



Naval Facilities Engineering Command Southwest
BRAC PMO West
San Diego, CA

DRAFT

Work Plan

Phase IV Non-Time Critical Removal Action, Solid Waste
Disposal Area Westside, Installation Restoration Site 12
Former Naval Station Treasure Island
San Francisco, CA
June 2020

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Acronyms and Abbreviations

²²⁶ Ra	radium-226
uR/hr	microroentgens per hour
AHA	activity hazard analysis
APP	Accident Prevention Plan
APTIM	Aptim Federal Services
ASTM	American Society for Testing and Materials
bgs	below ground surface
BMP	best management practice
BRAC	Base Realignment and Closure
CB&I	CB&I Federal Services
CCSF	City and County of San Francisco
CDPH	California Department of Public Health
COC	chemical of concern
COR	Contracting Officer Representative
CQCP	Contractor Quality Control Plan
CSO	Treasure Island Caretaker Site Office
DoD	U.S. Department of Defense
DCP	Dust Control Plan
DTSC	California Department of Toxic Substances Control
EPA	U.S. Environmental Protection Agency
ESS	Explosives Safety Submittal
Gilbane	Gilbane Federal
IR	Installation Restoration
LLRO	low-level radioactive object
LLRW	low-level radioactive waste
MDAS	material documented as safe
MEC	munitions and explosives of concern
MPPEH	material potentially presenting an explosive hazard
MRS	Munitions Response Site
Navy	U.S. Department of the Navy

NSTI	Naval Station Treasure Island
NTCRA	non-time critical removal action
OSHA	U.S. Occupational Health and Safety Administration
OTIE	Oneida Total Integrated Enterprises
PAH	polycyclic aromatic hydrocarbon
PCB	polychlorinated biphenyl
PCSR	post-construction summary report
PMO	Program Management Office
PPE	personal protective equipment
QA	quality assurance
QC	quality control
RASO	Radiological Affairs Support Office
RCA	radiologically controlled area
ROICC	Resident Officer in Charge of Construction
RPM	BRAC Remedial Project Manager
RPP	Radiation Protection Plan
RSY	radiological screening yard
SAP	Sampling and Analysis Plan
SSHO	Site Safety and Health Officer
SSHP	Site Safety and Health Plan
SWDA	solid waste disposal area
SWMP	Storm Water Management Plan
TIDA	Treasure Island Development Authority
TPH	total petroleum hydrocarbons
UFGS	United Facilities Guide Specification
USACE	U.S. Army Corps of Engineers
UXO	unexploded ordnance
UXOSO	Unexploded Ordnance Safety Officer
VOC	volatile organic compound
WMP	Waste Management Plan

1.0 Introduction

This work plan describes the implementation of the last phase of a non-time critical removal action (NTCRA), known as Phase IV, to excavate and dispose of total petroleum hydrocarbon (TPH)-contaminated soil from Solid Waste Disposal Area (SWDA) Westside in Installation Restoration (IR) Site 12 at the former Naval Station Treasure Island (NSTI) on Treasure Island in San Francisco, California (see **Figure 1**). This work plan was prepared by Gilbane Federal (Gilbane) for the U.S. Department of the Navy (Navy) Base Realignment and Closure (BRAC) Program Management Office (PMO) West under Radiological Environmental Multiple Award Contract N62473-17-D-0005, Contract Task Order N62473-18-F5271, with the Naval Facilities Engineering Command Southwest.

The Navy's decision to undertake an NTCRA at SWDA Westside, formerly known as SWDA A&B, is documented in the *Action Memorandum/Interim Remedial Action Plan: Non-Time Critical Removal Action for Solid Waste Disposal Areas Installation Restoration Site 12 Old Bunker Area Naval Station Treasure Island San Francisco, California* (Action Memorandum; Navy, 2007). The purpose of the NTCRA is to address potential human health risk to a resident or utility worker from direct contact with soil near the ground surface under the current land use and utility configuration. The NTCRA will reduce potential risks to human health by excavating and removing debris and contaminated soil from SWDA Westside. By doing this, the removal action will substantially eliminate identified pathways of exposure to hazardous substances for current and future residents and utility workers to the chemicals of concern (COCs). The COCs at IR Site 12 include dioxins, lead, polychlorinated biphenyls (PCBs), and polycyclic aromatic hydrocarbons (PAHs) in soil.

This work is being performed pursuant to the National Oil and Hazardous Substances Pollution Contingency Plan and in accordance with Navy guidance, based on the findings of potential exposure of current and future residents and utility workers to the COCs.

This Phase IV project is considered an extension of the Phase I, Phase II, and Phase III of the NTCRA. The Phase IV-specific objectives are to:

1. Remove existing soil stockpiles and radiological screening yard (RSY) pads that remain from the Phase III;
2. Excavate and dispose of TPH-contaminated soil;
3. Restore SWDA Westside areas excavated during Phase I and Phase III of the NTCRA to the pre-excavation grade (at which the adjacent housing is constructed); and
4. Release SWDA Westside from active radiological controls that exist at present.

The *Final Post-Construction Summary Report, Non-Time Critical Removal Action for Solid Waste Disposal Areas Westside Drive, Bayside Drive, and North Point Drive, Radiological Characterization, Building Demolition, and Remediation, Installation Restoration Site 12 (Phase III), Former Naval Station Treasure Island, San Francisco, California* (Aptim Federal Services [APTIM], 2019) documents existing soil stockpiles and RSY pads generated during the excavation of debris and contaminated soil in Phase III that remain in place. These will be removed prior to the excavation of additional debris and contaminated soil.

Alternative 3 of the Action Memorandum (Navy, 2007) calls for excavating soil from the common areas, roadways, and backyards to a depth of 4 feet below ground surface (bgs), or the grade at which the adjacent housing is constructed, and replacing it with clean fill. However, based on findings from Phase III (see **Section 2.1.4**) that encountered debris down to approximately 15 feet bgs, the excavations will extend vertically beyond the depth of 4 feet bgs and up to a maximum depth of 15 feet bgs as long as debris or contaminated soil, as indicated by visible TPH, is encountered. The excavations will extend laterally using the same indicators of debris and TPH-contaminated soil. Radiological and chemical data will be collected at the limits of the excavation to characterize the left-in-place conditions.

Following the removal of the debris and/or TPH-contaminated soil, SWDA Westside will be restored to its pre-excavation elevation and graded to implement proper drainage. Clean imported fill will be brought onto the site, compacted, and seeded to return the site to a safe and stable condition.

A radiological survey will be conducted and necessary actions taken to release SWDA Westside from active radiological controls. Radiological postings will be removed once concurrence is received from the Navy. The existing fence will remain. Passive controls such as groundcover, hardscape, and rental agreement house rules will remain in place.

1.1 Site Location and Description

The former NSTI is located on Treasure Island, which is a 403-acre man-made island located adjacent to Yerba Buena Island, in San Francisco Bay (see **Figure 1**). Treasure Island was constructed with sediments (hydraulic fill) dredged from the San Francisco Bay from 1936 to 1937 for the Golden Gate International Exposition of 1939 and 1940. In 1940, the Navy began leasing the island from the City and County of San Francisco (CCSF) and constructed NSTI. Later, during World War II, the Navy gained full ownership. In 1993, NSTI was designated for closure under the Base Realignment and Closure Act of 1990. It was closed on September 30, 1997.

1.1.1 IR Site 12

IR Site 12 is located on the northwest portion of the former NSTI on a relatively flat 93-acre area (see **Figure 1**). The site consists of multiplex housing units with private

backyards and common area front yards, side yards, and surrounding greenbelts. The area was originally used as a parking lot during the Golden Gate International Exposition of 1939 and 1940. After Navy occupation of the island in 1940, the area was used for bunker storage of munitions and other materials, vehicle and equipment storage, recreational playing fields, and disposal and burning of waste. Beginning in the 1960s, areas of IR Site 12 were incrementally developed into housing for Navy personnel and their dependents. Currently, IR Site 12 is proposed for reuse as residential, open space, and publicly-oriented uses.

1.1.2 SWDA Westside

SWDA Westside is an approximately 4.5-acre area on the west side of IR Site 12 abutting Perimeter Road (see **Figure 2**) which includes areas previously excavated and partially backfilled during earlier NTCRA phases. SWDA Westside has nine former residential buildings within its boundary:

- Buildings 1119, 1121, 1123, 1125, and 1127 around Lester Court;
- Building 1317 on Gateview Drive; and
- Buildings 1319, 1321, and 1323 facing Westside Drive and Perimeter Road

With the exception of Building 1317, which is still standing but unoccupied, these buildings were demolished during earlier NTCRA phases to facilitate the excavation of debris and contaminated soil. The buildings were radiologically surveyed and abated for asbestos and lead prior to their demolition. Building 1317 was subject to radiological survey of its interior.

- Phase I: Buildings 1123 and 1321
- Phase II: Buildings 1121 and 1323
- Phase III: Buildings 1119, 1125, and 1319
- Post-Phase III: Building 1127 (fire damaged and subsequently demolished)

Previous phases of the NTCRA found that debris and contaminated soil in SWDA Westside is co-located with low-level radioactive objects (LLROs) containing radium-226 (^{226}Ra ; e.g., foils, deck markers, instrument gauges, and metal fragments also referenced as radioactive “commodities/objects” in project documentation); and associated localized ^{226}Ra contaminated soil. Additionally, portions of SWDA Westside may contain discreet munitions-related items based on past findings. Those areas where munition-related items were encountered are identified as Munitions Response Site (MRS) 1 shown on **Figure 2**. Consequently, radiological and munitions screening of excavated debris and contaminated soil is required for disposal purposes and is incidental to the NTCRA.

1.2 Statutory Framework

As the lead federal agency, the Navy works with the California Department of Toxic Substances Control (DTSC), the San Francisco Regional Water Quality Control Board,

and other regulatory agencies under the terms of the 1992 Federal Facility Site Remediation Agreement. In addition, the California Department of Public Health (CDPH), specifically the Environmental Management Branch, works with DTSC to provide technical support on the radiological clean-up. Also within CDPH, the Radiologic Health Branch performs inspection, compliance, and enforcement related to Gilbane's State of California radioactive material license. Other public agencies and organizations also provide support to the environmental clean-up program, including the Treasure Island Development Authority (TIDA), and the Treasure Island Community Development.

Work activities will be coordinated with the BRAC Remedial Project Manager (RPM) and other appropriate Navy points of contact, including the Radiological Affairs Support Office (RASO), the Resident Officer in Charge of Construction (ROICC), and the Treasure Island Caretaker Site Office (CSO), to ensure proper work oversight. This includes identifying, establishing, and maintaining temporary facilities for the storage and handling of radioactive material in its possession.

Navy policy requires the contractor performing radiological work to maintain independent license authority. Gilbane possesses a current radioactive material license from the State of California (License No. 7948-07). The existing memorandum of understanding between Gilbane and other radioactive material licensees working at Treasure Island will be modified as appropriate to ensure proper interfacing of radioactive material handling responsibilities.

Munitions and explosives of concern (MEC) and material potentially presenting an explosive hazard (MPPEH) have been encountered during previous NTCRA phases. Therefore, munitions-related work will be conducted in accordance with the approved *Explosives Safety Submission, Phase IV Non-Time Critical Removal Action for Solid Waste Disposal Area Westside, Installation Restoration Site 12, Former Naval Station Treasure Island, San Francisco, California* (ESS; Gilbane, in preparation). The ESS will comply with U.S. Department of Defense (DoD) requirements and policies for the handling of MPPEH/MEC, and the certifying of material documented as safe (MDAS) by qualified unexploded ordnance (UXO) technicians. The ESS will be developed using guidance issued by the Naval Ordnance Safety and Security Activity and the DoD Explosives Safety Board.

1.3 Work Plan Organization

This work plan is organized into the following:

- **Section 1.0, Introduction** – introduces the objectives of the project, the site location and description, and the statutory framework of the project.
- **Section 2.0, Site Background** – describes the previous investigations and removal actions; site characteristics; and the source, nature, and extent of contamination.

- **Section 3.0, Non-Time Critical Removal Action Implementation** – describes the implementation of the NTCRA activities from work planning through final inspection and site close-out.
- **Section 4.0, Environmental Protection** – introduces measures that will be taken to manage storm water, and to monitor air and dust during execution of the work.
- **Section 5.0, Traffic Control** – describes the traffic control measures for the NTCRA.
- **Section 6.0, Health and Safety** – describes the health and safety measures that will be implemented for the NTCRA.
- **Section 7.0, Quality Control** – references the Contractor Quality Control Plan (CQCP) that will be implemented for quality control during the NTCRA.
- **Section 8.0, Post-Construction Summary Report** – describes the Post-Construction Summary Report (PCSR) that will be prepared after completion of the NTCRA to document the removal action.
- **Section 9.0, References** – includes a list of documents used to compile this work plan.
- **Figures** – includes figures referenced in the main text of this work plan.
- **Appendix A, Radiation Protection Plan** – addresses radiation protection activities such as personnel dosimetry, radiation monitoring, contamination control, air sampling, and respiratory protection.
- **Appendix B, Data Management Plan** – describes how environmental data will be named, stored, and managed.
- **Appendix C, Sampling and Analysis Plan** – details soil sampling requirements, analytical methods, and quality assurance/quality control procedures for implementation of the NTCRA.
- **Appendix D, Waste Management Plan** – describes anticipated procedures applicable to the generation, storage, characterization, transportation, and disposal of waste generated from the NTCRA activities.
- **Appendix E, Site Backfill and Grading Plan** – details how excavations will be backfilled and import fill placed to restore the site to its pre-excavation grade.
- **Appendix F, Environmental Protection Plan** – describes the measures that will be implemented to management storm water and monitor air and dust during work execution.
- **Appendix G, Traffic Plan** –addresses road closures and/or restrictions, routing, other relevant traffic disruptions, and controls for traffic into/out of SWDA Westside.

- **Appendix H, Contractor Quality Control Plan** – addresses lines of communication, technical review procedures, activity documentation, definable work features, quality control (QC) staff and their responsibilities, proposed outside organizations (vendors, subcontractors) and their responsibilities and reporting requirements, project inspection requirements, required submittals, and other procedures to be followed to ensure quality throughout the project.
- **Appendix I, Response to Comments** – includes the responses to regulatory comments on the draft work plan (provided in the final submittal only)

2.0 Site Background

This section presents a general description of previous investigations, removal actions, and the waste delineation investigation which form the basis of this NTCRA.

2.1 Previous Investigations and Removal Actions

Several investigations and removal actions have been conducted to delineate and remove contamination in the area of IR Site 12 that is included in the footprint covered by this work plan (i.e., SWDA Westside).

2.1.1 Previous Investigations and Interim Measures

In 1992 and 1995, several remedial investigations were conducted throughout IR Site 12 to identify suspect debris disposal areas. These investigations, which included aerial photograph interpretation and subsurface sampling and analysis, identified several areas of suspected debris disposal (i.e., SWDAs), one of which was SWDA Westside. In 2003, an investigation that included the excavation of nearly 600 exploration trenches, step-out trenches, and step-out hand auger locations was conducted to characterize contamination and to make decisions about further remedial efforts at IR Site 12. The results of the trenching investigation, documented in the *Final Data Summary Report, Site 12 Housing Area, Sitewide Investigation, Treasure Island, San Francisco, California* (Shaw Environmental, 2004) were used to further refine the boundaries of SWDA Westside.

In October 2000, the Navy worked with DTSC to develop a plan for interim measures in IR Site 12 around four identified SWDAs, including SWDA Westside, addressing solid waste, dioxin, lead, PCBs, PAHs, and soil gas sampling that showed localized soil contamination within the SWDAs. The interim measures are documented in the *Revised Engineering Evaluation and Cost Analysis, Solid Waste Disposal Areas, Installation Restoration Site 12, Old Bunker Storage Area, Naval Station Treasure Island, San Francisco, California* (SulTech, 2006). A remedial action objective was established to reduce the potential for human contact with chemically contaminated soil near the ground surface within the four SWDAs at IR Site 12 under the then-current land and utility configuration (i.e., current and future resident and utility worker exposure scenarios).

2.1.2 NTCRA Phase I

Known as Phase I of the NTCRA, portions of SWDA Westside were subject to removal actions which began in March 2007 and continued through September 2014. The results of these actions are documented in the *Final Post-Construction Summary Report, Installation Restoration Site 12, Solid Waste Disposal Area Westside, Naval Station Treasure Island, San Francisco, California* (CB&I Federal Services [CB&I], 2014). Soil was excavated to a pre-determined maximum depth of 4 feet bgs, and

extending to pre-planned excavation boundaries. Radiological screening for ^{226}Ra contaminated soil was performed, and excavation floor and sidewall soil samples were collected and analyzed for COCs and ^{226}Ra . Material potentially presenting an explosive hazard (MPPEH) was encountered during the soil excavation and screening process. Work had to be paused and secured in order for an ESS to be prepared and implemented during the removal of the remaining soil. Only a small portion of the excavated area was backfilled under Phase I. Additional excavation, radiological soil screening, radiological survey, munitions oversight and screening, and confirmation sampling were identified as needing to be completed during Phase III to address the specific areas with residual elevated sample results, and to fully address the remaining extent of the SWDA Westside footprint.

2.1.3 NTCRA Phase II

Phase II of the NTCRA included the demolition of two buildings (Buildings 1121 and 1323) at SWDA Westside, which is documented in the *Final Post Construction Summary Report for Solid Waste Disposal Area A&B, Non-Time Critical Removal Action Phase II, and Radiological Removal near Buildings 1128, 1303, and 1306, Former Naval Station Treasure Island, San Francisco, California* (TetraTech EC, 2014). Phase II did not include additional excavation at SWDA Westside. Rather, its focus was on activities primarily outside of SWDA Westside. Consequently, there was no progress toward the NTCRA goal of contaminated soil removal.

2.1.4 NTCRA Phase III

Phase III of the NTCRA (APTIM, 2019) was conducted beginning in August 2014 through May 2016. Its goal was to substantially eliminate the identified exposure pathways by removing chemically- and radiologically-contaminated material from SWDA Westside (and other SWDAs). Incorporated within its scope was to complete the unfinished activities from Phase I, including addressing the remaining areas identified with residual elevated sample results. Buildings 1119, 1125, and 1319 were radiologically surveyed, sampled, abated for asbestos and lead, and demolished. Excavation within the building footprints and other areas within SWDA Westside was performed to determine extent of debris and contamination and to complete remediation. Oily material was encountered southeast of the former Building 1123 footprint (see **Figure 2**). Excavation work was paused to re-evaluate best management practices (BMPs) for TPH removal under the water table and potential segregation of this waste. However, further removal did not continue under Phase III since the limits of the oily material were unknown.

As a result of the historical encounters of MPPEH at SWDA Westside during Phase I, Phase III was executed under the provisions of an ESS. MRS 1 (see **Figure 2**) was identified to include the portions of SWDA Westside where munitions-related items were likely to be encountered. These areas included the footprints of former Buildings 1123 and 1321 and the area east and northeast of Building 1321 (reference: *After Action*

Report, Non-Time Critical Removal Action for Solid Waste Disposal Areas Westside, Bayside, and North Point, Radiological Characterization, Building Demolition, and Remediation Installation Restoration Site 12 (Phase III), Former Naval Station Treasure Island, San Francisco, California [APTIM, 2018]). Excavated material was screened for MPPEH, munitions and explosives of concern (MEC), and LLROs. Large amounts of heavy industrial debris such as large cables and metal pieces such as vehicle bumpers were encountered down to approximately 15 feet bgs from an area at the northwest corner of former Building 1321 and removed. MPPEH and LLROs also were identified and recovered during the excavation.

Phase III NTCRA activities at SWDA Westside were halted in May 2016 following the discovery of two MEC objects that exceeded the limitations of the project ESS. As a result of safety limitations, the excavations were not completed. Instead, the work area was backfilled only enough to prevent surface water ponding, BMPs (e.g., straw waddles, privacy fencing, signage, etc.) were installed and contractor-owned equipment and materials were removed from the site. Additional excavation of debris and contaminated soil, radiological soil screening, radiological survey, munitions oversight and screening, and sampling were identified as needing to be completed during Phase IV, in addition to screening and disposing of existing Phase III stockpiled soil and RSY pads.

2.1.5 Waste Delineation Investigation

In 2016 during Phase III NTCRA activities, the Navy refined the conceptual site model for SWDA Westside after excavation activities revealed solid waste extending down to a depth of approximately 15 feet bgs in an area near former Building 1321. Although the previous conceptual site model assumed debris did not extend much deeper than 4 feet bgs, these findings are consistent with historic documentation of this area being a primary disposal area during early Navy operations.

In late 2017, an investigation into the extent of waste, both horizontally and vertically, of the entire SWDA Westside was launched. Its results are documented in the *Final Technical Memorandum: Investigation of Lateral and Vertical Extent of Solid Waste Solid Waste Disposal Areas (SWDAs) Westside, Bayside, and North Point Former Naval Station Treasure Island San Francisco, California* (Oneida Total Integrated Enterprises [OTIE], 2018). A series of borings were completed, ranging in depth from 2.5 to 19 feet bgs. These borings generally were located around the periphery of previous removals at the southern end of SWDA Westside. Visible debris was observed to a maximum depth of 17 feet bgs. Several borings showed evidence of petroleum/solvent impacts in addition to approximately 5% or less debris. Approximately one-third of the borings contained less than 10% visible inert debris greater than 1 foot bgs (but less than 5 feet bgs) and were interpreted to have been placed as a result of grading operations for construction of the Navy housing in IR Site 12. Typical debris encountered included concrete and asphalt, ceramic dishware, rusty nails/bolts, metal debris, and other common construction materials. Investigation results indicate that

much of the SWDA Westside vicinity has been affected by inert debris burial and/or redistribution by grading. Several borings had little to no debris, indicating no further removal actions are required.

2.2 Site Characteristics

The following describes the site characteristics at Treasure Island based on information from the Action Memorandum (Navy, 2007).

2.2.1 Land Use

Portions of IR Site 12 are leased to TIDA, who subsequently subleases select housing units. Currently, IR Site 12 contains residential buildings (about 900 housing units) that are two-story structures constructed with slab-on-grade foundations with four to eight residential units per building. Buildings within the subject construction area were not subject to lease.

Following environmental restoration of the site, the entirety of IR Site 12 will be transferred to the CCSF for residential/open space/publicly oriented uses. The surrounding land use includes various commercial/industrial uses.

2.2.2 Geology/Soil

Treasure Island is a relatively flat man-made island, consisting primarily of sand dredged from the San Francisco Bay and retained by a perimeter of rock and sand dikes. Treasure Island was constructed on the Yerba Buena Shoals, a sand spit extending north and northwest of Yerba Buena Island. Treasure Island ranges in elevation from 9 to 12 feet above mean sea level. Subsurface materials at Treasure Island are composed of dredged sand fill, Yerba Buena shoal sands, and Bay mud.

2.2.3 Hydrogeology

The estimated depth to groundwater at IR Site 12 ranges from about 2.5 to 7.5 feet bgs. The water table is unconfined. The groundwater flow is radial from the center of Treasure Island toward the shoreline. Groundwater recharge occurs primarily from infiltration of precipitation, with some contribution from landscape irrigation. Perched groundwater conditions may exist locally above the shallow water table because of the presence of relatively impermeable silt and clay lenses.

2.2.4 Climate and Meteorology

The climate on Treasure Island is dominated by the Pacific Ocean, which produces a maritime climate characterized by little temperature variation. The average annual temperature is 56 to 58 degrees Fahrenheit, with an annual frost-free period ranging from 300 to 330 days.

The prevailing wind direction for the San Francisco Bay Area is from the northwest. Wind speed is less than 6 miles per hour for more than 50 percent of the time and exceeds 12 miles per hour for approximately 10 percent of the time. The strongest winds are associated with winter storms. In the winter, winds from the north and east sometimes bring low temperatures to the San Francisco Bay Area. Westerly winds predominate during the summer, when cool marine air flows east toward the warm Central Valley region of California. These winds are strongest in the late afternoon and early evening.

The average annual precipitation is about 25 to 30 inches. Approximately 90 percent of the annual precipitation occurs from November to April. Localized showers are infrequent, and storms are moderate in duration and intensity. Mean annual evaporation is 48 inches. The greatest evaporation occurs during July.

Relative humidity during the winter is approximately 50 to 60 percent during the day, increasing to approximately 80 to 90 percent at night. Humidity decreases in spring; however, by summer, it increases, particularly at night or in the morning, when frequent fogs occur. Humidity is lowest in the fall, ranging from approximately 50 percent during the day to 70 percent at night.

2.3 Source, Nature, and Extent of Contamination

The NTCRA was implemented to remediate chemicals in soil associated with chemical/fuel storage and disposal or burning of waste in SWDAs on the western portion of IR Site 12. In 2017, the Navy performed an investigation to delineate the horizontal and vertical extent of contamination associated with buried waste and debris at SWDA Westside (OTIE, 2018). The results indicated several areas of varying subsurface conditions and contamination characteristics, including the following: (1) greater than 10% debris, (2) debris deeper than 5 feet bgs relative to normal grade, (3) evidence of chemical impacts, or (4) radiological impact. Boring results identified waste debris at depths ranging from 12 to 15 feet bgs. Further removal of the saturated soil will eliminate a potential source of surface water contamination. Removal of debris and other materials is a coincidental effect of this phase of work. Results from this last phase of the NTCRA will be used to develop the IR Site 12 Feasibility Study.

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3.0 Non-Time Critical Removal Action Implementation

The Phase IV NTCRA consists of three primary components:

- Pre-Excavation Activities – activities performed prior to and in preparation for excavation, including work planning; mobilization and site set-up; field support activities, screening of existing Phase III stockpiled soil and RSY pads; site clearing, grubbing, debris removal, and grading; and construction of RSY replacement (or laydown) pads and soil storage areas. Pre-excavation activities are described in **Section 3.1** through **Section 3.6**.
- Excavation Activities – activities performed during excavation, including excavation of debris and TPH contaminated soil, post-excavation sampling for COCs, backfill of excavated areas, and screening of excavated soil and transport for offsite disposal. Excavation activities are described in **Section 3.7** through **Section 3.9**.
- Post-Excavation Activities – activities performed following the completion of excavation, including site restoration, site radiological survey, and final inspection/site close-out. Post-excavation activities are described in **Section 3.10** through **Section 3.12**.

The project schedule is provided in **Figure 3**.

3.1 Work Planning

Work planning includes preparing work plans, obtaining appropriate authorizations, making notifications, and conducting community outreach.

3.1.1 Work Plan Preparation

This work plan presents the overall approach, requirements, and methods for executing the work and achieving the project objectives. Supporting documents include the stand-alone ESS (Gilbane, in preparation) and the *Site Safety and Health Plan, Phase IV Non-Time Critical Removal Action for Solid Waste Disposal Area Westside, Installation Restoration Site 12, Former Naval Station Treasure Island, San Francisco, California* (SSHP; Gilbane, 2020a). Additional supporting documents are included as appendices. Together, the supporting documents are designed to protect workers, the public, and the environment.

Once approved by the Navy and regulatory agencies, changes to this work plan and/or its supporting documents will require a field change request, which may include both text and figures based on the nature of the plan change. The Navy, with technical assistance from Gilbane, will coordinate field change requests with the regulatory agencies, as required, and provide notification of any other additional requirements to facilitate regulatory agency coordination.

3.1.2 Authorizations and Notifications

Appropriate authorizations will be obtained and notifications made prior to mobilization. The Navy complies with the substantive requirements of applicable and relevant permits. Necessary authorizations will be obtained from the ROICC and the CSO for implementing and completing the work. The appropriate Navy personnel, including the RPM, RASO, and the CSO will be notified regarding the planned schedule prior to mobilization. On-site activities (i.e., personnel and equipment mobilization) will begin once notice to proceed is received from the Navy.

An annual excavation permit from the California Occupational Safety and Health Administration is maintained by Gilbane and required notifications will be made before excavation begins. A dig permit will be submitted and the San Francisco Public Utilities Commission will be notified as appropriate before excavation work begins. Permits will be obtained, as needed, for connections to necessary services provided by utility companies serving the project.

3.1.3 Community Outreach

Community outreach will be conducted to keep the public informed of activities related to the removal action. Regular updates will be provided to the Navy on the progress of field work at a frequency and in such a manner as to foster Navy communication with its stakeholders. Communications with parties outside of the Navy will be coordinated through the Navy.

Work notices will be prepared in English, Spanish, and Cantonese and distributed door-to-door to the IR Site 12 residents in the vicinity of SWDA Westside to make them aware of the nature of work, exact location, and time constraints. Extraordinary work-arounds (i.e., non-routine work evolutions impacting island residents) caused by utility disruptions, severe weather, and other events will be coordinated with the Navy to minimize their impact on island residents. Environmental monitoring data (air and dust) will be made available to the public via the BRAC website.

3.2 Mobilization and Site Set-Up

Mobilization and site set-up include establishing temporary facilities, instituting site and work area security, completing a pre-construction environmental survey, completing an underground utility clearance, decommissioning monitoring wells, if necessary, and setting up munitions-related engineering controls.

3.2.1 Temporary Facilities

Temporary field facilities will be established at Building 570 (see **Figure 1**) and the associated secured fenced area will be used as a material and equipment storage area. A shipping container, if needed, will be staged at SWDA Westside as a working space and for storage of tools, small equipment, and materials. Private vehicles of workers will

be parked around Building 570 and only Gilbane and subcontractor vehicles will be used at work areas. Residential parking areas will be avoided.

3.2.2 Pre-Construction Environmental Survey

Prior to starting work, a pre-construction environmental survey will be performed with the Contracting Officer Representative (COR), CSO, and Field Safety Navy Technical Representative. Photos will be taken showing existing environmental conditions on and adjacent to the site. The results of the survey will be submitted in accordance with the applicable Contract Data Requirements List.

3.2.3 Utility Clearance

Underground utility clearance will be completed before intrusive activities are initiated. It includes:

- Review of utility maps, including historical maps from previous work;
- Use of geophysical methods, such as electromagnetic induction, magnetometry, and ground-penetrating radar, to clear the proposed limit of intrusive activity of potential subsurface obstructions prior to soil excavation or saw-cutting;
- Marking of the proposed limits of intrusive activity and the utility lines in the immediate vicinity, using color-coded surveyor paint;
- Notifying Underground Service Alert North; and
- Scheduling a meeting with interested parties (e.g., affected residents, utility providers) that may be affected by excavation activities.

Utilities will be identified, excavated around to the extent possible, and supported or shored, as required, to ensure integrity.

3.2.4 Monitoring Well Decommissioning

Existing groundwater monitoring wells are located in SWDA Westside on the edges of areas to be excavated (e.g., 12-MW08 and 12-MW30; see **Figure 2**). Efforts will be made to preserve and protect the wells to the extent possible with excavation activities limited to no closer than 5 feet from the well. The monitoring wells will be marked and access to them retained during site grading and backfilling activities. The monitoring wells will be evaluated as the excavation progresses. In the event they are damaged or compromised, they will be decommissioned and replaced in consultation with the Navy.

If needed, well decommissioning (and replacement) activities will be performed by a California-licensed drilling subcontractor in accordance with California Department of Water Resources regulations. The wells will be destroyed by extracting the casings and screens. A hollow-stem auger will be used to drill out the annular materials from the ground surface to the bottom of the excavation. The remaining well casing either will be

grouted or removed below the base of the excavation. The wells will be replaced in coordination with the Navy following appropriate permitting procedures. Once the excavation is backfilled, the replacement well will be installed in the same location. The well will be installed using a hollow-stem auger drill rig and will be completed using Schedule 40 polyvinyl chloride casing and screens. Screen slots will be machine cut to 0.01-inch to 0.02-inch sized openings. Wells will be constructed with screen at the same depth intervals as the original wells. The sand pack at each well will consist of #2/12 or #2/16 sized sand from the bottom of each borehole to approximately 1 foot above the screened interval. The sand will be topped with bentonite chips that are allowed to hydrate using either formation water or potable water for one hour. The well will be completed as a surface flush mount and developed no sooner than 48 hours after well construction is completed.

3.2.5 Munitions-Related Engineering Controls

Munitions-related engineering controls will be instituted in accordance with the ESS (Gilbane, in preparation). Prior to conducting any munitions-related field work, empty metal shipping containers (e.g., Sealand containers or Conex boxes) will be positioned and used as blast barriers to mitigate the potential explosive hazard (see example illustration in **Figure 4**). The shipping containers will be placed in a linear fashion and abutted in a protective manner around the proposed munitions-related field work boundaries. The actual positioning of the shipping containers as barriers will be established based on where the munitions-related work is occurring within the site. This approach essentially reduces the exclusion zone (see **Section 3.3.2**) to within the boundaries formed by the shipping containers. Additionally, the adjacent Perimeter Road and Gateview Avenue will be closed to public vehicular traffic and recreational use and monitored respectively during business hours and/or ground disturbing activities. See **Figure 4** for road closure and entry control point locations.

Mechanized equipment will be shielded using no less than 0.51 inches of acrylic sheet (e.g., PlexiGlas) to provide full frontal fragmentation penetration protection for equipment operators. This will provide full fragmentation shielding during excavation and soil movement activities.

3.3 Field Support Activities

Field support activities include site security, explosives safety, UXO construction support, radiation protection, mitigating odor impacts, site housekeeping, and data management. Radiological controls, dust mitigation, air monitoring, and other measures appropriate to mitigate potential contamination will be instituted and maintained during soil excavation and handling activities.

3.3.1 Site Security

Security will be provided for both workers and equipment and material before, during, and after working hours. Six-foot high fencing with privacy fabric will be installed around

work areas, including excavations and equipment laydown and storage areas where needed and not presenting existing. As a general rule, material and equipment in danger of theft will be stored inside the secured fenced area. After-hours security will be provided by a contract security company.

3.3.2 Explosives Safety

Explosives safety measures will be instituted in accordance with the ESS (Gilbane, in preparation). The ESS is designed to provide the maximum possible protection to people and property considering past findings of MPPEH/MEC at the site and its close proximity to occupied housing and public areas. A trained and qualified UXO Safety Officer (UXOSO) will be dedicated to the project whose sole responsibility will be to establish and ensure compliance with site-specific explosives safety requirements.

An exclusion zone will be in place while excavation and UXO clearance activities involving munitions-impacted soil are being performed. Operational areas will be fenced and personnel entry will be controlled. Access to the exclusion zone will be limited to personnel essential to the operation being conducted and authorized visitors on a case-by-case basis, granted access by the UXOSO or a qualified designee. Based on the risk posed by the munitions response operation underway, the UXOSO may determine that access to the exclusion zone is unsafe for visitors.

3.3.3 UXO Construction Support

As a precaution, UXO personnel will observe munitions-impacted soil handling activities and respond should suspected MPPEH/MEC be encountered. UXO technicians will observe operations from behind a 0.51-inch acrylic sheet barrier and double hearing protection will be used during excavation. A minimum distance of 12 feet (or other appropriate distance specified in the ESS) will be maintained between the excavation and the observer, as well as the distance from the excavator operator. The UXO team will be in constant communication with equipment operators during the handling of munitions-impacted soil.

Roads, parking lots, and public access points that are affected by operations and the exclusion zone will be monitored by UXO personnel and will be marked and/or manned according to the ESS (Gilbane, in preparation). In the event of an emergency, the Navy will be notified and will coordinate public notification and emergency response actions. High traffic areas will be controlled by flaggers and stationary blockades and barriers with signage and contact information.

3.3.4 Radiation Protection

Radiological controls will be instituted in accordance with the Radiation Protection Plan (RPP) provided in **Appendix A**. The RPP addresses radiation protection activities such as personnel dosimetry, radiation monitoring, contamination control, air sampling, and respiratory protection, as well as measures to maintain exposures to radiation and

radioactive material as low as reasonably achievable. Personnel surveys, equipment and material surveys, and decontamination are also addressed in the RPP. Corporate-level radiation safety procedures provide controls necessary for radiologically safe operations that will be followed for implementation of field work.

A trained and qualified Project/Site Radiation Safety Officer will ensure that radiological hazards are identified during work planning, and that appropriate controls are implemented to maintain worker exposure to these hazards.

Existing fencing and radiological postings at SWDA Westside will be maintained for the duration of the project. A series of routine radiological surveys will be performed. Weekly surveys of the SWDA Westside radiologically controlled area (RCA) boundary will be performed. Office areas, break rooms, restrooms/portable toilets, etc., used by workers who work within the RCA will be surveyed as least monthly. Quarterly surveys of radioactive sources and work vehicles will be performed per the RPP. Surveys will be documented and available on-site for inspection.

Work activities within an RCA will be conducted in accordance with a radiation work permit that details the radiological requirements and protective measures to be applied to the job. Trucks, machinery, and equipment will be radiologically surveyed prior to initial use and upon release from the project or before leaving the site.

3.3.5 Mitigating Odor Impacts

It is anticipated that TPH will be encountered as free product and dissolved phases in soil and groundwater. If encountered, TPH impacted soil may produce significant petroleum odors. Odor mitigation will be performed in tandem with dust control measures. Engineering controls, such as the use of physical barriers, oil skimmers, absorbent booms/pads, aerosol misting systems, soil applications, soil sealants, or similar measures will be taken to limit the odor nuisance to the nearby residents and members of the public that could be impacted by the excavation and removal activities. Coordination with TIDA and Treasure Island housing managers may be required to facilitate action to minimize impacts on local residents and the public.

During excavation activities, misting around the perimeter of the work area will be performed to neutralize odors from exposed vapor-containing materials. The odor control system uses high-pressure fog lines that will be set atop fences or shipping containers around the work area perimeter. An odor neutralizing compound (e.g., ODOR ARMOR) mixed with pressurized water will be used to create a fine fog that works to eliminate the odor through a series of chemical reactions. A surface capping material (e.g., Gorilla Snot) will be used to seal stockpiled material. Photoionization detector (or equivalent) measurements will be performed in ambient air for organic compound emissions during excavation activities, and odor control activities will be modified accordingly.

3.3.6 Housekeeping

Work areas will be kept tidy and free from litter or trash and material and equipment stored in designated locations. Routine clean-up will be performed as needed (but weekly at a minimum) to keep the buildings, site, and adjacent properties free from accumulations of waste materials, rubbish, and windblown debris. Sidewalks and streets, especially common areas, affected by the work will be swept clean and returned to a condition that is acceptable to the Navy.

Routine inspections of SWDA Westside will be performed to ensure fencing around the RCA is maintained and repaired as needed, and storm water controls are in place and effective. These activities will include fence panel repair, locks (as needed), placement and maintenance of straw waddles or equivalent measures to prevent storm water runoff from SWDA Westside.

3.3.7 Data Management

Data management will be performed in accordance with the Data Management Plan provided in **Appendix B**, which describes how environmental data will be named, stored, and managed. The plan addresses the type of database to be used, software programs, sample tracking, and how the data will be analyzed and displayed. In addition, the plan elaborates on the proposed method to efficiently manage data and information collected from all phases of the work process from the initial excavation, soil screening, laboratory analysis, backfilling, and reporting. Data will be reported to the Navy on a routine (daily) basis as it is generated and field activities are completed.

3.4 Screening of Existing Phase III Stockpiled Soil and RSY Pads

Approximately 3,300 cubic yards of stockpiled soil and RSY pads remain on-site following the Phase III NTCRA work (APTIM, 2019). Off-site disposal was not completed due to the halting of munitions screening following the discovery of MEC not addressed in the ESS. Therefore, this material is being addressed under Phase IV. The estimated volumes of the stockpiled soil and RSY pads are listed in Exhibit 1.

Exhibit 1: Stockpiled Phase III Soil and RSY Pads Requiring Screening

Phase III Material	Location Description ^a	Estimated Volume (cubic yards)
Stockpiled Soil ^b	RSY 1	1,310
	RSY 3	842
	Stockpiles 1, 2, and 3	956
	Stockpile 4	55
RSY Pads ^c	RSY 1	150
	RSY 3	150

Notes:

^a refer to **Figure 4** for approximate locations

^b requires munitions screening

^c requires radiological screening

The stockpiled soil was radiologically cleared by RASO for disposal as other than low-level radioactive waste (LLRW) during Phase III, though munitions screening is still required. The RSY pads require radiological screening, but not munitions screening. Once Navy concurrence is received, the screened material will be transported offsite for disposal.

3.4.1 Munitions Screening

Munitions screening will be performed through the use of visual inspection and hand held detectors such as Schonstedt GA-52Cx Magnetic Locator and/or Whites DXF-300 All Metals Locator in accordance with the ESS (Gilbane, in preparation). The soil will be cleared in lifts no thicker than 10 inches to ensure a measurable response with the hand held instruments.

Debris separated from the soil, including dirt and clay clumps, will be visually inspected for MPPEH/MEC and checked using the Schonstedt GA-52Cx (or equivalent) if metallic debris is indicated. Larger debris that could mask or hide MPPEH/MEC (e.g., pipes, hoses, containers) will be mechanically separated (e.g., using a wet saw) and classified as MDAS so that the contents can be inspected to ensure there are no hazardous components. If determined safe to move, the MPPEH/MEC will be manually removed and hand carried to the temporary safe holding area or the intentional consolidated shot detonation area for disposition.

Recovered MPPEH/MEC will be securely stored and dispositioned in accordance to the ESS so not to be accidentally comingled with MDAS. Stockpiled soil cleared as MDAS will be labeled or posted as such. MPPEH/MEC and related debris will be logged and tracked from origin to final disposition. Any MPPEH/MEC identified will be logged into the project database via a smart phone data collection application. The data will include origin, type, status, depth, storage and disposition. It also will include information pertaining to demolition activities of MPPEH/MEC (see ESS [Gilbane, in preparation]).

3.4.2 Radiological Screening

Radiological screening will be performed to identify and remove any potential LLROs. The soil will be laid out and screened in approximately 9-inch thick lifts. The radiological screening will be performed using a Radiation Solutions, Inc. RS-700 self-contained mobile gamma-ray detection system. The RS-700 consists of a digital gamma-ray spectrometer/multi-channel analyzer coupled to a sodium iodide gamma scintillation detector. The RS-700 system is mounted on a cart pulled behind a small tractor. The detectors are mounted at a height of 4 inches above the surface and moved over the surface at a speed of 18 inches per second, with each pass overlapping the previous pass to assure 100-percent coverage of the area being surveyed. Changes to the scan height or scan rate may be made to improve detection response if necessary to accommodate field conditions, such as soil composition and moisture content.

The RS-700 automatically captures gamma scan data at one-second intervals and position-correlates the data by means of a global positioning system mounted to the unit. Pre-set regions of interest within the energy spectrum programmed into the RS-700 will be used to identify and track specific gamma-ray emissions associated with the gamma-emitting progeny of ^{226}Ra . The RS-700 data will be used to aid in field investigations of radiation anomalies as well as to facilitate the selection of biased soil sample locations.

Recovered LLROs will be removed and stored for disposal as LLRW. Waste information will be compiled for each LLRO to detail the analytical information about the source to include photographs of the source, radionuclide identification, estimated curie content, and radiological survey information.

A minimum of 20 samples will be collected for each 600 cubic yards of soil. Sample results will be used to confirm the soil can be disposed as non-LLRW. The samples will be collected in a random-start systematic pattern across each 9-inch thick lift of soil spread out over an area of approximately 21,500 square feet. Up to five samples also will be collected at biased locations identified for further investigation by radiological screening.

3.4.3 Sampling and Analysis

The focus of the excavation is driven by visual evidence of debris and TPH-contaminated soil. However, post-excavation and waste characterization samples will be collected and analyzed for COCs and/or ^{226}Ra . Sampling and analysis will be performed according to the Sampling and Analysis Plan (SAP) provided in **Appendix C**. The SAP, prepared in the *Uniform Federal Policy for Quality Assurance Project Plans* (EPA et al, 2005) format, describes how sampling will be performed and details the laboratory operations that will support the sampling activities, including checks to ensure the quality of laboratory work performed. The SAP includes data quality objectives for sampling and analysis activities and provides guidelines for data evaluation.

The SAP describes the sample collection methodology to be used. Each sample will be labeled and assigned a unique sample identification number. Samples will be turned over to a laboratory accredited under the DoD Environmental Laboratory Accreditation Program and the California State Environmental Laboratory Accreditation Program, using proper chain-of-custody procedure. Samples will be prepared and analyzed for COCs and/or ^{226}Ra using accepted methods listed in the SAP.

3.4.4 Data Package

A data package will be prepared for radiologically screened soil once survey and sampling activities are complete and the resulting data are assessed. Data packages are designed as stand-alone documents to allow independent review and verification of survey results. As a minimum, they will include radiological screening data and soil

sample analytical results. Data packages will be provided to the Navy for review and concurrence prior to soil disposal.

3.4.5 Waste Management

Once screened for munitions and LLROs, excavated soil and other waste material will be handled, stored, and disposed of in a manner protective of human health and the environment and in accordance with applicable regulatory requirements as described in the Waste Management Plan (see **Appendix D**). The WMP outlines the day-to-day management of waste materials in support of the removal action, including storage and transportation of LLRW. The WMP covers both removal action and investigation-derived waste, for example: personal protective equipment (PPE), plastic sheeting, decontamination water, disposable sampling equipment and other assorted waste such as TPH free product (floating petroleum) that may be generated during this project. Samples will be collected as necessary to characterize the waste for disposal.

3.5 Site Clearing, Grubbing, Debris Removal, and Grading

Areas of SWDA Westside outside of the designated excavation areas will be cleared of vegetation and debris, grubbed, and graded in preparation for the construction of laydown pads, storage areas, and transport paths across the SWDA.

Chain saws, power trimmers, pole saws, and other manually operated cutting tools in addition to heavy equipment may be used to cut vegetation. Shrubs/bushes will be removed using appropriately sized construction equipment (e.g., backhoe, loader, and/or excavator) by pulling them from the ground and/or digging them out. Large shrubs and trees will be cut down using saws with smaller vegetation removed by blade scraping. Mature trees and shrubbery will be left in place whenever possible.

Remnant roots will be loaded into dump trucks and transported to a waste soil/debris staging area for off-site disposal with the associated contaminated soil. The main body of the vegetation also may be loaded into dump trucks and transported to the waste soil/debris staging area for eventual off-site disposal. The wood and larger organic debris will be sampled and analyzed for radioactive material uptake prior to disposal.

3.6 Constructing Laydown Pads and Storage Areas

Laydown pads and storage areas will be constructed to handle the excavated soil for screening and storage prior to disposal.

3.6.1 Laydown Pads

Up to five laydown pads, each approximately 21,500 square feet in size, will be constructed for use in screening excavated soil. Each laydown pad will be constructed with a single 10-mil layer of high-density polyethylene or polyvinyl chloride liner placed directly on the ground surface and covered with an approximately 6-inch sacrificial layer

of clean imported soil to protect the liner during pad loading and unloading activities. Berms will be constructed around the perimeter of the laydown pad using hay bales or straw wattles and lined to form a containment area. This area will prevent run-on/off during precipitation events. A radiological survey of the constructed pad will be performed prior to use to establish baseline radiological conditions and to assure that there are no radiation anomalies (i.e., irregularities) that may impact its use. The survey results will be submitted to RASO for review and concurrence prior to use.

3.6.2 Stockpile Areas

Stockpile areas to be used for storage of soil that has been screened and cleared of munitions and LLROs will be constructed using 10-mil liner placed on the ground surface and wrapped and secured with hay bales or straw wattles. A layer of soil will be laid down on top of the liner to protect it during stockpiling activities. Until Navy concurrence on soil disposition is received, stockpiled soil will remain separate and segregated (i.e., each stockpile will consist of soil from a single screening on a given laydown pad) from other screened soil to retain data integrity and ensure there is no cross-contamination. Environmental protection measures will be implemented and maintained while the soil is stockpiled.

3.7 Soil Excavation

The results of the SWDA Westside waste delineation investigation (OTIE, 2018) form the basis for the two designated areas to be excavated and their depths (see **Figure 2**). Excavation activities in both the north and south excavation areas will begin where the highest TPH contamination is anticipated. Soil will be excavated within the designated excavation areas that are within the fenced area of SWDA Westside. The decision criteria for the limits of soil removal are based on visual evidence of TPH free- product or debris during the excavation; and not by a pre-determined clean-up goal or confirmation sampling.

An excavator will be used to advance the excavation to remove debris and visually stained TPH-impacted soil encountered within the excavation area. Soil will be removed in 1-foot deep lifts to aid in systematic visual inspections for debris and MPPEH/MEC. The excavation area will be initially excavated to approximately 4 feet bgs, which is nominally just above the water table and will be the working level at which the excavator will dig out the remaining TPH-contaminated soil as needed. The limits of the excavation will continue laterally and vertically under the direction of the project engineer until TPH free product is no longer observed, or to a maximum depth of 15 feet bgs, or until Bay mud is encountered.

The width and depth of the excavation will be monitored by a designated competent person, as defined by 29 CFR 1926 in order to maintain sidewall integrity in accordance with U.S. Occupational Safety and Health Administration (OSHA) guidelines. Daily inspection documentation for inclusion in the daily report will be generated by the

project engineer. No worker entry into the excavation will be allowed. To minimize soil sloughing, the excavation will be backfilled as soon as possible once post-excavation characterization samples are collected (see **Section 3.7.4**).

When operating within fine-grained saturated soils and shallow groundwater, metal plates, swamp mats, and long-reach excavators may be used as necessary to minimize the impact of localized liquefaction and cross-contamination through downward migration.

Where appropriate, a perforated excavator bucket will be used and the excavated soil will be placed outside, but immediately adjacent to, the excavation to allow water to drain from the saturated soil back into the excavation. The soil will then be loaded and transported to a laydown pad for screening for munitions and LLROs. If suitable conditions exist, the excavated soil will be cleared for UXO outside the excavation and prior to transport to a laydown pad.

A UXO technician will act as a spotter and look for any munitions-related debris encountered during excavation activities, guide excavation equipment in the vicinity of identified utilities, and watch for buried utilities as soil is removed. If the excavator operator or the spotter technician recognizes suspect MPPEH/MEC, all work will stop and the operator will request assistance from the UXO technician to properly identify any potential munitions item. Precautions to protect existing utilities will be implemented as necessary during excavation.

3.7.1 Debris Retrieval, Inspection, and Segregation

The excavator operator will use the excavator bucket to pick out and remove larger debris from the excavation, if encountered. The debris will be placed outside of the excavation area to undergo MPPEH/MEC inspection and LLRO screening. A team of UXO and radiation safety technicians will perform debris inspection and segregation. Debris will be sorted by waste type (e.g., hazardous, non-hazardous, MPPEH/MEC, MDAS, or recyclable) and disposed of appropriately.

3.7.2 Handling Groundwater

Since tidally-influenced groundwater is anticipated at approximately 5 feet bgs and the proposed excavation depths are up to a maximum of 15 feet bgs, dewatering methods to prevent water intrusion into the excavation are not practical. The use of a perforated excavator bucket will allow excess water to drain from the saturated soil prior to stockpiling. To aid in dewatering, the excavated soil will be spread using heavy equipment prior to the material being transported to and placed on a laydown pad for screening. Any excess water will be directed back into the open excavation or will evaporate as the soil dries; thus, no liquid collection or disposal is anticipated.

3.7.3 Handling TPH Free Product

TPH is anticipated to be present in the form of chemically impacted soil and as free product at the surface of the groundwater interface within the excavation. Prior to backfilling an excavation, visible free product will be removed from the groundwater interface. Excavations will not remain open solely for the purpose of removing free product. In areas where higher concentrations of TPH are encountered (e.g., sludge or emulsion), a water pump will be used to collect free-phase (non-aqueous) liquid or TPH-contaminated water whenever there is significant accumulation present, including any accumulation prior to commencing removals at the beginning of each day. This will minimize contaminant migration during the excavation and ascertain whether TPH-contaminated soil is present. In areas where lower concentrations of TPH are encountered (e.g., sheen or discoloration on surface of water), oil-absorbent booms or soil berms/barriers will be constructed to collect and restrict movement of water entering the open excavation.

Removed contaminated water will be held in temporary tanks for characterization prior to proper final management. Contained water will be sampled and analyzed in accordance with the WMP (see **Appendix D**).

3.7.4 Post-Excavation Sampling

Soil samples will be collected and analyzed for COCs and ²²⁶Ra to characterize the left-in-place chemical and radiological conditions (see **Section 3.4.3**) at the horizontal and vertical extent of the debris/soil staining removal. The sample locations and depths will be recorded as part of the sampling documentation. The excavation floor will be sampled at a frequency of one sample per 2,500 square feet. In other words, one sample will be collected along the bottom of one or more excavations whose total floor surface area equals 2,500 square feet. In addition, the excavation sidewalls will be sampled at a frequency of one sample per 50 linear feet. In other words, one sample will be collected along the sidewall of one or more excavations that form the lateral excavation boundary whose total length equals 50 linear feet. If the excavation depth is less than 7 feet bgs, one discrete soil sample will be collected along the sidewall. If the excavation depth is equal to or greater than 7 feet bgs, then two discrete soil samples will be collected along the sidewall – one at less than 7 feet bgs and one at more than 7 feet bgs.

3.8 Backfilling

Each excavation will be backfilled once the removal of debris and TPH contaminated soil has been visually verified by the project engineer, post-excavation sampling performed, and a land survey of the excavation boundaries has been completed by a California-registered land surveyor. Backfilling will be performed in accordance with the Site Backfill and Grading Plan (see **Appendix E**). Where groundwater is encountered, crushed rock/gravel will be placed as bridging material. Self-compacting bridging sand

may be used to help control the excavation and facilitate backfilling. Geotextile fabric will be used where appropriate to stabilize the transition from the crushed rock/gravel to bridging sand. Backfill material will meet the standards described in the *Information Advisory Clean Impacted Fill* (DTSC, 2001). The fill material will be placed into the excavation and compacted up to the ground surface. The fill material will be compatible with the existing material and allow for adequate compaction (90% standard proctor) for areas above and below the water table. Recycled materials such as crushed concrete (3-inch minus) may be used for backfill in deeper fills, as appropriate.

3.9 Screening of Excavated Soil

During the removal process for TPH-contaminated soil, MPPEH/MEC, LLROs, construction debris, and other types of landfill waste may be encountered and/or comingled within the contaminated soil. Excavated soil will be moved from the excavation to a laydown pad, spread out approximately 9 inches thick, and radiologically screened in accordance with **Section 3.4.2**. Munitions-impacted soil excavated within MRS 1 will be screened for munitions in accordance with **Section 3.4.1**, and released as MDAS prior to radiological screening. UXO clearance of munitions-impacted soil may be performed at the excavation or once the soil is moved to a laydown pad. The excavated soil will be chemically and radiologically characterized for waste disposal in accordance with **Section 3.4.3**.

3.10 Site Restoration

Once the excavation is complete to existing grade, import fill soil will be placed over the SWDA Westside footprint (**Figure 2**) as needed to match the grade at which the adjacent housing is constructed, and to provide proper site drainage utilizing the existing storm drain infrastructure. Fill will be placed in 1-foot lifts under the observation of the project engineer and compacted via mechanical means. Field density testing will be required at the project engineer's discretion and must meet or exceed a minimum of 90-percent relative compaction based on the American Society for Testing and Materials (ASTM) Standard D1557, *Standard Test Methods for Laboratory Compaction Characteristics of Soil Using Modified Effort (56,000 ft-lbf/ft³ (2,700 kN-m/m³))* (ASTM, 2012). Further details are provided in the Site Backfill and Grading Plan (**Appendix E**).

Existing roadways, sidewalks, curbs, gutters, drainage culverts, and utilities will remain and be modified as needed to transition to open space allowing for vegetation growth. An approved seed mix will be applied by hydro-seeding to restore the area in coordination with project stakeholders. The seed mix will be consistent with that previously used at Treasure Island and will be submitted to the Navy for concurrence prior to application.

3.11 Radiological Survey

A radiological survey will be performed at finish grade, covering SWDA Westside in its entirety, and documented under this NTCRA. The radiological survey will be performed to determine the need for continuing active radiological controls at the site. The survey will consist of radiation exposure rate measurements collected from across the area. The survey results will be documented in the PCSR with recommendations as to whether active radiological controls remain necessary within the fenced area at SWDA Westside.

The radiological survey will be performed using a Ludlum Model 19 exposure rate meter (or equivalent) to verify general area (i.e., waist-level) exposure rates are no more than 11 microroentgens per hour (uR/hr), which is a Navy-established action level threshold. Any area identified above this action level will be delineated and investigated further in an attempt to determine the source of the elevated exposure rate. The offending material will be removed for further investigation, characterization, and disposal, as warranted.

3.12 Final Inspection/Site Close-Out

Following site restoration, a pre-final inspection will be held with the Navy to verify that field work has been completed in accordance with the approved work plan. Any concerns noted by the Navy that do not conform to specifications and any other requirements will be documented and presented to Gilbane. Gilbane will be responsible to correct any findings prior to a follow-on final inspection. Sign-off on the final inspection will serve as acceptance of site work and task completion.

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4.0 Environmental Protection

Environmental protection measures will be implemented according to the Environmental Protection Plan provided in **Appendix F** and be maintained during soil excavation and handling activities. The Environmental Protection Plan includes a Storm Water Management Plan (SWMP) and a Dust Control Plan (DCP) that meet the substantive requirements for storm water and dust mitigation as well as the unique needs encountered based on the public's concerns regarding work at Treasure Island.

4.1 Storm Water Management

The SWMP describes the BMPs to be implemented, including erosion and sediment control, waste management and disposal spill responses, post-removal/remediation controls, site inspection and monitoring programs, responsible personnel, training requirements, and certifications and compliance requirements. The SWMP implements the substantive provisions of the California State Water Resources Control Board National Pollutant Discharge Elimination System General Permit for discharge to surface water.

Appropriate perimeter BMPs will be installed along the perimeter of work areas prior to the start of excavation activities. During excavations, run-on will be adequately managed or diverted upstream of the excavation locations by utilizing diversion berms, such that the risk of erosion by upslope storm water is eliminated. Fiber rolls, silt fences, and/or sandbags will be installed along the fenced perimeter to prevent storm water from entering or leaving project work zones. The BMPs will minimize or prevent sediment entrainment in run-off entering the San Francisco Bay.

4.2 Air and Dust Monitoring

Air and dust monitoring will be performed in accordance with the DCP. The DCP describes the dust mitigation requirements, along with dust control practices and integrating air monitoring requirements for soil/debris excavation, stockpiling and movement. The DCP addresses the substantive requirements of air quality for construction and environmental remediation operations and Title 17, California Code of Regulations, Section 93105, *Asbestos Airborne Toxic Control Measures for Construction, Grading, Quarrying, and Surface Mining Operations*. Air monitoring will be performed at upwind and downwind locations during activities with the potential for generating dust.

A rigorous air sampling program with upwind and downwind monitoring will be performed and consist of both real-time monitoring of work zones, as well as work area perimeters. Monitoring will be performed for dust (particulate matter less than 10 microns in diameter) and VOCs. The collection and analysis of air samples for lead, PCBs, PAH, and ²²⁶Ra, and dioxin also will be performed to confirm dust mitigation activities are effective.

Wind speed will be monitored using an anemometer mounted on a wind pole or otherwise available to measure wind speed near the work site. Work will be stopped any time the wind speed is sustained over 25 mph. If a warning of gale force winds is issued, precautions will be taken to minimize any danger to persons, and to protect the work and any nearby Government property.

Water will be applied to the soil surfaces any time excavation or soil movement activities have the potential to create airborne dust. Soil stockpiles will be sprayed with a soil stabilizing agent to help prevent airborne dust due to wind when piles are not being moved during the work week or over weekends. A water truck or water buffalo will be used to wet down road surfaces in and around the work area.

5.0 Traffic Control

Traffic control will be implemented in accordance with the Traffic Plan provided in **Appendix G**. The Traffic Plan addresses road closures and/or restrictions, routing, other relevant traffic disruptions, and controls for traffic into/out of SWDA Westside. It identifies approved truck routes, truck holding and queuing areas, possible lay-down areas for construction materials, and wash-down and dust control areas as needed. In addition, it contains sufficient detail to ensure that impact to street traffic from site work will be minimized, and that there is safe traffic flow in the construction areas. The plan includes methods of maintaining traffic circulation such as lane reductions, shifts and alterations, traffic-pattern changes (flow directions, detours, and speed limits), signage, barricades, and signals. The Traffic Plan will be updated as needed in response to Treasure Island development detours.

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6.0 Health and Safety

Health and safety measures will be implemented in accordance with the SSHP (Gilbane, 2020a). The SSHP is a stand-alone document that includes an Accident Prevention Plan (APP). An activity hazard analysis (AHA) for tasks to be performed is included in the APP. The SSHP will be immediately accessible to workers at the site at all times during the project. A trained and qualified Site Safety and Health Officer (SSHO) will be dedicated to the project whose sole responsibility will be site safety during all site activities. In accordance with the SSHP, the SSHO has full authority to stop work immediately if conditions warrant and will immediately report any accidents to the ROICC, CSO, and the COR.

The SSHP will be maintained as a “living” document, with field changes documented and added/amended to the AHA and the APP as appropriate. Using information gathered as part of the AHA, engineering controls, work practices, PPE, or a combination of these will be implemented to protect workers by eliminating or effectively controlling the identified hazards. Engineering controls and work practices will be used to the greatest extent possible, supplemented by PPE as appropriate, to maintain a safe work site. References used to develop the SSHP include, but are not limited to Title 29, Code of Federal Regulations, Section 1910.120 (Hazardous Waste Operations and Emergency Response), the U. S. Army Corps of Engineers (USACE) *Safety and Health Requirements*, Manual No. 385-1-1 (USACE, 2014), and United Facilities Guide Specification (UFGS) 01 35 26, *Governmental Safety Requirements* (UFGS, 2017a).

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7.0 Quality Control

Quality assurance (QA)/QC will be performed according to the CQCP provided in **Appendix H**. The CQCP, prepared in accordance with United Facilities Guide Specification (UFGS) 01 45 00.00 20, *Quality Control* (UFGS, 2017b), describes:

- The QA/QC organization, including a chart showing lines of authority;
- The name, qualifications, duties, authorities, and responsibilities of each person assigned a QC function; and
- A schedule for managing submittals, testing, inspections, and other QA functions (including those of subcontractors, fabricators, suppliers, purchasing agents, etc.) that involve assuring quality workmanship, verifying compliance with the plans and specifications, and other QC objectives.

A QA surveillance plan with a list of definable features of the work will be implemented by conducting inspections to verify compliance with work elements and planning document requirements. Reporting includes summary reports of field work with corresponding site photos, a schedule of data submissions, inspection data sheets, problem identification and corrective measures reports, evaluation reports, acceptance reports, and final documentation.

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8.0 Post-Construction Summary Report

A PCSR summarizing the activities conducted for the removal action will be prepared following the completion of the field work. The PCSR will document a detailed narrative of the NTCRA, including sample data, data validation results, radiological survey data, copies of manifests and daily monitoring logs. The PCSR will also include post removal conditions detailed from site restoration activities and achievements of the NTCRA objectives.

The PCSR will, at a minimum, include the following sections:

- Overview - This section will include a very brief discussion of site characteristics, Conceptual Site Model, contaminants of concern, major findings, and results of previous site investigation and NTCRA activities.
- Removal Action Objectives - This section will detail the removal action objectives specified in the Action Memorandum (Navy, 2007), and subsequent modifications, if any.
- Removal Actions - This section will summarize actions taken to meet the objectives.
- Demonstration of Completion - This section will include information needed to demonstrate attainment of objectives (e.g., final sampling report, visual inspection report).
- Ongoing Activities - This section will describe the activities, if any, still being performed or to be performed.
- Community Relations - This section will briefly summarize the public outreach activities conducted at the Site (e.g., community relations plan), and provide the dates of public meetings.
- Certification Statement - This section will have a statement by a service representative authorized to sign decision documents, certifying that the PCSR memorializes the completion of the NTCRA

The appendices will include, but not be limited to, the record drawings (survey), photographs, waste manifests, field sampling logs, laboratory analytical results, radiological data packages, permits, and response to agency comments on the draft report. A copy of load tickets for construction material, including fill soil, and waste disposal also will be provided.

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9.0 References

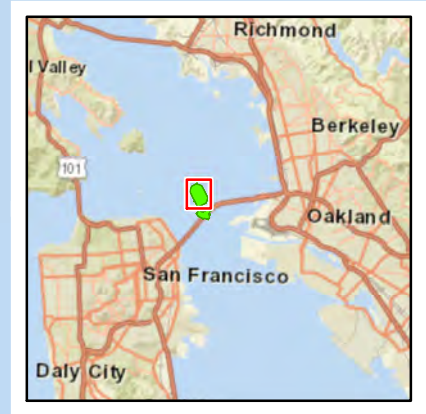
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FIGURES

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San Francisco Bay



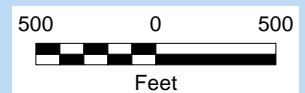
SWDA Westside

Site 12

Building 570
Gilbane Site Office

-  SWDA Westside
-  Site 12
-  Building

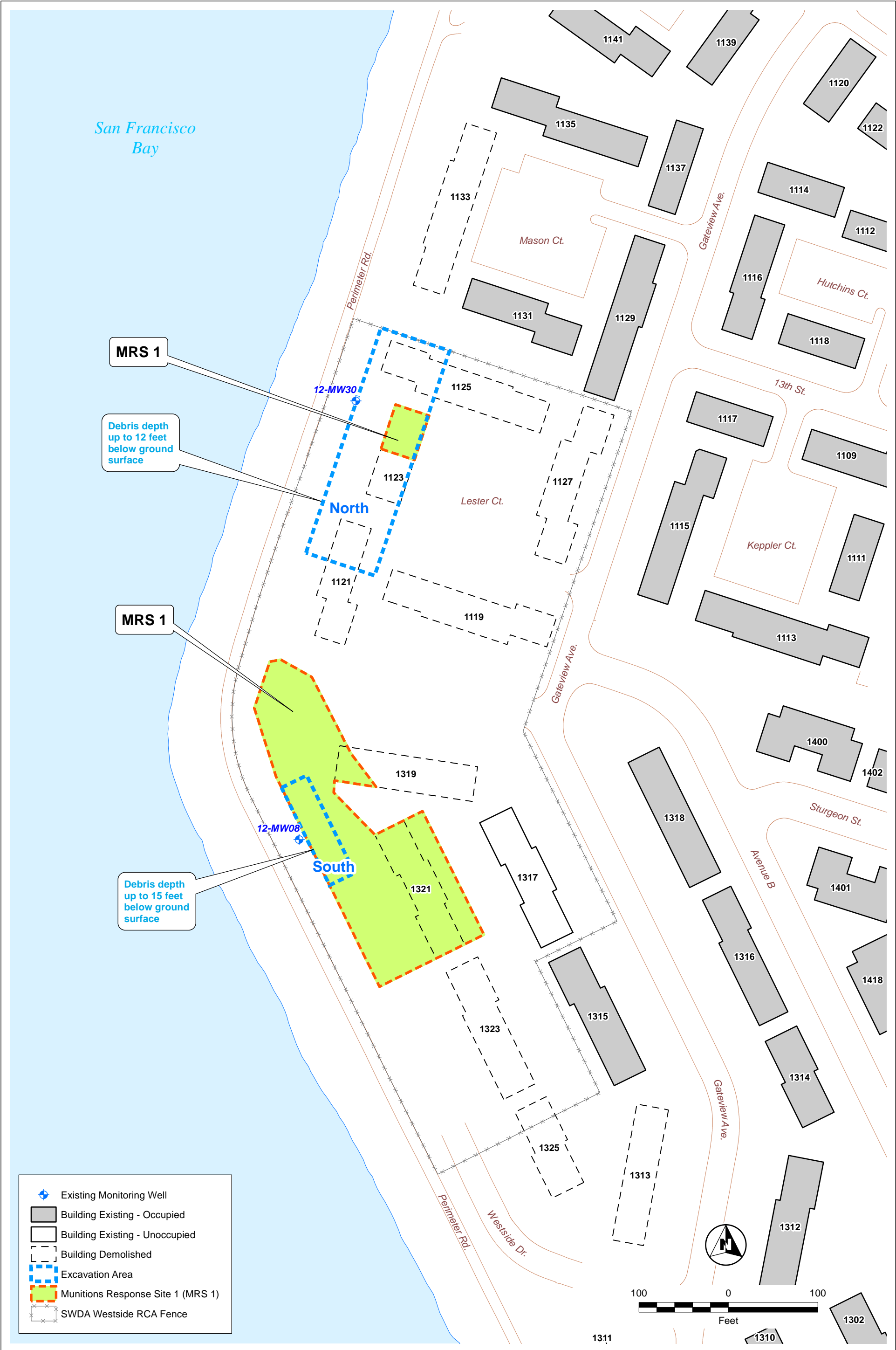
Note:
Background map source: ESRI® and Partners, 2020.



**Phase IV NTCRA for SWDA Westside at
IR Site 12**
Former Naval Station Treasure Island
San Francisco, CA

Figure 1
IR Site 12
SWDA Westside
Location Map

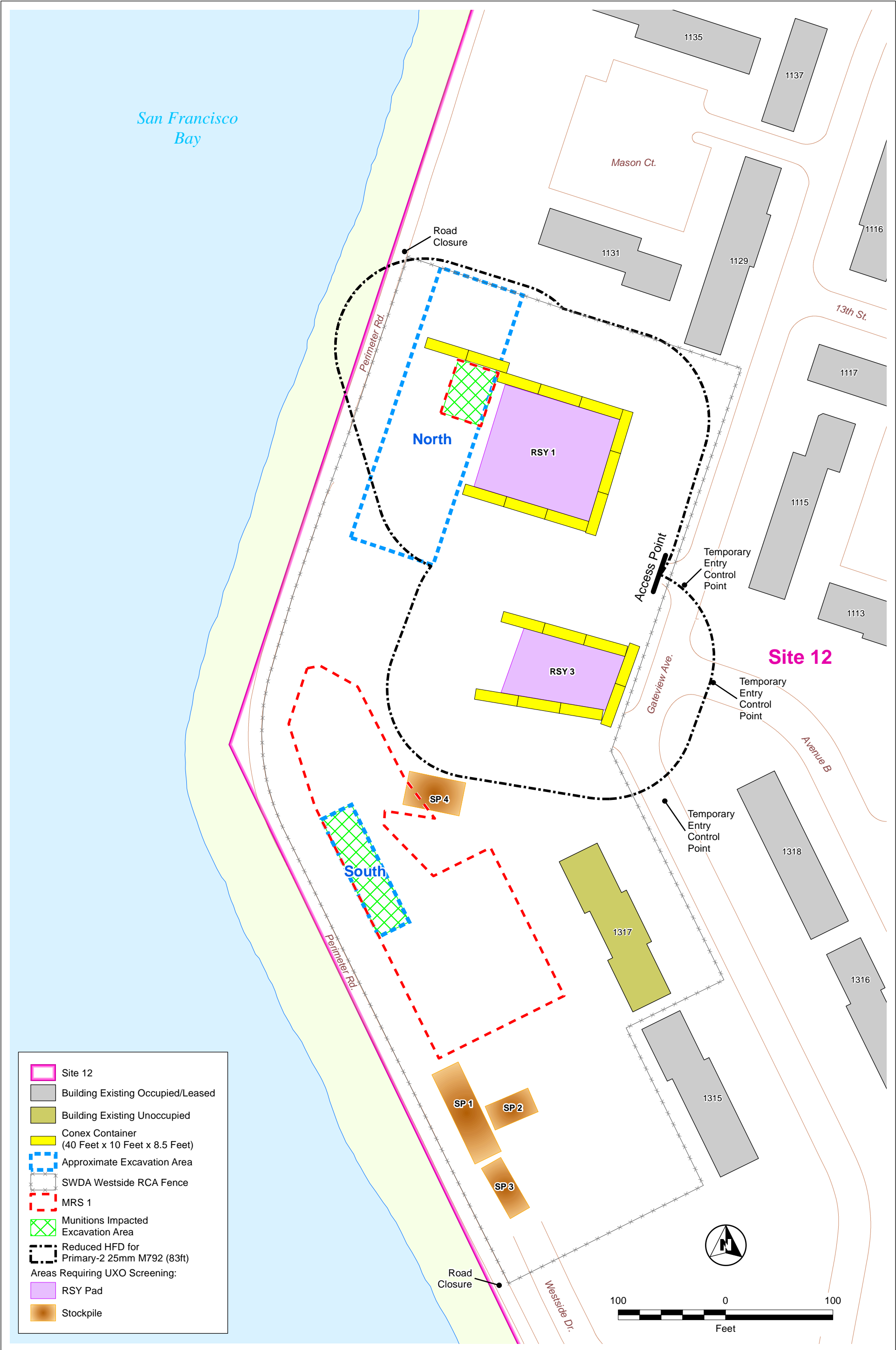
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FIGURE 3
PROJECT SCHEDULE
(To be Provided in Final Document)

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APPENDIX A

RADIATION PROTECTION PLAN

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Naval Facilities Engineering Command Southwest
BRAC PMO West
San Diego, CA

DRAFT

Radiation Protection Plan

Phase IV Non-Time Critical Removal Action, Solid Waste
Disposal Area Westside, Installation Restoration Site 12
Former Naval Station Treasure Island
San Francisco, CA
June 2020



Naval Facilities Engineering Command Southwest
BRAC PMO West
San Diego, CA

DRAFT

Radiation Protection Plan

Phase IV Non-Time Critical Removal Action, Solid Waste
Disposal Area Westside, Installation Restoration Site 12
Former Naval Station Treasure Island
San Francisco, CA
June 2020

DCN: GLBN-0005-F5271-0002

Prepared for:



Department of the Navy
Naval Facilities Engineering Command Southwest
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Prepared by:



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1655 Grant Street, Suite 1200
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Contract Number: N62473-17-D-0005; Task Order No. N6247318F5271

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Exhibit 5	Acceptable Surface Contamination Limits

List of Attachments

Attachment 1	Radiation Safety Manual
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Acronyms and Abbreviations

ALARA	as low as reasonably achievable
ALI	Annual Limit on Intake
CDE	committed dose equivalent
CEDE	committed effective dose equivalent
CFR	Code of Federal Regulations
cm	centimeter(s)
cm ²	square centimeter(s)
DAC	Derived Air Concentration
DDE	deep dose equivalent
dpm	disintegrations per minute
in	inch(es)
IR	Installation Restoration
Gilbane	Gilbane Federal
LDE	lens dose equivalent
mg/cm ²	milligram per square centimeter
M&E	material and equipment
mrem	millirem
N/A	not applicable
NaI(Tl)	sodium iodide (thallium activated)
NIST	National Institute of Standards and Technology
NRC	U.S. Nuclear Regulatory Commission
NTCRA	non-time critical removal action
PPE	personal protective equipment
RASO	Naval Sea Systems Command Detachment, Radiological Affairs Support Office
RCA	Radiologically controlled area
RPP	Radiation Protection Plan
RSO	Radiation Safety Officer
RWP	radiation work permit
SWDA	solid waste disposal area

TEDE	total effective dose equivalent
TODE	total organ dose equivalent
ZnS(Ag)	zinc sulfide (silver activated)

1.0 Introduction

This Radiation Protection Plan (RPP) describes the radiation protection measures to be applied in support of the Phase IV non-time critical removal action (NTCRA) for chemical and radioactive contaminants at Solid Waste Disposal Area (SWDA) Westside in Installation Restoration (IR) Site 12 at the former Naval Station Treasure Island in San Francisco, California. The RPP will be implemented consistent with the Gilbane Federal (Gilbane) Radiation Safety Program governing work performed in accordance with the requirements of Gilbane's State of California Radioactive Materials License 7948-07: Part 20 of Title 10 of the Code of Federal Regulations (10 CFR 20), *Standards for Protection Against Radiation*; and California Code of Regulations, Title 17, Division 1, Chapter 5, Subchapter 4, *Radiation*.

This RPP was prepared by Gilbane Federal (Gilbane) for the U.S. Department of the Navy (Navy) Base Realignment and Closure Program Management Office West under Radiological Environmental Multiple Award Contract N62473-17-D-0005, Contract Task Order N62473-18-F-5271, with the Naval Facilities Engineering Command Southwest.

1.1 Policy

Work with radioactive materials must be purposeful; performed in a manner that protects project staff, members of the general public, and the environment; and implemented in a manner that reduces health or safety risks associated with radioactive materials and ionizing radiation to levels that are as low as is reasonably achievable (ALARA). No activities will be conducted that could result in an occupational exposure of individuals to ionizing radiation without the expectation of an overall benefit from the activity causing the exposure. Both individual and collective exposures should be maintained as far below regulatory limits as social, technical, economic, practical, and public policy considerations permit.

1.2 Scope

The radiation protection measures described in this RPP apply to operational radiological activities performed by Gilbane under its radioactive materials license in support of the Phase IV NTCRA at SWDA Westside in IR Site 12. Gilbane possesses a current radioactive material license from the State of California (License No. 7948-07). Radioactive material possessed by Gilbane under its radioactive materials license will consist primarily of accumulated contaminated material and equipment (M&E), soil, and debris generated by remediation activities. The acceptable surface contamination levels given in this RPP (**Exhibit 5**) apply to tools, equipment, and materials.

1.3 Implementation

IR Site 12 is radiologically impacted as documented in the *Final Historical Radiological Assessment Supplemental Technical Memorandum, Naval Station Treasure Island, San*

Francisco, California (TriEco-Tt, 2014). Potential sources of radioactivity in SWDA Westside are discrete radioactive objects containing radium-226 such as deck markers, foils coated with radium salts, instrument gauges, and soil contamination from degraded objects.

Radiological controls will be applied to M&E and personnel involved in remediation and survey activities. Restricted areas around each work area will be established as needed with temporary fencing, warning placards, and controlled access. Where the potential for contamination exists, equipment will be surveyed before being brought into a restricted area. M&E and personnel will be surveyed for contamination before leaving a restricted area. Restricted areas may be de-posted and released from radiological controls once acceptable surface contamination levels given in **Exhibit 5** are met.

2.0 Program Administration

The Gilbane Radiation Safety Program will be implemented for this project through competent individuals who are trained in the program requirements and execute program requirements according to approved written plans and procedures approved by the Naval Sea Systems Command Detachment, Radiological Affairs Support Office (RASO).

2.1 Organization and Responsibilities

Qualified and experienced individuals will staff the positions described below to ensure consistent and successful implementation of radiation protection measures. Individuals will have the requisite skills necessary to perform the functions of their position. Specific duties and responsibilities are delineated in Gilbane's Radiation Safety Manual (see **Attachment 1**).

2.1.1 Corporate Radiation Safety Officer

The Gilbane Corporate Radiation Safety Officer (RSO) is responsible for the implementation and oversight of the Radiation Safety Program, under which this RPP provides guidance for radiation protection measures. The Corporate RSO may delegate the project-specific management of the Radiation Safety Program to a qualified individual; however, the responsibility for implementation of and compliance with the Radiation Safety Program remain with the Corporate RSO. The Corporate RSO will issue a letter of designation indicating the scope of the actions and decisions for which the individual is authorized to act on behalf of the Corporate RSO.

2.1.2 Gilbane Project Manager

The Gilbane Project Manager, with the support of the Project/Site RSO, will ensure that work is planned and conducted in accordance with the RPP and complies with radiation protection requirements.

2.1.3 Project/Site RSO

The Project/Site RSO reports to the Corporate RSO and is responsible for implementing the RPP and directing all project-related radiological activities in support of the Gilbane Project Manager. The Project/Site RSO will ensure that radiological hazards are identified during work planning, and that appropriate controls are implemented to maintain worker exposure to these hazards ALARA. The Project/Site RSO is responsible for providing on-site radiation protection support to the Gilbane Project Manager, implementing the RPP in the field, and ensuring that qualified personnel perform radiological work using adequate radiological controls according to the Radiation Safety Manual (**Attachment 1**).

The Project/Site RSO is the designated Authorized User under the radioactive materials license and will supervise and ensure the proper use of licensed materials. In accordance with RASO requirements, the Project/Site RSO or a qualified designee will be on-site during radiological work activities.

2.1.4 Radiation Safety Technicians

Radiation safety technicians will function as part of the field teams reporting to the Project/Site RSO and are responsible for assisting and guiding workers in the radiological aspects of their jobs. Radiation safety technicians are responsible for the protection of workers and the general public by implementing RPP requirements and the Radiation Safety Manual (**Attachment 1**) as directed by the Project/Site RSO.

2.1.5 Radiation Safety Professionals

Radiation safety professionals (e.g., radiation safety managers, radiological engineers, and/or health physicists) will be assigned to assist the Project/Site RSO in implementing the RPP, as appropriate. Such assistance may include:

- Providing health physics guidance on an as-needed basis.
- Conducting required radiological safety training.
- Conducting radiation incident investigations, project inspections, and program audits.

These individuals will be designated by the Corporate RSO based on their experience and education.

2.1.6 Radiation Workers (Field Personnel)

Radiation workers (i.e., field personnel who access restricted areas to perform work) are responsible for following RPP requirements and the Radiation Safety Manual (**Attachment 1**) to the best of their ability and knowledge. This responsibility includes proper use of protective and personnel monitoring equipment, notifying management of any potential or real radiation hazards or improper practices, and maintaining the individual radiation exposure of themselves and others ALARA. Radiation workers will use licensed materials under the supervision of the Project/Site RSO.

2.2 Training and Qualifications

Radiation protection personnel identified above will possess the technical competence and experience required to establish, implement, and maintain their applicable functional areas of the Radiation Safety Program, and the management skills to direct aspects of the Radiation Safety Program within their range of responsibility. Project-specific training will be provided commensurate with the anticipated duties and assignments of these personnel.

Radiation workers will be trained in radiation awareness commensurate with the requirements of Title 17, California Code of Regulations, Section 30255, *Notices, Instructions, and Reports to Personnel*. Visitors and escorted persons will receive a site briefing and will be assigned to a qualified radiation worker when in a restricted area.

2.2.1 Qualifications

The Corporate RSO will evaluate the qualifications of radiation protection personnel. Qualifications for each position will be based on the following factors as they apply to each individual's duties:

- The level of radiological hazard.
- The extent to which the individual must be able to work independently to ensure his or her own radiation safety.
- The extent to which the individual must be able to provide radiation protection guidance to others.

The resume for the Project/Site RSO will be submitted to RASO upon request. RASO will be notified of any changes in Project/Site RSO.

2.2.2 Radiation Worker Training

Radiation workers will be trained in radiation safety commensurate with their assigned duties and the radiological hazards the individual is expected to encounter during his/her work. Individuals will receive the appropriate training prior to assuming job duties involving radioactive materials.

The following radiation safety topics will be included in training, as applicable, commensurate with the individual's assigned tasks:

- Fundamentals of radiation safety,
- Radiation detection instruments,
- Radiation protection equipment and its use,
- U. S. Nuclear Regulatory Commission (NRC) and State of California regulations (as applicable),
- Operating and emergency procedures, and
- Case histories and lessons learned related to operations.

Training may be delivered as formal classroom instruction, videotape, or self-study. Methods of evaluation may include classroom examinations, on-the-job training and practical examinations, and discussion and/or drills on emergency procedures. Retraining may be required on topics where an individual is found to be deficient in the practical and/or classroom areas. Additional training will be provided whenever there is

a significant change in duties, regulations, or the terms and conditions of the license. Refresher training will be provided annually for on-site personnel.

Training will be conducted by qualified individuals (e.g., individuals whom the Corporate RSO determines are qualified by their experience and education). Training records will be maintained on-site.

2.2.3 Site Briefing

An RPP site briefing is designed for an escorted person and is presented when access to a restricted area is needed. Specific to the area(s) of concern where access is needed, the RPP brief will cover at a minimum:

- Applicable portions of 10 CFR 19.12, the RPP, the Radiation Safety Manual (**Attachment 1**) or other written procedures, and specific radiation work permits (RWPs) relevant to the radiological hazards associated with the restricted area(s).
- A description of radiation exposure risks and monitoring requirements.
- Access/egress protocol specific to the restricted area(s).

2.3 Procedures and Work Instructions

The policy and procedures that implement Gilbane's radiation safety program are referred to collectively as the Radiation Safety Manual (**Attachment 1**), which describes how Gilbane complies with regulatory requirements. Additional guidance, where appropriate, may be developed in the form of task-specific work instructions that are outside of, but supplement, the Radiation Safety Manual (**Attachment 1**). Work instructions will be approved by the Corporate RSO and captured and maintained as project records.

3.0 Dose Limits

The dose received by radiation workers assigned to complete activities involving exposure to radiation and/or to radioactive material from licensed and unlicensed radiation sources will be managed within regulatory and administrative limits defined for occupational doses. Occupational doses for radiation workers do not include any dose received from background radiation, medical practice/service, or as a member of the public.

3.1 Regulatory Limits

The occupational dose received by occupationally exposed individuals will be controlled such that the regulatory limits in **Exhibit 1**, specified in millirem (mrem), are not exceeded in a single year. Regulatory limits are specified for the total effective dose equivalent (TEDE) and the total organ dose equivalent (TODE) for occupationally exposed individuals. The TEDE is the sum of the deep dose equivalent (DDE) from external exposures and the committed effective dose equivalent (CEDE) from internal exposures. The TODE is the sum of the DDE and the committed dose equivalent (CDE) to any individual organ and tissue.

Exhibit 1: Regulatory Dose Limits

Exposure Type	Monitoring Required (mrem)	Annual Regulatory Dose Limits ⁽¹⁾ (mrem)
TEDE (DDE + CEDE)	500	5,000
TODE (DDE + CDE) to any Organ ⁽²⁾	5,000	50,000
Lens (of the Eye) Dose Equivalent (LDE)	1,500	15,000
Shallow-Dose Equivalent to the Skin	5,000	50,000
Shallow-Dose Equivalent to any Extremity	5,000	50,000

Notes:

⁽¹⁾ from 10 CFR 20.1201

⁽²⁾ except lens of the eye because unique monitoring requirements and annual legal exposure limits exist for this organ; see subsequent table row

3.1.1 Unborn Child/Declaration of Pregnancy

The dose to an embryo/fetus during the entire pregnancy will not exceed 500 mrem due to the occupational exposure of a declared pregnant woman. Declaration of pregnancy will be entirely at the discretion of the woman (medical proof is not required). To declare pregnancy, the woman must inform her supervisor or the Project/Site RSO, in writing, of the pregnancy and an estimated date of conception so that the estimated

dose to the embryo/fetus prior to declaration can be determined. A woman may withdraw her declaration of pregnancy at any time and for any reason by notifying her supervisor or the Project/Site RSO in writing. Any woman who does not declare her pregnancy will be subject to the normal occupational dose limits and will not be subject to special controls or treatment with respect to work assignments involving exposure to radiation even if she is pregnant.

3.1.2 Planned Special Exposures

No anticipated event within the work scope subject to this RPP will require use of a planned special exposure. In the event it is necessary to initiate such a need, an activity-specific work instruction including a formal ALARA review and an RWP will be prepared by the Project/Site RSO and approved by the Corporate RSO prior to submittal to RASO for acceptance.

3.1.3 Minors

The dose to a minor will be controlled administratively so that exposure does not exceed 10% of the dose limits for occupationally exposed adults.

3.1.4 Members of the General Public

The dose to individual members of the general public will not exceed 100 mrem in a year, excluding background and medical exposures, and will not exceed 2 mrem in any one hour in any unrestricted area.

3.2 Administrative Limits

Administrative limits for occupationally exposed adults, shown in **Exhibit 2**, will be used to control doses to ensure that regulatory limits are not exceeded and that occupational exposures are maintained ALARA. The administrative limits also serve to alert radiation protection personnel to practices or trends in the work environment that are resulting in additional or excessive exposure to individuals. Gilbane's goal is that no individual will exceed the administrative limits.

Extensions to the administrative limits will be considered on a case-by-case basis when written justification for the need to extend the individual's dose limit is provided by the individual's supervisor. Approval by the Corporate RSO is required if the quarterly or annual administrative limits will be exceeded. Extensions will be permitted only after an individual's accrued dose for the current year has been determined.

Exhibit 2: Administrative Dose Limits

Exposure Type	Annual Administrative Limit (mrem)
TEDE	1,000
LDE ⁽¹⁾	3,000
Skin Dose	10,000
TODE	10,000

Notes:

⁽¹⁾ Dose equivalent to the lens of the eye

3.3 Dose Investigations

A dose investigation will be performed for individuals whose exposure exceeds the levels in **Exhibit 3**.

Exhibit 3: Dose Investigation Levels

Quarterly Dosimeter Reading	Investigation Level (% of Administrative Limit)
<3 months	>25%
3 to 6 months	>50%
6 to 9 months	>75%
9 to 11 months	>90%

The Corporate RSO or designee will work with the individual's supervisor to determine if action is required to minimize the individual's dose, to monitor future dose closely, or to pursue an extension to the administrative limits.

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4.0 Dose Monitoring

An individual's exposure to internal and/or external sources of radiation will be monitored if the occupational dose is likely to exceed 10% of the administrative limit, as listed in **Exhibit 2**. Dose monitoring requirements will be specified in the RWP.

4.1 Internal Exposure Monitoring

Monitoring of internal exposure normally will be conducted by use of air samples, particularly samples collected within the breathing zone. When a potential or actual condition exists in which an individual could have received an unmonitored intake of radioactive material, the intake will be determined by measurements of quantities of radionuclides excreted from or retained in the body (e.g., in-vitro bioassay and/or in-vivo bioassay monitoring). The measurements and methods used will be directed by the Corporate RSO.

Internal exposure does not include exposure due to medical administration of radionuclides. Therefore, individuals will inform the Project/Site RSO prior to entering a restricted area when medical treatments involving radionuclides have been administered. Where possible, individuals will be reassigned to work in areas outside of restricted areas until the administered radionuclide is eliminated from the body to the extent that it will not significantly affect dosimeter or radiation detection measurements.

4.2 External Exposure Monitoring

External exposure monitoring will be accomplished using a thermoluminescent dosimeter or optically-stimulated luminescence dosimeter worn on the front of the upper torso. Radiological surveys may be performed to supplement personnel monitoring when work is being performed in areas where individuals are required to be monitored. Before a dosimeter is issued to an individual, the individual to be monitored will satisfactorily complete radiation worker training. Satisfactory completion is based on receiving a passing score on a competency exam administered by a qualified individual.

Personnel dosimetry will be processed and evaluated at least quarterly by a dosimetry processor holding current accreditation from the National Voluntary Laboratory Accreditation Program of the National Institute of Standards and Technology (NIST) for the type(s) of radiation for which the individual wearing the dosimeter is monitored.

4.3 Determination of Prior Occupational Exposure

The occupational dose during the current year will be determined for individuals who are likely to receive a dose in excess of 10% of the administrative limit. The individual's dose histories for prior years will be obtained and documented on NRC Form 4 or equivalent. The record will show each period in which the individual received an occupational dose, and will be signed by the individual who received the exposure.

4.4 Records and Reports

Records of occupational exposure monitoring results will be maintained for the duration of Gilbane's radioactive materials license. Reports of individual monitoring results will be made available to monitored individuals on an annual basis. Quarterly exposure reports will be generated to alert the Project/Site RSO of doses to individual workers that are approaching the administrative limits.

5.0 Instruments and Equipment

Commercially available portable and laboratory instruments and equipment will be used to perform three general types of measurements: (1) radiation, (2) contamination, and (3) airborne radioactivity. Instrumentation will be used only by individuals qualified in the use of the instrument.

5.1 Selection

Radiation detection and measurement instrumentation will be selected based on reliable operation, detection sensitivity, operating characteristics, and expected performance in the field. As a general rule, instruments will be capable of detecting radiation at levels anticipated to be present. Typical instrumentation that may be used is identified in **Exhibit 4**.

Exhibit 4: Typical Survey Instrumentation

Measurement Type	Detector Type	Effective Detector Area and Window Density	Instrument Model	Detector Model
Gamma Scan	NaI(Tl) scintillation	2 in (5.1 cm) dia x 2 in length N/A	Ludlum 2221	Ludlum 44-10
Alpha/Beta Scan/Static	Gas flow proportional	582 cm ² 3.4 mg/cm ² aluminized Mylar	Ludlum 2360	Ludlum 43-37
	Dual phosphor scintillation	100 cm ² 1.2 mg/cm ² aluminized Mylar	Ludlum 2360	Ludlum 43-93
Alpha/Beta Smears	ZnS(Ag) scintillation	5.1cm diameter 0.4 mg/cm ²	Protean WPC-9550	N/A
	Dual phosphor scintillation	5.1 cm diameter 0.4 mg/cm ²	Ludlum 2929	Ludlum 43-10-1
Exposure Rate	NaI(Tl) scintillation	1 in (2.5 cm) dia x 1 in length N/A	Ludlum 19	N/A

Notes:

- cm² – square centimeter
- cm – centimeter
- in – inches
- mg/cm² – milligrams per square centimeter
- ZnS(Ag) – zinc sulfite (silver activated)
- NaI(Tl) – sodium iodide (thallium activated)
- N/A – not applicable

5.2 Inventory and Control

A sufficient inventory and variety of operable and calibrated portable, semi-portable, and fixed radiation detection and measurement instrumentation will be established and maintained to satisfy the following considerations:

- Effective measurement of radiation exposure and control of radioactive material with instrumentation appropriate to enable the assessment of alpha, beta, and gamma radiation at the energies and intensities anticipated;
- Maximum number of personnel and separate work areas requiring surveillance;
- Frequency and types of surveys or measurements required to support normal and anticipated activities;
- Allowance for repair and calibrations; and
- Efforts to minimize delays in personnel access to and egress from radiological areas.

5.3 Calibration and Maintenance

Instruments and detectors will be calibrated for the radiation types and energies of interest using NIST-traceable sources and maintained to manufacturers' specifications. Anticipated radionuclide mixture ratios and varying energies will be accounted for during calibration by using a calibration source with a conservative and/or representative average energy as compared to the weighted average energy of the anticipated radionuclide mixture.

Each instrument will be inspected prior to use to ensure that it is in proper working condition. Instruments will be protected against inclement weather conditions in the field. Sufficient instrumentation redundancy will be maintained to preclude the need for repair and maintenance in the field. Maintenance and/or repair of equipment will be performed by the equipment manufacturer or authorized representative.

Calibration records will include the type of instrument and manufacturer; instrument model and serial number, date of calibration, calibration due date, and the name of the worker performing the calibration.

5.4 Response Checks

Instrument response checks will be conducted to assure constancy in instrument response, to verify that the detector is operating properly, and to demonstrate that measurement results are not the result of detector contamination.

Instrument response will be checked before the instrument is used each day, using a check source that emits the same type(s) of radiation (i.e., alpha, beta, and/or gamma) to be measured and that gives a similar instrument response. The check source does not necessarily use the same radionuclide as the radionuclide being measured. The

response check will be performed in a specified location using a specified source-detector alignment that can be repeated easily.

Prior to the initial use of a scaler instrument, a minimum of 20 measurements will be made using the source, and another 20 measurements will be made without the source to determine the instrument's expected response to ambient background. An acceptable range for both source and background instrument response will be developed using the chi-squared test. Response checks will be performed and evaluated against the acceptance criteria. If instrument measurements fall outside the acceptance criteria, the response check may be repeated or the instrument will be removed from service. If the instrument is removed from service, it will not be used until the problem is resolved or repaired.

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6.0 Radiological Monitoring

Radiological monitoring will be performed to identify, quantify, and evaluate the potential hazards associated with the radiological conditions in and around work areas. The survey data will be used to minimize the potential for exposure of personnel and to evaluate the effectiveness of site controls.

6.1 Radiological Surveys

Survey information will be used to inform individuals of the radiological conditions/hazards in the area, to determine any required area postings, to determine the type(s) of personal protective equipment (PPE) necessary, and to ensure that personnel exposures to radiation and radioactive materials are maintained ALARA. Surveys to assess radiation and contamination levels may be performed separately or jointly as warranted by expected radiological conditions and job requirements.

6.1.1 Radiation Surveys

General area surveys will be used to assess the nominal radiation fields, to verify that radiological conditions have not changed, and to establish specific radiological controls for work to be performed. Contact dose rate surveys will be used to locate and identify (1) the maximum radiation levels to which personnel could be exposed and (2) localized sources of radiation that present unique radiological concerns.

6.1.2 Contamination Surveys

Contamination surveys will be performed to measure fixed and removable levels of surface contamination. Removable contamination surveys will be performed to detect and quantify removable alpha/beta surface contamination using one of two methods:

- Smear Surveys - A smear is obtained by using an absorbent filter disk to wipe with moderate pressure across the area or item to be evaluated. The smear usually is wiped over an area of 100 cm² to quantify the level of removable contamination in units of disintegrations per minute (dpm) per 100 cm².
- Wipe Surveys - A wipe is obtained by wiping an absorbent pad or towel over a large area or the entire surface of the item being surveyed. Loose-surface contamination surveys (i.e., large area wipes) are performed periodically as a qualitative measure to ensure that contamination has not spread.

Both smear and wipe samples will be evaluated for gross alpha/beta activity, unless otherwise directed by the Project/Site RSO.

6.1.3 Survey Frequencies

A routine survey program consisting of daily, weekly, and monthly surveys will be implemented to monitor radiological conditions in designated work areas. Surveys will be conducted at a frequency commensurate with the hazard(s) present and the personnel occupancy in a given area. The performance frequency and detail level of surveys will consider the likelihood of changing conditions, the radiological hazards of specific areas, and the personnel occupancy within various areas, in order to anticipate personnel radiation doses and to ensure and verify that personnel exposure to radiation or radioactive materials is adequately controlled and monitored.

Routine survey frequencies will be augmented with additional surveys, under the direction of the Project/Site RSO, when any of the following conditions apply:

- Contamination above action levels is found outside of a restricted area.
- Unexpected, significant increases in radiation levels, contamination levels, or airborne radioactivity levels occur.
- Increased maintenance activities or changes in work scope may change radiological conditions (i.e., operations that may increase area dose rates, loose surface contamination, airborne radioactivity, etc.).

If conditions warrant, temporary changes to established survey frequencies will be approved by the Project/Site RSO.

6.1.4 Survey Documentation and Review

Surveys will be documented legibly, accurately, and timely. Survey documents will include the following information at a minimum:

- Date, time, and location of the survey.
- Instrument(s) used, including serial number and calibration date.
- Name and signature of the surveyor.
- Name and signature of the Project/Site RSO or designee reviewing the survey.
- Results of the measurements and analyses.
- Maps and drawings indicating the area(s) and items surveyed, as appropriate.

Documentation of surveys performed by trainees (i.e., non-qualified personnel) will be reviewed and approved by a qualified individual. Documentation methods, supervisory reviews, and the distribution of data will be standardized.

6.2 Air Sampling

Air sampling of general and localized areas will be performed when and/or where the generation of airborne radioactive material is possible. These samples will be used to

verify that radioactive material is being confined effectively and to provide warning of elevated concentrations for planning or response actions. The sampling points will be located in the airflow pathway near the known or suspected release point(s). As necessary, more than one air sample location may be used in order to provide a reasonable estimate of the general concentration of radioactive material in air. Breathing-zone air samples will be the primary method for monitoring workers' potential inhalation intake of radioactive material. The samples will be collected under known physical conditions (e.g., filter type, sample time, flow rate). The flow meters of air samplers will be calibrated annually at a minimum. Calibration will also be performed after repair or modification of the flow meter.

The limit for airborne radioactivity is based on radium-226 and will be 3×10^{-10} microcuries per milliliter, which represents a derived air concentration (DAC) of 1.0 (reference 10 CFR 20, Appendix B, Table 1). In the event the airborne levels reach a DAC of 0.1, the use of PPE (respirators) will be considered and/or work practices will be evaluated and modified if necessary to ensure the limit is not reached. DAC-hour tracking will be performed for individuals with breathing zone air sample results of 1 DAC or higher (see Section 7.7 for further discussion regarding DACs). A breathing-zone air sample result of 10 DAC-hours or higher without respiratory protection will result in the individual being restricted from work involving potential exposure to airborne radioactive material unless approved by the Project/Site RSO.

6.3 Monitoring Dose to the Public

Based on the scope of planned work, the limited activity of radionuclides expected, and the low concentrations of naturally occurring radioactive material anticipated, a public dose associated with tasks performed under this RPP is not anticipated.

To protect members of the public, survey and sampling protocols will be implemented in areas of intrusive work. Intrusive work locations that require monitoring (e.g., areas where soil excavations and/or handling, etc., may disturb sources of radioactive material) will be posted in conspicuous locations, and access will be restricted. Survey and sampling results and frequencies will be reviewed with RASO to ensure that the established controls are effective.

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7.0 Postings

Areas to which access should be limited for the purpose of protecting individuals against undue risks from exposure to radiation and radioactive materials will be restricted areas and will be posted as such.

7.1 General Requirements

Postings will consist of standardized signs or labels bearing the standard radiological trefoil symbol in magenta, purple, or black on a yellow background. Postings will provide information concerning a specific radiological hazard and include the wording: "Caution: Controlled Area" or "Caution: Restricted Area." These postings will also contain a contact telephone number. Supplemental information as specified by the Project/Site RSO may also be included as magenta (preferred), purple, or black markings on a yellow (preferred) or white background.

Barriers, signs, gates, doors, fences, etc., will be used to identify the boundaries of restricted areas and to control access of personnel into those areas. A minimum of one sign will be posted on each straight run of the boundary of a restricted area. Additional signs will be placed at approximately 30-meter (100-feet) intervals on long runs of the boundary. For waterfront areas, signs will be posted at areas accessible by watercraft. Areas not typically accessed by pedestrians (e.g., windows) do not requiring posting.

An access control area will be established to provide control over the entry to and exit from the restricted area. The access control point allows for the accountability of personnel, tools, and equipment that pass into and out of the area and also may function as a contamination control boundary between zones of differing contamination levels. During periods of inactivity, the access control point will be secured.

7.2 Radiologically Controlled Area

Any area accessible to individuals in which radiation levels could result in an individual receiving a dose equivalent of 100 mrem or more in a single year (excluding natural background and medical exposures) will be posted as a radiologically controlled area (RCA). For external sources, the RCA typically will be posted when the exposure rate exceeds 50 microroentgens per hour at a distance of 30 centimeters (cm) from the radiation source or from any surface that the radiation penetrates. However, this dose rate may be modified at the discretion of the Project/Site RSO based on accurately assessed occupancy factors.

7.3 Radiation Area

Any area accessible to individuals in which radiation levels could result in an individual receiving a dose equivalent in excess of 5 mrem in 1 hour at a distance of 30 cm from

the radiation source or from any surface that the radiation penetrates will be posted as a Radiation Area.

7.4 High Radiation Area

Any area accessible to individuals in which radiation levels could result in an individual receiving a dose equivalent in excess of 100 mrem in 1 hour at a distance of 30 cm from the radiation source or from any surface that the radiation penetrates will be posted as a High Radiation Area.

7.5 Contamination Area

Any area accessible to individuals in which removable surface contamination levels exceed the average acceptable surface contamination levels specified in Table 3 of Regulatory Guide 8.23, *Radiation Safety Surveys at Medical Institutions* (NRC, 1981), but do not exceed 100 times those values, will be posted as a Contamination Area. Regulatory Guide 8.23 values are reproduced in this document as **Exhibit 5**.

7.6 High Contamination Area

Any area accessible to individuals in which removable surface contamination levels exceed or are likely to exceed 100 times the average acceptable surface contamination levels specified in Table 3 of Regulatory Guide 8.23 (NRC, 1981) will be posted as a High Contamination Area. Regulatory Guide 8.23 values are reproduced in this document as **Exhibit 5**.

7.7 Airborne Radioactivity Area

A room, enclosure, or area will be posted as an Airborne Radioactivity Area if airborne radioactive materials composed wholly or partly of licensed material are present at the following concentrations:

- In excess of the DAC specified in state and federal regulatory documents, or
- At such a level that an individual present in the area without respiratory protective equipment could exceed an intake of 0.6 percent of the annual limit on intake (ALI) or 12 DAC-hours during the hours an individual is present in one week.

An ALI is defined as the amount of radioactive material taken into the body of an adult worker by inhalation or ingestion in a year that results in a CEDE of 5,000 mrem, or a CDE of 50,000 mrem to any individual organ or tissue. A DAC is defined as the product of the concentration of radioactive material in air (expressed as a fraction or multiple of the derived air concentration for each radionuclide) and the time of exposure to that radionuclide, in hours. One ALI is represented by 2,000 DAC-hours. DAC values are specified in Appendix B of 10 CFR 20.

7.8 Radioactive Materials Area

An area or room in which licensed material is used or stored, and in which the quantity of radioactive material exceeds 10 times (100 times for natural uranium or thorium) the quantity of such material specified in Appendix C of 10 CFR 20, will be posted as a Radioactive Materials Area.

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8.0 Work Planning and Performance

Radiological work activities will be planned in consultation with the Project/Site RSO and other radiation protection personnel tasked with oversight responsibilities. Work performed in restricted areas will require an RWP that details radiologically-based requirements and protective measures commensurate with the hazards associated with the specific activities being conducted.

8.1 Radiation Work Permits

RWPs will be used to inform individuals of the radiological conditions that exist in a restricted area and to detail the protective measures and controls needed to perform tasks in the restricted area. The RWP will describe the scope of work, including supporting tasks that must be performed in a restricted area, and will include, as necessary, supporting information and documentation such as work procedures, drawings, and diagrams. RWPs will be approved by the Project/Site RSO.

General RWPs will be used for the performance of routine activities such as observation, inspection, operator rounds, or tours where radiological conditions are stable. A general RWP normally will be valid for one calendar year, and will be rewritten at the end of the calendar year if necessary. Specific RWPs will be used for the performance of defined activities in specific locations and normally are valid for the duration of the activity or until the end of the calendar year in which they are written.

Work supervisors will review the provisions of specific RWPs with their workers before the related work begins. The Project/Site RSO may conduct a briefing prior to the start of the job to explain special requirements, engineering controls, or work restrictions. If the scope of the job changes due to any unexpected conditions, the work supervisor will notify the Project/Site RSO so that the impact on the provisions of the job can be evaluated. Workers or supervisors also will notify the Project/Site RSO of any unexpected or unusual conditions.

A copy of the RWP will be available at the entry/exit point of the restricted area or other location deemed appropriate by the Project/Site RSO. Individuals will read and sign the RWP before entering the restricted area or performing the tasks covered by the RWP.

8.2 ALARA Review

An ALARA review will be performed for any work involving radioactive material. The ALARA review will ensure that industry-recognized principles are applied to the radiological work so that exposure to ionizing radiation is purposeful; performed in a manner sufficient to ensure the protection of staff, members of the public, and the environment; and is maintained ALARA.

The ALARA review will include a review of the description of the scope, the sequence of events for the job evolution, and the radiological controls necessary to ensure the safety of the workers and the environment. The ALARA review will not only consider radiation safety requirements, but engineering and administrative controls that may be applied to reduce internal and/or external doses. The ALARA review will ensure that basic dose reduction strategies are employed using the ALARA concepts of time, distance, and shielding. The ALARA review may be conducted and/or documented as part of the RWP review and approval process.

8.3 Stop Work Authority

All radiation workers have the authority and responsibility to stop work when controls are inadequate or imminent danger exists. Radiation protection personnel will be responsible for stopping work whenever work being performed is outside the scope of the RWP, when workers are not complying with the provisions of an RWP, or when conditions vary from those described on the RWP and pose an immediate hazard to individuals in the area.

When work has been stopped, the work area will be placed in a safe configuration and work will not be resumed until proper controls have been established. Work re-start will be authorized by the Project/Site RSO with RASO concurrence.

8.4 Atypical Work Site Conditions

Radiological work will be suspended immediately where any of the following atypical work site conditions is encountered.

- An individual TEDE exceeds 500 mrem.
- The collective TEDE for the job exceeds 1,000 mrem.
- Airborne radioactivity above the minimum detectable concentration is identified/verified on an air sample.
- Conditions warrant the radiological posting of an area as a High Radiation Area or a High Contamination Area.

RASO will be notified immediately. The Corporate RSO, with concurrence from RASO, will authorize work resumption.

9.0 Radiation and Contamination Control

Wherever possible, radiation and contamination will be controlled at the source. Administrative procedures (e.g., establishing restricted areas) and engineering controls (e.g., shielding, containment, ventilation) will be used.

9.1 Restricted Areas

Restricted areas will be established for the purpose of protecting individuals against unnecessary risks from exposure to radiation and radioactive materials.

9.1.1 Access Control

Restricted area boundaries will be demarcated using yellow and magenta floor tape, rope, ribbon, barricades, or other suitable identifiers. Hoses or electrical leads which cross restricted area boundaries will be secured to prevent the hose or lead from being pulled into or out of area inadvertently and spreading contamination. An entry/exit control point (e.g., a swing gate or rope between two stanchions) will be established. Suitable radiation detection instruments will be provided at or near the entry/exit point.

9.1.2 Tools, Equipment, and Materials

Tools, equipment, and materials entering and exiting restricted areas will be controlled to ensure that unnecessary items are not brought into the restricted area and that contaminated items are not removed from the restricted area inadvertently.

Only tools, equipment, and material necessary to accomplish the planned task will be allowed into a restricted area. Work activities will be pre-planned to minimize the number of tools and/or equipment items and the quantity of material taken into restricted areas. Personal items will not be allowed. Container wrappings, packing, and similar materials will be segregated from essential items before the items are brought into a restricted area. The use of wooden pallets and other materials difficult to survey and decontaminate will be minimized inside restricted areas.

Non-radioactive materials will not be stored in a restricted area. Tools or equipment present within a restricted area will be fully used, rather than introducing additional tools or equipment to the restricted area. Containers of temporarily stored restricted area tools and equipment, such as toolboxes and crates, will be labeled as radioactive materials. Items brought into a restricted area will be removed as soon as practical to reduce the likelihood that the items will become contaminated inadvertently.

9.1.3 Housekeeping

Good housekeeping practices will be used at all times. Work areas will be cleaned up and housekeeping performed after each task is completed.

9.2 Contamination Areas

Contamination Areas will be established to minimize the spread of contamination during work activities, minimize the need for respiratory protection devices, and maintain internal personnel exposures ALARA. The number and size of Contamination Areas will be limited to reduce the amount of material that may become contaminated during use and to reduce the resources that are expended to decontaminate contaminated items and areas.

9.2.1 Set-Up

Surface coverings using plastic sheets or absorbent material, strippable coatings, containers to collect leakage of radioactive materials, and similar measures will be used based on the type of work to be performed and the location of the work.

An entry/exit point will be established at the boundary of the Contamination Area. Placement of the entry/exit point will take into consideration radiation levels in the area. A step-off-pad will be provided at the entry/exit control point. Step-off pads will be treated as non-contaminated. Protective clothing will be removed before the individual exits onto the step-off pad. In areas where more than one set of protective clothing is used (e.g., High Contamination Area), additional step-off pads may be used to prevent the spread of contamination. Appropriate receptacles will be provided to collect used protective clothing. Radioactive or contaminated trash will not be allowed to accumulate to avoid potential contribution to increased ambient radiation levels. The appropriate radiation detection instruments will be provided at or near the step-off-pad or work area and at the entry/exit point in a manner that will facilitate their use by radiation workers.

9.2.2 Access/Egress

Appropriate PPE will be donned prior to entry into and removed prior to egress from a Contamination Area. Individuals will be monitored for contamination upon exit. Individuals with open wounds or sores generally will not be granted access to a Contamination Area. Entry may be authorized by the Project/Site RSO on a case-by-case basis, if appropriate protection of the wound or sore is verified, planned work activities are unlikely to compromise the protection, and there is no other medical reason to restrict entry.

9.2.3 Tools and Equipment

Tools or equipment will be bagged, sleeved, covered, or coated, as appropriate, before being taken into a Contamination Area. Contaminated or unmonitored items will be bagged and properly labeled before being removed from a Contamination Area unless otherwise directed by radiation protection personnel. Bags will indicate the presence of contaminated or potentially contaminated materials.

9.3 Contamination Control Measures

Surfaces, tools, equipment, and materials will meet the acceptable surface contamination levels given in **Exhibit 5** or will be controlled as contaminated. **Exhibit 5** values are from Table 3 of Regulatory Guide 8.23 (NRC, 1981).

9.3.1 Surveys of Tools and Equipment

Where the potential for contamination exists, incoming surveys will be performed on equipment before it is put into service in a restricted area. Where surface contamination above the limits in **Exhibit 5** is identified, the equipment will be returned to the supplier for replacement or decontamination.

Tools and equipment will be surveyed for contamination prior to removal from a restricted area to minimize the spread of contamination. Survey extent will be established by the Project/Site RSO as appropriate to site and work conditions. Potentially contaminated tools and equipment will be wrapped or bagged until it is determined by survey that the surface contamination limits in **Exhibit 5** are not exceeded.

A contamination survey will be performed and documented for tools and equipment being released from the project. Where the surface contamination limits in **Exhibit 5** are exceeded, the items will be decontaminated before leaving the radiological work area or stored for disposal as radioactive waste. If decontamination methods are unsuccessful, a contaminated item may be retained for future use within the restricted area. If it is not feasible or cost-effective to control or decontaminate an item, the item will be disposed of as radioactive waste.

Exhibit 5: Acceptable Surface Contamination Limits

Nuclide ^a	Average ^{b,c}	Maximum ^{b,d}	Removable ^{b,e}
U-nat, U-235, U-238, and associated decay products	5,000 dpm α/100 cm ²	15,000 dpm α/100 cm ²	1,000 dpm α/100 cm ²
Transuranics, Ra-226, Ra-228, Th-230, Th-228, Pa-231, Ac-227, I-125, I-129	100 dpm/100 cm ²	300 dpm/100 cm ²	20 dpm/100 cm ²
Th-nat, Th-232, Sr-90, Ra-223, Ra-224, U-232, I-126, I-131, I-133	1,000 dpm/100 cm ²	3,000 dpm/100 cm ²	200 dpm/100 cm ²
Beta-gamma emitters (nuclides with decay modes other than alpha emission or spontaneous fission) except Sr-90 and others noted above.	5,000 dpm β-γ/100 cm ²	15,000 dpm β-γ /100 cm ²	1,000 dpm β-γ/100 cm ²

Notes:

- ^a Where surface contamination by both alpha- and beta-gamma emitting nuclides exists, the limits established for alpha and beta-gamma emitting nuclides should be applied independently.
- ^b As used in this table, dpm (disintegrations per minute) means the rate of emission by radioactive material as determined by correcting the counts per minute observed by an appropriate detector for background, efficiency, and geometric factors associated with the instrumentation.
- ^c Measurements of average contamination level should not be averaged over more than 1 square meter. For objects of less surface area, the average should be derived for each object.
- ^d The maximum contamination level applies to an area of not more than 100 cm².
- ^e The amount of removable radioactive material per 100 cm² of surface area should be determined by wiping that area with dry filter or soft absorbent paper, applying moderate pressure, and assessing the amount of radioactive material on the wipe with an appropriate instrument of known efficiency. When removable contamination on objects of less surface area is determined, the pertinent levels should be reduced proportionally and the entire surface should be wiped.

9.3.2 Survey of Vehicles and Materials

Vehicles will be surveyed upon exit from a restricted area. These surveys will include any material conveyed by the vehicle. In some cases, stockpiles of soil, debris, and miscellaneous materials may not be surveyed because the likelihood of contamination is very low. Such exceptions, if any, will be approved by the Project/Site RSO.

9.3.3 Personnel Monitoring

Individuals will be monitored for contamination on their person and on hand-carried items when exiting a restricted area. The nature and extent of the monitoring will be dictated by the radiological posting and/or the RWP. Individuals who are not qualified to self-monitor will be monitored by a radiation safety technician. No level of detectable contamination is acceptable on individuals. Hand-carried items leaving a restricted area will meet the limits in **Exhibit 5** or will be decontaminated as described in the following section. RASO will be notified of any personnel contamination incident.

9.3.4 Decontamination

Appropriate decontamination techniques may include low pressure spray (power wash), high-pressure spray, ultra-high pressure spray, scabbling, scarification, abrasive blast methods, needle gun, and removal of strippable coatings.

Contaminated equipment and tools will be brought to a designated area for decontamination. The amount of detergent or other decontamination agent used will be limited to the minimum required for the task. Decontamination agents will be collected, monitored, and properly disposed.

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10.0 Radioactive Material Control

Radioactive material controls will be established to provide positive control of radioactive material, prevent inadvertent release of radioactive material to unrestricted areas, ensure protection of members of the public and workers, and minimize the amount of radioactive waste generated during survey and remediation activities.

10.1 Discrete Radioactive Source Control

It is anticipated that discrete radioactive sources (e.g., radioactive objects that have high specific activity and localized dose rates) may be encountered during excavation and material screening activities. Enhanced radiation protection measures, such as shielding, remote handling (e.g., tongs), and extremity dosimetry, will be evaluated for use in the retrieval and handling of these sources. Discrete radioactive sources will be inventoried and assayed radiologically to characterize for disposal. Information such as a description of the item and exposure rates on contact and at distances of 10 cm and 30 cm will be recorded. Sources will be stored in locked container.

10.2 Sealed Radioactive Sources

It is not anticipated that field projects will receive radioactive material shipments other than exempt-quantity radioactive check sources, which will be controlled, stored, posted, and managed as radioactive material. Check sources will be used on field projects only for the period of time necessary to execute the planned work, will not be introduced onto a project location prior to project initiation, and will be returned to the provider immediately following the completion of planned field activities. Radioactive check sources will be packaged and shipped in accordance with U.S. Department of Transportation regulations via commercial carriers. Off-site shipment of radioactive materials other than exempt-quantity radioactive check sources by Gilbane is not anticipated.

10.3 Containment and Ventilation

Operations that routinely produce airborne radioactivity will utilize engineered containment and ventilation systems to minimize airborne releases. Containment and ventilation systems will be designed to accommodate the work area and operating conditions. Ventilation systems will be checked routinely for proper operation and airflow.

10.4 Spills

Steps will be taken to prevent and promptly clean up spills of radioactive material in either solid or liquid form. SWIM is an easy acronym that outlines the immediate actions to be taken:

- Stop the leak (e.g., shut off the valve, shut off ventilation)

- Warn others
- Isolate the area (e.g., rope off the spill site, divert flow from catch basin or drain)
- Minimize personnel exposure with PPE and safe practices

The ALARA principles of time, distance, and shielding will be used. Supplementary actions will include performing radiological surveys in the immediate and adjacent areas, including downwind. RASO will be notified of any spills involving radioactive material that occur outside an RCA.

11.0 Personal Protective Equipment

The primary level of PPE, Modified Level D, historically has been sufficient for radiological work activities. Any additional requirements will be specified in activity-specific RWPs based on the radiological conditions and field tasks required to perform planned activities.

11.1 Protective Clothing

Individuals will wear PPE commensurate with contamination hazards associated with the work area and the planned activity. Activities that require heavy physical effort or that have an increased potential for damage to PPE may require additional layers or different PPE materials, even in areas of low contamination. Protective clothing will be required for work in Contamination Areas. Site- or task-specific PPE requirements beyond the minimum traditionally used will be detailed in a corresponding RWP.

11.2 Respiratory Protection

The use of respiratory protection is not anticipated. However, in the event respiratory protection equipment is used to control inhalation of radioactive material, respiratory protection requirements and the selection of equipment will be made by the Project/Site RSO. Respiratory protection devices will be permitted for jobs where an ALARA review has been conducted and has determined that respirator use is compliant with ALARA principles.

11.2.1 Training, Medical Clearance, and Fit Testing

Before using a respirator, individuals will have (1) been trained in the proper use respiratory protection equipment and its limitations, (2) received medical clearance to wear a respirator, and (3) been respirator fit-tested within the last 12 months.

11.2.2 Respirator Selection and Use

Respirators will be selected from those approved by the National Institute for Occupational Safety and Health and or the Mine Safety and Health Administration for the contaminant(s) or situation to which the worker may be exposed. Selection will be based on the physical, chemical, and physiological properties of the contaminant, the contaminant concentration likely to be encountered, and the likely physical conditions of the workplace environment in which the respirator will be used. The potential or observed airborne radioactivity concentration also will be considered in selecting the type of respiratory protection equipment to be issued.

11.2.3 Respirator Inspection, Cleaning, and Maintenance

Each respirator will be inspected with regard to operability before, and routinely after, each use, and after cleaning. Respiratory protection equipment will be cleaned and

disinfected before being reissued. Replacement or repair will be done only by experienced persons, with parts specifically designed for the brand and model of the respirator requiring repair.

12.0 References

Gilbane (Gilbane Federal), 2020. *Phase IV Non-Time Critical Removal Action for Solid Waste Disposal Area Westside, Installation Restoration Site 12, Former Naval Station Treasure Island, San Francisco, California*. Tentatively June.

NRC (U.S. Nuclear Regulatory Commission), 1981. *Radiation Safety Surveys at Medical Institutions*. Regulatory Guide 8.23, Revision 1. January.

TriEco-Tt, 2014. *Final Historical Radiological Assessment – Supplemental Technical Memorandum, Naval Station Treasure Island, San Francisco, California*. July.

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ATTACHMENT 1

RADIATION SAFETY MANUAL

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Radiation Safety



Jerry C. Cooper, Corporate Radiation Safety Officer

05 Dec 2019

Date

Version	Changes	Affects Section	Effective Date
0.0	Initial Publication	Not Applicable	01 Jan 2016
1.0	Updated regulatory references and California license conditions	A, C, D, E, F	15 Nov 2019
2.0	Updated NRC license conditions	A, B, E, F	05 Dec 2019

Rescission
CO-ITSI-RSP-001.03 ITSI Gilbane Radiation Safety Program, Revision 3/MC1, dated 16 May 2014

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A. Introduction and Purpose

The Radiation Safety Manual consists of the Gilbane Federal ("Company") policy on radiation safety and the procedures that implement that policy. They are listed below and incorporated into the Radiation Safety Manual by reference. The policy and procedures constitute the Company's radiation safety program.

RADIATION SAFETY PROGRAM POLICY AND PROCEDURES		
Number	Title	Version
PO-RP-100	Radiation Safety	R00
PR-RP-110	Radiation Safety Program Administration	R02
PR-RP-120	Radiation Dose Limits and Personnel Monitoring	R01
PR-RP-130	Radiation Work Planning and Control	R00
PR-RP-140	Radiation Instruments and Equipment	R01
PR-RP-150	Radiological Survey and Sampling	R01
PR-RP-160	Radiation and Contamination Control	R01
PR-RP-170	Radioactive Material Control	R02
PR-RP-180	Personnel Protection and Emergency Response	R02
PR-RP-190	Radiological Records, Notifications, and Reports	R02

The Company holds radioactive material licenses issued by the U.S. Nuclear Regulatory Commission (NRC) and the State of California. These licenses are limited scope licenses and, as such, require approval of procedures by the licensing authority prior to their use under the respective license. To maintain program continuity, the Company maintains a single set of procedures for use under either license.

B. NRC Radioactive Materials License 04-29353-01

Except as specifically provided otherwise in the license, the Company conducts its program in accordance with the statements, representations, and procedures contained in the documents, including any enclosures, listed below. The NRC's regulations govern unless the statements, representations, and procedures in the Company's application and correspondence are more restrictive than the regulations.

- Application dated May 31, 2019. [NRC License Condition 25.A]

The indicated versions of the procedures referenced in Section A were approved by the NRC and incorporated into the NRC license by Amendment 08, dated November 22, 2019. A cross-reference of license conditions to the procedures is provided in Attachment 1. A cross-reference to the Company's radiation safety program, originally provided in the license application, is provided as Attachment 2.

C. California Radioactive Materials License 7948-07

Except as specifically provided otherwise by the license, the Company possesses and uses radioactive material under license in accordance with the statements, representations, and procedures contained in the documents listed below. The regulations of the California Department of Public Health (CDPH) govern unless the statements, representations, and procedures in the Company's license application and correspondence are more restrictive than the regulations.

- Application with attachments dated November 9, 2012, signed by Jay Pride, Corporate Radiation safety officer. The letters with attachments dated April 23, 2013, May 29, 2013, July 7, 2013, and July 26, 2013, all signed by Jay Pride, Corporate Radiation Safety Officer, regarding the Decommissioning Funding Plan, Financial Assurance, and commitment to follow Governor Davis' Executive Order dated 2002. [**California License Condition 13(a)**]
- Letters with attachments dated December 27, 2013, and January 9, 2014, both signed by Jay Pride, Corporate Radiation Safety Officer, regarding updated Radiation Work Instructions: ITSI-NSTI-033 dated October 1, 2013, and ITSI-NSTI-064 dated October 24, 2013. [**California License Condition 13(b)**]
- Letters with attachments dated February 25, 2014, and May 6, 2014, both signed by Jay Pride, Corporate Radiation Safety Officer, regarding the updated Radiation Safety Program and Radiation Safety Operating Procedures. [**California License Condition 13(c)**]
- Letters with attachments dated May 20, 2015, June 9, 2015, and June 23, 2015, all signed by Mike Salmon, Senior Vice President, regarding the Delegation of Authority for the Radiation Safety Officer and the updated Emergency Contact List (Revision 13 July 2015). [**California License Condition 13(d)**]
- Letter with attachments, dated September 3, 2015, regarding a revised Radiation Safety Plan, and change of address, signed by Jerry Cooper, CHP, Radiation Safety Officer. [**California License Condition 13(e)**]
- Letters with attachments, dated April 5, 2016 and April 13, 2016, both signed by Jerry C. Cooper, Corporate Radiation Safety Officer, regarding the updated Emergency Contact List (Revised April 2016). [**California License Condition 13(f)**]

- Letter with attachment, dated February 16, 2018, signed by Jerry C. Cooper, Corporate Radiation Safety Officer, regarding operational testing of survey instruments. [**California License Condition 13(g)**]
- Letter dated November 7, 2018, with attachments, letter dated December 12, 2018; and letter dated December 21, 2018, with attachments, all signed by Jerry C. Cooper, CHP, Corporate Radiation Safety Officer, regarding the appointment of a new Alternate Radiation Safety Officer, with the Delegation of Authority, updated 24-hour call list, and the Electronic Mail dated December 31, 2018, sent by Jerry C. Cooper, Corporate Radiation Safety Officer, providing additional information on estimated SNM activities, requested by RHB. [**California License Condition 13(h)**]
- Letter dated May 31, 2019, with attachments, signed by Jerry C. Coper, Corporate Radiation Safety Officer, as modified by email dated October 1, 2019, with attachments, from Jerry C. Cooper regarding updated radiation safety program procedures. [**California License Condition 13(i)**]

The indicated versions of the procedures referenced in Section A were approved by the CDPH Radiologic Health Branch and incorporated into the California license by Amendment 13, dated November 15, 2019. They replace the procedures submitted with the license application and subsequent revisions to them submitted via license correspondence (see above). A cross-reference of license conditions to the procedures is provided in Attachment 3. A cross-reference to the Company's radiation safety program, originally provided in the license application, is updated and provided as Attachment 4.

D. Acronyms and Definitions

Terms used in the Company's radiation safety program are defined in the glossary of terms in Attachment 5.

E. References

U.S. Nuclear Regulatory Commission (NRC) Radioactive Materials License No. 04-29353-01, Amendment 08, dated November 22, 2019.

State of California Radioactive Material License No. 7948-07, Amendment 13, dated November 15, 2019.

NUREG-1556, Vol. 18, Rev. 1, *Consolidated Guidance About Materials Licenses, Program-Specific Guidance About Service Provider Licenses*, NRC, August 2017.

- Section 8.10, Item 10: Radiation Safety Program
- Appendix D, Criteria for Acceptable Training and Experience for Authorized Users
- Appendix F, Radiation Monitoring Instrument Specifications and Model Survey Instrument and Air Sampler Calibration Program
- Appendix G, Model Leak Test Program
- Appendix M, Model Waste Disposal Program

Regulatory Guide 8.23, *Radiation Safety Surveys at Medical Institutions*. Revision 1. January 1981.

Gilbane Federal Policy PO-RP-100, Radiation Safety

Gilbane Federal Procedures

- PR-RP-110, Radiation Safety Program Administration
- PR-RP-120, Radiation Dose Limits and Personnel Monitoring
- PR-RP-130, Radiation Work Planning and Control
- PR-RP-140, Radiation Instruments and Equipment
- PR-RP-150, Radiological Survey and Sampling
- PR-RP-160, Radiation and Contamination Control
- PR-RP-170, Radioactive Material Control
- PR-RP-180, Personnel Protection and Emergency Response
- PR-RP-190, Radiological Records, Notifications, and Reports

F. Attachments

Attachment 1 – Program Cross-Reference: NRC License Conditions

Attachment 2 - Program Cross-Reference: NRC License Application and Supporting Correspondence

Attachment 3 – Program Cross-Reference: California License Conditions

Attachment 4 - Program Cross-Reference: California License Application and Supporting Correspondence

Attachment 5 – Glossary of Terms

Attachment 1 – Program Cross-Reference: NRC License Conditions

LICENSE CONDITION	SUMMARY OF CONDITION	PROCEDURE REFERENCE(S)
6,7,8	Licensed material types and maximum quantities	PR-RP-110: G
9,10	Authorized use of licensed material	PR-RP-110: G
11	Prohibition of taking possession of radioactive material from clients	PR-RP-110: C.5; PR-RP-170: C.1
12	Exemption from decommissioning financial assurance	Not Applicable
13	Limitation on use of licensed material at temporary job sites	PR-RP-110: C.5; PR-RP-170: C.1, C.6
14.A	Requirement to use licensed material by or under supervision of trained individuals	PR-RP-110: C.5
14.B	Designation of License Radiation Safety Officer	PO-RP-100: V
15	Restriction on use of licensed material to less than 10 CFR 30.72 quantities	PR-RP-110, G
16	14-day notification of initiation of license activities at temporary job site	PR-RP-110: C.5
17	Written agreement coordinating licensed activities for site with multiple licensees	PR-RP-110: C.5
18	Records of information important to decommissioning	PR-RP-190: C.2, D
19	Authorization for emergency departure from licensed requirements	PR-RP-180: C.5
20	30-day notification of termination of license activities at temporary job site	PR-RP-110: C.5
21	Prohibition to open sealed sources or remove sources from source holders	PR-RP-170: C.3
22	Inventory of sources and devices under license	PR-RP-170: C.2, D; PR-RP-190: G
23	Sealed source leak tests	PR-RP-170: C.3, D, G; PR-RP-190: G
24	Prohibition to import byproduct, source, and special nuclear material wastes	PR-RP-110: C.5; PR-RP-170: C.1
25	License program to be conducted in accordance with prior commitments	MA-RP-101: B

Attachment 2 – Program Cross-Reference: NRC License Application and Supporting Correspondence

A radiation safety program was established and submitted to NRC as part of the license renewal application. The program is commensurate with the scope and extent of activities for the use of licensed materials and specific to the Company's types of operations. The following table reflects commitments made in Items 7, 8, 10, and 11 of the Company's license renewal application to the NRC dated May 31, 2019, and not stated directly in the license.

PROGRAM ELEMENT	PROCEDURE REFERENCE(S)
ITEM 7 – INDIVIDUAL(S) RESPONSIBLE FOR RADIATION SAFETY PROGRAM AND THEIR TRAINING AND EXPERIENCE	
Authorized Users and Radiation Workers	
Before using licensed material, authorized users will receive the training described in Appendix D of NUREG–1556, Vol. 18, Rev. 1, <i>Consolidated Guidance About Materials Licenses: Program-Specific Guidance About Service Provider Licenses</i> .	PR-RP-110, C.2, G
ITEM 8 – TRAINING FOR INDIVIDUALS WORKING IN OR FREQUENTING RESTRICTED AREAS	
Before working in the vicinity of licensed materials, personnel will have successfully completed training commensurate with assigned duties.	PR-RP-110, C.2, G
ITEM 10 – RADIATION SAFETY PROGRAM	
Development, implementation, and maintenance of written operating and emergency procedures to address all likely situations.	PR-RP-110, C.3; G PR-RP-180, C.4, C.5
Development and implementation of an access authorization program, if required.	PR-RP-170, C.2
Development and implementation of an ALARA program.	PR-RP-110, C.1, C.2; G PR-RP-120, C.2; PR-RP-130, C.2; PR-RP-160, C.1 to C.7; PR-RP-180, C.2, C.4
Description of equipment and facilities adequate to protect personnel, the public and the environment.	PR-RP-160, C.6, C.7; PR-RP-180, C.1, C.2
Confirmation that licensed activities are conducted only by individuals qualified by training and experience.	PR-RP-110, C.1, C.2, G
Description of organization structure and individuals responsible for ensuring day-to-day oversight of the radiation safety program.	PR-RP-110, C.1, C.2
Establishment and management of a radiation safety and decommissioning records system.	PR-RP-190, C.1, C.2

Attachment 2 – Program Cross-Reference: NRC License Application and Supporting Correspondence

PROGRAM ELEMENT	PROCEDURE REFERENCE(S)
Implementation of an audit program to ensure that, at least annually, the radiation safety program is reviewed.	PR-RP-110, C.4
Development of a sample agreement letter between applicant and the applicant's customer acknowledging the use of radioactive materials at the customer's site (see NRC License Condition 17).	PR-RP-110, C.5
Development and implementation of a program to ensure the security and control of licensed material.	PR-RP-170, C.2
Operating and Emergency Procedures	
Procedure for obtaining an agreement with customers outlining the responsibilities of both the customer and service provider, when performing service operations at a customer's facility.	PR-RP-110, C.5
Instructions for handling and using licensed materials.	PR-RP-170, C.1 to C.6
Instructions for maintaining security during storage and transportation.	PR-RP-170, C.2, C.4
Instructions to keep licensed material under control and immediate surveillance during use.	PR-RP-170, C.1 to C.6
Instructions for posting areas and labeling containers	PR-RP-160, C.3 PR-RP-170, C.4
Steps to take to keep radiation exposures ALARA.	PR-RP-110, C.1, C.2; G PR-RP-120, C.2; PR-RP-130, C.1 to C.4; PR-RP-160, C.1 to C.7; PR-RP-180, C.2, C.4
Steps to maintain accountability during use.	PR-RP-170, C.1 to C.6
Steps to control access to work sites.	PR-RP-160, C.1 to C.4
Steps to take and whom to contact when an emergency occurs.	PR-RP-180, C.4; PR-RP-190, C.3
Instructions for using remote handling tools when handling sealed sources, except low-activity calibration sources.	PR-RP-170, C.3
Methods and occasions for conducting radiation surveys, including surveys for detecting contamination.	PR-RP-150, C.1 to C.7
Procedures to minimize personnel exposure during routine use and in the event of an incident, including exposures from inhalation and ingestion of licensed unsealed materials.	PR-RP-130, C.1 to C.4; PR-RP-160, C.1 to C.7; PR-RP-180, C.1 to C.4

Attachment 2 – Program Cross-Reference: NRC License Application and Supporting Correspondence

PROGRAM ELEMENT	PROCEDURE REFERENCE(S)
Methods and occasions for locking and securing stored licensed materials.	PR-RP-170, C.2
Procedures for personnel monitoring, including bioassays, and the use of personnel monitoring equipment.	PR-RP-120, C.1 to C.8
Procedures for transporting licensed materials to temporary job sites, packaging of licensed materials for transport in vehicles (private or common carrier), placarding of vehicles when needed, and physically securing licensed materials in transport vehicles during transportation to prevent accidental loss, tampering, or unauthorized removal.	PR-RP-170, C.4
Procedures for picking up, receiving, and opening packages containing licensed materials, in accordance with 10 CFR 20.1906.	PR-RP-170, C.1
Instructions for maintaining records in accordance with the regulations and the license conditions.	PR-RP-190, C.1
Procedures for identifying and reporting to NRC defects and non-compliance as required by 10 CFR 21.21(a).	PR-RP-190, C.3, C.4
Procedures and actions to be taken in an emergency situation that will cover all likely scenarios, including actions to prevent the spread of contamination and minimize inhalation and ingestion of licensed materials and actions to obtain suitable radiation survey instruments.	PR-RP-140, C.1; PR-RP-180, C.1 to C.5
Instructions for the proper storage and disposal of radioactive waste.	PR-RP-170, C.5
Procedures to be followed in the event of uncontrolled release of radioactive unsealed licensed material to the environment, including notification of the RSO, NRC, and other Federal and State agencies.	PR-RP-180, C.4; PR-RP-190, C.3
Procedures for identifying and reporting to NRC incident notifications (see Table 8-2 of NUREG-1556, Vol. 18, Rev. 1, <i>Consolidated Guidance About Materials Licenses: Program-Specific Guidance About Service Provider Licenses</i> , for a description of the typical incident notifications required by NRC regulations).	PR-RP-190, C.3, C.4

Attachment 2 – Program Cross-Reference: NRC License Application and Supporting Correspondence

PROGRAM ELEMENT	PROCEDURE REFERENCE(S)
<p>Procedures for the implementation of and adherence to good health physics practices while performing service operations:</p> <ul style="list-style-type: none"> – Minimization of distance to areas, to the extent practicable, where licensed materials are used and stored – Maximization of survey frequency, within reason, to enhance detection of contamination – Segregation of radioactive material in waste storage areas – Segregation of sealed sources and tracer materials to prevent cross-contamination – Separation of radioactive material from explosives – Separation of potentially contaminated areas from clean areas by barriers or other controls. 	<p>PR-RP-110, C.2; G PR-RP-130, C.1 to C.4; PR-RP-150, C.1 to C.7; PR-RP-160, C.1 to C.7; PR-RP-170, C.3, C.5;</p>
Method for reviewing the entire radiation safety program annually.	PR-RP-110, C.4
Material Receipt and Accountability	
We will develop, implement, and maintain procedures for ensuring accountability of licensed materials at all times.	PR-RP-170, C.2, C.5
Radiation Monitoring Instruments	
We will use instruments that meet the radiation monitoring instrument specifications published in Appendix F of NUREG-1556, Vol. 18, Rev. 1, <i>Consolidated Guidance About Materials Licenses: Program-Specific Guidance About Service Provider Licenses</i> . We reserve the right to upgrade our survey instruments as necessary.	PR-RP-140, C.1 to C.7
Surveys	
We will conduct surveys and maintain contamination levels in accordance with the survey frequencies and contamination levels published in Section 8.10.4 of NUREG-1556, Vol. 18, Rev. 1, <i>Consolidated Guidance About Materials Licenses: Program-Specific Guidance About Service Provider Licenses</i> .	PR-RP-150, C.3, C.6 PR-RP-160, C.2 to C.5
Leak Tests	
We will follow the model procedures in Appendix G of NUREG-1556, Vol. 18, Rev. 1, <i>Consolidated Guidance About Materials Licenses: Program-Specific Guidance About Service Provider Licenses</i> .	PR-RP-170, C.3, G

Attachment 2 – Program Cross-Reference: NRC License Application and Supporting Correspondence

PROGRAM ELEMENT	PROCEDURE REFERENCE(S)
Occupational Dosimetry	
We will monitor individuals in accordance with the criteria in Section 8.10.6 of NUREG-1556, Vol. 18, Rev. 1, <i>Consolidated Guidance About Materials Licenses: Program-Specific Guidance About Service Provider Licenses</i> .	PR-RP-120, C.3
Public Dose	
No response is required from the applicant in a license application.	PR-RP-150, C.5
Transportation	
No response is needed from applicants during the licensing phase.	PR-RP-170, C.4, G
Maintenance	
We will implement and maintain procedures for conducting routine maintenance of devices according to each manufacturer's (or distributor's) written recommendations and instructions.	PR-RP-170, C.3
We will have the device manufacturer (or distributor) or other person authorized by NRC or an Agreement State perform non-routine maintenance.	PR-RP-170, C.3
Audit and Review of Program	
No response is needed from applicants during the licensing phase.	PR-RP-110, C.4
Security Program for Category 1 and Category 2 Materials	
No response is required from applicant or licensee.	PR-RP-170, C.2
ITEM 11 – WASTE MANAGEMENT	
We will use the model waste procedures published in Appendix M of NUREG-1556, Vol. 18, Rev. 1, <i>Consolidated Guidance About Materials Licenses: Program-Specific Guidance About Service Provider Licenses</i> .	PR-RP-170, C.5

Attachment 3 – Program Cross-Reference: California License Conditions

LICENSE CONDITION	SUMMARY OF CONDITION	DOCUMENT and SECTION(S)
6,7,8	Licensed material types and maximum quantities	PR-RP-110: G
9	Authorized use of licensed material	PR-RP-110: G
10	Radioactive material use only in areas not under exclusive federal jurisdiction	PR-RP-110: C.5
11	Annual fee for sources of radioactive material	Not Applicable
12	Individuals authorized to use/supervise use radioactive material under license	PR-RP-110: C.5
13	License program to be conducted in accordance with prior commitments	MA-RP-101: C
14	Designation of License Radiation Safety Officer	PO-RP-100: V
15	Sealed source leak tests	PR-RP-170: G; and PR-RP-190: G
16	Limitation on use of licensed material at temporary job sites	PR-RP-110: C.5; and PR-RP-170: C.1, C.6
17(a)	14-day notification of initiation of license activities at temporary job site	PR-RP-110: C.5
17(b)	30-day notification of termination of license activities at temporary job site	PR-RP-110: C.5
18	Written agreement coordinating licensed activities for site with multiple licensees	PR-RP-110: C.5
19	Records of information important to decommissioning	PR-RP-190: C.2, D
20	Authorization to transport licensed material in accordance with 17 CCR 30373	PR-RP-170: G
21	Restriction on total mass of special nuclear material	PR-RP-110, G
22	Determination of decision on jurisdictional status	PR-RP-110: C.5
23	Acceptable financial instrument for decommissioning	Not Applicable
24	Annual low-level radioactive waste reports	PR-RP-190: C.6
25	Vacating any (permanent) address of use	Not Applicable
26	Records and documents available for inspection	PR-RP-190: C.1

Attachment 4 – Program Cross-Reference: California License Application and Supporting Correspondence

A radiation safety program is established and submitted to the CDPH Radiologic Health Branch as part of the license application. The program is commensurate with the scope and extent of activities for the use of licensed materials and specific to the Company's types of operations. The following table updates Items 6 through 13 of the Company's license application to CDPH dated November 9, 2012.

PROGRAM ELEMENT	DOCUMENT REFERENCES(S)
Item 6 – Radiation Detection Instruments	
(Discussion in original license application remains current; procedure reference updated regarding additional information and descriptions of typical instruments.)	PR-RP-140, C.1, C.2
Item 7 – Method, frequency, and standards used in calibrating instruments listed above	
(Discussion in original license application remains current; procedure reference updated regarding Company requirements for instrument operation, maintenance, and calibration.)	PR-RP-140, C.3 to C.7
Item 8 – Personnel monitoring and bioassay procedures	
(Discussion in original license application remains current; procedure reference updated regarding personnel monitoring and bioassay procedures.)	PR-RP-120, C.1 to C.8
Item 9 – Facilities and equipment	
(Discussion in original license application remains current; procedure reference added regarding personnel protection and engineering controls.)	PR-RP-160, C.6, C.7; PR-RP-180, C.1, C.2
Item 10 – Radiation safety program	
Development and implementation of an ALARA program	PR-RP-110, C.1, C.2; G PR-RP-120, C.2; PR-RP-130, C.2; PR-RP-160, C.1 to C.7; PR-RP-180, C.2, C.4
Description of equipment and facilities adequate to protect personnel, the public, and the environment	PR-RP-160, C.6, C.7; PR-RP-180, C.1, C.2
Confirmation that licensed activities are conducted only by individuals qualified by training and experience	PR-RP-110, C.1, C.2; G

Attachment 4 – Program Cross-Reference: California License Application and Supporting Correspondence

PROGRAM ELEMENT	DOCUMENT REFERENCES(S)
Item 10 – Radiation safety program (cont.)	
Development and maintenance of written operating and emergency procedures	PR-RP-110, C.3; G PR-RP-180, C.4, C.5
Implementation of an audit program to ensure that, at least annually, the radiation safety program is reviewed	PR-RP-110, C.4
Description of organization structure and individuals responsible for ensuring day-to-day oversight of the radiation safety program	PR-RP-110, C.1, C.2
Establishment and management of a radiation safety and decommissioning records system	PR-RP-190, C.1, C.2
Methods or procedures for preventing the release of contaminated material and equipment	PR-RP-150, C.3, C.6; PR-RP-160, C.1 to C.7
Methods or procedures for preventing personnel contamination. Radiation safety procedures and the authorized users responsibilities unique to each type of service operation requested in the application	PR-RP-150, C.3; PR-RP-160, C.1 to C.7; PR-RP-180, C.1, C.3
Radiation safety procedures	PR-RP-XXX procedures
Equipment, techniques, and corresponding radiation safety procedures associated with providing services involving either sealed sources or unsealed materials.	PR-RP-170, C.1 to C.6
We will use instruments that meet the radiation monitoring instrument specification published in Appendix J of NUREG-1556, Vol. 18, “Consolidated Guidance About Service Provider Licenses,” dated November 2000. Additionally, we will implement the model survey meter calibration program published in Appendix J of NUREG-1556, Vol. 18, “Consolidated Guidance About Service Provider Licenses,” dated November 2000. We reserve the right to upgrade our survey instruments as necessary.	PR-RP-140, C.1 to C.7
Ordering licensed material and package receipt and opening will follow the model procedures in Appendix K of NUREG-1556, Vol. 18, “Consolidated Guidance About Service Provider Licenses,” dated November 2000	PR-RP-170, C.1
For unsealed licensed material, submit a description of procedure(s) for ensuring material accountability	PR-RP-170, C.2, C.5

Attachment 4 – Program Cross-Reference: California License Application and Supporting Correspondence

PROGRAM ELEMENT	DOCUMENT REFERENCES(S)
Item 10 – Radiation safety program (cont.)	
We will have a prospective evaluation and determine that unmonitored individuals are not likely to receive, in one year, a radiation dose in excess of 10% of the allowable limits in 10 CFR Part 20, or we will monitor individuals in accordance with the criteria in the section entitled ‘Occupational Dose’ in NUREG-1556, Vol. 18, “Consolidated Guidance About Service Provider Licenses,” dated November 2000	PR-RP-120, C.3
Contract with an outside group for bioassay services. Provide a commitment that each vendor is licensed or otherwise authorized by NRC or Agreement State	PR-RP-120, C.4
Procedure for obtaining an agreement with customers outlining the responsibilities of both the customer and service provider, when performing service operations at a customer’s facility	PR-RP-110, C.5
Instructions for handling and using licensed materials	PR-RP-170, C.1 to C.6
Instructions for maintaining security during storage and transportation	PR-RP-170, C.2, C.4
Instructions to keep licensed material under control and immediate surveillance during use	PR-RP-170, C.1 to C.6
Steps to take to keep radiation exposures ALARA	PR-RP-110, C.1, C.2, G; PR-RP-120, C.2; PR-RP-130, C.1 to C.4; PR-RP-160, C.1 to C.7; PR-RP-180, C.2, C.4
Steps to maintain accountability during use	PR-RP-170, C.1 to C.6
Steps to control access to work sites	PR-RP-160, C.1 to C.4
Steps to take and whom to contact when an emergency occurs	PR-RP-180, C.4 PR-RP-190, C.3
Instructions for using remote handled tools when handling sealed sources, except low-activity calibration sources	PR-RP-170, C.3
Methods and occasions for conducting radiation surveys, including surveys for detecting contamination	PR-RP-150, C.1 to C.7

Attachment 4 – Program Cross-Reference: California License Application and Supporting Correspondence

PROGRAM ELEMENT	DOCUMENT REFERENCES(S)
Item 10 – Radiation safety program (cont.)	
Procedures to minimize personnel exposure during routine use and in the event of an incident, including exposures from inhalation and ingestion of licensed unsealed materials	PR-RP-130, C.1 to C.4; PR-RP-160, C.1 to C.7; PR-RP-180, C.1 to C.4
Methods and occasions for locking and securing stored licensed materials	PR-RP-170, C.2
Procedures for implementation of and adherence to good health physics practices while performing service operations: - Minimization of distance to areas, to the extent practicable, where licensed materials are used and stored - Maximization of survey frequency, within reason, to enhance detection of contamination - Segregation of radioactive material in waste storage areas - Segregation of sealed sources and tracer materials to prevent cross-contamination - Separation of radioactive material from explosives - Separation of potentially contaminated areas from clean areas by barriers or other controls	PR-RP-110, C.2; G PR-RP-130, C.1 to C.4; PR-RP-150, C.1 to C.7; PR-RP-160, C.1 to C.7; PR-RP-170, C.3, C.5;
Personnel monitoring, including bioassays, and the use of personnel monitoring equipment	PR-RP-120, C.1 to C.8
Transportation of licensed materials to temporary job sites, packaging of licensed materials for transport in vehicles, placarding of vehicles when needed, and physically securing licensed materials in transport vehicles during transportation to prevent accidental loss, tampering, or unauthorized removal	PR-RP-170, C.4
Procedures for picking up, receiving, and opening packages containing licensed materials, in accordance with 10 CFR 20.1906	PR-RP-170, C.1
Instructions for maintaining records in accordance with regulations and license conditions	PR-RP-190, C.1
Procedures for identifying and reporting to State defects and noncompliance as required by 10 CFR 21.21(a) of this chapter	PR-RP-190, C.3, C.4

Attachment 4 – Program Cross-Reference: California License Application and Supporting Correspondence

PROGRAM ELEMENT	DOCUMENT REFERENCES(S)
Item 10 – Radiation safety program (cont.)	
Procedures and actions to be taken if a sealed source is ruptured, including actions to prevent the spread of contamination and minimize inhalation and ingestion of licensed materials and actions to obtain suitable radiation survey instruments	PR-RP-140, C.1; PR-RP-180, C.1 to C.5
Instructions for the proper storage and disposal of radioactive waste	PR-RP-170, C.5
Procedures to be followed in the event of uncontrolled release of radioactive unsealed licensed material to the environment, including notification of the RSO, state and other Federal agencies	PR-RP-180, C.4; PR-RP-190, C.3
Procedures for identifying and reporting to State defects and noncompliance. See Table 8.4, which describes the typical incident notifications required by State regulations.	PR-RP-190, C.3, C.4
“We will survey our facility and maintain contamination levels in accordance with the survey frequencies and contamination levels published in NUREG-1556, Vol. 18, ‘ <i>Consolidated Guidance About Materials Licenses: Program-Specific Guidance About Service Provider Licenses</i> ,’ dated November 2000.”	PR-RP-150, C.3, C.6; PR-RP-160, C.2 to C.5
“Leak testing will follow the model procedures in Appendix O of NUREG-1556, Vol. 18, ‘ <i>Consolidated Guidance About Materials Licenses: Program-Specific Guidance About Service Provider Licenses</i> ,’ dated November 2000”	PR-RP-170, C.3, G
“We will have the device manufacturer (or distributor) or other person authorized by NRC or an Agreement State to perform non-routine maintenance on our devices.”	PR-RP-170, C.3
Before using licensed material, authorized users will receive training described in Appendix H of NUREG-1556, Vol. 18, ‘ <i>Consolidated Guidance About Material Licenses: Program-Specific Guidance About Service Provider Licenses</i> ,’ dated November 2000	PR-RP-110, C.2, G

Attachment 4 – Program Cross-Reference: California License Application and Supporting Correspondence

PROGRAM ELEMENT	DOCUMENT REFERENCES(S)
Item 10 – Radiation safety program (cont.)	
“Before using licensed materials, ancillary personnel will have successfully completed the classroom training portion of the training course described in Appendix H of NUREG-1556, Vol. 18, <i>‘Consolidated Guidance About Material Licenses: Program-Specific Guidance About Service Provider Licenses,’</i> dated November 2000.”	PR-RP-110, C.2, G
Item 11 – Effluent and environmental monitoring	
(Discussion in the original license application remains current; procedure reference updated regarding effluent and environmental monitoring.)	PR-RP-150, C.1 to C.7
Item 12 – Waste disposal	
(Discussion in the original license application remains current; procedure reference added regarding waste disposal.)	PR-RP-170, C.5, C.6
Item 13 – Decommissioning and decontamination plans	
(Discussion in the original license application remains current.)	Not Applicable

Attachment 5 – Glossary of Terms

The following terms are defined as they are used in the Company's radiation safety program. Many definitions are quoted or adapted from the NRC Glossary <http://www.nrc.gov/reading-rm/basic-ref/glossary.html>.

Administrative Controls. Formal procedures or rules established and monitored by the Company to ensure safety and controlled operations.

Administrative Dose Limit. A dose limit established by the Company for the purpose of maintaining personnel radiation dose below regulatory limits.

Agreement State. A State that has signed an agreement with the NRC authorizing the State to regulate certain uses of radioactive materials within the State.

Airborne Radioactivity Area. A room, enclosure, or area in which airborne radioactive materials, composed wholly or partially of licensed material, exist in concentrations that (1) exceed the derived air concentration (DAC) limits, or (2) would result in an individual present in the area without respiratory protection exceeding, during those hours while present in the area, 0.6 percent of the annual limit on intake (ALI) or 12 DAC-hours.

Annual Limit on Intake (ALI). The derived limit for the amount of radioactive material taken into the body of an adult worker by inhalation or ingestion in a year. ALI is the smaller value of intake of a given radionuclide in a year by the "reference man" that would result in a committed effective dose equivalent (CEDE) of 5 rem or a committed dose equivalent (CDE) of 50 rem to any individual organ or tissue. ALI values for intake by ingestion and inhalation are given in Table 1, Columns 1 and 2, of Appendix B to 10 CFR 20.

As Low as Reasonably Achievable (ALARA). Efforts made to maintain individual exposures to radiation as far below regulatory limits as is practical consistent with the purpose for which the work is undertaken.

Background Radiation. The natural radiation that is always present in the environment. It includes cosmic radiation from the sun and stars; terrestrial radiation (e.g., radon), which comes from the Earth; global fallout from nuclear testing and accidents; and the internal radiation that exists in all living things.

Bioassay. The determination of kinds, quantities, or concentrations (and, in some cases, locations) of radioactive material in the human body, whether by direct measurement (in vivo counting) or by analysis and evaluation of materials excreted or removed (in vitro) from the human body.

Attachment 5 – Glossary of Terms

Breathing Zone. An individual's breathing environment (i.e., the vicinity of the nose and mouth), where it is generally assumed that a contaminant's concentration is homogeneous and equivalent to the concentration inhaled by the worker.

Byproduct Material. Any radioactive material (except enriched uranium or plutonium) produced by a nuclear reactor. It also includes the tailings or wastes produced by the extraction or concentration of uranium or thorium or the fabrication of fuel for nuclear reactors. Additionally, it is any material that has been made radioactive through the use of a particle accelerator, or any discrete source of radium-226 used for a commercial, medical, or research activity.

California Code of Regulations (CCR). The codification of the general and permanent rules and regulations (sometimes called administrative law) announced in the California Regulatory Notice Register by California state agencies. Of specific interest here is Title 17, Division 1, Chapter 5, Subchapter 4, *Radiation*; and more specifically Groups 1 through 4, consisting of Sections 30100 to 30373. Regulations are cited using title and section number. For example, 17 CCR 30253 refers to Title 17, Section 30253.

- Title 17, Public Health
 - Section 30100, *General Definitions*
 - Section 30253, *Standards for Protection Against Radiation*
 - Section 30255, *Notices, Instructions, and Reports to Personnel*
 - Section 30256, *Vacating Installations: Records and Notice*
 - Section 30275, *Surveys and Tests*
 - Section 30293, *Records*
 - Section 30295, *Notification of Incidents*
 - Section 30373, *Transportation Regulations*

Check Source. A radioactive source, not necessarily calibrated, that is used to confirm the continuing satisfactory operation of an instrument.

Code of Federal Regulations (CFR). The codification of the general and permanent rules and regulations (sometimes called administrative law) published in the Federal Register by the executive departments and agencies of the federal government of the United States. Of interest here are specific chapters in Title 10, *Energy*; Title 29, *Labor*; and Title 49, *Transportation*. Regulations are cited using title and part number. For example, "10 CFR 20" refers to Title 10, Part 20.

- Title 10, Chapter I, Nuclear Regulatory Commission:
 - Part 19, *Notices, Instructions and Reports to Workers; Inspection and Investigations.*
 - Part 20, *Standards for Protection Against Radiation.*

Attachment 5 – Glossary of Terms

- Part 30, *Rules of General Applicability to Domestic Licensing of Byproduct Material*.
- Part 40, *Domestic Licensing of Source Material*.
- Part 70, *Domestic Licensing of Special Nuclear Material*.
- Part 71, *Packaging and Transportation of Radioactive Material*.
- Title 29, Chapter XVII, Occupational Safety and Health Administration:
 - Part 1910, Occupational Safety and Health Standards
- Title 49, Chapter I, Pipeline and Hazardous Materials Safety Administration:
 - Part 172, *Hazardous Materials Table, Special Provisions, Hazardous Materials Communications, Emergency Response Information, Training Requirements, and Security Plans*
 - Part 173, *Shippers – General Requirements for Shipments and Packagings*
 - Part 177, *Carriage by Public Highway*

Committed Dose Equivalent (CDE). The dose to some specific organ or tissue of reference that will be received from an intake of radioactive material by an individual during the 50-year period following the intake.

Committed Effective Dose Equivalent (CEDE). The sum of the products of the CDEs for each of the body organs or tissues that are irradiated multiplied by the weighting factors applicable to each of those organs or tissues.

Contamination. Undesirable radioactive material that is either airborne or deposited in (or on the surface of) structures, objects, soil, water, or living organisms (people, animals, or plants) in a concentration that may harm people, equipment, or the environment.

Contamination Area. Any area in which removable activity exceeds the acceptable surface contamination levels specified in Table 3 of Regulatory Guide 8.23, but does not exceed 100 times those values.

Controlled Area. See “Radiologically Controlled Area.”

Cosmic Radiation. A source of natural background radiation, which originates in outer space and is composed of penetrating ionizing radiation (both particulate and electromagnetic).

Declared Pregnant Woman. A woman who is a radiation worker and has voluntarily informed her employer, in writing, of her pregnancy and the estimated date of conception.

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Deep-Dose Equivalent (DDE). The external whole-body exposure dose equivalent at a tissue depth of 1 centimeter (cm) averaged over an area of 1 square centimeter (cm²) (1,000 milligrams per square centimeter [mg/cm²]).

Derived Air Concentration (DAC). The concentration of a given radionuclide in air which, if breathed by the “reference man” for a working year of 2,000 hours under conditions of light work (with an inhalation rate of 1.2 cubic meters of air per hour), results in an intake of one ALI. DAC values are specified in Appendix B of 10 CFR 20.

Derived Air Concentration-Hour (DAC-hour). The product of the concentration of radioactive material in air (expressed as a fraction or multiple of the DAC for each radionuclide) and the time of exposure to that radionuclide, in hours. Two thousand (2,000) DAC-hours represents one ALI, equivalent to a CEDE of 5 rem.

Dose Equivalent. A measure of the biological damage to living tissue as a result of radiation exposure. Also known as the “biological dose,” the dose equivalent is calculated as the product of absorbed dose in tissue multiplied by a quality factor and then sometimes multiplied by other necessary modifying factors at the location of interest. The dose equivalent is expressed numerically in rem (see 10 CFR 20.1003).

Dosimeter. A small device, such as a thermoluminescent dosimeter (TLD) or a self-reading dosimeter (e.g., pocket ionization chamber), worn by a single individual and used to measure and record the total accumulated personal dose of ionizing radiation. Also commonly referred to as dosimetry.

Engineering Controls. A general class of devices and associated methods used to reduce individual exposure to radiation and radioactive materials. Examples of engineering controls are local ventilation, glove boxes, remote handling tools, and enclosures.

Exposure Rate. The amount of radiation (exposure) delivered at a given point per unit of time. Typical units are microroentgens per hour (μR/hr).

External Radiation. Exposure to ionizing radiation when the radiation source is located outside the body.

Extremity. The hand, elbow, arm below the elbow, foot, knee, or leg below the knee.

Fixed Contamination. See “Surface Contamination, Fixed.”

Attachment 5 – Glossary of Terms

High Contamination Area. Any area in which removable activity exceeds or is likely to exceed 100 times the acceptable surface contamination levels specified in Table 3 of Regulatory Guide 8.23.

High Efficiency Particulate Air (HEPA). Being, using, or containing a filter usually designed to remove 99.97% of airborne particles measuring 0.3 microns or greater in diameter passing through it.

High Radiation Area. Any area with a dose rate greater than 100 millirem (mrem) in one hour 30 cm from the source or from any surface through which the ionizing radiation penetrates.

Hot Spot. The region in a radiation area or a contamination area where the level of radiation or contamination is significantly greater than in neighboring regions in the area. Note: Use of this term to identify areas outside a radiologically controlled area (RCA) may generate confusion regarding whether the proper radiological controls are being applied.

Instrument Efficiency. The rate of particles detected by the instrument relative to the surface (2π) particle emission rate of the calibration source. The surface particle emission rate is a value measured and certified by the source manufacturer.

Internal Deposition. Radioactive material that has been taken into and deposited in the body through inhalation, ingestion, absorption through the skin, or through wounds.

International Standards Organization (ISO). An international standard-setting body composed of representatives from various national standards organizations.

Lens Dose Equivalent (LDE). The external exposure dose equivalent to the lens of the eye at a tissue depth of 0.3 cm averaged over an area of 1 cm^2 (300 mg/cm^2).

Licensing Authority. For the NRC license, NRC, Region IV; or for the California license, the California Department of Public Health, Radiologic Health Branch.

Licensed Material. Radioactive material that is received, possessed, used, transferred, or disposed of under a general license or specific license issued by the NRC or an Agreement State.

Multi-Agency Radiation Survey and Site Investigation Manual (MARSSIM). A multi-agency consensus document that was developed collaboratively by four Federal agencies having authority and control over radioactive materials: Department of Defense (DOD), Department of Energy (DOE), Environmental Protection Agency (EPA), and NRC. The objective of MARSSIM is

Attachment 5 – Glossary of Terms

to describe a consistent approach for planning, performing, and assessing building surface and surface soil final status surveys to meet established dose or risk-based release criteria, while at the same time encouraging an effective use of resources.

Member of the Public. An individual not receiving or expected to receive an occupational dose.

Minimum Detectable Concentration (MDC): An *a priori*-determined minimum concentration of radioactivity that a specific instrument and technique are expected to detect under actual conditions of use 95% of the time. Note: The definition of this term may be modified to accommodate project-specific applications.

Minor. An individual less than 18 years of age.

National Institute of Standards and Technology (NIST). Formerly known as the National Bureau of Standards, a unit of the U.S. Department of Commerce that promotes and maintains measurement standards.

National Voluntary Laboratory Accreditation Program (NVLAP). A system for accrediting laboratories found competent to perform specific tests or calibrations or types of tests or calibrations.

(U.S.) Nuclear Regulatory Commission (NRC). The federal government agency whose role is to protect public health and safety related to nuclear energy.

NUREG. An NRC technical report designation.

Occupational Dose. The dose of ionizing radiation received by radiation workers in the course of employment. Occupational dose does not include any dose received from background radiation, as a patient from medical practices, from voluntary participation in medical research programs, or as a member of the public.

Physical Controls. Barriers, signs, gates, doors, fences, etc., used to control the access of personnel into areas of a facility (e.g., Restricted Areas, High Radiation Areas, etc.).

Policy. A statement of intent or management directive that is implemented as a procedure.

Posting. A standardized sign or label that bears the standard trefoil symbol in magenta, purple, or black on a yellow background and provides information concerning a specific radiological hazard.

Attachment 5 – Glossary of Terms

Procedure. A process-specific document detailing the step(s) taken and/or method(s) used to achieve policy objectives.

Protective Clothing. Clothing designed to prevent personnel contamination. Standard protective clothing includes Tyvek coveralls; plastic booties with overshoes; and latex and/or rubber anti-contamination gloves.

Public Dose. The dose received by a member of the public from exposure to radiation or to radioactive material released by the Company, or to any other source of radiation under the control of the Company. Public dose does not include occupational dose or doses received from background radiation, from any medical administration the individual has received for the purpose of medical diagnosis or therapy, or from voluntary participation in medical research programs.

Qualified. Possessing the education, experience, and skills; and/or having received classroom, on-the-job, and/or hands-on training sufficient to achieve and maintain proficiency, as determined by the Corporate RSO or Project/Site RSO.

Rad (radiation absorbed dose). One of the two units used to measure the amount of radiation absorbed by an object or person, known as the “absorbed dose,” which reflects the amount of energy that radioactive sources deposit in materials through which they pass. The radiation-absorbed dose (rad) is the amount of energy (from any type of ionizing radiation) deposited in any medium (e.g., water, tissue, air). An absorbed dose of 1 rad means that 1 gram of material absorbed 100 ergs of energy (a small but measurable amount) as a result of exposure to radiation. The related international system unit is the gray (Gy), where 1 Gy is equivalent to 100 rad.

Radiation. Energy emitted from a source. Energy in the form of waves (e.g., gamma rays, x-rays) or particles (e.g., alpha particles, beta particles, neutrons, high-speed electrons) capable of producing ions is referred to as ionizing radiation. Energy in the form of radio waves, microwaves, or visible, infrared, or ultraviolet light with insufficient energy to cause ionization is referred to as non-ionizing radiation.

Radiation Area. Any area with a radiation level greater than 5 mrem in one hour at 30 cm from the source or from any surface through which the radiation penetrates.

Radiation Protection Personnel. Project/site personnel trained in radiation protection who are responsible for the field implementation of the radiation safety program.

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Radiation Safety Manual. A reference document (MA-RP-101) incorporating the Company's policy on radiation safety and the procedures that implement that policy. The policy and procedures constitute the Company's radiation safety program.

Radiation Safety Officer (RSO). A Company-designated individual qualified by training and experience in radiation protection who ensures that activities involving radiation and radioactive material are performed safely in accordance with Company policies and procedures, and that regulatory requirements are met.

Radiation Work Permit (RWP). A document prepared by radiation protection personnel to inform radiation workers of the radiological conditions that exist in the work area and the radiological requirements and protective measures to be applied to the job.

Radiation Worker. An individual receiving or expected to receive an occupational dose.

Radioactive Contamination. See "Contamination."

Radioactive Material. Any solid, liquid, or gas that emits radiation spontaneously.

Radioactive Material Area (RMA). An area or room in which licensed material is used or stored and which contains radioactive material in an amount exceeding 10 times (100 times for natural uranium or thorium) the quantity of such material specified in Appendix C of 10 CFR 20.

Radiologically Controlled Area (RCA). An area in which radiation levels could result in an individual receiving a total effective dose equivalent (TEDE) in excess of 100 mrem in a single year (excluding natural background and medical exposures). For external sources, the RCA typically is posted when the radiation level exceeds 50 μ R/hr at a distance of 30 cm from the radiation source or from any surface that the radiation penetrates. However, this radiation level may be modified at the discretion of the Project/Site RSO based on accurately assessed occupancy factors.

Record. Information created, received, and maintained as evidence and information by an organization or person, in pursuance of legal obligations, regulatory compliance or the transaction of business.

Records of Information Important to Decommissioning. Information identifying radionuclides, quantities, forms, and concentrations important to decommissioning the facility, equipment or site. This includes: (1) records of spills or other occurrences involving the spread of contamination in and around the facility, equipment, or site, including descriptions of any

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instances when contamination remains after any cleanup procedures or when there is reasonable likelihood that contaminants may have spread to inaccessible areas, as for example, possible seepage into porous materials such as concrete; (2) as-built drawings and modifications of structures and equipment in restricted areas where radioactive materials are used and/or stored, and of locations of possible inaccessible contamination such as buried pipes which may be subject to contamination; (3) designated and formerly designated restricted areas; and (4) areas outside of restricted areas that contain material, including buried waste, such that, if the license expired, the licensee would be required to decontaminate the area to unrestricted release levels.

Regulatory. Related to requirements specified in the Company's radioactive material licenses and governing federal and state regulations, specifically Title 10, Code of Federal Regulations, Parts 19, 20, 30, 40, 70, and 71; and Title 17, California Code of Regulations, Division 1, Chapter 5, Subchapter 4. See also "Code of Federal Regulations" and "California Code of Regulations."

Rem (Roentgen equivalent man). One of the two standard units used to measure the dose equivalent (or effective dose), which combines the amount of energy (from any type of ionizing radiation that is deposited in human tissue), along with the medical effects of the given type of radiation. For beta and gamma radiation, the dose equivalent is the same as the absorbed dose. By contrast, the dose equivalent is larger than the absorbed dose for alpha and neutron radiation, because these types of radiation are more damaging to the human body. Thus, the dose equivalent (in rem) is equal to the absorbed dose (in rad) multiplied by the quality factor of the type of radiation (see 10 CFR 20.1004). The related international system unit is the sievert (Sv), where 100 rem is equivalent to 1 Sv.

Removable Contamination. See "Surface Contamination, Removable."

Restricted Area. Any area to which access is controlled for the protection of individuals from exposure to radiation and radioactive materials.

Roentgen (R). A unit of exposure to ionizing radiation. It is the amount of gamma or x-rays required to produce ions resulting in a charge of 0.000258 coulombs/kilogram of air under standard conditions.

Sealed Source. Any radioactive material that is permanently encased in a capsule or bonded and in a solid form designed to prevent leakage or escape of the material.

Shallow-Dose Equivalent (SDE). The external exposure dose equivalent to the skin or an extremity at a tissue depth of 0.007 cm averaged over an area of 1 cm² (7 mg/cm²).

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Source Material. Uranium or thorium, or any combination thereof, in any physical or chemical form, or ores that contain, by weight, one-twentieth of one percent (0.05 percent) or more of (1) uranium, (2) thorium, or (3) any combination thereof. Source material does not include special nuclear material.

Special Nuclear Material (SNM). Plutonium, uranium-233, or uranium enriched in the isotopes uranium-233 or uranium-235.

Stop Work Authority. The authority and responsibility given to all workers to stop work when controls are inadequate or imminent danger exists. The Project/Site RSO authorizes work re-start once proper controls have been established.

Surface Contamination, Fixed. Radioactive material that has been deposited onto a non-porous surface and is not readily removed by applying light to moderate pressure when wiping with a paper or cloth disk swipe or Masslinn®.

Surface Contamination, Removable. Radioactive material that has been deposited onto a non-porous surface and is readily removed by applying light to moderate pressure when wiping with a paper or cloth disk swipe or Masslinn®.

Surface Contamination, Total. The sum of fixed and removable surface contamination.

Surface Efficiency. The rate of particles emerging from the surface of interest in the field relative to the rate of particles being generated from the total (4π) activity present on the surface. Optimally, the surface efficiency is an experimentally determined value specific to the field surface that accounts for its backscatter characteristics as well as geometry influences (e.g., a scabbled concrete surface). In the absence of an experimentally determined value, a surface efficiency of 0.25 for alpha emitters and 0.5 for beta emitters with maximum beta energy greater than 0.15 MeV are used. These values are recommended in the International Organization for Standardization (ISO) 7503-1, *Evaluation of surface contamination – Part 1: Beta-emitters (maximum beta energy greater than 0,15 MeV) and alpha-emitters*.

Thermoluminescent Dosimeter (TLD). A small device used to measure radiation by measuring the amount of visible light emitted from a crystal in the detector when exposed to ionizing radiation.

Total Effective Dose Equivalent (TEDE). The sum of the DDE (external dose) and the CEDE (internal dose).

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Total Efficiency. The product of the instrument efficiency and the surface efficiency.

Total Organ Dose Equivalent (TODE). The sum of the DDE and CDE to the organ receiving the highest dose.

Work Instruction. A detailed description of the steps required to complete a work task (or group of tasks).

Work Suspension. An event where work is suspended immediately due to the discovery of atypical work site radiological conditions which have not be previously assessed in an ALARA review. The Corporate RSO, with concurrence from the project manager and client, authorizes work resumption once conditions have been assessed, corrected, and/or appropriate controls instituted.



POLICY
Document PO-RP-100
Version R00

Radiation Safety

Michael C. Salmon

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Michael C. Salmon, Senior Vice President
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September 3, 2015

Date

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Rescission

CO-ITSI-RSP-001.03 Gilbane Radiation Safety Program, Revision 3.1, dated 16 May 2014

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I. Statement of Policy

It is the policy of Gilbane Federal ("Company") that activities involving radiation or radioactive material be purposeful and be conducted in a manner that protects its employees, contractors, members of the public, and the environment; and that individual exposure to radiation be maintained as low as reasonably achievable (ALARA).

The following elements comprise the Company's overall policy in the implementation of its radiation safety program:

- (1) **Program Administration.** A designated radiation safety officer (RSO) ensures day-to-day program oversight and is the point of ultimate responsibility. Training is provided to individuals consistent with their program responsibilities. Written procedures and instructions are developed appropriate to the radiological hazards and commensurate with the education, training, and skills of the individuals exposed to those hazards. A review of program effectiveness is conducted at least annually. The Company's radioactive material license is invoked in a controlled, deliberate manner.
- (2) **Dose Limits and Personnel Monitoring.** Radiation exposures to radiation workers and members of the public are managed within regulatory and Company administrative dose limits. Individual exposures to internal and/or external sources of radiation are monitored where the occupational dose is likely to exceed 10% of the applicable regulatory limit(s).
- (3) **Work Planning and Control.** Work activities in restricted areas are conducted under a radiation work permit. An ALARA review is performed for work involving radiation or radioactive material to ensure that sound radiation protection principles are applied. Radiation workers have the authority and responsibility to stop work when controls are inadequate or imminent danger exists. Work is suspended immediately when unassessed atypical work site radiological conditions are encountered.
- (4) **Instruments and Equipment.** Commercially available portable and laboratory instruments and equipment are used to quantitatively measure radiation. Instruments are calibrated for the radiation types and energies of interest, maintained to manufacturers' specifications, and response-checked prior to use to assure proper operation.

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- (5) **Survey and Sampling.** Surveys and sampling are performed by radiation protection personnel to identify, quantify, and evaluate radiological hazards in and around work areas.
 - (6) **Radiation and Contamination Control.** Wherever possible, radiation and contamination are controlled at their source. Administrative procedures (e.g., restricted areas, barriers, postings) and engineered controls (e.g., shielding, containment, ventilation) are used, to the maximum extent practicable, to minimize contamination of the work area and the environment and the generation of radioactive waste.
 - (7) **Radioactive Material Control.** Positive control of radioactive material is established to prevent inadvertent release of radioactive material to unrestricted areas, and ensure protection of radiation workers and members of the public. Licensed material is transferred between licensees and transported in accordance with regulatory requirements.
 - (8) **Personnel Protection and Emergency Response.** Personal protective clothing and equipment are commensurate with the radiological hazards in the work area and the work activities being conducted, and are used to minimize individual exposure to radiation and radioactive material during routine use. Protective measures are identified for conditions requiring emergency response.
 - (9) **Records, Notifications, and Reports.** Records are maintained, notifications are made, and reports are submitted in accordance with Company policy and regulatory requirements.

This policy applies to Company employees, contractors, and other individuals working under the Company's direction and/or control.

II. Definitions

Terms as they are used in the Company's radiation safety program are defined in the glossary of terms in MA-RP-100, Radiation Safety Manual.

III. Implementation

The Senior Vice President - Environmental Business Unit is responsible for establishment and maintenance of Company's radiation safety policy.

The Corporate RSO is responsible for the implementation and annual review of this policy.

IV. Related Policies and Procedures

- PR-RP-110, Radiation Safety Program Administration
- PR-RP-120, Radiation Dose Limits and Personnel Monitoring
- PR-RP-130, Radiation Work Planning and Control
- PR-RP-140, Radiation Instruments and Equipment
- PR-RP-150, Radiological Survey and Sampling
- PR-RP-160, Radiation and Contamination Control
- PR-RP-170, Radioactive Material Control
- PR-RP-180, Personnel Protection and Emergency Response
- PR-RP-190, Radiological Records, Notifications, and Reports

V. Attachments

Attachment 1 - Delegation of Authority to Gilbane Federal Radiation Safety Officer

Attachment 1 – Delegation of Authority to Gilbane Federal Radiation Safety Officer



INTERNAL MEMORANDUM

May 20, 2015

To: Gilbane Federal
From: Mike Salmon, Senior Vice President

Subject: Delegation of Authority to Gilbane Federal Radiation Safety Officer, Jerry Cooper

You Jerry Cooper have been appointed Radiation Safety Officer (RSO) for Gilbane Federal. You will replace former RSO, Jay Pride. You are responsible for ensuring the safe use of radiation. You must ensure that radiation safety activities are being performed safely according to approved policies and procedures, and that all regulatory requirements are met. You are responsible for managing the radiation protection program; identifying radiation protection problems; initiating, recommending, or providing corrective actions; verifying implementation of corrective actions; stopping unsafe activities; and ensuring compliance with regulations.

You are hereby delegated the authority necessary to meet those responsibilities, including prohibiting the use of radioactive materials by employees who do not meet the necessary requirements and shutting down operations when justified by radiation safety or that may result in non-compliance with regulatory requirements.

You are required to notify management of situation where staff are not cooperating and not addressing radiation safety issues. In addition, you are free to raise issues with the NRC, Military Master Licenses or Agreement States (e.g. California Department of Health Services, Radiologic Health Branch) at any time. You will directly manage the radiation safety program and be physically present (or your representative) at any temporary use location where notification has been given by your license conditions and whatever time as may be necessary to ensure that the radiation protection activities are performed.

Please acknowledge your acceptance below.

A handwritten signature in blue ink, appearing to read "Mike Salmon".

Mike Salmon
Senior Vice President
Gilbane Federal

Radiation Safety Officer Acceptance

I accept the responsibilities as stated above.

A handwritten signature in blue ink, appearing to read "Jerry Cooper".

Digitally signed by Jerry Cooper, CN=Jerry Cooper, CHP, o, ou,
email=jerryccooper@comcast.net, c=US
Date: 2015.05.20 14:11:51 -0700

20 May 2015

Jerry Cooper (Signature)

Date



PROCEDURE
Document PR-RP-110
Version R02

Radiation Safety Program Administration

A handwritten signature in blue ink, appearing to read "Jerry C. Cooper".

Jerry C. Cooper, Corporate Radiation Safety Officer

05 Dec 2019

Date

Version	Changes	Affects Section	Effective Date
0.0	Initial Publication	Not Applicable	01 Jan 2016
1.0	Updated regulatory references and California license conditions	C, D, F, G	01 Nov 2019
2.0	Updated NRC license conditions	C, F	05 Dec 2019

Rescission

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A. Purpose and Scope

This procedure describes the implementation of the overall policy of Gilbane Federal ("Company") regarding its radiation safety program. A designated radiation safety officer (RSO) for the Company ensures day-to-day program oversight and is the point of ultimate responsibility. Training is provided to individuals consistent with their program responsibilities. Written procedures and work instructions are developed appropriate to the radiological hazards and commensurate with the education, training, and skills of the individuals exposed to those hazards. A review of program effectiveness is conducted at least annually. The Company's radioactive material license is invoked in a controlled, deliberate manner.

This procedure applies to Company employees, contractors, and other individuals working under the Company's direction and/or control who are involved in the implementation of the Company's radiation safety program. Bolded information in brackets, e.g., [**NRC License Condition 25**], references the source for the procedural provision.

B. Responsibilities

B.1 Corporate RSO

The Corporate RSO is responsible for ensuring that the Company's radiation safety program is administered in accordance with this procedure, and for issuing letters of delegation indicating the scope of the actions and decisions for which the Project/Site RSO is authorized to act on behalf of the Corporate RSO.

B.2 Project/Site RSO

The Project/Site RSO is responsible for implementing the radiation safety program of the assigned project/site, as delegated by the Corporate RSO.

B.3 Radiation Protection Personnel

Radiation protection personnel are responsible for assisting the Project/Site RSO in the field implementation of the radiation safety program.

C. Method

The radiation safety program is implemented through competent individuals who are trained in program requirements and execute the program according to written procedures approved by the licensing authority (i.e., U.S. Nuclear Regulatory Commission [NRC], Region IV or California Department of Public Health, Radiologic Health Branch).

C.1 Organization and Responsibilities

Individuals with suitable qualifications and training staff the positions described below to ensure consistent and successful implementation of the radiation safety program. Individuals must have the requisite skills necessary to perform the functions of their position. **[NUREG-1556, Vol. 18, Rev. 1, Section 8.7]**

- **Corporate RSO.** The Corporate RSO is responsible for the implementation and oversight of the radiation safety program. The Corporate RSO may delegate project/site-specific management of the radiation safety program to a qualified Project/Site RSO; however, the responsibility for overall implementation of and compliance with the radiation safety program remains with the Corporate RSO. The Corporate RSO issues a letter of delegation indicating the scope of the actions and decisions for which the Project/Site RSO is authorized to act on behalf of the Corporate RSO.
- **Project/Site RSO.** The Project/Site RSO is appointed by and acts for the Corporate RSO in implementing the radiation safety program of the assigned project/site. Radioactive material is used by or under the supervision of the Project/Site RSO, who ensures that radiological hazards are identified during work planning, and that appropriate controls are implemented to maintain worker exposure to these hazards as low as reasonably achievable (ALARA). While their duties are essentially the same, a Project RSO is assigned to a specific project, which may include one or more temporary job sites; and a Site RSO is assigned to a temporary job site, which may include one or more projects.
- **Radiation Protection Personnel.** Radiation protection personnel (e.g., health physicists, radiological engineers, and radiation technicians) are trained in radiation protection and assist the Project/Site RSO in the field implementation of the radiation safety program. Assistance may include:
 - Conducting radiation safety training,

- Preparing written procedures and work instructions, and
- Performing work planning and control, project reviews, and audits.
- **Radiation Workers.** Radiation workers (e.g., field personnel, including RSOs and radiation protection personnel, that routinely access restricted areas) perform work in accordance with radiation safety program requirements, including proper use of protective and personnel monitoring equipment, notifying the Project/Site RSO of unexpected radiation hazards, and maintaining their radiation exposure ALARA. Radiation workers use radioactive material under the supervision of the Project/Site RSO.

C.2 Qualifications and Training

The Corporate RSO must be qualified to act as RSO for the NRC and California radioactive material licenses. Other qualifications for the Corporate RSO are determined by the Environmental Business Unit Leader (e.g., Certification by the American Board of Health Physicists, completion of a bachelor's and/or master's degree in the sciences with at least one year of experience in the conduct of a radiation safety program of comparable size and scope).

The qualifications required for Project/Site RSOs, radiation protection personnel, and radiation workers are determined by the Corporate RSO based on the following factors as they apply to each individual's duties:

- The expected radiation hazards;
- The extent to which the individual must be able to work independently to ensure his or her own radiation safety; and
- The extent to which the individual must be able to provide radiation protection guidance to others.

The minimum required training is outlined in Attachments 1 and 2. The qualifications and training of individuals designated as Project/Site RSOs are documented.

Individuals are trained (i.e., informed about radiation hazards and appropriate precautions) before working in the vicinity of licensed materials. Training is conducted by qualified individuals. Training may be delivered as formal classroom instruction, live or recorded video/on-line presentation, or self-study, and may include supervised hands-on experience performing the task(s).

[NUREG-1556, Vol. 18, Rev. 1, Section 8.8 and Appendix D]

Methods of evaluation include classroom examinations, on-the-job training and practical examinations, discussions, and/or drills to ensure that the individual is knowledgeable of the radiation safety aspects of the work. Incorrect answers or responses are reviewed with the student. **[NUREG-1556, Vol. 18, Rev. 1, Appendix D]**

Retraining may be required on topics where an individual is found to be deficient in either practical or classroom training. Additional training is provided whenever there is a significant change in duties, regulations, or the terms and conditions of the license. Refresher training is provided annually. **[NUREG-1556, Vol. 18, Rev. 1, Appendix D]**

C.3 Written Procedures and Work Instructions

The policy and procedures that implement the Company's radiation safety program are referred to collectively as the Company's Radiation Safety Manual. The Radiation Safety Manual describes how the Company complies with license conditions and regulatory requirements. Changes to Radiation Safety Manual procedures are approved by the Corporate RSO, but also must be approved by the licensing authority when a procedure involves working with licensed materials. **[NUREG-1556, Vol. 18, Rev. 1, Section 8.10.1]**

Additional guidance, where appropriate, may be developed in the form of task-specific work instructions that are outside of, but supplement, the Radiation Safety Manual. Work instructions are approved by the Corporate RSO and may be corporate-wide or specific to a program, project, or site.

C.4 Annual Program Review

A review of the content and implementation of the Company radiation safety program is conducted at least annually. The review ensures that the following elements are satisfied:

- Compliance with NRC (or California) and U.S. Department of Transportation (DOT) regulations (as applicable), and the terms and conditions of the license; and
- Occupational doses and doses to members of the public are ALARA.

Applicable elements of the sample audit checklist in Appendix L of NUREG 1556, Vol. 18, Rev. 1 are used as a model for the programmatic reviews. Program reviews may include unannounced site audits to determine whether the

radiation safety program is being implemented properly. **[NUREG-1556, Vol. 18, Rev. 1, Section 8.10.10]**

Violations of license requirements identified by the program review are evaluated for their safety significance in order to set priorities and identify resources to correct these violations. NRC Information Notice 96-28 is used as a guide in this process. Certain identified problems or potential violations may require notification or a report to NRC (see PR-RP-190, Radiological Records, Notifications, and Reports). **[NUREG-1556, Vol. 18, Rev. 1, Section 8.10.10]**

C.5 License Implementation at Temporary Job Sites

Before work begins at a temporary job site, the following information is reviewed by the Corporate RSO to verify proper license implementation.

- Project/Site RSO – The qualifications of the Project/Site RSO to use or supervise use of licensed material is verified. **[NRC License Condition 14.A]**

For temporary job sites worked under the California license, the Project/Site RSO is named in the license. **[California License Condition 12]**

- Location of Temporary Job Site – Licensed materials may be used only at temporary job sites of the Company within the United States where the NRC maintains jurisdiction for regulating the use of licensed material, including areas of exclusive federal jurisdiction within Agreement States. Authorization for use of radioactive materials at job sites in Agreement States not under exclusive federal jurisdiction must be obtained from the appropriate state regulatory agency. **[NRC License Condition 13]**

For temporary job sites within California, radioactive material may be used under the California license only in areas not under exclusive federal jurisdiction. **[California License Condition 10]**

- Jurisdictional Status (if at a federal facility) – Before radioactive material may be used at a temporary job site at any federal facility, the jurisdictional status of the job site must be determined. If jurisdiction status of a federal facility within an Agreement State (which includes California) is unknown, the federal agency controlling the job site in question must be contacted to determine whether the proposed job site is an area of exclusive federal jurisdiction. **[NRC License Condition 13]**

For temporary job sites within California, the response must be obtained in writing or a record made of the name and title of the person at the federal agency who provided the determination and the date that it was provided. **[California License Condition 22]**

- **Authorized Types, Quantities, and Uses of Radioactive Material** – See Attachment 3 for authorized types, quantities, and uses of radioactive material.
- **Multiple Licensees at Temporary Job Site** – For temporary job sites with multiple radioactive material licensees, a written agreement must be established between the Company and other licensees specifying which license activities are performed under whose license and supervision. The agreement includes a commitment by the Company to ensure safety and to help clean up the temporary job site if there is an accident. A copy of this agreement is included in the notification to the licensing authority. **[NRC License Condition 17; California License Condition 18]**
- **Licensing Authority Initiating Notification** – The licensing authority must be notified in writing at least 14 days before activities under license are initiated at a temporary job site. This notification includes:
 - Estimated type, quantity, and physical/chemical forms of licensed material to be used;
 - Specific site location;
 - Description of planned activities, including waste management and disposition;
 - Estimated start date and completion date for the job;
 - Name and title of a point of contact managing radiological operations at the temporary job site, including information on how to contact the individual (i.e., address and phone number);
 - If multiple licensees at temporary job site, a copy of the written agreement between the Company and other licensee(s); and
 - If not previously approved, Radiation Safety Manual procedures implementing the Company's radiation safety program. **[NRC License Condition 16; California License Condition 17]**
- **Possession of Licensed Material** – The Company may not take possession of radioactive material from its clients without prior written approval from the NRC. **[NRC License Condition 11]**

- Origin of Licensed Material – Except for calibration sources, reference standards, and radioactively contaminated equipment owned by the Company, use of licensed material at each temporary job site is limited to material originating from each site. This material may either be transferred to an authorized recipient or remain at the site after Company activities are completed. [NRC License Condition 13; California License Condition 16]
- Import of Licensed Material - Byproduct material, source material, and special nuclear material wastes may not be imported into the U.S. or onto the temporary job site. [NRC License Condition 24]
- Licensing Authority Terminating Notification – Within 30 days of completing activities under license at a temporary job site, the licensing authority must be notified in writing of the radiological status of the temporary job site and the disposition of any licensed material. [NRC License Condition 20; California License Condition 17]

D. Required Records

Corporate RSO delegations of authority; training records; written policies and procedures; corporate and program-level task-specific work instructions; and records of license implementation are retained in accordance with PR-MO-003, Records Retention.

Project/site-specific work instructions are retained with project records.

Records of program reviews and audits are retained for at least 3 years from the date of the record, and include the date of the audit, the name(s) of the person(s) who conducted the audit, persons contacted by the auditor(s), areas audited, audit findings, corrective actions, and follow-up. [NUREG-1556, Vol. 18, Rev. 1, Section 8.10.10]

E. Acronyms and Definitions

Terms as they are used in the Company's radiation safety program are defined in the glossary of terms in MA-RP-101, Radiation Safety.

F. References

U.S. Nuclear Regulatory Commission (NRC) Radioactive Materials License No. 04-29353-01, Amendment 08, dated November 22, 2019.

State of California Radioactive Material License No. 7948-07, Amendment 13, dated November 15, 2019.

Code of Federal Regulations, Title 10 (10 CFR)

- 10 CFR 19, *Notices, Instructions and Reports to Workers; Inspection and Investigations*
- 10 CFR 20, *Standards for Protection Against Radiation*
- 10 CFR 20, Appendix B, *Annual Limits on Intake (ALIs) and Derived Air Concentrations (DACs) of Radionuclides for Occupational Exposure; Effluent Concentrations; Concentrations for Release to Sewerage*
- 10 CFR 20, Appendix C, *Quantities of Licensed Material Requiring Labeling.*
- 10 CFR 30, *Rules of General Applicability to Domestic Licensing of Byproduct Material*
- 10 CFR 40, *Domestic Licensing of Source Material*
- 10 CFR 70, *Domestic Licensing of Special Nuclear Material*
- 10 CFR 71, *Packaging and Transportation of Radioactive Material*

Code of Federal Regulations, Title 49 (49 CFR)

- 49 CFR 172, *Hazardous Materials Table, Special Provisions, Hazardous Materials Communications, Emergency Response Information, Training Requirements, and Security Plans*
- 49 CFR 173, *Shippers—General Requirements for Shipments and Packaging*
- 49 CFR 177, *Carriage by Public Highway*

California Code of Regulations, Title 17 (17 CCR)

- 17 CCR 30253, *Standards for Protection Against Radiation*
- 17 CCR 30255, *Notices, Instructions, and Reports to Personnel*
- 17 CCR 30256, *Vacating Installations: Records and Notice*
- 17 CCR 30275, *Surveys and Tests*
- 17 CCR 30293, *Records*
- 17 CCR 30295, *Notification of Incidents*
- 17 CCR 30373, *Transportation Regulations*

NUREG-1556, Vol. 18, Rev. 1, *Consolidated Guidance About Materials Licenses, Program-Specific Guidance About Service Provider Licenses*, NRC, August 2017.

- Section 8.7, Item 7: Individual(s) Responsible for Radiation Safety Program and Their Training and Experience
- Section 8.8, Item 8: Training for Individuals Working in or Frequenting Restricted Areas
- Section 8.10.1, Operating and Emergency Procedures
- Section 8.10.10, Audit and Review of Program

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- Appendix D, Criteria for Acceptable Training and Experience for Authorized Users
 - Appendix L, Suggested Service Provider Audit Checklist

Regulatory Guide 8.23, *Radiation Safety Surveys at Medical Institutions*, Atomic Energy Commission. Revision 1. January 1981.

NRC Information Notice 96-28, *Suggested Guidance Relating to Development and Implementation of Corrective Action*, dated May 1, 1996.

Gilbane Federal Policy PO-RP-100, Radiation Safety.

Gilbane Federal Manual MA-RP-101, Radiation Safety.

Gilbane Federal Procedures

- PR-RP-190, Radiological Records, Notifications, and Reports.
- PR-MO-003, Records Retention.

G. Attachments

Attachment 1 – Radiation Safety Program Implementation Training

Attachment 2 - Radiation Worker Training [**NUREG-1556, Vol. 18, Rev. 1, Appendix D**]

Attachment 3 – Authorized Types, Quantities, and Uses of Licensed Material [**NRC License Conditions 6, 7, 8, 9, 10, and 15; California License Conditions 6, 7, 8, 9, and 21**]

Attachment 1 – Radiation Safety Program Implementation Training

Company employees, contractors, and other individuals working under the Company's direction and/or control who are responsible for implementation of the Company's radiation safety program are trained in the following Company policies and procedures:

POLICY/PROCEDURE TRAINING MATRIX			
Policy/Procedure	Project/ Site RSOs	Radiation Protection Personnel	Radiation Workers
PO-RP-100, Radiation Safety	✓	✓	✓
PR-RP-110, Radiation Safety Program Administration	✓	✓	
PR-RP-120, Radiation Dose Limits & Personnel Monitoring	✓	✓	✓
PR-RP-130, Radiation Work Planning and Control	✓	✓	✓
PR-RP-140, Radiation Instruments and Equipment	✓	✓	
PR-RP-150, Radiological Survey and Sampling	✓	✓	
PR-RP-160, Radiation and Contamination Control	✓	✓	
PR-RP-170, Radioactive Material Control	✓	✓	
PR-RP-180, Personnel Protection and Emergency Response	✓	✓	✓
PR-RP-190, Radiological Records, Notifications, & Reports	✓	✓	

Attachment 2 – Radiation Worker Training

The following radiation safety topics are included in radiation worker training, as applicable, commensurate with the individuals' assigned tasks and the expected radiation hazards:

- Fundamentals of radiation safety:
 - Characteristics of radiation;
 - Units of radiation dose and quantity of radioactivity;
 - Hazards of exposure to radiation;
 - Levels of radiation from licensed material;
 - Methods of controlling radiation dose (time, distance, and shielding);
 - ALARA concept.
- Radiation detection instruments:
 - Operation;
 - Calibration;
 - Limitations of radiation survey instruments;
 - Radiation survey techniques for measuring radiation field;
 - Radiation survey techniques for measuring removable/fixed contamination;
 - Handling and proper use of personnel monitoring equipment.
- Radiation protection equipment and use:
 - Proper use of protective equipment;
 - Decontamination of contaminated protective equipment.
- Applicable regulatory requirements:
 - 10 CFR 19, 20, 30, 40, 70, and 71;
 - 17 CCR 30253, 30255, 30256, 30275, 30293, 30295, and 30373.
- Company operating and emergency procedures.
- Case histories and lessons learned relevant to operations.
- Briefing on site-specific conditions and requirements.

Escorted visitors may receive abbreviated radiation worker training which, at a minimum, includes information regarding radiological hazards and access/egress protocols specific to the restricted area(s) to be entered, and instruction to remain with the assigned escort at all times and to follow directions provided by the escort.

Attachment 3 – Authorized Types, Quantities, and Uses of Licensed Material

The authorized types, quantities, and uses of licensed material by the Company are as follows:

LICENSED MATERIAL TYPES AND QUANTITIES			
License	Condition	Provision	
NRC	6,7,8	<u>Licensed Material</u>	<u>Maximum</u>
		Hydrogen-3	330 curies (Ci)
		Any byproduct material with atomic numbers 3 through 83, with exceptions	500 Ci
		Any byproduct material with atomic numbers 84 through 96, with exceptions	2.2 Ci
		Carbon-14	110 Ci
		Radium-226 (²²⁶ Ra)	200 Ci
		Americium-241	250 Ci
		Any special nuclear material	350 grams uranium-235 (²³⁵ U), or 200 grams plutonium (Pu), or 200 grams uranium-233 (²³³ U), or any combination of these provided the sum of the ratios of the quantities does not exceed unity
NRC	15	In addition to the limits in Item 8, the licensee shall further restrict the use of licensed material to quantities below the limits specified in 10 CFR 30.72, which require consideration of the need for an emergency plan for responding to a release of licensed material.	
California	6,7,8	<u>Licensed Material</u>	<u>Maximum</u>
		Any radionuclides with atomic numbers 1 through 83	Total not to exceed 20 millicuries (mCi)
		Any radionuclides with atomic numbers 84 through 103	Total not to exceed 20 mCi
		²²⁶ Ra	Total not to exceed 60 mCi
		Any special nuclear material	Not to exceed 1 gram ²³³ U, 1.75 grams ²³⁵ U, or 0.9 grams Pu. Total not to exceed 400 mCi

Attachment 3 – Authorized Types, Quantities, and Uses of Licensed Material

LICENSED MATERIAL TYPES AND QUANTITIES		
License	Condition	Provision
California	21	The total mass of special nuclear material possessed under this license at any one time or at any one authorized location of use shall not exceed that stated in the following formula: The number of grams of ²³⁵ U divided by 350, plus the number of grams of ²³³ U divided by 200, plus the number of grams of Pu (all isotopes) divided by 200, shall not exceed one (i.e. unity).

LICENSED MATERIAL USE		
License	Condition	Provision
NRC	10	For receipt, storage and use incidental to any activity as follows: (1) Any activity related to site characterization, decontamination, and decommissioning of facilities, equipment, and containers; (2) Packaging and repackaging of customer waste for transport; (3) Transport in packages or containers approved for use under the provisions of 10 CFR 71 for transfer to licensees authorized to receive the materials, in accordance with the terms and conditions of licenses issued by the NRC or an Agreement State; (4) For use in performing leak tests and sample analysis as a commercial service for any person as defined in 10 CFR 30.4, and (5) Instruction of individuals.
California	9	To be used for site characterization, decontamination, decommissioning, final status survey, and collection of samples from various media as a customer service. Packaging of radioactive waste in DOT approved containers for transport as a customer service.
California	9	To be used for operational testing of survey instruments.



PROCEDURE
Document PR-RP-120
Version R01

Radiation Dose Limits and Personnel Monitoring



Jerry C. Cooper, Corporate Radiation Safety Officer

01 Oct 2019

Date

Version	Changes	Affects Section	Effective Date
0.0	Initial Publication	Not Applicable	01 Jan 2016
1.0	Modified requirements regarding visitor monitoring and secondary dosimetry use	C, F	01 Nov 2019

Rescission

CO-ITSI-RSOP-10.01 Personnel Exposure Program, Revision 1, dated 18 Feb 2014

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Attachment 1	Regulatory and Administrative Dose Limits

A. Purpose and Scope

This procedure describes the implementation of the Gilbane Federal (“Company”) policy that radiation exposures to radiation workers and members of the public be managed within regulatory and Company administrative dose limits (see Attachment 1). Individual exposures to internal and/or external sources of radiation are monitored where the occupational dose is likely to exceed 10% of the applicable regulatory limit(s).

This procedure applies to Company and contractor radiation safety officers (RSOs), radiation protection personnel, and radiation workers. Bolded information in brackets, e.g., **[NRC License Condition 25]**, references the source for the procedural provision.

B. Responsibilities

B.1 Corporate RSO

The Corporate RSO is responsible for authorizing individual extensions to the administrative dose limits, and specifying measurements and methods to assess internal exposures.

B.2 Project/Site RSO

The Project/Site RSO is responsible for:

- Establishing individual monitoring requirements;
- Ensuring that appropriate personnel monitoring devices are issued and exchanged at proper intervals;
- Monitoring individual exposures to ensure that the administrative dose limits are not exceeded; and
- Performing exposure investigations, as required.

B.3 Radiation Protection Personnel

Radiation protection personnel are responsible for issuing and exchanging personnel monitoring devices.

B.3 Radiation Workers

Radiation workers are responsible for informing the Project/Site RSO prior to entering a restricted area when medical treatments involving radionuclides have been administered to them.

C. Method

The doses received by individuals assigned to complete activities involving exposure to radiation and/or to radioactive material from licensed and unlicensed radiation sources are managed within regulatory and administrative limits defined for occupational doses. Occupational doses do not include any dose received from background radiation, from medical exposures, or as a member of the public.

C.1 Regulatory Dose Limits

Occupational doses are controlled such that the regulatory dose limits, given in Attachment 1, are not exceeded. [10 CFR 20.1201]

Occupational doses to minors are controlled such that they do not exceed 10% of the regulatory dose limits (see Attachment 1). [10 CFR 20.1207]

The dose to an embryo/fetus of a declared pregnant woman is not to exceed a total effective dose equivalent (TEDE) of 500 millirem (mrem) during the entire pregnancy (see Section C.7). [10 CFR 20.1208]

The dose to individual members of the general public is not to exceed a TEDE of 100 mrem in a year, excluding background and medical exposures. [10 CFR 20.1301(a)(1)]

There are no provisions for planned special exposures under the Company's radiation safety program.

C.2 Administrative Dose Limits

Administrative dose limits, shown in Attachment 1, are used to ensure that regulatory dose limits are not exceeded and that occupational doses are maintained as low as reasonably achievable (ALARA). Administrative dose limits serve to alert radiation protection personnel to practices or trends in the work environment that are resulting in additional or excessive exposure to individuals. The Company goal is that the dose to any individual does not exceed the administrative dose limits.

Extensions to the administrative dose limits are considered on a case-by-case basis when written justification for the need to extend the individual's dose limit is provided by the individual's supervisor. Extensions may be authorized only by

the Corporate RSO, and only after an individual's accrued dose for the current year has been determined.

C.3 Monitoring Requirements

The Project/Site RSO establishes individual monitoring requirements. As a good practice, individual monitoring is performed if the occupational dose is likely to exceed a TEDE of 100 mrem.

Monitoring of individual exposures to internal and/or external sources of radiation is required if the occupational dose is likely to exceed 10% of the regulatory dose limit (see Attachment 1). Monitoring of minors is required if the occupational dose is likely to exceed a TEDE of 100 mrem. Additional requirements for monitoring minors are found in Attachment 1. Monitoring of a declared pregnant woman is required if the occupational dose is likely to exceed a TEDE of 100 mrem during the entire pregnancy. [10 CFR 20.1502(a); NUREG-1556, Vol. 18, Rev. 1, Section 8.10.6]

Visitors, i.e., individuals who on an infrequent basis enter a restricted area where monitoring is required and who do not perform or supervise [intrusive work](#) or [handling of radioactive material](#), do not require individual monitoring. Visitors may enter a restricted area without dosimetry provided they remain at all times with an escort authorized by the Project/Site RSO who has been issued dosimetry, and do not enter a high radiation area or a contamination area.

C.4 Internal Exposure Monitoring

Monitoring of internal exposure normally is conducted by use of air samples, particularly samples collected within the breathing zone. When a potential or actual condition exists in which an individual could have received an unmonitored intake of radioactive material in excess of 10% of an annual limit on intake (ALI), the intake is determined by measurements of quantities of radionuclides excreted from or retained in the body (e.g., in-vitro and/or in-vivo bioassay monitoring). The measurements and methods used are directed by the Corporate RSO. Bioassay sample analysis is performed by a laboratory holding current accreditation from the National Voluntary Laboratory Accreditation Program (NVLAP) of the National Institute of Standards and Technology (NIST). [NUREG-1556, Vol. 18, Rev. 1, Section 8.10.6]

Internal exposure does not include exposure due to medical administration of radionuclides. Therefore, radiation workers are required to inform the Project/Site RSO prior to entering a restricted area when medical treatments involving radionuclides have been administered. Where possible, individuals are reassigned to work in areas outside of restricted areas until the administered radionuclide is eliminated from the body to the extent that it will not significantly affect dosimeter or radiation detection measurements.

C.5 External Exposure Monitoring

External exposure monitoring is accomplished using approved radiation dosimetry, such as a thermoluminescent dosimeter (TLD), worn on the front of the upper torso. Radiation protection personnel issue TLDs to radiation workers and exchange them on schedule approved by the Project/Site RSO, but no longer than a calendar quarterly basis. Radiological surveys may be performed to supplement personnel monitoring when work is being performed in areas where individuals are required to be monitored.

Secondary dosimetry, such as a self-reading dosimeter, is issued to individuals working in [restricted](#) areas with [general area](#) dose rates in excess of 2 mrem/hour, or which likely could result in a dose of 300 mrem. When both primary and secondary dosimeters are issued, the secondary dosimeter is placed in close proximity to the primary dosimeter to facilitate comparison of results.

Multiple primary dosimeters (multi-badging) may be issued when approved by the Project/Site RSO where dose rates to different parts of the body are likely to differ by more than 100 mrem/hour.

Personnel dosimetry is processed and evaluated by a dosimetry processor holding current NVLAP accreditation for the type(s) of radiation for which the individual wearing the dosimeter is monitored. [**10 CFR 20.1501(d); NUREG-1556, Vol. 18, [Rev. 1, Section 8.10.6](#)**]

C.6 Dosimetry Issue

Before dosimetry is issued, the individual to be monitored must satisfactorily complete radiation worker training. Satisfactory completion is based on receiving a passing score on a competency exam administered by a qualified individual (e.g., an individual whom the Corporate RSO or Project/Site RSO determines is qualified by experience and education).

The occupational dose received during the current calendar year (i.e., year-to-date) is determined for individuals who are likely to receive a dose in excess of 10% of the administrative limit. The year-to-date dose history for the current year is obtained and documented on NRC Form 4 or equivalent. The record shows each period during which the individual received an occupational dose, and is signed by the individual who received the exposure.

C.7 Exposure Investigations

The Project/Site RSO performs an exposure investigation for individuals with a lost or damaged dosimeter, or who exceed the levels in the following table.

Year-to-Date TLD Readings	Investigation Level (% of Administrative Limit)
< 3 months	> 250 mrem (25%)
3 to 6 months	> 500 mrem (50%)
6 to 9 months	> 750 mrem (75%)
9 to 11 months	> 900 mrem (90%)

The Project/Site RSO works with the individual's supervisor to determine if action is required to minimize the individual's dose, to monitor future dose closely, or to pursue an extension to an administrative dose limit.

C.8 Reports

Reports of individual monitoring results are made available to monitored individuals on an annual basis. Quarterly exposure reports are generated and reviewed by the Project/Site RSO to ensure that the administrative dose limits are not exceeded. [\[NUREG-1556, Vol. 18, Rev. 1, Section 8.10.6\]](#)

C.9 Declared Pregnant Woman

A declaration of pregnancy is entirely at the discretion of the woman (medical proof is not required). To declare pregnancy, the woman informs her supervisor or the Project/Site RSO, in writing, of the pregnancy and an estimated date of conception so that the estimated dose to the embryo/fetus prior to declaration can be determined. A woman may establish or withdraw her declaration of pregnancy at any time and for any reason by notifying her supervisor or the Project/Site RSO in writing. Any woman who does not declare her pregnancy is

subject to the normal occupational dose limits and is not subject to special controls or treatment with respect to work assignments involving exposure to radiation even if she is pregnant. [\[NUREG-1556, Vol. 18, Rev. 1, Section 8.10.6\]](#)

D. Required Records

Records of individual monitoring results are maintained until termination of the license. **[10 CFR 20.2106]**

E. Acronyms and Definitions

Terms as they are used in the Company's radiation safety program are defined in the glossary of terms in MA-RP-101, Radiation Safety.

F. References

Code of Federal Regulations, Title 10 (10 CFR)

- 10 CFR 20.1201, *Occupational Dose Limits for Adults*
- 10 CFR 20.1207, *Occupational Dose Limits for Minors*
- 10 CFR 20.1208, *Dose Equivalent to an Embryo/Fetus*
- 10 CFR 20.1501, *General*
- 10 CFR 20.1502, *Conditions Requiring Individual Monitoring of External and Internal Occupational Dose*
- 10 CFR 20.2106, *Records of Individual Monitoring Results*

NUREG-1556, Vol. 18, [Rev. 1](#), *Consolidated Guidance About Materials Licenses, Program-Specific Guidance About Service Provider Licenses*, NRC, [August 2017](#).

- [Section 8.10.6](#), Occupational Dose

Gilbane Federal Policy PO-RP-100, Radiation Safety.

Gilbane Federal Manual MA-RP-101, Radiation Safety.

G. Attachments

Attachment 1 – Regulatory and Administrative Dose Limits **[10 CFR 20.1201(a); 10 CFR 20.1207; 10 CFR 20.1502(a)]**

Attachment 1 – Regulatory and Administrative Dose Limits

Exposure Type	Annual Regulatory Limit ^a (mrem)	Monitoring Required (mrem) ^b	Annual Administrative Limit (mrem) ^c
TEDE (DDE + CEDE)	5,000	500	1,000
TODE (DDE + CDE) to any Organ ^d	50,000	5,000	10,000
Lens (of the Eye) Dose Equivalent (LDE)	15,000	1,500	3,000
Shallow-Dose Equivalent to the Skin (SDE)	50,000	5,000	10,000

Notes:

^a From 10 CFR 20.1201(a); dose to minors is limited to 10% of annual regulatory limits per 10 CFR 20.1207; dose to the embryo/fetus of a declared pregnant woman during the entire pregnancy is limited to 500 mrem per 10 CFR 20.1208.

^b From 10 CFR 20.1502(a); monitoring of minors is required at 100 mrem TEDE and at 10% of the LDE and SDE monitoring requirements; monitoring of a declared pregnant woman is required at 100 mrem TEDE.

^c Established at 20% of the regulatory limit; a declared pregnant woman is administratively limited to 100 mrem during the entire pregnancy.

^d Except the lens of the eye, because unique monitoring requirements and annual legal exposure limits exist for this organ; see subsequent table row.



PROCEDURE
Document PR-RP-130
Version R00

Radiation Work Planning and Control


Jerry C. Cooper, Corporate Radiation Safety Officer

18 Dec 2015

Date

Version	Changes	Affects Section	Effective Date
0.0	Initial Publication	Not Applicable	01 Jan 2016

Rescission

CO-ITSI-RSOP-01.01 Radiation Work Permit, Revision 2/MC-1, dated 29 Jul 2014; and
CO-ITSI-RSOP-02.02 ALARA Job Review, Revision 2, dated 18 Feb 2014

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A. Purpose and Scope

This procedure describes the implementation of the Gilbane Federal (“Company”) policy that work activities in restricted areas be conducted under a radiation work permit (RWP), and that an As Low As Reasonably Achievable (ALARA) review be performed for work involving radiation or radioactive material to ensure that sound radiation protection principles are applied. Radiation workers have the authority and responsibility to stop work when controls are inadequate or imminent danger exists. Work is suspended immediately when unassessed atypical work site radiological conditions are encountered.

This procedure applies to Company and contractor radiation safety officers (RSOs), radiation protection personnel, and radiation workers. Bolded information in brackets, e.g., **[NRC License Condition 25]**, references the source for the procedural provision.

B. Responsibilities

B.1 Corporate RSO

The Corporate RSO is responsible for approving independent ALARA reviews (see Section C.2). The Corporate RSO also is responsible for authorizing resumption of work, with concurrence from the project manager and client, following a work suspension due to the discovery of atypical work site radiological conditions.

B.2 Project/Site RSO

The Project/Site RSO is responsible for:

- Ensuring that the qualifications of radiation protection personnel in radiation work planning and control are sufficient for project/site needs;
- Approving RWPs;
- Where appropriate, conducting briefings prior to the start of the job to explain special requirements, engineering controls, or work restrictions; and
- With project manager concurrence, re-starting work following work stoppage.

B.3 Radiation Protection Personnel

Radiation protection personnel are responsible for performing radiation work planning and control activities under the direction of the Project/Site RSO, and for stopping work whenever it is not in compliance with the RWP.

B.4 Radiation Workers

Radiation workers are responsible for:

- Reviewing the provisions of job-specific RWPs before the job begins;
- If the scope of the job changes due to any unexpected conditions, notifying the Project/Site RSO so that the impact on the provisions of the job can be evaluated; and
- Stopping work when controls are inadequate or imminent danger exists.

C. Method

Work activities involving radiation and/or radioactive material will be planned using the following methods in consultation with the Project/Site RSO and other radiation protection personnel tasked with oversight responsibilities.

C.1 Radiation Work Permits (RWPs)

Work activities in restricted areas are conducted under an RWP that details radiologically based requirements and protective measures commensurate with the hazards associated with the specific activities being conducted.

RWPs are used to inform individuals of the radiological hazards that exist in a restricted area and to detail the protective measures and controls needed to perform the tasks in the restricted area safely. The RWP describes the scope of work, including supporting tasks that must be performed in a restricted area, and includes, as necessary, supporting information and documentation such as previous survey data, drawings, and diagrams. Dose monitoring requirements are specified in the RWP. RWPs are approved by the Project/Site RSO.

General RWPs are used for the performance of routine activities such as observation, inspection, or routine tours where radiological conditions are stable. A general RWP normally is valid for one calendar year, and is rewritten at the end of the calendar year if necessary. Job-specific RWPs are used for the performance of defined activities in specific locations and normally are valid for the duration of the activity or until the end of the calendar year in which they are written.

Radiation workers review the provisions of general RWPs prior to initial use, and review job-specific RWPs before the related work begins. The Project/Site RSO may conduct a briefing prior to the start of the job to explain special

requirements, engineering controls, or work restrictions. If the scope of the job changes due to any unexpected conditions, affected radiation workers must notify the Project/Site RSO so that the impact on the provisions of the job can be evaluated. Radiation workers also must notify the Project/Site RSO of any unexpected or unusual conditions.

A copy of the RWP normally is available at the entry/exit point of the restricted area. Individuals must read and sign the RWP before entering the restricted area or performing any of the tasks covered by the RWP.

C.2 ALARA Review

An ALARA review is performed by the Project/Site RSO for work involving radiation and/or radioactive material to ensure that industry-recognized principles are applied. The ALARA review includes a review of the description of the scope, the sequence of events for the job evolution, and the radiological controls necessary to ensure the safety of the workers, the public, and the environment. The ALARA review not only considers radiation safety requirements, but engineering and administrative controls that may be applied to reduce internal and/or external doses. The ALARA review ensures that basic dose reduction strategies are employed using the ALARA concepts of time, distance, and shielding. The ALARA review may occur and/or be documented as part of the RWP review and approval process.

An independent ALARA review, approved by the Corporate RSO, is performed and documented if any of the following conditions are expected:

- The individual dose estimate for a specific task exceeds 100 millirem (mrem) total effective dose equivalent (TEDE);
- The collective dose estimate for the job exceeds 500 mrem TEDE;
- Work activities involve entry into areas where whole-body dose rates for personnel could exceed 1 rem/hour;
- Airborne radioactivity levels exceed a derived air concentration (DAC) of 1,000 DAC.
- A radioactive release to the environment or exposure to member(s) of the public is possible;
- Highly complex, infrequent, or new/first-time radiological work activities are involved;
- Installation or removal of shielding is required; or
- The Corporate RSO determines that such a review is necessary.

C.3 Stop Work Authority

All radiation workers have the authority and responsibility to stop work when controls are inadequate or imminent danger exists. Radiation protection personnel are responsible for stopping work whenever work being performed is outside the scope of the governing RWP, when workers are not complying with the provisions of the RWP, or when conditions vary from those described in the RWP and pose an immediate hazard to individuals in the area.

The work area is placed in a safe configuration. Once stopped, work may not be resumed until proper controls have been established. Work re-start is authorized by the Project/Site RSO with project manager concurrence.

C.4 Work Suspension

Work is suspended immediately where any of the following atypical work site radiological conditions are encountered, unless previously assessed in an ALARA review:

- An individual TEDE exceeds 500 mrem;
- The collective TEDE for the job exceeds 1,000 mrem;
- Airborne radioactivity exceeds 1,000 DAC; or
- Conditions warrant the radiological posting of an area as a High Radiation Area or a High Contamination Area.

The Corporate RSO is notified and, with concurrence from the project manager and client, authorizes work resumption once conditions have been assessed, corrected, and/or appropriate controls instituted.

D. Required Records

RWPs and ALARA reviews are maintained until termination of the license. **[10 CFR 20.2102]**

E. Acronyms and Definitions

Terms as they are used in the Company's radiation safety program are defined in the glossary of terms in MA-RP-101, Radiation Safety.

F. References

Code of Federal Regulations, Title 10 (10 CFR)

- 10 CFR 20.2102, *Records of Radiation Protection Programs*

Gilbane Federal Policy PO-RP-001, Radiation Safety.

Gilbane Federal Manual MA-RP-101, Radiation Safety.


G. Attachments

None.



PROCEDURE
Document PR-RP-140
Version R01

Radiation Instruments and Equipment



Jerry C. Cooper, Corporate Radiation Safety Officer

01 Oct 2019

Date

Version	Changes	Affects Section	Effective Date
0.0	Initial Publication	Not Applicable	01 Jan 2016
1.0	Updated smear counting efficiency calcs, MDCs based on low background count, daily instrument response checks, and references	C, F, G	01 Nov 2019

Rescission

CO-ITSIRSOP-03.02 Radiation Safety Instrumentation, Revision 2, dated 18 Feb 2014

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Attachment 1	Alternate Method to Calculate MDCs Based on Low Background Count

A. Purpose and Scope

This procedure describes the implementation of the Gilbane Federal (“Company”) policy that commercially available portable and laboratory instruments and equipment be used to measure radiation quantitatively. Instruments are calibrated for the radiation types and energies of interest, maintained to manufacturers’ specifications, and response-checked prior to use to assure proper operation.

This procedure applies to Company and contractor radiation safety officers (RSOs) and radiation protection personnel. Bolded information in brackets, e.g., [**NRC License Condition 25**], references the source for the procedural provision.

B. Responsibilities

B.1 Project/Site RSO

The Project/Site RSO is responsible for:

- Ensuring that instruments and equipment are appropriate to the type(s) of radiological survey(s) and sampling to be performed;
- Establishing a program for instrument calibration, initial set-up, and daily response checks;
- Ensuring that the qualifications of radiation protection personnel in the use of instruments and equipment are sufficient for project/site needs.

B.3 Radiation Protection Personnel

Radiation protection personnel are responsible for performing initial set-up and ongoing response checks of instruments and equipment under the direction of the Project/Site RSO.

C. Method

Commercially available portable and laboratory instruments and equipment are used to measure radiation quantitatively, calibrated for the radiation types and energies of interest, and maintained to manufacturers’ specifications. Instruments are used only by individuals qualified in their use.

C.1 Selection

The Project/Site RSO selects radiation instruments and equipment appropriate to the type(s) of radiological survey(s) and sampling to be performed. The selection is based on reliable operation, detection sensitivity, operating characteristics, and expected performance in the field. As a general rule, only instruments are capable of detecting radiation at levels anticipated to be present should be considered for use. Typical instrumentation used is listed in the following table. [NUREG-1556, Vol. 18, Rev. 1, Appendix F]

Measurement Type	Detector Type	Detector Size/ Active Area	Instrument Model	Detector Model
Gamma scan	NaI(Tl) scintillation	68 cm x 16 cm x 16 cm crystal	RSI RSX-1	RSI RS-700
		5.1 cm diameter x 5.1 cm length crystal	Ludlum 2221	Ludlum 44-10
Alpha/beta scan/static	Gas flow proportional	582 cm ² active area	Ludlum 2360	Ludlum 43-37
	Dual phosphor scintillation	100 cm ² active area	Ludlum 2360	Ludlum 43-93
Alpha/beta smears	Gas flow proportional	20 cm ² window	Protean WPC-9550	N/A
	Dual phosphor scintillation	20 cm ² window	Ludlum 2929	Ludlum 43-10-1
Exposure/ dose rate	NaI(Tl) scintillation	2.5 cm diameter x 2.5 cm length crystal	Ludlum 19	N/A
	Tissue-equivalent scintillation	N/A	Bicron MicroRem®	N/A
Air sampling	Low-volume air sampler	N/A	F&J LV-1	N/A
Neutron dose rate	Helium-3 proportional	1.6 cm diameter x 2.5 cm length tube	Ludlum 12-4	N/A

Notes:

cm – centimeter(s)

cm² – square centimeter(s)

NaI(Tl) – sodium iodide (thallium-activated)

ZnS (Ag) – zinc sulfide (silver-activated)

C.2 Inventory and Control

A sufficient inventory and variety of operable and calibrated portable, semi-portable, and fixed radiation detection and measurement instruments is maintained at the site to satisfy the following considerations:

- Effective measurement of radiation exposure and control of radioactive material with instrumentation appropriate to enable the assessment of alpha, beta, and gamma radiation at the energies and intensities anticipated;
- Maximum number of personnel and separate work areas requiring surveillance;
- Frequency and types of surveys or measurements required to support normal and anticipated activities;
- Allowance for repair and calibrations; and
- Minimization of delays in personnel access/egress from restricted areas.

[NUREG-1556, Vol. 18, Rev. 1, Section 8.10.3]

C.3 Calibration and Maintenance

Instruments are calibrated prior to first use; following any repair, maintenance, or modifications that could affect calibration; and after failure of a response check requiring adjustments or repair that could affect calibration. Instruments are calibrated using National Institute of Standards and Technology (NIST)-traceable sources and maintained to manufacturers' specifications. Instruments are re-calibrated at least annually (every 12 months). Calibrated instruments are marked with a calibration sticker and accompanied by a calibration certification. Each instrument is inspected prior to use to ensure that it is in proper working condition. Instruments are protected against inclement weather conditions in the field. [10 CFR 20.1501(c); NUREG-1556, Vol. 18, Rev. 1, Section 8.10.3]

Calibration, maintenance, and repair of equipment are performed by the equipment manufacturer, authorized representative, or an approved vendor. Only modifications approved by the manufacturer may be made to an instrument. Minor maintenance (e.g., Mylar replacement) may be made in the field by qualified individuals.

Calibration records include the type of instrument and manufacturer, the instrument model and serial number, date of calibration, calibration due date, and the name of the worker performing the calibration. [NUREG-1556, Vol. 18, Rev. 1, Appendix F]

C.4 Initial Set-Up

Instruments and equipment are set up upon initial receipt and after calibration, repairs, or major maintenance is performed. Instrument response to radiation types and energies of interest is accounted for during set-up. A check source is used that emits the same type of radiation (i.e., alpha, beta, and/or gamma) to be measured and that gives a similar instrument response. The check source does not necessarily use the same radionuclide as the radionuclide(s) being measured; however, it should have a conservative and/or representative average energy as compared to the weighted average energy of the anticipated radionuclide mixture to be measured in the field.

A series of measurements is made using the check source, and another series of measurements is made without the check source to determine the instrument's expected response to background radiation. Where possible, a radiologically non-impacted area with radiological characteristics (i.e., materials and/or surfaces [e.g., concrete, brick, drywall, metal, etc.]) similar to the area(s) to be surveyed is used to determine the instrument's expected response to background radiation.

Measurements are made using a source-geometry configuration similar to the configuration to be used for the field measurements to be collected. For scaler instruments, an acceptable range (generally two times the standard deviation; i.e., $\pm 2\sigma$ around the expected value) for both source and background instrument response is established and verified as statistically suitable using the chi-squared test. For ratemeter instruments, an acceptable range (generally ± 20 percent around the expected value) is established.

Response checks are performed and evaluated against the acceptance criteria. If instrument measurements fall outside the acceptance criteria, the response check is repeated. If it falls outside the acceptance criteria a second time, it is removed from service and is not used until the problem is resolved or repaired.

C.5 Detection Efficiency

Total efficiency values for the detection of alpha/beta-emitting surface radioactivity are developed using International Organization for Standardization (ISO) 7503-1, *Evaluation of surface contamination – Part 1: Beta-emitters (maximum beta energy greater than 0,15 MeV) and alpha-emitters* (ISO, 1988).

ISO 7503-1 defines total efficiency (ϵ_T) as the product of two terms: the instrument efficiency (ϵ_i) and the surface efficiency (ϵ_s).

Equation 1

$$\epsilon_T = \epsilon_i \times \epsilon_s$$

The instrument efficiency is determined based on an average rate of particles detected by the instrument relative to the surface (2π) particle emission rate of the calibration source. The surface particle emission rate is a value measured and certified by the source manufacturer.

The surface efficiency (ϵ_s) is determined based on the rate of particles emerging from the surface of interest in the field relative to the rate of particles being generated from the total (4π) activity present on the surface. Optimally, the surface efficiency is an experimentally determined value specific to the field surface that accounts for its backscatter characteristics as well as geometry influences (e.g., a scabbled concrete surface). In the absence of an experimentally determined value, the following values recommended in ISO 7503-1 are used:

- 0.25 for alpha emitters and beta emitters with a maximum beta energy between 0.15 mega-electron volts (MeV) and 0.4 MeV, and
- 0.5 for beta emitters with maximum beta energy greater than 0.4 MeV

For smear counting, an absolute counting efficiency may be used in lieu of the total efficiency. The absolute counting efficiency (ϵ_A) is the ratio of the measured count rate and the contained activity [total dpm] of the source. The efficiency is given as 4π and measured according to guidelines found in American National Standards Institute (ANSI) N42.25-1997, *Calibration and Usage of Alpha/Beta Proportional Counters*. Beta efficiencies include backscatter. Alpha efficiencies include recoil.

C.6 Minimum Detectable Concentration

An *a priori* minimum detectable concentration (MDC) for each instrument and technique is calculated prior to its use. The MDC is the concentration that a specific instrument and technique can be expected to detect 95 percent of the time under actual conditions of use. MDC calculation equations may be modified to accommodate project-specific applications.

- Gamma Scan MDC for Land Areas – The MDC for gamma scan measurements is calculated using the methodology described in NUREG-1507, *Minimum Detectable Concentrations with Typical Radiation Survey Instruments for Various Contaminants and Field Conditions*, Section 6.8.2.
- Beta Scan MDC for Building and Structure Surfaces - The MDC for beta scan measurements is calculated using NUREG-1575, *Multi-Agency Radiation Survey and Site Investigation Manual* (MARSSIM), Equation 6-10, in units of disintegrations per minute per 100 square centimeters (dpm/100 cm²):

Equation 2

$$MDC_{scan} = \frac{d' \sqrt{B_i}}{\sqrt{p} \epsilon_i \epsilon_s (W_A/100) i}$$

where:

- d' = scan performance criteria (see MARSSIM, Section 6.7.2.1)
- B = number of background counts in time interval i (counts)
- p = surveyor efficiency, 0.5 (see NUREG-1507, Section 6.6)
- ϵ_i = instrument efficiency (counts per particle)
- ϵ_s = surface efficiency (particles per disintegration)
- W_A = active area of the detector window (cm²)
- i = time interval while detector passes over source (min)

- Alpha/Beta Static MDC for Building and Structure Surfaces – The MDC for alpha/beta static measurements is calculated using NUREG-1507, Equation 3-11, in units of dpm/100 cm²:

Equation 3

$$MDC = \frac{3 + 3.29 \sqrt{R_B T_{S+B} \left(1 + \frac{T_{S+B}}{T_B}\right)}}{\epsilon_i \epsilon_s \frac{W_A}{100 \text{ cm}^2} T_{S+B}}$$

where:

- R_B = background count rate (cpm)
- T_B = background counting time (minute [min])
- T_{S+B} = sample counting time (min)
- ϵ_i = instrument efficiency (counts per particle)
- ϵ_s = surface efficiency (particles per disintegration)
- W_A = active area of the detector window (cm²)

- Alpha/Beta Smears of Building and Structure Surfaces – The MDC for smear counting is calculated as described above for static measurements, in units of dpm/smear, with ϵ_i and ϵ_s terms replaced by the absolute counting efficiency (ϵ_A), if being used.
- Alpha Scan MDC for Building and Structure Surfaces – The MDC for alpha scan measurements is calculated using the methodology described in MARSSIM, Section 6.7.2.2

When the background count is low (< 100 counts), an alternate method provided in Attachment 1 may be used to calculate MDCs unless directed otherwise by the Project/Site RSO.

C.7 Daily Response Checks

Instrument response checks are conducted to assure constancy in instrument response, to verify that the detector is operating properly, and to demonstrate that measurement results are not the result of detector contamination.

Instrument response is checked at least daily when the instrument is in use, using the same check source(s) used to set up the instrument. The response check is performed in a specified location using a specified source-detector alignment that can be repeated easily. It may be performed at the beginning of, at the end of, or in some planned mix throughout the day.

D. Required Records

Radiation instrument and equipment calibration, set-up, and response check records are maintained for a minimum of 3 years after the record is made. **[10 CFR 20.2103]**

E. Acronyms and Definitions

Terms as they are used in the Company's radiation safety program are defined in the glossary of terms in MA-RP-101, Radiation Safety.

F. References

Code of Federal Regulations, Title 10 (10 CFR)

- 10 CFR 20.1501, *General*
- 10 CFR 20.2103, *Records of Surveys*

NUREG-1507, *Minimum Detectable Concentrations with Typical Radiation Survey Instruments for Various Contaminants and Field Conditions*, U.S. Nuclear Regulatory Commission (NRC), January 1998.

NUREG-1556, Vol. 18, Rev. 1, *Consolidated Guidance About Materials Licenses, Program-Specific Guidance About Service Provider Licenses*, NRC, August 2017.

- Section 8.10.3, Radiation Monitoring Instruments
- Appendix F, Radiation Monitoring Instrument Specifications and Model Radiation Survey Instrument and Air Sampler Calibration Program

NUREG-1575, *Multi-Agency Radiation Survey and Site Investigation Manual (MARSSIM)*, U.S. Department of Defense, U.S. Department of Energy, U.S. Environmental Protection Agency, and NRC, Revision 1, August 2000.

NUREG-1575, Supplement 1, *Multi-Agency Radiation Survey and Assessment of Materials and Equipment Manual (MARSAME)*, U.S. Department of Defense, U.S. Department of Energy, U.S. Environmental Protection Agency, and NRC, January 2009.

Gilbane Federal Policy PO-RP-001, Radiation Safety.

Gilbane Federal Manual MA-RP-101, Radiation Safety.

ANSI N42.25-1997, *Calibration and Usage of Alpha/Beta Proportional Counters*. American National Standards Institute (ANSI), 1997.

ISO-7503-1, *Evaluation of surface contamination – Part 1: Beta-emitters (maximum beta energy greater than 0,15 MeV) and alpha-emitters*. International Organization for Standardization (ISO), 1988.

G. Attachments

Attachment 1 – Alternate Method to Calculate MDCs Based on Low Background Count

Attachment 1 – Alternate Method to Calculate MDCs Based on Low Background Count

When the background count is high (> 100 counts), traditional equations used to calculate the MDC work well. However, at lower background levels such equations can produce a high rate of Type I errors, meaning that too often a decision is made that there is radioactivity present when it actually is not. When the background count is low (< 100 counts), the minimum detectable number of counts used to calculate the MDC values may be determined using Equation 3 from Table 7.6 from NUREG-1575, Supplement 1, *Multi-Agency Radiation Survey and Assessment of Materials and Equipment Manual* (MARSAME), given as Equation 4 below:

Equation 4

$$S_D = 2.71 \left(1 + \frac{T_{S+B}}{T_B} \right) + 3.29 \sqrt{N_B \frac{T_{S+B}}{T_B} \left(1 + \frac{T_{S+B}}{T_B} \right)}$$

where:

- S_D = minimum detectable number of counts (counts)
- T_B = background counting time (min)
- T_{S+B} = sample counting time (min)
- N_B = background counts (counts)

The MDC for alpha/beta static measurements may be calculated using a modified Equation 3 to convert the minimum detectable number of counts, S_D , to a concentration over 100 cm²:

Equation 5

$$\text{Static MDC} = \frac{S_D}{\epsilon_i \epsilon_s \frac{W_A}{100 \text{ cm}^2} T_{S+B}}$$

where variables not previously defined:

- ϵ_i = instrument efficiency (counts per particle)
- ϵ_s = surface efficiency (particles per disintegration)
- W_A = active area of detector window (cm²)

The MDC for smear counting may be calculated using Equation 5, in units of dpm/smear, replacing the ϵ_i and ϵ_s terms with the smear counter's absolute counting efficiency (ϵ_A).

Similar modifications to equations used to calculate the MDC for automated and manual scan measurements may be made using the minimum detectable number of counts, S_D .



PROCEDURE
Document PR-RP-150
Version R01

Radiological Survey and Sampling

A handwritten signature in blue ink, appearing to read "Jerry C. Cooper".

Jerry C. Cooper, Corporate Radiation Safety Officer

01 Oct 2019

Date

Version	Changes	Affects Section	Effective Date
0.0	Initial Publication	Not Applicable	01 Jan 2016
1.0	Updated regulatory references	C, F	01 Nov 2019

Rescission

CO-ITSI-RSOP-04.01 Radiological Air Sampling, Revision 1, dated 13 Apr 2013;
CO-ITSI-RSOP-05.01 Radiological Monitoring, Revision 1, dated 12 Apr 2013; and
CO-ITSI-RSOP-09.01 Effluent and Environmental Monitoring, Revision 1/MC2, dated 17 Jul 2014

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A. Purpose and Scope

This procedure describes the implementation of the Gilbane Federal (“Company”) policy that surveys and sampling are performed by radiation protection personnel to identify, quantify, and evaluate radiological hazards in and around work areas.

This procedure applies to Company and contractor radiation safety officers (RSOs) and radiation protection personnel. Bolded information in brackets, e.g., [**NRC License Condition 25**], references the source for the procedural provision.

B. Responsibilities

B.1 Project/Site RSO

The Project/Site RSO is responsible for:

- Ensuring that the qualifications of radiation protection personnel in radiological survey and sampling are sufficient for project/site needs;
- Verifying that the types of measurements, measurement techniques, and detection sensitivities are appropriate for project/site needs; and
- Establishing survey and sampling frequencies, reviewing and approving results, and authorizing exceptions, if any, to survey and sampling requirements.

B.2 Radiation Protection Personnel

Radiation protection personnel are responsible for performing radiological survey and sampling activities under the direction of the Project/Site RSO.

C. Method

Radiological survey and sampling are performed to identify, quantify, and evaluate the potential hazard(s) associated with the radiological conditions in and around work areas. [**10 CFR 20.1501(a); NUREG-1556, Vol. 18, Rev. 1, Section 8.10.4**]

C.1 Preparation

Survey and sampling information is used to:

- Inform radiation workers of the radiological conditions/hazards in the area;
- Determine any required area postings;
- Determine appropriate personal protective equipment (PPE); and

- Ensure that individual exposures to radiation and radioactive materials are maintained as low as reasonably achievable (ALARA).

Survey and sampling to assess radiation and contamination levels may be performed separately or jointly as warranted by expected radiological conditions and job requirements.

The types of measurements to be performed, the measurement techniques to be used, and the detection sensitivities of the selected instruments are verified by the Project/Site RSO to be appropriate to the project/site needs.

- Measurement Types – The three basic types of measurements used are scan measurements, static measurements, and samples. The following apply as a general rule:
 - Scan (i.e., moving) measurements are qualitative (i.e., provide information regarding the presence and nature of radioactivity).
 - Static (i.e., fixed point) measurements are quantitative (i.e., provide information regarding the amount of radioactivity). Static measurements provide numerical results that can be compared to an investigation level, release criterion, or other numerical limit.
 - Samples can be either qualitative or quantitative in nature depending on how they are collected and analyzed.
- Measurement Technique – The measurement technique depends on the selected instrument, the type of measurement, and a variety of field variables. These include:
 - Project-specific requirements (i.e., client/contract specifications);
 - Spatial location (i.e., where the measurement is collected);
 - Count time (i.e., how long the measurement is collected);
 - Scan rate (i.e., speed at which the instrument is moved over the surface);
 - Area or volume over which the measurement is collected; and
 - Characteristics of the surface or material being measured (e.g., source geometry, surface characteristics, and material background).
- Detection Sensitivity - The detection sensitivity depends on the characteristics of the selected instrument and the measurement technique. As a general rule, the detection sensitivity should be less than the investigation level, release criterion, or other applicable limit; otherwise compensatory measures are taken.

C.2 Radiation Surveys

Radiation surveys are performed using a combination of two methods: general area surveys and surface contact surveys. General area surveys are used to assess radiation fields representative of the area, to determine whether radiological conditions have changed over time, and to establish specific radiological controls for work to be performed. Surface contact surveys are used to locate and identify (1) the maximum radiation levels to which radiation workers could be exposed and (2) localized sources of radiation that present unique radiological concerns.

C.3 Contamination Surveys

Contamination surveys are performed to measure total and removable alpha/beta surface contamination. Surface (or near surface) contact surveys are performed to measure total (fixed plus removable) surface contamination. Where total and removable surface contamination measurements are collocated, the total surface contamination measurement is performed prior to the removable surface contamination measurement. Removable surface contamination may be detected and quantified using one of the following two methods:

- Smear Sample (quantitative method) - A smear is obtained by using an absorbent filter disk to wipe with moderate pressure across the area or item to be evaluated. The smear usually is wiped over an area of 100 square centimeters (cm²).
- Large Area Wipe (qualitative method) - A wipe is obtained by wiping an absorbent pad, towel, or Masslinn® over a large area or the entire surface of the item being surveyed. This qualitative method (i.e., solely indicating whether removable contamination may be present or is absent) may be performed periodically to ensure that contamination has not spread.

Both smear samples and large area wipes are evaluated for alpha/beta activity, unless otherwise directed by the Project/Site RSO.

C.4 Air Sampling

Air sampling of general and localized areas is performed when and/or where the generation of airborne radioactive material is possible. These samples are used

to verify that radioactive material is being confined effectively and to provide warning of elevated concentrations for planning or response actions. The sampling points are located in the airflow pathway near the known or suspected release point(s). As necessary, more than one air sample location may be used in order to provide a reasonable estimate of the general concentration of radioactive material in air.

Breathing zone air samples are the primary method for monitoring workers' potential inhalation intake of radioactive material. The samples are collected under known physical conditions (e.g., filter type, sample time, flow rate). The flow meters of air samplers are calibrated annually, at a minimum. Calibration is also performed after repair or modification of the flow meter.

In the event that airborne levels reach a derived air concentration (DAC) of 0.1, work controls and PPE (e.g., respirators) are evaluated and modified as necessary to ensure that a DAC limit of 1 DAC/day is not reached. DAC-hour tracking is performed for individuals with breathing zone air sample results of 1 DAC or higher. If a breathing zone air sample reports a result of 10 DAC-hours or higher without respiratory protection, the individual is restricted from work involving potential exposure to airborne radioactive material unless approved by the Project/Site RSO.

C.5 Monitoring Dose to the Public

To protect members of the public, survey and sampling protocols are implemented in areas of intrusive work. Intrusive work locations that require monitoring (e.g., areas where soil excavations and/or handling, etc., may disturb sources of radioactive material) are posted in conspicuous locations, and access is restricted.

Surveys of radiation levels outside of restricted areas and of radioactive materials in effluents released from restricted areas are performed to demonstrate compliance with the dose limits for individual members of the public. [10 CFR 20.1302; NUREG-1556, Vol. 18, Rev. 1, Section 8.10.7]

C.6 Survey and Sampling Frequencies

A routine program consisting of daily, weekly, and monthly survey and sampling is implemented under the direction of the Project/Site RSO. Survey and

sampling are conducted at a frequency commensurate with the hazard(s) present and the personnel occupancy in a given area.

The performance frequency and detail level of surveys are designed to anticipate doses to radiation workers and members of the public and to ensure and verify that individual exposure to radiation or radioactive materials is adequately controlled and monitored. Considerations include:

- Likelihood of changing conditions;
- Radiological hazards of specific areas; and
- Occupancy within various areas.

Routine survey and sampling frequencies are augmented if any of the following conditions applies:

- Contamination above action levels is found outside of a restricted area;
- Unexpected, significant increases in radiation levels, contamination levels or airborne radioactivity levels occur; or
- Increased maintenance activities or changes in work scope may change radiological conditions (e.g., operations which may increase area dose rates, loose surface contamination, airborne radioactivity).

C.7 Documentation and Review

Surveys are documented legibly accurately, and timely. Survey documents include the following information at a minimum:

- Date, time, and location of the survey;
- Instrument(s) used, including serial number and calibration date;
- Name and signature of the surveyor(s);
- Name and signature of the Project/Site RSO reviewing the survey;
- Results of the measurements and analyses; and/or
- Maps and drawings indicating the area(s) and items surveyed, as appropriate.

Standardized documentation methods, supervisory reviews, and information distribution are used.

D. Required Records

Records from surveys describing the location and amount of subsurface residual radioactivity identified at the temporary job site are kept with records important for decommissioning. [10 CFR 20.1501(b)]

Survey and sampling records are maintained until termination of the license. **[10 CFR 20.2103]**

E. Acronyms and Definitions

Terms as they are used in the Company's radiation safety program are defined in the glossary of terms in MA-RP-101, Radiation Safety.

F. References

Code of Federal Regulations, Title 10 (10 CFR)

- 10 CFR 20.1302, *Compliance with Dose Limits for Individual Members of the Public*
- 10 CFR 20.1501, *General*
- 10 CFR 20.2103, *Records of Surveys*

NUREG-1556, Vol. 18, Rev. 1, *Consolidated Guidance About Materials Licenses, Program-Specific Guidance About Service Provider Licenses*, NRC, August 2017.

- Section 8.10.4, Surveys
- Section 8.10.7, Public Dose

Gilbane Federal Policy PO-RP-100, Radiation Safety.

Gilbane Federal Manual MA-RP-101, Radiation Safety.

G. Attachments

None.



PROCEDURE
Document PR-RP-160
Version R01

Radiation and Contamination Control

A blue ink signature of Jerry C. Cooper, written in a cursive style.

Jerry C. Cooper, Corporate Radiation Safety Officer

01 Oct 2019

Date

Version	Changes	Affects Section	Effective Date
0.0	Initial Publication	Not Applicable	01 Jan 2016
1.0	Updated regulatory references and terms; added 10 CFR 20.1903 exceptions	C, F, G	01 Nov 2019

Rescission

CO-ITSI-RSOP-07.02 Unrestricted Release Program, Revision 2, dated 18 Feb 2014

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Attachment 1	Acceptable Surface Contamination Levels

A. Purpose and Scope

This procedure describes the implementation of the Gilbane Federal (“Company”) policy that, wherever possible, radiation and contamination are controlled at their source. Administrative procedures (e.g., restricted areas, barriers, postings) and engineering controls (e.g., shielding, containment, ventilation) are used, to the maximum extent practicable, to minimize contamination of the work area and the environment and the generation of radioactive waste.

This procedure applies to Company and contractor radiation safety officers (RSOs) and radiation protection personnel. Bolded information in brackets, e.g., **[NRC License Condition 25]**, references the source for the procedural provision.

B. Responsibilities

B.1 Project/Site RSO

The Project/Site RSO is responsible for:

- Ensuring that the qualifications of radiation protection personnel in radiation and contamination controls are sufficient for project/site needs;
- Verifying that the radiation levels at which a radiologically controlled area (RCA) is established are appropriate;
- Verifying that radiological postings contain appropriate information;
- Establishing and authorizing any exceptions to restricted area access/egress requirements; and
- Approving the use of shielding, containment devices, and ventilation systems, including high-efficiency particulate air (HEPA) filtered vacuums, inside a restricted area.

B.3 Radiation Protection Personnel

Radiation protection personnel are responsible for implementing radiation and contamination controls under the direction of the Project/Site RSO.

C. Method

Restricted areas are established to protect individuals from exposure to radiation and radioactive materials, and to maintain individual exposures as low as reasonably achievable (ALARA). Measures are instituted to monitor and control contamination. **[10 CFR 20.1406; NUREG-1556, Vol. 18, Rev. 1, Section 8.10.4]**

C.1 Restricted Areas Controlled for External Radiation Exposure

The dose to individual members of the general public is not to exceed 2 mrem in any one hour outside a restricted area. [10 CFR 20.1301(a)(2)]

A restricted area established to control external radiation exposure is posted as an RCA, a Radiation Area, or a High Radiation Area.

- RCA - Any area in which radiation levels could result in an individual receiving a dose equivalent in excess of 100 millirem (mrem) in a single year (excluding natural background and medical exposures) is posted as an RCA. For external sources, the RCA typically is posted when the radiation level exceeds 50 microroentgens per hour ($\mu\text{R/hr}$) at a distance of 30 centimeters (cm) from the radiation source or from any surface that the radiation penetrates. However, this radiation level may be modified at the discretion of the Project/Site RSO based on accurately assessed occupancy factors.
- Radiation Area - Any area with radiation levels greater than 5 mrem in one hour at 30 cm from the source or from any surface through which radiation penetrates is posted as a Radiation Area. [10 CFR 20.1003]
- High Radiation Area - Any area with dose rates greater than 100 mrem in one hour 30 cm from the source or from any surface through which the ionizing radiation penetrates is posted as a High Radiation Area. [10 CFR 20.1003]

C.2 Restricted Areas Controlled for Internal Radiation Exposure

A restricted area established to control internal radiation exposure is posted as a Contamination Area, High Contamination Area, Airborne Radioactivity Area, or Radioactive Materials Area.

- Contamination Area - Any area in which removable activity exceeds the acceptable surface contamination levels specified in Table 3 of Regulatory Guide 8.23, *Radiation Safety Surveys at Medical Institutions*, but do not exceed 100 times those values, is posted as a Contamination Area. Regulatory Guide 8.23 values are reproduced in Attachment 1.
- High Contamination Area - Any area in which removable activity exceeds or is likely to exceed 100 times the acceptable surface contamination levels specified in Table 1 of Regulatory Guide 8.23 is posted as a High

Contamination Area. Regulatory Guide 8.23 values are reproduced in Attachment 1.

- Airborne Radioactivity Area - A room, enclosure, or area is posted as an Airborne Radioactivity Area where airborne radioactive materials composed wholly or partly of licensed material are present in concentrations in excess of a derived air concentration (DAC) specified in state and federal regulatory documents, or at such a level that an individual present in the area without respiratory protective equipment could exceed an intake of 0.6 percent of the annual limit on intake (ALI) or 12 DAC-hours during the hours an individual is present in one week. [10 CFR 20.1003]
- Radioactive Materials Area - An area or room in which licensed material is used or stored, and in which the quantity of radioactive material exceeds 10 times (100 times for natural uranium or thorium) the quantity of such material specified in Appendix C of 10 CFR 20 is posted as a Radioactive Materials Area. [10 CFR 20.1902(e)]

C.3 Boundaries, Barriers, and Postings

Restricted area boundaries are demarcated using yellow and magenta floor tape, rope, ribbon, barricades, or other suitable identifier. Hoses or electrical leads which cross restricted area boundaries are secured to prevent the hose or lead from being pulled into or out of area inadvertently and spreading contamination.

Barriers, signs, gates, doors, fences, etc., are used to identify the boundary of the restricted area and to control access of personnel into the area. A minimum of one sign is posted on each straight run of the boundary of the restricted area. Additional signs are placed at approximately 30-meter (100-feet) intervals on long runs of the boundary. For waterfront areas, signs are posted at areas accessible by watercraft. Areas not typically accessed by pedestrians (e.g., windows) do not requiring posting.

Postings consist of standardized signs or labels bearing the standard radiological trefoil symbol in magenta, purple, or black on a yellow background. Postings provide information concerning a specific radiological hazard and include the wording: "Caution: Controlled Area" or "Caution: Restricted Area."

Supplemental information as specified by the Project/Site RSO also may be included as magenta (preferred), purple, or black markings on a yellow (preferred) or white background. [10 CFR 20.1901; 10 CFR 20.1902]

Posting for a period of less than 8 hours is not required when the area is under the continuous observation and control of an individual knowledgeable of, and empowered to implement, required access and exposure control measures. **[10 CFR 20.1903(a)]**

Posting of a room or area is not required because of the presence of sealed sources provided the radiation level does not exceed 50 μ R/hr at a distance of 30 cm from the surface of the source container or housing. **[10 CFR 20.1903(c)]**

C.4 Access and Egress

An access control point is established to provide control over entry to and exit from the restricted area. The access control point allows for accountability of personnel, tools, and equipment that pass into and out of the area and also may function as a contamination control boundary between zones of differing contamination levels. Placement of the access control point takes into account radiation levels in the area. During periods of inactivity, the access control point is secured.

Individuals are monitored for contamination on their person when exiting a restricted area in accordance with PR-RP-180, Personnel Protection and Emergency Response.

Hand-carried items are monitored for contamination when being removed from a restricted area. Suitable radiation detection instruments are provided at or near the access control point. The nature and extent of the monitoring are dictated by the radiological posting and/or the radiation work permit (RWP). Hand-carried items leaving a restricted area must meet the average and removable levels for acceptable surface contamination specified in Table 1 of Regulatory Guide 8.23 (see Attachment 1).

A step-off-pad is provided at the access control point of restricted areas controlled for contamination. Step-off pads are treated as non-contaminated. Protective clothing is donned prior to entry to the restricted area and removed prior to exiting onto the step-off pad. In areas where more than one set of protective clothing is used (e.g., High Contamination Area), additional step-off pads may be used to prevent the spread of contamination. Appropriate receptacles are provided to collect used protective clothing. Radioactive or contaminated trash is not allowed to accumulate, to avoid a potential

contribution to increased ambient radiation levels. The appropriate radiation detection instruments are provided at or near the step-off-pad or work area in a manner that will facilitate their use by radiation workers.

Individuals with open wounds or sores generally are not granted access to a restricted area controlled for contamination. Entry may be authorized by the Project/Site RSO on a case-by-case basis, if appropriate protection of the wound or sore is verified, planned work activities are unlikely to compromise the protection, and there is no other medical reason to restrict entry.

C.5 Tools, Equipment, and Materials

Tools, equipment, and materials entering and exiting restricted areas are controlled to ensure that unnecessary items are not brought into the restricted area and that contaminated items are not removed from the restricted area inadvertently. Good housekeeping practices are used at all times. Work areas are cleaned up and housekeeping performed after each task is completed.

Only tools, equipment, and material necessary to accomplish the planned task are allowed into a restricted area. Work activities are pre-planned to minimize the number of tools and/or equipment and the quantity of material taken into restricted areas. Personal items are not allowed. Container wrappings, packing, and similar materials are segregated from essential items before the items are brought into a restricted area. The use of wooden pallets and other materials difficult to survey and decontaminate is minimized inside restricted areas.

Non-radioactive materials are not stored in a restricted area. As far as possible, tools or equipment present within a restricted area are used, rather than introducing additional tools or equipment to the restricted area. Containers of temporarily stored restricted area tools and equipment, such as toolboxes and crates, are labeled as radioactive materials. Items brought into a restricted area are removed as soon as practical to reduce the likelihood that they become contaminated inadvertently.

Vehicles and large pieces of equipment (including rental equipment) are surveyed upon entry to and exit from a restricted area. These surveys include any material conveyed by the vehicle. In some cases, soil, debris, and miscellaneous materials may not be surveyed because the likelihood of contamination is very low. Exceptions, if any, are approved by the Project/Site RSO. Where the average and removable acceptable surface contamination

levels in Attachment 1 are not met, the vehicle or equipment is returned to the supplier for replacement or decontamination.

Tools and equipment are bagged, sleeved, covered, or coated, as appropriate, before being taken into a restricted area and surveyed for contamination prior to removal. Survey intensity is established by the Project/Site RSO as appropriate to site and work conditions. Where the average and removable acceptable surface contamination levels in Attachment 1 are not met, items are decontaminated before leaving the restricted area or stored for disposal as radioactive waste.

Appropriate decontamination techniques may include dry wipes, low pressure spray (power wash), high-pressure spray, ultra-high pressure spray, scabbling, scarification, abrasive blast methods, needle gun, and removal of strippable coatings. If decontamination methods are unsuccessful, the contaminated items may be retained for future use within the restricted area. If it is not feasible or cost-effective to control or decontaminate an item, the item is disposed of as radioactive waste.

C.6 Shielding

Shielding may be used to reduce radiation levels in and around work areas. Temporary shielding normally consists of lead blankets, aprons, drapes, and is specified in the respective RWP for each job requiring shielding. The type of shielding and the manner of its use are approved by the Project/Site RSO. A survey is performed following the installation, modification, and removal of shielding to verify proper radiological postings.

C.7 Ventilation and Containment

Ventilation systems and containment devices (e.g., glove boxes, containment tents, catch basins) are approved by the Project/Site RSO and used, where appropriate, to control the movement of airborne radioactivity and prevent or minimize the spread of contamination. Ventilation systems are checked routinely for proper operation and airflow. Containments are inspected for integrity and proper function. Only HEPA-filtered vacuums are used inside a restricted area, unless otherwise approved by the Project/Site RSO. HEPA-filtered vacuums used within the restricted area are labeled as radioactive material and maintained (e.g., emptied, filter changed) using appropriate radiological controls.

D. Required Records

None.

E. Acronyms and Definitions

Terms as they are used in the Company's radiation safety program are defined in the glossary of terms in MA-RP-101, Radiation Safety.

F. References

Code of Federal Regulations, Title 10 (10 CFR)

- 10 CFR 20.1003, *Definitions*
- 10 CFR 20.1301, *Dose Limits for Individual Members of the Public*
- 10 CFR 20.1406, *Minimization of Contamination*
- 10 CFR 20.1901, *Caution Signs*
- 10 CFR 20.1902, *Posting Requirements*
- 10 CFR 20.1903, *Exceptions to Posting Requirements*
- 10 CFR 20, Appendix C, *Quantities of Licensed Material Requiring Labeling*.

NUREG-1556, Vol. 18, Rev. 1, *Consolidated Guidance About Materials Licenses, Program-Specific Guidance About Service Provider Licenses*, NRC, August 2017.

- Section 8.10.4, Surveys

Regulatory Guide 8.23, *Radiation Safety Surveys at Medical Institutions*. Revision 1. January 1981.

Gilbane Federal Policy PO-RP-100, Radiation Safety.

Gilbane Federal Manual MA-RP-101, Radiation Safety.

Gilbane Federal Procedure PR-RP-180, Personnel Protection and Emergency Response.

G. Attachments

Attachment 1 - Acceptable Surface Contamination Levels

Attachment 1 – Acceptable Surface Contamination Levels

(Source: Regulatory Guide 8.23, *Radiation Safety Surveys at Medical Institutions*)

Nuclide ^a	Average ^{b,c}	Maximum ^{b,d}	Removable ^{b,e}
U-nat, U-235, U-238, and associated decay products	5,000 dpm α /100 cm ²	15,000 dpm α /100 cm ²	1,000 dpm α /100 cm ²
Transuranics, Ra-226, Ra-228, Th-230, Th-228, Pa-231, Ac-227, I-125, I-129	100 dpm/100 cm ²	300 dpm/100 cm ²	20 dpm/100 cm ²
Th-nat, Th-232, Sr-90, Ra-223, Ra-224, U-232, I-126, I-131, I-133	1,000 dpm/100 cm ²	3,000 dpm/100 cm ²	200 dpm/100 cm ²
Beta-gamma emitters (nuclides with decay modes other than alpha emission or spontaneous fission) except Sr-90 and others noted above.	5,000 dpm β - γ /100 cm ²	15,000 dpm β - γ /100 cm ²	1,000 dpm β - γ /100 cm ²

Notes:

^a Where surface activity by both alpha (α) and beta-gamma (β - γ) emitting nuclides exists, the levels established for α and β - γ emitting radionuclides should be applied independently.

^b As used in this table, dpm (disintegrations per minute) means the rate of emission as determined by correcting the counts per minute observed by an appropriate detector for background, efficiency, and geometric factors associated with the instrumentation.


^c Measurements of average activity should not be averaged over more than 1 square meter. For objects of less surface area, the average should be derived for each object.

^d The maximum contamination level applies to an area of not more than 100 square centimeters (cm²).

^e The amount of removable activity per 100 cm² of surface area should be determined by wiping that area with dry filter or soft absorbent paper, applying moderate pressure, and assessing the amount of radioactivity on the wipe with an appropriate instrument of known efficiency. When removable activity on objects of less surface area is determined, the pertinent levels should be reduced proportionally and the entire surface should be wiped.



Radioactive Material Control



Jerry C. Cooper, Corporate Radiation Safety Officer

05 Dec 2019

Date

Version	Changes	Affects Section	Effective Date
0.0	Initial Publication	Not Applicable	01 Jan 2016
1.0	Updated/added regulatory references, license conditions, 10 CFR 37 security requirements	B, C, D, F, G	01 Nov 2019
2.0	Updated NRC license conditions	C, F	05 Dec 2019

Rescission
CO-ITSI-RSOP-06.02 Radioactive Material Control, Storage, Receipt and Transportation, Revision 2, dated 18 Feb 2014

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Attachment 4	Transport of Radioactive Material in Aggregate Form (California only)

A. Purpose and Scope

This procedure describes the implementation of the Gilbane Federal (“Company”) policy that positive control of radioactive material be established to prevent inadvertent release of radioactive material to unrestricted areas, and ensure the protection of radiation workers and members of the public. Licensed material is transferred between licensees and transported in accordance with regulatory requirements.

This procedure applies to Company and contractor radiation safety officers (RSOs) and radiation protection personnel. Bolded information in brackets, e.g., [**NRC License Condition 25**], references the source for the procedural provision.

B. Responsibilities

B.1 Corporate RSO

The Corporate RSO is responsible for:

- Assessing the radionuclide activity thresholds in Title 10, Code of Federal Regulations (10 CFR) Part 37, Appendix A to determine specific requirements to protect licensed material from theft or diversion; and
- Authorizing Company transport of radioactive material NOT exempt from 10 CFR 71; and Title 17, California Code of Regulations (17 CCR), Section 30373.

B.2 Project/Site RSO

The Project/Site RSO is responsible for:

- Ensuring that the qualifications of radiation protection personnel in radioactive material control are sufficient for project/site needs;
- Verifying that radioactive material introduced onto the temporary job site complies with license conditions;
- Notifying the client/site owner prior to introducing radioactive material onto the temporary job site and ensuring that the radioactive material is secure;
- Ensuring licensed material is protected from theft or diversion;
- Ensuring that radioactive material is transported in accordance with applicable regulatory requirements, including authorizing Company transport of radioactive material exempt from 10 CFR 71 and 17 CCR 30373; and
- Authorizing the transfer of licensed material to another licensee.

B.3 Radiation Protection Personnel

Radiation protection personnel are responsible for implementing radioactive material control under the direction of the Project/Site RSO.

C. Method

Radioactive material in both sealed and unsealed form, including exempt quantity sources, typically is controlled as licensed material. As a general rule, material in the form of excavated soil, building materials, debris, stockpiles, etc. that may contain radioactive material is controlled as radioactive material until it has been cleared or otherwise determined to be exempt from regulatory requirements for purposes of licensing and/or transportation.

C.1 Receipt and Inspection

The Project/Site RSO ensures that the type(s) and uses of radioactive material received at the temporary job site are authorized by license and that the possession limits are not exceeded (see Attachment 3 to PR-RP-110, Radiation Safety Program Administration). **[NUREG-1556, Vol. 18, Rev. 1, Appendix E]**

The Company may not take possession of radioactive material from its clients without prior written approval from the U.S. Nuclear Regulatory Commission (NRC). **[NRC License Condition 11]**

Except for calibration sources, reference standards, and radioactively contaminated equipment owned by the Company, use of licensed material at each temporary job site is limited to material originating from that site. **[NRC License Condition 13; California License Condition 16]**

Byproduct material, source material, and special nuclear material wastes may not be imported into the U.S. or onto the temporary job site. **[NRC License Condition 24]**

Radioactive material is inspected upon receipt by radiation protection personnel using the guidelines in Attachment 1. If there are any obvious signs of damage to the package, the Project/Site RSO is notified immediately and no further action is taken until directed by the Project/Site RSO. **[10 CFR 20.1906(e)]**

C.2 Security and Accountability

Where the total quantity of licensed material possessed at a temporary job site may exceed 10.8 curies (0.4 terabecquerels), the Corporate RSO assesses the radionuclide activity thresholds in 10 CFR 37 Appendix A to determine the specific requirements for access to, and use, transfer, and transport of licensed material to protect it from theft or diversion. **[10 CFR 37; NUREG-1556, Vol. 18, Rev. 1, Section 8.10.11]**

The Project/Site RSO notifies the client/site owner prior to introducing radioactive material onto the temporary job site and ensures that the radioactive material is stored in a restricted area under secure conditions (i.e., controlled access). **[10 CFR 20.1801]**

Discrete radioactive sources (e.g., objects or items containing radioactive material with high specific activity and localized dose rates) are stored under lock and key. An inventory is maintained that includes descriptive information for each source (e.g., description of the item; radiation levels on contact and at 30 centimeters). **[10 CFR 20.1801]**

A physical inventory is conducted by radiation protection personnel every six months (or at other intervals approved by the NRC) to account for sources and/or devices received and used under license. **[NRC License Condition 22]**

C.3 Sealed Sources

Sealed sources or detector cells containing licensed material are not to be opened, and sources are not to be removed from source holders. **[NRC License Condition 21]**

Remote handling tools are used when handling sealed sources, except low-activity calibration sources. **[NUREG-1556, Vol. 18, Rev. 1, Section 8.10.1]**

Routine maintenance of devices sealed sources or devices are conducted according to manufacturer (or distributor) written recommendations and instructions. Routine maintenance includes the cleaning, lubrication, changing batteries, relays, or fuses. **[NUREG-1556, Vol. 18, Rev. 1, Section 8.10.9]**

Non-routine maintenance of sealed sources or devices are performed by the manufacturer (or distributor) or other person authorized by the NRC (or State of California, if under the California License). Non-routine maintenance includes the repair, removal, replacement, or alteration involving activities during which

personnel could receive radiation doses exceeding NRC limits. [NUREG-1556, Vol. 18, Rev. 1, Section 8.10.9]

Sealed sources are tested for leakage and/or contamination by radiation protection personnel as described in Attachment 2 except if they contain one or more of the following:

- Hydrogen-3 only;
- Radioactive material in a gaseous form only;
- A radionuclide with a half-life of 30 days or less;
- Not more than 100 microcuries (μCi ; 3,700 kilobecquerels [kBq] or 2.22×10^8 disintegrations per minute [dpm] of beta and/or gamma-emitting material); or
- Not more than 10 μCi (370 kBq or 2.22×10^7 dpm) of alpha-emitting material. [NRC License Condition 23.D]

C.4 Transport

As a general rule, the Company does not transport radioactive material, except:

- Exempt and non-exempt quantity radioactive check sources (to/from temporary job site locations);
- Discrete radioactive sources recovered during remediation (to a radioactive material area from the recovery location); and
- Bulk quantities of potentially radioactive material in aggregate form (to/from survey and/or stockpile areas).

The Project/Site RSO authorizes Company transport of radioactive material exempt from 10 CFR 71 under NRC license and 17 CCR 30373 under California license, i.e., where the activity concentrations do not exceed the values for exempt material given in the following table. [10 CFR 71.14(a); 17 CCR 30373(a)]

Radionuclide	Activity (pCi/g) ^a	Radionuclide	Activity (pCi/g) ^a
Am-241	27 (1 Bq/g)	Sr-90 ^d	2,700 (100 Bq/g)
Cs-137 ^b	270 (10 Bq/g)	Th-232	270 (10 Bq/g)
Pu-239	27 (1 Bq/g)	U-235 ^e	270 (10 Bq/g)
Ra-226 ^c	270 (10 Bq/g)	U-238 ^f	270 (10 Bq/g)

Notes

^a values taken from 10 CFR 71, Appendix A, Table A-2, and shown in units of picocuries per gram (pCi/g) and becquerels per gram (Bq/g); exempt material activity concentrations for radionuclides not listed here may be found in Table A-2.

^b includes progeny Ba-137m

Radionuclide	Activity (pCi/g) ^a	Radionuclide	Activity (pCi/g) ^a
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^c includes progeny Po-218, Pb-214, Bi-214, Po-214, Pb-210, Bi-210, and Po-210

^d includes progeny Y-90

^e includes progeny Th-231

^f includes progeny Th-234 and Pa-234m

Company transport of radioactive material under NRC license that is NOT exempt from 10 CFR 71, or under California license that is NOT exempt from 17 CCR 30373, may be authorized only by the Corporate RSO.

Transport of radioactive material over public roads and highways is performed in accordance with Attachment 3. For California only, transport of radioactive material in aggregate form (i.e., rock fragments, pebbles, sand, dirt, gravel, cobbles, crushed base, asphalt, and other similar materials), is performed in accordance with both Attachment 3 and Attachment 4.

C.5 Waste Management

Waste is handled, stored, and disposed of in a manner protective of human health and the environment and in accordance with regulatory requirements. Contact with radioactive material or exposure to ionizing radiation is maintained as low as reasonably achievable (ALARA) and consistent with technology, cost, and operational requirements. [NUREG-1556, Vol. 18, Rev. 1, Section 8.11 and Appendix M]

- **Solid Waste**. Radioactive objects and radioactively contaminated material such as sediment, discarded protective clothing, and investigation-derived waste generated during radiological screening activities are disposed as solid low-level radioactive waste (LLRW). Where possible, waste materials are loaded into roll-off boxes, drums, or other appropriate containers at the point of generation. Wastes that are not loaded directly into transport containers are stored in covered containers or in covered stockpiles. Containers are covered before being moved from the point of generation. Once containerized, waste is stored at a suitable location designated by the client and remains under the Company's control until transferred.
- **Liquid Waste**. As a general rule, radiological work activities are designed to avoid the use of significant quantities of liquids requiring treatment and/or disposal. Minimal use of water is anticipated for dust control activities; however, the generation of free water is avoided. Accumulated water is managed in a manner similar to storm water run-off. Collected water may be

used for dust control or is filtered or otherwise treated and sampled to verify compliance with the release criteria prior to discharge. Filters and sediment from water treatment are handled as solid waste.

- **Handling and Storage.** Waste may be stored temporarily at client-designated locations for sampling and analysis, to accumulate sufficient quantities for economical transportation and disposal, or to coordinate transportation between the carrier and the disposal site. Access to handling and storage areas is controlled. Waste storage areas are enclosed and/or roped-off, and appropriately posted. An inventory of containers storing radioactive waste is maintained and available for on-site inspection. A waste information fact sheet is prepared for each discrete radioactive object/high specific activity material that is recovered and includes information such as photographs of the source, radionuclide identification, estimated curie content, and radiological survey information.
- **Disposal.** As a general rule, waste disposal is accomplished by transferring the wastes to a licensed commercial waste treatment, storage, or disposal facility.

C.6 Transfer

The Project/Site RSO authorizes the transfer of radioactive material to another licensee. Transfers involving transport of radioactive material are made in accordance with Section C.4.

Except for calibration sources, reference standards, and radioactively contaminated equipment owned by the Company, licensed material at each temporary job site may either be transferred to an authorized recipient or remain at the site after Company activities are completed. **[NRC License Condition 13; California License Condition 16]**

Licensed material possessed at a temporary job site incident to performing services may not be transferred to the Company unless the Company is preparing it to be shipped AND will be the shipper of record (i.e., signing the Shipper's Certification on the shipping papers). **[NUREG-1556, Vol. 18, Rev. 1, Appendix E]**

D. Required Records

Records of inventories of sealed sources and devices are maintained for 3 years from the date of each inventory and include the radionuclides, quantities, manufacturer's name and model numbers, and the date of the inventory. **[NRC License Condition 22]**

Records of sealed source leak test results are kept in units of microcuries (becquerels) and maintained available for inspection by the NRC for 5 years. **[NRC License Condition 23.H]**

E. Acronyms and Definitions

Terms as they are used in the Company's radiation safety program are defined in the glossary of terms in MA-RP-101, Radiation Safety.

F. References

U.S. Nuclear Regulatory Commission (NRC) Radioactive Materials License No. 04-29353-01, Amendment 08, dated November 22, 2019.

State of California Radioactive Material License No. 7948-07, Amendment 13, dated November 15, 2019.

Code of Federal Regulations, Title 10 (10 CFR)

- 10 CFR 20.1801, *Security of Stored Material*
- 10 CFR 20.1802, *Control of Material Not in Storage*
- 10 CFR 20.1906, *Procedures for Receiving and Opening Packages*
- 10 CFR 30.50, *Reporting Requirements*
- 10 CFR 32.210, *Registration of Product Information*
- 10 CFR 37, *Physical Protection of Category 1 and Category 2 Quantities of Radioactive Material*
- 10 CFR 71, *Packaging and Transportation of Radioactive Material*

Code of Federal Regulations, Title 49 (49 CFR)

- 49 CFR 172, *Hazardous Materials Table, Special Provisions, Hazardous Materials Communications, Emergency Response Information, Training Requirements, and Security Plans*
- 49 CFR 173, *Shippers – General Requirements for Shipments and Packagings*
- 49 CFR 177, *Carriage by Public Highway*

California Code of Regulations, Title 17 (17 CCR)

- 17 CCR 30373, *Transportation Regulations*

California Vehicle Code (CVC) Section 23114, *Spilling Loads on Highways*.

NUREG 1556, Volume 18, Rev. 1, *Consolidated Guidance About Materials Licenses, Program-Specific Guidance About Service Provider Licenses*, U.S. Nuclear Regulatory Commission. August 2017.

- Section 8.10.2, Material Receipt and Accountability
- Section 8.10.1, Operating and Emergency Procedures
- Section 8.10.5, Leak Tests
- Section 8.10.9, Maintenance
- Section 8.10.11, Security Program for Category 1 and Category 2 Materials
- Section 8.11, Item 11: Waste Management
- Appendix E, Material Ordering and Package Receipt and Opening
- Appendix G, Model Leak Test Program
- Appendix M, Model Waste Disposal Program

Gilbane Federal Policy PO-RP-100, Radiation Safety.

Gilbane Federal Manual MA-RP-101, Radiation Safety.

Gilbane Federal Procedure PR-RP-110, Radiation Safety Program Administration.

G. Attachments

Attachment 1 – Receipt Inspection Guidelines [**NUREG-1556, Vol. 18, Rev. 1, Appendix E**]

Attachment 2 – Sealed Source Leak Testing [**NRC License Condition 23; California License Condition 15; NUREG-1556, Vol. 18, Rev. 1, Section 8.10.5 and Appendix G**]

Attachment 3 – Transport of Radioactive Material Over a Public Road or Highway [**NRC License Condition 10.C; California License Condition 20; 49 CFR 172, 173, and 177**]

Attachment 4 – Transport of Radioactive Material in Aggregate Form (California only) [**California Vehicle Code (CVC) Section 23114**]

Attachment 1 – Receipt Inspection Guidelines

Visual Inspection of Unopened Package

The package is visually inspected for any sign of damage (e.g. crushing, punctures), with precautions to prevent contamination, such as gloves to prevent hand contamination. If damage to the package is noted, the Project/Site RSO is notified and no further action is taken. The carrier, if still present, is asked to remain until it can be confirmed that neither the carrier nor the vehicle is contaminated.

Contamination and Radiation Survey

A contamination and radiation survey of the external surfaces of the package is performed as soon as practicable, but not later than 3 hours after receipt of the package. A package that is not labeled or is labeled as an excepted package (UN 2910), and does not show signs of any damage does not require a radiation and contamination survey. **[NUREG-1556, Vol. 18, Rev. 1, Section 8.10.2]**

Acceptable Contamination and Radiation Levels

The limits for non-fixed (removable) radioactive contamination on the external surfaces of the package are 2,400 disintegrations per minute per 100 square centimeters (dpm/100 cm²) beta-gamma and 240 dpm/100 cm² alpha. The limits for radiation levels are 200 millirem per hour (mrem/hr) at any point on the external surface of the package and 10 mrem/hr at 1 meter from any surface. **[49 CFR 173.443; 10 CFR 71.87(i); 10 CFR 71.47(a)]**

Opening Package

The outer package is opened following the supplier's directions, if provided, and the packing slip is removed. The inner package is opened to verify that the contents do not exceed license possession limits (e.g., by comparing the contents with the packing slip and/or the label on the container). The integrity of the final source container is verified (e.g., by inspecting for breakage of seals or vials, loss of liquid, discoloration of packaging material, or a high count rate on a smear).

Notifications

The Project/Site RSO is notified if anything unexpected is found. The carrier and the NRC are notified within 24 hours if acceptable limits for contamination and/or radiation are exceeded. **[10 CFR 20.1906(d)]**

Attachment 2 – Sealed Source Leak Testing

Leak testing of sealed sources is performed by qualified radiation protection personnel only.
[NRC License Condition 23.G; California License Condition 15]

Testing Prior to Initial Use

In the absence of a certificate from a transferor indicating that a leak test has been made prior to the transfer within the intervals specified in the certificate of registration issued by the NRC under 10 CFR 32.210 or by an Agreement State, a sealed source received from another licensee may not be put into use until it is tested for leakage and/or contamination and the test results are received and approved. **[NRC License Condition 23.C]**

Testing Frequency

Sealed sources designed to emit alpha particles are tested for leakage and/or contamination at intervals not to exceed 3 months. All other sealed sources are tested at intervals not to exceed 6 months, or at such other intervals as are specified by the certificate of registration referred to in 10 CFR 32.210. **[NRC License Conditions 23.A and 23.B]**

Sealed sources do not require testing if they are in storage and are not being used. However, when they are removed from storage for use or transferred to another licensee, and have not been tested within the required leak test interval, they are tested before use or transfer. Additionally, no sealed source may remain in storage for a period of more than 10 years without being tested for leakage and/or contamination. **[NRC License Condition 23.E]**

Test Performance

Tests for leakage and/or contamination are performed by the Company or by other persons specifically licensed by the NRC or an Agreement State to perform such services. The leak test must be capable of detecting the presence of 0.005 μCi (185 Bq or 11,000 dpm) of radioactive material on the test sample. **[NRC License Conditions 23.F and 23.G]**

Tests are conducted by wiping the most accessible area (but not directly from the surface of the source) where contamination would accumulate if the sealed source were leaking (e.g., the storage container). **[NUREG-1556, Vol. 18, Rev. 1, Appendix G]**

Leakage and/or Contamination

If the test reveals the presence of 0.005 μCi (185 Bq or 11,000 dpm) or more of removable contamination, the source is removed immediately from service and decontaminated, repaired,

Attachment 2 – Sealed Source Leak Testing

or disposed of in accordance with NRC regulations. A report is filed with the NRC in accordance with 10 CFR 30.50(c)(2) within 5 days of the date the leak test result is known. The report is sent to the NRC at the address given in the NRC license. The report specifies the source involved, the test results, and corrective action taken. [**NRC License Condition 23.F**]

Attachment 3 – Transport of Radioactive Material Over a Public Road or Highway

Transport of radioactive material over a public road or highway is performed in accordance with the following regulatory requirements, as applicable, based on the type and quantity of radioactive material and its mode of transportation.

- DOT – 49 CFR 172, 173, and 177; and
- State regulations and local ordinances (based on temporary job site location).

In addition to the above, unless otherwise exempt, licensed material is transported in accordance with the provisions of 10 CFR Part 71. [**NRC License Condition 10.C**]

In addition to the above, unless otherwise exempt, radioactive materials transported or delivered to a carrier for shipment in California must comply with the packaging, marking, labeling, loading, storage, placarding, monitoring, and accident reporting requirements of 17 CCR 30373. Shipping papers are maintained for inspection pursuant to DOT requirements (49 CFR 172.200 through 172.204). [**California License Condition 20**]

The following provisions apply to the transport of radioactive material:

- Transport is performed by a DOT hazmat-trained person. [**49 CFR 177.816; 49 CFR 172 Subpart H**]
- The shipment is accompanied by shipping papers. [**49 CFR 177.817; 49 CFR 172 Subpart C**]
- The shipment is properly loaded and unloaded. [**49 CFR 177.834 and 177.842**]
- A survey of the transport container is performed prior to transport. [**49 CFR 177.843**]
- The shipment is properly packaged. [**49 CFR 173 Subpart I**]
- The package, freight container, and transport vehicle are properly marked. [**49 CFR 172 Subpart D**]
- The package or containment device is properly labeled. [**49 CFR 172 Subpart E**]
- The transport vehicle is properly placarded. [**49 CFR 172 Subpart F**]
- The shipment is accompanied by emergency response information. [**49 CFR 172 Subpart G**]

Attachment 4 – Transport of Radioactive Material in Aggregate Form (California only)

Transport of radioactive material in aggregate form (i.e., rock fragments, pebbles, sand, dirt, gravel, cobbles, crushed base, asphalt, and other similar materials), over public roads and highways must be conducted safely, without loss of material during transport.

The transport vehicle must be constructed, loaded, and/or covered to prevent any of its contents or load from dropping, sifting, leaking, blowing, spilling, or otherwise escaping from the vehicle. **[CVC Section 23114(a)]**

Aggregate material is carried only in the cargo area of the transport vehicle. The cargo area must not contain any holes, cracks, or openings through which that material may escape, regardless of the degree to which the vehicle is loaded. **[CVC Section 23114(b)(1)]**

The transport vehicle must be equipped with the following:

- Properly functioning seals on any openings used to empty the load, including, but not limited to, bottom-dump release gates and tailgates;
- Splash flaps behind every tire, or set of tires, regardless of the position on the truck, truck tractor, or trailer;
- Center flaps at a location to the rear of each bottom-dump release gate as to trucks or trailers equipped with bottom-dump release gates;
- Fenders starting at the splash flap, that cover the tops of tires not already covered by the truck, truck tractor, or trailer body, with the leading edge of the fenders extending forward beyond the center of the axle;
- Complete enclosures on all vertical sides of the cargo area, including, but not limited to, tailgates; and
- Shed boards designed to prevent aggregate materials from being deposited on the vehicle body during top loading. **[CVC Section 23114(b)(2)]**


Transport vehicles with full rigid enclosures do not require center flaps or shed boards. **[CVC Section 23114(c)]**

The material must be covered unless the load, where it contacts the sides, front, and back of the cargo container area, remains six inches from the upper edge of the container area, and does not extend, at its peak, above any part of the upper edge of the cargo container area. **[CVC Section 23114(e)(1) and (4)]**



PROCEDURE
Document PR-RP-180
Version R02

Personnel Protection and Emergency Response



Jerry C. Cooper, Company Radiation Safety Officer

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Date

Version	Changes	Affects Section	Effective Date
0.0	Initial Publication	Not Applicable	01 Jan 2016
1.0	Updated regulatory references and license conditions	C, F	01 Nov 2019
2.0	Updated NRC license conditions	F	05 Dec 2019

Rescission

CO-ITSI-RSOP-08.01 Radiological Emergency Response, Revision 1, dated 12 Apr 2013

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A. Purpose and Scope

This procedure describes the implementation of the Gilbane Federal (“Company”) policy that personal protective clothing and equipment be commensurate with the radiological hazards in the work area and the work activities being conducted, and be used to minimize individual exposure to radiation and radioactive material during routine use. Protective measures are identified for conditions requiring emergency response.

This procedure applies to Company and contractor radiation safety officers (RSOs), radiation protection personnel, and radiation workers. Bolded information in brackets, e.g., **[NRC License Condition 25]**, references the source for the procedural provision.

B. Responsibilities

B.1 Corporate RSO

The Corporate RSO is responsible for approving any departure from license conditions and providing notification to the licensing authority of such departure.

B.2 Project/Site RSO

The Project/Site RSO is responsible for:

- Determining respiratory protection measures and the appropriate equipment to be used;
- Supervising personnel decontamination; and
- Notifying the Corporate RSO of personnel contamination events.

B.3 Radiation Protection Personnel

Radiation protection personnel are responsible for:

- Determining the appropriate personal protective clothing and equipment to be used;
- Administering the respiratory protection program under the direction of the Project/Site RSO;
- Performing personnel contamination monitoring; and
- Notifying the Project/Site RSO of conditions requiring emergency response, and directing the on-scene response.

B.4 Radiation Workers

Radiation workers are responsible for ensuring their personal protective clothing and equipment, including respiratory protection, is proper, and for immediately responding to and informing radiation protection personnel of conditions requiring emergency response.

C. Method

C.1 Personal Protective Clothing and Equipment

Individuals are to wear personal protective clothing and equipment commensurate with contamination hazards associated with the work area and the planned activity. Personal protective clothing and equipment are selected based on the contamination levels in the work area, the anticipated work activity, worker health considerations (including possibility of heat stress), and regard for non-radiological hazards that may be present. Area and activity hazards include heat, flame, hazardous chemicals, physical obstructions, electrical shock, and limited visibility.

The primary level of protection, Modified Level D, historically has been sufficient for radiological work activities. Additional measures, if any, are determined by radiation protection personnel and specified in activity-specific radiation work permits (RWPs) based on the radiological conditions and field tasks required to perform the planned activities.

Activities that require heavy physical effort or that have an increased potential for damage to protective clothing may require additional layers or different materials, even in areas of low contamination. Protective clothing, at a minimum, is required for work in areas posted as a Contamination Area or a High Contamination Area. Site- or task-specific requirements beyond the minimum traditionally used are detailed in the site-specific health and safety plan or RWP.

C.2 Respiratory Protection

Respiratory protection equipment may be used to control inhalation of radioactive material in a manner consistent with the respiratory protection program outlined in the Gilbane Federal Corporate Health and Safety Plan. Respiratory protection measures and the appropriate equipment to be used are determined by the Project/Site RSO. The respiratory protection program is

compliant with the requirements of the Occupational Safety and Health Administration regulations, Title 29, CFR Part 1910.134. Respiratory protection devices are permitted for jobs where an ALARA review has been conducted and has determined that respirator use is ALARA. Radiation protection personnel administer the respiration protection program as described in the Gilbane Federal Corporate Health and Safety Plan. It is described as follows:

- Training, Medical Clearance, and Fit Testing - Before using a respirator, individuals (1) are trained in the proper use of respiratory protection equipment and its limitations, (2) have received medical clearance to wear a respirator, and (3) have been respirator fit-tested within the last 12 months.
- Respirator Selection and Use - Respirators are selected from those approved by the National Institute for Occupational Safety and Health (NIOSH) and/or the Mine Safety and Health Administration (MSHA) for the situation or the contaminant to which the worker may be exposed. Selection is based on the physical, chemical, and physiological properties of the contaminant, the contaminant concentration likely to be encountered, and the likely physical conditions of the workplace environment in which the respirator will be used. The potential or observed airborne radioactivity concentration also is considered in selecting the type of respiratory protection equipment to be issued.
- Respirator Inspection, Cleaning, and Maintenance – The radiation worker inspects his/her respirator with regard to operability before use. Before being reissued, radiation protection personnel clean, disinfect, and inspect each respirator with regard to operability after cleaning. Replacement or repair is done only by qualified individuals, with parts specifically designed for the brand and model of the respirator requiring repair.

C.3 Personnel Contamination Monitoring

Each individual is monitored for contamination on his/her person when exiting a restricted area. The nature and extent of the monitoring is dictated by the radiological posting, the site-specific health and safety plan, and/or the RWP. Individuals who are not qualified to self-monitor are monitored by radiation protection personnel. In the event of an emergency condition, individuals may exit a restricted area without monitoring; however, subsequent monitoring is performed by radiation protection personnel. Any level of detectable radioactivity on an individual (i.e., the skin) is considered contamination.

Where personnel monitoring is required, the palms and backs of the hands and the bottoms of shoes are monitored, at a minimum. Where a whole-body frisk is required, it should take at least two to three minutes and be performed as follows:

- Verify that the instrument is in service and is set to the proper scale, and that the audio output can be heard during frisking.
- Frisk the hands before picking up the probe.
- Hold the probe approximately 1/4 inch from the body and move it over the rest of the body in the following general sequence, at approximately 2 inches per second:
 - Head (pause at mouth and nose for approximately 5 seconds);
 - Neck and shoulders;
 - Arms (pause at each elbow);
 - Chest and abdomen;
 - Back, hips, and seat of pants;
 - Legs (pause at each knee);
 - Shoe tops;
 - Shoe bottoms (pause at sole and heel); and
 - Personnel and supplemental dosimeters.
- If the count rate increases during frisking, pause for 5 to 10 seconds over the area to provide adequate time for instrument response.
- If the count rate increases rapidly or the instrument alarms, remain in the area and notify radiation protection personnel.
- If the frisk is completed with no alarms, return the probe to its holder and leave the area. The probe should be placed on the side or face up to allow the next person to monitor his/her hands before handling the probe.

Hand-carried items are monitored for contamination in accordance with PR-RP-160, Radiation and Contamination Control.

C.4 Emergency Response

Radiation workers take immediate action and inform radiation protection personnel of conditions requiring emergency response. Radiation protection personnel notify the Project/Site RSO and direct the on-scene response. Directions provided on-scene by radiation protection personnel take precedence over guidance provided here. **[NUREG-1556, Vol. 18, Rev. 1, Section 8.10.1]**

- Radioactive Material Spill – A spill of radioactive material is usually in the form of a liquid or dry material. This may occur from a leak, a breach of a

radioactive system, or a rupture of a sealed source. Steps are taken to prevent and promptly clean up spills of radioactive material in either solid or liquid form. Immediate actions include stopping the spill, containing the spread of material, and isolating the area using barrier tape or rope. The ALARA principles of time, distance, and shielding are used. Supplementary actions include performing radiological surveys in the immediate and adjacent areas, including downwind.

- Contaminated Individual – A contaminated individual is usually discovered during routine contamination monitoring or as a result of an accident or injury. Once an individual is confirmed to be contaminated, immediate actions are necessary to minimize exposure to the individual and the spread of the contamination. These include isolating the individual, containing the contamination, and performing decontamination. As a rule, first aid/medical response always takes precedence over contamination concerns. Measures taken to contain the contamination are not to impede medical response. Radiation protection personnel provide contamination control and radiation protection support to medical personnel both on-scene and at the hospital, as needed.

No level of detectable contamination is acceptable on individuals. Radiation protection personnel survey the individual—including any injuries or wounds—to determine the extent of contamination, and perform decontamination. The Project/Site RSO supervises decontamination efforts. Appropriate decontamination techniques include tape presses, moist wipes, and soap and warm water. Supplementary actions include documenting the contamination event and performing radiological surveys in the immediate area. The Project/Site RSO notifies the Corporate RSO of personnel contamination events.

- High Radiation – This condition is defined as radiation that exceeds expected conditions (including rapidly increasing radiation levels) and warrants immediate action to minimize external exposure. It usually is discovered by a real-time radiation alarm, routine radiation monitoring using hand-held instruments, or a high supplemental dosimeter reading. Immediate actions include evacuating and isolating the area. Radiation protection personnel then assess the radiological conditions and determine the proper protective measures and supplementary actions to be instituted. Supplementary actions include evaluating affected individuals for external exposures, investigation the cause, and implementing appropriate corrective measures

to restore the area to previous conditions.

- **High Airborne Radioactivity** – This condition is defined as airborne radioactivity levels that exceed expected conditions and warrant immediate action to minimize internal exposure. It usually is detected by real-time air monitoring or determined by passive air samplers where the sampling results are found to exceed expected conditions and/or are excessive for the respirator protection devices being worn. Immediate actions include evacuating and isolating the area and securing forced air ventilation. Supplementary actions include evaluating affected individuals for internal exposures, investigating the cause, and implementing appropriate corrective measures to restore the area to previous conditions.

C.5 Departure from License Conditions

Radiation protection personnel may take reasonable actions that depart from license conditions in an emergency when immediate action is needed to protect public health and safety and no action consistent with license conditions that can provide adequate or equivalent protection is immediately apparent. The Corporate RSO approves the actions and notifies the licensing authority before, if practicable, and in any case, immediately after, such emergency action is taken. Notifications are made in accordance with PR-RP-190, Radiological Records, Notifications, and Reports. **[NRC License Condition 19]**

D. Required Records

Records of respiratory protection activities (i.e., medical clearance, respirator fit test forms, and training documentation) and personnel contamination events are maintained until termination of the license or 30 years from the date of an individual's employment termination, whichever is later. **[10 CFR 20.2102; 29 CFR 1910.1020(d)(1)]**

E. Acronyms and Definitions

Terms as they are used in the Company's radiation safety program are defined in the glossary of terms in MA-RP-101, Radiation Safety.

F. References

U.S. Nuclear Regulatory Commission (NRC) Radioactive Materials License No. 04-29353-01, Amendment 08, dated November 22, 2019.

Code of Federal Regulations, Title 10 (10 CFR)

- 10 CFR 20, *Standards for Protection Against Radiation*

Code of Federal Regulations, Title 29 (29 CFR)

- 29 CFR 1910, *Occupational Safety and Health Standards*

NUREG-1556, Vol. 18, Rev. 1, *Consolidated Guidance About Materials Licenses, Program-Specific Guidance About Service Provider Licenses*, NRC, August 2017.

- Section 8.10.1, Operating and Emergency Procedures

Gilbane Federal Corporate Health and Safety Plan.

Gilbane Federal Policy PO-RP-100, Radiation Safety.

Gilbane Federal Manual MA-RP-101, Radiation Safety.

Gilbane Federal Procedure PR-RP-160, Radiation and Contamination Control.


Gilbane Federal Procedure PR-RP-190, Radiological Records, Notifications, and Reports.

G. Attachments

None.



Radiological Records, Notifications, and Reports


Jerry Cooper, Corporate Radiation Safety Officer

05 Dec 2019
Date

Version	Changes	Affects Section	Effective Date
0.0	Initial Publication	Not Applicable	01 Jan 2016
1.0	Updated regulatory references, license conditions, and 10 CFR 20.1906 reporting	C, D, F, G	01 Nov 2019
2.0	Updated NRC license conditions	F	05 Dec 2019

Rescission

None

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A. Purpose and Scope

This procedure describes the implementation of the Gilbane Federal (“Company”) policy that records are maintained, notifications are made, and reports are submitted in accordance with Company policy and regulatory requirements.

This procedure applies to Company and contractor radiation safety officers (RSOs) and radiation protection personnel. Bolded information in brackets, e.g., [**NRC License Condition 25**], references the source for the procedural provision.

B. Responsibilities

B.1 Corporate RSO

The Corporate RSO is responsible for establishing the types of radiological records to be created and their retention requirements; and for ensuring that notifications are made and/or written reports are prepared and submitted to the Company and regulatory agencies as required.

B.2 Project/Site RSO

The Project/Site RSO is responsible for:

- Ensuring that radiological records are created and maintained;
- Notifying the Corporate RSO of conditions that warrant notifications and/or reports to the Company and regulatory agencies; and
- Preparing written reports under the direction of the Corporate RSO.

B.3 Radiation Protection Personnel

Radiation protection personnel are responsible for creating and maintaining radiological records under the direction of the Project/Site RSO.

C. Method

Records are created, used, and dispositioned in accordance with PR-MO-003, Record Retention. There is no equivalent Company procedure for notifications and reports.

C.1 Radiological Records

Records are created and maintained to substantiate compliance with relevant laws and regulatory requirements, and are retained for at least the minimum period stated in Company policy or any applicable statute or regulation.

The types of records and their retention requirements are established by the Corporate RSO and are listed in Attachment 1. As a general rule, radiation safety program records are retained for a minimum of 3 years or until license termination. The Project/Site RSO maintains records readily accessible at the temporary job site for the duration of field work to support program reviews/audits or inspections.

Records must be legible and include all pertinent information and approvals, such as stamps, initials, and signatures. **[10 CFR 20.2110]**

Records are to use the units curie, rad, and rem, including multiples and subdivisions (versus International System of Units [SI] units), and clearly indicate the units of all quantities. **[10 CFR 20.2101(a)]**

Regarding dose records, a clear distinction is made among the dose quantities entered on the records (e.g., total effective dose equivalent, shallow-dose equivalent, lens dose equivalent, deep-dose equivalent, committed effective dose equivalent). **[10 CFR 20.2101(d)]**

Records may be scanned and retained as scans, provided that the scan preserves an accurate image of the original record, including signatures, other written or graphic images, and the back of the document if information is recorded there.

A copy of the California license and records and documents pertaining to that license are maintained available for inspection at 1655 Grant Street, 12th Floor, Concord, CA 94520 **[California License Condition 26]**

C.2 Records of Information Important to Decommissioning

Records of information important to decommissioning each temporary job site are maintained at the job site and made available for inspection and to the client upon request. The records are transferred to the client for retention upon completion of activities at a temporary job site. **[NRC License Condition 18; California License Condition 19]**

Records from surveys describing the location and amount of subsurface residual radioactivity identified at the temporary job site are kept with records important for decommissioning. [10 CFR 20.1501(b)]

Records of information important to decommissioning are described in Attachment 2. In the event the Company terminates its radioactive materials license, those records will be transferred to the new licensee (if applicable). [17 CCR 30256(a)]

C.3 Immediate Notifications

Events or conditions requiring immediate notifications are listed in Attachment 3. The Project/Site RSO notifies the Corporate RSO of conditions that warrant immediate notification to the Company and/or regulatory agencies. Notifications are made by or under the direction of the Corporate RSO.

As appropriate, immediate notifications are made by telephone to:

- NRC Operations Center at (301) 816-5100 or (301) 951-0550;
- (in California) California Department of Public Health (CDPH) Radiologic Health Branch at (510) 620-3416;
- Agreement State and/or other appropriate state agencies;
- Client; and
- Other licensees on-site.

To the extent that the information is available at the time of notification, the information provided in the notification includes:

- Caller's name and call-back telephone number;
- Description of the event, including date and time;
- Exact location of the event;
- Isotopes, quantities, and chemical and physical form of the licensed material involved; and
- Any personnel radiation exposure data available.

C.4 Written Reports

Events or conditions requiring written reports are listed in Attachment 3. The Project/Site RSO notifies the Corporate RSO of conditions that warrant a written report to the Company and regulatory agencies. Written reports are prepared by the Project/Site RSO and submitted under the direction of the Corporate RSO.

To the extent possible, the information provided in the written report includes:

- A description of the event, including the probable cause;
- Date and time of the event;
- The exact location of the event;
- The manufacturer and model number (if applicable) of any equipment that failed or malfunctioned;
- The isotopes, quantities, and chemical and physical form of the licensed material involved;
- The extent of exposure of individuals to radiation or to radioactive materials (without identification of individuals by name); and
- Corrective actions taken or planned and the results of any evaluation or assessment.

C.5 Notices of Violation

Notices of violation of the regulations, and responses to such notices, are to be posted within two working days after receipt or dispatch of the documents.

These documents are to remain posted for a minimum of five working days or until the violation has been completely corrected, whichever is later. **[10 CFR 19.11(g); 17 CCR 30255(b)(4)]**

C.6 Annual Low-Level Radioactive Waste Report (California only)

Low-level radioactive waste (LLRW) reports containing information included in NRC Forms 540, 541, and 542 (and any successor forms that describe the LLRW stored and shipped by the generator) are provided to CDPH on an annual basis for both shipped and stored LLRW via the online LLRW Tracking System at <https://llrwts.cdph.ca.gov/>. **[California License Condition 24; California Health and Safety Code Section 115000.1(h)]**

D. Required Records

Records of information important to decommissioning are transferred to the client for retention following completion of activities at temporary job sites. **[NRC License Condition 18; California License Condition 19]**

E. Acronyms and Definitions

Terms as they are used in the Company's radiation safety program are defined in the glossary of terms in MA-RP-101, Radiation Safety.

F. References

U.S. Nuclear Regulatory Commission (NRC) Radioactive Materials License No. 04-29353-01, Amendment 08, dated November 22, 2019.

State of California Radioactive Material License No. 7948-07, Amendment 13, dated November 15, 2019.

Code of Federal Regulations, Title 10 (10 CFR):

- 10 CFR 19, *Notices, Instructions and Reports to Workers; Inspection and Investigations;*
- 10 CFR 20, *Standards for Protection Against Radiation;*
- 10 CFR 30, *Rules of General Applicability to Domestic Licensing of Byproduct Material;*
- 10 CFR 40, *Domestic Licensing of Source Material;*
- 10 CFR 70, *Domestic Licensing of Special Nuclear Material;*
- 10 CFR 71, *Packaging and Transportation of Radioactive Material.*

Code of Federal Regulations, Title 29 (29 CFR):

- 29 CFR 1910, *Occupational Safety and Health Standards.*

California Code of Regulations, Title 17 (17 CCR):

- 17 CCR 30253, *Standards for Protection Against Radiation;*
- 17 CCR 30255, *Notices, Instructions, and Reports to Personnel;*
- 17 CCR 30256, *Vacating Installations: Records and Notice;*
- 17 CCR 30275, *Surveys and Tests;*
- 17 CCR 30293, *Records;*
- 17 CCR 30295, *Notification of Incidents;*
- 17 CCR 30373, *Transportation Regulations.*

California Health and Safety Code, Chapter 8, *Radiation Control Law*, Article 3

- Section 115000.1

NUREG-1556, Vol. 18, Rev. 1, *Consolidated Guidance About Materials Licenses, Program-Specific Guidance About Service Provider Licenses*, NRC, August 2017.

- Section 8.10.1, *Operating and Emergency Procedures*

Gilbane Federal Policy PO-RP-100, *Radiation Safety*.

Gilbane Federal Manual MA-RP-101, *Radiation Safety*.

Gilbane Federal Procedure PR-MO-003, Records Retention.

G. Attachments

Attachment 1 – Radiological Records and Retention Periods [**NRC License Conditions 22 and 23.H; 10 CFR 20; 29 CFR 1910**]

Attachment 2 – Records of Information Important to Decommissioning [**17 CCR 30256(a)**]

Attachment 3 – Incident Notifications and Reports [**10 CFR 20 and 30; 17 CCR 30295; NUREG-1556, Vol. 18, Section 8.10.6**]

Attachment 1 – Radiological Records and Retention Periods

RADIATION PROTECTION PROGRAM ADMINISTRATION		
Record	Retention Period	Regulatory Basis
Radiation protection program records (Corporate and Project/Site RSO delegations of authority; training records; written policies, procedures, and work instructions; license implementation records)	License termination	10 CFR 20.2102(b)
Training records	License termination; or 30 years from date of individual's employment termination, whichever is later	10 CFR 20.2102(b); 29 CFR 1910.1096(i)(2)
Program reviews and audits	3 years	10 CFR 20.2102(b)
RADIATION DOSE LIMITS AND PERSONNEL MONITORING		
Records of individual monitoring results	License termination; or 30 years from date of individual's employment termination, whichever is later	10 CFR 20.2106; 29 CFR 1910.1096(n)
Records showing the results of bioassays	License termination	10 CFR 20.2103(b)(3)
Determination of prior occupational dose as maintained on NRC Form 4 or equivalent	License termination	10 CFR 20.2104(f)
Determination of prior occupational dose; records used to prepare the NRC Form 4	3 years	10 CFR 20.2104(f)
NRC Form 5 records of doses received by all individuals for whom monitoring was required	License termination	10 CFR 20.2106(c)
Records of dose to an embryo/fetus with the records of dose to the declared pregnant woman	License termination	10 CFR 20.2106(e)
RADIATION WORK PLANNING AND CONTROL		
Radiation work permits and ALARA reviews	License termination	10 CFR 20.2102(b)

Attachment 1 – Radiological Records and Retention Periods

RADIATION INSTRUMENTS AND EQUIPMENT		
Record	Retention Period	Regulatory Basis
Radiation instrument and equipment calibration, set-up, and response check records	3 years	10 CFR 20.2103(a)
RADIOLOGICAL SURVEY AND SAMPLING		
Survey and sampling records	License termination	10 CFR 20.2103
Records of dose to individual members of the public	License termination	10 CFR 20.2107(b)
Records showing the results of surveys and calibrations	3 years	10 CFR 20.2103(a)
Records of results of surveys to determine dose from external sources and used in assessment of individual dose equivalents	License termination	10 CFR 20.2103(b)(1)
Records of the results of measurements and calculations used to determine individual intakes of radioactive material and used in the assessment of internal dose	License termination	10 CFR 20.2103(b)(2)
Records showing the results of air sampling, surveys, and bioassays	License termination	10 CFR 20.2103(b)(3)
Records of the results of measurements and calculations used to evaluate the release of radioactive effluents to the environment	License termination	10 CFR 20.2103(b)(4)
RADIATION AND CONTAMINATION CONTROL		
None.	N/A	N/A
RADIOACTIVE MATERIAL CONTROL		
Records of inventories of sealed sources and devices	5 years	NRC License Condition 22
Records of sealed source leak test results	3 years	NRC License Condition 23.H
Receipt and transfer of licensed material	3 years	10 CFR 20.2102
Records of disposal of licensed material	License termination	10 CFR 20.2108(b)

Attachment 1 – Radiological Records and Retention Periods

PERSONNEL PROTECTION AND EMERGENCY RESPONSE		
Record	Retention Period	Regulatory Basis
Records of respiratory protection activities (i.e., medical clearance, respirator fit test forms, and training documentation) and personnel contamination events	License termination, or 30 years after individual's employment termination	10 CFR 20.2102(b); 29 CFR 1910.1020(d)(1)
OTHER RECORDS		
Department of Energy radiological protection program, radiological contamination remediation, and demolition or decommissioning of radiologically contaminated facilities records	When final disposition is authorized by DOE Contracting Officer	(reference PR-MO-003)

Attachment 2 – Records of Information Important to Decommissioning

Records of information important to decommissioning include:

- Records of spills or other unusual occurrences involving the spread of contamination in and around the site, including but not limited to a description of any instances when contamination remains after any clean-up procedures or when there is reasonable likelihood that contaminants may have spread to inaccessible areas (e.g., possible seepage into porous materials such as concrete). These records shall include any known information on identification of involved nuclides, quantities, forms, and concentrations.
- As-built drawings and modification drawings of structures and equipment in restricted areas where radioactive materials are used or stored, and of locations of possible inaccessible contamination such as buried pipes that may be subject to contamination. If required drawings are referenced, each relevant document need not be indexed individually. If drawings are not available, the licensee shall substitute appropriate records of available information concerning these areas and locations.
- Except for areas containing only sealed sources (provided the sources have not leaked or no contamination remains after any leak) or any radioactive materials having only half-lives of less than 65 days, a list of the following, contained in a single document and updated every 2 years:
 - Areas designated and formerly designated as restricted areas;
 - Areas outside of restricted areas;
 - Areas outside of restricted areas where current and previous wastes have been buried, as documented under 10 CFR 20.2108; and
 - Areas outside of restricted areas that contain material such that, if the license expired, the licensee would be required either to decontaminate the area to unrestricted release levels or apply for approval for disposal under 10 CFR 20.2002.

Attachment 3 – Incident Notifications and Reports

Event	Telephone Notification	Written Report	Regulatory Agency	Regulatory Citation
Package received with removable radioactive surface contamination exceeding the limits of 10 CFR 71.87(i) or external radiation levels exceeding the limits of 10 CFR 71.47	Immediate	None	NRC	10 CFR 20.1906(d)
Theft or loss of material	Immediate	30 days	NRC	10 CFR 20.2201(a)(1)(i)
Whole body dose > 25 rem	Immediate	30 days	NRC	10 CFR 20.2202(a)(1)(i)
Extremity dose > 250 rem	Immediate	30 days	NRC	10 CFR 20.2202(a)(1)(iii)
Whole body dose > 5 rem in 24 hours	24 hours	30 days	NRC	10 CFR 20.2202(b)(1)(i)
Extremity dose > 50 rem in 24 hours	24 hours	30 days	NRC	10 CFR 20.2202(b)(1)(iii)
Whole body dose > 5 rem	None	30 days	NRC	10 CFR 20.2203(a)(2)(i)
Dose to individual member of public greater than 100 mrem	None	30 days	NRC	10 CFR 20.2203(a)(2)(iv)
Filing petition for bankruptcy under United States Code Title 11	None	Immediate	NRC	10 CFR 30.34(h)
Decision to terminate license activities or to not acquire or possess and use authorized material	None	60 days	NRC	10 CFR 30.36(d)
No principal activities conducted for 24 months	None	60 days	NRC	10 CFR 30.36(d)
Event that prevents immediate protective actions necessary to avoid exposure to radioactive materials that could exceed regulatory limits	Immediate	30 days	NRC	10 CFR 30.50(a)
	Immediate	30 days	California	17 CCR 30295(a)
An unplanned contamination event that requires access to the contaminated area to be restricted for more than 24 hours	24 hours	30 days	NRC	10 CFR 30.50(b)(1)
	24 hours	30 days	California	17 CCR 30295(b)(1)
Equipment is disabled or fails to function as designed when	24 hours	30 days	NRC	10 CFR 30.50(b)(2)

Attachment 3 – Incident Notifications and Reports

Event	Telephone Notification	Written Report	Regulatory Agency	Regulatory Citation
required to prevent radiation exposure in excess of regulatory limits	24 hours	30 days	California	17 CCR 30295(b)(2)
An event that requires unplanned medical treatment (at a medical facility) of an individual with spreadable radioactive contamination on the individual's clothing or body	24 hours	30 days	NRC	10 CFR 30.50(b)(3)
	24 hours	30 days	California	17 CCR 30295(b)(3)
Unplanned fire or explosion that affects the integrity of any licensed material or device, container, or equipment with licensed material	24 hours	30 days	NRC	10 CFR 30.50(b)(4)
	24 hours	30 days	California	17 CCR 30295(b)(4)

APPENDIX B

DATA MANAGEMENT PLAN

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Naval Facilities Engineering Command Southwest
BRAC PMO West
San Diego, CA

DRAFT

Data Management Plan

Phase IV Non-Time Critical Removal Action, Solid Waste
Disposal Area Westside, Installation Restoration Site 12
Former Naval Station Treasure Island
San Francisco, CA
June 2020

Approved for public release; distribution is unlimited



Base Realignment and Closure
Program Management Office West
San Diego, California

DRAFT

Radiation Protection Plan

Phase IV Non-Time Critical Removal Action for Solid
Waste Disposal Area Westside, Installation Restoration
Site 12

Former Naval Station Treasure Island
San Francisco, California

June 2020

DCN: GLBN-0005-F5271-0002

Prepared for:



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List of Attachments

Attachment 1 Electronic Data Deliverable (EDD) Specification for Upload into eDMS

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Acronyms and Abbreviations

ADR	automated data review
COC	chain-of-custody
DMP	Data Management Plan
EDD	electronic data deliverable
eDMS	environmental data management system
EPA	U.S. Environmental Protection Agency
Esri	Environmental Systems Research Institute, Inc.
PQCM	Project QC Manager
Gilbane	Gilbane Federal
GIS	geographic information system
LAN	local area network
PM	Program Manager
QA	quality assurance
QC	quality control
SOP	standard operating procedure
WAN	wide area network

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1.0 Introduction

This Data Management Plan (DMP) is designed to provide guidelines for managing both historical information and new environmental information gathered as part of environmental activities at various project sites. This DMP addresses requirements for data management systems, including tracking, storing, and retrieving data in project files and in the Gilbane Federal (Gilbane) environmental data management system (eDMS).

The processes described in this DMP expand on those included in project-specific documents that might include sampling and analysis plans, site-specific work plans, or Uniform Federal Policy Quality Assurance Project Plans. This DMP serves as a companion document to these project documents.

1.1 Purpose And Objectives

This document outlines the processes that will be followed to establish the site database, incorporate historical data from previous activities at various project work sites, and establish procedures for incorporating future information from ongoing activities. A systematic approach to data management will ensure that required data are collected and available for current and future reporting requirements.

The following data management objectives are critical to the success of the data management process.

- **Standardize and systematize data collection.** Use standard field forms, and provide guidance for formatting, reviewing, and transferring data collected in the field to the project files and eDMS—a centralized project database.
- **Monitor and ensure quality of the dataset.** Implement quality assurance (QA) and quality control (QC) measures to ensure the quality and accuracy of the data collected and stored in the project files and eDMS. QA/QC procedures include manual review of field forms used to collect field parameters, periodic audits of field activities, and Project Chemist review of both field parameters and laboratory data for completeness and accuracy. Electronic QA/QC measures are incorporated by subjecting electronic data deliverables (EDDs) from the contracted analytical laboratories to automated data review (ADR) against the project document QC requirements when uploaded to eDMS.
- **Provide data that are well documented.** Documentation relating to the proper collection of field parameters and analytical samples support the technical and legal defensibility of the data. This includes transport of analytical samples to the laboratory and proper analysis of the analytical samples by the laboratories. This documentation will be maintained in the project files, on eDMS, and/or on shared project-specific Web-based data portals.

- **Provide staff and client access to the data.** Access to the data from a single source for tabular report generation and geographic information system (GIS) mapping will be provided including screening against project screening criteria, statistical analyses, and waste-management activities.

1.2 Scope of Data Management Plan

This DMP outlines the processes and guidelines for tracking, storing, accessing, delivery, and reporting of analytical and spatial data generated during field investigations and other environmental data collection events at various work sites. This DMP also identifies software to be used, minimum data requirements, data formats, and backup data management. This DMP includes the following information.

- Definitions of roles and responsibilities.
- Process flow diagram for collecting, reviewing, and entering environmental data into eDMS. The process flow diagram is presented as Figure 1 in the Sample Tracking and Electronic Data Management standard operating procedure (SOP) (Gilbane, 2016).
- EDD format used by commercial laboratories to transfer analytical data electronically for incorporation into eDMS (**Attachment 1**).
- Description and use of GIS devices and data.
- Management and archiving procedures for hard-copy and electronic project documentation.

1.3 Organization

Project team members involved in the collection, analysis, and/or reporting of analytical results and field parameters all have a role in the proper execution of this DMP. The SOP flow diagram outlines the flow of sample information and laboratory results from initial sampling through reporting the validated results. Responsibilities, by role, for managing the accurate collection and reporting of the data are discussed below.

1.3.1 Project Manager

The Project Manager (PM) is responsible for:

- Establishing the goals and objectives of the sampling event and communicating them to the team;
- Providing specifics regarding the number and type of samples, analytical methods, and any special reporting requirements;
- Working with the Data Manager and Project Chemist to develop a list of required reporting elements (tables, figures, diagrams, custom queries, and reports); and
- Managing technical evaluation of the data.

1.3.2 Project QC Manager

The Project QC Manager (PQCM) or designee is responsible for:

- Reviewing the project plans;
- Planning and executing field activities, including ensuring that the field team has the equipment necessary to complete the work consistent with the requirements of the project documents and SOPs;
- Ensuring the collection of QC samples consistent with the project documents;
- Ensuring that the sampling crew has the proper type and number of sample containers for the scheduled sampling activities, as identified in the project document;
- Tracking the samples from time of collection through laboratory acceptance;
- Reviewing field forms and chain-of-custody (COC) forms for accuracy and completeness;
- Reconciling the cooler inventory against COC forms before transfer to the laboratory;
- Submitting samples to the laboratory;
- Sending COC forms, sample collection logs, and other field forms to the PM, Project Chemist, and Quality Control Manager;
- Resolving completeness issues with the laboratory and/or Project Chemist (e.g., broken bottles, missing samples, etc.); and
- Completing and submitting daily production and QC paperwork packages to the PM, Project Chemist, and Quality Control Manager.

1.3.3 Field Personnel

Field personnel are responsible for:

- Reviewing the project plans;
- Collecting environmental samples in a manner consistent with the approved project document and SOPs; and
- Completing accurate, defensible documentation of sample collection consistent with project documents and SOPs.

1.3.4 Project Chemist

The Project Chemist or designee will be responsible for:

- Reviewing the project plans;

- Establishing the planned sample table in conjunction with the project team;
- Configuring the site in eDMS, loading QC requirements from the project plans into eDMS, and establishing the sample schedule in eDMS;
- Serving as the primary point of contact for laboratories;
- Reviewing and approving COC and field information in eDMS;
- Ensuring sufficient QC samples were collected;
- Reviewing laboratory information system login reports, and updating sample tracking logs once samples are in the laboratory and for subsequent activities (e.g., data validation);
- Resolving analytical issues with laboratories;
- Reviewing and approving results/validation qualifiers; and
- Releasing data for use.

1.3.5 Data Manager

The Data Manager or designated data management staff will be responsible for:

- Establishing and maintaining the project database;
- Importing historical datasets;
- Working with the Project Chemist to configure the project setup;
- Establishing any new user accounts needed based on staffing of the project team and stakeholders;
- Working with the Project Chemist to designate sample IDs (and new location IDs, if required);
- Helping the laboratories upload EDDs;
- Running completeness tests on EDDs against COC forms to verify that all data have been received;
- Working with the PM, Project Chemist, GIS Analyst, and other assigned technical staff to develop custom queries, tabular reports, trend graphs, and other tools for accessing, reviewing, and publishing project data; and
- Exporting data to or sharing data with federal or regulatory agencies if needed.

1.3.6 GIS Analyst

The GIS Analyst will be responsible for:

- Creating and maintaining geospatial data, project maps, and figures;

- Acquiring or digitizing maps and features (buildings, roads, property boundaries, surface waters, topography), and formatting and editing the spatial database;
- Using survey data to locate and check the placement of surveyed features;
- Combining spatial data with attribute data from the project database to display features with their attributes;
- Customizing maps to include color, tabular information, and interpretive features (such as buffer zones and isoconcentration lines);
- Producing customized figures and maps according to the needs of the project, and supervising final maps submitted in deliverables;
- Working with the PM, PQCM, Project Chemist, and Data Manager to update data displayed on maps and figures; and
- Working with the Data Manager to implement QC procedures to ensure high-quality and accurate maps and figures.

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2.0 Management of Environmental Data

Data management tasks will follow a defined work flow, starting with planning activities and ending with the release of fully reviewed and validated data to team members for use in reports, maps, and other project deliverables. Data management tasks are shown on the Gilbane SOP.

2.1 Management of Field Data

Field data entered into eDMS will be managed as follows.

- **Activity Setup**—Features (e.g., monitoring wells, sample ports) identified as part of the scope of work for a specific activity that are not already in eDMS are added to eDMS by creating a new location ID file for each feature. The location ID files are created by the data management staff using available information (e.g., boring logs, well completion diagrams, and survey results) provided by the project team. These files are then reviewed against the source files and certified. A location map is then prepared in GIS using data directly from eDMS to visually confirm the identification of each feature and its location relative to previous site maps.
- **QC of Activity Setup**—The identification and location of each new feature is then reviewed by the PM or PQCM, and the other information on the location ID files is checked against the source information. If accurate, the new features are then approved and available for use.
- **Planning**—The individual features needed for each new field activity are identified, appropriate actions for each feature are selected and input into the planning module (such as groundwater sample collection with specific analyses designated), and appropriate field forms are identified and pre-populated where possible.
- **Field Event**—Field data are collected on the field forms, reviewed by the field team members, and submitted to the PQCM (for data relating to the collection of environmental samples and for other forms of data).
- **QC of Field Data**—The PQCM reviews the field forms for legibility, completeness, and technical accuracy.
- **Entry of Field Data**—Once reviewed and approved, the associated data are entered into eDMS, reviewed against the field forms to ensure accurate entry of the data, and approved by the data management team.
- **QC of Entered Field Data**—The Project Chemist reviews the data on the field forms against the entries in eDMS for completeness and accuracy, use of appropriate valid values, and proper units. If the entries accurately reflect the data provided on the field forms, the Project Chemist then approves the data.

- **Data Use**— Once approved by the Project Chemist, the data are available in eDMS for use. The data manager notifies the project team that the data are available for use in reports, figures, maps, risk assessments, modeling, and other project deliverables.

Although most field data are entered into eDMS for ready availability to the project team, some information collected in the field may not need to be entered into eDMS (e.g., waste profiles and transportation and disposal records for spent carbon). These field data that will not be entered into eDMS will be managed as follows.

- **Planning**—The required activities are reviewed and appropriate field forms are identified.
- **Field Event**—Field data are collected on the field forms, reviewed by the field team members, and forwarded to the PQCM.
- **QC Checking**—The field forms are reviewed for legibility, completeness, and technical accuracy by the PQCM.
- **Data Retention**—Electronic copies of the field forms are created and uploaded to the project server, and the hard-copy field sheets are stored in the project file.
- **Data Use**—The PQCM notifies the project team that the data are available for use in reports and other project deliverables.

2.2 Management of Chemical Analytical Data

Analytical data to be entered into eDMS will be managed as follows.

- **Activity Setup**—Features (e.g., newly installed monitoring wells) identified as part of the scope of work for a specific activity not already in eDMS, and not added into eDMS as part of the field data setup activities, are added to eDMS by creating a new location ID file for each feature. The location ID files are created by the data management staff using available information (e.g., boring logs, well completion diagrams, and survey results) provided by the project team. These files are reviewed against the source files and certified. A location map is then prepared in GIS using data directly from eDMS to visually confirm the identification of each feature and its location relative to previous site maps.
- **QC of Activity Setup**—The identification and location of each new feature is then reviewed by the PM or PQCM, and the other information on the location ID files is checked against the source information. If the information is accurate, the new features are then approved and available for use.
- **Planning**—The individual features needed for each new field activity are identified, appropriate actions for each feature are selected and input into the planning module (such as groundwater sample collection with specific analyses designated), and appropriate sample collection forms (i.e. COC forms) and sample labels are identified and pre-populated where possible.

- **Field Event**—Samples, including any required QC samples (e.g., matrix spike/matrix spike duplicate) are collected in proper containers as specified in the project document and placed in an iced cooler. Samples are immediately entered on a COC form and analyses are assigned as specified in the project document.
- **QC of Sample Information**—The PQCM reviews the COC for completeness and technical accuracy against the planned sample table from the project documents.
- **Entry and QC of Sample Information**—Once reviewed and approved, the sample information (e.g., sample ID, date and time collected, analyses requested) is entered into eDMS, and reviewed against the COC and the laboratory login receipt by the data management staff to ensure accurate entry of the sample information.
- **Laboratory Coordination**—The Project Chemist resolves any discrepancies between the COC information and the laboratory login receipt documentation with the laboratory.
- **Upload of Sample Results**—Once login issues are resolved by the Project Chemist and sample analyses are complete, laboratory results may be loaded and associated with the samples. The laboratory directly uploads the EDD into eDMS through a secure Web portal.
- **QC of Sample Results**—Upon upload, eDMS performs ADR, comparing the results against the QC criteria set up in eDMS as part of the initial activity setup. Once the laboratory data successfully pass through the ADR, the data are available for use, that is, validated to an equivalent of stage 2B electronic data review (U.S. Environmental Protection Agency [EPA], 2009) or for further validation, depending on the criteria established in the project document.
- **Data Use**—Once the sample results have successfully passed through the required data validation steps, the Project Chemist or data management staff will notify the project team that the data are available for use in reports, figures, maps, and other project deliverables.

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3.0 Reporting of Environmental Data

Once the environmental data (e.g., well stabilization parameters, water levels, analytical results) have been fully reviewed and approved, the information is available to the project team in a variety of ways.

- Preformatted summary tables and trend graphs will be developed consistent with previous report formats for ease of review by regulatory agencies and other stakeholders. These will be available as reports that are run from within eDMS. The advantage of such preformatted documents is that once the template is developed and gone through QC processes, the data in any specific report will be both accurate and consistent. Also, there is a potential for errors to be introduced if the data are imported into another software program for development of presentation tables and graphs, increasing QC requirements on the end product. These preformatted tables and graphs will be available directly via eDMS to the end user of the data, typically the technical staff developing the periodic reports, but available to all team members with appropriate permissions.
- GIS maps will be developed consistent with previous formats, again for ease of review by regulatory agencies and other stakeholders. The GIS maps are live-linked with the data in eDMS, assuring that only the latest validated and approved data are used to generate maps for the periodic reports.
- The preformatted summary tables, trend graphs, and GIS maps are available through the eDMS secure Web portal to those with appropriate permissions.

Development of unique data presentations and/or the use of specialty software (e.g., ProUCL) that requires data in specific formats will be closely coordinated with both the Data Manager and Project Chemist to ensure that the latest data are used and that the correct fields are pulled from the database (this is especially important relative to detection/reporting limits). Use of data outside eDMS carries a requirement of greater QC to ensure transfer of the correct data and units, and preservation of appropriate significant figures.

The historical dataset will be georeferenced in the GIS system, and coordinates referenced in the project-required coordinate system.

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4.0 Structure of the Data Management System

The data management system for the various project work sites is designed to provide routine and secure access to data while being flexible to accommodate periodic customized needs. The data management system for the works sites will consist of the following:

- A database in Gilbane's enterprise-wide eDMS that can store many types of data;
- Importing and exporting capabilities that allow any format required;
- Project-specific reporting capabilities that meet reporting and document style requirements;
- Enterprise-wide GIS capabilities with live links to eDMS;
- Remote access to project work site data through a secure Web-based portal; and
- A hard-copy filing system that will allow easy retrieval of project records and provide secure storage.

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5.0 Data Administration and Security

5.1 Database Administration

The Data Manager will oversee the administration of eDMS and will implement and maintain the project work site database and electronic data management processes. Database maintenance will consist of the following:

- Adding, altering, and deleting users, roles, and privileges;
- Providing routine backup of eDMS to online storage and tape backup;
- Maintaining an approved list of valid values for data consistency; and
- Assisting with custom queries for unique data needs.

Valid values are critical to relational databases. Inconsistencies in naming conventions, analyte or method spelling differences, and the use of non-standard abbreviations can result in misclassified data and potentially incorrect conclusions. Most tables and forms in eDMS use look-up tables for acceptable valid values and will not allow the entry of data that do not conform. A current list of valid values is available from the Data Manager on request.

Data from third-party sources may use alternative valid values and require conversion prior to use in eDMS. In this case, the original data are stored in eDMS, and the conversion occurs in interim tables, converting the stored information into the appropriate valid values consistent with the information stored in eDMS. This provides the ability to audit third-party information back to the raw source.

5.2 Data Management and Archiving Procedures

The PQCM, Project Chemist, and Data Manager will collect hard-copy and electronic data and verify that the incoming records are legible and are in suitable condition for storage. Project-related information generated during implementation of the scope of work will be managed and stored within Gilbane's offices. Record storage will be performed in two stages:

- Storage during the project; and
- Permanent storage of project records.

Physical records, including copies of field documents (e.g., completed traffic reports, COCs, field forms, site photographs), written communications (e.g., letters, telephone conversation logs), and other project-related documents will be secured in steel file cabinets labeled with the project identification.

Electronic data consists of plans, reports, and other documents created in Microsoft Office and other office productivity software packages, and electronic versions or scans

of physical data saved in adobe acrobat format. These files are stored on Gilbane servers located in a Class 4 data center in Culpepper Virginia (for older projects), or in Bluebeam Studio, a cloud-based storage system hosted on Amazon Elastic Compute Cloud (EC2) and accessed through Bluebeam Revu desktop application (for newer projects). Electronic data also consists of chemical data, provided as electronic data deliverables (EDDs), and other field-generated raw electronic data. Chemical data are stored in eDMS.

5.3 Data Security Procedures

Site data will be stored in secure locations. Physical data is stored in steel file cabinets located within Gilbane's offices. Each office has key card access control during the day, and key card and passcode access control for after hours. Access is logged by each individual's key card, and video surveillance covers the entrance and work areas.

Access to Gilbane's servers is controlled by domain security for both access to Gilbane's local area network (LAN), and for access via the LAN to the wide-area network (WAN) and the data center. For remote users, access to the servers located on Gilbane's WAN is via a secure virtual private network (VPN). Access to Bluebeam Studio is controlled through the Bluebeam Revu application, with each user entering their unique username and password to initiate connectivity with the Bluebeam Studio cloud. The user password is transmitted in an encrypted form, and communications between Revu and the Studio cloud is via encrypted HTTPS protocol. Electronic access to eDMS is controlled by both domain-level security for overall access, and by application-level granular security within the Microsoft SQL database based on individual user permissions established on the basis of the individual's assigned role. Only users with explicit permission (typically the Data Manager only) can alter records in eDMS, and all changes to stored data are logged regarding what changed and who made the changes.

5.4 Data Backup and Recovery

Electronic data files, whether stored on Gilbane's servers (such as eDMS) or on Bluebeam Studio, are backed up to online storage nightly.

6.0 References

EPA, 2009. *Guidance for Labeling Externally Validated Laboratory Analytical Data for Superfund Use*. EPA 540-R-08-005. January.

Gilbane Federal, 2016. *Standard Operating Procedure: Sample Tracking and Electronic Data Management, PR-TC-02.12.02.00 v 2.2*. October.

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ATTACHMENT 1

ELECTRONIC DATA DELIVERABLE (EDD) SPECIFICATION FOR UPLOAD INTO eDMS

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FieldName	VVL?	Required?	Column Width	Start Position	End Position	Description
AFIID	True	Yes	5	1	5	Facility identification is the unique code used to represent an installation, plant, or base.
LOCID		Conditional	15	7	21	Location Identification is the unique identifier assigned to a location within a USAF installation where measurements or samples are taken.
LOGDATE		Conditional	11	23	33	The date/time that the sample was taken. The format for LOGDATE field entry is [DD-MMM-YYYY] where YYYY is the calendar year, MMM is the abbreviated month and DD is the numeric date.
LOGTIME		Conditional	4	35	38	Log Time is the time of the day (24 hour clock) that a sample is collected, a field measurement is made, or a quality control sample is created. This value is expressed in the HHMM format of the Local Time.
MATRIX	True	Yes	2	40	41	Sampling Matrix is a coded value identifying the specific sample medium actually being analyzed. I.e., soil, water, drill cuttings, waste water, etc.
SBD		No	8	43	50	Sample Beginning Depth is the upper depth in feet from the ground surface or the water surface at which a sample is collected or recovered.
SED		No	8	52	59	Sample Ending Depth is the lower depth in feet from the ground surface or the water surface at which a
SACODE	True	Yes	2	61	62	Sample Code is a coded value identifying whether the sample is a QC or normal and the type is QC.
SAMPNO		Yes	2	64	65	Sample Number is the numerical identifier for the sample taken.
LOGCODE	True	No	4	67	70	Logging Company Code is the coded value identifying the company performing the field tests.
SMCODE	True	No	2	72	73	Sample Method Code is a coded value identifying the sampling method used to collect a sample.
FLDSAMPID		Yes	30	75	104	Field Sample Identification is a unique number assigned to the sample in the field. This number will be a reference to the specific sample regardless of the sample date or location.
COCID		No	12	106	117	Chain of Custody Identification is a unique identification reference to the chain of custody describing the transport of the sample to the laboratory.
COOLER		No	2	119	120	Cooler Number is the unique number assigned to the cooler transporting the sample.
ABLOT		No	8	122	129	Ambient Blank Field Lot Identifier is used in ERPTools to relate the lot of normal samples (collected in the field) to the related ambient blank. There will only be an entry for normal samples that are associated to an ambient blank. This field in the sample record for the ambient blank itself will be left blank. Entries in this field will be formatted as date and SAMPNO (DDMMYYNN). The format for the ABLOT, EBLOT, and TBLOT field entries is [DDMMYYNN] where DD is the numeric date of the month, MM is the number for the month, YY is the last two digits of the calendar year, and NN is the sequentially assigned number for the lot.

FieldName	VVL?	Required?	Column Width	Start Position	End Position	Description
EBLOT		No	8	131	138	Equipment Blank Field Lot Identifier is used, in ERPTools, to relate the lot of normal samples (collected in the field) to the related equipment blank(s). There will only be an entry for normal samples that are associated to an equipment blank. This field will be left blank in the sample record for the equipment blanks themselves. Entries in this field will be formatted as date and SAMPNO (DDMMYYNN). The format for the ABLOT, EBLOT, and TBLLOT field entries is [DDMMYYNN] where DD is the numeric date of the month, MM is the number for the month, YY is the last two digits of the calendar year, and NN is the sequentially assigned number for the lot.
TBLLOT		No	8	140	147	Test Blank Field Lot Identifier is used in ERPTools to relate the lot of normal samples (collected in the field) to the related trip blank(s). There will only be an entry for normal samples that are associated to a trip blank. This field will be left blank in the sample record for the equipment blanks themselves. Entries in this field will be formatted as date and SAMPNO (DDMMYYNN). The format for the ABLOT, EBLOT, and TBLLOT field entries is [DDMMYYNN] where DD is the numeric date of the month, MM is the number for the month, YY is the last two digits of the calendar year, and NN is the sequentially assigned number for the lot.
REMARKS		No	240	149	388	Contains comments about the sample.
SDG		No	20	390	409	A lab created code used to identify a group or selection of samples. The SDG is used for processing and reporting accuracy by labs. This value is included in a prime project file for integrity references.
LABCODE	True	Yes	4	411	414	Analytical Laboratory Code is a coded value identifying the laboratory which performed the analysis of the samples.
ANMCODE	True	Yes	7	416	422	Analytical method code is a coded value representing the method of analyses of a given parameter.
EXMCODE	True	Yes	7	424	430	Extraction Method Code is a coded value representing the method used to extract or prepare a sample.
LCHMETH	True	Yes	7	432	438	Leachate Method is a coded value identifying the leachate method used in the test. In ERPTools, leachate method has been added as a key field. In addition, the field widths for analytical, extraction, and leachate methods have been expanded to 7 (allowing for the indication of method version and (or) method revisions). For all versions of the data-loading handbook there are basically four categories of extraction method codes that determine which extraction method code should be used, (or leachate method if applicable).
RUN_NUMBER		Yes	2	440	441	This information is stored in the test procedure class and is replaced by the use of test sequence.
LABSAMPID		Yes	20	443	462	Lab Sample Identification is a unique number assigned to a sample by a laboratory and included in the reporting of the results. This number is the prime number that the Lab will use to reference a specific sample for tests and results.
EXTDATE		Conditional	11	464	474	Extraction Date is the data that represents the start of an extraction test or other preparation methods. This is used for compatibility with DESCIM. The format for EXTDATE field entry is [DD-MMM-YYYY] where YYYY is the calendar year, MM is the numeric month and DD is the numeric date.
EXTTIME		Conditional	4	476	479	Extraction Time is the time of day (24 hour clock) that represents the start of an extraction test or other preparation methods. This value is expressed in HHMM of the local time. This is used for compatibility with DESCIM.
LCHDATE		Conditional	11	481	491	Leachate Date is the date which a sample was leachated. The format for LCHDATE field entry is [DD-MMM-YYYY] where YYYY is the calendar year, MM is the numeric month and DD is the numeric date.

FieldName	VVL?	Required?	Column Width	Start Position	End Position	Description
LCHTIME		Conditional	4	493	496	Leachate Time is the time of day (24 hour clock) that represents the time a sample was leached. This value is expressed in HHMM of the local time.
LCHLOT		Conditional	10	498	507	Leachate Lot is the batch designator of an autonomous group of environmental samples and associated quality control samples leached together.
ANADATE		Yes	11	509	519	Analyze Date is a date that represents the start of a test or procedure. The Date represents the date the sample or extraction is analyzed in the laboratory. The format for ANADATE field entry is [DD-MMM-YYYY] where YYYY is the calendar year, MMM is the abbreviated month and DD is the numeric date.
ANATIME		Yes	4	521	524	Analyze Time is the time of day (24 hour clock) that represents the start of a test or procedure. This value is expressed in HHMM of the local time. TEST: The Date represents the date the sample or extraction is analyzed in the laboratory.
ANALOT		Yes	10	526	535	Analyze Lot is the batch designator of an autonomous group of environmental samples and associated quality control samples analyzed together.
LABLOTCTL		Yes	10	537	546	Lab Lot Control is a more general identifier to indicate extractions or other preparation methods during the testing process. For compatibility with DEDCIM.
CALREFID		No	10	548	557	Calibration Reference Identification is a coded value which establishes a reference link between environmental and quality control samples and their corresponding calibration records. Note: This field is a Legacy requirement that has not been removed from the database structure for integrity reasons. It should only be included if an CALREFID already exists for the test being performed.
RTTYPE	True	No	5	559	563	Remediation Technology Type is a coded value describing the type of remediation technology being used. This value is the coded value for remediation technology like slurry wall, in situ vitrification, bio-reactor, etc.
BASIS	True	Yes	1	565	565	Basis is a coded value detailing whether tissue or solid sample results are reported on a wet (W) or dry (D) basis.
PARLABEL	True	Yes	12	567	578	Parameter Label Code is an abbreviated, common acronym representing a parameter/analyte.
PRCCODE	True	Yes	3	580	582	Parameter Class Code is a coded value identifying a class or group that a parameter is associated with. I. e., ORG, MET, STD, etc.
PARVQ	True	Yes	2	584	585	Parameter Value Qualifier is a coded value qualifying the analytical results field (PARVAL). The following list details the most commonly used Parameter Value Qualifier (PARVQ) codes. Note that in general, the PARVQ does not indicate QC failures or deficiencies such as accuracy, precision, blank contamination, or holding time violations. The field PRIME_FLAG, exists for the purpose of adding additional CLP data qualifiers as needed.
PARVAL		Yes	17	587	603	Parval is the value of a calculated parameter reported in units consistent with UNITMEAS.
PARUN		Conditional	13	605	617	Par Uncertainty is a value which measures the uncertainty of the measurement. This value is expressed as positive (+) or negative (-) some value.
PRECISION_		Yes	1	619	619	Precision is number indicating the number of digits after the decimal point of the results.
EXPECTED		Conditional	17	621	637	Expected Result is a number indicating the target result for a quality control sample or surrogate spike.
EVPREC		Conditional	1	639	639	Expected Value Precision is a number indicating the number of digits after the decimal point in the results of a test.

FieldName	VVL?	Required?	Column Width	Start Position	End Position	Description
MDL		Yes	17	641	657	Method detection limit is the smallest quantity of an analyte that can be detected from a prepared sample.
RL		Yes	17	659	675	AFCEE Reporting Limit is a number which is the smallest quantity of an analyte that should be reported in accordance with the AFCEE QAPP.
UNITS	True	Yes	10	677	686	Units of Measure refers to type of unit used for measuring a specific value during the ERPIMs process. In each of the tables Units refers to a specific field in each record. The UNITS field refers to the units used for the parameter value.
VQ_1C	True	Conditional	2	688	689	1C Value Qualifier is a coded value qualifying the analytical results field (VAL_1C).
VAL_1C		Conditional	17	691	707	First Column Parameter Value is a number field which represents the primary or initial value for a analyte generated from a Gas Chromatography or Gas Chromatography/Mass Spectroscopy results.
FCVALPREC		Conditional	1	709	709	First Column Value Precision is a number indicating the number of digits after the decimal point of the results of a test.
VQ_CONFIRM	True	Conditional	2	711	712	Value Qualifying Confirmation is a coded value qualifying the confirming analytical result.
VAL_CONFIRM		Conditional	17	714	730	Confirming Value is a number value of a chromatographic analytical result that requires second column confirmation.
CNFVALPREC		Conditional	1	732	732	Confirm Value Precision is a number indicating the number of digits after the decimal point of the results of a test.
DILUTION		Yes	17	734	750	Dilution Required is a numeric expression of the amount of dilution required to bring the analyte concentration in the sample into analysis range.
PRIME_DQT	True	No	5	752	756	Prime Data Qualifier Type is a coded value identifying the type of data qualifier that the prime used
PRIME_FLAG	True	No	6	758	763	Prime Flags are codes that are assigned during chemistry data validation.
LAB_DQT	True	No	5	765	769	Laboratory Data Qualifier Type is a coded value indicating the specific QAPP or DQO document from which the entered performance criteria data originates.
LAB_QC_FLAG	True	Conditional	6	771	776	Laboratory Quality Control Flag is coded values entered by the laboratory to indicate the existence of a specific quality control exception or condition.
BEST_RESULT		Yes	1	778	778	Best Result is a single value that has been determined to be the best result. I.e., the value reported in the prime contractor's final report for the sampling event in focus. Appropriate Values are Y (Yes) or N (No)
REASON_CODE		No	30	780	809	Reason Code is a coded value that indicates why a laboratory or contractor flag was issued to a data point.
PERCENT_RECOVERY		Conditional	15	811	825	Percent Recovery is the calculated recovery value for the spiked or surrogate analyte. This value is expressed in percent plus 2 decimals.
RPD		Conditional	15	827	841	Relative Percent Difference is a measure of variability that adjust for the magnitude of observations. This value is used to assess total and analytical precision of duplicate measurements.
UPPER_RPD		Conditional	15	843	857	Upper Relative Percent Difference is a number representing the upper performance limit for relative percent difference.
UPPER_ACCURACY		Conditional	15	859	873	Accuracy Upper Limit is a number representing the upper control limit of percent recovery as measured for a known target analyte spiked into a quality control sample.
LOWER_ACCURACY		Conditional	15	875	889	Accuracy Lower Limit is a number representing the lower control limit of percent recovery as measured for a known target analyte spiked into a quality control sample.

FieldName	VVL?	Required?	Column Width	Start Position	End Position	Description
SPIKE_ADDED		Conditional	17	891	907	Spike Amount Added is a number value of a final concentration of an analyte spiked into a sample.
SPIKE_ADDED_PREC		Optional	1	909	909	Spike Amount Added Precision is number indicating the number of digits after the decimal point of the spike added.
VALCODE	True	No	4	911	914	Coded value identifying the company validating analytical results.
TIC_NAME		No	54	916	969	Name of the Tentatively Identified Compound being reported.
RETENTION_TIME		No	6	971	976	Retention time of a Tentatively Identified Compound.
LOD	False	No	17	978	994	Limit of Detection

Valid values will be specified by the project team, and are generally based on the most current valid values released for ERPIMS. Valid values can be found on the project portal; navigate to Tools/Query Tool and select the field name of interest from the list of Saved Queries.

For non-ERPIMS projects, radiochemistry PARVALs should be reported as the detection (positive or negative) as reported by the instrument.

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APPENDIX C

SAMPLING AND ANALYSIS PLAN

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SAP Worksheet #1 – Title and Approval Page

DRAFT
Sampling and Analysis Plan
(Field Sampling Plan and Quality Assurance Project Plan)
Naval Station Treasure Island Phase IV Non-Time Critical Removal Action
(NTCRA) Solid Waste Disposal Area (SWDA) Westside Site 12
May 2020

Prepared for:

Base Realignment and Closure Program Management Office - West
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Prepared by:

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Document Control Number: GLBN-0005-5271-0002

Prepared under:

Contract Number N62473-17-D-0005
Contract Task Order CTO N6247318F5271

Reviewed by:



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Executive Summary

Introduction

This Sampling and Analysis Plan (SAP) provides guidance to Gilbane Federal (Gilbane) and its project subcontractors in the performance of sampling and analysis during Phase IV of the Non-Time Critical Removal Action (NTCRA) activities to be performed in the non-Solid Waste Disposal Areas (SWDA) Westside within Installation Restoration (IR) Site 12 at the former Naval Station Treasure Island (NSTI) in San Francisco, California (Figure 1). This SAP was prepared by Gilbane, as requested by the United States Department of the Navy (Navy) under the Radiological Multiple Award Contract (RADMAC II) Number N62473-17-D-0005, Contract Task Order (CTO) N6247318F5271, based on the Performance Work Statement (PWS) received from the Navy on August 9, 2018.

This Phase IV project is considered an extension of the NTCRA Phase III and focuses on the removal of soil stained with total petroleum hydrocarbon (TPH) and co-mingled with radioactive contamination and buried debris. The primary objective of Phase IV is to excavate soil with chemical and radioactive contaminants and replace it with clean fill pursuant to the *Action Memorandum/Interim Remedial Action Plan: Non-Time Critical Removal Action for Solid Waste Disposal Areas Installation Restoration Site 12 Old Bunker Area Naval Station Treasure Island San Francisco, California* (Action Memo [Navy, 2007]), Alternative 3. Alternative 3 calls for excavating soil from the common areas, roadways, and backyards to a depth of 4 feet (ft) below ground surface (bgs) and replacing it with clean fill. However, the excavation will be extended laterally if buried debris and/or TPH contamination is visually present, and vertically to a maximum depth of 15 ft bgs. Radiological and chemical data will be collected for characterization purposes to document the as-left surfaces.

Secondary objectives include performing munitions screening and disposing of munitions-impacted soil (see Figure 2) from previously excavated Phase III soil stockpiles within SWDA Westside; and down-posting the radiologically controlled area (RCA) at SWDA Westside. This SAP will provide guidance for the sampling associated with the characterization sampling and import material characterization. Radiological screening, munitions screening, and waste characterization will be described in the Work Plan (Gilbane, 2018).

The remedial action objective for the NTCRA per the Action Memo (Navy, 2007) is as follows:

- Reduce the potential for human contact with chemically-contaminated soil near the ground surface within the four SWDAs at Site 12 under the current land and utility configuration.

Because the initial stages of the NTCRA have shown chemically-contaminated soil to be co-located with debris (KCH, 2015), and to achieve the secondary goal of TPH source removal, excavation will continue until no debris and/or TPH stained soil is visually observed to a maximum depth of 15 ft bgs. When excavation is completed, bottom and sidewall characterization samples for Radium-226 (Ra-226), lead (Pb), polycyclic aromatic hydrocarbons (PAHs), polychlorinated biphenyls (PCBs), dioxins/furans, and volatile organic compounds (VOCs) will be collected for the purposes of documenting left-in-place conditions.

This SAP (Appendix A of the Work Plan [Gilbane, 2018]) addresses critical requirements such as project organization and responsibilities, data quality objectives (DQOs), sampling design, field and analytical procedures, quality control (QC), and quality assurance (QA) for sampling at the former NSTI. The QA/QC elements in this SAP were prepared in accordance with the U.S. Environmental Protection Agency (EPA) *Uniform Federal Policy for Quality Assurance Project Plans* (UFP-QAPP; EPA, 2005) and *Requirements for Quality Assurance Project Plans, EPA QA/R-5* (EPA, 2001) to ensure that all data collected are precise, accurate, representative, complete, and comparable to meet their intended use.

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Acronyms and Abbreviations

°C	degrees Celsius
°F	degrees Fahrenheit
%R	percent recovery
µg/kg	micrograms per kilogram
ACM	asbestos-containing material
amu	atomic mass unit
BaP	benzo(a)pyrene
BAP(eq)	benzo(a)pyrene equivalency
bgs	below ground surface
BCT	Base Closure Team
BCY	bank cubic yard
BRAC	Base Realignment and Closure
BSC	Background Subtraction Count
CA	corrective action
CARB	California Air Resources Board
CAS	Chemical Abstracts Service
CCB	continuing calibration blank
CCSF	City and County of San Francisco
CCV	continuing calibration verification
CDPH	California Department of Public Health
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act of 1980
CFR	Code of Federal Regulations
COC	chain of custody; chemical of concern
CSO	Caretaker Site Office
CTO	Contract Task Order
CVAA	cold vapor atomic absorption
D	difference
DER	Duplicate Error Ratio
DL	detection limit

DoD	Department of Defense
DOE	Department of Energy
DOT	Department of Transportation
dpm	disintegrations per minute
DQI	data quality indicator
DQO	data quality objective
DTSC	California Department of Toxic Substances Control
EDD	electronic data deliverable
EDL	estimated detection limit
ELAP	Environmental Laboratory Accreditation Program
EPA	United States Environmental Protection Agency Region 9
EPM	Environmental Program Manager
ESS	Explosives Safety Submission
EWI	Environmental Work Instruction
FCR	Field Change Request
FWHM	Full width at Half Maximum
g	gram(s)
GC	gas chromatograph
Gilbane	Gilbane Federal
GPS	Global Positioning System
HASL	Health and Safety Laboratory
HERO	Human and Ecology Risk Office
HHRA	Human Health Risk Assessment
HpCDD	heptachlorodibenzo-p-dioxin
HpCDF	heptachlorodibenzofuran
HRGC	high resolution gas chromatography
HRMS	high resolution mass spectrometry
HxCDD	hexachlorodibenzo-p-dioxin
HxCDF	hexachlorodibenzofuran
ICAL	initial calibration
ICB	initial calibration blank
ICC	Instrument Contamination Check

ICP	inductively coupled plasma spectrophotometer
ICP/MS	inductively coupled plasma/mass spectrometer
ICS	interference check solution
ICV	initial calibration verification
IR	Installation Restoration
IS	internal standard
IUPAC	International Union of Pure and Applied Chemistry
keV	kilo-electron volt(s)
L	liter
LCS	laboratory control sample
LCSD	laboratory control sample duplicate
LDR	Linear Dynamic Range
LOD	limit of detection
LOQ	limit of quantitation
LRPM	Lead Remedial Project Manager
m	meter(s)
MARLAP	Multi-Agency Radiological Laboratory Analytical Protocols
MB	method blank
MDC	minimum detectable concentration
MDL	method detection limit
mg/kg	milligrams per kilogram
mL	milliliters
MRS 1	Munitions Response Site 1
MS	mass spectrometer
MS/MSD	matrix spike/matrix spike duplicate
NA	not applicable
NAVFAC SW	Naval Facilities Engineering Command Southwest
Navy	United States Department of the Navy
NCR	Nonconformance Report
NEDD	Navy Electronic Data Deliverable
NIRIS	Naval Installation Restoration Information Solution
NL	not listed

NSTI	Naval Station Treasure Island
NTCRA	Non-time critical removal action
OCDD	octachlorodibenzodioxin
OCDF	octachlorodibenzofuran
OSHA	Occupational Safety and Health Organization
oz.	ounce(s)
Pace National	Pace Analytical National Center for Testing and Innovation
PAH	polycyclic aromatic hydrocarbon
PARCCS	precision, accuracy, representativeness, completeness, comparability, and sensitivity
PeCDD	pentachlorodibenzo-p-dioxin
PeCDF	pentachlorodibenzofuran
PEF	potency equivalence factors
Pb	lead
PCB	polychlorinated biphenyl
pCi/g	picocuries per gram
PLM	polarized light microscopy
PM	Project Manager
PMO-W	Program Management Office - West
PPE	personal protective equipment
PQCM	Project Quality Control Manager
PSL	project screening limit
PT	performance testing
PWS	performance work statement
QA	quality assurance
QAO	Quality Assurance Officer
QC	quality control
QCPM	Quality Control Program Manager
QCSR	Quality Control Summary Report
QL	quantitation limit
QSM	Quality Systems Manual
Ra-226	Radium-226

RAB	Restoration Advisory Board
RADMAC	Radiological Multiple Award Contract
RASO	Radiological Affairs Support Office
RCA	radiologically controlled area
RER	relative error ratio
ROICC	Resident Officer in Charge of Construction
RPD	relative percent difference
RPM	Remedial Project Manager
RSD	relative standard deviation
RSL	regional screening level
RSO	Radiation Safety Officer
RT	retention time
RO	radioactive object
SAP	Sampling and Analysis Plan
SDG	sample delivery group
Shaw	Shaw Environmental, Inc.
SOP	standard operating procedure
SSHO	Site Safety and Health Officer
SVOC	semivolatile organic compounds
SWDA	Solid Waste Disposal Area
TBD	to be determined
TCDD	tetrachlorodibenzo-p-dioxin
TCDF	tetrachlorodibenzofuran
TEF	toxicity equivalency factors
TEQ	toxicity equivalent
TI	Treasure Island
TICD	Treasure Island Community Development
TIDA	Treasure Island Development Authority
TPH	total petroleum hydrocarbons
TPH-e	total petroleum hydrocarbons - extractable
TPH-p	total petroleum hydrocarbons – purgeable
TSA	technical systems audit

UFP-QAPP	Uniform Federal Policy for Quality Assurance Project Plans
UXO	unexploded ordnance
Validata	Validata Chemical Services, Inc.
VOA	volatile organic analysis
VOC	volatile organic compounds
Water Board	San Francisco Bay Regional Water Quality Control Board

SAP Worksheet #2 – SAP Identifying Information

Site Name/Number:	Former Naval Station Treasure Island
Operable Unit:	Installation Restoration Site 12
Contractor Name:	Gilbane Federal
Contract Number:	N62473-17-D-0005
Contract Title:	RAD MAC II
Work Assignment Number (optional):	CTO N6247318F5271

1. This SAP was prepared in accordance with the requirements of the *Uniform Federal Policy for Quality Assurance Project Plans* (UFP-QAPP; EPA, 2005) and *EPA Guidance for Quality Assurance Project Plans, EPA QA/G-5* (EPA, 2002). Additional guidance taken from the following sources:

- *Quality Systems Manual for Environmental Laboratories, Version 5.1* (QSM; U.S. Department of Defense [DoD]/Department of Energy[DOE], 2017)
- *Quality Systems Manual for Environmental Laboratories, Version 5.1.1* (QSM; DoD/DOE, 2018)
- *Guidance on Systematic Planning Using the Data Quality Objectives Process* (EPA, 2006)
- *Environmental Work Instruction (EWI) #2, Review, Approval, Revision, and Amendment of Sampling and Analysis Plans EV3.2* (NAVFACSW, 2011)

2. Identify regulatory program: Comprehensive Environmental Response, Compensation, and Liability Act of 1980 (CERCLA)

3. This SAP is a: Project-Specific SAP.

4. List dates of scoping sessions that were held:

Scoping/kickoff meeting

10/16/2018

5. List dates and titles of any SAP documents written for previous site work that are relevant to the current investigation.

Title	Date
<u><i>Final Sampling and Analysis Plan Site 12 Final Remedy Removal Action, Treasure Island, San Francisco, California</i> (Shaw Environmental, Inc. [Shaw], 2007)</u>	<u>2/26/2007</u>
<u><i>Final Sampling and Analysis Plan Non-Time Critical Removal Action Solid Waste Disposal Areas, Westside, Bayside, and North Point, Former Naval Station Treasure Island, San Francisco</i> (CB&I, 2015)</u>	<u>6/9/2015</u>

6. List organizational partners (stakeholders) and connection with lead organization: Oversight by the California Department of Toxic Substances Control (DTSC); California Department of Public Health (CDPH), Division of Drinking Water and Environmental Management; the California Regional Water Quality Control Board-San Francisco Bay Region (Water Board); and the United States Environmental Protection Agency (EPA), and the Treasure Island Development Authority (TIDA)

7. Lead organization:

U.S. Department of the Navy (Navy)

8. If any required SAP elements or required information are not applicable to the project or are provided elsewhere, then note the omitted SAP elements and provide an explanation for their exclusion below: No SAP elements are omitted or provided elsewhere; therefore, no crosswalk is provided.

SAP Worksheet #3 – Distribution List

Name of SAP Recipient	Title/Role	Organization	Telephone Number	E-mail Address or Mailing Address
David Clark	Navy Lead Remedial Project Manager (LRPM)	Base Realignment and Closure (BRAC) PMO West	619-524-6870	david.j.clark2@navy.mil
Brandon Mills	Navy Remedial Project Manager (RPM)	BRAC PMO West	619-524-5887	brandon.s.mills1@navy.mil
Teresie Walker	Navy Quality Assurance Officer (QAO)	NAVFAC LANT	757-322-4699	teresie.walker@navy.mil
Kimberly Noble	Navy Environmental Program Manager (EPM)	Navy Radiological Affairs Support Office (RASO)	714-606-7226	Kimberly.k.noble1@navy.mil
Izzat Amadea	Navy Resident Officer in Charge of Construction (ROICC)	Navy San Francisco Bay Area	510-749-5947	izzat.amadea@navy.mil
Glenwood "Thomas" Ivey	Point of Contact	Navy Caretaker Site Office (CSO)	415-743-4729	glenwood.ivey@navy.mil
Sheetal Singh	Regulatory Agency Representative	CDPH	916-449-5691	sheetal.singh@cdph.ca.gov
Nina Bacey	Regulatory Agency Representative	DTSC	510-540-2480	juanita.bacey@dtsc.ca.gov
Nadia Hollan Burke	Regulatory Agency Representative	EPA	415-972-3187	burke.nadiahollan@epa.gov
Myriam Zech	Regulatory Agency Representative	Water Board	510-622-5684	mzech@waterboards.ca.gov
Robert P. Beck	Treasure Island Director	TIDA	415-274-0662	bob.beck@sfgov.org
John Baur	Project Manager (PM)	Gilbane	925-946-3212	jbaur@gilbaneco.com
Laura Tryboski	Quality Control Program Manager (QCPM)	Gilbane	925-946-3192	ltryboski@gilbaneco.com
Christopher Bryson	Site Radiation Safety Officer (RSO)	Envirachem	925-784-7719	chris.bryson@envirachem.com
Chuck Clyde	Project Quality Control Manager (PQCM)	Gilbane	925-383-7604	cclyde@gilbaneco.com

Name of SAP Recipient	Title/Role	Organization	Telephone Number	E-mail Address or Mailing Address
David Clark	Navy Lead Remedial Project Manager (LRPM)	Base Realignment and Closure (BRAC) PMO West	619-524-6870	david.j.clark2@navy.mil
Kristen Carlyon Peyton	Program/Project Chemist	Gilbane	925-946-3180	kcarlyon@gilbaneco.com
Tony Olmstead	Site Superintendent	Gilbane	925-946-3365	tolmstead@gilbaneco.com
Teresa Ruha	Site Health & Safety	Gilbane	925-946-3177	truha@gilbaneco.com

SAP Worksheet #4 – Project Personnel Sign-Off Sheet

Name	Organization/Title/Role	Telephone Number (optional)	Signature/email receipt	SAP Section Reviewed	Date SAP Read
John Baur	Gilbane/PM	925-946-3212		All	
Chris Bryson	Envirachem/Site RSO	925-784-7719		All	
Tony Olmstead	Gilbane/Site Superintendent	925-946-3365		All	
Chuck Clyde	Gilbane/PQCM	925-383-7604		All	
Jennifer Gambill ¹	Pace Analytical National Center for Testing and Innovation (Pace National) – Mt. Juliet and Minnesota /Laboratory PM	615-773-9670		All	
Shantall Carpenter	A&B Environmental Services, Inc. (A&B Labs) / Laboratory PM	713-453-6060		All	
Kevin Harmon	Validata Chemical Services, Inc. (Validata)/Data Validator (3 rd Party)	770-232-0130		All	
TBD ^{2,3}	Gilbane/Field Crew	Various		All	
TBD ^{2,3}	Gilbane/Field Crew	Various		All	
TBD ^{2,3}	Gilbane/Field Crew	Various		All	

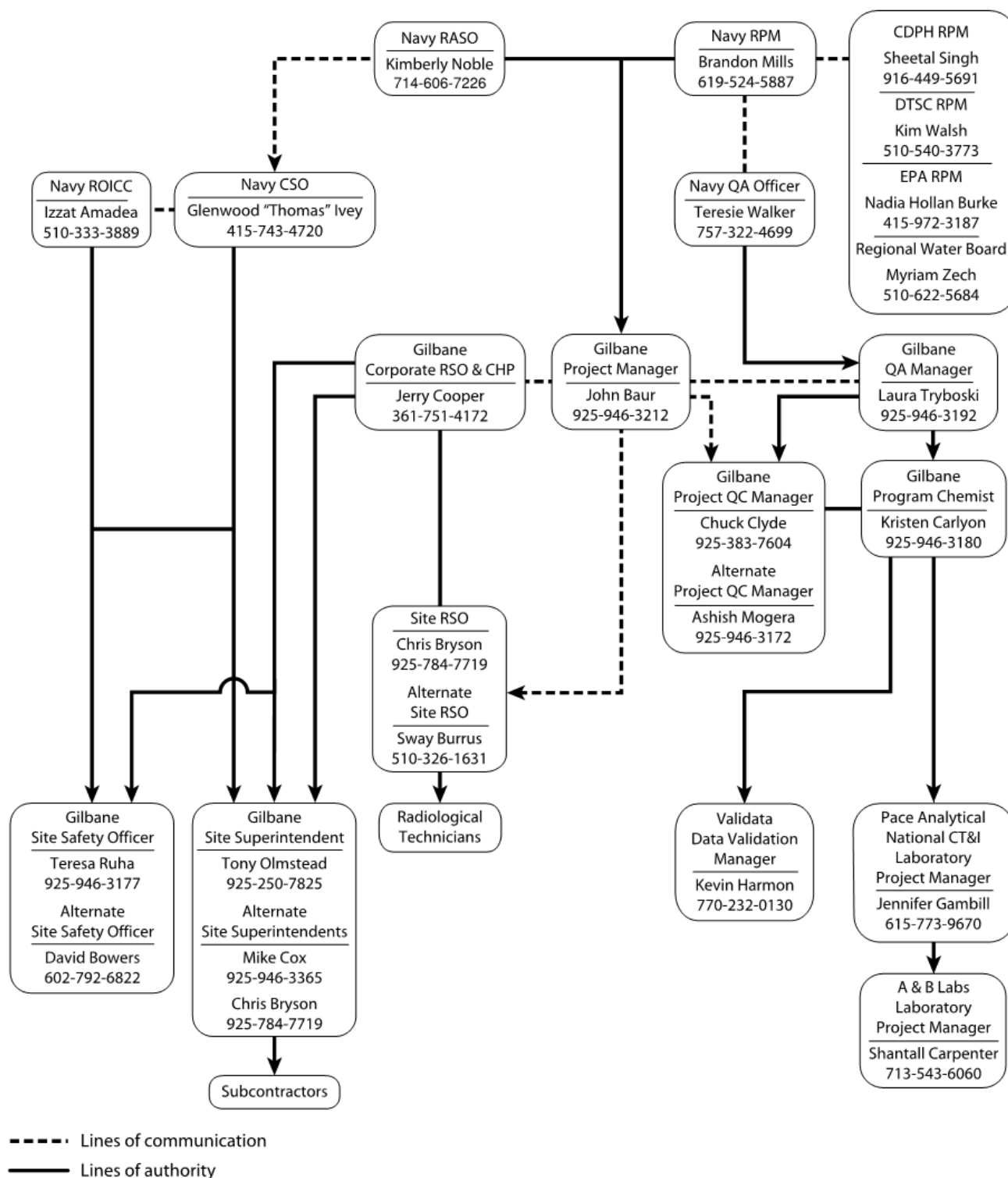
Note:

¹Subcontracted Laboratory PMs will be provided SAP and will sign off; however, primary communication will be through the Pace National Laboratory PM.

²To be determined. The indicated team member has yet to be selected. These fields will be completed before submission of the Final SAP.

³ Field crew members will be selected at task startup. Persons identified by the PQCM will read the SAP and sign this worksheet as required. Their identities and the number of required personnel have not been determined at the time of publication.

SAP Worksheet #5 – Project Organizational Chart



SAP Worksheet #6 – Communication Pathways

Communication Drivers	Responsible Affiliation	Name	Phone Number and/or e-mail	Procedure
Approvals	Navy QAO Navy RPM RASO	Teresie Walker Brandon Mills Kimberly Noble	teresie.walker@navy.mil brandon.a.mills1@navy.mil Kimberly.k.noble1@navy.mil	Gilbane PM to request approval of SAP from QAO and approval of Work Plan from Navy RPM via email, with radiological review and concurrence of both plans to be obtained from RASO. Draft WP/Appendices to be submitted as hard copy to regulatory agencies for comments via email in 30 days. Gilbane PM to respond to regulatory comments via email within 2 weeks and revise as necessary to obtain concurrence on WP/Appendices. PM to acquire approval to initiate field work from Navy RPM and QAO after appropriate agency concurrence has been obtained.
Project management	Gilbane PM	John Baur	jbaur@gilbaneco.com	If changes are necessary, the PM communicates the changes via phone and/or email to the project staff and is authorized to stop work, if necessary. PM to provide Navy RPM and ROICC with all project-required notifications within 24 hours.

SAP Worksheet #6 – Communication Pathways (Continued)

Communication Drivers	Responsible Affiliation	Name	Phone Number and/or e-mail	Procedure
SAP review	Gilbane Program Chemist	Kristen Carlyon Peyton	kcarlyon@gilbaneco.com	The SAP is written by the Program Chemist and reviewed by the QCPM prior to submittal to the Navy QAO for review.
	Gilbane QCPM	Laura Tryboski	ltryboski@gilbaneco.com	
SAP procedure revision during field activities	Gilbane Project Chemist	Kristen Carlyon Peyton	kcarlyon@gilbaneco.com	The Project Chemist (or designee) will prepare a Field Change Request (FCR) for any changes that occur due to conditions in the field or laboratory that do not affect DQOs. FCRs affecting the SAP will be loaded to NIRIS for QAO review/approval.
SAP amendments	Gilbane Project Chemist	Kristen Carlyon Peyton	kcarlyon@gilbaneco.com	Significant changes to the SAP that affect DQOs, will require that the Project Chemist prepare an addendum, which will be reviewed and approved by the Navy QAO prior to initiating the affected field activities. Any SAP addendum also will be submitted to the regulatory agencies for review. Regulatory agencies will be notified via email within 1 week of all significant changes to the SAP that do not require approval.

SAP Worksheet #6 – Communication Pathways (Continued)

Communication Drivers	Responsible Affiliation	Name	Phone Number and/or e-mail	Procedure
Notification of non-usable analytical data	Gilbane Project Chemist	Kristen Carlyon Peyton	kcarlyon@gilbaneco.com	If significant problems are identified by the laboratory or the project team that impact the usability of the data (i.e., the data is rejected or the DQOs are not met), the Project Chemist will notify the PM; Navy RPM and Navy QAO will be notified within 24 hours or the next business day.
Coordination of laboratory supplies for field sampling activities	Gilbane Project Chemist	Kristen Carlyon Peyton	kcarlyon@gilbaneco.com	The Project Chemist will contact the subcontract laboratory to provide necessary sample containers and appropriate shipping materials (such as coolers and bubble wrap) to be delivered to the site prior to commencement of field sampling activities and throughout the course of the project.
Reporting laboratory data quality issues or analytical corrective actions	Pace National – Mt. Juliet & Minnesota/ Laboratory PM A&B Labs/Laboratory PM	Jennifer Gambill Shantall Carpenter	jgambill@pacenational.com scarpenter@ablabs.com	Data quality issues will be reported to the Project Chemist within 24 hours. Any corrective actions will be documented and verified by the Project Chemist, who will notify the PQCM, Site RSO (if applicable) and the PM in writing. The PM will notify the BRAC RPM and RASO (if applicable).

SAP Worksheet #6 – Communication Pathways (Continued)

Communication Drivers	Responsible Affiliation	Name	Phone Number and/or e-mail	Procedure
Stop work issues	All Gilbane and Navy Staff	Various	Various	The initiator of a stop work order verbally notifies Navy ROICC and Gilbane PM immediately. For stop work initiated by the Navy, the Contracting Officer must be verbally notified within 24 hours.
Field corrective actions	Gilbane PQCM	Chuck Clyde	ccllyde@gilbaneco.com	Field corrective actions will be documented in writing by the PQCM, who will notify the Site RSO (if applicable) and PM in writing. The PM will notify the BRAC RPM and RASO (if applicable). Regulatory agencies will be notified via email within one week of all substantive field corrective actions.

SAP Worksheet #7 – Personnel Responsibilities and Qualifications Table
(UFP-QAPP Manual Section 2.4.3)

Name	Title/Role	Organizational Affiliation	Responsibilities
Chris Yantos	Navy Project Manager	BRAC PMO	Oversees project execution and coordination with site representatives, regulatory agencies, and Navy management. Actively participates in the DQO process, and provides management and technical oversight during data collection. Is notified of delays in or changes to field work and has authority to stop work and initiate corrective action at any time.
Teresie Walker	Navy QAO	NAVFAC LANT	Responsible for QA issues for all Navy work; provides government oversight of the QA program for contractors. Reviews and approves the SAP and any significant SAP modifications or amendments; has the authority to suspend project activities if Navy quality requirements are not met.
Kimberly Noble	Navy EPM	RASO	Reviews radiological laboratory on a routine basis. Performs on-site reviews of all radiological site operations. Reviews and approves final reports. Performs quality reviews on chains of custody (COCs) to ensure samples are handled in accordance with the Work Plan (WP) and SAP. Provides review and concurrence on data for proposed radiological actions. Ensures that all necessary sample results are provided and are consistent with proposed radiological actions.
Laura Tryboski	QC Program Manager (QCPM)	Gilbane	Reviews QC processes, issues corrective action orders; assures adherence to requirements of the QC program, including the QA/QC Plan, and SAP, as appropriate. Can receive communication from the PM, Program Chemist, PQCM, and field staff. Has the authority to stop work and initiate corrective action.
Chuck Clyde	PQCM	Gilbane	Implements field-related quality control activities, issues nonconformance reports (NCRs), initiates necessary rework and/or corrective actions, and communicates with the PM, QCPM, Superintendent, and Project Chemist.

SAP Worksheet #7 – Personnel Responsibilities and Qualifications Table (Continued)

Name	Title/Role	Organizational Affiliation	Responsibilities
John Baur	Project Manager	Gilbane	Develops and implements all Task Order documents and activities. Assures overall project quality, implementation of three-phase quality control activities, and compliance with project schedule; and performs contract management, technical oversight, and report generation. Responsible for notifying the RPM of significant project information, including (but not limited to) project progress, schedule compliance, modifications to work, delays, analytical data quality issues, and safety-related issues.
Kristen Carlyon Peyton	Program/Project Chemist	Gilbane	Assists the QC Program Manager and assesses the propriety of the proposed analytical methodology; assists in the preparation of the SAP and with management of project tasks associated with sampling; reviews preservation requirements; coordinates SAP review/approval and other QA issues with the Navy QAO; conducts general oversight of and communication with the field personnel in relation to sampling activities; coordinates sample collection and analysis with the analytical laboratory; implements appropriate quality control activities and corrective actions; coordinates data validation activities and the uploading of data to appropriate databases. Coordinates third-party data validation of all definitive laboratory data. Reviews data validation reports.

SAP Worksheet #7 – Personnel Responsibilities and Qualifications Table (Continued)

Name	Title/Role	Organizational Affiliation	Responsibilities
Jerry Cooper	Corporate RSO/Certified Health Physicist	Gilbane	Oversees overall radiological operations. Insures that Site RSO and filed sampling personnel have adequate training in radiological sample collection. Oversees the preparation of remediation plans and the performance of remediation activities when sampling activities indicate the presence of radioactive materials at levels above the release criteria. Acts as a technical resource for radiological data collection for analysis, and technical discussions with stakeholders. Reviews radiological data to ensure the DQOs have been met. Provides critical analysis and interpretation of radiological data.
Chris Bryson	Site RSO	Envirachem	Supervises day-to-day radiological operations. Oversees performance of radiological static surveys. Collects and maintains records of instrument calibration and maintenance. Collects and maintains completed survey forms, chain of custody records, field log sheets, and other field data. Integrates field, graphical information system (GIS), and global positioning system (GPS) data and plot data on maps.
Tony Olmstead	Superintendent	Gilbane	Conducts oversight of all field activities; ensures implementation of individual elements of site or task-specific work plans; oversees the work of subcontractors performing field-related tasks; and oversees the collection of samples and coordinates shipments with laboratories. Ensures that the sampling protocol is followed in accordance with the SAP.

SAP Worksheet #7 – Personnel Responsibilities and Qualifications Table (Continued)

Name	Title/Role	Organizational Affiliation	Responsibilities
Jennifer Gambill Shantall Carpenter	Laboratory PM	Pace National (Mt. Juliet and Minnesota) A&B Labs	Performs chemical analyses; assures compliance with project requirements regarding performance of analytical procedures; supplies sample containers; handles and preserve samples in accordance with project-specified protocols.
Kevin Harmon	Data Validator	Validata	Performs data validation on analytical data used for project decisions.

SAP Worksheet #8 – Special Personnel Training Requirements Table

Project Function	Specialized Training by Title or Description of Course	Training Provider	Training Date	Personnel / Groups Receiving Training	Personnel Titles / Organizational Affiliation	Location of Training Records / Certificates
Radiation-impacted area sampling	Radiation Safety Awareness Training	Gilbane	Prior to field work	All project field personnel	Gilbane / Excavation or Demolition Subcontractors	Gilbane, Concord, CA
UXO-impacted area sampling	Military Munitions Hazards Training	Gilbane	Prior to field work	All project field personnel	Gilbane / Excavation or Demolition Subcontractors	Gilbane, Concord, CA

Training Requirements

Project personnel are required to meet the U.S. Occupational Safety and Health Organization (OSHA) training requirements defined in Title 29 Code of Federal Regulations (Title 29 CFR) Part 1910.120(e). These requirements are: (1) 40 hours of formal off-site instruction; (2) a minimum of 3 days of actual on-site field experience under the supervision of a trained and experienced field supervisor; and (3) 8 hours of annual refresher training. In addition, training will be provided to all field team members working in radiation-impacted areas according to the requirements of 10 Code of Federal Regulations (CFR) 19.12, Instructions to Workers.

SAP Worksheet #9 – Project Scoping Session Participants Sheet

Project Name: Phase IV Non-Time Critical Removal Action for Solid Waste Disposal Area Westside

Site Name: Installation Restoration Site 12

Site Location: Former Naval Station Treasure Island, San Francisco, California

Projected Date(s) of Sampling: June 2019 through November 2019

Project Manager: John Baur

Date of Scoping Session: October 16, 2018

Scoping Session Purpose: Describe scope of work

Scoping Session Participants:

Name	Title	Affiliation	Phone	E-mail Address	Project Role
Chris Yantos	RPM	Navy BRAC	619-524-6870	Christopher.yantos@navy.mil	RPM
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Glennwood Ivey	CSO	Navy CSO	415-743-4729	Glennwood.ivey@navy.mil	CSO
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Izzat Amadeya	ROICC	Navy ROICC	510-749-5947	izzat.amadeya@navy.mil	ROICC
John Baur	Project Manager	Gilbane	925-946-3212	jbaur@gilbaneco.com	PM
Tony Olmstead	Site Superintendent	Gilbane	925-250-7875	Aolmstead@gilbaneco.com	Site Superintendent
Jerry Grose	UXO Operations Manager	Gilbane	303-256-6142	jgrose@gilbaneco.com	UXO Operations Manager
Ed Palser	Radiological Operations Manager	Gilbane	505-400-4076	ePSLser@gilbaneco.com	Rad Operations Manager
Arvind Acharya	Program Manager	Gilbane	510-719-6858	aacharya@gilbaneco.com	Program Manager

SAP Worksheet #9 – Project Scoping Session Participants Sheet (Continued)

Comments/Decisions:

No decisions were made at the scoping meeting. The following issues were discussed:

- Community relations logistics will need to be worked out and closely coordinated by Gilbane and Navy.
- Project Schedule and sequencing of field work.
- Submittal of Project Plans.
- Logistics of dewatering, unexploded ordnance (UXO) clearance, odor suppression during excavation of petroleum products.

Action Items:

- (1) Gilbane to update project schedule to provide more detailed information relative to field work and planning documents.
- (2) Gilbane to submit internal draft work plans by end of November 2018.

Consensus Decisions:

No consensus decisions were reached at the meeting.

SAP Worksheet #10 – Problem Definition

Environmental investigation and remediation activities are conducted at Treasure Island under the U.S. Department of Defense (DoD) IR Program in accordance with CERCLA and National Oil and Hazardous Substances Pollution Contingency Plan (NCP) requirements in Title 40 Code of Federal Regulations, Part 300.415(b)(2). Under Executive Order 12580, the Navy is the lead agency responsible for implementation of the IR Program and removal actions.

As the lead federal agency, the Navy, including RASO, is working with DTSC and the Water Board to implement Phase IV of the NTCRA at SWDA Westside. These entities make up the planning group. The Navy coordinates activities at NSTI with the regulatory agencies under the terms of the 1992 Federal Facility Site Remediation Agreement. Navy, DTSC, and Water Board representatives are collectively referred to as the BRAC Cleanup Team (BCT) for NSTI. In addition, the CDPH works with DTSC to provide technical support on the radiological program. Other agencies and organizations also provide support to the BCT and the environmental program, including TIDA, the Treasure Island Community Development (also known as TICD), the Restoration Advisory Board (also known as RAB), the EPA, and other public groups.

This Phase IV project is considered an extension of the NTCRA Phase III and focuses on the removal of soil stained with total petroleum hydrocarbon (TPH) and co-mingled with radioactive contamination and buried debris. As described in the Action Memo (Navy, 2007), the NTCRA will substantially eliminate potential human health risks to a resident or utility worker from direct contact with soil by substantially eliminating the identified pathways of exposure to hazardous substances for current and future residents and utility workers and radiological contaminants identified in the initial stages of the NTCRA.

10.1 Site Description and History

IR Site 12 is located on the northwest portion of Treasure Island on a relatively flat 93-acre area (see Figure 1). The site consists of multi-plex housing units with private backyards and common area front yards, side yards and surrounding greenbelts. The area was originally used as a parking lot during the Golden Gate International Exposition. After the Navy gained full ownership of the island in 1940, the area was developed for bunker storage of munitions and other materials, vehicle and equipment storage, recreational playing fields, and disposal and burning of waste. Beginning in the 1960s, areas of IR Site 12 were incrementally developed into housing for Navy personnel and their dependents.

IR Site 12 was included in the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) process in 1988 because of findings documenting the potential for soil and groundwater contamination from debris that may not have been entirely removed during housing construction. In 2002, the IR Site 12

SAP Worksheet #10 – Problem Definition (Continued)

boundary was expanded to include all existing residential areas. The environmental restoration at IR Site 12 is ongoing in accordance with the National Oil and Hazardous Substances Pollution Contingency Plan (NCP) and CERCLA.

The focus of the initial work within IR Site 12 was the removal of chemicals that exceeded clean-up goals. However, during chemical investigation and remediation activities, radioactive material in the form of discrete radioactive objects (ROs) and associated localized radium-226 (Ra-226) contamination in adjacent soil was identified. Generally, the presence of radioactive material in IR Site 12 has been found to be co-located with chemical contaminants and visible debris.

10.2 Previous Investigations and Removal Actions

SWDA Westside is an approximately 4.5-acre area on the west side of IR Site 12 abutting Perimeter Road (see Figure 2), and includes previously excavated and backfilled areas west of Building 1133 (Mason Court) and west of Buildings 1323 and 1325. SWDA Westside contained chemical contaminants (predominantly lead) which were co-located with radiological objects (ROs) containing Ra-226 (e.g., foils, deck markers, instrument gauges, metal debris; also referenced as radioactive “commodities” in project documentation) and associated localized Ra-226 contaminated soil. Additionally, portions of SWDA Westside contain munitions-impacted soil labeled as Munitions Response Site 1 (MRS 1) shown on Figure 2.

A NTCRA was initiated at the SWDA Westside, then known as SWDA A/B, in May 2006. Phase I was initiated to remediate chemicals in soil associated with chemical/fuel storage and disposal or burning of waste on the western portion of IR Site 12. Excavation and chemical confirmation sampling were performed around Buildings 1119, 1121, 1123, 1125, 1319, 1321, 1323, and 1325. Phase II consisted of radiological surveys of two buildings (Buildings 1121 and 1323) and their subsequent demolition. The foundations from those buildings were removed during Phase III. Other Phase III activities included the radiological survey, demolition, and foundation slab removal of Buildings 1119, 1125, and 1319. Excavation within the building footprints and other areas within SWDA Westside was performed as required to determine extent of debris and contamination and to complete remediation. Phase III NTCRA activities at SWDA Westside were halted in May 2016 following the discovery of MEC not included in the ESS. Excavations were not completed. Instead, the work area was backfilled only enough to prevent ponding, BMPs (straw waddles, privacy fencing, signage, etc.) were installed, and contractor-owned equipment and materials were removed from the site.

In June 2000, the Navy collected soil gas samples from 70 locations within Site 12. Volatile organic compounds (VOCs) were reported at concentrations that exceeded screening criteria in samples collected near Building 1323 in SWDA Westside. The results from a subsequent soil gas investigation conducted in 2002 showed elevated

SAP Worksheet #10 – Problem Definition (Continued)

concentrations of VOCs in soil gas samples collected from either side of the roadway in front of Building 1323 (Sullivan Consulting Group and Tetra Tech EM, Inc., 2006).

In 2016 during Phase III NTCRA activities, the Navy decided to re-define the conceptual site model for SWDA Westside after excavation activities revealed solid waste extending down to a depth of approximately 15 feet bgs in an area near former Building 1321. An investigation into the extent of waste, both horizontally and vertically, of the entire SWDA Westside was launched (OTIE, 2018).

In September and October 2017, a series of borings was completed to define the horizontal and vertical extent of solid waste at SWDA Westside. The borings ranged in depth from 2.5 to 19 ft bgs. Visible debris was observed to a maximum depth of 17 ft bgs. Several borings showed evidence of petroleum/solvent impacts in addition to approximately 5% or less debris. Approximately one-third of the borings contained less than 10% visible inert debris greater than 1 ft bgs (but less than 5 ft bgs), interpreted to have been placed as a result of grading operations for construction of the IR Site 12 Navy housing. Typical debris encountered included concrete and asphalt, ceramic dishware, rusty nails/bolts, metal debris, and other common construction materials. Investigation results indicate that much of the SWDA Westside vicinity has been affected by inert debris burial and/or redistribution by grading. Several borings had little to no debris, indicating no further removal actions are required. These borings were generally located around the periphery of previous removals at the southern end of SWDA Westside.

The results of the waste delineation investigation (OTIE, 2018) form the basis for the areas to be excavated and their depths (see Figure 2). These areas exhibited: (1) greater than 10% debris, (2) debris deeper than 5 ft bgs relative to normal grade, (3) evidence of chemical impacts, or (4) radiological impact. Boring results identified waste debris at depths up to 12 to 15 ft bgs.

10.3 Remedial Action Objectives

The remedial action objective for the NTCRA per the Action Memo is as follows:

- Reduce the potential for human contact with chemically-contaminated soil near the ground surface within the four SWDAs at Site 12 under the current land and utility configuration.

Because the initial stages of the NTCRA have shown chemically-contaminated soil to be co-located with debris (KCH, 2015), and to achieve the secondary goal of TPH source removal, excavation will continue until no debris and/or TPH stained soil is visually observed to a maximum depth of 15 ft bgs. When excavation is completed, bottom and sidewall characterization samples for Ra-226, Pb, PAHs, PCBs,

SAP Worksheet #10 – Problem Definition (Continued)

dioxin/furans, and VOCs will be collected for the purposes of documenting left-in-place conditions.

SAP Worksheet #11 – Project Quality Objectives/Systematic Planning Process Statements

Step 1 | State the Problem

Environmental investigations for Site 12 (Figure 1) identified several areas of suspected debris disposal. After further site investigations and Phases I, II, and III of the NTCRA, additional activities are required in SWDAs Westside as part of Phase IV of the NTCRA, based on Alternative 3 of the *Action Memorandum/Interim Remedial Action Plan: Non-Time Critical Removal Action for Solid Waste Disposal Areas Installation Restoration Site 12 Old Bunker Area Naval Station Treasure Island San Francisco, California* (Navy, 2007) which calls for excavating soil from the common areas, roadways, and backyards to a depth of 4 feet (ft) below ground surface (bgs) and replacing it with clean fill. However, the excavation will extend laterally as long as buried debris and/or TPH contamination is visually present, and vertically to a maximum depth of 15 ft bgs. Radiological and chemical data will be collected for characterization purposes to document the as-left excavation surfaces. Although the Navy intends to remove TPH-contaminated soil from the site, the decision criteria for the limits of soil removal are based on past characterization data and visual evidence during the excavation; and not by a pre-determined clean-up goal and post-confirmation sampling.

In addition, approximately 3,300 cubic yards of stockpiled soil is left from the previous Phase III NTCRA work (APTIM, 2019). Munitions screening of the soil was halted following the discovery of munitions and explosives of concern (MEC) that could not be handled under the Phase III ESS. This resulted in existing soil stockpiles generated during the Phase III NTCRA being left in place that now require disposal under this Phase IV NTCRA. The existing stockpiles will be screened for munitions under an ESS approved for Phase IV and scanned for radioactive contamination under the work plan. They cannot be moved without proper engineering controls (ECs), but once cleared, will be transported offsite for disposal. This will be completed prior to the TPH-contaminated soil removal action.

Step 2 | Identify the Goals of the Study

The goals of the sampling activities are:

- Remove TPH-stained soil and debris from the four excavation areas in the SWDA Westside (SAP Worksheet # 18 and Figure 2) to a maximum depth of 15 feet.
- Characterize final concentrations of PAHs, PCBs, dioxin/furans, VOCs, Pb, and radium-226 left-in-place in completed excavation areas (shown on Figure 2).
- Confirm imported fill materials meet sampling and acceptance criteria per the Information Advisory Clean Imported Fill Material (DTSC, 2001).

SAP Worksheet # 11 – Project Quality Objectives/Systematic Planning Process Statements (Continued)

Step 2	Identify the Goals of the Study (Continued)
<p>The Study Questions Are:</p> <ul style="list-style-type: none"> • Are there no visual signs of debris or TPH staining or has a maximum depth of 15 ft bgs been reached? • Has original fill or Bay Mud been encountered? • Have final characterization samples been collected in accordance with SAP Worksheet 18? • Do imported fill materials meet the acceptance criteria presented on SAP Worksheet #15.7 through #15.15? 	
Step 3	Identify Information Inputs
<p>The following inputs will be used to make decisions for this project:</p> <ul style="list-style-type: none"> • Visual indications of TPH-staining and debris. • Sampling of imported fill material per DTSC guidance (dependent on source of fill). 	
Step 4	Define the Boundaries of the Study
<p>Figure 2, “Excavation Areas,” shows the estimated extent of excavation area one through four in SWDA Westside. The total estimated volume of soil and debris to be removed for all proposed excavations is 22,000 bank cubic yards (BCY). The depth of excavations needed to attain NTCRA objectives will be determined by the extent of debris discovered during excavation; however, will not exceed 15 feet. The project duration for the field activities is approximately 5 months, with excavation expected to begin in early August of 2020 and end in January 2021.</p>	
Step 5	Develop the Analytical Approach
<p>The decision rules for this RA are:</p> <p><i>Excavation and Characterization Sampling:</i></p> <ul style="list-style-type: none"> • If there is visible indication of staining or debris and the excavation has not reached a maximum depth of 15 feet bgs, excavation will continue in accordance with the Work Plan (Gilbane, 2018). • If there is no visible indication of staining or debris, then the excavation is complete and characterization samples will be collected in accordance with SAP Worksheets 17 and 18. • If the maximum depth of 15 feet bgs has been reached, then the excavation is complete and characterization samples will be collected in accordance with SAP Worksheets 17 and 18. <p><i>Imported fill material:</i></p> <ul style="list-style-type: none"> • If the required sampling frequency meets the requirements outlined in the DTSC <i>Information Advisory Clean Imported Fill Material</i> and the results meet the acceptance criteria presented in Worksheets #15-7 through #15-15, then the source will be considered suitable for backfill. 	

SAP Worksheet # 11 – Project Quality Objectives/Systematic Planning Process Statements (Continued)

Step 5	Develop the Analytical Approach (Continued)
	<ul style="list-style-type: none"> If the required sampling frequency does not meet the requirements outlined in the DTSC Information Advisory Clean Imported Fill Material, and/or results do not meet the acceptance criteria presented in Worksheets #15-7 through #15-15, then the source will not be considered suitable for backfill, and another source will be evaluated.
Step 6	Specify Performance or Acceptance Criteria
	<p>To limit uncertainty in obtained environmental data, criteria for the precision, accuracy, representativeness, completeness, comparability, and sensitivity parameters and reporting limits for the chemicals of concern have been developed to meet the PSLs for the RA. Measurement errors will be controlled by using appropriate sampling and analytical methods, adhering to the Department of Defense (DoD) Quality Systems Manual (QSM) (versions 5.1 and 5.1.1 as applicable), following established SOPs, and having third-party data validation to verify laboratory processes. The field crews will review the SAP before sample collection to limit sample collection errors. The subcontract analytical laboratory will have a copy of the SAP and will adhere to DoD QSM guidance to limit measurement errors.</p>
Step 7	Develop Plan for Obtaining Data
	<p>A resource-effective plan for collecting data sufficient to fulfill study objective developed in Steps 1 through 6 of the DQO process is described in SAP Worksheet #17.</p>

SAP Worksheet #12 – Measurement Performance Criteria Table

Measurement Performance Criteria Table – Field QC Samples

Matrix: Soil

QC Sample ¹	Analytical Group	Frequency	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample (Assesses Error for Sampling [S], Analytical [A] or both [S&A])
Field Duplicate ¹	All	Not Applicable	Precision	Not Applicable	S & A
Equipment Rinse Blanks	Dioxin/Furans, VOCs, SVOCs/PAHs, TPH, Pesticides, PCBs, Metals	None if disposable sampling equipment used; 1 per day if using non-disposable sampling equipment	Accuracy/Bias – Contamination	< ½ limit of quantitation (LOQ)	S
Matrix Spikes ²	Dioxin/Furans, Metals, VOCs, SVOCs/PAHs, PCBs, Pesticides, TPH	5%; one per 20 samples collected	Precision/Accuracy	Laboratory statistically derived control limits	A
Temperature Blanks ³	Mercury, VOCs, SVOCs, PCBs, Pesticides, TPH, Cyanide	Every cooler shipped to the laboratory	Representativeness	< 6 degrees Celsius (°C)	S

Notes:

¹ Due to the heterogeneous distribution of contaminants typically found in soil matrix, field duplicates for soil samples are not considered reliable for determining precision, and will not be collected for this project. Sample data are not qualified based on field duplicate precision; matrix spike duplicate or lab replicate data will be used to assess sample precision.

²Per DoD QSM 5.1.1 Table B-17 matrix spikes are not required for gamma spectroscopy.

³There are no temperature preservation requirements for metals, radionuclides, or asbestos; therefore, no temperature blanks are required.

SAP Worksheet #13 – Secondary Data Criteria and Limitations Table

Secondary Data	Data Source (originating organization, report title and date)	Data Generator(s) (originating organization, data types, data generation / collection dates)	How Data Will Be Used	Limitations on Data Use
Historical Data	<i>Final Site 12 Removal Site Evaluation and Action Memorandum for Time Critical Removal of Lead Contaminated Soil Near Building 1133, San Francisco, California</i> (November 1999)	TetraTech EMI	Assessment of site conditions	None
Historical Data	<i>Treasure Island Naval Station Historical Radiological Assessment, Former Naval Station Treasure Island</i> (February 2006)	Weston	Assessment of site conditions	None
Historical Data	<i>Final Historical Radiological Assessment—Supplemental Technical Memorandum, Naval Station Treasure Island, San Francisco, California</i> (July 2014)	TriEco-Tt, a Joint Venture of TriEco LLC and Tetra Tech EM, Inc.	Assessment of site conditions	None
Historical Data	<i>Final Post-Construction Summary Report, Installation Restoration Site 12, Solid Waste Disposal Area Westside, Former Naval Station Treasure Island, San Francisco, California.</i> (December 2014)	CB&I	Assessment of site conditions	None
Historical Data	<i>Final Phase III Non-Time Critical Removal Action, Solid Waste Disposal Areas, IR Site 12, Former Naval Station Treasure Island, San Francisco, California.</i> (June 2015)	CB&I	Assessment of site conditions	None.

SAP Worksheet #13 – Secondary Data Criteria and Limitations Table

Secondary Data	Data Source (originating organization, report title and date)	Data Generator(s) (originating organization, data types, data generation / collection dates)	How Data Will Be Used	Limitations on Data Use
Design Plans	<i>Revised Engineering Evaluation and Cost Analysis, Solid Waste Disposal Areas, Installation Restoration Site 12, Old Bunker Storage Area, Naval Station Treasure Island, San Francisco, California (October, 2006)</i>	SulTech, A Joint Venture of Sullivan Consulting Group and Tetra Tech EM Inc.	To guide work yet to be completed.	None
Design Plans	<i>Final Action Memorandum/Interim Remedial Action Plan: Non-Time Critical Removal Action for Solid Waste Disposal Areas Installation Restoration Site 12 Old Bunker Area, Naval Station Treasure Island, San Francisco, California (February, 2007)</i>	Navy	To guide work yet to be completed.	None

SAP Worksheet #14 – Summary of Project Tasks

14.1 Major Project Tasks

Tasks applicable to sampling activities performed by Gilbane for this project at will include the following:

- Screening of remnant Phase III soil per the Work Plan and Explosives Safety Submission (ESS; Appendix A to the Work Plan) for munitions and/or low-level radioactive objects.
- Pre-excavation activities including notifications, utility clearance, and construction of laydown pads per the Work Plan (Gilbane, 2020).
- Excavation until no debris and/or TPH stained soil is visually observed up to a maximum of 15 ft bgs.
- Screening of excavated soil per the Work Plan and ESS, as applicable.
- Collection of post-excavation characterization samples for Ra-226, Pb, PAHs, PCBs, dioxins, and VOCs.
- Conducting of a site wide radiological scan of SWDA in accordance with the Work Plan (Gilbane, 2020) for the purposes of removing radiological controls from the SWDA Westside RCA.
- Backfilling of excavated areas with clean imported fill.
- Soil cover installation and site restoration per the Work Plan.
- Waste characterization and disposal per the Waste Management Plan (Appendix F of the Work Plan).
- Validation of data and complete data quality assessment.
- Uploading of data to Naval Installation Restoration Information Solution (NIRIS).

14.1.1 Sample Collection Procedures

The following sections provide the sampling procedures for collection of samples associated with remediation activities for this project. Samples will be labeled, documented, and packaged in accordance with the procedures in SAP Worksheets #27 and 29. The types of samples to be collected are listed below:

Excavation Soil Samples

Soil samples will be collected from excavation sidewalls and bottom using disposable plastic scoops in accordance with Gilbane SOP PR-TC-02.02.01.01 (Attachment 1). Samples will be collected from the backhoe/excavator bucket using the general sampling technique described below:

1. Put on a new (unused) pair of sampling gloves and other appropriate personal protective equipment (PPE).
2. Obtain a new (unused) disposable sampling scoop (or other non-disposable decontaminated sampling equipment).

SAP Worksheet #14 – Summary of Project Tasks (Continued)

3. If the excavation is less than 4 feet in depth or is sloped, the excavation may be entered for sample collection.
4. If the excavation is greater than 4 feet and un-sloped, then the excavation will not be entered for sampling. Direct the excavator operator to obtain a sample from the desired location. Allow excess water, if present, to drain from the soil before collecting the sample.
5. Collect sample from soil in center of excavator bucket.
6. Soil samples for VOC or TPH-p analyses will be collected using TerraCore® sampling devices (or equivalent) following the procedure described in Gilbane SOP PR-TC-02.02.01.05 (Attachment 1).
7. Collect the sample for the remaining analysis into the appropriate sample containers using a disposable sampling scoop. Sample containers are listed in SAP Worksheet #19.
8. Label, package, and prepare the samples for shipment to the laboratory in accordance with SOP PR-TC-02.04.01.01 (Attachment 1).
9. Place the chemistry samples in an ice-filled cooler in accordance with SOP PR-TC-02.04.01.01 (Attachment 1) after collection.

All samples will be documented, handled, and shipped in accordance with the provisions set forth by Worksheet #27.

14.1.2 Decontamination Procedures

Prior to decontamination, non-disposable sampling equipment will be wiped down and radiologically surveyed. Total radioactivity will be measured using a hand-held alpha/beta survey meter. A smear sample will be collected and analyzed for removable radioactivity. If radioactivity exceeding the release limits shown below in Table 14-1 is detected, the sampling equipment will be wiped down again and re-surveyed.

SAP Worksheet #14 – Summary of Project Tasks (Continued)

Table 14-1		
Type of Radiation	Removable (disintegrations per minute [dpm]/100cm ²)	Total (dpm/100cm ²)
Alpha	20	100
Beta	1,000	5,000

Note: To eliminate the need for isotopic identification, alpha radioactivity is assumed to be Ra-226. An industry-standard default value is used for beta radioactivity since there are no beta-emitting ROCs.

Once radioactivity is below the release limits, the non-disposable sampling equipment that comes into contact with samples will be decontaminated to prevent the introduction of extraneous material into samples, and to prevent cross-contamination between samples. All equipment will be decontaminated by steam cleaning or by washing with a non-phosphate detergent such as Liquinox™ or equivalent. Decontamination water will be collected in 55-gallon U.S. Department of Transportation (DOT)-approved drums or a poly tank and handled as liquid waste in accordance with the Waste Management Plan.

The following procedures will be used to decontaminate non-disposable sampling equipment:

1. If mud or soil is adhering to the sampling equipment, first rinse with potable water. This step will decrease the gross contamination and reduce the frequency at which the non-phosphate detergent and water solution need to be changed.
2. Wash with the non-phosphate detergent and water solution. This step will remove remaining contamination from the equipment. Dilute the non-phosphate detergent as directed by the manufacturer.
3. Rinse with potable water. Change the water frequently.
4. Rinse twice with deionized water. This step will rinse any detergent solution and potable water residues. Rinsing will be done by applying the deionized water from a clean squeeze bottle (or equivalent) while holding equipment over a bucket.
5. Store unused decontaminated equipment in plastic or designated storage container to prevent contamination until next use.

SAP Worksheet #14 – Summary of Project Tasks (Continued)

14.4 Post-Sampling Field Tasks

Waste characterization and disposal will be performed as detailed in the Waste Management Plan (Appendix B of the RAWP). Site restoration and surveying will be performed as detailed in Section 8.1, and Section 6.0, respectively, of the project RAWP.

14.2 Analytical Tasks

The handling of the samples and transferring of custody must be well documented given the evidentiary nature of the analytical data. The integrity and traceability of samples from the time they are collected through the time the data are reported are essential in any sampling and analysis program. Sample custody and procedures are described in WS #27. Sampling locations and analytical methods are described further in WS #18 through #20.

14.3 Quality Control Tasks

Analytical methods will require the applicable QC tasks described in the respective methods and DoD QSM 5.1 or 5.1.1 as applicable, including initial calibrations, continuing calibrations, tuning, reagent blanks, surrogates, replicates, control spikes, and others, as necessary.

Media-specific field quality control samples (as described on SAP Worksheet #12) include field duplicates to assess sampling and analytical precision, as well as trip blanks and equipment blanks to assess sampling and analytical accuracy.

14.4 Data Management and Review Tasks

Analytical data generated by the fixed analytical laboratory will be reviewed by the laboratory using three levels of document review and reporting. Review processes will be documented using appropriate checklists, forms, or logbooks, which will be signed and dated by the reviewers. Field surveying data, field forms, and chain-of-custody (COC) records will be reviewed by the PQCM and/or the Project Chemist and maintained in the Gilbane project file in accordance with Gilbane standard operating procedure (SOP) PR-TC-01.04.01.00, Field Documentation.

The Site Superintendent or PQCM will e-mail a copy of the COC records to the Project Chemist the day any samples are sent to the laboratory. The Project Chemist will maintain a copy of the COC record until submitted to the Navy Administrative Record along with the hard-copy packages as described in SAP Worksheet #29. The laboratory will e-mail definitive analytical results within the turnaround time to the Project Chemist. This submittal will include surrogates, and matrix spike/matrix spike duplicates (MS/MSDs). The Project Chemist will review prior to distribution to the project team. Following this submittal, the laboratory will be required to submit a DoD Stage 4-suitable data packages package within 10 business days of the sample collection date as described in SAP Worksheet #29.

SAP Worksheet #14 – Summary of Project Tasks (Continued)

Information from the COC records for samples analyzed by the laboratory will be uploaded into a database following the procedures outlined in Gilbane SOP PC-TR-02.12.02.00, Sample Tracking and Electronic Data Management. The laboratories will provide electronic data deliverables (EDDs) to upload analytical results into the Gilbane database. The EDD will be checked for required values and project-specific requirements. Any discrepancies in the EDD will be corrected by the laboratory. Validated results will be exported for upload to NIRIS in accordance with Environmental Work Instruction (EWI) #6, Environmental Data Management and Required Electronic Delivery Standards (Naval Facilities Engineering Command Southwest [NAVFAC SW], 2005). The database will be backed up on electronic media or an independent server.

Survey data will be recorded by on-site personnel for all sample locations. Horizontal control information for upload into the database will be captured in the State Plane Coordinate System in feet, and vertical control standards will be in mean sea level. Survey data for upload into the NIRIS will be in accordance with EWI #6, (NAVFAC SW, 2005).

DoD Stage 4 hard-copy data packages will be stored until subsequent submittal to the Navy Administrative Record as described in SAP Worksheet #29.

14.5 Third Party Data Validation

Analytical results that represent definitive data will be validated by a third-party data validation service provider. The validation report is described in SAP Worksheet #29, and the validation qualifiers will be entered electronically in the Gilbane project database by the validator to the EDDs loaded by the laboratory.

SAP Worksheet #15.1 – Reference Limits and Evaluation Table

Matrix: Soil (Characterization Sampling)

Analytical Group: Polycyclic Aromatic Hydrocarbons (PAHs) by EPA 8270CSIM

Analyte	Chemical Abstracts Service (CAS) Number	Project Screening Limit ² (mg/kg)	Project Screening Limit Reference	Project Quantitation Limit Goal (mg/kg)	Laboratory-specific		
					LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
Acenaphthene	80-23-9	NA	NA	0.0066	0.006	0.003	0.0006
Anthracene	120-12-7	NA	NA	0.0066	0.006	0.003	0.0006
Benzo(a)anthracene	56-55-3	NA	NA	0.0066	0.006	0.003	0.0006
Benzo(a)pyrene	50-32-8	NA	NA	0.0066	0.006	0.003	0.0006
Benzo(b)fluoranthene	205-99-2	NA	NA	0.0066	0.006	0.003	0.0006
Benzo(k)fluoranthene	207-08-9	NA	NA	0.0066	0.006	0.003	0.0006
Benzo(g,h,i)perylene	191-24-2	NA	NA	0.0066	0.006	0.003	0.0006
Chrysene	218-01-9	NA	NA	0.0066	0.006	0.003	0.0006
Dibenz(a,h)anthracene	53-70-3	NA	NA	0.0066	0.006	0.003	0.0006
Fluoranthene	206-44-0	NA	NA	0.0066	0.006	0.003	0.0006
Fluorene	86-73-7	NA	NA	0.0066	0.006	0.003	0.0006
Indeno(1,2,3-cd)pyrene	193-39-5	NA	NA	0.0066	0.006	0.003	0.0006
Naphthalene	91-20-3	NA	NA	0.0066	0.02	0.01	0.002
Pyrene	129-00-0	NA	NA	0.0066	0.006	0.003	0.0006
PAHs as BAP (EQ) ²	NL	NA	NA ¹	NA	NA	NA	NA

Notes:

¹NA = Not Applicable; the results of characterization sampling are informational only to document left-in-place conditions and will not trigger step-out sampling.

²PAHs as benzo(a)pyrene equivalency [BAP (EQ)] will be calculated as described on Worksheet 17.2.

³Results will be reported on a dry-weight basis for comparison to the project action limits.

mg/kg = milligrams per kilogram

NA = not applicable

NL = not listed

SAP Worksheet #15.2 – Reference Limits and Evaluation Table

Matrix: Soil (Characterization Sampling)

Analytical Group: Dioxin/Furans by EPA Method 8290

Analyte	Chemical Abstracts Service (CAS) Number	Project Screening Limit ² (pg/g)	Project Screening Limit Reference	Project Quantitation Limit Goal (pg/g)	Laboratory-specific		
					LOQ (pg/g)	LOD (pg/g)	EDL ³ (pg/g)
2,3,7,8-TCDD	1746-01-6	NA	NA	1	2.0	0.5	TBD
1,2,3,7,8-PeCDD	40321-76-4	NA	NA	5	5.0	0.5	TBD
1,2,3,4,7,8-HxCDD	39277-28-6	NA	NA	5	5.0	1	TBD
1,2,3,6,7,8-HxCDD	57653-85-7	NA	NA	5	5.0	0.5	TBD
1,2,3,7,8,9-HxCDD	19408-74-3	NA	NA	5	5.0	1	TBD
1,2,3,4,6,7,8-HpCDD	35822-39-4	NA	NA	5	5.0	0.5	TBD
OCDD	3268-87-9	NA	NA	10	10.0	2.0	TBD
2,3,7,8-TCDF	51207-31-9	NA	NA	1	2.0	0.5	TBD
1,2,3,7,8-PeCDF	57117-41-6	NA	NA	5	5.0	0.5	TBD
2,3,4,7,8-PeCDF	57117-31-4	NA	NA	5	5.0	1.0	TBD
1,2,3,4,7,8-HxCDF	70648-26-9	NA	NA	5	5.0	0.5	TBD
1,2,3,6,7,8-HxCDF	57117-44-9	NA	NA	5	5.0	1.0	TBD
1,2,3,7,8,9-HxCDF	72918-21-9	NA	NA	5	5.0	2.0	TBD
2,3,4,6,7,8-HxCDF	60851-34-5	NA	NA	5	5.0	1.0	TBD
1,2,3,4,6,7,8-HpCDF	67562-39-4	NA	NA	5	5.0	0.5	TBD
1,2,3,4,7,8,9-HpCDF	55673-89-7	NA	NA	5	5.0	1.0	TBD
OCDF	39001-02-0	NA	NA	10	10.0	1.0	TBD
Dioxins as 2,3,7,8-TCDD TEQ	NL	NA	NA ¹	NA	NA	NA	NA

Notes:

¹NA = Not Applicable; the results of characterization sampling are informational only to document left-in-place conditions and will not trigger step-out sampling.

The 2,3,7,8-TCDD TEQ (Dioxin TEQ) is based on the 2,3,7,8-TCDD congener and is calculated as described in Worksheet 17.

SAP Worksheet #15.2 – Reference Limits and Evaluation Table (Continued)

²Results will be reported on a dry-weight basis for comparison to the project action limits.

³Estimated Detection Limit (EDL) – For each chemical not detected, an EDL is calculated. The sample specific EDL is an estimate made by the laboratory of the concentration of a given chemical that would have to be present to produce a signal with a peak height of at least 2.5 times the background signal level. The estimate is specific to a particular analysis of the sample and will be affected by sample size, dilution, etc. Because of the toxicological significance of dioxins, the EDL value is reported for nondetected chemicals rather than reporting the quantitation limit.

pg/g = picograms per gram

DL = detection limit

EMPC = estimated maximum potential
concentration

HpCDD = heptachlorodibenzo-p-dioxin

HpCDF = heptachlorodibenzofuran

HxCDD = hexachlorodibenzo-p-dioxin

HxCDF = hexachlorodibenzofuran

LOD = limit of detection

NA = not applicable

NL = not listed

OCDF = octachlorodibenzofuran

PeCDD = pentachlorodibenzo-p-dioxin

PeCDF = pentachlorodibenzofuran

QL = quantitation limit

TCDD = tetrachlorodibenzo-p-dioxin

TCDF = tetrachlorodibenzofuran

TBD = to be determined

SAP Worksheet #15.3 – Reference Limits and Evaluation Table

Matrix: Soil (Characterization Sampling)

Analytical Group: PCBs by EPA 8082

Analyte	Chemical Abstracts Service (CAS) Number	Project Screening Limit ³ (mg/kg)	Project Screening Limit Reference	Project Quantitation Limit Goal (mg/kg)	Laboratory-specific		
					LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
Aroclor 1016	12674-11-2	NA	NA	0.033	0.017	0.0085	0.003504
Aroclor 1221	11104-28-2	NA	NA	0.033	0.017	0.0085	0.005367
Aroclor 1232	11141-16-5	NA	NA	0.033	0.017	0.0085	0.004168
Aroclor 1242	53469-21-9	NA	NA	0.033	0.017	0.0085	0.003176
Aroclor 1248	12672-29-6	NA	NA	0.033	0.017	0.0085	0.003148
Aroclor 1254	11097-69-1	NA	NA	0.033	0.017	0.0085	0.004722
Aroclor 1260	11096-82-5	NA	NA	0.033	0.017	0.0085	0.004942
Total Aroclors ¹	NL	NA	NA ²	NA	NA	NA	NA

Notes:

¹ Total Aroclors will be calculated by summation of the seven listed Aroclors. Non-detected values will be included in the sum as one-half the value of the LOD.

² NA = Not Applicable; the results of characterization sampling are informational only to document left-in-place conditions and will not trigger step-out sampling.

³ Results will be reported on a dry-weight basis for comparison to the project action limits.

DL = detection limit

LOD = limit of detection

LOQ = limit of quantitation

NA = not applicable

NL = not listed

mg/kg = milligram per kilogram

PCB = polychlorinated biphenyls

SAP Worksheet #15.4 – Reference Limits and Evaluation Table

Matrix: Soil (Characterization Sampling)

Analytical Group: Lead by EPA Method 6010B

Analyte	CAS Number	Project Screening Limit ² (mg/kg)	Project Screening Limit Reference	Project Quantitation Limit Goal (mg/kg)	Laboratory-specific		
					LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
Lead	7439-92-1	NA	NA ¹	1	0.50	0.25	0.19

Notes:

¹NA = Not Applicable; the results of characterization sampling are informational only to document left-in-place conditions and will not trigger step-out sampling.

²Results will be reported on a dry-weight basis for comparison to the project screening limits.

DL = detection limit

LOD=limit of detection

LOQ=limit of quantitation

mg/kg = milligrams per kilogram

SAP Worksheet #15.5 – Reference Limits and Evaluation Table

Matrix: Soil (Characterization Sampling)

Analytical Group: VOCs by EPA Method 8260B

Analyte	CAS Number	Project Screening Limit ² (mg/kg)	Project Screening Limit Reference	Project Quantitation Limit Goal (mg/kg)	Laboratory-specific		
					LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
1,1,1,2-Tetrachloroethane	630-20-6	NA	NA ¹	0.005	0.0025	0.00125	0.0005
1,1,1-Trichloroethane	71-55-6	NA	NA ¹	0.005	0.0025	0.00125	0.000275
1,1,2,2-Tetrachloroethane	79-34-5	NA	NA ¹	0.005	0.0025	0.00125	0.00039
1,1,2-Trichloroethane	79-00-5	NA	NA ¹	0.005	0.0025	0.00125	0.000883
1,1-Dichloroethane	75-34-3	NA	NA ¹	0.005	0.0025	0.00125	0.000575
1,1-Dichloroethene	75-35-4	NA	NA ¹	0.005	0.0025	0.00125	0.0005
1,2,3-Trichlorobenzene	87-61-6	NA	NA ¹	0.005	0.0025	0.00125	0.000625
1,2,4-Trichlorobenzene	120-82-1	NA	NA ¹	0.005	0.0125	0.00625	0.00482
1,2,4-Trimethylbenzene	95-63-6	NA	NA ¹	0.005	0.0025	0.00125	0.00116
1,2-Dibromo-3-chloropropane	96-12-8	NA	NA ¹	0.010	0.025	0.0125	0.0051
1,2-Dibromoethane (EDB)	106-93-4	NA	NA ¹	0.010	0.0025	0.00125	0.000525
1,2-Dichlorobenzene	95-50-1	NA	NA ¹	0.005	0.005	0.0025	0.00145
1,2-Dichloroethane	107-06-2	NA	NA ¹	0.005	0.0025	0.00125	0.000475
1,2-Dichloropropane	78-87-5	NA	NA ¹	0.005	0.005	0.0025	0.00175
1,3,5-Trimethylbenzene	108-67-8	NA	NA ¹	0.005	0.005	0.0025	0.00108
1,3-Dichloropropane	142-28-9	NA	NA ¹	0.005	0.005	0.0025	0.00175
1,3-Dichlorobenzene	541-73-1	NA	NA ¹	0.005	0.005	0.0025	0.0017
1,4-Dichlorobenzene	106-46-7	NA	NA ¹	0.005	0.005	0.0025	0.00197
2-Butanone (MEK)	78-93-3	NA	NA ¹	0.010	0.025	0.0125	0.0025
2-Chlorotoluene	95-49-8	NA	NA ¹	0.005	0.0025	0.00125	0.00092
2-Hexanone	591-78-6	NA	NA ¹	0.010	0.025	0.0125	0.01
4-Chlorotoluene	106-43-4	NA	NA ¹	0.005	0.005	0.0025	0.00113
4-Methyl-2-pentanone	108-10-1	NA	NA ¹	0.010	0.025	0.0125	0.00625
Acetone	67-64-1	NA	NA ¹	0.020	0.025	0.0188	0.0137

SAP Worksheet #15.5 – Reference Limits and Evaluation Table (Continued)

Matrix: Soil (Characterization Sampling)

Analytical Group: VOCs by EPA Method 8260B

Analyte	CAS Number	Project Screening Limit ² (mg/kg)	Project Screening Limit Reference	Project Quantitation Limit Goal (mg/kg)	Laboratory-specific		
					LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
Benzene	71-43-2	NA	NA ¹	0.005	0.001	0.0005	0.0004
Bromobenzene	108-86-1	NA	NA ¹	0.005	0.0125	0.00625	0.00105
Bromochloromethane	74-97-5	NA	NA ¹	0.005	0.005	0.0025	0.00113
Bromodichloromethane	75-27-4	NA	NA ¹	0.005	0.0025	0.00125	0.000788
Bromoform	75-25-2	NA	NA ¹	0.005	0.025	0.0125	0.00598
Bromomethane	74-83-9	NA	NA ¹	0.005	0.0125	0.00625	0.0037
Carbon disulfide	75-15-0	NA	NA ¹	0.01	0.0125	0.00625	0.00406
Carbon tetrachloride	56-23-5	NA	NA ¹	0.005	0.005	0.0025	0.00108
Chlorobenzene	108-90-7	NA	NA ¹	0.005	0.0025	0.00125	0.000573
Chloroform	67-66-3	NA	NA ¹	0.005	0.0025	0.00125	0.000415
Chloromethane	74-87-3	NA	NA ¹	0.005	0.0125	0.00625	0.00139
cis-1,2-Dichloroethene	156-59-2	NA	NA ¹	0.005	0.0025	0.00125	0.00069
cis-1,3-Dichloropropene	10061-01-5	NA	NA ¹	0.005	0.0025	0.00125	0.000678
Dibromochloromethane	124-48-1	NA	NA ¹	0.005	0.0025	0.00125	0.00045
Dibromomethane	74-95-3	NA	NA ¹	0.005	0.005	0.0025	0.001
Dichlorodifluoromethane	75-71-8	NA	NA ¹	0.005	0.0025	0.00125	0.000818
Ethylbenzene	100-41-4	NA	NA ¹	0.005	0.0025	0.00125	0.00053
Hexachlorobutadiene	87-68-3	NA	NA ¹	0.005	0.025	0.00125	0.000425
Methyl tert-Butyl ether	1634-04-4	NA	NA ¹	0.005	0.001	0.0005	0.000295
Methylene chloride	75-09-2	NA	NA ¹	0.005	0.025	0.0125	0.00664
m,p-Xylenes	179601-23-1	NA	NA ¹	0.005	0.004	0.002	0.0015
o-Xylene	95-47-6	NA	NA ¹	0.005	0.0025	0.00125	0.001
Styrene	100-42-5	NA	NA ¹	0.005	0.0125	0.00625	0.00273
Tetrachloroethene	127-18-4	NA	NA ¹	0.005	0.0025	0.00125	0.0007

SAP Worksheet #15.5 – Reference Limits and Evaluation Table (Continued)

Matrix: Soil (Characterization Sampling)

Analytical Group: VOCs by EPA Method 8260B

Analyte	CAS Number	Project Screening Limit ² (mg/kg)	Project Screening Limit Reference	Project Quantitation Limit Goal (mg/kg)	Laboratory-specific		
					LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
Toluene	108-88-3	NA	NA ¹	0.005	0.005	0.0025	0.00125
trans-1,2-Dichloroethene	156-60-5	NA	NA ¹	0.005	0.005	0.0025	0.00143
Trichloroethene	79-01-6	NA	NA ¹	0.005	0.001	0.0005	0.0004
Trichlorofluoromethane	75-69-4	NA	NA ¹	0.005	0.0025	0.00125	0.0005
Vinyl chloride	75-01-4	NA	NA ¹	0.005	0.0025	0.00125	0.0005

Notes:

¹NA = Not Applicable; the results of characterization sampling are informational only to document left-in-place conditions and will not trigger step-out sampling.

²Results will be reported on a dry-weight basis for comparison to the project screening limits.

DL = detection limit

LOD = limit of detection

LOQ = limit of quantitation

mg/kg = milligram per kilogram

SAP Worksheet #15.6 – Reference Limits and Evaluation Table

Matrix: Soil (Characterization Sampling)

Analytical Group: Radium-226 by HASL 300 GA-01-R

Analyte ¹	CAS Number	Project Screening Limit ² (pCi/g)	Project Screening Limit Reference	Project Quantitation Limit Goal (pCi/g)	Laboratory-specific
					MDC (pCi/g)
Radium-226	13982-63-3	NA	NA ²	0.5	0.5

Notes:

¹ Radium-226 will be reported based on the 609 keV bismuth-214 gamma energy peak after 21-day in-growth.

²NA = Not Applicable; the results of characterization sampling are informational only to document left-in-place conditions and will not trigger step-out sampling. keV = kilo-electron volts

MDC = minimum detectable concentration

NA = not applicable

pCi/g = picocuries per gram

SAP Worksheet #15.7 – Reference Limits and Evaluation Table

Matrix: Soil (testing of imported fill material)

Analytical Group: Radium-226 by HASL 300 GA-01-R

Analyte ¹	CAS Number	Project Screening Limit ² (pCi/g)	Project Screening Limit Reference	Project Quantitation Limit Goal (pCi/g)	Laboratory-specific
					MDC (pCi/g)
Radium-226	13982-63-3	1.69	Sitewide Radiation Protection Plan ³	0.5	0.5

Notes:

¹ Radium-226 will be reported based on the 609 keV bismuth-214 gamma energy peak after 21-day in-growth.

²Project Screening level is background plus the derived concentration guideline level (DCGL).

³*Sitewide Radiation Protection Plan – Revision 1, Treasure Island, San Francisco, California* (CB&I, 2014)

keV = kilo-electron volts

MDC = minimum detectable concentration

NA = not applicable

pCi/g = picocuries per gram

SAP Worksheet #15.8 – Reference Limits and Evaluation Table

Matrix: Soil (testing of import fill material)

Analytical Group: VOCs by EPA Method 8260B

Analyte	CAS Number	Project Screening Limit ⁴ (mg/kg)	Project Screening Limit Reference	Project Quantitation Limit Goal (mg/kg)	Laboratory-specific		
					LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
1,1,1,2-Tetrachloroethane	630-20-6	2.0	HERO HHRA Note 3 ²	1	0.0025	0.00125	0.0005
1,1,1-Trichloroethane	71-55-6	8,100	EPA Residential RSL ¹	4,000	0.0025	0.00125	0.000275
1,1,2,2-Tetrachloroethane	79-34-5	0.61	HERO HHRA Note 3 ²	0.3	0.0025	0.00125	0.00039
1,1,2-Trichloroethane	79-00-5	1.1	EPA Residential RSL ¹	0.5	0.0025	0.00125	0.00883
1,1-Dichloroethane	75-34-3	3.6	HERO HHRA Note 3 ²	1.3	0.0025	0.00125	0.000575
1,1-Dichloroethene	75-35-4	230	EPA Residential RSL ¹	100	0.0025	0.00125	0.0005
1,2,3-Trichlorobenzene	87-61-6	63	HERO HHRA Note 3 ²	30	0.0025	0.00125	0.000625
1,2,4-Trichlorobenzene	120-82-1	24	EPA Residential RSL ¹	12	0.0125	0.00625	0.00482
1,2,4-Trimethylbenzene	95-63-6	300	EPA Residential RSL ¹	150	0.005	0.0025	0.00116
1,2-Dibromo-3-	96-12-8	0.0053	EPA Residential RSL ¹	0.005	0.0025	0.0125	0.0051
1,2-Dibromoethane (EDB)	106-93-4	0.036	EPA Residential RSL ¹	0.018	0.0025	0.00125	0.000525
1,2-Dichlorobenzene	95-50-1	1,800	EPA Residential RSL ¹	900	0.005	0.0025	0.00145
1,2-Dichloroethane	107-06-2	0.46	EPA Residential RSL ¹	0.23	0.0025	0.00125	0.000525
1,2-Dichloropropane	78-87-5	2.5	EPA Residential RSL ¹	1.2	0.005	0.0025	0.00127
1,3,5-Trimethylbenzene	108-67-8	270	EPA Residential RSL ¹	130	0.005	0.0025	0.00108
1,3-Dichloropropane	142-28-9	1,600	EPA Residential RSL ¹	800	0.005	0.0025	0.00175
1,3-Dichlorobenzene	541-73-1	16	TI SSC ³	8	0.005	0.0025	0.0017
1,4-Dichlorobenzene	106-46-7	2.6	EPA Residential RSL ¹	1.3	0.005	0.0025	0.00197
2-Butanone	78-93-3	27,000	EPA Residential RSL ¹	13,000	0.025	0.0125	0.00025
2-Chlorotoluene	95-49-8	480	HERO HHRA Note 3 ²	200	0.0025	0.00125	0.000793
2-Hexanone	591-78-6	200	EPA Residential RSL ¹	100	0.025	0.0125	0.01
4-Chlorotoluene	106-43-4	440	HERO HHRA Note 3 ²	200	0.005	0.0025	0.00113
4-Methyl-2-pentanone	108-10-1	33,000	EPA Residential RSL ¹	15,000	0.025	0.0125	0.01
Acetone	67-64-1	61,000	EPA Residential RSL ¹	30,000	0.025	0.0188	0.0137

SAP Worksheet #15.8 – Reference Limits and Evaluation Table (Continued)

Matrix: Soil (testing of imported fill material)

Analytical Group: VOCs by EPA Method 8260B

Analyte	CAS Number	Project Screening Limit ⁴ (mg/kg)	Project Screening Limit Reference	Project Quantitation Limit Goal (mg/kg)	Laboratory-specific		
					LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
Benzene	71-43-2	0.33	HERO HHRA Note 3 ²	0.015	0.001	0.0005	0.0004
Bromobenzene	108-86-1	290	EPA Residential RSL ¹	150	0.0125	0.00625	0.00104
Bromochloromethane	74-97-5	150	EPA Residential RSL ¹	75	0.005	0.0025	0.00113
Bromodichloromethane	75-27-4	0.30	HERO HHRA Note 3 ²	0.15	0.0025	0.00125	0.000788
Bromoform	75-25-2	20	HERO HHRA Note 3 ²	10	0.025	0.0125	0.00598
Bromomethane	74-83-9	6.8	EPA Residential RSL ¹	3.4	0.0125	0.00625	0.0037
Carbon disulfide	75-15-0	770	EPA Residential RSL ¹	330	0.0125	0.00625	0.00406
Carbon tetrachloride	56-23-5	0.099	HERO HHRA Note 3 ²	0.005	0.005	0.0025	0.00107
Chlorobenzene	108-90-7	280	EPA Residential RSL ¹	140	0.0025	0.00125	0.000573
Chloroform	67-66-3	0.32	EPA Residential RSL ¹	0.15	0.0025	0.00125	0.000415
Chloromethane	74-87-3	110	EPA Residential RSL ¹	50	0.0125	0.00625	0.00139
cis-1,2-Dichloroethene	156-59-2	19	HERO HHRA Note 3 ²	10	0.0025	0.00125	0.00069
cis-1,3-Dichloropropene	10061-01-5	1.8	EPA Residential RSL ¹	0.9	0.0025	0.00125	0.000678
Dibromochloromethane	124-48-1	8.3	EPA Residential RSL ¹	4.0	0.0025	0.00125	0.00045
Dibromomethane	74-95-3	24	EPA Residential RSL ¹	12	0.005	0.0025	0.001
Dichlorodifluoromethane (Freon 12)	75-71-8	87	EPA Residential RSL ¹	43	0.0025	0.00125	0.000818
Ethylbenzene	100-41-4	5.8	EPA Residential RSL ¹	2.0	0.0025	0.00125	0.00053
Hexachlorobutadiene	87-68-3	1.2	HERO HHRA Note 3 ²	0.6	0.025	0.00125	0.000425
Methyl tert-Butyl ether (MTBE)	1634-04-4	47	EPA Residential RSL ¹	23	0.001	0.0005	0.000295
Methylene chloride	75-09-2	1.9	HERO HHRA Note 3 ²	1.0	0.025	0.0125	0.00664
m,p-Xylenes	179601-23-1	550	EPA Residential RSL ¹	275	0.004	0.002	0.0015
o-Xylene	95-47-6	650	EPA Residential RSL ¹	325	0.0025	0.00125	0.001
Styrene	100-42-5	6,000	EPA Residential RSL ¹	3,000	0.0125	0.00625	0.00273
Tetrachloroethene	127-18-4	0.59	HERO HHRA Note 3 ²	0.30	0.0025	0.00125	0.0007

SAP Worksheet #15.8 – Reference Limits and Evaluation Table (Continued)

Matrix: Soil (testing of imported fill material)

Analytical Group: VOCs by EPA Method 8260B

Analyte	CAS Number	Project Screening Limit ⁴ (mg/kg)	Project Screening Limit Reference	Project Quantitation Limit Goal (mg/kg)	Laboratory-specific		
					LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
Toluene	108-88-3	1,100	HERO HHRA Note 3 ²	500	0.005	0.0025	0.00125
trans-1,2-Dichloroethene	156-60-5	1,300	HERO HHRA Note 3 ²	600	0.005	0.0025	0.00143
Trichloroethene	79-01-6	0.94	EPA Residential RSL ¹	0.45	0.001	0.0005	0.0004
Trichlorofluoromethane	75-69-4	1,200	HERO HHRA Note 3 ²	600	0.0025	0.00125	0.0005
Vinyl chloride	75-01-4	0.0088	HERO HHRA Note 3 ²	0.005	0.0025	0.00125	0.0005

Notes:

¹ Values are from the EPA Region 9 Regional Screening Level for Residential Soil as presented in Regional Screening Level (RSL) Summary Table (TR=1E-6, HQ=1) May 2018.

² Values are from the Human and Ecology Risk Office (HERO) Human Health Risk Assessment (HHRA) Note 3 (DTSC, 2018).

³ TI SSC established for TI remediation projects (Shaw Environmental, Inc., 2005).

⁴ Results will be reported on a dry-weight basis for comparison to the project screening limits.

DL = detection limit

LOD = limit of detection

LOQ = limit of quantitation

mg/kg = milligram per kilogram

RSL = Regional Screening Level

SAP Worksheet #15.9 – Reference Limits and Evaluation Table

Matrix: Soil (testing of imported fill material)

Analytical Group: SVOCs by EPA Method 8270C with PAHs by 8270CSIM

Analyte	CAS Number	Project Screening Limit ⁴ (mg/kg)	Project Screening Limit Reference	Project Quantitation Limit Goal (mg/kg)	Laboratory-specific		
					LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
2,4,5-Trichlorophenol	95-95-4	6,300	EPA Residential RSL ¹	3,200	0.333	0.167	0.0104
2,4,6-Trichlorophenol	88-06-2	75	HERO HHRA Note 3 ²	35	0.333	0.167	0.00779
2,4-Dichlorophenol	120-83-2	190	EPA Residential RSL ¹	95	0.333	0.167	0.00746
2,4-Dimethylphenol	105-67-9	1,300	EPA Residential RSL ¹	650	0.333	0.167	0.047
2,4-Dinitrophenol	51-28-5	130	EPA Residential RSL ¹	65	0.333	0.167	0.098
2,4-Dinitrotoluene	121-14-2	1.7	EPA Residential RSL ¹	0.85	0.333	0.167	0.00607
2,6-Dinitrotoluene	606-20-2	0.36	EPA Residential RSL ¹	0.33	0.333	0.167	0.00737
2-Chloronaphthalene	91-58-7	4,800	EPA Residential RSL ¹	2,400	0.033	0.0167	0.00639
2-Chlorophenol	95-57-8	390	EPA Residential RSL ¹	190	0.333	0.167	0.00831
2-Methylnaphthalene	91-57-6	240	EPA Residential RSL ¹	120	0.333	0.167	0.00861
2-Methylphenol	95-48-7	3,200	EPA Residential RSL ¹	1,600	0.333	0.167	0.00986
2-Nitroaniline	88-74-4	630	EPA Residential RSL ¹	310	0.333	0.167	0.00755
3,3'-Dichlorobenzidine	91-94-1	1.2	HERO HHRA Note 3 ²	1.6 ⁵	0.333	0.167	0.0794
3/4-Methylphenol	15831-10-4	3,200	EPA Residential RSL ¹	1,600	0.333	0.167	0.00783
4-Chloroaniline	106-47-8	2.7	EPA Residential RSL ¹	1.3	0.333	0.167	0.0352
4-Nitroaniline	100-01-6	27	EPA Residential RSL ¹	13	0.333	0.167	0.00639
Acenaphthene (PAH)	83-32-9	3,600	EPA Residential RSL ¹	1800	0.006	0.003	0.0006
Anthracene (PAH)	120-12-7	18,000	EPA Residential RSL ¹	9,000	0.006	0.003	0.0006
Benzo(a)anthracene (PAH)	56-55-3	1.1	EPA Residential RSL ¹	0.5	0.006	0.003	0.0006
Benzo(a)pyrene (PAH)	50-32-8	0.11	EPA Residential RSL ¹	0.05	0.006	0.003	0.0006
Benzo(b)fluoranthene (PAH)	205-99-2	1.1	EPA Residential RSL ¹	0.5	0.006	0.003	0.0006
Benzo(k)fluoranthene (PAH)	207-08-9	11	EPA Residential RSL ¹	5.5	0.006	0.003	0.0006
Benzoic acid	65-85-0	250,000	EPA Residential RSL ¹	125,000	1.67	0.835	0.111
Benzyl alcohol	100-51-6	6,300	EPA Residential RSL ¹	3,000	0.333	0.167	0.0075

SAP Worksheet #15.9 – Reference Limits and Evaluation Table (Continued)

Matrix: Soil (testing of imported fill material)

Analytical Group: SVOCs by EPA Method 8270D with PAHs by 8270CSIM

Analyte	CAS Number	Project Screening Limit ⁴ (mg/kg)	Project Screening Limit Reference	Project Quantitation Limit Goal (mg/kg)	Laboratory-specific		
					LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
bis(2-Chloroethoxy)methane	111-91-1	190	EPA Residential RSL ¹	95	0.333	0.167	0.0077
bis(2-Chloroethyl)ether	111-44-4	0.23	EPA Residential RSL ¹	0.33 ⁵	0.333	0.167	0.00896
bis(2-Ethylhexyl)phthalate	117-81-7	39	EPA Residential RSL ¹	19	0.333	0.167	0.012
Chrysene (PAH)	218-01-9	110	EPA Residential RSL ¹	55	0.006	0.003	0.0006
Dibenz(a,h)anthracene (PAH)	53-70-3	0.11	EPA Residential RSL ¹	0.05	0.006	0.003	0.0006
Dibenzofuran	132-64-9	73	EPA Residential RSL ¹	35	0.333	0.167	0.00518
Diethylphthalate	84-66-2	51,000	EPA Residential RSL ¹	25,000	0.333	0.167	0.00691
Dimethylphthalate	131-11-3	100,000	TI SSC ³	50,000	0.333	0.167	0.0054
Di-n-butylphthalate	84-74-2	6,300	EPA Residential RSL ¹	3,200	0.333	0.167	0.0109
Di-n-octylphthalate	117-84-0	6,300	EPA Residential RSL ¹	3,200	0.333	0.167	0.00907
Fluoranthene (PAH)	206-44-0	2,400	EPA Residential RSL ¹	1,200	0.006	0.003	0.0006
Fluorene (PAH)	86-73-7	2,400	EPA Residential RSL ¹	1,200	0.006	0.003	0.0006
Hexachlorobenzene	118-74-1	0.21	EPA Residential RSL ¹	0.33 ⁵	0.333	0.167	0.00856
Hexachlorobutadiene	87-68-3	1.2	EPA Residential RSL ¹	0.6	0.333	0.167	0.01
Hexachloroethane	67-72-1	1.8	EPA Residential RSL ¹	0.9	0.333	0.167	0.0134
Indeno(1,2,3-cd)pyrene (PAH)	193-39-5	1.1	EPA Residential RSL ¹	0.5	0.006	0.003	0.0006
Isophorone	78-59-1	570	EPA Residential RSL ¹	280	0.333	0.167	0.00522
Naphthalene (PAH)	91-20-3	3.8	EPA Residential RSL ¹	1.9	0.02	0.01	0.002
Nitrobenzene	98-95-3	5.1	EPA Residential RSL ¹	2.5	0.333	0.167	0.00695
n-Nitrosodiphenylamine	86-30-6	1,100	EPA Residential RSL ¹	550	0.333	0.167	0.00594
Pentachlorophenol	87-86-5	1.0	EPA Residential RSL ¹	1.6 ⁵	0.333	0.167	0.048
Phenol	108-95-2	19,000	EPA Residential RSL ¹	9,500	0.333	0.167	0.00695
Pyrene (PAH)	129-00-0	1,800	EPA Residential RSL ¹	900	0.006	0.003	0.0006

SAP Worksheet #15.9 – Reference Limits and Evaluation Table (Continued)

Notes:

¹ Values are from the EPA Region 9 Regional Screening Level for Residential Soil as presented in Regional Screening Level (RSL) Summary Table (TR=1E-6, HQ=1) May 2018.

² Values are from the Human and Ecology Risk Office (HERO) Human Health Risk Assessment (HHRA) Note 3 (DTSC, 2018).

³ TI SSC established for TI remediation projects (Shaw Environmental, Inc., 2005).

⁴ Results will be reported on a dry-weight basis for comparison to the project screening limits.

⁵ The LOQ does not meet the PSL; however, the LOD and DL are sufficient to meet the PSL. Non-detects will be reported to the LOD.

DL= detection limit

EPA = U.S. Environmental protection Agency

LOD = limit of detection

LOQ = limit of quantitation

mg/kg = milligram per kilogram

PAH = polycyclic aromatic hydrocarbons

RSL = regional screening level

SAP Worksheet #15.10 – Reference Limits and Evaluation Table

Matrix: Soil (testing of imported fill material)

Analytical Group: PCBs by EPA Method 8082

Analyte	CAS Number	Project Screening Limit ² (mg/kg)	Project Screening Limit Reference	Project Quantitation Limit Goal (mg/kg)	Laboratory-specific		
					LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
Aroclor 1016	12674-11-2	4.1	EPA Residential RSL ¹	2.0	0.017	0.0085	0.003504
Aroclor 1221	11104-28-2	0.2	EPA Residential RSL ¹	0.1	0.017	0.0085	0.005367
Aroclor 1232	11141-16-5	0.17	EPA Residential RSL ¹	0.08	0.017	0.0085	0.004168
Aroclor 1242	53469-21-9	0.23	EPA Residential RSL ¹	0.12	0.017	0.0085	0.003176
Aroclor 1248	12672-29-6	0.23	EPA Residential RSL ¹	0.12	0.017	0.0085	0.003148
Aroclor 1254	11097-69-1	0.24	EPA Residential RSL ¹	0.12	0.017	0.0085	0.004722
Aroclor 1260	11096-82-5	0.24	EPA Residential RSL ¹	0.12	0.017	0.0085	0.004942

Notes:

¹ Values are from the EPA Region 9 Regional Screening Level for Residential Soil as presented in Regional Screening Level (RSL) Summary Table (TR=1E-6, HQ=1) May 2018.

² Results will be reported on a dry-weight basis for comparison to the project screening limits.

EPA = U.S. Environmental protection Agency

LOD = limit of detection

LOQ = limit of quantitation

mg/kg = milligram per kilogram

PCBs = polychlorinated biphenyls

SAP Worksheet #15.11 – Reference Limits and Evaluation Table

Matrix: Soil (testing of imported fill material)

Analytical Group: Pesticides by EPA Method 8081A

Analyte	CAS Number	Project Screening Limit ³ (mg/kg)	Project Screening Limit Reference	Project Quantitation Limit Goal (mg/kg)	Laboratory-specific		
					LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
4,4'-DDE	72-54-8	1.9	EPA Residential RSL ¹	0.9	0.02	0.01	0.00154
4,4'-DDD	72-55-9	2.0	EPA Residential RSL ¹	1.0	0.02	0.01	0.00156
4,4'-DDT	50-29-3	1.9	EPA Residential RSL ¹	0.9	0.02	0.01	0.002
Aldrin	309-00-2	0.039	EPA Residential RSL ¹	0.019	0.02	0.01	0.00135
alpha-BHC	319-84-6	0.086	EPA Residential RSL ¹	0.040	0.02	0.01	0.00136
beta-BHC	319-85-7	0.3	EPA Residential RSL ¹	0.015	0.02	0.01	0.0016
Chlordane (technical)	12789-03-6	1.7	EPA Residential RSL ¹	0.85	0.2	0.1	0.039
Dieldrin	60-57-1	0.034	EPA Residential RSL ¹	0.017	0.02	0.01	0.00152
Endosulfan I	959-98-8	470	EPA Residential RSL ¹ for Endosulfan	235	0.02	0.01	0.00149
Endosulfan II	33213-65-9	470	EPA Residential RSL ¹ for Endosulfan	235	0.02	0.01	0.0016
Endrin	72-20-8	19	EPA Residential RSL ¹	9	0.02	0.01	0.00157
gamma-BHC	58-89-9	0.57	EPA Residential RSL ¹	0.28	0.02	0.01	0.00145
Heptachlor	76-44-8	0.13	EPA Residential RSL ¹	0.065	0.02	0.01	0.00154
Heptachlor epoxide	1024-57-3	0.070	EPA Residential RSL ¹	0.035	0.02	0.01	0.00161
Methoxychlor	72-43-5	320	EPA Residential RSL ¹	160	0.02	0.01	0.00178
Toxaphene	8001-35-2	0.49	EPA Residential RSL ¹	0.24	0.4	0.2	0.036

Notes:

¹Values are from the EPA Region 9 Regional Screening Level for Residential Soil as presented in Regional Screening Level (RSL) Summary Table (TR=1E-6, HQ=1) May 2018.

²The LOQ does not meet the PSL; however, the LOD and DL are sufficient to meet the PSL. Non-detects will be reported to the LOD.

³Results will be reported on a dry-weight basis for comparison to the project screening limits.

DL= detection limit

EPA = U.S. Environmental Protection Agency

LOD = limit of detection

LOQ = limit of quantitation

mg/kg = milligram per kilogram

RSL = regional screening level

SAP Worksheet #15.12 – Reference Limits and Evaluation Table

Matrix: Soil (testing of imported fill material)

Analytical Group: Total Petroleum Hydrocarbons (TPH) by EPA Method 8015B

Analyte	CAS Number	Project Screening Limit ³ (mg/kg)	Project Action Limit Reference	Project Quantitation Limit Goal (mg/kg)	Laboratory-specific		
					LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
TPH as gasoline range organics (C6-C10)	-3544 ¹	100	Water Board Tier 1 ESL ²	1	0.1	0.055	0.0217
TPH as diesel range organics (C10-C28)	-3527 ¹	230	Water Board Tier 1 ESL ²	25	4	2	1.61
TPH as oil range organics (C28-C40)	-3528 ¹	5,100	Water Board Tier 1 ESL ²	25	10	5	1.07

Notes:

¹ CAS number listed is from the Navy Electronic Data Deliverable (NEDD) valid value list since a CAS number is not available for this analyte.

²Water Board Tier 1 Environmental Screening Level (ESL) based on residential land use (Feb. 2016, Rev. 3)

³Results will be reported on a dry-weight basis for comparison to the project screening limits.

DL = detection limit

LOD=limit of detection

LOQ=limit of quantitation

mg/kg = milligrams per kilogram

SAP Worksheet #15.13 – Reference Limits and Evaluation Table

Matrix: Soil (testing of imported fill material)

Analytical Group: Metals by EPA Method 6020/7471A

Analyte	CAS Number	Project Screening Limit ³ (mg/kg)	Project Screening Limit Reference	Project Quantitation Limit Goal (mg/kg)	Laboratory-specific		
					LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
Antimony	7440-36-0	31	EPA Residential RSL ¹	15	0.1	0.05	0.0316
Arsenic	7440-38-2	0.11	HERO HHRA Note 3 ²	1.0 ⁵	0.1	0.05	0.0025
Barium	7440-39-3	15,000	EPA Residential RSL ¹	7,500	0.2	0.1	0.032
Beryllium	7440-41-7	1,600	HERO HHRA Note 3 ²	800	0.1	0.05	0.012
Cadmium	7440-43-9	2,100	HERO HHRA Note 3 ²	1,000	0.1	0.05	0.016
Chromium	7440-47-3	120,000	EPA Residential RSL ¹	6,000	0.2	0.1	0.05
Cobalt	7440-48-4	23	EPA Residential RSL ¹	10	0.1	0.05	0.026
Copper	7440-50-8	3,100	EPA Residential RSL ¹	1,500	0.2	0.1	0.05
Lead	7439-92-1	80	HERO HHRA Note 3 ²	40	0.1	0.05	0.024
Mercury	7439-97-6	1	HERO HHRA Note 3 ²	0.5	0.02	0.01	0.0028
Molybdenum	7439-98-7	390	EPA Residential RSL ¹	190	0.2	0.1	0.014
Nickel	7440-02-0	1,500	HERO HHRA Note 3 ²	750	0.1	0.05	0.035
Selenium	7782-49-2	390	EPA Residential RSL ¹	190	0.1	0.05	0.038
Silver	7440-22-4	390	HERO HHRA Note 3 ²	190	0.1	0.05	0.031
Thallium	7440-28-0	0.78	EPA Residential RSL ¹	0.50	0.1	0.05	0.019
Vanadium	7440-62-2	390	HERO HHRA Note 3 ²	190	0.2	0.1	0.018
Zinc	7440-66-6	23,000	EPA Residential RSL ¹	11,000	1	0.5	0.256

Notes:

¹ Values are from the EPA Region 9 Regional Screening Level for Residential Soil as presented in Regional Screening Level (RSL) Summary Table (TR=1E-6, HQ=1) May 2018.

² Values are from the Human and Ecology Risk Office (HERO) Human Health Risk Assessment (HHRA) Note 3 (DTSC, 2018).

³ Results will be reported on a dry-weight basis for comparison to the project screening limits.

⁵ The LOQ does not meet the PSL; however, the method represents the best available technology. Non-detects will be reported to the LOD and uncertainty in any non-detects will be addressed in the QCSR.

DL = detection limit

EPA = U.S. Environmental Protection Agency

LOD = limit of detection

LOQ = limit of quantitation

mg/kg = milligrams per kilogram

SAP Worksheet #15.14 – Reference Limits and Evaluation Table
(UFP-QAPP Manual Section 2.8.1)

Matrix: Soil (testing of imported fill material)

Analytical Group: Asbestos by CARB 435 or equivalent

Analyte	CAS Number	Project Screening Limit (%)	Project Screening Limit Reference	Project Quantitation Limit Goal (%)	Laboratory-specific	
					QL (%)	MDL
Asbestos	132207-33-1	0.25	Bay Area Air Quality Management District	0.25	0.25	1 fiber

Notes:

QL = quantitation limit

MDL = method detection limit

SAP Worksheet #15.15 – Reference Limits and Evaluation Table
(UFP-QAPP Manual Section 2.8.1)

Matrix: Soil (testing of imported fill material)

Analytical Group: pH by EPA Method 9045C

Analyte	CAS Number	Project Screening Limit (pH Units)	Project Screening Limit Reference	Project Quantitation Limit Goal (pH Units)	Laboratory-specific		
					LOQ (pH Units)	LOD (pH Units)	DL (pH Units)
pH	-9 ¹	6.5 < pH < 8.5	Water Board ²	0.1	0.1	NA	NA

Notes:

¹ CAS number listed is from the Navy Electronic Data Deliverable (NEDD) valid value list since a CAS number is not available for this analyte.

²Remediation goal from the *San Francisco Bay (Region 2) Water Quality Control Plan (Basin Plan)* (Water Board, 2011).

Water Board = San Francisco Bay Water Quality Control Board

SAP Worksheet #16 – Project Schedule /Timeline Table
(UFP-QAPP Manual Section 2.8.2)

The project schedule is included as Figure 3 of this SAP.

SAP Worksheet #17 – Sampling Design and Rationale

The following sections describe the sampling designed to meet the project objectives.

17.1 Excavation of Contaminated Soils

Excavation will be conducted per the Work Plan (Gilbane, 2018) to which this SAP is an appendix. Slot trenching in a step-out fashion will be used to remove debris and stained soil across the excavation area. Excavation activities in both northern and southern excavation areas will begin at the point of highest contamination or concern based on past characterization data from previous removal actions. This information will be provided by the Navy. Explosive hazard precautions will be implemented in accordance with the ESS (see Appendix A of the Work Plan).

17.1.1 Chemical and Radiological Characterization Sampling

Soil samples will be collected to characterize the as-left chemical and radiological conditions at the lateral and vertical extent of the debris/soil staining removal. The vertical extent will be sampled at a frequency of one sample per 2,500 square ft. In other words, one sample will be collected along the bottom of one or more trenches whose total floor surface area equals 2,500 square ft. In addition, the lateral extent will be sampled at a frequency of one sample per 50 linear ft. In other words, one sample will be collected along the sidewall of one or more trenches that form the lateral excavation boundary whose total length equals 50 linear ft. If the excavation depth is less than 7 ft, one discrete soil sample will be collected along the sidewall. If the excavation depth is equal to or greater than 7 ft, then two discrete soil samples will be collected along the sidewall – one at less than 7 ft deep and one at more than 7 ft deep. Samples will be collected from the backhoe/excavator bucket in accordance with SAP Worksheet # 14. After sample collection, spoils will be returned to the excavation.

All characterization samples will be sent to an off-site DoD and California Environmental Laboratory Accreditation Program (ELAP)-certified laboratory for analysis. Characterization samples will be analyzed for the following: (1) PAHs by EPA Method 8270CSIM, (2) Dioxin/Furans by EPA 8290, (3) VOCs by EPA 8260B, (4) PCBs by EPA 8082, and (5) lead by EPA 6010B. The samples will also be analyzed for Ra-226 by DOE Health and Safety Laboratory (HASL) 300 Method GA-01-R.

17.2 Benzo(a)pyrene and Dioxin Toxicity Equivalency Calculation

PAHs will be reported as the BAP(eq) in the after-action report for consistency with historical site data. The BAP(eq) will be calculated by summing concentrations of carcinogenic PAH compounds after multiplying each concentration by their potency equivalence factors (PEFs). The PEFs used for this Project are from California Office of Environmental Health Hazard Assessment *Air Toxics Hot Spots Program Risk Assessment Guidelines* (2003).

The PEFs for the carcinogenic PAHs are as follows:

- Benzo(a)pyrene 1.0

SAP Worksheet #17 – Sampling Design and Rationale (Continued)

- Benzo(a)anthracene 0.1
- Benzo(b)fluoranthene 0.1
- Benzo(k)fluoranthene 0.1
- Chrysene 0.01
- Dibenz(a,h)anthracene 1.0
- Indeno(1,2,3-cd)pyrene 0.1

Two BAP(eq) values are calculated: BAPeq(0) is calculated using only the detected values of all seven analytes and BAPeq($\frac{1}{2}$) is calculated using one-half of LOD values for analytes that are not detected.

Dioxins/furans will be reported as the 2,3,7,8-TCDD TEQ for consistency with historical site data. The dioxin toxicity equivalency factors (TEFs) will be calculated in accordance with Gilbane Work Instruction *Calculating Toxic Equivalence (TEQ) for Dioxins, Furans and Dioxin-Like Compounds* (SAP Attachment 1). The Dioxin-TEF will be calculated by summing concentrations of carcinogenic chlorinated dioxin and furan compounds after multiplying each concentration by their toxic equivalence factor. The TEFs used for this project are from California Office of Environmental Health Hazard Assessment *Air Toxics Hot Spots Program Risk Assessment Guidelines* (2015) using the 2005 World Health Organization TEFs. The WHO/05 TEFs for the dioxins and furans are listed in the Gilbane Dioxin TEQ Work Instruction included in Attachment 1.

17.3 Import Material Sampling

All excavations will be backfilled with imported soil that meets the project's acceptance criteria. Imported fill material testing will include collection of samples for analysis of the site-specific chemicals of concern, and other contaminants based on the nature of the fill source at the frequency presented in the *Information Advisory Clean Imported Fill Material* (DTSC, 2001). The imported fill material acceptance criteria for the project are presented in Worksheets #15-7 through #15-15.

SAP Worksheet #18 – Sampling Locations and Methods/SOP Requirements Table

Sampling Location/ID Number ³	Matrix	Depth (feet bgs)	Analytical Group	Estimated Number of Samples	Sampling SOP Reference
SWDA Excavation Area 1 Characterization Sampling					
WS1-SW-001 through -010	Soil	(Sidewalls) TBD ¹	PAHs, PCBs, VOCs, Pb, Dioxin/furans, and Ra-226	10	SAP Worksheet #17
WS1-BS-001 through -003	Soil	(Bottom) TBD ¹	PAHs, PCBs, VOCs, Pb, Dioxin/furans, and Ra-226	3	SAP Worksheet #17
SWDA Excavation Area 2 Characterization Sampling					
WS2-SW-001 through -012	Soil	(Sidewalls) TBD ¹	PAHs, PCBs, VOCs, Pb, Dioxin/furans, and Ra-226	12	SAP Worksheet #17
WS2-BS-001 through -007	Soil	(Bottom) TBD ¹	PAHs, PCBs, VOCs, Pb, Dioxin/furans, and Ra-226	7	SAP Worksheet #17
SWDA Excavation Area 3 Characterization Sampling					
WS3-SW-001 through -010	Soil	(Sidewalls) TBD ¹	PAHs, PCBs, VOCs, Pb, Dioxin/furans, and Ra-226	10	SAP Worksheet #17
WS3-BS-001 through -003	Soil	(Bottom) TBD ¹	PAHs, PCBs, VOCs, Pb, Dioxin/furans, and Ra-226	3	SAP Worksheet #17
SWDA Excavation Area 4 Characterization Sampling					
WS4-SW-001 through -008	Soil	(Sidewalls) TBD ¹	PAHs, PCBs, VOCs, Pb, Dioxin/furans, and Ra-226	8	SAP Worksheet #17
WS4-BS-001 through -003	Soil	(Bottom) TBD ¹	PAHs, PCBs, VOCs, Pb, Dioxin/furans, and Ra-226	3	SAP Worksheet #17
Import Material					
IF-YYY-ZZZ, where YYY indicates the source number, and ZZZ is the sequential sample number per WS # 27	Soil	Not Applicable	VOCs, SVOCS, PAHs, PCBs, Pesticides, Metals, TPH, Asbestos, pH, Ra-226	91 estimated	SAP Worksheet #17

Notes:

¹TBD = To be determined. The exact depths of the excavations will be determined by field conditions and professional judgment of the project team, but will not exceed 15 feet bgs.

SAP Worksheet #19 – Analytical SOP Requirements Table

Matrix	Analytical Group	Analytical and Preparation Method // SOP Reference	Containers (number, size, and type)	Sample volume ¹ (units)	Preservation Requirements ² (chemical, temperature, light protected)	Maximum Holding Time (preparation / analysis)
Soil	PAHs	EPA 8270C SIM/3550B// 330345	1 X 4-oz Jar	30 g	<6°C	14 days/40 days
Soil	Dioxin/Furans	EPA 8290// BRL SOP-00406	1 X 4-oz Jar	30 g	<6°C	30 days/45 days
Soil	Pesticide	EPA 8081A/3550B// 330344	1 X 4-oz Jar	30 g	<6°C	14 days/40 days
Soil	PCBs	EPA 8082/3550B// 330343	1 X 4-oz Jar	30 g	<6°C	None
Soil	Pb	EPA 6010B/3050B// 340386	1 x 2-oz Jar	2 g	None	180 days ³
Soil	Metals	EPA 6020/3050B// 340390	1 X 2-oz Jar	2 g	None	180 days ³
Soil	Mercury	EPA 7471A// 340384B			<6°C	28 days ³
Soil	VOCs	EPA 8260B/5035// ENV-SOP-MTJL-0100	3 X 5-g TerraCore™ Samplers ^{4,5,7} or equivalent	5 grams (g)	<6°C	48 hours/14 days
Soil	SVOCs	EPA 8270C/3550B// 330345	1 X 4-oz Jar	30 g	<6°C	14 days/40 days
Soil	TPH-purgeable	EPA 8015B/5035// ENV-SOP-MTJL-0087	3 X 5-g TerraCore™ Samplers ^{4,5} or equivalent	5 g	<6°C	14 days
Soil	TPH-extractable	EPA 8015B/3550B// ENV-SOP-MTJL-0089	1 X 4-oz Jar	30 g	<6°C	14 days/40 days
Soil	Asbestos	CARB 435 or equivalent	1 X 8-oz Jar or plastic baggie	8 ounce (oz.)	None	Not Applicable

SAP Worksheet #19 – Analytical SOP Requirements Table (Continued)

Matrix	Analytical Group	Analytical and Preparation Method // SOP Reference	Containers (number, size, and type)	Sample volume ¹ (units)	Preservation Requirements ² (chemical, temperature, light protected)	Maximum Holding Time (preparation / analysis)
Soil	pH	EPA 9045C// 340335	1 X 2-oz Jar	10 g	<6°C	7 days
Soil	Gamma Spectroscopy	HASL 300 GA-01-R//RAD_04-13	Gallon Ziploc bag	~1,000g (bag)	None	None

Notes:

¹ Minimum sample volume or mass requirement if different from the container volume.

²Temperature compliance will be measured using temperature blanks included in the coolers used to ship the samples to the laboratory.

³ The time listed is the maximum holding time for the analysis. Preparation time is included in the analytical method holding time.

⁴ If TerraCore samplers cannot be used due to saturated soil, then only the 8-ounce jar (which will be filled without headspace) will be used, and VOC analysis will be conducted from the jar sample.

⁵TerraCore samples will be immediately transferred to suitable vials for transportation to laboratory per SOP PR-TC-02.02.01.05.

⁶Percent moisture will only be collected in a separate container when samples are to be analyzed for VOCs or TPH-purgeable only.

⁷Terracore kits for VOCs will consist of two unpreserved and one methanol-preserved vial because acid preservation may cause the chemical breakdown of certain reactive VOC compounds in the soil sample, specifically styrene, acrylonitrile, vinyl chloride, and 2-chloroethylvinyl ether (SOP PR-TC-02.02.01.05).

SAP Worksheet #20 – Field Quality Control Sample Summary Table

Matrix	Analytical Group	No. of Sampling Locations ¹	No. of MS/MSDs	No. of Equipment Blanks	Total No. of Samples to Lab ³
Soil (characterization)	PAHs, PCBs, VOCs, Pb, Dioxin/furans, and Ra-226	56	3	None or 1 per/day ²	56
Soil Imported Fill Material	VOCs, SVOCs, PAHs, PCBs, Pesticides, Metals, TPH, pH, Ra-226	91	2	None or 1 per/day ²	TBD
Soil Imported Fill Material	Asbestos	91	None	None	TBD

Notes:

¹Actual sampling location (and thus the number of total samples to the laboratory) will be determined in the field based on the amount of excavated soil and the number of imported fill material sources sampled.

²None if disposable sampling equipment used; 1 per day if using non-disposable sampling equipment

³The total number shipped to lab does not include MS/MSDs.

PAH = polycyclic aromatic hydrocarbons

PCB = polychlorinated biphenyl

PT = performance testing

Ra-226 = radium-226

TPH = total petroleum hydrocarbons

SVOC = semivolatile organic compound

VOA = volatile organic analysis

VOC = volatile organic compound

SAP Worksheet #21 – Project Sampling SOP References Table

Reference Number	Title, Revision Date and / or Number	Originating Organization	Equipment Type	Modified for Project Work? (Y/N)	Comments
PR-TC-01.04.04.00	Field Documentation	Gilbane	Not Applicable	N	Attachment 1 of SAP
PR-TC-02.12.02.00	Sample Tracking and Electronic Data Management	Gilbane	Not Applicable	N	Attachment 1 of SAP
PR-TC-02.04.01.01	Sample Handling, Packaging and Shipping	Gilbane	Not Applicable	N	Attachment 1 of SAP
PR-TC-04.01.00.00	Review, Verification, and Validation of Chemical Data	Gilbane	Not Applicable	N	Attachment 1 of SAP
PR-TC-04.01.02.00	Review, Verification, and Validation of Radiological Data	Gilbane	Not Applicable	N	Attachment 1 of SAP
PR-TC-02.02.01.05	Volatile Organic Compound (VOC) Sampling	Gilbane	Per EPA 5035	N	Attachment 1 of SAP
PR-TC-02.02.01.01	Surface Soil: Sampling with Trowel or Spoon	Gilbane	Various	N	Attachment 1 of SAP
Work Instruction	Calculating Toxic Equivalence (TEQ) for Dioxins, Furans and Dioxin-Like Compounds	Gilbane	Not Applicable	N	Attachment 1 of SAP

SAP Worksheet #22 – Field Equipment Calibration, Maintenance, Testing, and Inspection Table

Field Equipment	Activity	Frequency	Acceptance Criteria	Corrective Action	Resp. Person	SOP Reference	Comments
Photoionization Detector (PID)	<ul style="list-style-type: none"> • Calibration Check • Daily cleaning during field use • Proper storage when not in use 	Daily (prior to field use)	Within 10% of the calibration gas concentration true value	Recalibrate instrument, if still out, return instrument	Superintendent	(See Comments column)	Calibration procedure stated in the instrument manufacturer's operational instructions will be followed. Inoperable equipment will be removed from use and replaced.
Global Positioning System (GPS)	<ul style="list-style-type: none"> • No calibration Required • Charge batteries • Clean of dust, dirt, and grease • Store instrument in case when not in use 	Daily (prior to field use)	± 10 mm horizontally and 15 mm vertically	If the instrument can not connect to satellites, then the secondary unit will be used to verify that there are no connections	Superintendent	(See Comments column)	Calibration procedure stated in the instrument manufacturer's operational instructions will be followed. Inoperable equipment will be removed from use and replaced.
Sodium Iodide Detector	Calibration Check	Daily (prior to field use)	Within 10% of the calculated activity isotope	Follow procedure as outlined in the manufacturer's instruction manual or contact vendor for technical support.	Radiological Technician	(See Comments column)	Calibration procedure stated in the instrument manufacturer's operational instructions will be followed. Inoperable equipment will be removed from use and replaced.

SAP Worksheet #23 – Analytical SOP References Table

Lab SOP Number ¹	Title, Revision Date, and / or Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Variance from DoD QSM? (Y/N)
ENV-SOP-MTJL-0089	Method for Determination of Extractable Petroleum Hydrocarbons BY GC/FID With 1:1 Diesel/Motor Oil - Modified 8015B, 8015C & 8015D with provisions for: AZ, CA, IN (ERO), ND (TEH/TEM - client specific), OH, TN, and Waste Oil Analysis, 1/25/2019, Revision 02	Definitive	Soil /TPH-extractable	Gas Chromatograph (GC)	Pace National	N
ENV-SOP-MTJL-0087	BTEX (Method 8021B, 602, SM6200C 2th) and Gasoline Range Organics (Method 8015B, 8015C, 8015D) by GC (with provisions for Calif-Lo, NWTPH-Gx, OA1, WI GRO (synthetic), Wyoming LAUST Req., GRO Louisiana, AK101 GRO), 1/1/2019, Revision 00	Definitive	Soil /TPH-purgeable	GC	Pace National	N
330343	Polychlorinated Biphenyls (PCBs), 3/23/18, Revision 17	Definitive	Soil /PCBs	GC	Pace National	N
330344	Pesticides, 7/17/18, Revision 21	Definitive	Soil/Pesticides	GC	Pace National	N
330345	Semi-Volatile Organic Compounds by GC/MS, 3/22/18, Revision 26	Definitive	Soil/SVOCs & PAHs	GC/Mass Spectrometer (MS)	Pace National	N

SAP Worksheet #23 – Analytical SOP References Table (Continued)

Lab SOP Number	Title, Revision Date, and / or Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Variance from DoD QSM? (Y/N)
ENV-SOP-MTJL-0100	Volatile Organic Compounds by GC/MS (EPA 8260B, 8260C, 624, 624.1 and SM 6200B), 2/14/2019, Revision 01	Definitive	Soil/VOCs	GC/MS	Pace National	N
340390	Metals by ICP/MS, 7/27/18, Revision 19	Definitive	Soil /ICPMS Metals	ICP/Mass Spectrometer (MS)	Pace National	N
340386	Metals by ICP/AES, 9/10/18, Revision 21	Definitive	Soil/ICP Metals	Inductively Coupled Plasma (ICP) Spectrophotometer	Pace National	N
340384B	Mercury in Soil Samples, 7/27/18, Revision 13	Definitive	Soil/Mercury	Cold Vapor Atomic Absorption (CVAA)	Pace National	N

SAP Worksheet #23 – Analytical SOP References Table (Continued)

Lab SOP Number ¹	Title, Revision Date, and / or Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Variance from DoD QSM? (Y/N)
ENV-SOP-MIN4-0026	Preparation and Analysis of Samples for the Determination of Dioxins and Furans by USEPA Method 8290/8290A/1613B (Revision 01)	Definitive	Soil/Dioxins/Furans	High Resolution (HR) GC/HRMS	Pace Analytical Minnesota	N
340335	pH, 4/9/18, Revision 16	Definitive	Soil/pH	Probe	Pace National	N
RAD_04-12	Gamma Spec, Rev 13, 10/19/18	Definitive	Soil/Gamma Spectroscopy	Gamma Spectrometer	Pace National	N
SOP 109	Asbestos Bulk Sample Analysis, PLM, Version 12, Effective: 6/30/17	Definitive	Soil/Asbestos	Polarized Light Microscopy (PLM)	A&B Labs	N

Notes:

¹Laboratory SOPs are provided as Attachment 3 to this SAP.

SAP Worksheet #24- Analytical Instrument Calibration Table

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference
Gamma Spectrometer	Initial Calibration Verification (ICAL) for Energy, Efficiency, and FWHM peak resolution	Prior to initial use, following repair or loss of control and upon incorporation of new or changed instrument settings	The energy difference should be within 0.05% for all calibration points or within 0.2 keV. Peak energy difference is within 0.1 keV of reference energy for all points. Peak Full width at Half Maximum (FWHM) < 2.5 keV at 1332 keV. The efficiency difference should be within 8% of the true value for each point unless T.C.C calibration is performed.	Correct problem, then repeat ICAL.	Analyst and/or Supervisor	RAD_04-12
Gamma Spectrometer	Initial Calibration Verification (ICV)	After ICAL for energy/efficiency and prior to analysis of samples.	Observed peaks of second source standard fall within $\pm 10\%$ of initial calibration value relative to the true value.	Verify second source standard and repeat ICV to check for errors. If that fails, identify and correct problem and repeat ICV or ICAL and ICV as appropriate.	Analyst and/or Supervisor	RAD_04-12
Gamma Spectrometer	Continuing Calibration Verification (CCV) (Daily Check)	Daily or prior to use. When working with long count times or batch sequences that run more than a day, CCV is performed at the beginning and end of each analytical batch; but no less than once per week.	Energy: ± 0.5 keV at 60 keV; $\pm .75$ keV at 1332 keV. FW HM: $\pm 1.2x$ at 60 keV; $\pm 1.8x$ at 662 keV; $\pm 2.3x$ at 1332 keV. Activity Difference: %difference between the source activity and the reported activity $\pm 5\%$	Correct problem, rerun CCV. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification.	Analyst and/or Supervisor	RAD_04-12

SAP Worksheet #24- Analytical Instrument Calibration Table (Continued)

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference
Gamma Spectrometer	Background Subtraction Count Measurement (BSC) (Long count for subtracting background from blanks or test sources)	Immediately after ICAL and then performed on at least a monthly basis.	Background count rate of the entire spectrum with $\pm 3\sigma$ of the average.	Recount and check control chart for trends. Determine cause, correct problem, re-establish BSC. If background activity has changed, re-establish BSC and reanalyze or qualify all impacted samples since last acceptable BSC.	Analyst and/or Supervisor	RAD_04-12
Gamma Spectrometer	Instrument Contamination Check (ICC) (Short count for controlling gross contamination)	Daily or when working with long count times before and after each analytical batch. Check after counting high activity samples.	No extraneous peaks identified (i.e., no new peaks in the short background spectrum compared to previous spectra); background count rate of the entire spectrum with $\pm 3\sigma$ of the average.	Recount the background. If still out of control, locate and correct problem; reanalyze or qualify all impacted samples since last acceptable ICC. If background activity has changed, re-establish BSC and reanalyze samples.	Analyst and/or Supervisor	RAD_04-12
HCGC/HRMS	Resolving Power	Prior to ICAL and at the beginning and the end of each 12-hour period of analysis.	Static resolving power $\geq 10,000$ (10% valley) for identified masses.	Retune instrument and verify. Rerun affected samples.	Analyst and/or Supervisor	BRL SOP-00406

SAP Worksheet #24- Analytical Instrument Calibration Table (Continued)

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference
HRGC/HRMS	Calibration Verification (CCV)	At the beginning of each 12-hour period, and at the end of each analytical sequence.	<p>Ion abundance specified in the method must be met.</p> <p>For unlabeled standards, RF within $\pm 20\%$ D of RF established in ICAL; <u>and</u></p> <p>For labeled standards, RF within $\pm 30\%$ D of RF established in ICAL.</p>	<p>Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails or if two consecutive CCVs cannot be run, perform corrective action(s) and repeat CCV and all associated samples since last successful CCV.</p> <p>Alternately, recalibrate if necessary; then reanalyze all associated samples since the last acceptable CCV</p> <p><u>End-of-run CCV</u>: If the RF for unlabeled standards $\leq 25\%$ RPD and the RF for labeled standards $\leq 35\%$ RPD (relative to the RF established in the ICAL), the mean RF from the two daily CCVs must be used for quantitation of impacted samples instead of the ICAL mean RF value. If the starting and ending CCV RFs differ by more than 25% RPD for unlabeled compounds or 35% RPD for labeled compounds, the sample may be quantitated against a new initial calibration if it is analyzed within two hours.</p> <p>Otherwise analyze samples with positive detections, if necessary.</p>	Analyst and/or Supervisor	BRL SOP-00406
HRGC/HRMS	Sample Estimated Maximum Possible Concentration (EMPC)	Every sample with a response $S/N \geq 2.5$ for both quantitation ions	Identification criteria per method must be met, and the S/N of response for both quantitation ions must be ≥ 2.5	NA	Analyst and/or Supervisor	BRL SOP-00406

SAP Worksheet #24- Analytical Instrument Calibration Table (Continued)

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference
GC/MS	Tune Check	Prior to ICAL and prior to each 12-hour period of sample analysis	Specific ion abundance criteria of BFB or DFTPP from method.	Retune instrument and verify.	Analyst and/or Supervisor	SOPs 330345 & ENV-SOP-MTJL-0100
GC/MS	Performance Check (Method 8270 only)	At the beginning of each 12-hour period, prior to analysis of samples.	Degradation $\leq 20\%$ for DDT. Benzidine and pentachlorophenol shall be present at their normal responses and shall not exceed a tailing factor of 2.	Correct problem, then repeat performance checks.	Analyst and/or Supervisor	SOPs 330345 & ENV-SOP-MTJL-0100
GC/MS	Initial Calibration (ICAL) – Minimum 5 levels for linear and 6 levels for quadratic.	Initial calibration prior to sample analysis for all analytes including surrogates	Each analyte must meet one of the three options below: <u>Option 1:</u> RSD for each analyte $\leq 15\%$; <u>Option 2:</u> linear least squares regression for each analyte: $r^2 \geq 0.99$; <u>Option 3:</u> non-linear least squares regression (quadratic) for each analyte: $r^2 \geq 0.99$.	Repeat calibration	Analyst and/or Supervisor	SOPs 330345 & ENV-SOP-MTJL-0100
GC/MS	Initial Calibration Verification (ICV)	Once after each initial calibration, analysis of a second source standard prior to sample analysis.	All reported analytes within $\pm 20\%$ of true value	Rerun initial calibration verification (ICV) one time, second failure requires recalibration.	Analyst and/or Supervisor	SOPs 330345 & ENV-SOP-MTJL-0100

SAP Worksheet #24- Analytical Instrument Calibration Table (Continued)

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference
GC/MS	Continuing Calibration Verification (CCV)	Daily before sample analysis; after every 12 hours of analysis time; and at the end of the analytical batch run.	All reported analytes and surrogates within $\pm 20\%$ of true value. All reported analytes and surrogates within $\pm 50\%$ for end of analytical batch CCV.	Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails or if two consecutive CCVs cannot be run, perform corrective action(s) and repeat CCV and all associated samples since last successful CCV. Alternately, recalibrate if necessary; then reanalyze all associated samples since the last acceptable CCV.	Analyst and/or Supervisor	SOPs 330345 & ENV-SOP-MTJL-0100
GC/MS	Retention Time window position establishment	Once per ICAL and at the beginning of the analytical sequence	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	N/A	Analyst and/or Supervisor	SOPs 330345 & ENV-SOP-MTJL-0100
GC/MS	Evaluation of Relative Retention Times (RRT)	With each sample.	RRT of each reported analyte within ± 0.06 RRT units.	Correct problem, then rerun ICAL.	Analyst and/or Supervisor	SOPs 330345 & ENV-SOP-MTJL-0100

SAP Worksheet #24- Analytical Instrument Calibration Table (Continued)

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference
GC	Breakdown check (Endrin/DDT Method 8081 only)	Before sample analysis and at the beginning of each 12-hour shift.	Degradation of DDT and Endrin must each be $\leq 15\%$.	Correct problem, then repeat breakdown checks.	Analyst and/or Supervisor	SOP 330344
GC	Initial Calibration (ICAL) Minimum 5 levels for linear and 6 levels for quadratic. Quantitation for multicomponent analytes such as chlordane, toxaphene, and Aroclors must be performed using a 5-point ICAL. Results may not be quantitated using a single point.	Initial calibration prior to sample analysis	ICAL must meet one of the three options below: <u>Option 1</u> : RSD for each analyte $\leq 20\%$; <u>Option 2</u> : linear least squares regression for each analyte: $r^2 \geq 0.99$; <u>Option 3</u> : non-linear least squares regression (quadratic) for each analyte: $r^2 \geq 0.99$.	Correct problem then repeat ICAL	Analyst and/or Supervisor	SOPs 330343, 330344, ENV-SOP-MTJL-0087, & ENV-SOP-MTJL-0089
GC	Retention Time window position establishment	Once per ICAL and at the beginning of the analytical sequence	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	N/A	Analyst and/or Supervisor	SOPs 330343, 330344, ENV-SOP-MTJL-0087, & ENV-SOP-MTJL-0089

SAP Worksheet #24- Analytical Instrument Calibration Table (Continued)

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference
GC	Retention Time (RT) window width	At method set-up and after major maintenance (e.g. column change)	RT width is ± 3 times standard deviation for each analyte RT from the 72-hour study or 0.03 minutes, whichever is greater.	N/A	Analyst and/or Supervisor	SOPs 330343, 330344, ENV-SOP-MTJL-0087, & ENV-SOP-MTJL-0089
GC	Initial Calibration Verification (ICV)	Once after each initial calibration, analysis of a second source standard prior to sample analysis.	All reported analytes within established RT windows. All reported analytes within $\pm 20\%$ of true value	Rerun initial calibration verification (ICV) one time, second failure requires recalibration	Analyst and/or Supervisor	SOPs 330343, 330344, ENV-SOP-MTJL-0087, & ENV-SOP-MTJL-0089
GC	Continuing Calibration Verification (CCV)	Before sample analysis, after every 10 field samples, and at the end of the analysis sequence, except for CCVs for Pesticide multicomponent analytes (i.e., Toxaphene, Chlordane and Aroclors other than 1016 and 1260), which are only required before sample analysis.	All reported analytes within established RT windows. All reported analytes within $\pm 20\%$ of true value	Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, or if two consecutive CCVs cannot be run, perform corrective action(s) and repeat CCV and all associated samples since last successful CCV. Alternately, recalibrate if necessary; then reanalyze all associated samples since the last acceptable CCV.	Analyst and/or Supervisor	SOPs 330343, 330344, ENV-SOP-MTJL-0087, & ENV-SOP-MTJL-0089

SAP Worksheet #24- Analytical Instrument Calibration Table (Continued)

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference
GC	Confirmation of positive results (second column)	All results > the DL must be confirmed (except for single column methods such as TPH by Method 8015 where confirmation is not an option or a requirement).	Calibration and QC criteria for second column are the same as for initial or primary column analysis. Results between primary and secondary column RPD $\leq 40\%$.	Evaluate data, then report the higher result with a flag to denote RPD > 40%. Narrate obvious matrix issues.	Analyst and/or Supervisor	SOPs 330343, 330344, ENV-SOP-MTJL-0087, & ENV-SOP-MTJL-0089
ICP/AES	Linear Dynamic Range (LDR) or high-level check standard	At initial set up and checked every 6 months high a high standard at the upper limit of the range	Within $\pm 10\%$ of true value	Dilute samples within the calibration range, or re-establish/verify the LDR	Analyst and/or Supervisor	SOP 340386
ICP/AES	Initial Calibration (ICAL) – minimum one high standard and a calibration blank	Daily initial calibration prior to sample analysis	If more than one calibration standard is used, $r^2 \geq 0.99$.	Recalibrate	Analyst and/or Supervisor	SOP 340386
ICP/AES	Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All reported analytes within $\pm 10\%$ of true value.	Correct problem. Rerun ICV. If that fails, repeat ICAL.	Analyst and/or Supervisor	SOP 340386

SAP Worksheet #24- Analytical Instrument Calibration Table (Continued)

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference
ICP/AES	Continuing Calibration Verification (CCV)	After every 10 samples and at the end of the analysis sequence	All analytes within $\pm 10\%$ of expected value	<p>Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails or if two consecutive CCVs cannot be run, perform corrective action(s) and repeat CCV and all associated samples since last successful CCV.</p> <p>Alternately, recalibrate if necessary; then reanalyze all associated samples since the last acceptable CCV.</p>	Analyst and/or Supervisor	SOP 340386
ICP/AES	Low-level Calibration Check Standard \leq LOQ	Daily	All analytes within $\pm 20\%$ of true value	Correct problem and repeat ICAL	Analyst and/or Supervisor	SOP 340386

SAP Worksheet #24- Analytical Instrument Calibration Table (Continued)

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference
ICP/AES	Calibration Blanks (ICB/CCB)	Immediately after the ICV and immediately after every CCV.	The absolute values of all analytes must be $< \frac{1}{2}$ LOQ or $< 1/10$ th the amount measured in any sample.	ICB: Correct problem and repeat ICV/ICB analysis. If that fails, rerun ICAL. All samples following the last acceptable Calibration Blank must be reanalyzed. CCBs may not be reanalyzed without reanalysis of the associated samples and CCV(s).	Analyst and/or Supervisor	SOP 340386
ICP/AES	Interference Check Solutions (ICS)	After ICAL and prior to sample analysis	ICS-A: Absolute value of concentration for all non-spiked project analytes $< 1/2$ LOQ (unless they are a verified trace impurity from one of the spiked analytes); ICS-AB: Within $\pm 20\%$ of true value.	Terminate analysis; locate and correct problem; reanalyze ICS, reanalyze all samples	Analyst and/or Supervisor	SOP 340386
ICP-MS	Linear Dynamic Range (LDR) or high-level check standard	At initial set-up and checked every 6 months with a high standard at the upper limit of the range.	Within $\pm 10\%$ of true value	Dilute samples within the calibration range, or re-establish/verify the LDR	Analyst and/or Supervisor	SOP 340390
ICP-MS	Tuning	Prior to ICAL	Mass calibration < 0.1 atomic mass unit (amu) from the true value; resolution < 0.9 amu full width at 10% peak height	Retune instrument and verify	Analyst and/or Supervisor	SOP 340390

SAP Worksheet #24- Analytical Instrument Calibration Table (Continued)

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference
ICP-MS	Initial Calibration (ICAL) – minimum one high standard and a calibration blank	Daily initial calibration prior to sample analysis	If more than one calibration standard is used, $r^2 \geq 0.99$	Recalibrate	Analyst and/or Supervisor	SOP 340390
ICP-MS	Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All reported analytes within $\pm 10\%$ of true value.	Correct problem. Rerun ICV. If that fails, repeat ICAL.	Analyst and/or Supervisor	SOP 340390
ICP-MS	Continuing Calibration Verification (CCV)	After every 10 samples and at the end of the analysis sequence	All reported analytes within $\pm 10\%$ of true value.	Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails or if two consecutive CCVs cannot be run, perform corrective action(s) and repeat CCV and all associated samples since last successful CCV. Alternately, recalibrate if necessary; then reanalyze all associated samples since the last acceptable CCV.	Analyst and/or Supervisor	SOP 340390
ICP-MS	Low-level Calibration Check Standard \leq LOQ	Daily	All reported analytes within $\pm 20\%$ of the true value.	Correct problem and repeat ICAL	Analyst and/or Supervisor	SOP 340390

SAP Worksheet #24- Analytical Instrument Calibration Table (Continued)

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference
ICP-MS	Calibration Blanks (ICB/CCB)	Immediately after the ICV and immediately after every CCV.	The absolute values of all analytes must be $< \frac{1}{2}$ LOQ or $< 1/10$ th the amount measured in any sample.	ICB: Correct problem and repeat ICV/ICB analysis. If that fails, rerun ICAL. All samples following the last acceptable Calibration Blank must be reanalyzed. CCBs may not be reanalyzed without reanalysis of the associated samples and CCV(s).	Analyst and/or Supervisor	SOP 340390
ICP-MS	Interference Check Solutions (ICS)	After ICAL and prior to sample analysis	ICS-A: Absolute value of concentration for all non-spiked project analytes $< 1/2$ LOQ (unless they are a verified trace impurity from one of the spiked analytes); ICS-AB: Within $\pm 20\%$ of true value.	Terminate analysis; locate and correct problem; reanalyze ICS, reanalyze all samples	Analyst and/or Supervisor	SOP 340390
Cold Vapor AA	Initial Calibration (ICAL) – Minimum of 5 standards	Daily initial calibration prior to sample analysis	$R^2 \geq 0.99$	Correct problem, then recalibrate.	Analyst and/or Supervisor	SOP 340384A & 340384B
Cold Vapor AA	Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All reported analytes within $\pm 10\%$ of the true value.	Correct problem. Rerun ICV. If that fails, rerun ICAL.	Analyst and/or Supervisor	SOP 340384A & 340384B

SAP Worksheet #24- Analytical Instrument Calibration Table (Continued)

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference
Cold Vapor AA	Low-level Calibration Check Standard \leq LOQ	Daily	All reported analytes within $\pm 20\%$ of the true value.	Correct problem and repeat ICAL.	Analyst and/or Supervisor	SOP 340384A & 340384B
Cold Vapor AA	Continuing Calibration Verification (CCV)	After every 10 samples and at the end of the analysis sequence.	All reported analytes within $\pm 10\%$ of the true value.	<p>Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails or if two consecutive CCVs cannot be run, perform corrective action(s) and repeat CCV and all associated samples since last successful CCV.</p> <p>Alternately, recalibrate if necessary; then reanalyze all associated samples since the last acceptable CCV.</p>	Analyst and/or Supervisor	SOP 340384A & 340384B

SAP Worksheet #24- Analytical Instrument Calibration Table (Continued)

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference
Cold Vapor AA	Calibration Blanks (ICB/CCB)	Immediately after the ICV and immediately after every CCV.	The absolute values of all analytes must be $< \frac{1}{2}$ LOQ or $< 1/10$ th the amount measured in any sample.	ICB: Correct problem and repeat ICV/ICB analysis. If that fails, rerun ICAL. All samples following the last acceptable Calibration Blank must be reanalyzed. CCBs may not be reanalyzed without reanalysis of the associated samples and CCV(s).	Analyst and/or Supervisor	SOP 340384A & 340384B
pH Probe	Initial Calibration (ICAL)	Daily initial calibration prior to sample analysis	The slope must be 95-105%	Recalibrate	Analyst and/or Supervisor	SOP 340335

SAP Worksheet #24- Analytical Instrument Calibration Table (Continued)

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference
pH Probe	Continuing Calibration Verification (CCV)	After every 10 samples and at the end of the analysis sequence.	The measured concentration must be within 0.1su of the true value.	<p>Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails or if two consecutive CCVs cannot be run, perform corrective action(s) and repeat CCV and all associated samples since last successful CCV.</p> <p>Alternately, recalibrate if necessary; then reanalyze all associated samples since the last acceptable CCV.</p>	Analyst and/or Supervisor	SOP 340335

SAP Worksheet #24- Analytical Instrument Calibration Table (Continued)

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference
Polarized Light Microscope (PLM)	a) The substage condenser and iris diaphragm are centered in the optic axis; b) Set microscope for Kohler illumination (when applicable); c) The substage analyzer and polarizer are oriented at 90 degrees to each other; d) The ocular crosshairs coincide with the privileged directions of the polarizer and analyzer; e) The objectives are centered to prevent any grains from leaving the field of view during stage rotation.	Daily before use	a) The substage condenser and iris diaphragm are centered in the optic axis; b) Set microscope for Kohler illumination (when applicable); c) The substage analyzer and polarizer are oriented at 90 degrees to each other; d) The ocular crosshairs coincide with the privileged directions of the polarizer and analyzer; e) The objectives are centered to prevent any grains from leaving the field of view during stage rotation;	Maintenance	PLM Supervisor	SOP 109; §6.1.1
PLM	Refractive index liquids must be calibrated with an accuracy of 0.004.	Monthly	+/- 0.004	Purchase new RI liquids	PLM Supervisor	SOP109; §6.1.2

SAP Worksheet #25 – Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table

Instrument / Equipment	Activity (Maintenance / Testing / Inspection)	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference
Gamma Spectrometer	Clean cave; fill dewar with N2	Weekly	Acceptable background	Recalibrate	Analyst and/or Supervisor	RA_04-12
Gamma Spectrometer	QA Check of background and source (check deviation)	Daily	Within 3 sigma of measured population	Instrument maintenance and consult with Technical director	Analyst and/or Supervisor	RA_04-12
GC-MS	Change septum, clip column, clean the detector, changing injection port liners, etc.	As needed	Method dependent	Perform needed maintenance and recalibrate the instrument	Analyst and/or Supervisor	SOP 330345 & 330363
GC	Change septum, clip column, clean the detector, changing injection port liners, etc.	As needed	Method dependent	Perform needed maintenance and recalibrate the instrument	Analyst and/or Supervisor	SOP 330343, 330344, 330350A, & 330351
ICP/MS	Change pump tubing, replace torch, clean the nebulizer, etc.	As needed	Method dependent	Perform needed maintenance and recalibrate the instrument	Analyst and/or Supervisor	SOP 340390
ICP/AES	Change pump tubing, replace torch, clean the nebulizer, etc.	As needed	Method dependent	Perform needed maintenance and recalibrate the instrument	Analyst and/or Supervisor	SOP 340386
CVAA	Change pump tubing, clean optical cell, replace lamp, etc.	As needed	Method dependent	Perform needed maintenance and recalibrate the instrument	Analyst and/or Supervisor	SOP 340384A & 340384B
HRGC/HRMS	Parameter Setup	Initially; prior to DCC	Correct Parameters	Reset if incorrect	Analyst and/or Supervisor	BRL SOP-00406
HRGC/HRMS	Tune Check	Initially; prior to DCC	Compliance to ion abundance criteria	Correct the problem and repeat tune check	Analyst and/or Supervisor	BRL SOP-00406

SAP Worksheet #25 – Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table (Continued)

Instrument / Equipment	Activity (Maintenance / Testing / Inspection)	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference
pH probe	Clean or replace electrode	As needed	Method dependent	Perform needed maintenance and recalibrate the instrument	Analyst and/or Supervisor	SOP 340335
PLM	Microscope physical check	Annual	Acceptable functionality	Maintenance or purchase new microscope	QA Dept /Approved vendor	SOP 109

SAP Worksheet #26 – Sample Handling System

SAMPLE HANDLING SYSTEM

SAMPLE COLLECTION, PACKAGING, AND SHIPMENT
Sample Collection (Personnel/Organization): Sampler / Gilbane
Sample Packaging (Personnel/Organization): Sampler / Gilbane
Coordination of Shipment (Personnel/Organization): Sampler / Gilbane
Type of Shipment/Carrier: Overnight shipping service such as FedEx or Laboratory Courier
SAMPLE RECEIPT AND ANALYSIS
Sample Receipt (Personnel/Organization): Laboratory receipt clerk / Pace National/A&B Labs
Sample Custody and Storage (Personnel/Organization): Laboratory technician or custodian / Pace National/A&B Labs
Sample Preparation (Personnel/Organization): Laboratory technician / Pace National/ A&B Labs
Sample Determinative Analysis (Personnel/Organization): Analyst / Pace National/A&B Labs
SAMPLE ARCHIVING
Field Sample Storage (No. of days from sample collection): 90 calendar days
Sample Extract/Digestate Storage (Number of days from extraction/digestion): Up to 40 calendar days depending on method holding times
Biological Sample Storage (Number of days from sample collection): NA
SAMPLE DISPOSAL
Personnel/Organization: Sample Custodian / Pace National/A&B Labs
Number of Days from Analysis: 90 calendar days

Notes:

NA = Not applicable

SAP Worksheet #27 – Sample Custody Requirements Table

Field Sample Custody Procedures (sample collection, packaging, shipment, and delivery to laboratory):

Standardized sample custody procedures will be followed from sample collection, through transfer, storage, and analysis, to ultimate disposal in accordance with SOP PR-TC-02.04.01.01. Sample containers will be properly labeled, and collected samples will be stored on ice if required. Temperature blanks will be used in coolers containing samples requiring preservation at reduced temperature. Samples will be accompanied by a chain-of-custody record. When samples are transferred, both the individual relinquishing and the individual receiving the samples will sign, date, and note the transfer time on the COC record. Samples will be packaged for shipment with completed sample labels for each sample container, sample containers carefully packed upright and on ice (as required), and with a COC record in a Ziploc bag.

Custody seals will be used and will be placed on the cooler so that the seals must be broken before the cooler can be opened. The seals will be signed and dated. Samples will be shipped to the laboratory(s) using an overnight shipper.

Laboratory Sample Custody Procedures (receipt of samples, archiving, disposal):

A designated laboratory sample custodian will accept custody of the samples and verify that the information on the sample labels matches that on the chain-of-custody form(s). Pertinent information as to sample condition, shipment, pickup, and courier will also be checked on the chain-of-custody form. The temperature inside the cooler will be measured immediately after the cooler is opened by measuring and recording the temperature of the temperature blank, as required. Information on the date and time of receipt, method of shipment, and sample condition will also be recorded on a sample receipt form. The custodian will then enter the appropriate data into the laboratory sample tracking system. The sample custodian will use the sample numbers on the sample labels for tracking and assign a unique laboratory number to each sample. The custodian will then transfer the samples to the proper analyst(s) or store the samples in the appropriate secure area. A login verification sheet which includes the sample receipt form will be forwarded to the Gilbane Project Chemist within 48 hours of sample receipt. Data sheets and laboratory records will be retained by the laboratory as part of the permanent documentation for a period of at least three years. Samples and extracts will be retained by the analytical laboratory for a minimum of 90 days after the laboratory reports the data. Unless notified otherwise, samples may be disposed of by the laboratory in a manner consistent with local government regulations.

SAP Worksheet #27 – Sample Custody Requirements Table (Continued)

Chain-of-Custody Procedures:

A COC form will be completed for every group of samples sent to the analytical laboratory, to document sample possession from the time of collection to sample receipt by the laboratory; and a copy of the form will accompany the shipment. Each completed COC form will contain the following information: sample identification number(s); name(s) and signature(s) of collectors, samplers, or recorders; Gilbane project number, project name,

and location of project; project manager's name and contact information; date and time of collection; sample type(s) and analyses requested; and signatures of persons relinquishing and receiving the samples. When samples are transferred, the individuals relinquishing and receiving the samples will sign, date, and note the transfer time on the COC form.

In addition to providing a custody exchange record for the samples, the COC record serves as a formal request for sample analysis. The COC records will be completed, signed, and distributed as described in SOP PR-TC-02.12.02.00, *Sample Handling and Electronic Data Management*.

Sample Numbering

The sample number will be recorded in the field logbook, on the labels, and on the chain-of-custody record at the time of sample collection. A complete description of the sample and sampling conditions will be recorded in the field logbook and referenced using the unique sample identification number.

Samples will be uniquely designated using a numbering system that identifies the CTO number, location/type of sample, and a sequential number as follows:

- Soil samples collected from four SWDA Westside excavations: **WSX-YY-ZZZ**, where:
WSX– **W**est **S**ide followed by a one-digit number indicating the excavation area (1 through 4)
YY – two-character designation indicating either **S**ide **W**all or **B**ottom **S**ample
ZZZ – three-character consecutive number (begins with 001) to designate the number of the sample collected from the excavation
- Soil samples collected from imported fill material sources: **IF-YYY-ZZZ**, where:
IF – represents Import Fill
YYY- three-character consecutive number (begins with 001) to designate the number of the source
ZZZ – three-character consecutive number (begins with 001) to designate the number of the sample collected from the specific source
- RSY Survey Pads (provided for use in waste characterization only): **RSY-QQQQ-UU**, where:
QQQQ – Four-character consecutive pad number 0001; continues consecutively throughout the CTO with no repeated numbers
UU – Two-character consecutive sample number starting with 01 (number of samples collected from each pad)

SAP Worksheet #27 – Sample Custody Requirements Table (Continued)

Sample Packaging – Chemical Samples

Chemical samples will be packaged and shipped in accordance with SOP PR-TC-02.04.01.01, *Sample Handling, Packaging, and Shipping*.

Sample Packaging - Radiological Samples

Samples will be delivered for analysis to the laboratory via cooler, box, or other similar container (ice is not required if only radiological analyses will be performed), along with the completed COC. Samples to be sent off site will be packaged in accordance with applicable Department of Transportation (DOT) and International Air Transport Association (IATA) procedures. At a minimum, sample containers will be placed in a box, cooler, or similar container for shipment and packaged with bubble wrap or other materials as necessary to prevent container breakage.

For samples transported via commercial carrier, two custody seals will be taped across the lid of the box or cooler: one seal in the front and one seal on the side. The COC will include the air bill number, and the "Received By" box will be labeled with the commercial courier's name. The COC will be sealed in a resealable bag and then taped to the inside of the sample cooler lid or placed inside the box. A copy of the COC will be maintained on site and a copy will be e-mailed to the Project Chemist. The box/cooler will be taped shut as necessary. The airbill will be completed for priority overnight delivery and placed in the pouch, which then will be secured to the box/cooler. If multiple boxes/coolers are being shipped, the original COC will be placed in one of the boxes/cooler, and copies of the COC will be placed in the other boxes/coolers. The number of packages should be included on each airbill (e.g., 1 of 2, 2 of 2). Prepared packages will be surveyed prior to shipment.

SAP Worksheet #28 – Laboratory QC Samples Table
(UFP-QAPP Manual Section 3.4)

Matrix	Soil					
Analytical Group	Gamma Spectroscopy					
Analytical Method / SOP Reference¹	HASL 300 Ga-01-R/ RAD_04-12					
QC Sample	Frequency / Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank (MB)	One per preparation batch	No analytes detected > target detection limit	Correct problem. If required, re-prepare and reanalyze MB and all samples processed with the contaminated blank.	Analyst, Supervisor	Representativeness	Acceptable results per stated QC Acceptance Limits
Laboratory Control Sample (LCS)	One per preparation batch	Recovery limits: 87-120% for Cs-137, 87-115% for Co-60, 87-116% for Am-241	Correct problem, then re-prepare and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	Analyst, Supervisor	Accuracy	Acceptable QC Acceptance Limits
Sample Duplicate	One per preparation batch	RPD limit of 40% or Relative Error Ratio <1	Correct problem, then re-prepare and reanalyze all samples in the associated preparatory batch, if not excursion not caused by sample matrix.	Analyst, Supervisor	Precision	Acceptable QC Acceptance Limits

SAP Worksheet #28 – Laboratory QC Samples Table (continued)

Matrix	Soil					
Analytical Group	Dioxin/Furans					
Analytical Method / SOP Reference¹	EPA 8290/ BRL SOP-00406					
QC Sample	Frequency / Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
MB	One per preparation batch	No target analytes detected > 1/2 LOQ or > 1/10 th of the associated regulatory limit or > 1/10 th of the sample result for the analyte, whichever is greater.	Correct problem. If required, reprep and reanalyze MB and all QC samples and field samples processed with the contaminated blank.	Analyst and/or Supervisor	Representative-ness	No target analytes > 1/2 LOQ
LCS	One per preparatory batch	Per DoD QSM/ laboratory limits as listed in Attachment 2 to this SAP.	Correct problem, then re-prepare and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes if sufficient sample material is available.	Analyst and/or Supervisor	Accuracy	Per DoD QSM/ laboratory limits as listed in Attachment 2 to this SAP.
Matrix spike/matrix spike duplicate (MS/MSD)	One per 20 field samples	Per DoD QSM/ laboratory limits as listed in Attachment 2 to this SAP.	Evaluate data, if samples non-detect and surrogate recovery is above upper limits, report with case narrative comment. If obvious chromatographic interference is present, report with narrative comment. Otherwise, re-extract and reanalyze.	Analyst and/or Supervisor	Accuracy/ Precision	Per DoD QSM/ laboratory limits as listed in Attachment 2 to this SAP.
Internal Standard (IS)	Every field sample, standard and QC sample	% Recovery for each IS in the original sample (prior to any dilutions) must be within 40-135% of the ICAL average RF.	Correct problem, then reprep and reanalyze the sample(s) with failed IS. If corrective action fails in field samples, data must be qualified and explained in the Case Narrative.	Analyst and/or Supervisor	Accuracy	40-135%

SAP Worksheet #28 – Laboratory QC Samples Table (continued)

Matrix	Soil					
Analytical Group	Dioxin/Furans					
Analytical Method / SOP Reference¹	EPA 8290/ BRL SOP-00406					
QC Sample	Frequency / Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Sample Estimated Maximum Possible Concentration (EMPC)	Every sample with a response S/N ≥ 2.5 for both quantitation ions.	Identification criteria per method must be met, and the S/N of response for both quantitation ions must be ≥ 2.5 .	Not Applicable.	Analyst and/or Supervisor	Sensitivity	Identification criteria per method must be met, and the S/N of response for both quantitation ions must be ≥ 2.5 .

SAP Worksheet #28 – Laboratory QC Samples Table (continued)

Matrix	Soil					
Analytical Group	VOCs					
Analytical Method / SOP Reference¹	EPA 8260B/ ENV-SOP-MTJL-0100					
QC Sample	Frequency / Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Internal Standards (IS)	Every field sample, standard, and QC sample.	Retention time within ± 10 seconds from retention time of the midpoint standard in the ICAL; EICP area within -50% to $+100\%$ of ICAL midpoint standard. On days when ICAL is not performed, the daily initial CCV can be used.	Inspect GC for malfunctions and correct problem. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	Analyst and/or Supervisor	Accuracy	Acceptable recoveries per stated QC Acceptance Limits
Method Blank (MB)	One per preparation batch	No analytes detected $> \frac{1}{2}$ LOQ or $> 1/10$ th the amount measured in any sample or $1/10$ th the regulatory limit, whichever is greater. Common contaminants must not be detected $> \text{LOQ}$.	Correct problem. If required, reprep and reanalyze MB and all QC samples and field samples processed with the contaminated blank.	Analyst and/or Supervisor	Accuracy / Sensitivity	Acceptable results per stated QC Acceptance Limits
Laboratory Control Sample (LCS)	One per preparation batch	Per DoD QSM/ laboratory limits as listed in Attachment 2 to this SAP.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes if sufficient sample material is available.	Analyst and/or Supervisor	Accuracy	Per DoD QSM/ laboratory limits as listed in Attachment 2 to this SAP.

SAP Worksheet #28 – Laboratory QC Samples Table (continued)

Matrix	Soil					
Analytical Group	VOCs					
Analytical Method / SOP Reference¹	EPA 8260B/ ENV-SOP-MTJL-0100					
QC Sample	Frequency / Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
MS/MSD	One per preparation batch	Per DoD QSM/ laboratory limits as listed in Attachment 2 to this SAP.	Evaluate the data to determine if the failed criteria are due to sample matrix or laboratory error.	Analyst and/or Supervisor	Precision/Accuracy	Per DoD QSM/ laboratory limits as listed in Attachment 2 to this SAP.
Surrogate	Every field sample and QC sample	Per DoD QSM/ laboratory limits as listed in Attachment 2 to this SAP.	Correct problem, then reprep and reanalyze all failed samples for all surrogates in the associated preparatory batch if sufficient sample material is available. If obvious chromatographic interference is present, reanalysis may not be necessary.	Analyst and/or Supervisor	Accuracy	Per DoD QSM/ laboratory limits as listed in Attachment 2 to this SAP.
IS	Every field sample, standard, and QC sample.	Retention time within ± 10 seconds from retention time of the midpoint standard in the ICAL; EICP area within – 50% to +100% of ICAL midpoint standard. On days when ICAL is not performed, the daily initial CCV can be used.	Inspect GC for malfunctions and correct problem. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	Analyst and/or Supervisor	Accuracy	Acceptable recoveries per stated QC Acceptance Limits

SAP Worksheet #28 – Laboratory QC Samples Table (continued)

Matrix	Soil					
Analytical Group	SVOCs					
Analytical Method / SOP Reference¹	EPA 8270C/ 330345					
QC Sample	Frequency / Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
IS	Every field sample, standard, and QC sample.	Retention time within ± 10 seconds from retention time of the midpoint standard in the ICAL; EICP area within – 50% to +100% of ICAL midpoint standard. On days when ICAL is not performed, the daily initial CCV can be used.	Inspect GC for malfunctions and correct problem. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	Analyst and/or Supervisor	Accuracy	Acceptable recoveries per stated QC Acceptance Limits
MB	One per preparation batch	No analytes detected $> \frac{1}{2}$ LOQ or $> 1/10$ th the amount measured in any sample or $1/10$ th the regulatory limit, whichever is greater. Common contaminants must not be detected $> \text{LOQ}$.	Correct problem. If required, reprep and reanalyze MB and all QC samples and field samples processed with the contaminated blank.	Analyst and/or Supervisor	Representativeness	Acceptable results per stated QC Acceptance Limits
MS/MSD	1 per twenty field samples	Per DoD QSM/ laboratory limits as listed in Attachment 2 to this SAP.	Evaluate the data to determine if the failed criteria are due to sample matrix or laboratory error.	Analyst and/or Supervisor	Precision/Accuracy	Per DoD QSM/ laboratory limits as listed in Attachment 2 to this SAP.

SAP Worksheet #28 – Laboratory QC Samples Table (continued)

Matrix	Soil					
Analytical Group	SVOCs					
Analytical Method / SOP Reference¹	EPA 8270C/330345					
QC Sample	Frequency / Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
LCS/LCSD	One LCS or LCS/LCSD pair per preparation batch per matrix	Per DoD QSM/ laboratory limits as listed in Attachment 2 to this SAP.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes if sufficient sample material is available.	Lab Manager/ Analyst	Precision/ Accuracy	Per DoD QSM/ laboratory limits as listed in Attachment 2 to this SAP.
Surrogate	Every field sample and QC sample	Per DoD QSM/ laboratory limits as listed in Attachment 2 to this SAP.	Correct problem, then reprep and reanalyze all failed samples for all surrogates in the associated preparatory batch if sufficient sample material is available. If obvious chromatographic interference is present, reanalysis may not be necessary.	Lab Manager/ Analyst	Accuracy	Per DoD QSM/ laboratory limits as listed in Attachment 2 to this SAP.

SAP Worksheet #28 – Laboratory QC Samples Table (continued)

Matrix	Soil					
Analytical Group	PAHs					
Analytical Method / SOP Reference¹	EPA 8270CSIM/ 330345					
QC Sample	Frequency / Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
IS	During acquisition of calibration standard, samples, and QC check samples	Retention time within ± 10 seconds from retention time of the midpoint standard in the ICAL; EICP area within - 50% to +100% of ICAL midpoint standard. On days with the ICAL is not analyzed, use the initial CCV.	Inspect GC for malfunctions and correct problem. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	Analyst and/or Supervisor	Accuracy	Acceptable recoveries per stated QC Acceptance Limits
MB	One per preparation batch	No analytes detected $> \frac{1}{2}$ LOQ or $> 1/10$ th the amount measured in any sample or $1/10$ th the regulatory limit, whichever is greater. Common contaminants must not be detected $> \text{LOQ}$.	Correct problem. If required, reprep and reanalyze MB and all QC samples and field samples processed with the contaminated blank.	Analyst and/or Supervisor	Representativeness	Acceptable results per stated QC Acceptance Limits
MS/MSD	1 per twenty field samples	Per DoD QSM/ laboratory limits as listed in Attachment 2 to this SAP.	Evaluate the data to determine if the failed criteria are due to sample matrix or laboratory error.	Analyst and/or Supervisor	Precision/Accuracy	Per DoD QSM/ laboratory limits as listed in Attachment 2 to this SAP.

SAP Worksheet #28 – Laboratory QC Samples Table (continued)

Matrix	Soil					
Analytical Group	PAHs					
Analytical Method / SOP Reference¹	EPA 8270CSIM/ 330345					
QC Sample	Frequency / Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
LCS/LCSD	One LCS or LCS/LCSD pair per preparation batch per matrix	Per DoD QSM/ laboratory limits as listed in Attachment 2 to this SAP.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes if sufficient sample material is available.	Analyst and/or Supervisor	Precision/Accuracy	Per DoD QSM/ laboratory limits as listed in Attachment 2 to this SAP.
Surrogate	Every field sample and QC sample	Per DoD QSM/ laboratory limits as listed in Attachment 2 to this SAP.	Correct problem, then reprep and reanalyze all failed samples for all surrogates in the associated preparatory batch if sufficient sample material is available. If obvious chromatographic interference is present, reanalysis may not be necessary.	Analyst and/or Supervisor	Accuracy	Per DoD QSM/ laboratory limits as listed in Attachment 2 to this SAP.

SAP Worksheet #28 – Laboratory QC Samples Table (continued)

Matrix	Soil					
Analytical Group	PCBs					
Analytical Method / SOP Reference¹	EPA 8082/ 330343					
QC Sample	Frequency / Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
MB	One per preparation batch	No analytes detected > ½ LOQ or >1/10th the amount measured in any sample or 1/10th the regulatory limit, whichever is greater.	Correct problem. If required, reprep and reanalyze MB and all QC samples and field samples processed with the contaminated blank.	Analyst and/or Supervisor	Representativeness	Acceptable results per stated QC Acceptance Limits
MS/MSD	1 per twenty field samples	Per DoD QSM/ laboratory limits as listed in Attachment 2 to this SAP.	Evaluate the data to determine if the failed criteria are due to sample matrix or laboratory error.	Analyst and/or Supervisor	Precision/ Accuracy	Per DoD QSM/ laboratory limits as listed in Attachment 2 to this SAP.
LCS/LCSD	One LCS or LCS/LCSD pair per preparation batch per matrix	Per DoD QSM/ laboratory limits as listed in Attachment 2 to this SAP.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes if sufficient sample material is available.	Analyst and/or Supervisor	Precision/ Accuracy	Per DoD QSM/ laboratory limits as listed in Attachment 2 to this SAP.

SAP Worksheet #28 – Laboratory QC Samples Table (continued)

Matrix	Soil					
Analytical Group	Gamma Spectroscopy					
Analytical Method / SOP Reference¹	HASL 300 Ga-01-R/ RAD_04-12					
QC Sample	Frequency / Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Surrogate	Every standard and sample	Per DoD QSM/ laboratory limits as listed in Attachment 2 to this SAP.	Correct problem, then reprep and reanalyze all failed samples for all surrogates in the associated preparatory batch if sufficient sample material is available. If obvious chromatographic interference is present, reanalysis may not be necessary.	Analyst and/or Supervisor	Accuracy	Per DoD QSM/ laboratory limits as listed in Attachment 2 to this SAP.

SAP Worksheet #28 – Laboratory QC Samples Table (continued)

Matrix	Soil					
Analytical Group	PCBs					
Analytical Method / SOP Reference¹	EPA 8082/ 330343					
QC Sample	Frequency / Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Internal Standards (IS)	Every field sample, standard, and QC sample.	Retention time within ± 0.06 RRT UNITS from retention time of the midpoint standard in the ICAL; Internal standard signal (area or height) within -50% to +100% of ICAL midpoint standard. On days when ICAL is not performed, the daily initial CCV can be used.	Inspect GC for malfunctions and correct problem. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	Analyst and/or Supervisor	Accuracy	Acceptable recoveries per stated QC Acceptance Limits

SAP Worksheet #28 – Laboratory QC Samples Table (continued)

Matrix	Soil					
Analytical Group	Pesticides					
Analytical Method / SOP Reference¹	EPA 8081A/ 330344					
QC Sample	Frequency / Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
IS	One per preparation batch	RT within ± 0.06 RRT units of RT of ICAL mid-point standard, Area within - 50% to +100% of ICAL midpoint standard. On days without ICAL, initial CCV may be used as reference.	Inspect GC for malfunctions and correct problem. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	Analyst and/or Supervisor	Accuracy	Acceptable recoveries per stated QC Acceptance Limits
MB	One per preparation batch	No analytes detected $> \frac{1}{2}$ LOQ or $> 1/10$ th the amount measured in any sample or $1/10$ th the regulatory limit, whichever is greater.	Correct problem. If required, reprep and reanalyze MB and all QC samples and field samples processed with the contaminated blank.	Analyst and/or Supervisor	Representativeness	Acceptable results per stated QC Acceptance Limits
MS/MSD	1 per twenty field samples	Per DoD QSM/ laboratory limits as listed in Attachment 2 to this SAP.	Evaluate the data to determine if the failed criteria are due to sample matrix or laboratory error.	Analyst and/or Supervisor	Precision/Accuracy	Per DoD QSM/ laboratory limits as listed in Attachment 2 to this SAP.
LCS and/or LCSD	One LCS or LCS/LCSD pair per preparation batch per matrix	Per DoD QSM/ laboratory limits as listed in Attachment 2 to this SAP.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes if sufficient sample material is available.	Analyst and/or Supervisor	Precision/Accuracy	Per DoD QSM/ laboratory limits as listed in Attachment 2 to this SAP.

SAP Worksheet #28 – Laboratory QC Samples Table (continued)

Matrix	Soil					
Analytical Group	Pesticides					
Analytical Method / SOP Reference¹	EPA 8081A/ 330344					
QC Sample	Frequency / Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Surrogate	Every field sample and QC sample	Per DoD QSM/ laboratory limits as listed in Attachment 2 to this SAP.	Correct problem, then reprep and reanalyze all failed samples for all surrogates in the associated preparatory batch if sufficient sample material is available. If obvious chromatographic interference is present, reanalysis may not be necessary.	Analyst and/or Supervisor	Accuracy	Per DoD QSM/ laboratory limits as listed in Attachment 2 to this SAP.

SAP Worksheet #28 – Laboratory QC Samples Table (continued)

Matrix	Soil					
Analytical Group	TPH					
Analytical Method / SOP Reference¹	EPA 8015B/ ENV-SOP- MTJL-0087 & - 0089					
QC Sample	Frequency / Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank (MB)	One per preparation batch	No analytes detected > ½ LOQ or >1/10th the amount measured in any sample or 1/10th the regulatory limit, whichever is greater.	Correct problem. If required, reprep and reanalyze MB and all QC samples and field samples processed with the contaminated blank.	Analyst and/or Supervisor	Accuracy / Sensitivity	Acceptable results per stated QC Acceptance Limits
Laboratory Control Sample (LCS)	One per preparation batch	Per DoD QSM/ laboratory limits as listed in Attachment 2 to this SAP.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes if sufficient sample material is available.	Analyst and/or Supervisor	Accuracy	Per DoD QSM/ laboratory limits as listed in Attachment 2 to this SAP.
MS/MSD	One per preparation batch	Per DoD QSM/ laboratory limits as listed in Attachment 2 to this SAP.	Evaluate the data to determine if the failed criteria are due to sample matrix or laboratory error.	Analyst and/or Supervisor	Precision/Accuracy	Per DoD QSM/ laboratory limits as listed in Attachment 2 to this SAP.
Surrogate	Every field sample and QC sample	Per DoD QSM/ laboratory limits as listed in Attachment 2 to this SAP.	Correct problem, then reprep and reanalyze all failed samples for all surrogates in the associated preparatory batch if sufficient sample material is available. If obvious chromatographic interference is present, reanalysis may not be necessary.	Analyst and/or Supervisor	Accuracy	Per DoD QSM/ laboratory limits as listed in Attachment 2 to this SAP.

SAP Worksheet #28 – Laboratory QC Samples Table (continued)

Matrix	Soil					
Analytical Group	Metals					
Analytical Method / SOP Reference¹	EPA 6010B & 6020/340386 & 340390					
QC Sample	Frequency / Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Internal Standards (IS)	Every field sample, standard, and QC sample.	IS intensity in the samples within 30-120% of intensity of the IS in the ICAL blank.	<p>If recoveries are acceptable for QC samples, but not field samples, the field samples may be considered to suffer from a matrix effect.</p> <p>Reanalyze sample at 5-fold dilutions until criteria is met.</p> <p>For failed QC samples, correct problem and rerun all associated failed field samples.</p>	Analyst and/or Supervisor	Accuracy	Acceptable recoveries per stated QC Acceptance Limits
Method Blank (MB)	One per preparation batch	No analytes detected > ½ LOQ or >1/10th the amount measured in any sample or 1/10th the regulatory limit, whichever is greater.	Correct problem. If required, reprep and reanalyze MB and all QC samples and field samples processed with the contaminated blank.	Analyst and/or Supervisor	Accuracy / Sensitivity	Acceptable results per stated QC Acceptance Limits
Laboratory Control Sample (LCS)	One per preparation batch	Per DoD QSM/ laboratory limits as listed in Attachment 2 to this SAP.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes if sufficient sample material is available.	Analyst and/or Supervisor	Accuracy	Per DoD QSM/ laboratory limits as listed in Attachment 2 to this SAP.

SAP Worksheet #28 – Laboratory QC Samples Table (continued)

Matrix	Soil					
Analytical Group	Metals					
Analytical Method / SOP Reference¹	EPA 6010B & 6020/340386 & 340390					
QC Sample	Frequency / Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
MS/MSD	One per preparation batch	Per DoD QSM/ laboratory limits as listed in Attachment 2 to this SAP.	Evaluate the data to determine if the failed criteria are due to sample matrix or laboratory error.	Analyst and/or Supervisor	Precision/Accuracy	Per DoD QSM/ laboratory limits as listed in Attachment 2 to this SAP.
Dilution Test (ICP & ICP/MS only)	One per preparatory batch if MS or MSD fails.	Five-fold dilution must agree within $\pm 10\%$ of the original measurement.	No specific CA. Qualify results appropriately.	Analyst and/or Supervisor	Accuracy	Acceptable recoveries per stated QC Acceptance Limits
Post Digestion Spike (ICP & ICP/MS only)	One per preparatory batch if MS or MSD fails.	Recovery within 80-120%.	No specific CA. Qualify results appropriately.	Analyst and/or Supervisor	Accuracy	Acceptable recoveries per stated QC Acceptance Limits

SAP Worksheet #28 – Laboratory QC Samples Table (continued)

Matrix	Soil					
Analytical Group	Metals					
Analytical Method / SOP Reference¹	EPA 7471A/ 340384B					
QC Sample	Frequency / Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank (MB)	One per preparation batch	No analytes detected > ½ LOQ or >1/10th the amount measured in any sample or 1/10th the regulatory limit, whichever is greater.	Correct problem. If required, reprep and reanalyze MB and all QC samples and field samples processed with the contaminated blank.	Analyst and/or Supervisor	Accuracy / Sensitivity	Acceptable results per stated QC Acceptance Limits
Laboratory Control Sample (LCS)	One per preparation batch	Per DoD QSM/ laboratory limits as listed in Attachment 2 to this SAP.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes if sufficient sample material is available.	Analyst and/or Supervisor	Accuracy	Per DoD QSM/ laboratory limits as listed in Attachment 2 to this SAP.
MS/MSD	One per preparation batch	Per DoD QSM/ laboratory limits as listed in Attachment 2 to this SAP.	Evaluate the data to determine if the failed criteria are due to sample matrix or laboratory error.	Analyst and/or Supervisor	Precision/Accuracy	Per DoD QSM/ laboratory limits as listed in Attachment 2 to this SAP.

SAP Worksheet #28 – Laboratory QC Samples Table (continued)
(UFP-QAPP Manual Section 3.4)

Matrix	Soil					
Analytical Group	pH					
Analytical Method / SOP Reference¹	EPA 9045C / WS-WC-0044					
QC Sample	Frequency / Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
LCS	One per preparation batch per matrix	The measured concentration must be within 0.1su of the true value.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes if sufficient sample material is available.	Analyst and/or Supervisor	Accuracy	Acceptable recoveries per stated QC Acceptance Limits
Duplicate	One per prep batch	RPD should be ≤ 1% for solid samples	Evaluate the data to determine if the failed criteria are due to sample matrix or laboratory error.	Analyst and/or Supervisor	Precision	Acceptable results per stated QC Acceptance Limits

SAP Worksheet #28 – Laboratory QC Samples Table (continued)
(UFP-QAPP Manual Section 3.4)

Matrix	Soil/Sediment					
Analytical Group	PLM					
Analytical Method / SOP Reference¹	EPA 600/R-93/116 / SOP 109					
QC Sample	Frequency / Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Friable Non-Asbestos Containing Material (ACM) blank	Daily, at beginning and end of day's work.	No asbestos fibers detected	Correct problem. If required, re-prepare and re-analyze Friable Non-ACM Blank and all samples processed with the contaminated blank.	Analyst, Supervisor	Accuracy/Bias/Contamination	No asbestos fibers detected
Non-friable Non-ACM blank	Every 20 non-friable material samples	No asbestos fibers detected	Correct problem. If required, re-prepare and re-analyze Non-friable Non-ACM Blank and all samples processed with the contaminated blank.	Analyst, Supervisor	Accuracy/Bias/Contamination	No asbestos fibers detected
Intra-analyst Duplicate	10% per month	Within one quantification range above or below that found on the initial analysis	Correct problem, then re-prepare and re-analyze all samples in the associated preparatory batch, if exceedance not caused by sample matrix.	Analyst, Supervisor	Precision/Accuracy/Bias	Within one quantification range above or below that found on the initial analysis
Inter-analyst Duplicate	10% of Intra-duplicate analyses	Within one quantification range above or below that found on the initial analysis	Correct problem, then re-prepare and re-analyze all samples in the associated preparatory batch, if exceedance not caused by sample matrix.	Analyst, Supervisor	Precision/Accuracy/Bias	Within one quantification range above or below that found on the initial analysis

SAP Worksheet #29 – Project Documents and Records Table

Document	Where Maintained
Work Plan, which includes this SAP	Project file, NAVFAC SW Administrative Record
Field forms	Project file
Chain-of-custody forms	Project file, laboratories; NAVFAC SW will receive a copy of definitive data chain-of-custody records
Shipping Records	Project file
Audit/assessment checklists/reports	Project file and laboratory (if applicable)
Corrective action forms/reports	Project file and laboratory (if applicable)
Field change request forms	Project file
Analytical laboratory data packages (DoD Stage 4)	Laboratory and project file; NAVFAC SW Administrative Record
Data validation reports	Validator and project file; NAVFAC SW Administrative Record

Field documentation associated with sampling activities includes field forms, sample labels, chain-of-custody records, sample shipping records, field surveillance reports, and Field Change Request (FCR) forms. In addition, laboratory and data validator documentation will be generated during this project. These types are described in the following sections.

Field Logbooks/Forms

Field forms (or logbooks) will be handled in accordance with SOP PR-TC-01.04.04.00, *Field Documentation* (included in Attachment 1 of this SAP).

Sample Labels

For radiological analysis of fill material, a resealable plastic bag will be used to collect soil samples. Sample labels will be generated at the time the chain-of-custody record is prepared, and affixed to the resealable plastic bag and the prepared sample container (if one is used during sample processing). The label will contain the following:

- Sample identification number.
- Sample collection date (month/day/year)
- Analytical method.
- Time of collection.
- Sampler's initials.

SAP Worksheet #29 – Project Documents and Records Table (Continued)

If containers are too small to fit all the sample information listed above, at a minimum the container will be labeled with the sample identification number, and the remaining information will be recorded on the chain of custody.

For chemical samples to be sent to the laboratory labels will be hand-written using indelible black or blue ink on a waterproof label and affixed to each sample container at the time of sample collection (or labels may be computer generated). The label will contain the following information:

- Sample identification number.
- Sample collection date (month/day/year).
- Time of collection (24-hour clock).
- Sampler's initials.
- Preservative (if any).

Chain of Custody

Chain-of-custody record information is described in SAP Worksheet #27.

Sample Shipping Records

For samples shipped via FedEx to the laboratory(s), the chain-of-custody record will be packaged within the cooler, and the sender's copy of the airbill will serve as custody documentation. The air bill number will be written on the COC, and the airbill will be maintained in the project file. Sample shipping procedures are detailed in SAP Worksheet #27.

Field Surveillance Reports

Field surveillances will be performed in accordance with the three phases of inspection as required by the QC program. A Preparatory Inspection will be performed by the PQCM prior to the first sampling activities. This will include a general orientation for health and safety. An Initial Inspection will be conducted at the beginning of field sampling activities for this project. Daily field inspections and subsequent surveillances will be performed at the discretion of the PQCM or the QCPM throughout the duration of the project. The PQCM will use the Initial Inspection Checklist during inspection.

Field Change Request

An FCR will be prepared by the Program Chemist, or a designee, if a change to the SAP is needed during sampling activities. Any changes will not result in a change DQOs for this project. The FCR will be reviewed and approved by the Navy QAO via NIRIS and will be acknowledged by the QCPM prior to field implementation.

SAP Worksheet #29 – Project Documents and Records Table (Continued)

Major changes to work scope affecting the DQOs or meeting criteria described in EWI #2, 3EVR.2, *Review, Approval, Revision, and Amendment of Sampling and Analysis Plans* (NAVFAC SW, 2006) will require preparation of a SAP Addendum. The SAP Addendum must be approved by the Navy QAO and the planning team identified on SAP Worksheet #10 prior to conducting sampling and analysis.

Laboratory Documentation

Relevant laboratory raw data and documentation, including but not limited to logbooks, data sheets, electronic files, and reports, will be maintained by the laboratory for at least 5 years. Gilbane must be notified 30 days before disposal of any relevant records. Laboratory data packages will be compliant the reporting requirements as described in Appendix A of the DoD QSM 5.1.1 or 5.1.

- Excavation characterization soil samples and imported fill material used for backfill – 100 percent suitable for Stage 4.
- Waste Characterization samples – 100 percent suitable for Stage 2A.

For Stage 2A- or 4-suitable data packages, an EDD will be uploaded directly to Gilbane's web-based portal at <http://edms.gilbaneco.com>. The hard-copy data package also will be uploaded in PDF format to the portal. Both the EDDs and the hard-copy data package will present results to two or three significant figures. For radiological results, at least three significant figures will be used. For organic results, at least two significant figures will be used. For inorganic results, at least two significant figures will be used for results less than 10, and at least three significant figures will be used for results greater than 10. Results for QC analyses (method blanks, MS/MSDs, LCSs, and duplicates) will be reported up to 3 significant figures.

When revisions to Stage 2A- or 4-suitable data packages are required, the report will be resubmitted in its entirety to both the portal and the Project Chemist, with the notation "amended or revised report." If the revisions affect the EDDs, the revised EDD also will be uploaded to the portal.

Data Validation Reports

Samples are to be validated in accordance with SAP Worksheet #36 and Gilbane SOP PR-TC-04.01.00.00 or PR-TC-04.01.02.00, as appropriate.

The validation report for definitive samples will include the data validation findings worksheets. Each laboratory sample delivery group (SDG) will have its own data validation report. The validation reports will contain the following information:

- Title page that contains project name, sample collection date, validator subcontractor name, report date, type of analysis, laboratory, sample delivery group (SDG), sample identifications (including MS/MSD, duplicate, reanalysis, or dilution samples), sample matrix (e.g., soil, water), and validation level (DoD Stage 2B or Stage 4).

SAP Worksheet #29 – Project Documents and Records Table (Continued)

- Introduction page including the number of samples per matrix, analytical method reference, validation guideline reference, section references to summary qualification flags, and identification of QC samples. The report body will include the acceptance criteria used to evaluate each QC parameter exceeding criteria, a list of all QC exceedances as well as the associated bias, the samples associated with each exceedance, and the qualifiers applied. Statements regarding flag classification (protocol/advisory) and whether a raw data check was performed also will be included.
- Evaluation and discussion of the following parameters:
 - Technical holding times.
 - GC/MS instrument performance check (tune) if applicable.
 - Calibration.
 - Laboratory blanks.
 - Accuracy and precision data for internal laboratory QC associated with each SDG.
 - Target compound identification.
 - System performance checks.
 - Analyte quantitation and quantitation limits (MDC and LOQs).
 - Field QC samples (if not applicable, report will note).
 - Overall assessment of data.
 - Qualifier classification.

The data validator will upload a PDF copy of the validation report to the project portal at <http://edms.gilbaneco.com>, and enter qualifiers directly to the database using the project portal.

The data validation subcontractor must maintain validation records for at least 5 years. Gilbane will be notified 30 days before disposal of any records.

SAP Worksheet #30 – Analytical Services Table

Matrix	Analytical Group	Sample Locations/ ID Number	Analytical Method	Data Package Turnaround Time	Laboratory / Organization (name and address, contact person and telephone number)	Backup Laboratory / Organization (name and address, contact person and telephone number)
Soil	PAHs, Pb, Metals, PCBs, Pesticides, VOCs, SVOCs, TPH, pH	Samples as listed on SAP WS #18	EPA 8270CSIM, 6010B, 6020/7471A, 8082, 8081A, 8260B, 8270C, 8015B, 9045C	10 Business Days	Pace National 12065 Lebanon Road Mt. Juliet, TN 37122 Jennifer Gambill 615-773-9670 jgambill@pacenational.com	TestAmerica West Sacramento 880 Riverside Parkway West Sacramento, CA 95605 Rhonda Ridenhower 314-787-8227 Rhonda.ridenhower@testamericainc.com
Soil	Dioxin/Furans	Samples as listed on SAP WS #18	EPA 8290	10 Business Days	Pace Analytical Services, LLC 1700 Elm St. SE, Suite 200 Minneapolis, MN 55414 Jennifer Gambill 615-773-9670 jgambill@pacenational.com	TestAmerica West Sacramento 880 Riverside Parkway West Sacramento, CA 95605 Rhonda Ridenhower 314-787-8227 Rhonda.ridenhower@testamericainc.com
Soil	Ra-226	Samples as listed on SAP WS #18	HASL 300 GA-01-R	20 Business Days	Pace National 12065 Lebanon Road Mt. Juliet, TN 37122 Jennifer Gambill 615-773-9670 jgambill@pacenational.com	TestAmerica St. Louis 13715 Rider Trail North Earth City, MO 63045 Rhonda Ridenhower 314-787-8227 Rhonda.ridenhower@testamericainc.com

SAP Worksheet #30 – Analytical Services Table

Matrix	Analytical Group	Sample Locations/ ID Number	Analytical Method	Data Package Turnaround Time	Laboratory / Organization (name and address, contact person and telephone number)	Backup Laboratory / Organization (name and address, contact person and telephone number)
Soil	Asbestos	Samples as listed on SAP WS #18	EPA 600/R-93/116	10 Business Days	A&B Labs 10100 East Freeway, Ste. 100 Houston, TX 77029 Shantall Carpenter 713-543-6060	NVL Laboratories, Inc. 4708 Aurora Ave N Seattle, WA 98103 206-547-0100

Selected laboratories will have successfully completed the DoD ELAP certification for the matrices and methods listed in SAP Worksheet #23 and will maintain current status throughout the duration of this project. Laboratories also will be certified by the California Water Board under the ELAP for all the analytical methods listed in SAP Worksheet #23. DoD and California ELAP certifications for the primary laboratory are presented in Attachment 4 of this SAP. Laboratories must provide the project QC and data deliverables required by this SAP and the DoD QSM for Environmental Laboratories version 5.1 or 5.1.1 as applicable per the laboratory's certification. Status of laboratory certifications/ accreditations will be verified prior to fieldwork and before samples are delivered to lab. Updates to lab accreditation to ensure the laboratory is qualified to perform the analysis will be made prior to sample testing.

SAP Worksheet #31 – Planned Project Assessments Table

Assessment Type	Frequency	Internal or External	Organization Performing Assessment	Person(s) Responsible for Performing Assessment (title and organizational affiliation)	Person(s) Responsible for Responding to Assessment Findings (title and organizational affiliation)	Person(s) Responsible for Identifying and Implementing Corrective Actions (CA) (title and organizational affiliation)	Person(s) Responsible for Monitoring Effectiveness of CA (title and organizational affiliation)
Field Sampling Surveillance	Annually; at least one technical systems audit (TSA) at the start of field activities, with discretionary follow-ups	Internal	Gilbane	PQCM (Gilbane)	Project Manager (Gilbane)	Project Manager (Gilbane)	Project Manager and PQCM (Gilbane)
Management Review	Once during the project	Internal	Gilbane	QCPM (Gilbane)	Project Manager (Gilbane)	Project Manager (Gilbane)	PQCM (Gilbane)

SAP Worksheet #32 – Assessment Findings and Corrective Action Responses

Assessment Type	Nature of Deficiencies Documentation	Individual(s) Notified of Findings (name, title, organization)	Time-frame of Notification	Nature of Corrective Action Response Documentation	Individual(s) Receiving Corrective Action Response	Time-frame for Response
Field Sampling Surveillance	Surveillance Report	Project Manager, Gilbane	7 days after completion of report	Corrective Action Report	Project Manager and QCPM, Gilbane	5 days after notification
Management Review	Surveillance Report	Project Manager, Gilbane	7 days after completion of report	Corrective Action Report	Project Manager, Gilbane	14 days after notification

SAP Worksheet #33 – QA Management Reports Table

Type of Report	Frequency (daily, weekly monthly, quarterly, annually, etc.)	Projected Delivery Date(s)	Person(s) Responsible for Report Preparation (title and organizational affiliation)	Report Recipient(s) (title and organizational affiliation)
Field Sampling Surveillance Report	Once, at startup of sampling	Determined during the project	PQCM, Gilbane	Project Manager and QCPM, Gilbane
Management Review Report	Once, after management review is completed	Determined during the project	QCPM, Gilbane	Project Manager and Program Manager, Gilbane

SAP Worksheet #34 – Verification (Step I) Process Table

Verification Input	Description	Internal / External	Responsible for Verification (name, organization)
Field notes/logbook	Field notes will be reviewed internally, at intervals as needed during the project and at the completion of the work and placed in the project file. A copy of the field notes will be attached to the final report.	I	PQCM, Gilbane
Chain-of-custody forms	Chain-of-custody forms will be reviewed internally in the field upon completion and verified against the packed sample coolers they represent. The shipper's signature on the chain-of-custody form will be initialed by the reviewer. A copy of the form will be retained in the project file, and the original and remaining copies will be taped inside the cooler for shipment.	I I	PQCM, Gilbane Second-level review by Project Chemist, Gilbane
Sample receipt	For samples shipped to the lab via FedEx, the Project Chemist will verify receipt of samples by the laboratory the day following the shipment.	I	Project Chemist, Gilbane
Sample logins	Sample log-in information will be reviewed and verified for accuracy and completeness in accordance with the requirements in this SAP.	E ¹	Laboratory PM
Laboratory analytical results prior to release	Laboratory analytical results will be reviewed to verify that the requirements in this SAP have been met. Prior to release, results will be verified as follows:	E ¹	Laboratory PM
	• Analytical results (100 percent) comply with the method- and project-specific requirements, and any deviation or failure to meet criteria is documented for the project file.	E ¹	Laboratory PM
	• Manual entries (100 percent) are free of transcription errors, and manual calculations are accurate; computer calculations are spot-checked to verify program validity; results reported are compliant with method- and project-specific QC requirements; raw data and supporting materials are complete; spectral assignments are confirmed; descriptions of deviations from method or project requirements are documented; significant figures and rounding have been used appropriately; reported values include dilution factors; and results are reasonable.	E ¹	Analyst

SAP Worksheet #34 – Verification (Step I) Process Table (Continued)
(UFP-QAPP Manual Section 5.2.1)

Verification Input	Description	Internal / External	Responsible for Verification (name, organization)
Laboratory analytical results prior to release (Continued)	<ul style="list-style-type: none"> Analytical results reported are compliant with method- and project-specific QC; analytical methods are implemented in compliance with approved SOPs. (This review may be conducted after release of results, since reviews are done only on 10 percent of the results.) 	E ¹	Laboratory PM
Laboratory analytical results due at turnaround time listed on chain-of-custody record	Laboratory analytical results will be verified as having been obtained following the protocols in this SAP and being of sufficient quality to satisfy DQOs.	I	Project Chemist, Gilbane
Laboratory Data Packages	Screening data reports and Stage 3- or 4-equivalent laboratory data packages will be verified by the laboratory performing the work for completeness and technical accuracy prior to submittal, in accordance with the requirements described in SAP Worksheet #29.	E ¹	Laboratory PM
Field and electronic data	One-hundred percent of manual entries will be reviewed against the hard-copy information, and 5 percent of electronic uploads will be checked against the hard copy.	I	Project Chemist, Gilbane
Analytical Procedures	Ensure that the analytical methods and deliverable requirements described in this SAP were followed (including holding times, analyte lists, and QC criteria).	E ¹	Laboratory PM
Laboratory Data Reports	Ensure that data reports are validated by the laboratory performing the work for technical accuracy and for meeting the requirements listed in SAP Worksheet #29 prior to submittal.	E ¹	Laboratory PM

Notes:

¹The Laboratory Project Manager may direct the use of a designee for verification inputs as needed.

SAP Worksheet #35 – Validation (Steps IIa and IIb) Process Table

Step IIa / IIb	Validation Input	Description	Responsible for Validation (name, organization)
IIa	Sample Collection	Ensure that the sampling procedures described in this SAP were used to collect samples and that any deviations from those procedures were documented in a FCR.	PQCM, Gilbane Project Chemist, Gilbane
IIa	Sample Handling	Ensure that the procedures described in this SAP for sample handling, packaging, and transport to the laboratory were followed.	PQCM, Gilbane Project Chemist, Gilbane
IIa	Sample Documentation	Ensure that the chain-of-custody record procedures described in this SAP were followed for sample collection, and that logbooks or field forms were completed as required.	PQCM, Gilbane Project Chemist, Gilbane
IIb	Sampling Procedures	Review sampling procedures to document any deviations that occurred and note any corrective actions required.	PQCM, Gilbane
IIb	Analytical Procedures	Review analytical procedures to document any deviations that occurred and note any corrective actions required.	Site RSO or designee, Envirachem/Gilbane Project Chemist, Gilbane Third Party Validator, Validata
IIb	Project Quantitation Limits and Laboratory QC Criteria	Ensure that project quantitation limits and laboratory QC criteria were followed and that any deviations were documented.	Site RSO or designee, Envirachem/Gilbane Project Chemist, Gilbane Third Party Validator, Validata

Notes:

The Laboratory PM may direct the use of a designee for verification inputs as needed.

SAP Worksheet #36 – Analytical Data Validation (Steps IIa and IIb) Summary Table

Step IIa / IIb	Matrix	Analytical Group	Validation Criteria	Data Validator (title and organizational affiliation)
IIa	All	All	In accordance with Laboratory SOPs listed in SAP Worksheet #23 and the DoD QSM 5.1 or 5.1.1, as applicable.	Site RSO or designee, Envirachem/Gilbane Project Chemist, Gilbane
IIb	All	Waste	In accordance with NAVFAC SW EWI #1, this SAP, and Gilbane SOP PR-TC-04.01.00.00 and PR-TC-04.01.02.00	Project Chemist, Gilbane
IIb	All	Definitive Data (Characterization and Import Fill Material Sampling)	In accordance with NAVFAC SW EWI #1, this SAP, and Gilbane SOP PR-TC-04.01.00.00 and PR-TC-04.01.02.00	Third-Party data validator, Validata

The validation strategy for definitive data for this project is 90% Stage 2B and 10% Stage 4, in accordance with Step IIb above. The 10% will be randomly chosen on the chain-of-custody record. Waste samples will be validated at a Stage 2A.

DoD Stage 4 Data Validation

DoD Stage 4 data validation is a full validation that will be performed on the TI Phase IV NTCRA summary and raw data packages. The data reviewer will request any missing information from the laboratory and copy the Project Chemist when missing information is requested. The data reviewer will validate all components of the data package even when an individual QC element has been rejected. An overall final qualification of results will encompass the impact of individual findings and will be determined using the professional judgment of a senior data reviewer.

SAP Worksheet #36 – Analytical Data Validation (Steps IIa and IIb) Summary Table (continued)

Stage 4 Data Validation Elements

The QC elements to be reviewed for Stage 4 validation are listed below.

Radiological Analyses

- Chain of Custody
- Case Narrative
- Initial calibration.
- Continuing calibration.
- Blanks.
- Duplicate sample RPD, duplicate error ratio (DER), or relative error ratio (RER), as appropriate.
- Laboratory control sample recovery.
- Reporting limits and uncertainty.
- Review of reagent traceability summary, if applicable.
- Calculation checks of quantified analytical data and QC samples.
- Overall assessment of data in the SDG.

Chemical Analyses

- Chain of custody.
- Case narrative.
- Holding times and preservation.
- Blanks.
- Lab QC.
- Field QC, if applicable.
- Surrogates and internal standards, where applicable.
- Initial and continuing calibrations.
- Instrument performance checks.
- Review of raw data.
- Review of reagent traceability summary, if applicable.
- Calculation checks of quantified analytical data and QC samples.

SAP Worksheet #36 – Analytical Data Validation (Steps IIa and IIb) Summary Table (continued)

DoD Stage 2B Data Validation

Stage 2B data validation is a validation that will be performed on the summary packages only (i.e., not on raw data). The Stage 2B encompasses sample completeness/compliance, field QC, method QC, and instrument-related QC. The data reviewer will request any missing information from the laboratory and copy the client's project manager when missing information is requested. The data reviewer will validate all components of the summary data package, even when an individual QC element has been rejected. An overall final qualification of results will encompass the impact of individual findings and will be determined using the professional judgment of a senior data reviewer.

Stage 2B Data Validation Elements

The QC elements to be reviewed for Stage 2B validation are listed below.

Radiological Analyses

- Chain of custody.
- Case narrative.
- Initial calibration.
- Continuing calibration.
- Blanks.
- Duplicate sample RPD, DER, or RER, as appropriate.
- Laboratory control sample recovery.
- Reporting limits.
- Overall assessment of data in the SDG.

Chemical Analyses

- Chain of custody.
- Case narrative.
- Holding times and preservation.
- Blanks.
- Lab QC.
- Field QC, if applicable.
- Surrogates and internal standards, where applicable.
- Initial and continuing calibrations.
- Instrument performance checks

SAP Worksheet #36 – Analytical Data Validation (Steps IIa and IIb) Summary Table (continued)

DoD Stage 2A Data Validation

Stage 2A data validation is a cursory validation that will be performed on the summary packages only (i.e., not on raw data). The Stage 2A validation encompasses sample completeness/compliance, field QC, and method QC. The data reviewer will request any missing information from the laboratory and copy the client's project manager when missing information is requested. The data reviewer will validate all components of the summary data package, even when an individual QC element has been rejected. An overall final qualification of results will encompass the impact of individual findings and will be determined using the professional judgment of a senior data reviewer.

Stage 2A Data Validation Elements

The QC elements to be reviewed for Stage 2B validation are listed below.

Radiological Analyses

- Chain of custody.
- Case narrative.
- Blanks.
- Duplicate sample RPD, DER, or RER, as appropriate.
- Laboratory control sample recovery.
- Reporting limits.
- Overall assessment of data in the SDG.

Chemical Analyses

- Chain of custody.
- Case narrative.
- Holding times and preservation.
- Blanks.
- Lab QC.
- Field QC, if applicable.
- Surrogates, where applicable.

SAP Worksheet #36 – Analytical Data Validation (Steps IIa and IIb) Summary Table (continued)

The following documents will be used as guidance for validating chemical analytical results:

- *General Data Validation Guidelines* (DoD, 2018)
- *Contract Laboratory Program National Functional Guidelines for Superfund Organic Methods Data Review*, EPA 540-R-2017-002 (EPA, 2017a).
- *Contract Laboratory Program National Functional Guidelines for Inorganic Superfund Data Review*, EPA 540-R-2017-001 (EPA, 2017b).
- *EWI #1, 3EN2.1, Chemical Data Validation* (NAVFACSW, 2001).
- *Test Methods for Evaluating Solid Waste, Physical Chemical Methods*, SW-846, Third Edition and final updates (EPA, 1986).
- QSM v. 5.1 (DoD/ DOE, 2017) or QSM v. 5.1.1 (DoD/DOE, 2018) as applicable per the laboratory certification.
- QC criteria specified in this SAP.

Radiological analytical results will be validated using the QC criteria specified in the SAP and QSM v. 5.1.1 (DoD/DOE, 2018), Gilbane SOP PR-TC-04.01.02.00, and Chapter 8 of the *Multi-Agency Radiological Laboratory Analytical Protocols Manual* (MARLAP), EPA 402-B-04-001A (EPA et al., 2004).

SAP Worksheet #37 – Usability Assessment

After the analytical results have been reviewed, verified, and validated in accordance with SAP Worksheets #34 through #36, a Quality Control Summary Report (QCSR) will be prepared as an appendix to the Post Construction Summary Report to assess data quality and usability. The QCSR will include review of the following, and will include enough information to support the data usability conclusions:

- Sample collection and analytical methods to verify that these were performed as discussed in SAP Worksheets #14 and #17.
- Project-specific QLs as listed in SAP Worksheets #15.1 through #15.18 to verify that project-specific remedial goals were met for each sample.
- DQOs to determine whether they have been achieved by the data collected.
- Project-specific data quality indicators for precision, accuracy, representativeness, completeness, comparability, and sensitivity (PARCCS) parameters (including, but not limited to, assessment of analytical DQOs) as discussed below.

Precision

Precision is defined as the degree of mutual agreement between individual measurements of the same property under similar conditions, and provides a measurement of the reproducibility of an analytical result. Precision will be evaluated through the analysis of field duplicate samples, LCS and LCSD (if LCSD is run), and MS/MSD samples, as applicable (see SAP Worksheet #20). QC criteria failures will be documented in the case narrative and included in the Comprehensive Analytical Report from the analytical laboratory. The precision measurement will be determined using the RPD, RER between the duplicate sample results as follows:

$$RPD = \frac{|A - B|}{(A + B)/2} \times 100\%$$

where: A = First duplicate concentration
B = Second duplicate concentration

$$DER = \frac{|(S - D)|}{\sqrt{(CSUs)^2 - (CSUd)^2}}$$

—
=

SAP Worksheet #37 – Usability Assessment (Continued)

where: S = Sample result
D = Duplicate (or lab replicate) result
CSUs = Combined standard uncertainty of the sample
CSUd = Combined standard uncertainty of the duplicate
DER = Duplicate error ratio

$$RER = (\text{result activity} - \text{duplicate activity}) / (\text{sample uncertainty} + \text{duplicate uncertainty})$$

using 2 sigma propagated uncertainty

As applicable, the RPD or RER limits for laboratory duplicates, MSDs, and LCSDs are presented in SAP Worksheet #28. Associated samples that do not meet the criteria will be evaluated by the validator.

Field duplicate precision will be evaluated for chemical and radiological analyses for those concentrations 5 times the reporting limit using the RPD presented on SAP Worksheet #12. For concentrations less than 5 times the reporting limit, the precision for chemical analyses will be evaluated by a “reporting limit check” in which the difference in concentration between the duplicate and the parent sample is compared to the reporting limit as the criterion. Field duplicate precision for gamma spectrometry with concentrations near the MDC will be evaluated using an RER criterion of less than or equal to 1.

For the MS and the MSD, sample heterogeneity and the presence of interfering compounds often negatively affect the precision of the analysis. Also, the presence of high levels of target compounds in the sample chosen for spiking may necessitate a dilution of the sample, or may otherwise result in errors in spiked compound recovery. For these reasons, MS samples may not be truly representative of the precision of the analytical process. When the RPD obtained from the results of MS/MSD are out of criteria and the RPD of the LCS/LCSD is within criteria, the poor variance is attributed to the matrix of the sample and the effect on the project objectives must be considered.

The overall precision will be discussed in the QCSR. If the precision is poor, the impacted data will be qualified as described in the EPA National Functional Guidelines. The impact will be documented along with the rationale for re-sampling or the limited or unlimited use of the data.

Accuracy

Accuracy is the degree of agreement between an analytical measurement and a reference accepted as a true value. The accuracy of a measurement system can be affected by errors introduced by field contamination, sample preservation, sample handling, sample preparation, or analytical techniques. A program of sample spiking will be conducted to evaluate laboratory accuracy. Accuracy will be evaluated by the percent recovery of the spiked compounds in the LCS, LCS duplicate, and MS/MSD

SAP Worksheet #37 – Usability Assessment (Continued)

samples. LCS and MS samples will be spiked prior to extraction with the method target compounds indicated in this SAP. MS/MSD and LCS or blank spike samples will be analyzed at a frequency of 5 percent or one per sample delivery group/analytical batch (sample sets are about 10 samples). The results of the spiked samples will be used to calculate the percent recovery for evaluating accuracy, using the following equation:

$$\text{Percent Recovery} = \frac{S-C}{T} \times 100$$

where:

S = Measured spike sample concentration
C = Sample concentration
T = True or actual concentration of the spike

SAP Worksheet #28 presents accuracy goals for this investigation based on the percent recovery of matrix and surrogate spikes. Results that fall outside the accuracy goals will be further evaluated based on other QC samples.

For MS and MSD, sample heterogeneity and the presence of interfering compounds often negatively affect the accuracy and precision of the analysis. Also, the presence of high levels of target compounds in the sample chosen for spiking may necessitate a dilution of the sample, or may otherwise result in errors in spiked compound recovery. For these reasons, MS/MSD samples may not be truly representative of the accuracy and/or precision of the analytical process.

If MS/MSD analyses do not meet the specified recovery criteria, the recoveries from the LCS will be evaluated. If the LCS accuracy criteria are met, the failure of the MS/MSD will be attributed to interference from the sample matrix, and no corrective action will be required. If the LCS accuracy criteria are not met, the associated primary and QC samples will be re-prepared and re-analyzed.

In cases where re-preparation and re-analysis of the samples is not possible, the QC criteria failures will be documented in the case narrative and included in the Comprehensive Analytical Report. The affected data will be qualified as described the guidelines described in SAP Worksheet #36, and the impact of the QC failures on the DQOs for the project will be assessed in the final report.

Trend Analysis for Precision and Accuracy

For each analytical method, the laboratory uses the MS/MSD and LCS/LCSD data to track and analyze trends in the laboratory. From these trends they can recognize deficiencies in the method and create in-house acceptance criteria. For this project, the limits are based on the most recent version of the DoD QSM if available. For methods

SAP Worksheet #37 – Usability Assessment (Continued)

where the limits are not available, the project criteria default to the laboratory criteria based on their tracked trending.

The precision and accuracy of the entire data set is used to determine if any systemic problems have occurred during the sampling event that will result in deficiencies in the data set. The occurrence of systemic problems and the resulting consequences will be discussed in the QCSR. The data reviewer will make every effort to identify any critical elements or trends that would result in non-usability of data as early as possible.

Representativeness

Representativeness expresses the degree to which sample data accurately and precisely represent the characteristics of a population, variations in a parameter at a sampling point, or an environmental condition that they are intended to represent. For this project, representative data will be obtained through careful selection of sampling locations and analytical parameters. Representative data also will be obtained through proper collection and handling of samples to avoid interference and minimize cross-contamination. Representativeness of data will also be ensured through consistent application of the appropriate established field and laboratory procedures. To aid in evaluating the representativeness of the sample results, field and laboratory blank samples will be evaluated for the presence of contaminants. Laboratory procedures will be reviewed to verify that standard operating procedures were followed and method requirements were met during the analysis of project samples. Laboratory sample storage practices, holding times, sub-sampling procedures, method blanks, and evidence of matrix interference will be assessed for potential impacts on the representativeness of the data.

Data determined to be non-representative will be used only if accompanied by appropriate qualifiers and limits of uncertainty.

Representativeness as it relates to field procedures refers to the collection of samples that allow accurate conclusions to be made regarding the composition of the sample media at the entire site. Representativeness will be assessed qualitatively by evaluating whether the procedures described in this SAP were followed. The site-sampling layout, including sampling locations, frequency of sampling, and timing of sampling activities, will be reviewed.

Completeness

Completeness is a measure of the percentage of project-specific data that are valid. Valid data are obtained when samples are collected and analyzed in accordance with the QC procedures outlined in this SAP and when none of the QC criteria that affect data usability is exceeded. When data validation is completed, the percent completeness value will be calculated by dividing the number of useable samples by the total number of samples planned for this investigation to calculate field completeness.

SAP Worksheet #37 – Usability Assessment (Continued)

Completeness will be evaluated by reviewing the tasks that contribute to the sampling event, such as chain-of-custody procedures, adherence to the Work Plan, and adherence to this Sampling and Analysis Plan. The QC parameters to be evaluated in determining completeness include holding times, initial calibrations, continuing calibrations, surrogate recoveries, LCS recoveries, MS/MSD recoveries and RPDs, and laboratory duplicate RPDs. Analytical completeness will be calculated based on the number of individual results (i.e., per analyte). The evaluation of completeness will help determine whether any limitations are associated with the decisions to be made based on the data collected. The completeness goal for this project is 95%.

Comparability

Comparability expresses the confidence with which one dataset can be compared with another. Comparability of data will be achieved by consistently following standard field and laboratory procedures and by using standard measurement units in reporting analytical data. Analytical methods selected for this field investigation are consistent with the methods used during previous investigations of this type. To ensure the comparability of laboratory data, the contract laboratory will use standard test methods and means of sample preservation; standard units, detection limits, calculation procedures, and reporting formats; and standard measures of accuracy and precision. Only laboratories that have been approved by the DoD ELAP will perform chemical analyses of environmental samples to produce definitive data in support of this CTO.

Sensitivity

The DL, LOD, and LOQ will be evaluated by the project team prior to sample analysis to determine if the laboratory is able to attain the required sensitivity for the project. If project decision limits are too sensitive, it will be determined prior to sample analysis whether a sensitivity variance will be issued to the laboratory based on the method chosen and the technology available.

The DL is the minimum quantity of an analyte that can be reliably distinguished from background noise or from zero for a specific analytical method at a 99 percent confidence level. The DL protects against false positives. The LOD is the minimum quantity of an analyte that can be reliably detected for a specific analytical method at a 99 percent confidence level that the value is not a false negative. The LOD should be equivalent to the concentration of the DL verification standard. The LOQ represents the smallest quantity of an analyte that can be accurately and reproducibly quantified in a given sample matrix (e.g., three to five times the

LOD). For this project, the minimum detectable concentration (MDC) pertains only to radiological analyses, defined as follows in accordance with the Multi-Agency Radiological Laboratory Analytical Protocols: the MDC is calculated as a sample specific value and typically these values assume both a Type I (α) and Type II (β) error

SAP Worksheet #37 – Usability Assessment (Continued)

of 5 percent. The LOD and/or the LOQ and MDC should be sensitive enough to meet the project decision limits (e.g., PSLs). The LOD, LOQ and MDC will be evaluated after sample analysis to determine if there were any matrix effects, operator errors, or analytical process errors that interfered with the ability to compare the results to the project decision limits. The LOD and MDC (as applicable) will be used to determine if no detectable amounts of contaminants of concern are present. If no detectable amounts are reported and all data are acceptable from the verification and validation, then the data is usable. The DL will be used to determine if any detectable amounts of contaminants of concern are present. If detectable amounts are reported and the verification and validation are acceptable, then the data is usable. Any detection falling between the DL and LOQ are qualified as estimated. If anomalies in sensitivity are present, the rationale for use or non-use of the affected samples will be discussed in the QCSR. Worksheet #15 presents the laboratory DL, LODs, LOQs, and MDCs (as applicable) for the selected analytical method(s) used to support the project decision limits.

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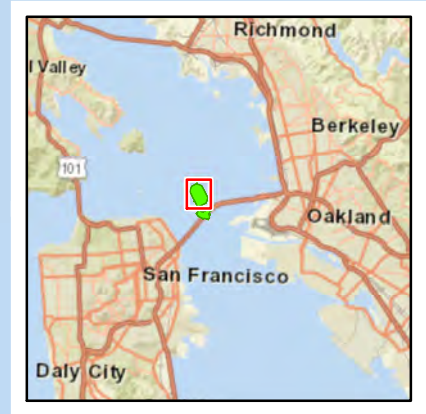
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FIGURES

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San Francisco Bay



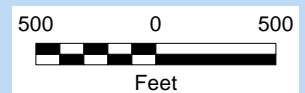
SWDA Westside

Site 12

Building 570
Gilbane Site Office

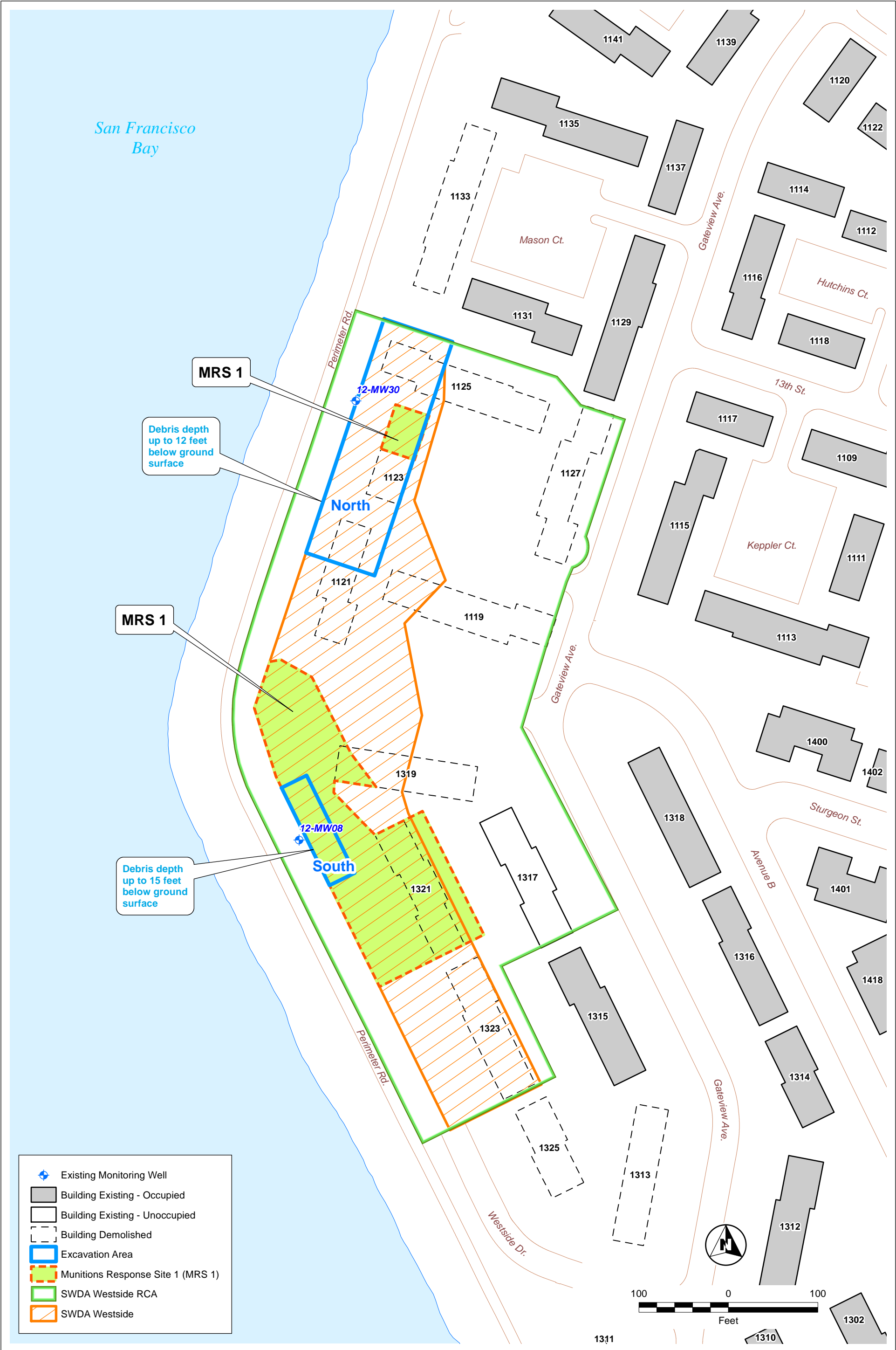
-  SWDA Westside
-  Site 12
-  Building

Note:
Background map source: ESRI® and Partners, 2020.



**Phase IV NTCRA for SWDA Westside at
IR Site 12**
Former Naval Station Treasure Island
San Francisco, CA

Figure 1
IR Site 12
SWDA Westside
Location Map



Phase IV NTCRA for SWDA Westside at
IR Site 12
Former Naval Station Treasure Island
San Francisco, CA

Figure 2
Excavation Areas

Figure 3 Project Schedule

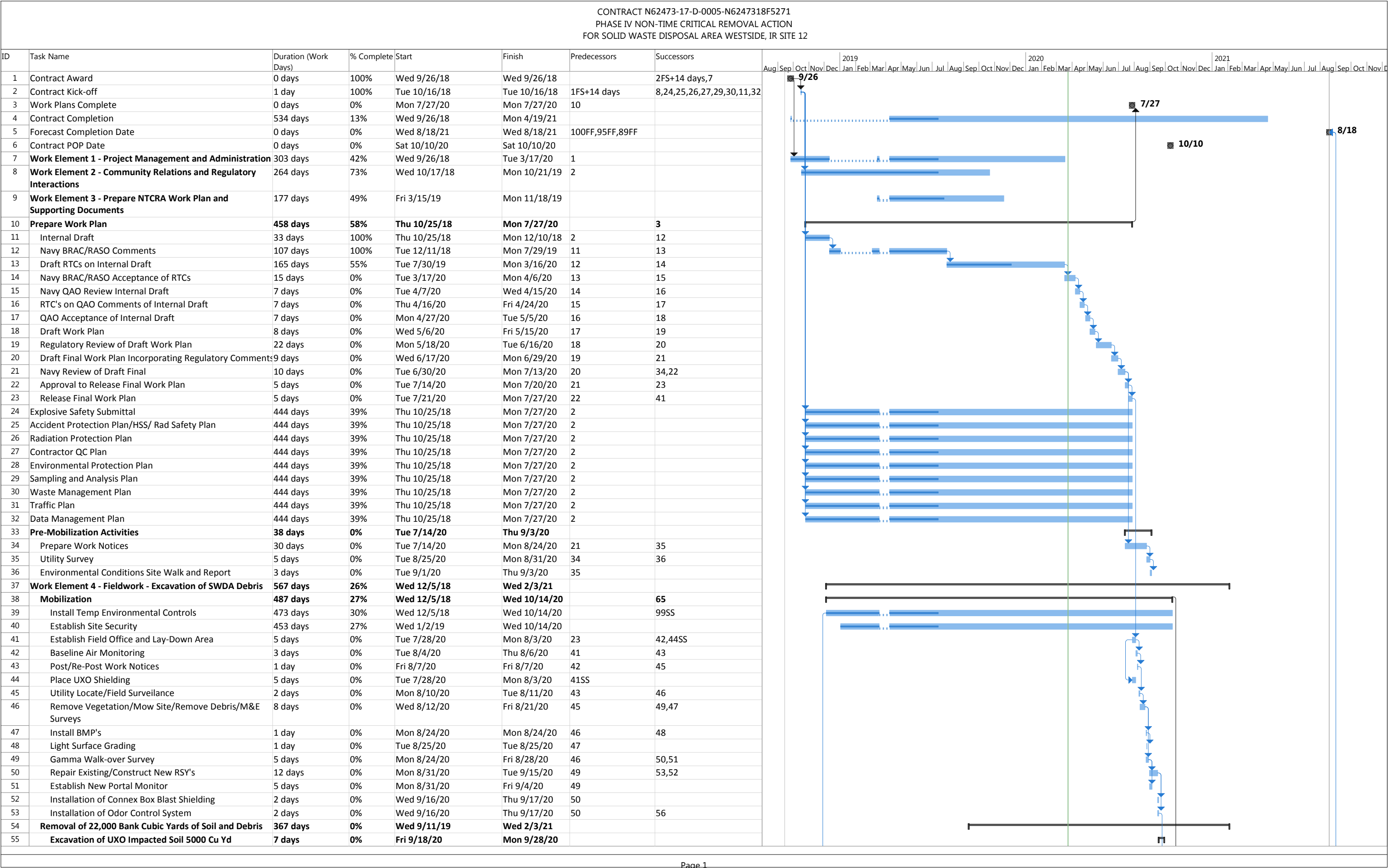


Figure 3 Project Schedule

CONTRACT N62473-17-D-0005-N6247318F5271
PHASE IV NON-TIME CRITICAL REMOVAL ACTION
FOR SOLID WASTE DISPOSAL AREA WESTSIDE, IR SITE 12

ID	Task Name	Duration (Work Days)	% Complete	Start	Finish	Predecessors	Successors																																																
56	Soil Excavation Dewatering and Inspection for UXO	5 days	0%	Fri 9/18/20	Thu 9/24/20	53	57																																																
57	Demilitarization and Debris Inspection	2 days	0%	Fri 9/25/20	Mon 9/28/20	56	58SS																																																
58	Transport Soil to RSY Pad	2 days	0%	Fri 9/25/20	Mon 9/28/20	57SS	60,73,66,67,68,69																																																
59	Excavation of Rad Impacted Soil 17000 Cu Yd	17 days	0%	Wed 9/11/19	Thu 10/3/19																																																		
60	Soil Excavation and Dewatering	17 days	0%	Tue 9/29/20	Tue 10/20/20	58	61SS+1 day																																																
61	Transport to RSY Pad	15 days	0%	Wed 9/30/20	Mon 10/19/20	60SS+1 day																																																	
62	Removal of Rad Controls	1 day	0%	Fri 12/25/20	Fri 12/25/20	100																																																	
63	Site Restoration/Installation and Grading of Final Cover	10 days	0%	Fri 12/25/20	Thu 1/7/21	100	64																																																
64	Demobilization	19 days	0%	Fri 1/8/21	Wed 2/3/21	63																																																	
65	Work Element 5 - Radiological Surveys	35 days	0%	Thu 10/15/20	Wed 12/2/20	38	96SS																																																
66	Gamma Walk-over Survey of UXO Impacted Soil	3 days	0%	Thu 10/15/20	Mon 10/19/20	58																																																	
67	Sample Colletion and Analysis	3 days	0%	Thu 10/15/20	Mon 10/19/20	58																																																	
68	Stockpiling for Pending Transport	2 days	0%	Thu 10/15/20	Fri 10/16/20	58																																																	
69	Gamma Walk-over Survey RAD Impacted Soil	15 days	0%	Thu 10/15/20	Wed 11/4/20	58	70,71																																																
70	Sample Colletion and Analysis	20 days	0%	Thu 11/5/20	Wed 12/2/20	69																																																	
71	Stockpiling for Pending Transport	20 days	0%	Thu 11/5/20	Wed 12/2/20	69	97SS+1 day,98SS+1 day																																																
72	Work Element 6 - Munitions Screening of Remaining Stockpiles	4 days	0%	Tue 9/10/19	Fri 9/13/19																																																		
73	Relocate Dewatering Plant to Stockpile Area	1 day	0%	Tue 9/29/20	Tue 9/29/20	58	74																																																
74	Soil Dewatering and Inspection for UXO	2 days	0%	Wed 9/30/20	Thu 10/1/20	73	75,76SS																																																
75	Demilitarization and Debris Inspection	2 days	0%	Fri 10/2/20	Mon 10/5/20	74																																																	
76	Stockpile Soil for Transport	3 days	0%	Wed 9/30/20	Fri 10/2/20	74SS																																																	
77	Work Element 7 - Prepare Post Construction Summary Report	163 days	0%	Tue 11/12/19	Thu 6/25/20																																																		
78	Internal Draft Post Construction Summary Report	45 days	0%	Fri 12/25/20	Thu 2/25/21	100	79																																																
79	Navy Comments	22 days	0%	Fri 2/26/21	Mon 3/29/21	78	80																																																
80	Draft RTCs on Internal Draft	7 days	0%	Tue 3/30/21	Wed 4/7/21	79	81																																																
81	Navy Acceptance of RTCs	7 days	0%	Thu 4/8/21	Fri 4/16/21	80	82																																																
82	Draft Post Construction Report	9 days	0%	Mon 4/19/21	Thu 4/29/21	81	83																																																
83	Regulatory Review of Draft Post Construction Report	22 days	0%	Fri 4/30/21	Mon 5/31/21	82	84																																																
84	Draft Final Post Construction Report Incorporating Regulatory Comments	9 days	0%	Tue 6/1/21	Fri 6/11/21	83	85																																																
85	Navy Review of Draft Final	10 days	0%	Mon 6/14/21	Fri 6/25/21	84	86																																																
86	Regulatory Review of Draft Final Post Construction Report	22 days	0%	Mon 6/28/21	Tue 7/27/21	85	87																																																
87	Approval to Release Final Post Construction Report	5 days	0%	Wed 7/28/21	Tue 8/3/21	86	88																																																
88	Release Final Post Construction Report	5 days	0%	Wed 8/4/21	Tue 8/10/21	87	89																																																
89	Update NIRIS	6 days	0%	Wed 8/11/21	Wed 8/18/21	88	5FF																																																
90	After-Action Report	55 days	0%	Tue 12/22/20	Mon 3/8/21																																																		
91	Draft After-Action Report	15 days	0%	Tue 12/22/20	Mon 1/11/21	98	92																																																
92	NOSSA/DDESB Review	22 days	0%	Tue 1/12/21	Wed 2/10/21	91	93																																																
93	RTCs and final for Navy Review	10 days	0%	Thu 2/11/21	Wed 2/24/21	92	94																																																
94	Navy Acceptance of RTCs	5 days	0%	Thu 2/25/21	Wed 3/3/21	93	95																																																
95	Final After-Action Report	3 days	0%	Thu 3/4/21	Mon 3/8/21	94	5FF																																																
96	Work Element 8 - Waste Characterization, Transportation and Disposal	60 days	0%	Thu 10/15/20	Wed 1/6/21	65SS																																																	
97	Transport and Disposal of Cal Haz	60 days	0%	Fri 11/6/20	Thu 1/28/21	71SS+1 day																																																	
98	Transport and Disposal of Class 2	32 days	0%	Fri 11/6/20	Mon 12/21/20	71SS+1 day	91,100																																																
99	Work Element 9 SWDA Westside RCA Controls and Down-posting	321 days	22%	Mon 4/8/19	Mon 6/29/20	39SS																																																	
100	Final Gamma Walkover Survey	3 days	0%	Tue 12/22/20	Thu 12/24/20	98	78,63,62,5FF																																																

AugSepOctNovDec2019JanFebMarAprMayJunJulAugSepOctNovDec2020JanFebMarAprMayJunJulAugSepOctNovDec2021JanFebMarAprMayJunJulAugSepOctNovDec

ATTACHMENT 1

Gilbane Standard Operating Procedures

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Standard Operating Procedure

Surface Soil: Sampling with Trowel or Spoon

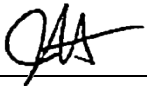
PR-TC-02.02.01.01 v2.2

Reviewed by: Clare Gilmore Date: 14 June 2013
Clare Gilmore, Senior Geologist

Reviewed by: Jim Schollard Date: 14 June 2013
Jim Schollard, Program QA/QC Manager

Approved by: Jeffrey Hess Date: 14 June 2013
Jeffrey Hess, Director Technical Services

Review / Revision History:

Version	Changes	Affects Section/Pages	Effective Date	Approval*
1.0	Initial Issue		1 Oct 2009	NA
2.0	Add provisions to retain weathered soil for non-VOC analysis, and placement of sample directly into appropriate sample containers.	Pages 2-4	14 Jun 2013	NA
2.1	Updated organization name. No other changes needed.	All	6 Aug 2014	J. Hess
2.1a	Reviewed. No changes needed.	–	14 Oct 2016	J. Hess
2.2	Minor editorial changes, and remove reference to logbook.	All	07 Jan 2019	

* Approval required for reviews and minor changes only. Substantive revisions to the technical requirements contained in the SOP require review and approval by the signatures to the SOP.

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1.0 PURPOSE AND SCOPE

The purpose of this standard operating procedure is to describe the methods and procedure for sampling of surface soils using trowels or spoons (scoops). Trowels or spoons can be used when soil matrices are composed of relatively soft and non-cemented formations, and to depths of up to 12 inches into the ground surface, depending on site conditions.

Note: Samples for VOC analysis should not be collected via trowel or spoon method. However, a trowel or spoon may be used to penetrate and expose the undisturbed material to the desired depth for sampling using an approved VOC sampling device.

1.1 LIMITATIONS

Samples from depths greater than 12 inches below the surface, or in matrices that are difficult to penetrate using a spoon or a trowel, should be collected using an alternative method (see SOP PR-TC-02.02.01.02, Surface Soils: Drive Sampler, Hand Auger or Test Pit, and SOP PR-TC-02.02.01.03, Subsurface Soils: Direct Push or Drill Rig.)

2.0 ACRONYMS AND DEFINITIONS

For purposes of this procedure, a number of terms and acronyms have the meanings defined below.

COC	chain-of-custody form
DAR	Daily Activity Report
GPS	global positioning system
HSP	Health and Safety Plan
PPE	personal protective equipment
SCL	Sample Collection Log
Scoop	Used interchangeably with “spoon.” A sample collection device with a round metal or plastic blade attached to a handle.
SOP	Standard Operating Procedure
Spoon	Used interchangeably with “scoop.” A sample collection device with a round metal blade attached to a handle.
Surface Soil	Soil that is removed from the surface (i.e., from a depth no greater than 12 inches below grade after vegetation, rocks, etc. have been cleared.
Trowel	A sample collection device with a curved and pointed metal or plastic blade attached to a handle. All trace environmental samples should be collected using stainless steel or single-use disposable plastic trowels.
VOC	volatile organic compound

3.0 PROCEDURES

The intent of these procedures is to identify the steps to be taken to assure that surface soil samples are collected efficiently and that the samples accurately reflect current conditions for the location and matrix from which they are collected.

3.1 SAMPLE COLLECTION

The following steps should be followed to collect samples of surface soil by hand using a trowel or spoon (NOTE: the words “trowel” and “spoon” are used interchangeably in the following text):

1. Don a pair of clean sampling gloves (e.g., Latex, Nitrile).
2. If desired, place plastic sheeting around the targeted location to keep sampled material in place. Use a knife or scissors to cut an access hole for the sample location.
3. If a larger volume of soil will be collected than necessary to fill the appropriate sample containers (due to heterogeneity of the soil at the sample location), place a clean stainless steel bowl large enough to hold the soil to be collected within reach of the sample location.
4. Remove any surficial debris (e.g., vegetation, rocks, etc.) from the sample location and surrounding area until the soil is exposed. Once exposed, the soil surface is designated as “at grade” or 0 inches.
5. If collecting a sample for volatile organic compounds (VOCs), use a trowel or spoon to scrape and remove the top 1/8 to 1/4 inch of weathered soil¹, if present. Otherwise, retain the material as part of the sample.
6. Collect all samples for VOC analysis first using an approved VOC sampling device.
7. Using a new/clean (i.e., disposable or decontaminated) trowel, place the point of the blade on the ground. While holding the handle of the trowel, partially rotate the blade in a clockwise/counter-clockwise motion while pushing downward at an angle until the blade is inserted to the required depth or the blade is nearly covered. Be certain that the trowel is not inserted to a depth where the soil will touch the handle or the sampler’s gloved hand.
8. With a prying motion, lift up the trowel with soil on the blade and place soil directly into the appropriate sample container(s) specified in the approved project plans or as provided by the analytical laboratory, or into the stainless steel mixing bowl.
9. Repeat steps 6 and 7 until the specified sampling depth is reached and the required amount of soil has been collected.
10. Measure the depth below grade of the sample location with an Engineer’s Rule or measuring tape to verify the sampling depth, obtain the GPS coordinates of the

¹ Weathered soil is often the top 1/8 to 1/4 inch of soil that can be affected by factors such as heat from the sun, rain, and/or foot or vehicular traffic that can result in loss of VOCs present in the shallow soil.

sample location, and record the depth/interval and GPS coordinates on the Sample Collection Log (SCL) and/or in the Daily Activity Report (DAR), as appropriate.

11. When using a stainless steel mixing bowl, homogenize the non-volatile organic compound (VOC) sample media first as specified in the approved project plans, then transfer the sample directly into the appropriate sample container(s) specified in the approved project plans or as provided by the analytical laboratory.
12. If using a wide-mouth glass jar, seal the jar with a Teflon-lined cap. If using a stainless steel sample tube, cap both ends of the tube with plastic caps lined with Teflon sheets.
13. Clean off the surface of the sample container; complete the sample label and chain-of-custody (COC) documentation; attach the label to the jar or tube; place the sample containers in Ziplock[®] Freezer Bag or equivalent and place the sample into a sample cooler maintained at 4 degrees Celsius.

3.2 EQUIPMENT

The following equipment should be used when collecting samples with a spoon or trowel:

- Decontaminated stainless steel or new disposable trowel or spoon.
- Engineer's Rule or stiff measuring tape.
- Decontaminated stainless steel mixing bowl.
- Sample container(s) as specified in the approved project plans.
- Sample collection supplies (e.g., caps, sample labels, coolers, ice, etc.).
- Plastic sheeting.
- DAR and SCLs for recording field notes and sample locations as specified in the approved project-specific project plans.
- GPS
- Digital camera/device to photograph the site, field activities and key features, as applicable
- Decontamination equipment & supplies.
- Personal protective equipment (PPE) as specified in the project-specific Health and Safety Plan (HSP).

3.3 QC SAMPLING

If non-disposable sampling equipment (e.g., trowel, mixing bowl) is re-used for multiple samples, then equipment rinsate should be collected and disposed of properly. Samples should be collected to verify that proper decontamination methods were performed between samples. A minimum of one sample per major sampling device should be collected per event, if not more frequently (i.e., one per day).

4.0 REQUIRED DOCUMENTATION

The following records generated as a result of implementation of this procedure must be maintained as quality records.

- GPS coordinates for each sample location
- Field notes provided on a DAR
- Sample Collection Form with descriptions of collected samples, depths, collection times, sample locations, etc.
- Chain-of-custody form

5.0 ATTACHMENTS/FORMS

None.


6.0 REFERENCES

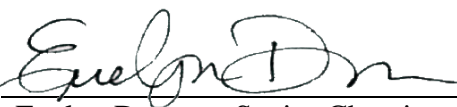
Innovative Technical Solutions, Inc. (ITSI), 2006. *Final Chemical Data Quality Management Plan*, 8(a) Remedial Action Contract Number N68711-005-D-6403. January.

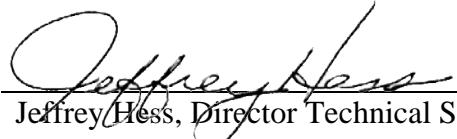
Standard Operating Procedure

Soil: Volatile Organic Compound (VOC) Sampling


PR-TC-02.02.01.05 v1.3

Reviewed by:  Date: 02 March 2010
Kristen Carlyon Peyton, Senior Chemist

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Approved by:  Date: 02 March 2010
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Review / Revision History:

Version	Changes	Affects Section/Pages	Date	Approval*
1.0	Initial Issue	NA	02 Mar 2010	NA
1.1	Addition of Terra Core System to SCDs	3.2 and 3.3	05 Oct 2011	JH
1.2	Updated procedures to incorporate SW846 Update 4 changes to preservation and hold times.	Pages 3, 5-8, 11-12	30 Jul 2013	JH
1.3	Updated organization name. No other changes necessary.	All	20 Jul 2015	

- Approval required for reviews and minor changes only. Substantive revisions to the technical requirements contained in the SOP require review and approval by the signatories to the SOP.

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1.0 PURPOSE AND SCOPE

The purpose of this standard operating procedure is to describe the requirements and procedures for the collection, packaging and transport of soil samples for the analysis of volatile organic compounds (VOCs). This SOP is focused on the preparation and preservation of soil samples in compliance with United States Environmental Protection Agency (USEPA) Method 5035 (USEPA, 1997) and updated USEPA Method 5035A (USEPA, 2002) and associated agency guidance documents such as the United States Department of Defense (USDoD) Quality Systems Manual, Version 5.0 (DoD, 2013).

Note: There are several approaches to the collection of VOC soil samples. However, the methods and their associated sampling and/or storage devices are not interchangeable. Also, each of the methods has limitations in their application and use, so the decision on which method to use should consider both the benefits and limitations of each method.

2.0 ACRONYMS AND DEFINITIONS

For purposes of this procedure, a number of terms and acronyms have the meanings defined below.

DQOs: data quality objectives

GPS: global positioning system

“high concentration”: refers to concentrations of a volatile organic compound (VOC) greater than 200 ug/kg

“low concentration”: refers to concentrations of a volatile organic compound (VOC) typically ranging from 0.5 ug/kg to 200 ug/kg

MFSD: multi-function sampling device

ml: milliliter

MS: matrix spike

MSD: matrix spike duplicate

PTFE: polytetrafluoroethylene

QAPP: Quality Assurance Project Plan

SCD: sub-coring device

USACE: United States Army Corps of Engineers

USEPA: United States Environmental Protection Agency

VOA: volatile organic analysis

VOC: volatile organic compound

3.0 PROCEDURES

The intent of this SOP is to present several acceptable procedures for the collection of VOC soil samples, consistent with EPA Method 5035A. The actual selected procedure will be specified in the project-specific QAPP. However, the following two steps are applicable to all the methods described in this SOP:

1. When the concentration of VOCs is unknown, a sample set typically is comprised of three (3) sub-samples of 5 grams of soil each.
2. To determine VOC concentrations on a dry weight basis, in the absence of an additional sample aliquot of sufficient volume to determine dry weight basis, a separate container (2-ounce jar or 40 ml vial) should be filled with soil that is co-located with each sample.

Each procedure has its own set of benefits and limitations. Selection of the appropriate procedure should be based primarily on which procedure best addresses the data quality objectives (DQOs) while considering the limitations imposed by field conditions and sampling requirements. Attachment A provides a brief discussion of some of the limitations of the specific procedures. Some of the limitations of the different procedures include:

3.1 USE OF MULTI-FUNCTION SAMPLING DEVICE

Multi-function sampling devices (MFSDs), such as the En Core[®] Sampler, act as both a coring tool and sample storage container, allowing for the collection of soil samples directly into the storage container with zero or minimal headspace. Currently approved MFSDs include the following:

- En Core[®] Sampler
- Core N' One[™] soil sampling system

The procedures for the collection of the soil samples using the approved MFSDs are provided below. A soil sample typically is comprised of a minimum of 3 MFSDs containing 5 grams of soil each.

3.1.1 Encore Sampler

3.1.1.1 Required Equipment

- En Core[®] T-Handle
- En Core[®] Samplers

3.1.1.2 Sampling Procedure

1. Assemble the En Core[®] Sampler by holding the coring body and pushing the plunger rod down until the small o-ring rests against the tabs. Depress the locking lever on the En Core[®] T-handle and place the plunger end of the coring body into the open end of the T-handle. Align the two slots on the coring body with the two locking pins in the T-handle. Twist the coring body clockwise to lock the pins in the slots. Finally, check to ensure that the coring body is locked in place.

2. Collect the En Core[®] sample by grasping the T-Handle with the open end of the coring body facing the soil sample. Using the T-Handle, push the sampler into soil until coring body is completely full. The coring body is full when the small o-ring is centered in the viewing hole in the T-Handle. Remove the sampler from the soil. Push and twist cap on the exposed end of the coring body until the ridges on the coring body snap into the grooves of the cap. Check to ensure that cap is properly secured.
3. Prepare the sample for shipment by removing the T-Handle from the En Core[®] Sampler by depressing the locking lever on T-Handle and then twist and pull the En Core[®] Sampler from T-Handle. Lock the plunger by rotating the extended plunger rod fully counter-clockwise until the wings rest firmly against the tabs. Attach the completed circular label to the cap of the coring body.
4. Return the full En Core[®] Sampler to the zipper bag. Seal the bag and immediately place the bag in an ice chest filled with ice.

3.1.2 Core N' One[™] Sampling System

3.1.2.1 Required Equipment

- Core N' One[™] Soil Handle
- Core N' One[™] Soil Capsules

3.1.2.2 Sampling Procedure

1. Remove capsule from zip lock and insert prongs into the slots of the Core N' One[™] handle. Make a one-quarter turn to the right to lock the capsule in place.
2. Unscrew the capsule cap and bore the beveled edge opening into the soil. You can determine if you have taken a full 5-gram sample by holding the capsule up to the light. The dark shading of the soil should be at the top level of the cap threads.
3. Screw the cap tight. The edge of the cap should touch the center rib of the capsule.
4. Insert the capsule into the zip lock for transport to the lab.

3.1.3 Additional Analytical Requirements

Using MFSDs require specific handling procedures for the samples after collection and by the analytical laboratory.

Field preservation options include the following:

- a) Place the samples in an iced cooler and chill and maintain the sample at the appropriate temperature for the specified method (as identified in the project-specific plans and/or as listed in SOP PR-TC-02.04.01.01, Sample Handling, Packaging and Shipping). Deliver the samples to the laboratory quickly to allow for analysis or extraction of the samples by the laboratory within 48 hours of sample collection time.
- b) Place the samples in a freezer and chill and maintain the sample at $< -7^{\circ}\text{C}$, and deliver the samples to the laboratory quickly to allow for analysis or extraction of the samples by the laboratory within 48 hours of sample collection time.

Although alternative a) is the normal procedure for most soils, alternative b) is necessary with biologically active soils potentially containing aromatic hydrocarbons (i.e., benzene).

Extraction options for the analytical laboratory include the following:

- 1) Analysis of the sample within 48 hours of sample collection.
- 2) Extrude core into unpreserved pre-tared VOA vial at the laboratory. For low-level analysis, the soil can be extruded, weighed and placed into a pre-tared VOA vial containing reagent-grade extractant water (the preferred low-level method, see Section 3.1.4 below). The laboratory then freezes the sample at $< -7^{\circ}\text{C}$. The sample must then be analyzed within 14 days of the sample collection date.
- 3) Extrude core into preserved VOA vial at the laboratory. For low-level analysis, the soil can be extruded, weighed and preserved in a pre-tared VOA vial containing sodium bisulfate solution (not recommended, see Section 3.1.4 below). For high-level analysis, the soil can be extruded, weighed, and preserved with methanol. After extrusion of the soil into an appropriate extraction fluid, the sample must be analyzed within 14 days of the sample collection date.

Additionally, in order to determine VOC concentrations on a dry weight basis, in the absence of one or more additional sample aliquots of sufficient volume to determine dry weight basis, a separate container (2-ounce jar or 40 ml vial) should be filled with soil that is co-located to each sample collection point. Sample nomenclature that links this sample to the VOC sample containers that comprise a sample set should be used.

3.1.4 Method Incompatibilities

Biologically active samples may result in the rapid loss of aromatic hydrocarbons during the initial 48 hours after sampling, and thus should be immediately chemically preserved or frozen using dry ice.

Sample preservation with sodium bisulfate solution presents four potential problems:

- i. Acid preservation may cause the chemical breakdown of certain reactive VOC compounds in the soil sample, specifically styrene, acrylonitrile, vinyl chloride, and 2-chloroethylvinyl ether.
- ii. In soil samples with a high proportion of organic material, acid preservation may generate acetone as a byproduct.
- iii. Calcareous soil samples may effervesce upon contact with sodium bisulfate solution and cause VOC loss.
- iv. Calcareous soil samples may increase the pH of the preservation fluid above 2.0, producing a sample in an unpreserved state. Accordingly, the soils at the site should be evaluated for potential problems prior to sampling activities. In cases where preservation by acid is a potential problem, an alternate sample collection method should be utilized.

3.2 USE OF SUB-CORING DEVICE AND FIELD PRESERVATION

An alternative to using MFSDs consists of using sub-coring devices (SCDs) and field preservation of the samples. Benefits of this method include less-expensive sampling devices (e.g., Lock N' LoadTM), and potentially longer holding times. However, SCDs and field preservation involves more physical handling of the samples in the field, including weighing of the samples, potentially impacting production rates. Also, preservatives for low- or high-level analysis pose significant problems themselves. This method should be used with caution and only after full consideration of the inherent problems in the method.

Approved SCDs include:

- Lock N' LoadTM sampling system
- EasyDraw Syringe[®]
- Terra Core[®] Sampler

The procedures for the collection of the soil samples using the approved SCDs are provided below. A soil sample typically is comprised of a minimum of 3 field preserved vials containing 5 grams of soil each.

3.2.1 Lock N' LoadTM Sampling System

3.2.1.1 Required Equipment

- Lock N' LoadTM soil handle
- Lock N' LoadTM soil syringe
- VOA vials with preservative (pre-tared)

3.2.1.2 Sampling Procedure

1. Insert Lock N' LoadTM Syringe into Lock N' LoadTM Handle at base opening. Turn the locking portion of the syringe into the O gram setting. Remove end cap from the Lock N' LoadTM Syringe. Position the Lock N' LoadTM Handle to the desired soil sample volume (5 grams in triplicate). To do this, slide the slot portion of the handle down the fitted track, then turn the handle one quarter to the right at the desired setting.
2. Push the syringe into the soil until the plunger portion of the syringe makes contact with the base of the Lock N' LoadTM Handle.
3. Transfer the soil from the syringe into a pre-tared 40 ml VOA vial containing the appropriate preservative, if any, by turning the Lock N' LoadTM Handle one quarter to the left (back to the fitted track) and pushing down. Slightly tilt the VOA vial to avoid splashing and potentially losing some of the preservative. Avoid getting dirt on the threads of the vial. Cap the vial and store the sample at the required temperature until time of analysis.

3.2.2 EasyDraw Syringe[®] and PowerStop Handle[®]

3.2.2.1 Required Equipment

- PowerStop Handle[®]

- EasyDraw Syringe[®]
- VOA vials with preservative (pre-tared)

3.2.2.2 Sampling Procedure

1. Load Sampling Device. Insert the EasyDraw Syringe[®] into the appropriate slot on the Powerstop Handle[®] and remove end cap from syringe. For low-level analysis, insert syringe into one of the three positions of the device for collection of 5 gram samples. Use the heavy position for dense clay, the light position for dry sandy soil, and the medium position for all others.
2. Collect Sample. Push the EasyDraw Syringe[®] into freshly exposed soil. Continue pushing until the soil column inside the syringe has forced the plunger to the stopping point. The soil plug should be flush with the mouth of the sampler. The EasyDraw Syringe[®] delivers approximately 5, 10 or 13 grams. Actual weight will be determined at the laboratory.
3. Eject Sample Into Vial. Remove the syringe from the Powerstop Handle[®]. Insert syringe into open end of a pre-tared 40-ml VOA vial containing the appropriate preservative, if any. Extrude the sample into the vial by pushing on the syringe plunger. Slightly tilt the VOA vial to avoid splashing and potentially losing some of the preservative. Avoid getting dirt on the threads of the vial. Cap the vial and store the sample at the required temperature until time of analysis.

3.2.3 Terra Core[®] Sampler

3.2.3.1 Required Equipment

- Terra Core[®] Sampler
- VOA vials with preservative (pre-tared)

3.2.3.2 Sampling Procedure

1. With the plunger seated in the handle, push the Terra Core[®] into freshly exposed soil until the sample chamber is filled. A filled chamber will deliver approximately 5 or 10 grams of soil.
2. Wipe all soil or debris from the outside of the Terra Core[®] sampler. The soil plug should be flush with the mouth of the sampler. Remove any excess soil that extends beyond the mouth of the sampler.
3. Rotate the plunger that was seated in the handle top 90° until it is aligned with the slots in the body. Place the mouth of the sampler into the 40ml VOA vial containing the appropriate preservative and extrude the sample by pushing the plunger down. Slightly tilt the VOA vial to avoid splashing and potentially losing some of the preservative, and quickly place the lid back on the 40ml VOA vial. Avoid getting dirt on the threads of the vial. Cap the vial and store the sample at the required temperature until time of analysis.

3.2.4 Additional Analytical Requirements

Using field preservation requires specific handling procedures for the samples after collection and by the analytical laboratory. Requirements vary depending on the specific preservation method, so care needs to be taken to follow the specific procedure carefully.

Preservation options include the following:

- b) Field preservation with methanol. After extruding the soil samples into pre-tared 40-ml VOA vials preserved with methanol, the vials are re-weighed in the field, and then are chilled at the required temperature in a cooler and shipped with adequate ice to ensure that the required temperature is maintained during transport to the laboratory. The samples must arrive at the laboratory within 48 hours of the sample collection time. The vials are weighed again at the stationary laboratory to verify that no methanol was lost during transport. The laboratory must prepare and analyze the samples within 14 days of the sample collection date. This technique applies only to high-level analysis so it should be used only if detection limits of greater than 200 ug/kg are appropriate.
- c) Field preservation with sodium bisulfate solution. After extruding the soil samples into pre-tared 40-ml VOA vials preserved with sodium bisulfate solution, the samples are kept chilled at the required temperature in a cooler and shipped with adequate ice to ensure that the required temperature is maintained during transport to the laboratory. The samples must arrive at the laboratory within 48 hours of the sample collection time. The laboratory must prepare and analyze the samples within 14 days of the sample collection date. This preservation technique provides detection limits to approximately 0.5 ug/kg (low-level analysis). However, sample preservation with sodium bisulfate solution presents four potential problems:
 - i. Acid preservation may cause the chemical breakdown of certain reactive VOC compounds in the soil sample, specifically styrene, acrylonitrile, vinyl chloride, and 2- chloroethylvinyl ether.
 - ii. In soil samples with a high proportion of organic material, acid preservation may generate acetone as a byproduct.
 - iii. Calcareous soil samples may effervesce upon contact with sodium bisulfate solution and cause VOC loss.
 - iv. Calcareous soil samples may increase the pH of the preservation fluid above 2.0, producing a sample in an unpreserved state. Accordingly, the soils at the site should be evaluated for potential problems prior to sampling activities. In cases where preservation by acid is a potential problem, an alternate sample collection method should be utilized.
- d) Field extraction into reagent water and analysis within 48 hours. After extruding the soil samples into pre-tared 40-ml VOA vials containing reagent-grade extractant water, the samples are kept chilled at the required temperature in a cooler and shipped with adequate ice to ensure that the required temperature is maintained during transport to the laboratory. Upon receipt of the samples, the laboratory chills the vials to the required temperature and analyzes the samples within 48 hours of the sample collection time. This technique applies to samples for low-level and high-level analysis.

Note: Extruding soil samples into vials containing reagent-grade extractant water may have an adverse effect on sample results in that water may actually promote bacterial degradation of aromatic hydrocarbons. Likewise, some VOCs may be unstable in reagent water, such as 1,1,2,2-tetrachloroethane. Accordingly, reagent water-filled vials should only be used for chemicals that do not readily biodegrade or breakdown.

- e) Field extraction into reagent water and field freezing. After extruding the soil samples into pre-tared 40-ml VOA vials containing reagent-grade extractant water, the samples are frozen to $<-7^{\circ}\text{C}$ in a cooler in the field and shipped with adequate dry ice to ensure that $<-7^{\circ}\text{C}$ is maintained during transport to the laboratory. The vials should not be frozen below -20°C due to potential problems with the vial seals. A temperature blank should be included with the samples so that the laboratory can verify the temperature upon receipt and the arrival temperature of the samples should be annotated on the chain-of-custody form. During the freezing process, the vials should be stored in a 45° angle to prevent water expansion from shattering the vials. To avoid potential rupture of the PTFE-lined septum caps, the dry ice should not directly contact the top of the vials. The laboratory must immediately freeze the sample vials to $<-7^{\circ}\text{C}$ upon receipt. The samples may be held at $<-7^{\circ}\text{C}$ for up to 7 days prior to analysis from the sample collection date. This technique applies to samples for low-level and high-level analysis. This option is used in the situations where it is difficult or impossible to deliver the samples to the laboratory within 48 hours of the sample collection time.
- f) Field extraction into reagent water and laboratory freezing. After extruding the soil samples into pre-tared 40-ml VOA vials containing reagent-grade extractant water, the samples are kept chilled at the required temperature in a cooler and shipped with adequate ice to ensure that the required temperature is maintained during transport to the laboratory. The laboratory must receive and immediately freeze the vials to $<-7^{\circ}\text{C}$ within 48 hours of the sample collection time. During the freezing process, the vials should be stored in a 45° angle to prevent water expansion from shattering the vials. The samples may be held at $<-7^{\circ}\text{C}$ for up to 7 days prior to analysis from the sample collection date. The vials should not be frozen below -20°C due to potential problems with the vial seals. This technique applies to samples for low-level and high-level analysis.

Additionally, in order to determine VOC concentrations on a dry weight basis, in the absence of one or more additional sample aliquots of sufficient volume to determine dry weight basis, a separate container (2-ounce jar or 40 ml vial) should be filled with soil that is co-located to each sample collection point. Sample nomenclature that links this sample to the VOC sample containers that comprise a sample set should be used.

3.2.5 Method Incompatibilities

3.2.4.1 Aromatic Hydrocarbons

Chemicals, such as aromatic hydrocarbons (e.g., benzene), are subject to VOC loss by biodegradation under certain sampling procedures. Accordingly, to obtain aromatic hydrocarbon soil concentrations that are representative of site conditions, only a subset of the available options are available for use. To reduce the biological activity in soil contaminated with aromatic hydrocarbons, soil samples should be preserved with methanol or sodium bisulfate solution in the field, or frozen in the field at $<-7^{\circ}\text{C}$ in non-preserved VOA vials. Under no circumstances

should soil samples contaminated with aromatic hydrocarbons be collected in the field with VOA vials containing reagent-grade extractant water. The introduction of unpreserved water to the soil sample may enhance the biodegradation of the aromatic hydrocarbons.

3.2.4.2 Chemical Reactions

Acid preservation of soil by sodium bisulfate solution, whether done in the field or in the stationary laboratory, may cause the chemical breakdown of certain compounds, including vinyl chloride. Some olefins, ketones, esters, ethers, and sulfides may react under low pH conditions, yielding analytical results that are not representative of soil conditions. Hence, precaution should be taken when preserving soil samples with sodium bisulfate solution when these compounds are present. If the degree of potential chemical reaction is unknown, an alternative procedure should be used.

3.2.4.3 Calcareous Soil

Calcareous soil samples may react upon contact with sodium bisulfate solution, causing VOC loss through effervescence and potentially cause failure of the VOA vial septum through pressure buildup. Additionally, when soil samples are highly calcareous in nature, the sodium bisulfate preservative solution may not be strong enough to reduce the pH of the aqueous solution to below 2.0, potentially rendering the preservative useless. If carbon dioxide is generated due to carbonate reaction with the acid, the carbon dioxide in the VOA vial may interfere with the detector of the analytical equipment. Hence, precaution should be taken when preserving soil samples with sodium bisulfate solution when carbonates are present.

3.3 USE OF SUB-CORING DEVICE AND EMPTY VIAL

An alternative to using SCDs with field preservation consists of field extruding the samples into clean VOA vials. Benefits of this method include use of less-expensive SCDs and potentially longer holding times. However, the empty vial method involves more physical handling of the samples in the field, including weighing of the samples, potentially impacting production rates. This method should be used with caution and only after full consideration of the inherent problems in the method.

Soil samples for the empty vial method are collected using a sub-coring device. Approved sub-coring devices include:

- Lock N' Load™ sampling system
- EasyDraw Syringe®
- Terra Core® Sampler

The procedures for the collection of the soil samples using the approved sub-coring devices are provided below. A soil sample typically is comprised of a minimum of 3 empty vials containing 5 grams of soil each.

3.3.1 Lock N' Load™ Sampling System

3.3.1.1 Required Equipment

- Lock N' Load™ soil handle

- Lock N' Load™ soil syringe
- VOA vials (pre-tared)

3.3.1.2 Sampling Procedure

1. Insert Lock N' Load™ Syringe into Lock N' Load™ Handle at base opening. Turn the locking portion of the syringe into the O gram setting. Remove end cap from the Lock N' Load™ Syringe. Position the Lock N' Load™ Handle to the desired soil sample volume (5 grams in triplicate). To do this, slide the slot portion of the handle down the fitted track, then turn the handle one quarter to the right at the desired setting.
2. Push the syringe into the soil until the plunger portion of the syringe makes contact with the base of the Lock N' Load™ Handle.
3. Transfer the soil from the syringe into a pre-tared empty 40 ml VOA vial by turning the Lock N' Load™ Handle one quarter to the left (back to the fitted track) and pushing down. Avoid getting dirt on the threads of the vial. Cap the vial and store the sample at the required temperature until time of analysis.

3.3.2 EasyDraw Syringe® and PowerStop Handle®

3.3.2.1 Required Equipment

- PowerStop Handle®
- EasyDraw Syringe®
- VOA vials (pre-tared)

3.3.2.2 Sampling Procedure

1. Load Sampling Device. Insert the EasyDraw Syringe® into the appropriate slot on the Powerstop Handle® and remove end cap from syringe. For low-level analysis, insert syringe into one of the three positions of the device for collection of 5 gram samples. Use the heavy position for dense clay, the light position for dry sandy soil, and the medium position for all others.
2. Collect Sample. Push the EasyDraw Syringe® into freshly exposed soil. Continue pushing until the soil column inside the syringe has forced the plunger to the stopping point. The soil plug should be flush with the mouth of the sampler. The EasyDraw Syringe® delivers approximately 5, 10 or 13 grams. Actual weight will be determined at the laboratory.
3. Eject Sample Into Vial. Remove the syringe from the Powerstop Handle®. Insert syringe into open end of a pre-tared empty 40-ml VOA vial. Extrude the sample into the vial by pushing on the syringe plunger. Avoid getting dirt on the threads of the vial. Cap the vial and store the sample at the required temperature until time of analysis.

3.3.3 Terra Core® Sampler

3.3.3.1 Required Equipment

- Terra Core® Sampler

- VOA vials (pre-tared)

3.3.3.2 Sampling Procedure

1. With the plunger seated in the handle, push the Terra Core® into freshly exposed soil until the sample chamber is filled. A filled chamber will deliver approximately 5 or 10 grams of soil.
2. Wipe all soil or debris from the outside of the Terra Core® sampler. The soil plug should be flush with the mouth of the sampler. Remove any excess soil that extends beyond the mouth of the sampler.
3. Rotate the plunger that was seated in the handle top 90° until it is aligned with the slots in the body. Place the mouth of the sampler into the 40ml VOA vial containing the appropriate preservative and extrude the sample by pushing the plunger down. Slightly tilt the VOA vial to avoid splashing and potentially losing some of the preservative, and quickly place the lid back on the 40ml VOA vial. Avoid getting dirt on the threads of the vial. Cap the vial and store the sample at the required temperature until time of analysis.

3.3.4 Additional Analytical Requirements

Using the empty vial method requires specific handling procedures for the samples after collection and by the analytical laboratory. Requirements vary depending on the specific preservation method, so care needs to be taken to follow the specific procedure carefully.

Handling options include the following:

- a) Field extraction and analysis within 48 hours. After extruding the soil samples into pre-tared empty 40-ml VOA vials, the samples are kept chilled at the required temperature in a cooler and shipped with adequate ice to ensure that the required temperature is maintained during transport to the laboratory. Upon receipt of the samples, the laboratory chills the vials to the required temperature and analyzes the samples within 48 hours of the sample collection time. This technique applies to samples for low-level and high-level analysis.
- b) Field extraction and laboratory freezing. After extruding the soil samples into pre-tared empty 40-ml VOA vials, the samples are kept chilled at the required temperature in a cooler and shipped with adequate ice to ensure that the required temperature is maintained during transport to the laboratory. The laboratory must receive and immediately freeze the vials to $<-7^{\circ}\text{C}$ within 48 hours of the sample collection time. The samples may be held at $<-7^{\circ}\text{C}$ for up to 14 days prior to analysis from the sample collection date. The vials should not be frozen below -20°C due to potential problems with the vial seals. This technique applies to samples for low-level and high-level analysis.
- c) Field extraction and laboratory preservation. After extruding the soil samples into pre-tared empty 40-ml VOA vials, the samples are kept chilled at the required temperature in a cooler and shipped with adequate ice to ensure that the required temperature is maintained during transport to the laboratory. The samples must arrive at the laboratory within 48 hours of the sample collection time. The laboratory then must preserve the sample using methanol. The laboratory must prepare and analyze the samples within 14

days of the sample collection date. This technique applies only to high-level analysis so it should be used only if detection limits of greater than 200 ug/kg are appropriate.

- d) Field extraction and field freezing. After extruding the soil samples into pre-tared empty 40-ml VOA vials, the samples are immediately frozen to $<-7^{\circ}\text{C}$ and shipped with adequate dry ice to ensure that $<-7^{\circ}\text{C}$ is maintained during transport to the laboratory. The laboratory must receive and immediately freeze the vials to $<-7^{\circ}\text{C}$. The samples may be held at $<-7^{\circ}\text{C}$ for up to 14 days prior to analysis from the sample collection date. The vials should not be frozen below -20°C due to potential problems with the vial seals. This technique applies to samples for low-level and high-level analysis.

Additionally, in order to determine VOC concentrations on a dry weight basis, in the absence of one or more additional sample aliquots of sufficient volume to determine dry weight basis, a separate container (2-ounce jar or 40 ml vial) should be filled with soil that is co-located to each sample collection point. Sample nomenclature that links this sample to the VOC sample containers that comprise a sample set should be used.

3.3.5 Method Incompatibilities

Biologically active samples may result in the rapid loss of aromatic hydrocarbons during the initial 48 hours after sampling, and thus should be immediately chemically preserved or frozen. Thus, the use of field freezing is required when using the empty vial method for biologically active samples suspected of containing aromatic hydrocarbons.

3.4 EQUIPMENT

Specific equipment requirements are discussed in the procedures above. In addition to the materials identified above, many of the procedures require the following:

- Appropriate clean laboratory-provided pre-tared VOA vials with selected preservative
- A scale capable of weighing 100 grams and accurate to ± 0.1 grams.

3.5 QUALITY CONTROL SAMPLES

QC samples are important to measure potential impacts to the accuracy and representativeness of the VOC samples collected. Specific QC samples important to the procedures presented in this SOP include:

- Trip blanks
- Temperature blanks
- MS/MSD samples
- Other recommended QC samples

3.5.1 Trip Blanks

Soil samples can be contaminated by diffusion of VOCs through the septum on VOA vials or through the seal on MFSDs during shipment and storage. Trip blanks are samples that accompany the environmental samples during the sampling operations and transport to the laboratory. The trip blanks may be prepared with laboratory grade methanol, sodium bisulfate solution, or reagent water, dependent on the field methods, and could also consist of laboratory-certified soil, and can be carried through sampling and handling protocols as a check on such contamination.

Generally one trip blank should be used for each field sample cooler, as specified in the QAPP. However, at a minimum, one trip blank should be used per day. The trip blank should remain unopened throughout sampling operations and labeled by date, such as TB022510-01 (for cooler no. 1 trip blank sent on February 25, 2010), or similar nomenclature as specified in the QAPP.

3.5.2 Temperature Blanks

Temperature blanks should be used so that the laboratory can verify the temperature upon receipt of the samples. In the case of field freezing, the temperature blanks should be frozen upon arrival at the laboratory. The temperature of the samples upon arrival should be annotated on the chain-of-custody form and also mentioned in the laboratory narrative that accompanies the analytical results. A temperature blank routinely consists of a vial filled with blank water (deionized water is acceptable).

3.5.3 Matrix Spike and Matrix Spike Duplicate Samples

An important measure of the performance of an analytical method relative to the specific sample matrix of interest is the matrix spike and matrix spike duplicate (MS/MSD). The MS/MSD is an important aspect of an overall quality assurance program for a project. When soil sampling, a MS/MSD sample should be collected for each analytical method at a frequency of five (5) percent of the field samples, unless otherwise specified in the site-specific QAPP. The MS/MSD sample should be prepared in a fashion similar to the other samples. Samples taken for MS/MSD should be labeled as such and specified on the chain-of-custody form. The primary purpose of MS/MSD analyses is to establish the applicability of the overall analytical approach to the specific sample matrix from the site.

Each sample set designated for MS/MSD analysis should be collected in triplicate (e.g., if using En Core[®] Samplers, then nine 5-gram sample containers would be required).

3.5.4 Other Field Quality Control Samples

Field quality control samples to demonstrate the integrity of the field samples should also be collected as required by the site-specific QAPP. Field duplicates should be collected at a minimum frequency of 10 percent of the samples. Field blanks and equipment rinsate blanks, if required, should be collected each day, or as specified in the site-specific QAPP.

4.0 REQUIRED DOCUMENTATION

The following records generated as a result of implementation of this procedure must be maintained as quality records.

- GPS or survey coordinates for each sample location
- Chains of Custody
- Sample collection logs, including recorded weights of samples placed in vials.
- Field notes

5.0 ATTACHMENTS

Attachment A. Limitations to VOC Soil Sampling Procedures

Attachment B. Various Options for the Collection of Soil Samples for VOC Analyses

6.0 FORMS

None.

7.0 REFERENCES

California Environmental Protection Agency, Department of Toxic Substances Control (DTSC), 2004. *Guidance Document for the Implementation of USEPA Method 5035: Methodologies for Collection, Preservation, Storage, and Preparation of Soils to be Analyzed for Volatile Organic Compounds*. November.

United States Department of Defense (USDoD), 2013. *Quality Systems Manual, Version 5.0* July.

United States Environmental Protection Agency (USEPA), Office of Solid Waste, 1997. *Method 5035, Closed-System Purge-and-Trap Extraction for Volatile Organics in Soil and Waste Samples*. June. Part of methods compendium SW-846, *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Update III* (Method 5035).

USEPA, Office of Solid Waste, 2002. *Method 5035A, Closed-System Purge-and-Trap Extraction for Volatile Organics in Soil and Waste Samples*. July. Part of methods compendium SW-846, *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Update III* (Method 5035). Updated method.

ATTACHMENT A. LIMITATIONS TO VOC SOIL SAMPLING PROCEDURES

Each of the sampling and preservation procedures has their own set of benefits and limitations. Selection of the appropriate procedure should be based primarily on which procedure best addresses the data quality objectives (DQOs) while considering the limitations imposed by field conditions and sampling requirements. Some of the limitations of the different procedures include:

- Multi-Functional Sampling Devices. When the MFSDs are received by the laboratory, the soil subcores within the MFSDs are extruded into VOA vials for analysis. As the soil subcores pass from the MFSDs to the VOA vials during the extrusion process, the soil subcores are open to ambient air and VOC loss could occur. This VOC loss could yield analytical results that are potentially biased low. Users of MFSDs must recognize this limitation when evaluating the data quality objectives for their project.
- Field Chemical Preservation. Chemical preservation of VOA vials in the field with sodium bisulfate solution (low-level analysis) or methanol (high-level analysis) is considered to yield better representativeness for VOC analysis of soil. The introduction of chemical preservatives in the field inhibits VOC loss by biodegradation. Also, VOC loss due to sample handling at the laboratory is minimized in that the sample aliquot is placed in a VOA vial in the field that contains the required preservative, sodium bisulfate or methanol, and stir stick and therefore each VOA vial does not need to be reopened by the laboratory as syringes may be used through the septum cap. However, field preservation has significant limitations that may ultimately prohibit its use on some sites. One issue is that storage of methanol used in preservation may absorb non-site specific VOCs during storage and transport. When the concentration levels of VOCs are not known to be low or high (above 200 ug/kg), multiple VOCs vials containing sodium bisulfate, methanol, and no preservative, will need to be processed in the field for each sample location.
- Empty Vial Technique. The extractant fluid, whether methanol, sodium bisulfate solution, or reagent water, must be added by the stationary laboratory to the VOA vials after the soil has been sealed into the vials in the field. To do this, the PTFE-lined septum caps must be pierced for the introduction of the extraction fluid into the VOA vials. After the introduction of the extraction fluid, the vials must be stirred or sonicated to promote the partitioning of the VOCs into the extraction fluid. Upon completion of the stirring or sonication, the sample is then analyzed for VOC concentration. During the stirring or sonication, VOCs can escape from the VOA vial through the pierced septum. Hence, the Empty Vial Technique may potentially yield analytical results that are biased low. Users of the Empty Vial Technique must recognize this limitation when evaluating the data quality objectives for their project.

ATTACHMENT B. VARIOUS OPTIONS FOR THE COLLECTION OF SOIL SAMPLES FOR VOC ANALYSES

Option	Sample Collection	Sample Container	Field Preservation	Laboratory Procedure	Holding Time (DTSC/EPA)	Reporting Limit
3.1(a)1	MFSD ⁽¹⁾	MFSD	Cool to 4 ± 2°C	Analyze w/in 48 hrs of sample collection	48 hours	Low/High
3.1(a)2	MFSD	MFSD	Cool to 4 ± 2°C	Extrude sample w/in 48 hrs into unpreserved VOA and freeze to < 7°C	7 days / 14 days	Low/High
3.1(a)3	MFSD	MFSD	Cool to 4 ± 2°C	Extrude sample w/in 48 hrs into methanol preserved VOA and cool to 4±2°C	14 days	High
3.1(a)3	MFSD	MFSD	Cool to 4 ± 2°C	Extrude sample w/in 48 hrs into sodium bisulfate preserved VOA and cool to 4±2°C	14 days	Low/High
3.1(a)3	MFSD	MFSD	Cool to 4 ± 2°C	Extrude sample w/in 48 hrs into reagent-grade extractant water VOA and cool to 4±2°C	14 days	Low/High
3.1(b)	MFSD	MFSD	Freeze to < 7°C	Use of any of the above laboratory procedures and associated holding times	— ⁽³⁾ / See above	See above
3.2(a)	SCD	VOA	Preserve with methanol and cool to 4 ± 2°C	Cool sample to 4±2°C	14 days	High
3.2(b)	SCD	VOA	Preserve with sodium bisulfate and cool to 4 ± 2°C	Cool sample to 4±2°C	14 days	Low/High
3.2(c)	SCD	VOA	Extract into reagent-grade water and cool to 4 ± 2°C	Analyze w/in 48 hrs of sample collection	48 hours	Low/High
3.2(d)	SCD	VOA	Extract into reagent-grade water and freeze to < 7°C	Freeze sample to < 7°C	7 days / 14 days	Low/High
3.2(e)	SCD	VOA	Extract into reagent-grade water and cool to 4 ± 2°C	Freeze sample w/in 48 hrs to < 7°C	7 days / 14 days	Low/High
3.3(a)	SCD	VOA	Cool to 4 ± 2°C	Cool to 4±2°C	48 hours	Low/High
3.3(b)	SCD	VOA	Cool to 4 ± 2°C	Freeze sample w/in 48 hrs to < 7°C	14 days	Low/High
3.3(c)	SCD	VOA	Cool to 4 ± 2°C	Extrude sample w/in 48 hrs into methanol preserved VOA and cool to 4±2°C	14 days	High
3.3(d)	SCD	VOA	Freeze to < 7°C	Freeze to < 7°C	14 days	Low/High

⁽¹⁾ Multi-Function Sampling Device (e.g., EnCore Sampler).

⁽²⁾ Sub-Coring Device (e.g., Lock N' Load sampling system)


⁽³⁾ Freezing of MFSDs are not recommended by DTSC due to the potential for damage to their seals.

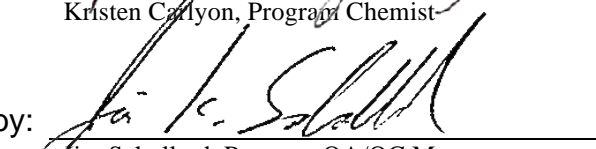


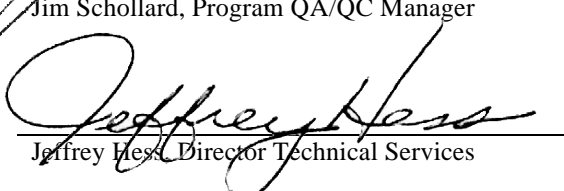
Standard Operating Procedure

Sample Handling, Packaging and Shipping

PR-TC-02.04.01.01 v2.3

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Review / Revision History:

Version	Changes	Affects Section/Pages	Effective Date	Approval*
1.0	Initial Issue	NA	30 Sep 2009	NA
1.1	Added perchlorate to the Sample Preservation and Storage Requirements Table.	Attachment A	24 Feb 2010	–
1.2	Remove references to SOPs currently under revision for inclusion of CLP procedures.	Pgs 4-5	06 Aug 2010	–
2.0	Added in SW846 Revision 4	Attachment A	13 Jun 2013	NA
2.1	Updated organization name. No other changes needed.	All	6 Aug 2014	–
2.2	Minor text changes, added methods to Attachment A	Pages 4-6, Attachment A	10 Apr 2017	–
2.3	Minor text changes, revised holding times in Attachment A	Pages 3,4 & 6, Attachment A	26 Oct 2018	

* Approval required for reviews and minor changes only. Substantive revisions to the technical requirements contained in the SOP require review and approval by the signatures to the SOP.

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1.0 PURPOSE

The objective of this procedure is to establish a uniform method for the handling of environmental samples. This includes using the appropriate sample containers and preservatives, following correct chain-of-custody procedures, and using appropriate sample shipment methods.

2.0 SCOPE AND APPLICABILITY

This procedure will be used during the collection and handling of all types of environmental media, including but not limited to, groundwater, surface water, soil, sediment, and air samples.

This procedure applies to the shipping and packing of all non-hazardous samples. Non-hazardous samples are those that do not meet any hazard class definitions found in 49 CFR 107-178, including materials designated as Class 9 materials and materials that represent Reportable Quantities (hazardous substances). In general, most soil, air, and aqueous samples do not meet any of DOT's hazardous materials definitions. However, samples for which screening has shown a potential hazard sufficient to meet a DOT definition or that are derived from a source known or suspected to meet a DOT definition must be packaged and shipped in accordance with applicable DOT and/or IATA requirements.

3.0 ACRONYMS AND DEFINITIONS

For purposes of this procedure, a number of terms and acronyms have the meanings defined below.

°C:	degrees Celcius
Bubble wrap:	Plastic sheeting with entrained air bubbles; used for protective packaging purposes.
CFR:	Code of Federal Regulations
CLP:	Contract Laboratory Program
COC:	Chain-of-custody
Cooler:	Any hard-sided insulated container meeting DOT or IATA packaging requirements.
DOT:	U.S. Department of Transportation.
IATA:	International Air Transport Association.
Packing material:	Styrofoam beads ("peanuts"), or equivalent
PPE:	Personal protective equipment.
QAPP:	Quality Assurance Project Plan
Shipping container:	<i>see</i> Cooler
VOA vial:	40-mL glass vial used for the collection of samples for volatile organic analysis.

4.0 EQUIPMENT AND MATERIALS

Equipment and materials that may be required to implement this SOP include the following:

- Bubble wrap
- Packing material
- Tape (packing tape, duct tape, or other tear-resistant material)
- Large plastic trash bags
- Ziploc bags (freezer grade, gallon and quart sizes)
- Shipping containers (e.g. coolers)
- Sample container(s) as specified in the approved project plans
- Ice
- Custody seals
- “This Side Up” arrows
- Address labels and/or airbills
- Chain-of-Custody forms
- Sample Collection Forms, Daily Activity Reports, activity-specific sampling forms
- Black waterproof pen (e.g., fine-point Sharpie marker).

5.0 PROCEDURE

5.1 GENERAL

The following method outlines general considerations for sample handling in the field and maintaining sample custody after collection.

Environmental samples are collected in the field in order to evaluate whether conditions in soil gas, soil, surface water, groundwater or atmosphere are hazardous. These samples therefore, should be handled with the utmost care to maintain sample integrity, so that analytical data represent field conditions as closely as possible. In addition, sample care, custody, and control are extremely important for establishing that sample integrity was maintained between field crews and the laboratory.

General considerations for handling during sampling are:

- Always wear proper PPE when handling samples.

- Wrap sample container in a way that is both protective of the sample container and other surrounding sample containers.
- Document all collection procedures thoroughly in sampling forms (e.g. Sample Collection Form) and general field notes in the Daily Activity Report (or field logbook, when applicable). There is never “too much information”.

Samples must be stabilized for transport from the field to the laboratory through the use of the proper sample containerization and preservation. This is due to the potential chemical and/or biological degradation that may occur after samples are collected. Typical sample containerization and preservation are presented in Appendix A. Unless otherwise indicated in the site-specific QAPP, sample containers should be cooled immediately after completion of sampling and maintained at a temperature not to exceed the temperature specified in Attachment A until received by the laboratory.

5.2 SAMPLE CONTAINERIZATION AND PRESERVATION

The appropriate sample container types, volumes, preservatives, and holding time requirements for soil and groundwater samples for the most commonly requested analyses are listed in Attachment A, Sample Preservation and Storage Requirements.

Methods of sample preservation are intended to retard biological action, retard hydrolysis, and reduce sorption effects. Preservation methods are generally limited to pH control, chemical addition, refrigeration, and protection from light.

All sample containers will be properly labeled and monitored for temperature control in the field and during laboratory transport and storage. Temperature blanks will be used in all coolers containing samples requiring preservation at reduced temperature.

5.3 SAMPLE IDENTIFICATION AND LABELS

All samples will be properly labeled to prevent misidentification of samples. Generally, preprinted sample labels are encouraged to enhance legibility and reduce transcription errors at the laboratory. The label will be affixed to the sample container prior to transportation to the laboratory and will generally contain the following information (except when using EPA Contract Laboratory Program [CLP]):

- Project name, number, and location
- Site name
- Name of collector
- Date and time of collection
- Sample identification number
- Preservative, if any
- Requested test methods or analyses.

Labels intended for CLP projects will not include any information which identifies the project, location, or site. See the site-specific QAPP for any additional sample identification protocols.

5.4 CHAIN OF CUSTODY

Chain-of-custody (COC) procedures are implemented to ensure that all samples are traceable from the time that they are collected until they, or their derived data, are used. A sample is considered to be “in custody” under the following conditions:

- It is in personal possession.
- It is in personal view after being in personal possession.
- It was in personal possession when it was properly secured.
- It is in a designated secure area.

Sample custody will be documented through the use of COC forms. These forms will be used to track sample custody from the point of sample collection through sample disposal. The security of samples will be ensured by the use of the procedures described below.

5.4.1 Chain-of-Custody Forms

A COC form will be filled out for and will accompany every group of samples sent to the analytical laboratory, to document sample care, custody, and control from the time of collection to sample receipt. See SOP PR-TC-01.04.05.00 for a copy of the latest COC form and on how to properly complete the form.

The following information will be recorded on the COC form:

- COC form number
- Company name, address, and telephone number
- Company contact person
- Laboratory name, address, and telephone number
- Laboratory contact person
- Sample identification
- Date and time of collection
- Sampler's name
- Analytical method(s) requested
- Sample volume (e.g., three 40-milliliter [mL] vials)
- Sample matrix (e.g., soil or groundwater)
- Preservative (e.g., hydrochloric acid [HCl])
- Request for matrix spike analysis or other QC analysis
- Signatures of individuals releasing and accepting samples

- Times of release and acceptance of samples
- Air bill number if shipping by commercial courier
- Any comments to identify special conditions or requests.

5.4.2 Custody Seals

Custody seals will be used when samples are shipped via courier service, and must be placed on the shipping container (cooler) so that the seals have to be broken before the container can be opened. The seal must be signed and dated by the field personnel. Custody seals are not deemed necessary when the samples will be in the continuous possession of project, field, or laboratory personnel.

5.5 PACKAGING FOR SHIPMENT

Samples will be packaged for shipment as follows:

- Use tape to seal off the cooler drain on the inside and outside to prevent leakage.
- Place packing material (bubble wrap) on the bottom of the shipping container (cooler) to provide a soft impact surface.
- Place a 55-gallon or equivalent plastic bag into the cooler (to minimize the possibility of leakage during transit).
- Place each sample bottle or set of volatile organic analysis (VOA) vials in a separate plastic bag and seal the bag. Squeeze air from the bag before sealing.
- Starting with the largest glass containers, wrap each container with sufficient bubble wrap to ensure the best chance to prevent breakage of the container.
- Pack the largest glass containers in bottom of the cooler, placing packing material between the containers to partially cover the sample containers (more than halfway) to avoid breakage from bumping. Cardboard separators may be placed between the containers at the discretion of the shipper.
- Double-bag ice chips or cubes in gallon or quart freezer-grade Ziploc plastic bags and wedge the ice bags between the sample containers.
- Add bagged ice across the tops of the samples.
- Continue filling the shipping container in the same manner (e.g., using bubble-wrap and ice) with smaller sample containers/vials.
- When the container is sufficiently full (or all samples have been packed), seal the inner protective plastic bag (with twist-ties and/or packing tape and custody seal), and place additional packing material on top of the bag to minimize shifting of containers during shipment.
- Tape a gallon Ziploc bag to the inside of the cooler lid, place one copy of the completed COC document for the shipment inside, and seal the bag shut.

- Tape the shipping container (cooler) shut using packing tape, duct tape, or other tear-resistant adhesive strips. Taping should be sufficient to ensure that the lid will not open during transport.
- In situations where samples will not be in the continuous possession of project, field, or laboratory personnel, place custody seals on two separate portions of the cooler, to provide evidence that the lid has not been opened prior to receipt by the intended recipient.

5.5.1 Field Quality Control

A second-party quality control (QC) check of the cooler contents is the ultimate responsibility of the field team lead.

The cooler contents will be reconciled against the COC before submitting the cooler to the laboratory as follows:

- Check that each sample in the cooler is accounted for on the COC.
- Conversely, check that each sample on the COC is in the cooler.
- Check that the label information for each container matches the COC (i.e., correct date, time, and sample identification)

The QC should be documented with an initial in the upper left-hand corner of the COC.

5.5.2 Labeling

Label the shipping container/cooler as follows:

- Attach a “This Side Up” arrow securely to each side of the cooler. Affix "Fragile" or other labels on the cooler as appropriate.
- Attach a label with the name and address of the receiver and the shipper to the top of the cooler.
- If the cooler is to be shipped by overnight carrier, attach a properly completed airbill to the top of the cooler. Alternatively, affix the properly completed airbill to the cooler handle with a FedEx-provided label mount and zip tie.

6.0 ATTACHMENTS

- Attachment A: Sample Preservation and Storage Requirements

7.0 FORMS

The following forms are attached:

- None

8.0 REFERENCES

U.S. Department of Transportation Regulations, 49 CFR Parts 108-178.

International Air Transport Association (IATA), Dangerous Goods Regulations, current edition.

Attachment A

Sample Preservation and Storage Requirements PR-TC-02.04.01.01

Matrix	Analytical Group	Analytical Method	Containers (number, size and type)	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time (preparation/analysis)
Water	VOC	Gasoline Range Organics (GRO) 8015B	3 X 40 mL VOA vials with PTFE septa	HCL to pH < 2 / $4 \pm 2^{\circ}\text{C}$	14 days analysis
Water	VOC	Gasoline Range Organics (GRO) 8015C	3 X 40 mL VOA vials with PTFE septa	HCL to pH < 2 / $\leq 6^{\circ}\text{C}$	14 days analysis
Water	VOC	Gasoline Range Organics (GRO) 8015D	3 X 40 mL VOA vials with PTFE septa	HCL to pH < 2 / $\leq 6^{\circ}\text{C}$	14 days analysis
Water	VOC	GCMS VOCs 8260B	3 X 40 mL VOA vials with PTFE septa	HCL to pH < 2 / $4 \pm 2^{\circ}\text{C}$	14 days analysis (7 days unpreserved)
Water	VOC	GCMS VOCs 8260C	3 X 40 mL VOA vials with PTFE septa	HCL to pH < 2 / $\leq 6^{\circ}\text{C}$	14 days analysis (7 days unpreserved) ^{a,b}
Water	VOC	GC VOCs 8021B (SW846 Update III)	3 X 40 mL VOA vials with PTFE septa	HCL to pH < 2 / $4 \pm 2^{\circ}\text{C}$	14 days analysis (7 days unpreserved)
Water	VOC	GC VOCs 8021B (SW846 Update IV)	3 X 40 mL VOA vials with PTFE septa	HCL to pH < 2 / $\leq 6^{\circ}\text{C}$	14 days analysis (7 days unpreserved) ^b
Water	SVOC	Phenols 8041A (SW846 Update III)	2 X 1.0 liter amber glass with PTFE-lined lid	$4 \pm 2^{\circ}\text{C}$	7 days extraction 40 days analysis (after extraction)
Water	SVOC	Phenols 8041A (SW846 Update IV)	2 X 1.0 liter amber glass with PTFE-lined lid	$\leq 6^{\circ}\text{C}$	7 days extraction 40 days analysis (after extraction)
Water	SVOC	Organochlorine Pesticides 8081A	2 X 1.0 liter amber glass with PTFE-lined lid	$4 \pm 2^{\circ}\text{C}$	7 days extraction 40 days analysis (after extraction)
Water	SVOC	Organochlorine Pesticides 8081B	2 X 1.0 liter amber glass with PTFE-lined lid	$\leq 6^{\circ}\text{C}$	7 days extraction 40 days analysis (after extraction)
Water	SVOC	Organochlorine Pesticides by high resolution 1699	2 X 1.0 liter amber glass with PTFE-lined lid	$\leq 6^{\circ}\text{C}$	7 days extraction 40 days analysis (after extraction)
Water	SVOC	Polychlorinated Biphenyls (PCBs) 8082	2 X 1.0 liter amber glass with PTFE-lined lid	$4 \pm 2^{\circ}\text{C}$	7 days extraction 40 days analysis (after extraction)
Water	SVOC	PCB Congeners by high resolution 1668C	2 X 1.0 liter amber glass with PTFE-lined lid	$\leq 6^{\circ}\text{C}$	7 days extraction 40 days analysis (after extraction)
Water	SVOC	Polychlorinated Biphenyls (PCBs) 8082A	2 X 1.0 liter amber glass with PTFE-lined lid	$\leq 6^{\circ}\text{C}$	None

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Sample Preservation and Storage Requirements PR-TC-02.04.01.01

Matrix	Analytical Group	Analytical Method	Containers (number, size and type)	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time (preparation/analysis)
Water	SVOC	Organophosphorus Pesticide 8141A	2 X 1.0 liter amber glass with PTFE-lined lid	$4 \pm 2^{\circ}\text{C}$	7 days extraction 40 days analysis (after extraction)
Water	SVOC	Organophosphorus Pesticide 8141B	2 X 1.0 liter amber glass with PTFE-lined lid	$\leq 6^{\circ}\text{C}$	7 days extraction 40 days analysis (after extraction)
Water	SVOC	Chlorinated Herbicides 8151A (SW846 Update III)	2 X 1.0 liter amber glass with PTFE-lined lid	$4 \pm 2^{\circ}\text{C}$	7 days extraction 40 days analysis (after extraction)
Water	SVOC	Chlorinated Herbicides 8151A (SW846 Update IV)	2 X 1.0 liter amber glass with PTFE-lined lid	$\leq 6^{\circ}\text{C}$	7 days extraction 40 days analysis (after extraction)
Water	SVOC	GCMS SVOC 8270C	2 X 1.0 liter amber glass with PTFE-lined lid	$4 \pm 2^{\circ}\text{C}$	7 days extraction 40 days analysis (after extraction)
Water	SVOC	GCMS SVOC 8270D	2 X 1.0 liter amber glass with PTFE-lined lid	$\leq 6^{\circ}\text{C}$	7 days extraction 40 days analysis (after extraction)
Water	SVOC	Dioxins and Furans 8280A; 8290	2 X 1.0 liter amber glass with PTFE-lined lid	$4 \pm 2^{\circ}\text{C}$, store in the dark	30 days extraction 45 days analysis (after extraction)
Water	SVOC	Dioxins and Furans 8280B; 8290A	2 X 1.0 liter amber glass with PTFE-lined lid	$\leq 6^{\circ}\text{C}$	None
Water	SVOC	Polycyclic Aromatic Hydrocarbons 8310 (SW846 Update III) ; 8270CSIM	2 X 1.0 liter amber glass with PTFE-lined lid	$4 \pm 2^{\circ}\text{C}$	7 days extraction 40 days analysis (after extraction)
Water	SVOC	Polycyclic Aromatic Hydrocarbons 8310 (SW846 Update IV); 8270DSIM	2 X 1.0 liter amber glass with PTFE-lined lid	$\leq 6^{\circ}\text{C}$	7 days extraction 40 days analysis (after extraction)
Water	SVOC	PFOS/PFOA 537 (like 8321B)	250 mL polypropylene bottle with poly lid	Trizma® buffer 5g/L / $< 6^{\circ}\text{C}$	14 days extraction 28 days analysis (after extraction)
Water	SVOC	Nitroaromatics and Nitroamines 8330A; 8330B	2 X 1.0 liter amber glass with PTFE-lined lid	$\leq 6^{\circ}\text{C}$	7 days extraction 40 days analysis (after extraction)
Water	SVOC	Diesel and Oil Range Organics (DRO and ORO) 8015B	2 X 1.0 liter amber glass with PTFE-lined lid	$4 \pm 2^{\circ}\text{C}$	7 days extraction 40 days analysis (after extraction)
Water	SVOC	Diesel and Oil Range Organics (DRO and ORO) 8015C	2 X 1.0 liter amber glass with PTFE-lined lid	$\leq 6^{\circ}\text{C}$	7 days extraction 40 days analysis (after extraction)

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Sample Preservation and Storage Requirements PR-TC-02.04.01.01

Matrix	Analytical Group	Analytical Method	Containers (number, size and type)	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time (preparation/analysis)
Water	SVOC	Diesel and Oil Range Organics (DRO and ORO) 8015D	2 X 1.0 liter amber glass with PTFE-lined lid	$\leq 6^{\circ}\text{C}$	7 days extraction 40 days analysis (after extraction)
Water	Metals	ICP-AES Metals 6010B; 6010C	1 X 500 mL plastic	HNO_3 to $\text{pH} < 2$	6 months analysis
Water	Metals	ICP-MS Metals 6020; 6020A	1 X 500 mL plastic	HNO_3 to $\text{pH} < 2$	6 months analysis
Water	Metals	Mercury by CVAA 7470A (SW846 Update III)	1 X 500 mL plastic	HNO_3 to $\text{pH} < 2$; $4 \pm 2^{\circ}\text{C}$	28 days analysis
Water	Metals	Mercury by CVAA 7470A (SW846 Update IV)	1 X 500 mL plastic	HNO_3 to $\text{pH} < 2$; $\leq 6^{\circ}\text{C}$	28 days analysis
Water	Metals	Methyl Mercury 1630	1 X 250 mL fluoropolymer or PETG	HCl (high-grade) to 0.4% in field or lab in 48 hrs; $\leq 6^{\circ}\text{C}$	6 months analysis
Water	Metals	Low Level Mercury 1631A	1 X 250 mL fluoropolymer or PETG	HCl (high-grade) to 0.4% in field or lab in 48 hrs; $\leq 6^{\circ}\text{C}$; then BrCl in lab in 28 days	90 days analysis
Water	Inorganic	Hexavalent Chromium 7196A; 7199	1 X 250 mL plastic	$4 \pm 2^{\circ}\text{C}$	24 hours analysis ^f
Water	Inorganic	Hexavalent Chromium 7196A; 7199	1 X 250 mL plastic	$\leq 6^{\circ}\text{C}$	24 hours analysis ^f
Water	Inorganic	Anions by IC 300.0 / 9056A (S846 Update III)	1 X 250 mL plastic	$4 \pm 2^{\circ}\text{C}$	48 hours for nitrate, nitrite, and orthophosphate analysis 28 days for chloride, sulfate, bromide, and fluoride analysis
Water	Inorganic	Anions by IC 300.0 / 9056A (SW846 Update IV)	1 X 250 mL plastic	$\leq 6^{\circ}\text{C}$	48 hours for nitrate, nitrite, and orthophosphate analysis 28 days for chloride, sulfate, bromide, and fluoride analysis
Water	Inorganic	Nitrate and Nitrite as N Total 353.2	1 X 250 mL plastic	H_2SO_4 to $\text{pH} < 2$ / $4 \pm 2^{\circ}\text{C}$	28 days analysis
Water	Inorganic	Kjeldahl Nitrogen 351.4 / SM 4500NH3-C	1 X 250 mL plastic	H_2SO_4 to $\text{pH} < 2$ / $4 \pm 2^{\circ}\text{C}$	28 days analysis

Attachment A

Sample Preservation and Storage Requirements PR-TC-02.04.01.01

Matrix	Analytical Group	Analytical Method	Containers (number, size and type)	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time (preparation/analysis)
Water	Inorganic	Chemical Oxygen Demand (COD) 410.4 / SM 5220D	1 X 250 mL plastic	H ₂ SO ₄ to pH < 2 / 4 ± 2°C	28 days analysis
Water	Inorganic	Alkalinity SM 2320B / 310.1	1 X 250 mL plastic	4 ± 2°C	14 days analysis
Water	Inorganic	Total Dissolved Solids (TDS) SM 2540C / 160.1	1 X 250 mL plastic	4 ± 2°C	7 days analysis
Water	Inorganic	pH SM 4500-H+B	1 X 250 mL plastic	None	15 minutes analysis
Water	Inorganic	pH 150.1	1 X 250 mL plastic	None	24 hour analysis
Water	Inorganic	Conductivity SM 2510B / 120.1	1 X 250 mL plastic	4 ± 2°C	28 days analysis
Water	Radiochem	Gross Alpha/Gross Beta 900.0	500-mL glass or plastic	HNO ₃ to pH < 2	6 months analysis ^e
Water	Radiochem	Gamma-Emitting Radionuclides 901.1	2 X 1-liter glass or plastic	HNO ₃ to pH < 2	6 months analysis ^e
Water	Radiochem	Radium-226 by Radon Emanation 903.1	2 X 1 liter glass or plastic	HNO ₃ to pH < 2	6 months analysis ^e
Water	Radiochem	Gamma Radioassay HASL300 GA-01-R	2 X 1 liter glass or plastic	HNO ₃ to pH < 2	6 months analysis ^e
Water	Radiochem	Radium-228 EPA 904.0	2 X 1 liter glass or plastic	HNO ₃ to pH < 2	6 months analysis ^e
Water	Radiochem	Strontium-90 905.0	2 X 1 liter glass or plastic	HNO ₃ to pH < 2	6 months analysis ^e
Water	Radiochem	Tritium 906.0	2 X 1 liter glass or plastic	None	6 months analysis ^e
Water	Radiochem	Plutonium 238 and 239/240 HASL 300-Pu-11	2 X 1 liter glass or plastic	HNO ₃ to pH < 2	6 months analysis ^e
Water	Radiochem	Uranium-234, -235, and -238 HASL 300 U-02-RC	2 X 1 liter glass or plastic	HNO ₃ to pH < 2	6 months analysis ^e

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Sample Preservation and Storage Requirements PR-TC-02.04.01.01

Matrix	Analytical Group	Analytical Method	Containers (number, size and type)	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time (preparation/analysis)
Soil	VOC	Gasoline Range Organics (GRO) 8015B	3 X 5g TerraCore® or equivalent	4 ± 2 °C	48 hours until transfer to glass vials – 14 days analysis / 7 days if no acid (including 48 hours)
Soil	VOC	Gasoline Range Organics (GRO) 8015C	3 X 5g TerraCore® or equivalent	≤ 6 °C	48 hours until transfer to glass vials – 14 days analysis / 7 days if no acid (including 48 hours)
Soil	VOC	Gasoline Range Organics (GRO) 8015D	3 X 5g TerraCore® or equivalent	≤ 6 °C	48 hours until transfer to glass vials – 14 days analysis / 7 days if no acid (including 48 hours)
Soil	VOC	GCMS VOCs 8260B	3 X 5g TerraCore® or equivalent	4 ± 2 °C	48 hours until transfer to glass vials – 14 days analysis / 7 days if no acid (including 48 hours)
Soil	VOC	GCMS VOCs 8260C	3 X 5g TerraCore® or equivalent	≤ 6 °C	48 hours until transfer to glass vials – 14 days analysis / 7 days if no acid (including 48 hours) ^a
Soil	VOC	GC VOCs 8021B (SW846 Update III)	3 X 5g TerraCore® or equivalent	4 ± 2 °C	48 hours until transfer to glass vials – 14 days analysis / 7 days if no acid (including 48 hours)
Soil	VOC	GC VOCs 8021B (SW846 Update IV)	3 X 5g TerraCore® or equivalent	≤ 6 °C	48 hours until transfer to glass vials – 14 days analysis / 7 days if no acid (including 48 hours)
Soil	SVOC	Phenols 8041A (SW846 Update III)	Sleeves ^c with PTFE TM end caps or 8 oz glass jar	4 ± 2 °C	extraction - 14 days analysis - 40 days
Soil	SVOC	Phenols 8041A (SW846 Update IV)	Sleeves ^c with PTFE TM end caps or 8 oz glass jar	≤ 6 °C	extraction - 14 days analysis - 40 days
Soil	SVOC	Organochlorine Pesticides 8081A	Sleeves ^c with PTFE TM end caps or 8 oz glass jar	4 ± 2 °C	extraction - 14 days analysis - 40 days
Soil	SVOC	Organochlorine Pesticides 8081B	Sleeves ^c with PTFE TM end caps or 8 oz glass jar	≤ 6 °C	extraction - 14 days analysis - 40 days
Soil	SVOC	Polychlorinated Biphenyls (PCBs) 8082	Sleeves ^c with PTFE TM end caps or 8 oz glass jar	4 ± 2 °C	extraction - 14 days analysis - 40 days
Soil	SVOC	Polychlorinated Biphenyls (PCBs) 8082A	Sleeves ^c with PTFE TM end caps or 8 oz glass jar	≤ 6 °C	None

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Sample Preservation and Storage Requirements PR-TC-02.04.01.01

Matrix	Analytical Group	Analytical Method	Containers (number, size and type)	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time (preparation/analysis)
Soil	SVOC	Organophosphorus Pesticides 8141A	Sleeves ^c with PTFE TM end caps or 8 oz glass jar	4 ± 2 °C	extraction - 14 days analysis - 40 days
Soil	SVOC	Organophosphorus Pesticides 8141B	Sleeves ^c with PTFE TM end caps or 8 oz glass jar	≤ 6 °C	extraction - 7 days analysis - 40 days
Soil	SVOC	Chlorinated Herbicides 8151A (SW846 Update III)	Sleeves ^c with PTFE TM end caps or 8 oz glass jar	4 ± 2 °C	extraction - 14 days analysis - 40 days
Soil	SVOC	Chlorinated Herbicides 8151A (SW846 Update IV)	Sleeves ^c with PTFE TM end caps or 8 oz glass jar	≤ 6 °C	extraction - 14 days analysis - 40 days
Soil	SVOC	GCMS SVOCs 8270C	Sleeves ^c with PTFE TM end caps or 8 oz glass jar	4 ± 2 °C	extraction - 14 days analysis - 40 days
Soil	SVOC	GCMS SVOCs 8270D	Sleeves ^c with PTFE TM end caps or 8 oz glass jar	≤ 6 °C	extraction - 14 days analysis - 40 days
Soil	SVOC	Dioxins and Furans 8280A; 8290	Sleeves ^c with PTFE TM end caps or 8 oz glass jar	4 ± 2 °C ; store in the dark	extraction - 30 days analysis - 45 days
Soil	SVOC	Dioxins and Furans 8280B; 8290A	Sleeves ^c with PTFE TM end caps or 8 oz glass jar	≤ 6 °C	None
Soil	SVOC	Polycyclic Aromatic Hydrocarbons 8310 (SW386 Update III); 8270CSIM	Sleeves ^c with PTFE TM end caps or 8 oz glass jar	4 ± 2 °C	extraction - 14 days analysis - 40 days
Soil	SVOC	Polycyclic Aromatic Hydrocarbons 8310 (SW386 Update IV); 8270DSIM	Sleeves ^c with PTFE TM end caps or 8 oz glass jar	≤ 6 °C	extraction - 14 days analysis - 40 days
Soil	SVOC	PFOS/PFOA 537 Mod. (like 8151A)	Sleeves ^c (not PTFE) with polypropylene end caps or 8 oz glass jar	≤ 6 °C	extraction - 14 days analysis - 40 days
Soil	SVOC	Nitroaromatics and Nitramines 8330A	Sleeves ^c with PTFE TM end caps or 8 oz glass jar	≤ 6 °C	extraction - 14 days analysis - 40 days
Soil	SVOC	Nitroaromatics and Nitramines 8330B	1.5 grams of soil in specially prepared locking plastic bag	≤ 6 °C	extraction - 14 days analysis - 40 days
Soil	SVOC	Diesel and Oil Range Organics 8015B	Sleeves ^c with PTFE TM end caps or 8 oz glass jar	4 ± 2 °C	extraction - 14 days analysis - 40 days

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Sample Preservation and Storage Requirements PR-TC-02.04.01.01

Matrix	Analytical Group	Analytical Method	Containers (number, size and type)	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time (preparation/analysis)
Soil	SVOC	Diesel and Oil Range Organics 8015C	Sleeves ^c with PTFE TM end caps or 8 oz glass jar	$\leq 6^{\circ}\text{C}$	extraction - 14 days analysis - 40 days
Soil	SVOC	Diesel and Oil Range Organics 8015D	Sleeves ^c with PTFE TM end caps or 8 oz glass jar	$\leq 6^{\circ}\text{C}$	extraction - 14 days analysis - 40 days
Soil	Metals	ICP-AES 6010B; 6010C	Sleeves ^c with PTFE TM end caps or 4 oz glass jar	None	analysis - 6 months
Soil	Metals	ICP-MS 6020; 6020A	Sleeves ^c with PTFE TM end caps or 4 oz glass jar	None	analysis - 6 months
Soil	Metals	Mercury by CVAA 7471A	Sleeves ^c with PTFE TM end caps or 4 oz glass jar	$4 \pm 2^{\circ}\text{C}$	analysis - 28 days
Soil	Metals	Mercury by CVAA 7471B	Sleeves ^c with PTFE TM end caps or 4 oz glass jar	$\leq 6^{\circ}\text{C}$	analysis - 28 days
Soil	Inorganics	Conductivity 9050A/ 9050A	1 X 4 oz glass jar	$4 \pm 2^{\circ}\text{C}$	analysis - 28 days
Soil	Inorganics	Hexavalent Chromium 7196A / 7199 (SW846 Update III)	1 X 4 oz glass jar	$4 \pm 2^{\circ}\text{C}$	3060A Extraction – 30 days analysis - 48 hours
Soil	Inorganics	Hexavalent Chromium 7196A / 7199 (SW846 Update IV)	1 X 4 oz glass jar	$\leq 6^{\circ}\text{C}$	3060A Extraction – 30 days analysis - 48 hours
Soil	Inorganics	pH 9045D	1 X 4 oz glass jar	None	analysis - immediately
Soil	Radiochem	Gamma-Emitting Radionuclides 901.1M	1 X 16 oz glass or plastic jar ^d	None	None
Soil	Radiochem	Radium-226 by Radon Emanation 903.1M	1 X 16 oz glass or plastic jar ^d	None	None
Soil	Radiochem	Gamma Radioassay HASL300 GA-01-R	1 X 16 oz glass or plastic jar ^d	None	None
Soil	Radiochem	Radium-228 904.0M	1 X 16 oz glass or plastic jar ^d	None	None
Soil	Radiochem	Strontium-90 905.0M	1 X 16 oz glass or plastic jar ^d	None	None

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Sample Preservation and Storage Requirements PR-TC-02.04.01.01

Matrix	Analytical Group	Analytical Method	Containers (number, size and type)	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time (preparation/analysis)
Soil	Radiochem	Tritium 906.0M	1 X 16 oz glass or plastic jar ^d	None	None
Soil	Radiochem	Plutonium 238 and 239/240 HASL 300-Pu-11	1 X 16 oz glass or plastic jar ^d	None	None
Soil	Radiochem	Uranium-234, -235, and -238 HASL 300 U-02-RC	1 X 16 oz glass or plastic jar ^d	None	None

Abbreviations and Notes:

AES = Atomic Emission Spectrometry

°C = degrees centigrade

CVAA = Cold Vapor Atomic Absorption

GC = Gas Chromatography

HCl = Hydrochloric Acid

H₂SO₄ = Sulfuric Acid

IC = Ion Chromatography

ICP = Inductively Coupled Plasma

mL = milliliters

Abbreviations and Notes:

MS = Mass Spectrometry

oz = ounce

SVOC = Semi-volatile Organic Compounds

VOA = Volatile Organic Analysis

VOC = Volatile Organic Compounds

^a If vinyl chloride, styrene, or 2-chloroethyl vinyl ether are analytes of interest, collect a second set of samples without acid preservatives and analyze as soon as possible (7 day hold time).

^b If carbonaceous materials are present (or if MTBE and other fuel oxygenate ethers are present and a high temperature sample preparative method is to be used), do not acid preserve the sample.

^c Sleeves may be stainless steel, acetate, brass or PTFE, depending on project needs.

^d Sample volume and container dependent on required site-specific reporting limits. See the site-specific plan for details or variances such as tuna cans.


^e Manual for the Certification of Laboratories Analyzing Drinking Water, EPA 815-B-97-001, March 1997 Criteria and Procedures Quality Assurance.


^f Hold time may be extended with appropriate buffer solution per <https://www.epa.gov/cwa-methods/hexavalent-chromium-questions-and-answers>.

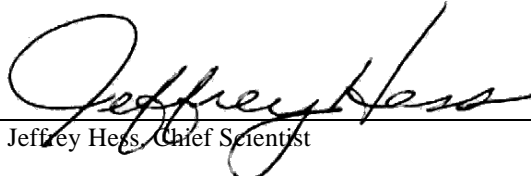
Standard Operating Procedure

Sample Tracking and Electronic Data Management

PR-TC-02.12.02.00 v3

Prepared by:  Date: 12 Sep 2018
 Kristen Carlyon Peyton, Senior Chemist

Reviewed by:  Date: 12 Sep 2018
 Wing Tse, Database Manager

Approved by:  Date: 12 Sep 2018
 Jeffrey Hess, Chief Scientist

Review / Revision History:

Version	Changes	Affects Section/Pages	Effective Date	Approval*
1.0	Initial Issue	NA	30 Sep 2009	NA
1.1	Clarification of Project Chemist responsibilities, and corrections to section numbering	Pages 2-4	30 Sep 2009	J Hess
2.0	Reorganized procedures and responsibilities	Sections 3-4	14 Jun 2013	NA
2.1	Updated definition of ERPIMS	Page 1	16 Jan 2014	J Hess
2.2	Updated field procedures and flow chart	Pages 3-5	14 Oct 2016	J Hess
3.0	Updated procedures relating to use of eDMS	All	12 Sep 2018	NA

* Approval required for reviews and minor changes only. Substantive revisions to the technical requirements contained in the SOP require review and approval by the signatures to the SOP.

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1	Gilbane Data Management Workflow

1.0 PURPOSE AND SCOPE

The purpose of this standard operating procedure is to describe the requirements and procedures for tracking environmental samples in a manner that will provide a systematic means of notifying our electronic data management group (including our chemists, database administrators, data management specialists, and other interested parties) of upcoming sampling events, ensuring the correct samples are collected and correct analyses are requested, tracking the receipt of analytical data from the laboratory for the sampling efforts, facilitate upload of electronic data to the database from the field crew and laboratories, and provide a reference for reconciliation of laboratory invoices.

2.0 ACRONYMS AND DEFINITIONS

For purposes of this procedure, a number of terms and acronyms have the meanings defined below.

COC:	chain-of-custody form
CDQMP:	Chemical Data Quality Management Plan
DQO:	data quality objective
EDD:	electronic data deliverable
eDMS:	Environmental Data Management System, Gilbane's in-house environmental data management system.
eQAPP:	electronic version of QAPP or SAP in eDMS (also known as "project setup")
ERPIMS:	Environmental Resources Program Information Management System
FUDSChem:	Formerly Used Defense Site Chemistry Database
Geotracker:	A database and geographic information system (GIS) hosted by the California State Water Resources Control Board (SWRCB) that provides online access to environmental data.
GPS:	global positioning system
LIMS:	laboratory information management system
NIRIS:	Naval Installation Restoration Information Solution
ORP:	oxidation-reduction potential
PID:	photo-ionization detector
QAPP:	Quality Assurance Project Plan
QC:	quality control
SAP:	Sampling and Analysis Plan
SEDD:	staged electronic data deliverable
SMO:	EPA Sample Management Office
SOP:	standard operating procedure

TAT: turn-around time
XRF: x-ray fluorescence

3.0 ROLES AND RESPONSIBILITIES

3.1 PROJECT MANAGER

Establishes and communicates the goals and objectives of the sampling event to the team, and provides specifics regarding the number and type of samples, analytical methods, and any special reporting requirements. Authorizes payment of laboratory and validation invoices upon successful submittal of completed EDD or ADRs, respectively.

3.2 FIELD PERSONNEL.

Responsible for the proper collection of environmental samples in accordance with the approved SAP or QAPP. Responsible for accurate, defensible documentation of sample collection per approved project plans and/or corporate SOPs.

3.3 SAMPLE COORDINATOR OR FIELD TEAM LEAD

Responsible for tracking the samples from time of collection through laboratory acceptance. Reconciles cooler content against COCs prior to transfer to laboratory. Submits samples to the laboratory. Sends COCs, Sample Collection Logs and other field forms to project team. Resolves completeness issues with laboratory (e.g. broken bottles, missing samples, etc.).

3.4 PROJECT CHEMIST (OR DESIGNEE)

Prepares SAP or QAPP and sample tracking log. Uploads project planning documents to Gilbane project portal. Reviews laboratory login report and resolves analytical issues with laboratory. Facilitates communication between lab and data group in upload of EDDs. Runs completeness test on EDDs against COCs to ensure all data has been received (this can be accomplished with the use of Event Planning reports). Reviews and finalizes results/validation qualifiers, releasing data for use. Generates Tracking Reports from eDMS or alternatively completes Sample Tracking Table (for cases like U.S. EPA Contract Laboratory Program work where sample receipts are not provided for event planning) once samples are in the laboratory. Notify Project Manager upon successful submittal of completed EDDs.

3.5 DATA MANAGEMENT STAFF

Work with Project Chemist on setting up project and/or site, if new, and updating existing projects as they evolve. Loads eQAPP using the project setup tool and sample schedule using event planning module into eDMS. Review COC and field information in eDMS. Identify EDD reporting requirements (ERPIMS, NIRIS, SEDD, etc.) based on Contract and Task Order requirements. Manage any new user accounts needed based on staffing of project team. Work with Project Chemist on designation of sample IDs (and new location IDs, if required).

Enter/review COC and field information in eDMS. Review LIMS login report and resolve login discrepancies with the laboratory/Project Chemist. Generate bottle labels by running the Bottle Label report from eDMS and send them to field personnel for sample collection. Upload the depth to water measurements and field parameter measurements to eDMS. Coordinate with GIS staff for location coordinates to be uploaded to eDMS.

4.0 PROCEDURES

Systematic sample tracking and efficient data management require that the procedures presented in this SOP be followed by all parties involved in the collection and reporting of environmental data. Figure 1 outlines the generalized flow of sample information and laboratory results from initial sampling through reporting of the validated results. Although not specifically shown in Figure 1, these procedures are applicable to other field or laboratory generated environmental data from field instruments (e.g, chemical, radiological, geotechnical, water quality, and air monitoring), mobile field laboratories, and observations.

Procedures for managing the accurate collection and reporting of the data are discussed below. Roles and specific responsibilities are presented in Sections 3.1-3.3.

- **Kickoff Meeting.** A meeting is held with the project manager or task lead, the project chemist, and a representative of the data management team to communicate the goals and objectives of the sampling event to the team, and provide specifics regarding the number and type of samples, analytical methods, and any special reporting requirements.
- **Preparation of Sample Guidance Documents.** The SAP or QAPP is prepared by the project chemist, is reviewed by the project manager or task lead for consistency with the work plan and other project documents, and goes through both the internal and client approval process. The information contained in the approved SAP or QAPP is used to develop a Planned Sample Table / Sample Tracking Log.
- **Setup of Project Database.** If the site does not already exist in eDMS (Gilbane's web-based environmental data management system), a new database is created. If the site exists, a new project is added to the previously existing database. The quality control (QC) criteria from the SAP or QAPP are entered into eDMS, and the information from the Planned Sample Table / Sample Tracking Log is entered into the Event Planning Tool in eDMS.
- **Sample Collection and Documentation.** Samples are collected in accordance with the approved site-specific project plans and chain-of-custody is properly documented.
- **Sample Tracking.** Samples are tracked from time of collection through laboratory acceptance. Issues with sample receipt (i.e., bottle breakage) are resolved.
- **Sample Entry.** COCs, Sample Collection Logs, and other field forms are submitted from the field team by scanning the field documentation and uploading to eDMS nightly. COC

information is then entered into eDMS daily. The depth to water and the field parameter measurements are also submitted from the field team to the data management staff to be uploaded to eDMS.

- Best practice (and required by some clients) is to collect GPS coordinates at time of sample collection even if a subsequent survey is planned later.
- COC Review. COC and field information is reviewed in eDMS. Laboratory LIMS login report is reviewed and analytical/login issues resolved with laboratory. The Event Planning Tool in eDMS is updated with required information (e.g., SDG associated with each sample, lab receipt date, review type, TAT).
- EDD Upload. The EDDs are uploaded to the project database. Upon successful upload, the laboratory report is available for review/validation.
- Release of Data. Review/validation is reviewed and approved. Sample Tracking Log is updated or Event Planning completeness reports are run. Once data is approved and shown to be complete, it can be released for use.
- Review and Publication of Data. Once data has been released for use, various reports can be run from eDMS to view the data in a variety of formats, and to review the data relative to applicable screening criteria and/or cleanup goals. The data is also available to incorporation into GIS for figure generation.
- Generation of Deliverable EDDs. Deliverable EDDs (i.e. NEDDs, ERPIMs, or SEDDs) are generated and uploaded as appropriate. Project Manager is notified of completion.

4.1 FIELD STAFF RESPONSIBILITIES

4.1.1 Notification of Sampling

At the beginning of each project involving the acquisition of environmental data, a preliminary meeting will be held by the project manager, project chemist and members of the electronic data management group to discuss the data quality objectives (DQOs), sampling requirements, and to plan preparation of the SAP or QAPP (including location and sample IDs). Once the plans are approved and the fieldwork is scheduled, a meeting between the project manager, project chemist and the field personnel prior to deployment will be conducted to discuss the specific requirements of the project. Specific information regarding the number and type of samples to be collected will be presented, along with recommended field procedures, sequence of work, and identity of the primary and secondary analytical laboratories. Sample naming protocols will be discussed to insure proper sample identification in the field and on COCs. The electronic data management group will be notified at this time of the start of sampling.

For multi-phase or recurring projects such as quarterly monitoring, both the project chemist and electronic data management group will be notified prior to the beginning of each sampling event, and a copy of the Planned Sample Table or Worksheet 18 from the QAPP/SAP will be provided to all interested parties prior to the initiation of sampling.

4.1.2 Chain-of-Custody

During field sampling activities, the COC will be uploaded daily by the sample coordinator or field team lead to eDMS for access by the project chemist and data management staff.

- With the use of the Event Planning tool in eDMS, an electronic COC can replace a hand-written COC. Several fields will be pre-populated based on the sample tracking table and the room for hand-written error is greatly reduced. Internet connectivity is required to use this feature either on site or at a hotel or office before sampling begins. The COCs will be uploaded to the server nightly.
- If paper COCs are used, then COCs with a unique identification number should be used and can be requested through the project chemist prior to the beginning of sampling. The location and sample IDs and the sample depths (top and bottom) for non-aqueous samples should be written on the copies of the COCs. The COCs will then be uploaded to eDMS nightly. In those cases where there is no internet access available, COCs are to be faxed daily and sent by FedEx at the end of each week.
- If Scribe is used, a copy of the output files in xml format should be forwarded electronically to the Project Chemist for upload to the SMO Portal along with a copy of the hard copy COC from the field printer to the electronic data management group.

The data management group will review and QC COCs daily in eDMS.

4.1.3 Sample Coordinates and Other Field Notes

The data management staff will either: 1) directly load location coordinates (from GPS) into eDMS; or 2) forward location coordinates (from surveys) to be loaded into eDMS for each sample collected, with the exception of waste and some process samples, or recurring sample locations where coordinates already exist (e.g., previously surveyed monitoring wells). Please confirm the existence of valid coordinates for each location prior to sampling, otherwise collect GPS coordinates just in case. In addition, all field notes including boring logs, water levels, and field measurements will be uploaded to eDMS or to the data management group for entry into the system.

4.2 PROJECT CHEMIST RESPONSIBILITIES

4.2.1 Development of SAP/QAPP and Appropriate Location and Sample IDs

The Project Chemist is responsible for preparation of the SAP or QAPP and development of appropriate location and sample IDs in consultation with the Project Manager or Task Lead. A list of location IDs and their associated sample IDs should be sent to the data manager or their designee for approval before they are incorporated in the sampling plan. The location and sample IDs should conform to the location and sample ID nomenclature requirements listed in SOP PR-TC-01.04.04.00. Once the project plans are approved by the client, the Project Chemist shall upload the eQAPP in eDMS and request that the data manager set up the site in the eDMS.

The project chemist shall set up the site in the eDMS and upload the work plan and/or QAPP/SAP to the eDMS portal.

4.2.2 Creation of Planned Sample Table

The project chemist shall develop a Planned Sample Table (PST) from the Work Plan or QAPP/SAP prior to the start of each sampling event. The project chemist shall submit the PST to the data management group for use in setting up the Event Planning for each sampling event, and for use in creating the bottle orders. Use Event Planning will allow for electronic COC and label creation, as well as electronic tracking of laboratory SDG status. An example of a PST is provided as Attachment 1. An example of an Event Planning report is provided as Attachment 2.

4.2.3 Event Planning, COC, and Label Generation

The Data Management Staff will plan the event by first creating an event to house the data. The next steps entail entering the planned locations, sampling methods, analyses, laboratory(s), and sampling company(s) into eDMS. Once event planning is complete, the Data Management Staff will generate electronic COCs and labels using eDMS reports.

Once the completed COC has been returned to the Data Management group, it will be combined with the laboratory log in receipt for data entry, QC, and data tracking.

4.2.2.1 QC of Entry of COCs in eDMS

The project chemist (or designee) will QC the data entry of the COC information entered into eDMS. The sample identifications, analyses requested, sampling methods, matrices, dates and times of sample collections, and proper assignment of quality control samples will all be checked. The Project Chemist will also verify that Event Planning has been updated with the SDG information from the laboratory log in receipt.

4.2.2.2 Cross-checking of Laboratory Receipt Form

Upon receipt of the samples by the laboratory, a completed COC and laboratory receipt form shall be forwarded to the Project Chemist and crosschecked to the online data management through use of Event Planning Reports (Attachment 2) within 48 hours. Transcription errors and any minor differences will be resolved right away and documented through email correspondence. Major problems will be documented using corrective action forms.

4.2.3 Receipt of Data and Data Uploads

As laboratory data packages are prepared and submitted to Gilbane, receipt of these data packages will be recorded by the online data management system. The Project Chemist will facilitate communication between the lab and data management group to successfully load and certify EDDs. If not uploaded to eDMS by the lab, the electronic data deliverables (EDDs) in acceptable format (Gilbane unless otherwise approved) will be forwarded to the electronic data

management group right away. The completeness of the EDDs will be verified upon receipt by the electronic data management group. eDMS will screen the results against the eQAPP. After errors (if any) are resolved, the EDD is certified by the person who uploaded it.

Validation using eDMS or a third-party validator occurs at this point. The validation codes are applied to eDMS and a validation report is prepared. The Project Chemist reviews and approves the qualifiers and again updates the Sample Tracking Log. At this point the data is approved by the Project Chemist for general use.

Upon completion of the receipt of the last sample for the sampling event (for example, one complete round of groundwater monitoring), a copy of the completed Sample Tracking Log will be forwarded to the electronic data management group for organization purposes

4.2.4 Reconciliation of Invoices

Upon receipt of laboratory invoices, the Project Chemist or his designee will cross-check the invoices against the sample tracking log to verify the receipt by Gilbane of all billed sample analyses, completed final data packages, and EDDs (accepted by the electronic data management group) before notifying the project manager that the invoices should be authorized for payment.

4.3 DATA MANAGEMENT GROUP RESPONSIBILITIES

4.3.1 Upload of Sample Information and Field Data to Database

The data management group will use the information in the planned sample table and/or Event Planning tool in eDMS and the COCs as they are received in preparation for the upload of the EDDs directly from the laboratory or from laboratory provided electronic files.

Upon receipt of the planned sample table, the data management group will review the table. Any immediate potential problems (for example, the use of the dash '-' instead of an underscore '_' in the laboratory data system) that may impact the successful upload of the EDD by the laboratory will be identified and corrected.

4.3.1.1 Entry of COCs

The data management group will enter the COC information into eDMS. The sample identifications, analyses requested, sampling methods, matrices, sample depths, dates and times of sample collections, and proper assignment of quality control samples will be cross-checked for accuracy. The Event Planning tool will be used to update eDMS as each COC is entered.

4.3.1.2 Entry of Other Field Data

Other data to be entered by the data management group includes water levels, field water quality parameters (dissolved oxygen, ORP, turbidity, etc.), well construction diagrams, borelogs,

lithological data, and GPS or survey coordinates. Additional data may include results of XRF field sampling, immunoassay test kit sampling, PID measurements, or other information required to be electronically provided to the client (typically any data used to make a regulatory decision), important for data review and analysis.

4.3.1.3 Reconciliation of Results in the EDD, COC Information Uploaded to eDMS and Event Planning Information

The data management group should run the “Current TAT Exceedance (Data Not Loaded)” Report to determine if there are any sample results the laboratory has not included in the uploaded EDD that we have requested. The data management staff should contact the Project Chemist to follow up with the laboratory if the report determines that there are samples that we have requested but the laboratory has not included in the uploaded EDD.

4.3.2 Creation of Final Data Package

The data management group will consolidate the validated EDDs from the in-house and/or third-party data validation firms with field information needed to complete the required data package. The final data package will then be submitted to the client in the required format (ERPIMS, NIRIS, SEDD, Geotracker, etc.)

5.0 REQUIRED DOCUMENTATION

The following records generated as a result of implementation of this procedure must be maintained as quality records.

- Completed COCs
- Sample Collection Logs
- GPS coordinates for each sample collected
- Sample Tracking Log (as appropriate).
- Field notes

6.0 ATTACHMENTS

Attachment 1: Example Planned Sample Table

Attachment 2: Example Event Planning Report

7.0 FORMS

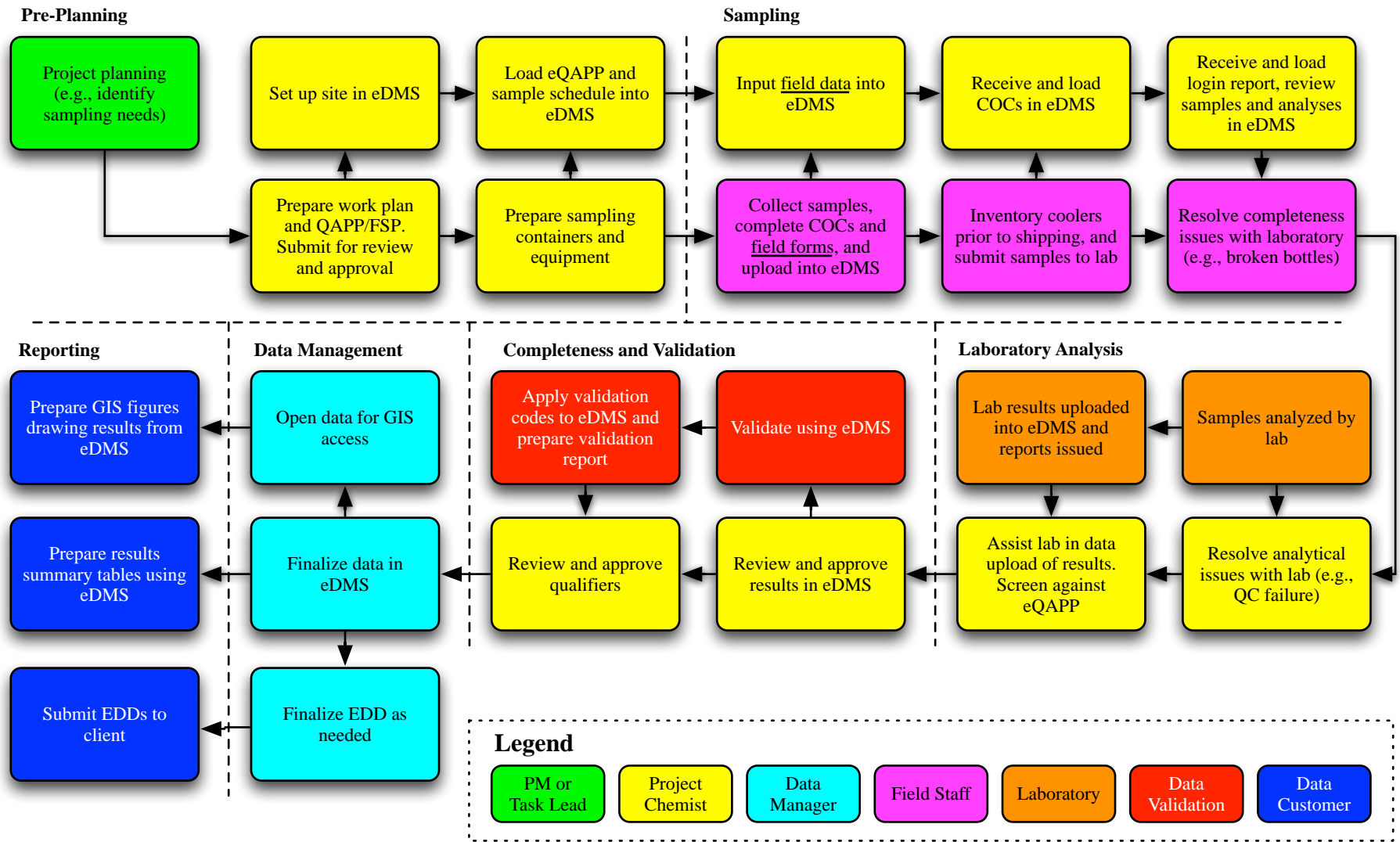
None

8.0 REFERENCES

None.

Figure 1. Gilbane Data Management Workflow

Figure 1. Gilbane Data Management Workflow



Field data includes environmental measurements collected during the course of collecting samples, and can include depth to water in a monitoring well, water quality parameters generated during purging, soil types classified during drilling, location information (by GPS), etc.

Field forms consist of the daily paperwork package generated during field events, including daily activity report (DAR), tailgate safety form, sample collection logs (as specified in the applicable SOPs), chain-of-custody forms, contractor production report (CPR), etc.

ATTACHMENT 1

EXAMPLE PLANNED SAMPLE TABLE

Attachment 1. Example Planned Sample Table

Well Number	Plume Name	Matrix	Sampling Method	BTEX+N	GRO	DRO	Total Chromium	Dissolved Chromium	Alkalinity	
MW-1		WG	LF	X	X	X	X	X	X	
MW-2		WG	LF	X	X	X	X	X	X	
MW-3R		WG	LF	X	X	X	X	X	X	
MW-4R		WG	LF	X	X	X				
MW6		WG	LF	X	X	X	X	X	X	
MW-13		WG	LF	X	X	X	X	X	X	
MW-14		WG	LF	X	X	X	X	X		
MW-20		WG	LF	X	X	X	X	X	X	MS/MSD
MW-23R		WG	LF	X	X	X				
MW-31		WG	LF	X	X	X				
MW-32		WG	LF				X	X	X	
MW-34		WG	LF	X	X	X				
MW-39		WG	LF	X	X	X	X	X	X	Dup
Piezometer A		WG	LF	X	X	X	X	X	X	
		WG	LF	X	X	X	X	X	X	Dup
		WG	LF	X	X	X	X	X	X	
TB-		WQ	NA	X	X					
TB-		WQ	NA	X	X					
TB-		WQ	NA	X	X					

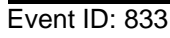
ANMCODE	EXMCODE	StrList
SW8260B	SW5030B	M
M8015D	SW3510C	B
M8015V	SW5030B	A
SW6010C	TOTAL/FLDFLT	B
A2320	NONE	A

	Samples	DUPS	MS	MSD	Total Samp	Bottles
BTEX+N	18	2	1	1	22	66
GRO	18	2	1	1	22	66
DRO	15	2	1	1	19	38
chromium	24	4	2	2	32	32
alkalinity	11	1	1	1	14	14

ATTACHMENT 2

EXAMPLE EVENT PLANNING REPORT

.



Total Location Count	5	7	7	8
-----------------------------	----------	----------	----------	----------


Site	Review Type S3/S4 Sample Count	Percent of S3/S4 Sample Count
██████████	0	0.00
██████████	0	0.00
██████████	0	0.00
██████████	0	0.00
Total	0	0.00




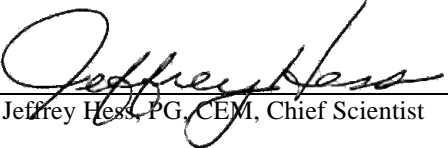
Standard Operating Procedure

Review, Verification, and Validation of Chemical Data

PR-TC-04.01.00.00 v3

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Approved by:  Date: 26 Apr 2018
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Review / Revision History:

Version	Changes	Affects Section/Pages	Effective Date	Approval*
1.0	Initial Issue	NA	27 Jul 2011	NA
1.1	Changed title to add the term “chemical data”		22 Nov 2013	J Hess
2.0	Revise procedures to document changes to process for updating validation codes.	Sections 4.2 & 4.3	17 Mar 2014	NA
2.1	Update validation checklists in eDMS	Section 6.0 & Attachments	13 Oct 2016	J Hess
3.0	Updated text to reflect updated government requirements and updated ADR procedures.	All	21 Mar 2018	NA

* Approval required for reviews and minor changes only. Substantive revisions to the technical requirements contained in the SOP require review and approval by the signatures to the SOP.

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1.0 PURPOSE AND SCOPE

The purpose of this standard operating procedure (SOP) is to describe the requirements and procedures for data review and data validation, using both automated and manual procedures. Use of the procedures outlined in this SOP for procedures other than by an experienced data reviewer should be done with great caution.

2.0 ACRONYMS AND DEFINITIONS

For purposes of this procedure, a number of terms and acronyms have the meanings defined below.

3rd Party: An independent party not involved in the collection or analysis of the samples.

ADR: automated data review; an automated validation of electronic data deliverables against project and contract requirements entered into Gilbane's database, eDMS.

COC: chain-of-custody

DAR: Data Assessment Report

DQAR: Data Quality Assessment Report

% D: percent difference (also termed percent drift)

DV: data validation; the analyte- and sample-specific process that determines the analytical quality of a specific set of data.

EDD: electronic data deliverable; an electronic file containing, in a specific electronic format, laboratory data; an EDD is produced for each sample delivery group, and is used for uploading data to the eDMS. .

eDMS: environmental data management system

GCMS: gas chromatography/mass spectrometry

ID: identification

MS/MSD: matrix spike/matrix spike duplicate

QA: quality assurance

QC: quality control

QAPP: Quality Assurance Project Plan

QRT: qualified results table

RPD: relative percent difference

RSD: relative standard deviation

SDG: sample delivery group; a group of samples that is reported together under one laboratory identification number.

SAP: Sampling and Analysis Plan

SOP: standard operating procedure

3.0 BACKGROUND

There are many competing procedures for the review and validation of chemical data. This SOP attempts to harmonize these differing procedures utilizing the most recent regulatory guidelines and the latest technology. The sections below provide the necessary background information used in developing these harmonized procedures.

3.1 GENERAL REQUIREMENTS

The general requirements for the review and/or validation of chemical data are outlined in the following documents (the relevant guidance will be identified in the SAP):

- *General Data Validation Guidelines* (Department of Defense [DoD], 2018)
- *Guidance for Labeling Externally Validated Laboratory Analytical Data for Superfund Use* (U.S. Environmental Protection Agency [EPA], 2009)

As described in these documents, some or all of following items (as applicable to the analytical method(s) used to generate the data) are considered and evaluated in a routine review of laboratory-generated data:

- Laboratory reports and chain of custody documentation, to check for errors and omissions;
- Laboratory case narratives, to check for anomalies and exceedances of QA/QC requirements;
- Laboratory reports, to check for correct reporting limits and units, and type of sensitivity limits (i.e. reporting limit/method detection limit versus limit of quantitation/limit of detection/detection limit);
- Extraction and analysis holding times;
- Method blank, trip blank, equipment blank, ambient blank, and rinse water blank data (to note any detected analytes and their respective concentrations and to check for frequency of collection);
- Surrogate compounds, the spiking levels, the resulting concentrations, and the percent recoveries;
- Laboratory control samples, the spiking levels, resulting concentrations, and percent recoveries;
- Laboratory duplicate samples, field duplicate samples, and relative percent differences (or replicates and relative standard deviation [RSD]).
- MS/MSD samples, the spiking levels, resulting concentrations, percent recoveries, and relative percent differences between the MS and MSD.

It should be noted that unless otherwise specified in the project-specific plans, as a policy, Gilbane has adopted the holding time decision criteria presented in the *General Data Validation Guidelines* (DoD, 2018).

In addition to the routine review procedure described above, more rigorous data validation will be conducted at a frequency prescribed in the project-specific sampling and analysis plan (SAP) or quality assurance project plan (QAPP). These reviews will evaluate the above items, plus each of the following (as applicable to the subject analytical methodology):

- Instrument initial calibrations, calibration levels, individual compound response factors for each level, and RSD or regression summaries of the response factors;
- Instrument continuing calibrations, calibration levels, individual compound response factors, and the percent difference (%D) or percent drift between the response factor and the response factor in the initial calibration;
- Initial and continuing blank summaries;
- Internal standard area counts and retention times, to compare to method-specified acceptance criteria;
- GC/MS tuning data and instrument performance checks, to compare to method-specified acceptance criteria;
- Data for serial dilution analyses, interference check samples, post-digestion spikes, and any method of standard additions (metals analyses only);
- Confirmation of positive results for second column or detector including percent difference between the two analytical concentrations that are greater than the detection limit;
- Raw data, including chromatograms, to check for correct transcription, interpretation, manual integrations, and compound identification;
- Injection logs for all instruments used for analysis of project sample;
- Preparation logs for all project samples and associated QA/QC samples;
- Date and time of analysis of project samples and associated QA/QC samples.

3.2 REGULATORY BASIS

The general requirements outlined in Section 3.1 were evaluated against guidelines and procedures outlines in the 2009 *Guidance for Labeling Externally Validated Laboratory Analytical Data for Superfund Use* by the U.S. Environmental Protection Agency (EPA) (EPA, 2009). This guidance document establishes specific terminology for data review and validation efforts. This terminology is being adopted here to facilitate a clear understanding by both the reviewer and the ultimate user of the data regarding the specific level of review and validation to which the data was subjected. This terminology is outlined below.

Table 1. Description of “Stages” Terminology

Stage	Validation Description	Label Codes
–	Not validated	NV
1	Completeness	S1VE, S1VM
2A	Completeness and sample-related QC	S2AVE, S2AVM, S2AVEM
2B	Completeness and both sample-related and instrument-related QC	S2BVE, S2BVM, S2BVEM
3	Completeness, both sample-related and instrument-related QC, and recalculation checks	S3VE, S3VM, S3VEM
4	Completeness, both sample-related and instrument-related QC, recalculation checks and review actual instrument outputs	S4VE, S4VM, S4VEM

The possible label codes are based on a combination of what “Stage” of review and/or validation was performed, and whether the review and/or validation was performed manually (M), electronically (E), or both (EM).

4.0 PROCEDURES

The specific protocols used are outlined below for the review and validation of analytical data consistent with the guidance presented in Section 3.1.

4.1 STAGE 1 VERIFICATION

Stage 1 verification, commonly referred to as a completeness check, of the laboratory analytical data package consists of verification of compliance and sample receipt conditions. The following items are considered and evaluated during Stage 1 verification of the sample delivery group (SDG):

- Chain-of-custody documentation including laboratory receipt information,
- Laboratory case narrative and summary report,
- Laboratory report signed by official laboratory representative,
- Requested samples analyzed,
- Requested analytes, sensitivity limits, sensitivity limit types and units.

Stage 1 can be completed either manually, electronically within eDMS, or a combination of the two.

4.1.1 Manual Stage 1

Specific procedures for manual Stage 1 verification are provided below:

1. Verify the laboratory report clearly identifies the laboratory receiving the samples and performing the analyses and that the laboratory report is signed by an official laboratory representative.
2. Review laboratory case narrative for anomalies and QC issues.

3. Verify the analytical methods specified in the SAP or QAPP were performed, and if substitutions were made, verify written instructions from the Gilbane Project Chemist specifying/allowing the substitution.
4. Review cooler receipts and sample login files for potential issues that may affect the usability of the samples (presence of custody seals, etc.) or validity of the reported results (receiving temperatures, etc.).
5. Verify all the target compounds identified in the SAP or QAPP for each of the analyses were reported by the laboratory.
6. Verify all the analyses for each of the samples listed on the chain-of-custodies were completed, the dates are present when the analyses were performed, the analyses were performed within the specified holding times, the requested reporting limits were obtained, and the results appropriately qualified.
7. Document review with Stage 1 Verification Checklist, included here in the Attachments.

4.1.2 Electronic Stage 1 Using eDMS

Much of a Stage 1 verification is by nature manual. For this reason, no specific procedures for electronic Stage 1 verification incorporating eDMS are provided.

Perform steps 1 through 4 as written above.

From the Gilbane Projects Portal (<https://edms.gilbaneco.com>), select the project in which you would like to run your report from the drop-down menu.

From the grey menu bar, select “Reports>Data Review Reports>ADR Report”.

A new screen will pop up. In “Status” choose “Loaded and Certified”, and a list of available SDGs should appear. Choose an SDG, and fill in the rest of the fields as appropriate. These are report headers and used for documentation purposes. At a minimum, fill in the data review quality control (QC) SIVEM (per Table 1 in Section 3.2). Select the “Display All” option of the Anomalies Display pull-down menu to generate a list of cases where the reported reporting limit (RL) exceeds that specified in the governing project document.

Use the last four fields as a case narrative for any qualifiers entered in the sample qualification tool that the ADR did not qualify.

Select “View Reports” and the bookmarked ADR Report will generate.

Verify that the total number of analyses presented in the “ADR Summary” match the total number of analyses requested on the COC. If there is a discrepancy, use the detailed “Batch Report” for more in-depth review.

Verify that there were no holding time exceedances by reviewing the “QC Outliers Report.”

Review the “Reporting Anomalies” to verify requested reporting limits were attained.

Document review using Stage 1 Validation Checklist, example presented in the Attachments. File completed checklist in the project library on eDMS and/or on server in project folder in Data Validation subfolder.

4.2 STAGE 2A VALIDATION

Stage 2A validation will evaluate and consider the items listed above for completeness, in addition to following:

- Extraction and analysis holding times,
- Method blank, trip blank, equipment blank and ambient blank data,
- Surrogates,
- Matrix spikes and matrix spike duplicates,
- Field duplicates,
- Laboratory control samples, and
- Laboratory duplicates.

Stage 2A can be performed manually, but is preferentially performed electronically using eDMS. The following steps apply when performing automated data review (ADR) for Stage 2A within eDMS:

Data Validator (Gilbane or 3rd Party)

1. From the Gilbane Projects Portal (<https://edms.gilbaneco.com>), select the project in which you would like to run your reports from the drop-down menu.
2. From the grey menu bar, select “Reports>Data Review Reports>ADR LOD Detail”
3. The status pull-down menu will default to the most commonly used status (“Loaded and Certified”). Select the desired SDG, and fill in the balance of the items as desired.
4. Click “View Report” on the left hand side of the screen to generate the report.
5. Review the resulting report for QC Outliers and RL anomalies. (The report can be exported to Excel or PDF for ease of review by clicking on the export icon above the report.
 - On the Batch Report, verify all samples are being reported with the correct IDs. Verify the correct parameters were evaluated.
 - Skip to “QC Outlier Report” and review. Verify that both the correct control limits and correct warning limits were used to qualify the data. Check the outliers against the case narrative and check 10% against the hard copy.

Note: If incorrect control or warning limits are discovered, send an email to the database manager or specialist with a correction request.
6. Return to the home screen and select Tools>Data – Review Checklist. Select the relevant project and event from left-hand side of the screen. Select whether it is the first or second review, or all if there will be only one.
7. Select the desired SDG from the left-hand side pull-down menu.
8. Select the SAP- or QAPP-specified validation level from the top-right hand Data Review QC Level pull-down menu, populate the balance of the upper fields, and click “ Save SDG Details”.

The screenshot shows a web-based form titled 'SDG Details'. On the left, there are four dropdown menus: 'Project' (selected: Parcel E2 Hot Spot Delin), 'Event' (selected: Parcel E-2 Air Monitoring), 'Review Status' (selected: First Review Only), and 'SDG' (selected: 1602674). The main form area contains several input fields and dropdowns: 'Data Review Contractor', 'Project Manager', 'Reviewer' (dropdown), 'Reviewer Title', 'Second Reviewer' (dropdown), 'Review Type (Planned)', 'Data Review QC Level' (dropdown), 'Review Date' (calendar icon), 'Lab Receipt Date' (calendar icon), 'Review Completion Date' (calendar icon), and 'Val. Report in Library' (calendar icon). There are two 'Save' buttons: 'Save SDG Details' and 'Save Comments'. Below the main form is a 'Test Method' dropdown and a text area for 'Additional Comments for Test Method' with a 'Save Comments' button. At the bottom left, there is an 'Editing Tips' link.

9. The Test Methods will become available once Save SDG Details has been clicked. Each method has an associated electronic checklist to be populated.
10. After the checklists are populated, edit/add qualifiers as necessary in the database by selecting “Tools>Sample Qualification” in the Gilbane Project Portal.
 - a. If there are any necessary changes to “Qualified Results”, notify the project chemist immediately to determine if project setup needs to be adjusted to correct the issues and prevent the issue in the future.
11. Generate a Field Duplicate Report, if applicable, by choosing “Reports>Data Review Reports>Field Duplicate Reports by SDG.” Fill in the desired title of the report, select the project and event from the pull-down menu, select the SDG from the next pull-down menu, and click “View Report. Check that the correct parent and duplicate sample are identified, and that the correct limits are displayed in the RPD columns. If updates are necessary then:
 - Notify the Project Chemist of any changes that need to be made to project setup;
 - Select “Field Duplicate Reports by SDG (user criteria)” from the “Data Review Reports”; enter appropriate information and the RPD values for the inorganic or organic methods. Click “View Report” to verify the report. Export the report in desired format (i.e. pdf, by clicking down arrow next to “Select a format” and then clicking on “Export.” Note if there are any qualified outliers.
12. From the grey menu bar, select “Reports>Data Review Reports>ADR”.
13. The following screen will appear:

Status	<input type="text" value="Loaded and Certified"/>	SDG	<input type="text" value="<Select a Value>"/>
2nd Signature on Report	<input type="radio"/> True <input checked="" type="radio"/> False	Second Reviewer Title	<input type="text"/>
Logo to Display	<input type="text"/>	Report Title	<input type="text" value="Data Validation Report"/>
Display Modified Qualifications Table	<input checked="" type="radio"/> True <input type="radio"/> False	Display Trace Values Table	<input type="radio"/> True <input checked="" type="radio"/> False

14. The status pull-down menu will default to the most commonly used status (“Loaded and Certified”). Select the desired SDG, and fill in the balance of the items as desired.
15. The “Project” logo choice will provide a Gilbane logo. The “Company” logo defaults to Gilbane as well, but can be customized for those projects using third party validation by contacting the Database Manager at htse@gilbaneco.com and providing a company logo.
16. Click on “View Report” on the left hand side of the screen to generate the ADR.
17. From the “Select a format” pull down menu, select “PDF”, and click on export.
18. Combine the final ADR and the Field Duplicate Report into a single PDF and collect all applicable electronic signatures.
19. When reports have been generated and review process is complete select <Finalize Qualifiers> from main screen.

Gilbane Project Chemist

20. Review the data validation report for consistency with project-specific requirements, validation guidelines (e.g., National Functional Guidelines, General DV Guidelines, etc.), and with this SOP. Work directly with the data validator to resolve any questions and any necessary changes. Once the data validation report is approved, the SDGs will be released for use by selecting <Approve SDG>.

4.3 STAGE 2B VALIDATION

For 2B validation, the following additional information will be reviewed:

- Initial and continuing calibration standards,
- Initial and continuing calibration blanks (for inorganic and metals analyses),
- Serial dilutions and post spikes, where applicable,
- Reporting limit verification standard, and
- Instrument tune and performance checks
- Internal standards (if summaries are available)
- Interference check standards

These additional steps apply when performing an ADR for 2B within eDMS:

Data Validator (Gilbane or 3rd Party)

1. Manually review those elements of the Stage 2B not covered by the ADR using the applicable sections of the Stage 2B Validation Checklist, example presented in the Attachments. These items include:
 - Initial and continuing calibration standards
 - Initial and continuing calibration blanks

- Serial dilutions and post spikes, where applicable
 - Reporting limit verification standard
 - Instrument tune and performance checks
 - Internal standards (if summaries are available)
2. Additional qualification of data may result from the manual review. Narration of any additional qualification should be presented in the appropriate checklist and case narrative, if applicable..
 3. Edit/add qualifiers as necessary in the database by selecting “Tools>Data - Sample Qualification” in the Gilbane Project Portal.
 4. To add QC Outliers to the ADR Report, use either the “Apply Contamination Qualifiers” or “Apply Qualifiers with Outlier” functions in the Data- Sample Qualification Tool, as appropriate.
 5. Generate a Field Duplicate Report, if applicable, by choosing “Reports> Data Review Reports>Field Duplicate Report by SDG.” Fill in the desired title of the report, select the event from the pull-down menu, select the SDG from the next pull-down menu, and click “View Report. Check that the correct parent and duplicate sample are identified and that the correct limits are displayed in the RPD columns. If updates are necessary then:
 - Notify the Project Chemist of any changes that need to be made to project setup;
 - Select “Field Duplicate Reports by SDG (user criteria)” from the “Data Review Reports”; enter appropriate information and the RPD values for the inorganic or organic methods. Click “View Report” to verify the report. Export the report in desired format (i.e. pdf, by clicking down arrow next to “Select a format” and then clicking on “Export.”
 6. When reports have been generated and review process is complete select <Finalize Qualifiers>.
 7. For the final report combine the ADR and Field Duplicate Report into one pdf file.

Note: Depending on the requirements of the project, the ADR pdf alone may suffice as the final deliverable. Check with the Project Chemist for the specific requirements of each project.

Gilbane Project Chemist

8. Review the data validation report for consistency with project-specific requirements, validation guidelines (e.g., National Functional Guidelines, General DV Guidelines, etc.), and with this SOP. Work directly with the data validator to resolve any questions and any necessary changes. Once the data validation report is approved, the SDGs will be released for use by selecting <Approve SDG>.

4.4 STAGE 3 AND 4 VALIDATION

Stage 3 validation will evaluate and consider the raw data associated with items reviewed in Stages 2A and 2B, in addition to following:

- Internal standards and retention times

- Manual integration
- Method of standard additions, linear range determinations, instrument detection limits, (metals analyses only)
- Method detection limit studies
- Sequence logs
- Preparation logs
- Raw data suitable for recalculation of results

For a Stage 4 review, add the review of the following instrument outputs:

- Raw data of chromatograms and spectra suitable for qualitative assessment

The following additional steps apply when performing the ADR within eDMS:

Data Validator (Gilbane or 3rd Party)

1. Manually review the raw data for calibrations, internal standards and retention times, tunes, serial dilutions, post-spikes, interference check samples, and MDLs, and perform raw data recalculations, and, if specified, review raw chromatograms and spectra using the Stage 3 and 4 Validation Checklists and Worksheets example presented in the Attachments.
2. Edit and review qualifiers as necessary in the database by following the instructions in Section 4.3 steps 3 and 4.
3. When reports have been generated and review process is complete, select <Finalize Qualifiers> from main screen.
4. For the final report, combine the ADR and Field Duplicate Report into one pdf file.

Gilbane Project Chemist

5. Review the data validation report for consistency with project-specific requirements, validation guidelines (e.g., National Functional Guidelines, General DV Guidelines, etc.), and with this SOP. Work directly with the data validator to resolve any questions and any necessary changes. Once the data validation report is approved, the SDGs will be released for use by selecting <Approve SDG>.

5.0 REQUIRED DOCUMENTATION

The following records generated as a result of implementation of this procedure must be maintained as quality records.

- ADR with checklists attached
- Field Duplicate Report (as applicable)

6.0 ATTACHMENTS

- Example eDMS checklists
- Example Manual Checklists
 - Stage 1 Verification Checklist
 - Stage 2 Validation Checklists
 - Stage 3 & 4 Validation Checklists and Worksheet

7.0 FORMS

None.

8.0 REFERENCES

U.S. Department of Defense (DoD), 2018. *General Data Validation Guidelines*. February.

U.S. Department of the Navy, Southwest Division (NAVFAC-SW), 2001. *Environmental Work Instruction #1, Data Validation Guidelines for Chemical Analysis of Environmental Samples*. 28 November.

U.S. Environmental Protection Agency (USEPA), 2009. EPA 540-R-8-005, *Guidance for Labeling Externally Validated Laboratory Analytical Data for Superfund Use*. January.

ATTACHMENTS

- Example eDMS checklists
- Example Manual Checklists
 - Stage 1 Verification Checklist
 - Stage 2 Validation Checklists
 - Stage 3 & 4 Validation Checklists and Worksheet

Example eDMS Checklists

Item No.	Review Questions	Yes/No/NA	Comment
SW6010C – Inductively Coupled Plasma-Atomic Emission Spectrometry			
Stage 2B VEM			
1	Stage 2 Review: COC - Custody Trail?		
2	Stage 2 Review: COC - Temperature/Condition?		
3	Stage 2 Review: COC - Receipt anomalies?		
4	Stage 2 Review: COC - Sample/Methods checked?		
5	Stage 2 Review: Case Narrative - Anomalies?		
6	Stage 2 Review: Samples - Collection date?		
7	Stage 2 Review: Samples - Extraction date?		
8	Stage 2 Review: Samples - Analysis date?		
9	Stage 2 Review: Samples - Holding time?		
10	Stage 2 Review: Samples - Batching?		
11	Stage 2 Review: Samples - Lab qualifiers?		
12	Stage 2 Review: Calibration - ICAL?		
13	Stage 2 Review: Calibration - ICV?		
14	Stage 2 Review: Calibration - CCV?		
15	Stage 2 Review: Blank - Method blank?		
16	Stage 2 Review: Blank - Trip blank?		
17	Stage 2 Review: Blank - Equipment blank?		
18	Stage 2 Review: Precision/Accuracy - MS/MSD?		
19	Stage 2 Review: Precision/Accuracy - LCS/LCSD?		
20	Stage 2 Review: Quantitation - PQLs?		
21	Stage 2 Review: Quantitation - Dilution Factor?		
22	Stage 2 Review: Quantitation - Results (i.e. correct analytes)?		
23	Stage 2 Review: Field Duplicates - RPD within limits?		

SW8260B – Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)			
Stage 2B VEM			
1	Stage 2 Review: COC - Custody Trail?		
2	Stage 2 Review: COC - Temperature/Condition?		
3	Stage 2 Review: COC - Receipt anomalies?		
4	Stage 2 Review: COC - Sample/Methods checked?		
5	Stage 2 Review: Case Narrative - Anomalies?		
6	Stage 2 Review: Samples - Collection date?		
7	Stage 2 Review: Samples - Extraction date?		
8	Stage 2 Review: Samples - Analysis date?		
9	Stage 2 Review: Samples - Holding time?		
10	Stage 2 Review: Samples - Batching?		
11	Stage 2 Review: Samples - Surrogate recoveries?		
12	Stage 2 Review: Samples - Lab qualifiers?		
13	Stage 2 Review: Calibration - ICAL?		
14	Stage 2 Review: Calibration - ICV?		
15	Stage 2 Review: Calibration - CCV?		
16	Stage 2 Review: Blank - Method blank?		
17	Stage 2 Review: Blank - Trip blank?		
18	Stage 2 Review: Blank - Equipment blank?		
19	Stage 2 Review: Precision/Accuracy - MS/MSD?		
20	Stage 2 Review: Precision/Accuracy - LCS/LCSD?		
21	Stage 2 Review: Quantitation - PQLs?		
22	Stage 2 Review: Quantitation - Dilution Factor?		
23	Stage 2 Review: Quantitation - Results (i.e. correct analytes)?		
24	Stage 2 Review: Field Duplicates - RPD within limits?		

Example Manual Checklists

- Stage 1 Verification Checklist
- Stage 2 Validation Checklists
- Stage 3 & 4 Validation Checklists and Worksheet

Stage 1 Verification Checklist

Project Name:

Project No.:

Laboratory:

SDG #:

Reviewer:

Organization:

CATEGORY		COMMENTS
SAMPLE RECEIPT FORM		
<input type="checkbox"/>	Cooler temperature within control limits	
<input type="checkbox"/>	Samples received intact within holding time	
RECONCILIATION OF CHAIN-OF-CUSTODY TO SDG		
<input type="checkbox"/>	Sample IDs correct	
<input type="checkbox"/>	Requested analysis received	
<input type="checkbox"/>	Sample time and date received correct	
<input type="checkbox"/>	Chain of custody maintained (i.e. signatures and line-outs)	
REVIEW OF SDG		
Review case narrative		
<input type="checkbox"/>	Check for analytical holding times	
Review laboratory package		
<input type="checkbox"/>	Verify analyses requested on COC for each sample were completed	
<input type="checkbox"/>	Verify analytical methods specified in QAPP were performed, else written variance on file.	
<input type="checkbox"/>	Verify all the target compounds identified in the QAPP for each of the analyses were reported by the laboratory.	
<input type="checkbox"/>	Verify requested reporting limits were obtained, and that results were appropriately reported and qualified.	
<input type="checkbox"/>	Check type of package requested (ie Level III, Level IV) was received.	

Stage 2 Review Checklist: Organics

Project Name:

Project No.:

Laboratory:

SDG #:

Reviewer:

Organization:

Category	Method_____	Notes
COC		
Custody Trail		
Temperature/Condition		
Receipt anomalies		
Sample/Methods check		
Case Narrative		
Anomalies		
Samples		
Collection date		
Extraction date		
Analysis date		
Holding time		
Batching		
Surrogate recoveries		
Lab qualifiers		
Calibration (Stage 2b only)		
ICAL		
ICV		
CCV		
Blank		
Method blank		
Trip blank		
Equipment blank		
Precision/Accuracy		
MS/MSD		
LCS/LCSD		
Quantitation		
PQLs		
Dilution Factor		
Results (i.e. correct analytes)		
Field Duplicates		
RPD within limits		

Stage 2 Organic Checklist 072711.doc

Stage 2 Review Checklist: Metals

Project Name:

Project No.:

Laboratory:

SDG #:

Reviewer:

Organization:

Category	Method _____	Notes
COC		
Custody Trail		
Temperature/Condition		
Receipt anomalies		
Sample/Methods check		
Case Narrative		
Anomalies (DQFs)		
Samples		
Collection date		
Extraction date		
Analysis date		
Holding time		
Batching		
Lab qualifiers		
Blank		
Method blank		
Equipment blank		
Calibration (Stage 2b only)		
ICAL		
ICV		
CCV		
LLCCV		
Precision/Accuracy		
MS/MSD		
LCS/LCSD		
Quantitation		
PQLs		
Dilution Factor		
Results		
Field Duplicates		
RPD in criteria		

Stage 2 Metals Checklist 072711.doc

Stage 3 & 4 Review Checklist: Organics

Project Name:

Project No.:

Laboratory:

SDG #:

Reviewer:

Organization:

Category	Method _____	Notes
COC		
Custody Trail		
Temperature/Condition		
Receipt anomalies		
Sample/Methods check		
Case Narrative		
Anomalies		
Samples		
Collection date		
Extraction date		
Analysis date		
Holding time		
Batching		
Surrogate recoveries		
Internal standard recoveries		
Lab qualifiers		
Blank		
Method blank		
Trip blank		
Equipment blank		
Calibration		
ICAL		
ICV		
CCV		
Precision/Accuracy		
MS/MSD		
LCS/LCSD		
Instrument		
Tune		
Breakdown Standard		

Stage 3 & 4 Review Checklist: Organics

Project Name:

Project No.:

Laboratory:

SDG #:

Reviewer:

Organization:

Quantitation		
PQLs		
Dilution Factor		
Results (RT, Ions, Rel. Intens.)		
Retention times		
Major ions present		
Relative intensities		
Column/Detector RPD		
Confirmation		
Result Recalculation		
Field Duplicates		
RPD within limits		
Review Chromatograms/Spectra (Stage 4 only)		

Stage 3-4 Organic Checklist 072711.doc

Stage 3 & 4 Review Checklist: Metals

Project Name:

Project No.:

Laboratory:

SDG #:

Reviewer:

Organization:

Category	Method_____	Notes
COC		
Custody Trail		
Temperature/Condition		
Receipt anomalies		
Sample/Methods check		
Case Narrative		
Anomalies (DQFs)		
Samples		
Collection date		
Extraction date		
Analysis date		
Holding time		
Internal standard		
Batching		
Lab qualifiers		
Blank		
Method blank		
Calibration blank		
Equipment blank		
Calibration		
ICAL		
ICV		
CCV		
LLCCV		
Precision/Accuracy		
MS/MSD		
LCS/LCSD		
Post digestion spike		
Serial dilution		
Reporting Limit Std.		
Instrument		
ICSA		
ICSAB		
Tune		

Stage 3 & 4 Review Checklist: Metals

Project Name:

Project No.:

Laboratory:

SDG #:

Reviewer:

Organization:

Quantitation		
PQLs		
Dilution Factor		
Results - Recalc		
Field Duplicates		
RPD in criteria		

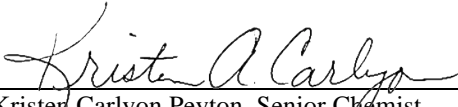
Stage 3-4 Metals Checklist 072711.doc




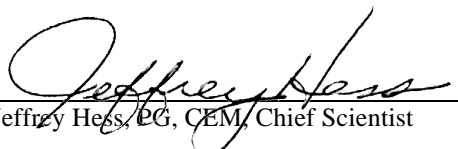
Standard Operating Procedure

Review, Verification, and Validation of Radiological Data


PR-TC-04.01.02.00 v1.0a

Reviewed by:  Date: 12 Oct 2016
Kristin Carlyon Peyton, Senior Chemist

Reviewed by:  Date: 12 Oct 2016
Evelyn Dawson, Senior Chemist

Approved by:  Date: 12 Oct 2016
Jeffrey Hess, PG, CEM, Chief Scientist

Review / Revision History:

Version	Changes	Affects Section/Pages	Effective Date	Approval*
1.0	Initial Issue	NA	12 Oct 2016	NA
1.0a	Reviewed, no update necessary	NA	21 Jun 2018	

* Approval required for reviews and minor changes only. Substantive revisions to the technical requirements contained in the SOP require review and approval by the signatures to the SOP.

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1.0 PURPOSE AND SCOPE

The purpose of this standard operating procedure (SOP) is to describe the requirements and procedures for the review and validation of radiological data using primarily manual procedures. Use of the procedures outlined in this SOP for procedures other than by an experienced data reviewer should be done with great caution.

2.0 ACRONYMS AND DEFINITIONS

For purposes of this procedure, a number of terms and acronyms have the meanings defined below.

ADR: automated data review; an automated validation of electronic data deliverables against project and contract requirements entered into Gilbane's database, eDMS.

ANSI: American National Standards Institute

Carrier: A known mass of a non-radioactive isotope of the analyte (or stable isotope of a chemically similar element) used in analyses to determine chemical yield.

CDQMP: Chemical Data Quality Management Plan

Chemical Yield: A measure for losses that might have occurred during sample processing, separation, and quantification, as measured using a tracer or carrier. Chemical yield is expressed as the percent recovery.

COC: chain-of-custody

CSU: combined standard uncertainty. Standard uncertainty of an output estimate calculated by combining the standard uncertainties (one-sigma uncertainties) of the input estimates. Expanded uncertainty is the combined standard uncertainty multiplied by a "coverage factor" (e.g., 2 or 3) to obtain the two-sigma [95% probability] or three-sigma [99% probability] expanded uncertainty.

critical value (L_c): The minimum measured value (e.g., of the instrument signal or the radionuclide concentration) required to give a specified probability that a positive (non-zero) amount of radioactivity is present in the material being measured. Also known as the critical level, decision level (DL), and decision level concentration (DCL).

DAR: Data Assessment Report

DER: duplicate error ratio. A measure of the reproducibility of results calculated using the primary and duplicate sample results and combined standard uncertainties. Also known as relative error ratio (RER)

DL: decision level (see critical value)

DPM: disintegrations per minute

DQA: data quality assessment

DQAR: Data Quality Assessment Report

DQO: data quality objective

% D: percent difference (also termed percent drift)

DV: data validation; the analyte- and sample-specific process that determines the analytical quality of a specific set of data.

EDD: electronic data deliverable; an electronic file containing, in a specific electronic format, laboratory data; an EDD is produced for each sample delivery group, and is used for uploading data to the eDMS.

eDMS: environmental data management system.

ID: identification

MARLAP: Multi-Agency Radiological Laboratory Analytical Protocols Manual

MARSAME: Multi-Agency Radiation Survey and Assessment of Materials and Equipment Manual

MARSSIM: Multi-Agency Radiation Survey and Site Investigation Manual

MB: method blank

MDA: minimum detectable amount. Also known as the minimum detectable activity. The minimum amount or concentration of a radionuclide required within a given confidence that the measurement result would be above the DL (detected). This is typically based on a confidence level of 95%. Correspondingly, the probability of a Type II error (probability of erroneously not concluding a radionuclide is detected in a sample that has the MDA quantity or concentration) is typically set at 0.05. Thus the alpha (Type I) and beta (Type II) probabilities are both typically set at 0.05.

MDC: minimum detectable concentration (see MDA). There are two types of MDC: (1) the “a priori” MDC calculated using nominal or typical analytical parameter values, typically used to evaluate the relative detection capabilities of specific analytical methods; and (2) the “a posteriori” sample-specific MDC (ssMDC) calculated for a specific sample and using the sample-specific Lc (ssLc) and parameter values for the individual sample.

MS/MSD: matrix spike/matrix spike duplicate

MQO: measurement quality objective

QA: quality assurance

QC: quality control

QAPP: Quality Assurance Project Plan

QRT: qualified results table

RDL: required detection limit. Also known as the required minimum detectable concentration (RMDC).

RPD: relative percent difference

RSD: relative standard deviation

SDG: sample delivery group; a group of samples that is reported together under one laboratory identification number.

SAP: Sampling and Analysis Plan

SOP: standard operating procedure

TPU: total propagated uncertainty (TPU). See CSU (MARLAP, 2004).

Tracer: A known amount of a radioactive isotope chemically similar to the analyte used in analyses to determine chemical yield.

3.0 BACKGROUND

Historically there have been few formalized procedures for the review and validation of radiological data. This SOP attempts to develop specific procedures that are consistent with the available standards in order to provide for more uniform verification and validation of radiological data by Gilbane personnel and 3rd-party validation firms.

In 2012, the American Nuclear Society (ANS), working with the American National Standards Institute (ANSI), published *Verification And Validation Of Radiological Data For Use In Waste Management And Environmental Remediation* (ANSI/ANS-41.5-2012). This standard complements recommended practices in Multi-Agency Radiological Laboratory Analytical Protocols Manual (MARLAP) (NUREG-1576, 2004) for planning radiological testing programs and for the laboratory analyses of radiological samples, and is adopted by Gilbane as the standard for data verification and validation of radiological data.

3.1 GENERAL REQUIREMENTS

3.1.1 Evaluation Criteria

As outlined in ANSI/ANS-41.5, the following items may be evaluated during the verification and validation of laboratory-generated radiological data:

- Sample Specific Parameters
 - Sample Preservation
 - Holding Time
 - Sample-specific Chemical Yield
 - Required Detection Limit (RDL)
 - Nuclide Identification
 - Quantification and Uncertainty
 - Detectability
 - Sample Aliquot Representativeness
- Batch Control Parameters
 - Laboratory Control Sample (LCS) Analysis
 - Matrix Spike (MS) Analysis
 - Duplicate and Matrix Spike Duplicate (MSD) Analysis
 - Batch Method Blank Analysis
- Instrument Parameters
 - Counting Efficiency Calibration
 - Energy Calibration
 - Background Determination

Additional evaluation steps are recommended as part of the laboratory selection and qualification process, and may be included in a laboratory desktop audit performed prior to or during preparation of the project-specific planning documents. See ANS/ANSI-41.5-2012 Sections 3.1 through 3.4.

3.1.2 Qualifying Radiological Data

The verification and validation process for radiological data involves a series of evaluations as outlined in Section 3.1.1, and culminates in a judgment of the quality and reliability of data, both individually and for an entire batch of data. Based on this judgment, appropriate qualifiers are assigned.

Radiological data should not be rejected without adequate cause, generally requiring a pattern of problems with a specific piece of data or batch of data to be rejected. As MARLAP indicates, “rejecting a result is an unconditional statement that it is not useable for the intended purpose. A result should only be rejected when the risks of using it are significant relative to the benefits of using whatever information it carries.” MARLAP provides some general guidance to consider when thinking about rejecting data:

1. Insufficient or only incorrect data are available to make fundamental decisions about data quality. For example, if correctly computed uncertainty estimates are not available, it is not possible to do most of the suggested tests. If the intended use depends on a consistent, high level of validation, it may be proper to reject such data. The missing data should be fundamental. For example, missing certificates for standards are unlikely to be fundamental if laboratory performance on spiked samples is acceptable. In contrast, if no spiked sample data is available, it may be impossible to determine if a method gives even roughly correct results, and rejection may be appropriate.
2. Available data indicate that the assumptions underlying the method are not true. For example, QC samples may demonstrate that the laboratory’s processes are out of control. Method performance data may indicate that the method simply does not work for particular samples. These problems should be so severe that it is not possible to make quantitative estimates of their effects.
3. A result is very unusually uncertain. It is difficult to say what degree of uncertainty makes a result unusable. Whenever possible, uncertain data should be rejected based on multiple problems with one result, patterns in related data, and the validator’s judgment, not the outcome of a single test. This requires radiochemistry expertise and knowledge of the intended use.

3.2 REGULATORY BASIS

The requirements outlined in Section 3.1 were evaluated against guidelines and procedures outlined in the 2009 *Guidance for Labeling Externally Validated Laboratory Analytical Data for Superfund Use* by the U.S. Environmental Protection Agency (EPA) (EPA, 2009). This guidance document establishes specific terminology for data review and validation efforts. This terminology is adopted here to facilitate a clear understanding by both the reviewer and the

ultimate user of the data regarding the specific level of review and validation to which the data was subjected. This terminology is outlined below.

Table 1. Description of “Stages” Terminology

Stage	Validation Description	Label Codes
–	Not validated	NV
1	Completeness	S1VE, S1VM
2A	Completeness and sample-related QC	S2AVE, S2AVM, S2AVEM
2B	Completeness and both sample-related and instrument-related QC	S2BVE, S2BVM, S2BVEM
3	Completeness, both sample-related and instrument-related QC, and recalculation checks	S3VE, S3VM, S3VEM
4	Completeness, both sample-related and instrument-related QC, recalculation checks and review actual instrument outputs	S4VE, S4VM, S4VEM

The possible label codes are based on a combination of what “Stage” of review and/or validation was performed, and whether the review and/or validation was performed manually (M), electronically (E), or both (EM).

3.3 SPECIFIC CLIENT REQUIREMENTS

Based on a review of various client requirements (e.g. Department of Defense data item descriptions, NAVFAC-SW Environmental Work Instruction #1, *Chemical Data Validation* (NAVFAC-SW, 2001), EM 200-1-10, etc.) and their governing CDQMPs, the following table outlines the general guidelines for the review and/or validation of chemical data for various clients:

Table 2. Cross-Walk of Validation Requirements

Client	Target Validation Level	Equivalent Stage
Department of Defense		
Air Force/AFCEC	Level III	2A
	Expanded Level III	2B
	Level IV	3
Army/USACE	Level III	2A
	Expanded Level III	2B
	Level IV	3
Navy	Level III	2B (3 rd party)
	Level IV	3 (3 rd party)
Other Federal Agencies		
EPA	Tier 1A	1
	Tier 1B	2A or 2B
	Tier 2	4 (on subset of results)
	Tier 3	4

There may be specific contracts that require components of SEDD 2B as part of their Level III validation specifications; **ALWAYS** check the master contract language when specifying DV services under this SOP. As contract-specific requirements are identified, please send them to j.hess@gilbaneco.com so they may be incorporated into this SOP in a future update.

4.0 PROCEDURES

The specific protocols used are outlined below for the review and validation of analytical data consistent with our CDQMP and the EPA “Stage” approach.

4.1 STAGE 1 VERIFICATION

Stage 1 verification, commonly referred to as a completeness check (or level II verification), of the laboratory analytical data package consists of verification of compliance and sample receipt conditions. The following items are considered and evaluated during Stage 1 verification of the sample delivery group (SDG):

- Laboratory report signed by official laboratory representative
- Chain-of-custody documentation including laboratory receipt information
- Laboratory case narrative and summary report
- Requested samples analyzed
- Requested analytes reported
- Results include:
 - requested analyte results and associated uncertainty (and type of uncertainty)
 - laboratory data qualifiers and definitions
 - reporting limits

- sample-specific critical value and sample-specific minimum detectable value, activity or concentration for results at or below the critical value
- chemical yield (if applicable to method), reference date and time
- units (both for results and associated uncertainty)
- sampling dates

At this time, Stage 1 is performed manually, until such time as Gilbane's environmental data management system (eDMS) supports electronic review of radiological data.

4.1.1 Manual Stage 1

Specific procedures for manual Stage 1 verification are provided below:

1. Verify the laboratory report clearly identifies the laboratory receiving the samples and performing the analyses and that the laboratory report is signed by an official laboratory representative.
2. Review chain-of-custodies for proper custody, and cooler receipts and sample login files for potential issues that may affect the usability of the samples (presence of custody seals, etc.) or validity of the reported results (receiving temperatures, sample condition, etc.).
3. Review laboratory case narrative for anomalies and QC issues.
4. Verify the analytical methods specified in the project plan were performed, and if substitutions were made, verify written instructions were received from the Gilbane Project Chemist specifying/allowing the substitution.
5. Verify all the target compounds identified in the project plan for each of the analyses were reported by the laboratory.
6. Verify all the analyses for each of the samples listed on the chain-of-custodies were completed. Verify the requested reporting limits were obtained.
7. For each analysis, verify the following information is provided:
 - the dates when the samples were collected and when analyses were performed
 - the requested analyte results and associated uncertainty (and type of uncertainty)
 - laboratory data qualifiers and definitions
 - sample-specific critical value and sample-specific minimum detectable value, activity or concentration for results at or below the critical value
 - chemical yield (if applicable to method)
 - units (both for results and associated uncertainty)
8. Document review with Stage 1 Verification Checklist, included in Attachment A. Upload completed checklist to eDMS.

4.1.2 Electronic Stage 1 Using eDMS

Currently performing Stage 1 electronically is not supported in eDMS. However, if the SDG is uploaded into eDMS, the verification checklist present in eDMS can be utilized.

4.2 STAGE 2A VALIDATION

Stage 2A validation (which can include some or all of the components of level III validation, depending on client) will evaluate and consider the items listed above for completeness, in addition to the following:

- Sample Specific Parameters
 - Sample Preservation
 - Holding Time
 - Sample Specific Chemical Yield
 - Required Detection Limit (RDL)
 - Nuclide Identification
 - Quantification and Uncertainty
 - Detectability
 - Sample Aliquot Representativeness
- Batch Control Parameters
 - Laboratory Control Sample (LCS) Analysis
 - Matrix Spike Sample (MSS) Analysis
 - Duplicate and Matrix Spike Duplicate (MSD) Sample Analysis
 - Batch Method Blank Analysis

Stage 2A is performed manually, until such time as Gilbane's eDMS supports electronic review of radiological data. Document review with Stage 2A Verification Checklist, included in Attachment A, or if the SDG is uploaded into eDMS, the verification checklist present in eDMS can be utilized. Data qualifiers generated during the validation process are entered into eDMS, as eDMS is the definitive source of data for generation of tables and figures for reporting purposes. Attachment B provides guidance for entering in qualifiers into eDMS.

4.2.1 Sample Specific Parameters

Perform the following activities, using either the provided checklists or equivalent.

1. Verify required **sample preservation** was performed per the approved project plans (or laboratory SOP if not specified in the project plans). Qualify as estimated ("J/UJ") with the appropriate reason code or reject ("R") based on professional judgment those results for samples improperly preserved
2. Verify analyses were performed within the required **holding times** as stipulated in the approved project plans (or laboratory SOP if not specified in the project plans). Qualify as estimated ("J/UJ") with the appropriate reason code those results that exceeded holding times.
3. Verify **sample-specific chemical yield** was performed consistent with the approved project plan or method requirements (or laboratory SOP) if not specified. Qualify as estimated ("J/UJ") with the appropriate reason code those results with sample-specific chemical yield varied from acceptable method requirements.

4. Verify the MDA meets the **required detection limit** (RDL) for all analytes of interest as stipulated in the approved project plans. Qualify as estimated (“J/UJ”) with the appropriate reason code those results for samples with the MDA not meeting the RDL.
5. Evaluate **detectability** by verifying results for all detected analytes of interest are greater than or equal to (“≥”) their Lc, and that those analytes of interest with results below their Lc are appropriately qualified as non-detect (“U”).

4.2.2 Batch Control Parameters

Verify QC parameters are within requirements stipulated in the approved project plans (or laboratory SOP if not specified in the project plans).

1. Verify the number of LCS analyses performed and the limits stipulated in the approved SAP (or laboratory SOP) were met.
 - Compare LCS analytical results to the bias and precision measurement quality objectives (MQOs) identified in the approved project plans. In general, individual LCS results should be compared with the least restrictive of the bias or precision MQO or overall measurement uncertainty MQO. Qualify as estimated (“J/UJ”) with the appropriate reason code those results associated with a batch with LCS results outside specified control limits.
2. Verify the number of MS analyses performed and the limits stipulated in the approved project plans (or laboratory SOP if not specified in the project plans) were met.
 - Compare MS analytical results to the bias and precision measurement quality objectives (MQOs) identified in the approved project plans. In general, individual MS results should be compared with the least restrictive of the bias or precision MQO or overall measurement uncertainty MQO. Qualify as estimated (“J”) with the appropriate reason code those results associated with a batch with MS results outside specified control limits.
3. Verify the number of duplicate and MSD analyses performed stipulated in the approved project plans (or laboratory SOP if not specified in the project plans) were met.
 - Compare relative percent difference (RPD) results for both duplicate and MSD analyses with the precision MQO stipulated in the approved project plans. If RPD is higher than stipulated in the approved project plans, evaluate duplicate results considering measurement uncertainty using a duplicate error ratio (DER) calculation against appropriate precision requirement. Use a DER limit of 2 (equivalent to 95% [2-sigma] confidence) if not otherwise stipulated in the approved SAP. Qualify as estimated (“J/UJ”) with the appropriate reason code those results associated with a batch with RSD results greater than specified in the approved project plans.
4. Verify the number of Method blanks (MBs) performed stipulated in the approved project plans (or laboratory SOP if not specified in the project plans) was met. Qualify as estimated (“J/UJ”) with the appropriate reason code those results associated with a batch with RSD results greater than specified in the approved project plans.

4.3 STAGE 2B VALIDATION

Most of Stage 2B is performed manually, until such time as Gilbane's eDMS supports electronic review of radiological data and associated QC. Document review with Stage 2B Verification Checklist, included in Attachment A, or if the SDG is uploaded into eDMS, the verification checklist present in eDMS can be utilized. Data qualifiers generated during the validation process are entered into eDMS, as eDMS is the definitive source of data for generation of tables and figures for reporting purposes. Attachment B provides guidance for entering in qualifiers into eDMS.

Stage 2B validation (which includes all of the components of level III validation, regardless of client) will evaluate and consider the items listed above for completeness and Stage 2A validation, in addition to the following:

- Instrument Parameters
 - Counting Efficiency Calibration
 - Energy Calibration
 - Background Determination

4.3.1 Counting Efficiency Calibration

Use the appropriate checklist provided in this SOP or checklist in Data Review/Checklist module in eDMS, or equivalent checklist (for 3rd party data validation) for verification of the following:

1. Verify instrument's efficiency calibrations are present and were performed within frequency required in approved project plans (or laboratory SOP if not specified in the project plans). Qualify as estimated ("J/UJ") with the appropriate reason code those results associated with a batch with less than required efficiency calibration.
2. Verify daily efficiency performance checks were performed. Qualify as estimated ("J/UJ") with the appropriate reason code those results associated with a batch with missing efficiency performance checks.
3. Verify check source counting statistics are present. Qualify as estimated ("J/UJ") with the appropriate reason code those results associated with a batch with missing check source counting statistics.
4. Verify efficiency performance checks were performed daily prior to counting samples. Qualify as estimated ("J/UJ") with the appropriate reason code those results associated with a batch with missing performance checks.

4.3.2 Energy Calibration

Use the appropriate checklist provided in this SOP, or equivalent checklist (for 3rd party data validation) for verification of the following:

1. Verify instrument's energy calibrations performed within frequency required in approved project plans (or laboratory SOP if not specified in the project plans). Qualify as estimated ("J/UJ") with the appropriate reason code those results associated with a batch with missing energy calibration.

2. Verify energy performance checks performed daily prior to counting samples. Qualify as estimated (“J”) with the appropriate reason code those results associated with a batch with missing energy performance checks.

4.3.3 Background Determination

Verify the following:

1. Verify background performance checks were performed at the required frequency identified in the approved project plans (or laboratory SOW if not specified in the project plans). Qualify as estimated (“J/UJ”) with the appropriate reason code those results associated with a batch with missing background performance checks.

4.4 STAGE 3 VALIDATION

Stage 3 validation builds upon the validation conducted in Stages 2A and 2B, and involves the verification by calculation of instrument and sample results from the laboratory instrument responses, and comparison of recalculated results to laboratory reported results. Document review with Stage 3 Validation Checklist, included in Attachment A, or if the SDG is uploaded into eDMS, the validation checklist present in eDMS can be utilized.

4.4.1 Sample Specific Parameters

1. Verify chemical yield is within required limits. If greater than 110%, qualify the sample result as estimated (J) or unusable (R) based on the amount of bias allowed by the MQOs.
2. For sample results at or below the Lc (non-detect), verify by calculation each analyte’s detectability. Estimate the Lc by multiplying the CSU by 1.65 (corresponding to 95% [2-sigma] probability). Qualify as estimated (“UJ”) with the appropriate reason code those results with $L_c < 1.65 \text{ times CSU}$. When appropriate background data (detector or blanks) is available, a more comprehensive evaluation of the Lc should be performed for those results qualified by the above calculation (see Section 4.7.2, ANSI/ANS-41.5-2012).
3. For sample results at or below the Lc (non-detect), determine whether the RDL has been met. Verify by calculation the CSU times 3.5 (corresponding to a 95% [2-sigma] probability) is less than or equal to (“ \leq ”) the RDL, or as otherwise stipulated in the approved project plans (or laboratory SOP if not specified in the project plans). Qualify as estimated (“UJ”) with the appropriate reason code those results when $3.5 \text{ times CSU} > \text{RDL}$ or as otherwise stipulated.
4. For sample results that are negative, evaluate results for excessive negative bias. Verify by calculation the absolute value of the negative result is less than (“ $<$ ”) 2 times the CSU, or as otherwise stipulated in the approved project plans (or laboratory SOP if not specified in the project plans). If absolute value of result is equal to or greater than 2 times CSU, qualify those results as estimated (“UJ”) with the appropriate reason code.

4.4.2 Batch Control Parameters

1. For a subset of the LCS analyses (10% or as otherwise stipulated in the approved SAP), verify recoveries are calculated correctly (see Section 5.1.1, ANSI/ANS-41.5-2013):

$$\%D = \frac{|LCS_M - LCS_E|}{LCS_E} \times 100$$

where:

LCS_M = measured concentration of each analyte in the LCS
LCS_E = expected concentration of each analyte in the LCS

If the LCS is calculated incorrectly, contact the laboratory for corrected results.

2. For a subset of the MS analyses (10% or as otherwise stipulated in the approved SAP), verify recoveries are calculated correctly (see Section 5.2.1, ANSI/ANS-41.5-2013):

$$\%D = \frac{|SSR - SR - SA|}{SA} \times 100$$

where:

SSR = spiked sample result
SR = sample result
SA = amount of spike added.

If the MS is calculated incorrectly, contact the laboratory for corrected results.

3. For a subset of the duplicate and MSD results (10% or as otherwise stipulated in the approved SAP), verify calculations for RPD and DER (see Section 5.3.1, ANSI/ANS-41.5-2013).

$$RPD = \frac{|S - D|}{[(S + D) / 2]} \times 100$$

and

$$DER = \frac{|S - D|}{\sqrt{(CSU_S)^2 + (CSU_D)^2}}$$

where:

RPD = relative present difference
DER = duplicate error ratio
S = first sample value
D = second sample value
CSU_S = first sample CSU
CSU_D = second sample CSU

If the RPD or DER is calculated incorrectly, contact the laboratory for corrected results.

4. For a subset of the MB analyses (10% or as otherwise stipulated in the approved project plans), verify by calculation the MB is less than 1.65 x CSU and/or within control limits.

Qualify as estimated (“J/UJ”) with the appropriate reason code those results with MB exceeding required limits.

4.4.3 Instrument Parameters

1. Evaluate whether the daily performance check results are within established tolerances [identified in the approved project plans (or laboratory SOP if not specified in the project plans)]. Qualify as estimated (“J/UJ”) with the appropriate reason code those results associated with a batch with efficiency checks are outside accepted tolerances.
2. Evaluate whether the reported counting uncertainty was less than or equal to 1/5 of the MQO [identified in the approved project plans]. Qualify as estimated (“J/UJ”) with the appropriate reason code those results associated with a batch with counting uncertainty outside acceptable criteria.
3. Verify the daily peak centroid or calculated energy for each peak in the performance check source or tolerance charts are within established tolerances identified in the approved project plans (or laboratory SOP if not specified in the project plans). Qualify as estimated (“J/UJ”) with the appropriate reason code those results associated with a batch with energy calibration outside acceptable criteria.

4.4.4 Background Determination

Use the appropriate checklist provided in this SOP or in eDMS, or equivalent checklist (for 3rd party data validation) for verification of the following:

- Verify instrument’s background was determined each time there was significant operational change and performed within frequency required in SAP (or laboratory SOW).
- Verify background performance check count-rate results are within established tolerances [identified in the SAP (or laboratory SOW)].
- Verify background performance check counting time was equal to or longer than sample counting time.

Qualify as estimated (“J”) with the appropriate reason code those results associated with a batch with one or more of the above issues related to instrument background determination.

4.4.5 Quantitation and CSU

1. Verify raw data and calculations used in developing the results, MDA, and CSU were included in the results.
2. Verify procedures and equations used are consistent with method requirements (or laboratory SOP).
3. Verify dates and time intervals used in calculations are correct.
4. Review by calculation a subset of the quantitation calculations (10% of target analytes or as otherwise stipulated in the approved project plans).

4.5 STAGE 4 VALIDATION

Stage 4 validation builds upon the validation conducted in Stage 3. Stage 4 validation of the laboratory analytical data package consists of the Stage 3 validation plus the evaluation of instrument outputs. Document review with Stage 4 Validation Checklist, included in Attachment A, or if the SDG is uploaded into eDMS, the verification checklist present in eDMS can be utilized.

4.5.1 Nuclide Identification

1. Verify the raw spectral data and/or peak search and identification reports were included in the results.
2. Review raw spectral data for potential errors, including but not limited to: misidentification of peaks, nonlinear energy response or skewed spectral peak positions, and unresolved overlapping peak interference.
3. Review the resolution and centroid position of peak associated with radio tracer (alpha spectroscopy).
4. Verify by calculation detector resolution and energy calibration parameters of spectrometry systems and peak centroid energy.

5.0 REQUIRED DOCUMENTATION

The following records generated as a result of implementation of this procedure must be maintained as quality records.

- Narrative summary
- QRT
- DAR or DQAR (as required)
- Validation and Verification Checklists

6.0 ATTACHMENTS

- A. Verification and Validation Checklists and Worksheets
- B. Entering Qualifiers into eDMS

7.0 FORMS

None.

8.0 REFERENCES

American Nuclear Society (ANS), 2012. *Verification and Validation of Radiological Data for Use in Waste Management and Environmental Remediation*, ANS/ANSI-41.5-2012, Approved by the American National Standards Institute, Inc. (ANSI). February.

Innovative Technical Solutions, Inc. (ITSI), 2010. *Chemical Data Quality Management Plan (CDQMP)*, March.

U.S. Department of the Navy, Southwest Division (NAVFAC-SW), 2001. *Environmental Work Instruction #1, Data Validation Guidelines for Chemical Analysis of Environmental Samples*. 28 November.

U.S. Army Corps of Engineers (USACE), 2005. EM 200-1-10, *Guidance for Evaluating Performance-Based Chemical Data*. 30 June.

U.S. Environmental Protection Agency (USEPA), 2009. EPA 540-R-8-005, *Guidance for Labeling Externally Validated Laboratory Analytical Data for Superfund Use*. January.

Attachments

Attachment A

Verification and Validation Checklists and Worksheets

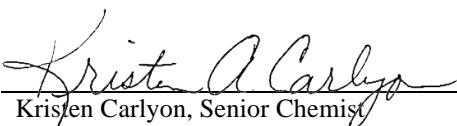
Item No.	Review Questions	Yes/No/NA	Comment
E901.1 – Gamma Emitting Radionuclides in Drinking Water			
Stage 2B VEM			
1	Sampling/Login: COC - Custody Trail?		
2	Sampling/Login: COC - Temperature/Condition?		
3	Sampling/Login: COC - Receipt anomalies?		
4	Sampling/Login: COC - Sample/Methods checked?		
6	Sampling/Login: Samples - Collection date?		
7	ICAL: Calibration - Verification source when calculated with the new efficiency must be $\pm 10\%$ of known value?		
8	ICAL: Full Width at the Half Maximum (FWHM) $< \pm 2$ sigma or FWHM ≤ 3.0 keV at 1332 KeV or reference manufactures specifications?		
9	ICAL: Peak Shape - FWHM difference < 0.5 keV for selected peaks – one low end (ie: 241Am), one middle (ie: 60Co) and one high (ie: 137Cs)		
10	ICAL: Background - Monthly - Long count and Daily - Short count ± 3 sigma of measured population.		
11	ICAL: Background counting time was equal to or longer than sample counting time?		
12	CCV: Daily Verification - Verification ± 3 sigma?		
13	Analysis: Samples - Extraction date?		
14	Analysis: Samples - Analysis date?		
15	Analysis: Samples - Holding time?		
16	Analysis: Samples - Batching?		
17	Analysis: Samples - Lab qualifiers?		
18	Analysis: Blank - Method blank $< \text{MDA}$?		
19	Analysis: Blank - Equipment blank $< \text{MDA}$?		
20	Analysis: Precision/Accuracy - MS/MSD?		
21	Analysis: Precision/Accuracy - LCS/LCSD (75-125%)?		
22	Analysis: Precision/Accuracy - Laboratory Duplicates (Act. $< 5 * \text{MDC}$ then RPD $< 100\%$, Act. $> 5 * \text{MDC}$ then $< 20\%$ RPD)?		
23	Analysis: Quantitation - PQLs (Lab RL $< \text{Project RL}$)?		
24	Analysis: Quantitation - MDA $< \text{required detection limit (RDL)}$. Formula $3.5 * \text{CSU} \leq \text{RDL}$? If this is not true then: If the result is less than L_c and the result plus 1.65 times its uncertainty is greater than the action level, it shall be qualified as (J/UJ).		
25	Analysis: Quantitation - Sample Uncertainty (Negative results less than $2X$ TPU flagged "U", $> 2X$ TPU flagged "UJ")?		
26	Analysis: Quantitation - Dilution Factor?		
27	Analysis: Field Duplicates - RPD in criteria?		
E905.0 – Radioactive Strontium			
Stage 2B VEM			
1	Sampling/Login: COC - Custody Trail?		
2	Sampling/Login: COC - Temperature/Condition?		
3	Sampling/Login: COC - Receipt anomalies?		
4	Sampling/Login: COC - Sample/Methods checked?		
5	Sampling/Login: Case Narratives - Anomalies (DQFs)?		
6	Sampling/Login: Samples - Collection date?		
7	ICAL: Calibration - Verification source when calculated with the new efficiency		

	must be $\pm 25\%$ of known value?		
8	ICAL: Background frequency performed at required interval?		
9	ICAL: Daily (short count) Weekly (long count) Verification - Background Check ± 3 sigma		
10	ICAL: Background updated upon instrument/operational change?		
11	ICAL: Background counting time was equal to or longer than sample counting time?		
12	CCV: Daily Verification - Efficiency Check ± 3 sigma		
13	Analysis: Samples - Extraction date?		
14	Analysis: Samples - Analysis date?		
15	Analysis: Samples - Holding time?		
16	Analysis: Samples - Batching?		
17	Analysis: Samples - Lab qualifiers?		
18	Analysis: Blank - Method blank $< \text{MDA}$?		
19	Analysis: Blank - Equipment blank $< \text{MDA}$?		
20	Analysis: Precision/Accuracy - MS/MSD?		
21	Analysis: Precision/Accuracy - LCS/LCSD (75-125%)?		
22	Analysis: Precision/Accuracy - Tracers/Carriers (25-125%)?		
23	Analysis: Precision/Accuracy - Laboratory Duplicates (Act. $< 5 \times \text{MDC}$ then RPD $< 100\%$, Act. $> 5 \times \text{MDC}$ then $< 20\%$ RPD or RER ≤ 3)?		
24	Analysis: Quantitation - PQLs (Lab RL $<$ Project RL)?		
26	Analysis: Quantitation - Sample Uncertainty (Negative results less than 2X TPU flagged "U", $> 2\text{X}$ TPU flagged "UJ")?		
27	Analysis: Quantitation - Dilution Factor?		
28	Analysis: Field Duplicates - RPD in criteria?		
HASL 300 – Alpha, Beta or Gamma Radioassay			
Stage 2B VEM			
1	Stage 2 Review: COC - Custody Trail?		
2	Stage 2 Review: COC - Temperature/Condition?		
3	Stage 2 Review: COC - Receipt anomalies?		
4	Stage 2 Review: COC - Sample/Methods checked?		
5	Stage 2 Review: Case Narrative - Anomalies?		
6	Stage 2 Review: Samples - Collection date?		
7	Stage 2 Review: Samples - Extraction date?		
8	Stage 2 Review: Samples - Analysis date?		
9	Stage 2 Review: Samples - Holding time?		
10	Stage 2 Review: Calibration - ICAL?		
11	Stage 2 Review: Calibration - ICV?		
12	Stage 2 Review: Calibration - CCV?		
13	Stage 2 Review: Blank - Method blank?		
14	Stage 2 Review: Blank - Trip blank?		
15	Stage 2 Review: Blank - Equipment blank?		
16	Stage 2 Review: Tracer/Carrier recoveries within limits?		
17	Stage 2 Review: Precision/Accuracy - MS/MSD?		
18	Stage 2 Review: Precision/Accuracy - LCS/LCSD?		
19	Stage 2 Review: Precision - Duplicate RPD Acceptable?		
20	Stage 2 Review: Reporting Limits Acceptable?		
21	Stage 2 Review: System Performance Acceptable?		
22	Stage 2 Review: Analysis: Quantitation - Sample Uncertainty (Negative results less than 2X TPU flagged "U", $> 2\text{X}$ TPU flagged "UJ")?		
23	Stage 2 Review: Overall Assessment of data in SDG?		
E903 – Total Alpha-Emitting Radium Isotopes			
Stage 2B VEM			
1	Stage 2 Review: COC - Custody Trail?		


2	Stage 2 Review: COC - Temperature/Condition?		
3	Stage 2 Review: COC - Receipt anomalies?		
4	Stage 2 Review: COC - Sample/Methods checked?		
5	Stage 2 Review: Case Narrative - Anomalies?		
6	Stage 2 Review: Samples - Collection date?		
7	Stage 2 Review: Samples - Extraction date?		
8	Stage 2 Review: Samples - Analysis date?		
9	Stage 2 Review: Samples - Holding time?		
10	Stage 2 Review: Calibration - ICAL?		
11	Stage 2 Review: Calibration - ICV?		
12	Stage 2 Review: Calibration - CCV?		
13	Stage 2 Review: Blank - Method blank?		
14	Stage 2 Review: Blank - Equipment blank?		
15	Stage 2 Review: Tracer/Carrier recoveries within limits?		
16	Stage 2 Review: Precision/Accuracy - MS/MSD?		
17	Stage 2 Review: Precision/Accuracy - LCS/LCSD?		
18	Stage 2 Review: Precision - Duplicate RPD Acceptable?		
19	Stage 2 Review: Reporting Limits Acceptable?		
20	Stage 2 Review: System Performance Acceptable?		
21	Stage 2 Review: Sample Uncertainty Review (Between 2 and 3 Sigma J/UJ over 3 Sigma R/R)?		
22	Stage 2 Review: Overall Assessment of data in SDG?		
E904 – Radium 228			
Stage 2B VEM			
1	Stage 2 Review: COC - Custody Trail?		
2	Stage 2 Review: COC - Temperature/Condition?		
3	Stage 2 Review: COC - Receipt anomalies?		
4	Stage 2 Review: COC - Sample/Methods checked?		
5	Stage 2 Review: Case Narrative - Anomalies?		
6	Stage 2 Review: Samples - Collection date?		
7	Stage 2 Review: Samples - Extraction date?		
8	Stage 2 Review: Samples - Analysis date?		
9	Stage 2 Review: Samples - Holding time?		
10	Stage 2 Review: Calibration - ICAL?		
11	Stage 2 Review: Calibration - ICV?		
12	Stage 2 Review: Calibration - CCV?		
13	Stage 2 Review: Blank - Method blank?		
14	Stage 2 Review: Blank - Equipment blank?		
15	Stage 2 Review: Tracer/Carrier recoveries within limits?		
16	Stage 2 Review: Precision/Accuracy - MS/MSD?		
17	Stage 2 Review: Precision/Accuracy - LCS/LCSD?		
18	Stage 2 Review: Precision - Duplicate RPD Acceptable?		
19	Stage 2 Review: Reporting Limits Acceptable?		
20	Stage 2 Review: System Performance Acceptable?		
21	Stage 2 Review: Sample Uncertainty Review (Between 2 and 3 Sigma J/UJ over 3 Sigma R/R)?		
22	Stage 2 Review: Overall Assessment of data in SDG?		

Work Instruction

Calculating Toxic Equivalence (TEQ) for Dioxins, Furans and Dioxin-Like Compounds

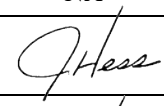
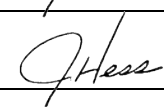
Reviewed by: 
Kristen Carlyon, Senior Chemist

Date: 27 Oct 2011

Approved by: 
Jeffrey Hess, Director Technical Services

Date: 27 Oct 2011

Review / Revision History:

Version	Changes	Affects Section/Pages	Review/ Revision Date	Approval*
1.0	Initial release		27 Oct 2011	NA
1.1	Add references to updated EPA National Functional Guidelines for dioxins and furans	Pages 2 and 4	27 June 2012	
1.2	Updated new company information. No other changes made.	All	04 Mar 2015	

* Approval required for reviews and minor changes only. Substantive revisions to the technical requirements contained in the work instruction require review and approval by the signatures to the work instruction.

1.0 PURPOSE AND SCOPE

The purpose of this work instruction is to describe the standard methodology for calculating the 2,3,7,8-tetrachlorodibenzo-p-dioxin (TCDD) toxic equivalency (TEQ). This methodology uses toxicity equivalence factors (TEFs) established for polychlorinated dibenzo-p-dioxins (PCDDs), polychlorinated dibenzofurans (PCDFs), and dioxin-like compounds (DLCs) by the World Health Organization (WHO) in 2005, and is consistent with U.S. Environmental Protection Agency (EPA) recommended practice.

There are several alternative methodologies for calculating the TEQ, some of which will be briefly discussed in this document. However, care should be taken in calculating TEQ using any of these alternative methodologies, and use of alternative methodologies should be clearly documented, including the rationale for use of the alternative methodology.

2.0 ACRONYMS AND DEFINITIONS

For purposes of this procedure, a number of terms and acronyms have the meanings defined below.

DLC: dioxin-like compound

EMPC: estimated maximum possible concentration

EDL: estimated detection limit

EQL: estimated quantitation limit

TCDD: 2,3,7,8-tetrachlorodibenzo-p-dioxin

TEF: toxicity equivalence factor

TEQ: toxic equivalency

PCDD: polychlorinated dibenzo-p-dioxin

PCDF: polychlorinated dibenzofuran

USEPA: U.S. Environmental Protection Agency

WHO: World Health Organization

3.0 BASIS OF CALCULATION

TEFs are published for seven 2,3,7,8-substituted isomers of PCDD's and ten 2,3,7,8-substituted isomers of PCDF's, and 12 polychlorinated biphenyls (PCBs) considered DLCs by WHO (2005) and recommended for use in human health risk assessment by EPA in *Recommended Toxicity Equivalence Factors (TEFs) for Human Health Risk Assessments of 2,3,7,8- Tetrachlorodibenzo-p-dioxin and Dioxin-Like Compounds* (2010). For PCDDs/PCDFs, the TEF's are used to convert concentrations of the specific isomers to "equivalent" concentrations of TCDD, the sum of which represents the samples TEQ.

As noted in EPA Method 8280B (revision 2) for *PCDDs and PCDFs by High-Resolution Gas Chromatography/ Low-Resolution Mass Spectrometry (HRGC/LRMS)*, when calculating TEQ for a sample:

...include only those 2,3,7,8-substituted isomers that were detected in the sample and met all of the qualitative identification criteria in Section 11.14.5. Do not include EMPC or EQL values in the TEQ calculation.

Section 11.14.5 of the method identifies a series of identification criteria that must be met for a peak to be "unambiguously identified" as a PCDD or PCDF. The criteria include: 1) retention times; 2) peak identification; 3) signal to noise ratio; 4) ion abundance ratios; and 5) other steps to minimize interference if the previous 4 steps are not met.

Further, as indicated in the *National Functional Guidelines for Chlorinated Dibenzo-p-Dioxins (CDDs) and Chlorinated Dibenzofurans (CDFs) Data Review* (EPA, 2005):

When calculating the 2,3,7,8-TCDD TEF-adjusted concentration of a sample, the laboratory must include only those 2,3,7,8-substituted isomers that were detected in the sample and that met all of the qualitative identification criteria. The laboratory does not include Estimated Maximum Possible Concentration (EMPC) or Estimated Detection Limit (EDL) values in the TEF calculations.

The updated *National Functional Guidelines for CDDs and CDFs Data Review* (EPA, 2011) reaffirmed EPA's position on the above methodology, but added in the option of considering EMPCs or EDLs in the calculation of TEQ as surrogates for non-detect results, depending on regional policies. The methodology specified in this work instruction is consistent with the methodology specified for the analytical laboratories and as identified in XII (C)(1)(a) of the National Functional Guidelines (EPA, 2011).

The published TEFs as established by the WHO in 2005 are provided below. These are for mammals, there are separate TEFs for fish and birds, so make sure the correct TEFs are used when performing ecological risk assessments.

PCDDs

<u>Compound</u>	<u>TEF</u>
2,3,7,8-TCDD	1
1,2,3,7,8-PeCDD	1
1,2,3,4,7,8-HxCDD	0.1
1,2,3,6,7,8-HxCDD	0.1
1,2,3,7,8,9-HxCDD	0.1
1,2,3,4,6,7,8-HpCDD	0.01
OCDD	0.0003

PCDFs

<u>Compound</u>	<u>TEF</u>
2,3,7,8-TCDF	0.1
1,2,3,7,8-PeCDF	0.03
2,3,4,7,8-PeCDF	0.3
1,2,3,4,7,8-HxCDF	0.1
1,2,3,6,7,8-HxCDF	0.1
1,2,3,7,8,9-HxCDF	0.1
2,3,4,6,7,8-HxCDF	0.1
1,2,3,4,6,7,8-HpCDF	0.01
1,2,3,4,7,8,9-HpCDF	0.01
OCDF	0.0003

Non-ortho-substituted PCBs

<u>Compound</u>	<u>TEF</u>
3,3',4,4'-TCB (77)	0.0001
3,4,4',5-TCB (81)	0.0003
3,3',4,4',5-PeCB (126)	0.1
3,3',4,4',5,5'-HxCB (169)	0.03

Mono-ortho-substituted PCBs

<u>Compound</u>	<u>TEF</u>
2,3,3',4,4'-PeCB (105)	0.00003
2,3,4,4',5-PeCB (114)	0.00003
2,3',4,4',5-PeCB (118)	0.00003
2',3,4,4',5-PeCB (123)	0.00003
2,3,3',4,4',5-HXCB (156)	0.00003
2,3,3',4,4',5'-HxCB (157)	0.00003
2,3',4,4',5,5'-HxCB (167)	0.00003
2,3,3',4,4',5,5'-HpCB (189)	0.00003

4.0 CALCULATION METHOD

The TEQ is calculated by multiplying the reported value of the 2,3,7,8-substituted isomer by the corresponding TEF shown above, and then summing the resulting values. As noted above, the TEF should only be calculated for isomers that were detected and met all of the identification criteria identified in the analytical method; EMPC or EDL values should not be included in the calculation of TEQ.

An example calculation is shown below, with the shaded portion typical of the information the laboratory will provide:

Isomer	Result	EMPC	Qualifier	DL	LOQ	TEF	Isomer-specific TEQ
2,3,7,8-TCDD	ND		U	0.176	0.539	1	–
1,2,3,7,8-PeCDD	ND		U	0.367	2.69	1	–
1,2,3,4,7,8-HxCDD	0.500		J	0.420	2.69	0.1	0.05
1,2,3,6,7,8-HxCDD	3.76			0.445	2.69	0.1	0.376
1,2,3,7,8,9-HxCDD	1.87		J	0.438	2.69	0.1	0.187
1,2,3,4,6,7,8-HpCDD	133			1.13	2.69	0.01	1.33
OCDD	734			1.83	5.39	0.0003	0.2202
2,3,7,8-TCDF		0.511	J	0.240	0.539	0.1	–
1,2,3,7,8-PeCDF	ND		U	0.205	2.69	0.03	–
2,3,4,7,8-PeCDF	ND		U	0.199	2.69	0.3	–
1,2,3,4,7,8-HxCDF		0.433	J	0.259	2.69	0.1	–
1,2,3,6,7,8-HxCDF	ND		U	0.243	2.69	0.1	–
1,2,3,7,8,9-HxCDF		0.435	J	0.260	2.69	0.1	–
2,3,4,6,7,8-HxCDF	ND		U	0.296	2.69	0.1	–
1,2,3,4,6,7,8-HpCDF	3.17			0.274	2.69	0.01	0.0317
1,2,3,4,7,8,9-HpCDF	ND		U	0.354	2.69	0.01	–
OCDF	4.01		J	1.00	5.39	0.0003	<u>0.001203</u>
						Total TEQ:	2.20

PCDD and PCDF results are typically reported in pg/g, equivalent to parts per trillion (ppt).

5.0 DISCUSSION

Inclusion of non-detect and estimated results in TEQ calculations varies by regulatory agency and ultimate use of the results. While the above methodology is consistent with:

- EPA National Functional Guidelines (EPA, 2005)(EPA, 2011)
- EPA Region II Data Validation SOP (EPA, 2006)
- EPA Method 8280B (EPA, 2007)
- Bay Area Clean Water Agencies (BACWA) Guidance (BACWA, 2010)

Other jurisdictions (including EPA Region 4 [EPA, 2008]) include both estimated and non-detect values, with the EDLs being used for non-detect values. Additionally, the State of Florida methodology for calculating TEQ (Florida Department of Environmental Protection [FDEP], 2005) uses older 1997 WHO TEF values and ½ the method detection limit for non-detect values (based on FDEP's excel-based *Dioxin Conversion Table* at http://www.dep.state.fl.us/waste/quick_topics/publications/wc/DioxinConversionTable.xls).

6.0 REFERENCES

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FDEP, 2005. *Technical Report: Development of Cleanup Target Levels (CTLs) For Chapter 62-777, F.A.C.*, February.

Van den Berg, et al, 2006. *The 2005 World Health Organization Re-evaluation of Human and Mammalian Toxic Equivalency Factors for Dioxins and Dioxin-like Compounds*. Toxicological Sciences Volume 93, Issue 2, pp. 223-241, October.

ATTACHMENT 2

DoD QSM Laboratory Limits

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Attachment 2.1 DOD QSM Laboratory Limits - Volatile Organics in Soil
Sampling and Analysis Plan
Phase IV NTCRA for SWDA Westside, IR Site 12
Former Naval Station Treasure Island, San Francisco, California

Analyte	Lower Limit	Upper Limit	RPD
1,1,1,2-Tetrachloroethane	78	125	20
1,1,1-Trichloroethane	73	130	20
1,1,2,2-Tetrachloroethane	70	124	20
1,1,2-Trichloroethane	78	121	20
1,1-Dichloroethane	76	125	20
1,1-Dichloroethene	70	131	20
1,2,3-Trichlorobenzene	66	130	20
1,2,3-Trichloropropane	73	125	20
1,2,4-Trichlorobenzene	67	129	20
1,2,4-Trimethylbenzene	75	123	20
1,2-Dibromo-3-chloropropane	61	132	20
1,2-Dibromoethane (EDB)	78	122	20
1,2-Dichlorobenzene	78	121	20
1,2-Dichloroethane	73	128	20
1,2-Dichloroethane-d4 (surrogate)	71	136	NA
1,2-Dichloropropane	76	123	20
1,3,5-Trimethylbenzene	73	124	20
1,3-Dichloropropane	77	121	20
1,4-Dichlorobenzene	75	120	20
2-Butanone	51	148	20
2-Chlorotoluene	75	122	20
2-Hexanone	53	145	20
4-Bromofluorobenzene(BFB) -(surrogate)	79	119	NA
4-Chlorotoluene	72	124	20
4-Methyl-2-pentanone	65	135	20
Acetone	36	164	20
Bromobenzene	78	121	20
Bromochloromethane	78	125	20
Bromodichloromethane	75	127	20
Bromoform	67	132	20
Bromomethane	53	143	20
Carbon disulfide	63	132	20
Carbon tetrachloride	70	135	20
Chlorobenzene	79	126	20
Chloroform	78	123	20
Chloromethane	50	136	20
cis-1,2-Dichloroethene	77	123	20
Dibromochloromethane	74	126	20
Dibromofluoromethane (surrogate)	78	119	NA
Dibromomethane	78	125	20
Dichlorodifluoromethane (Freon 12)	29	149	20
Ethylbenzene	76	122	20
Hexachlorobutadiene	61	135	20
Methyl tert-Butyl ether (MTBE)	73	125	20
Methylene chloride	70	128	20
m,p-Xylenes ³	77	124	20
o-Xylene	77	123	20

Attachment 2.1 DOD QSM Laboratory Limits - Volatile Organics in Soil
Sampling and Analysis Plan
Phase IV NTCRA for SWDA Westside, IR Site 12
Former Naval Station Treasure Island, San Francisco, California

Analyte	Lower Limit	Upper Limit	RPD
n-Butylbenzene	70	128	20
n-Propylbenzene	73	125	20
Styrene	76	124	20
Tetrachloroethene	73	128	20
Toluene	77	121	20
Toluene d8 (surrogate)	85	116	NA
trans-1,2-Dichloroethene	74	125	20
trans-1,3-Dichloropropene	71	130	20
Trichloroethene	77	123	20
Trichlorofluoromethane (Freon 11)	62	140	20
Vinyl chloride	56	135	20

Notes:

NA = not applicable

RPD = relative percent difference

Attachment 2.2 DOD QSM Laboratory Limits - Semivolatile Organics in Soil
Sampling and Analysis Plan
Phase IV NTCRA for SWDA Westside, IR Site 12
Former Naval Station Treasure Island, San Francisco, California

Analyte	Lower Limit	Upper Limit	RPD
2,4,5-Trichlorophenol	41	124	20
2,4,6-Tribromophenol (surrogate)	39	132	NA
2,4,6-Trichlorophenol	39	126	20
2,4-Dichlorophenol	40	122	20
2,4-Dimethylphenol	30	127	20
2,4-Dinitrophenol	Lab Limits	Lab Limits	20
2,4-Dinitrotoluene	48	126	20
2,6-Dinitrotoluene	46	124	20
2-Chloronaphthalene	41	114	20
2-Chlorophenol	34	121	20
2-Fluorobiphenyl (surrogate)	44	115	NA
2-Fluorophenol (surrogate)	35	115	NA
2-Nitroaniline	44	127	20
3,3'-Dichlorobenzidine	33	119	20
4-Chloroaniline	17	106	20
4-Nitroaniline	30	130	20
Benzyl alcohol	29	122	20
bis(2-Chloroethoxy)methane	36	121	20
bis(2-Chloroethyl)ether	31	131	20
bis(2-Ethylhexyl)phthalate	51	133	20
Dibenzofuran	44	120	20
Diethylphthalate	50	124	20
Di-n-butylphthalate	51	128	20
Hexachlorobenzene	45	122	20
Hexachlorobutadiene	32	123	20
Hexachloroethane	28	117	20
Isophorone	30	122	20
Nitrobenzene	34	122	20
Nitrobenzene-d5 (surrogate)	37	122	NA
n-Nitrosodiphenylamine	38	127	20
n-Nitroso-di-n-propylamine	36	120	20
Pentachlorophenol	25	133	20
Phenol	34	131	20
Phenol-d5 (surrogate)	33	122	NA
Terphenyl-d14(surrogate)	54	127	NA

Attachment 2.3 DOD QSM Laboratory Limits - PCBs and TPH in Soil
Sampling and Analysis Plan
Phase IV NTCRA for SWDA Westside, IR Site 12
Former Naval Station Treasure Island, San Francisco, California

PCBs in Soil

Analyte	Lower Limit	Upper Limit	RPD
Aroclor 1016	44	134	30
Aroclor 1260	53	130	30
Decachlorobiphenyl (surrogate)	Lab Limits	Lab Limits	NA
Tetrachloro-m-xylene (surrogate)	44	130	NA

TPH-GRO in Soil

Analyte	Lower Limit	Upper Limit	RPD
TPH-GRO	79	122	30
4-Bromofluorobenzene	67	134	NA

TPH-DRO, MO in Soil

Analyte	Lower Limit	Upper Limit	RPD
TPH-DRO	38	132	30
TPH-MO	39	106	30
o-Terphenyl	45	130	NA

Notes:

NA = not applicable

PCBs = polychlorinated biphenyls

RPD = relative percent difference

TPH = total petroleum hydrocarbons

Attachment 2.4 DOD QSM Laboratory Limits - Polycyclic Aromatic Hydrocarbons in Soil
Sampling and Analysis Plan
Phase IV NTCRA for SWDA Westside, IR Site 12
Former Naval Station Treasure Island, San Francisco, California

Analyte	Lower Limit	Upper Limit	RPD
Acenaphthene	44	111	20
Anthracene	50	114	20
Benzo(a)anthracene	54	122	20
Benzo(a)pyrene	50	125	20
Benzo(b)fluoranthene	53	128	20
Benzo(k)fluoranthene	56	123	20
Chrysene	57	118	20
Dibenz(a,h)anthracene	50	129	20
Fluoranthene	55	119	20
Fluorene	57	114	20
Indeno(1,2,3-cd)pyrene	49	130	20
2-Methylnaphthalene	39	114	20
Naphthalene	38	111	20
Pyrene	55	117	20
Nitrobenzene-d5 (surrogate)	Lab Limits	Lab Limits	NA
2-Fluorobiphenyl (surrogate)	Lab Limits	Lab Limits	NA
Terphenyl-d14 (surrogate)	Lab Limits	Lab Limits	NA

Notes:

NA = not applicable

PAHs = polycyclic aromatic hydrocarbons

RPD = relative percent difference

Attachment 2.5 DOD QSM Laboratory Limits - Pesticides in Soil
Sampling and Analysis Plan
Phase IV NTCRA for SWDA Westside, IR Site 12
Former Naval Station Treasure Island, San Francisco, California

Analyte	Lower Limit	Upper Limit	RPD
4,4'-DDD	56	139	30
4,4'-DDE	56	134	30
4,4'-DDT	50	141	30
Aldrin	45	136	30
alpha-BHC	45	137	30
beta-BHC	50	136	30
Chlordane (technical)	Lab Limits	Lab Limits	30
Dieldrin	56	136	30
Endosulfan I	53	132	30
Endosulfan II	53	134	30
Endrin	57	140	30
gamma-BHC (Lindane)	49	135	30
Heptachlor	47	136	30
Heptachlor epoxide	52	136	30
Methoxychlor	52	143	30
Toxaphene	33	141	30
Tetrachloro-m-xylene (surrogate)	42	129	NA
Decachlorobiphenyl (surrogate)	Lab Limits	Lab Limits	NA

Notes:

NA = not applicable

RPD = relative percent difference

Attachment 2.6 DOD QSM Laboratory Limits - Dioxins/Furans in Soil
Sampling and Analysis Plan
Phase IV NTCRA for SWDA Westside, IR Site 12
Former Naval Station Treasure Island, San Francisco, California

Analyte	Lower Limit	Upper Limit	RPD
2,3,7,8-TCDD	70	128	20
1,2,3,7,8-PeCDD	74	125	20
1,2,3,4,7,8-HxCDD	72	131	20
1,2,3,6,7,8-HxCDD	74	134	20
1,2,3,7,8,9-HxCDD	71	138	20
1,2,3,4,6,7,8-HpCDD	76	125	20
OCDD	73	135	20
2,3,7,8-TCDF	75	135	20
1,2,3,7,8-PeCDF	74	125	20
2,3,4,7,8-PeCDF	77	131	20
1,2,3,4,7,8-HxCDF	72	131	20
1,2,3,6,7,8-HxCDF	73	134	20
1,2,3,7,8,9-HxCDF	74	135	20
2,3,4,6,7,8-HxCDF	74	133	20
1,2,3,4,6,7,8-HpCDF	73	135	20
1,2,3,4,7,8,9-HpCDF	72	131	20
OCDF	66	144	20

Notes:

NA = not applicable

RPD = relative percent difference

Attachment 2.7 DOD QSM Laboratory Limits - Metals in Soil
Sampling and Analysis Plan
Phase IV NTCRA for SWDA Westside, IR Site 12
Former Naval Station Treasure Island, San Francisco, California

ICP in Soil

Analyte	Lower Limit	Upper Limit	RPD
Lead	81	112	20

ICPMS in soil

Analyte	Lower Limit	Upper Limit	RPD
Antimony	71	129	20
Arsenic	77	123	20
Barium	81	119	20
Beryllium	78	122	20
Cadmium	82	118	20
Chromium	81	119	20
Cobalt	79	121	20
Copper	81	119	20
Lead	78	122	20
Molybdenum	83	114	20
Nickel	81	119	20
Selenium	79	121	20
Silver	54	146	20
Thallium	83	118	20
Vanadium	82	118	20
Zinc	73	127	20

Mercury in Soil

Analyte	Lower Limit	Upper Limit	RPD
Mercury	80	124	20

Notes:

NA = not applicable

RPD = relative percent difference

ATTACHMENT 3

Laboratory Standard Operating Procedures

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SOP Minor Revision Summary

SOP:			
Title -	POLYCHLORINATED BIPHENYLS (PCBS) BY GAS CHROMATOGRAPHY (SOIL, WATER & OIL) (EPA METHODS 608, 608.3, 8082, & 8082A, SM 6431B)		
Number -	330343	Department -	SVOA
Revision -	17	Rev. Date -	3/23/2018

This Standard Operating Procedure has been amended to include changes required during normal business operations. These changes as defined by SOP 010103 (Document Control and Distribution) are routine modifications that will be incorporated into the SOP upon the next scheduled review.

Rev.	Date	Section	Brief Description
a	8/7/18	8.5	Add guidance for Aroclor and Chlordane Identification



Number: 330343
Analysis: PCBs
Date/rev: 3/23/18 R17
Page 1 of 51

Standard Operating Procedure

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**TITLE: POLYCHLORINATED BIPHENYLS (PCBS) BY GAS CHROMATOGRAPHY
(SOIL, WATER & OIL) (EPA METHODS 608, 608.3, 8082, & 8082A, SM 6431B)**

Reviewed by: Blake Judge, Chris Johnson, Steve Miller

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QA Department

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1.0 SCOPE AND APPLICATION

NOTE: EPA Methods 608 and 608.3 include the analysis of pesticides. For direction regarding pesticide analysis using these methods, see ESC SOP #330344, *Chlorinated Pesticides by GC*.

STATE NOTE: For samples analyzed in conjunction with the Ohio Voluntary Action Program (VAP) please utilize SOP #330343OH.

- 1.1 This standard operating procedure describes a gas chromatographic method for the determination of polychlorinated biphenyls (PCBs) as Aroclors. It is used for waste samples, waters, soils, sediments, and other solid samples. Compounds analyzed by this method and their typical reporting limits are found below (subject to change).

Analyte	CAS No./ IUPAC No.:	Soil mg/kg	Water mg/L
Aroclor 1016	12674-11-2	0.017	0.0005
Aroclor 1221	11104-28-2	0.017	0.0005
Aroclor 1232	11141-16-5	0.017	0.0005
Aroclor 1242	53469-21-9	0.017	0.0005
Aroclor 1248	12672-29-6	0.017	0.0005
Aroclor 1254	11097-69-1	0.017	0.0005
Aroclor 1260	11096-82-5	0.017	0.0005
Aroclor 1262* [†]	37324-23-5	--	0.0005
Aroclor 1268*	11100-14-4	--	0.0005

* See section 13.3.

[†] Not a target analyte in Method 608.3

- 1.2 Aroclors are multi-component mixtures. When samples contain more than one Aroclor, a higher level of analyst expertise is required to attain acceptable levels of qualitative and quantitative analysis. The same is true of Aroclors that have been subjected to environmental degradation ("weathering") or degradation by treatment technologies.

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Standard Operating Procedure

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**TITLE: POLYCHLORINATED BIPHENYLS (PCBS) BY GAS CHROMATOGRAPHY
(SOIL, WATER & OIL) (EPA METHODS 608, 608.3, 8082, & 8082A, SM 6431B)**

Such weathered multi-component mixtures may have significant differences in peak patterns than those of Aroclor standards.

- 1.3 Quantitation of PCBs as Aroclors is appropriate for many regulatory compliance determinations, but is particularly difficult when the Aroclors have been weathered by long exposure in the environment.
- 1.4 A Method Detection Limit (MDL) study must be completed at least annually or more frequently if major instrumentation changes occur. MDLs are performed based on ESC SOP #030206. Updated MDL records are filed and stored on ESC's intranet.
 - 1.4.1 Limit of Detection (LOD) and Limit of Quantitation (LOQ) studies are completed at the frequency required by the TNI standard per the procedure identified in the ESC SOP #030206, *Method Detection Limits (MDL), Limits of Detection (LOD) and Limits of Quantitation (LOQ)*. Should the procedure be utilized for DOD support; then the frequency of these studies must meet the requirements of the current DOD QSM (see Attachment II).
 - 1.4.2 Lower Limit of Quantitation (LOQ) – For analyses performed per the requirements of Method 8000D, the LLOQ is established at concentrations where both quantitative and qualitative requirements can consistently be met (see Sections 2.8 and 10.16).
 - 1.4.3 When analyzing the PCBs as Aroclors, it is only necessary to establish an MDL for one of the multi-component analytes (e.g., PCB 1254), or the mixture of Aroclors 1016 and 1260 may be used to establish MDLs for all of the Aroclors.

2.0 METHOD SUMMARY AND DEFINITIONS

- 2.1 A measured volume or weight of sample (approximately 100mL or 1L for liquids, up to 30g for solids) is extracted using the appropriate matrix-specific sample extraction technique.
- 2.2 Aqueous samples are extracted at neutral pH with methylene chloride using EPA method 3510C (separatory funnel) or other appropriate technique. Reduced volume (RV) extraction using EPA method 3510C that requires a smaller volume (usually 100mL) of field sample is also available for use where applicable. See section 13.4 of this procedure and ESC SOP #330702B. The resulting extracts are exchanged in Hexane for final solvent and concentrated using ESC SOP #330708, Buchi Syncore Concentration System.
- 2.3 Solid samples are extracted with methylene chloride using EPA methods 3546 (microwave). The extract is exchanged in Hexane for final solvent. Extracts from solid samples may or may not require concentration depending on instrumentation used for analysis and the data quality objectives of the client project. Non-concentrated extracts may utilize a Large Volume Injection (LVI) technique if instrumentation is equipped with proper inlet or if sensitivity at detection is not sufficient.
- 2.4 Oily matrices are subjected to waste dilution according to EPA method 3580A.

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(SOIL, WATER & OIL) (EPA METHODS 608, 608.3, 8082, & 8082A, SM 6431B)**

- 2.5 Extracts for PCB analysis may be subjected to a sulfuric acid/potassium permanganate cleanup (EPA Method 3665A), silica gel cleanup (EPA Method 3630C), and Sulfur Cleanup (EPA Method 3660B). These cleanup techniques remove many single component organochlorine/organophosphorus pesticides, sulfur and other non-target analytes that can interfere with the identification and quantitation of PCBs; therefore, cleaned extracts for analysis using Method 8082 are not applicable to the analysis of those compounds (see EPA Method 8081).
- 2.6 Routinely, an internal standard is added to the sample extract then the extract is injected into a gas chromatograph equipped with a capillary column and an electron capture detector; however in cases where there is an obvious interferent co-eluting with the internal standard peak, extracts without internal standard are analyzed and quantitation using external calibration is performed.
- 2.7 The chromatographic data may be used to determine the nine Aroclors in Sec. 1.1 or total PCBs.
- 2.8 Lower Limit of Quantitation (LLOQ) – For analyses performed according to the requirements of Method 8000D, the lowest concentration at which the laboratory has demonstrated target analytes can be reliably measured and reported with a certain degree of confidence, which must be greater than or equal to the lowest point in the calibration curve.
- 2.9 LVI: Large Volume Injection: any injection volume >5ul. Technique is dependent upon type of GC inlet used and sensitivity of detection.
- 2.10 See the current Quality Assurance Manual for definitions associated with terms found in this document.
- 3.0 HEALTH AND SAFETY
- 3.1 The toxicity or carcinogenicity of each reagent used in the laboratory has not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds must be as low as reasonably achievable. A reference file of safety data sheets (SDSs) is made available on ESC's intranet to all personnel. Use hazardous reagents in a fume hood whenever possible and if eye or skin contact occurs, flush with large volumes of water. Always wear safety glasses or a shield for eye protection, protective clothing and observe proper mixing protocols.
- 3.2 Many of the compounds determined by this methodology have been identified as known or putative carcinogens in man and/or animals. Exposure to these compounds must be reduced to a minimum. Neat standards should be handled in a fume hood. The analyst must use gloves to minimize the possibility of trans-dermal adsorption of these compounds.
- 3.3 Since the electron capture detector is a non-destructive detector, effluent from the gas chromatograph must be vented through an adsorption trap. Large quantities of the dichloromethane extraction solvent should be handled in the fume hood.

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- 3.4 Wear safety glasses, gloves, and laboratory coat to protect against physical contact with samples that contain potentially hazardous chemicals.
- 4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE
- 4.1 All samples must have been collected using a sampling plan that addresses the considerations of this method.
- 4.2 The sample containers must be glass or Teflon and have screw caps with Teflon-lined septa. Sample containers must be filled with care to prevent any portion of the collected sample contacting the sampler's gloves, thus causing possible contamination. Samples must not be collected or stored in the presence of exhaust fumes. If the sample contacts the sampler (e.g. if an automatic sampler is used), run organic-free reagent water through the sampler and utilize the rinsate as a field blank.
- 4.3 If residual chlorine is present, water samples are preserved with 3mL of 10% sodium thiosulfate per gallon and cooled to $4 \pm 2^{\circ}\text{C}$. Water samples are collected in a 1L amber bottle with Teflon lined caps and must be extracted within 365 days of collection and analyzed within 40 days following the extraction. See section 13.5.
- 4.4 Soils are collected in wide mouth jars with Teflon lined caps and are cooled to $4 \pm 2^{\circ}\text{C}$ upon collection. Soils must be extracted within 365 days of collection and analyzed within 40 days following extraction. See section 13.5.
- 4.5 All analytical glassware must be cleaned according to SOP #030701, *Glassware Cleaning*.
- 4.6 Samples submitted for analysis that do not meet the requirements contained within this section must be addressed before performing the logging process within the laboratory. In some cases, exceeding the appropriate preservation and storage criteria can cause significant bias in the resulting data. Clients may need to resubmit samples where the conditions during shipment cause uncertainty regarding sample integrity. If samples do not meet the requirements for preservation, sampling, shipment and storage and the client approves the completion of the analytical process, sample results can be qualified and possible bias is narrated per the ESC SOP #030201, *Data Handling and Reporting*.
- 4.7 Method 608.3 allows the use of hydrogen as a carrier gas in place of helium. If used, the laboratory should take the necessary precautions in dealing with hydrogen, and should limit hydrogen flow at the source to prevent buildup of an explosive mixture of hydrogen in air.
- 5.0 INTERFERENCES
- 5.1 Interferences co-extracted from the samples vary considerably from matrix to matrix. While general cleanup techniques are referenced or provided as part of this method, unique samples may require additional cleanup approaches to achieve desired degrees of discrimination and quantitation. Sources of interference in this method can be grouped into three broad categories.
- 5.1.1 Contaminated solvents, reagents, or sample processing hardware.

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- 5.1.2 Contaminated GC carrier gas, parts, column surfaces, or detector surfaces.
- 5.1.3 Compounds extracted from the sample matrix to which the detector will respond.
- 5.2 Interferences by phthalate esters introduced during sample preparation can pose a major problem in PCB determinations.
 - 5.2.1 Common flexible plastics contain varying amounts of phthalate esters that are easily extracted or leached from such materials during laboratory operations. Interferences from phthalate esters can best be minimized by avoiding contact with any plastic materials and checking all solvents and reagents for phthalate contamination.
 - 5.2.2 Exhaustive cleanup of solvents, reagents and glassware may be required to eliminate background phthalate ester contamination.
 - 5.2.3 Phthalate esters can be removed through the use of Method 3665A (sulfuric acid/permanganate cleanup).
- 5.3 Cross-contamination of clean glassware routinely occurs when plastics are handled during extraction steps, especially when solvent-wetted surfaces are handled. Glassware must be scrupulously cleaned. Clean all glassware as soon as possible after use by rinsing with the last solvent used. Detergent washing with hot water and rinses with tap water and organic-free reagent water follow. Drain the glassware, and dry it in an oven at 130°C for several hours, or rinse with methanol and drain. Store dry glassware in a clean environment.

NOTE: Oven-drying of glassware used for PCB analysis can increase contamination because PCBs are readily volatilized in the oven and spread to other glassware. Therefore, exercise caution and do not dry glassware from samples containing high concentrations of PCBs with glassware that may be used for trace analyses.
- 5.4 Elemental sulfur (S) is readily extracted from soil samples and may cause chromatographic interferences in the determination of PCBs. Sulfur can be removed using EPA Method 3660B. Other non-target contaminants can be cleaned from extracts using EPA Methods 3665A, 3620C, or 3630C. See the relevant ESC SOPs for more information regarding the use and procedure for these cleanup methods.
- 5.5 If co-elutions occur in analysis of a sample, a co-elution on one column is acceptable so long as effective separation of the co-eluting compounds can be achieved on the second column.

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6.0 EQUIPMENT AND SUPPLIES

6.1 Instrumentation (equivalent substitutions may be made)

Instrument name:	SVGC #18	SVGC #28
Use (method #'s):	8082, 608	8082, 608
Model #:	Agilent 6890	Agilent 7890
Column (type, brand, size):	STX-CLPesticides 30m x 0.32mm x 0.5um, STX-CLPesticides II 30m x 0.32mm x 0.25um	STX-CLPesticides 30m x 0.32mm x 0.5um, STX-CLPesticides II 30m x 0.32mm x 0.25um
Detector:	Dual Micro ECD	Dual micro ECD
Software name and version:	EnviroQuant Chemstation G1701DA	EnviroQuant Chemstation G1701EA
Software version:	D.00.01.27	E.02.00
Sample introduction system:	HP 7683 AS	Agilent 7693 AS
Computer name:	SVCOMPD	SVCOMPAT
Computer brand, and model #:	HP Compaq	HP Compaq
Gases used (grade and supplier):	N2 – Zero Grade/He/H ₂	N2 – Zero Grade/He/H ₂

- 6.2 Vials 10 - 15mL with Teflon lined screw caps
- 6.3 Syringes - Hamilton Gastight or equivalent: 1mL, 250µL, 100µL, 10µL
- 6.4 40mL vials with Teflon lined caps
- 6.5 9" VWR Disposable Pasteur Pipette, or equivalent
- 6.6 10mL Pyrex Disposable Pipette, or equivalent
- 6.7 1.8mL Wheaton ABC Vials with Teflon rubber lined caps or equivalent
- 6.8 10mL Pyrex Volumetric Flasks - Class "A" or equivalent.

7.0 REAGENTS AND STANDARDS

- 7.1 All reagents and standards must be recorded in the appropriate preparation log and assigned a unique number. See SOP #030230, *Standards Logger – Tree Operation*. Additional information regarding reagent preparation can be found in the Standards Logger (Tree) digital archive system. All spiking solutions and surrogate standard solutions should be replaced at least every 6 months or sooner if a problem is detected unless otherwise noted.
- 7.2 Pesticide grade chemicals are used in all tests.

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NOTE: Store the standard solutions (stock, composite, calibration, internal, and surrogate standards) according to manufacturer's guidance. Routinely, store PCB standard and spiking solutions at <6°C in polytetrafluoroethylene (PTFE)-sealed containers in the dark. When a lot of standards is prepared, it is recommended that aliquots of that lot be stored in individual small vials for protection from degradation and possible contamination.

7.3 Aroclor stocks, working and calibration standards - The laboratory working spike standard is made using 500uL, or 2.5uL for non-concentrated soil and RV of a 200ug/mL Pesticides Surrogate Standard Spiking Solution (Ultra Scientific Cat# ISM-320) and 100uL, or 0.5uL for RV, each of Aroclor 1016 & 1260 at 1000ug/mL. The final volume is 10mL in Hexane and the final concentration is 10ppm and 50ppb for non-concentrated soil and RV. Stock standards for each Aroclor are received at 1.0mg/mL and are diluted as follows for calibration working standards. Equivalent substitutions of purchased standards and calibration standard levels may be made.

- PCB 1016 - AccuStandard Cat# C-2165-H-10x
- PCB 1260 - AccuStandard Cat# C-2605-H-10x
- PCB 1221 - AccuStandard Cat# C-2215-H-10x
- PCB 1232 - AccuStandard Cat# C-2325-H-10x
- PCB 1242 - AccuStandard Cat# C-2425-H-10x
- PCB 1248 - AccuStandard Cat# C-2485-H-10x
- PCB 1254 - AccuStandard Cat# C-2445-H-10x
- PCB 1262 - AccuStandard Cat# C-262S-H-10X
- PCB 1268 - AccuStandard Cat# C-268S-H-10X

Calibration standards are produced using this solution at the concentrations below. Also see section 13.2.

For concentrated Soil and 1L extracted analyses:

Compound	Std1 µg/mL	Std2 µg/mL	Std3 µg/mL	Std4* µg/mL	Std5 µg/mL	Std6 µg/mL
Amount of Intermediate added (uL)	50uL	100uL	250uL	500uL	750uL	1000uL
Final Volume (mL)	1mL	1mL	1mL	1mL	1mL	1mL
Analyte Concentrations						
1016	.05ppm	.10ppm	.25ppm	.50ppm	.75ppm	1.0ppm
1260	.05ppm	.10ppm	.25ppm	.50ppm	.75ppm	1.0ppm
DCB	.05ppm	.10ppm	.25ppm	.50ppm	.75ppm	1.0ppm
TCMX	.05ppm	.10ppm	.25ppm	.50ppm	.75ppm	1.0ppm

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For non-concentrated Soil and RV analyses:

Compound	Std1 µg/mL	Std2 µg/mL	Std3* µg/mL	Std4 µg/mL	Std5 µg/mL	Std6 µg/mL
Amount of Intermediate added (uL)	40uL	80uL	100uL	200uL	400uL	1000uL
Final Volume (mL)	1mL	1mL	1mL	1mL	1mL	1mL
Analyte Concentrations						
1016	2ppb	4ppb	5ppb	10ppb	20ppb	50ppb
1260	2ppb	4ppb	5ppb	10ppb	20ppb	50ppb
DCB	2ppb	4ppb	5ppb	10ppb	20ppb	50ppb
TCMX	2ppb	4ppb	5ppb	10ppb	20ppb	50ppb

- Levels also used for ICV/CCV.

NOTE: A standard containing a mixture of Aroclor 1016 and Aroclor 1260 includes many of the peaks represented in the other five Aroclor mixtures. As a result, a multi-point initial calibration employing a mixture of Aroclors 1016 and 1260 at five concentrations is sufficient to demonstrate the linearity of the detector response without the necessity of performing initial calibrations for each of the nine Aroclors, but six points are routinely run for calibration. In addition, the 1016/1260 mixture is used as a standard to demonstrate that a sample does not contain peaks that represent any one of the Aroclors. This standard is used to determine the concentrations of either Aroclor 1016 or Aroclor 1260, if they are present in a sample. A 0.50ppm or 5ppb for non-concentrated soil and RV, single point calibration is used for all remaining Aroclors other than 1016/1260. These are analyzed following each new initial calibration curve.

METHOD NOTE: For Method 608.3, one of the calibration standards should be at a concentration at or below the method-defined minimum level (ML) specified in the table below, as specified by a regulatory/control authority, or in a permit:

Method 608.3 ML Values

Analyte	ML (ng/L)
PCB-1016	--
PCB-1221	--
PCB-1232	--
PCB-1242	95
PCB-1248	--
PCB-1254	--
PCB-1260	--
PCB-1268	--

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Alternatively, the laboratory may establish an ML for each analyte based on the concentration of the lowest calibration standard in a series of standards produced by the laboratory or obtained from a commercial vendor, again, provided that the ML does not exceed the method-defined ML, and provided that the resulting calibration meets the acceptance criteria in based on the RSD, RSE, or R^2 .

A separate standard near the MDL may be analyzed as a check on sensitivity, but should not be included in the linearity assessment. The solvent for the standards must match the final solvent for the sample extracts (e.g., isooctane or hexane).

7.3.1 PCB presence and ID: Where PCBs are suspected and do not match the 1016/1260 standards, select the Aroclor that is suspected and run a single calibration point using the calibration standards listed in section 7.3.

7.4 Laboratory Control Sample, Matrix Spike Solution and Second Source Calibration Verification Solution:

Method	Matrix	Supplier/ Concentration*	Dilution	Spike Conc.	Spike Volume
608/8082 PCBs	Water	NSI - 1.0mg/mL each Aroclor 1016 & 1260	Dilute 1.0mL standard to 200mL in Acetone	5µg/mL	100uL of Spike Solution to 1L DI water (LCS) or 1L of sample (MS) or 10uL to 100mL for 3510RV
8082 PCBs	Conc. Soil/ Solid	NSI - 1.0mg/mL each Aroclor 1016 & 1260	Dilute 1.0mL standard to 200mL in Acetone	5µg/mL	1.0mL of Spike Solution to 30g Ottawa sand (LCS) or 30g of sample (MS) also Waste Dil.
608/8082 PCBs	Non- Conc. Soil/ Solid	NSI - 1.0mg/mL each Aroclor 1016 & 1260	Dilute 1.0mL standard to 200mL in Acetone	5µg/mL	100uL of Spike Solution to 1L DI water (LCS) or 1L of sample (MS) or 10uL to 100mL for non- concentrated soil

* see section 13.2

7.5 Hexane - pesticide grade - VWR EM-HX0298-1 or equivalent

7.6 Concentrated sulfuric acid (H_2SO_4) - VWR VW6840-3 reagent grade or equivalent.

7.7 Stock Internal Standard: 1-Bromo-2-nitrobenzene at 5000 mg/L (Ultra Cat# PPS-351) or equivalent. Dilute the purchased stock standard 0.10mL, or 2uL for non-concentrated soil and RV, to 10mL for the intermediate standard. Add 10uL of the intermediate standard to each 1mL standard, field sample, method blank, and QC (LCS/LCSD/MS/MSD) extract.

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- 7.8 Sodium sulfate, reagent grade, granular anhydrous, rinsed with methylene chloride, baked in a shallow tray at 450°C for 1 hour minimum, cooled in a desiccator, and stored in a pre-cleaned glass bottle with screw cap which prevents moisture from entering.
- 7.8.1 If, after heating, the sodium sulfate develops a noticeable grayish cast (due to the presence of carbon in the crystal matrix), that batch of reagent is not suitable for use and should be discarded. Extraction with methylene chloride (as opposed to simple rinsing) and baking at a lower temperature may produce sodium sulfate suitable for use.
- 7.9 Method 608.3 Standard Requirements
- 7.9.1 Quality Control (QC) Check Sample Concentrate—Prepare one or more mid-level standard mixtures (concentrates) in acetone (or other water miscible solvent). The concentrate is used as the spiking solution with which to prepare the Demonstration of Capabilities (DOC) samples, the Laboratory Control Sample (LCS), and Matrix Spike (MS) and Matrix Spike Duplicate (MSD) samples. If prepared by the laboratory (as opposed the purchasing it from a commercial supplier), the concentrate must be prepared independently from the standards used for calibration, but may be prepared from the same source as the second source standard used for calibration verification.
- 7.9.2 Calibration Verification Standards— In order to verify the results of the initial calibration standards, prepare one or more mid-level standard mixtures in isooctane or hexane, using standards obtained from a second source (different manufacturer or different certified lot from the calibration standards). These standards will be analyzed to verify the accuracy of the calibration. As with the QC sample concentrate, multiple solutions may be required to address coelutions among all of the analytes.
- 7.9.3 Internal standard solution—If the internal standard calibration technique is to be used, prepare Pentachloronitrobenzene (PCNB) at a concentration of 10 mg/mL in ethyl acetate. Alternative and multiple internal standards (e.g., tetrachloro-m-xylene, 4,4'-dibromobiphenyl, and/or decachlorobiphenyl) may be used provided that the laboratory performs all QC tests and meets all QC acceptance criteria with the alternative or additional internal standard(s) as an integral part of this method.
- 7.9.4 Surrogate solution—Prepare a solution containing one or more surrogates at a concentration of 2mg/mL in acetone. Potential surrogates include: dibutyl chlorendate (DBC), tetrachloro-m-xylene (TCMX), 4,4'-dibromobiphenyl, or decachlorobiphenyl. Alternative surrogates and concentrations may be used, provided the laboratory performs all QC tests and meets all QC acceptance criteria with the alternative surrogate(s) as an integral part of this method. If the internal standard calibration technique is used, do not use the internal standard as a surrogate.

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7.9.5 DDT and endrin decomposition (breakdown) solution—Prepare a solution containing endrin at a concentration of 50ng/mL and 4,4'-DDT at a concentration of 100ng/mL, in isooctane or hexane. A 1-mL injection of this standard will contain 50 picograms (pg) of endrin and 100 pg of DDT. The concentration of the solution may be adjusted by the laboratory to accommodate other injection volumes such that the same masses of the two analytes are introduced into the instrument.

8.0 PROCEDURE

8.1 Sample extraction

8.1.1 In general, water samples are extracted at a neutral pH with methylene chloride using a separatory funnel (ESC SOPs #330702 or #330702B). Solid samples are extracted with methylene chloride by microwave (ESC SOP #330707). Oil samples are extracted according to EPA method 3580A (ESC SOP #330754).

8.1.2 Reference materials, field-contaminated samples, or spiked samples are used to verify the applicability of the selected extraction technique to each new sample type. Such samples are spiked with the compounds of interest in order to determine the percent recovery and the limit of detection for that sample type. When other materials are not available and spiked samples are used, they are spiked with the analytes (Aroclors) of interest. When the presence of specific Aroclors is not anticipated, the Aroclor 1016/1260 mixture is an appropriate choice for spiking.

8.2 Extract cleanup: For information on specific cleanup procedures, see SOP #330741, *Sulfur Cleanup*, SOP #330740, *Acid Cleanup*, and SOP #330739, *Silica Gel Cleanup*.

8.3 Current conditions can be found in CyberLab for the GC instrument.

8.4 Initial Calibration: Prepare and inject, minimally, a 5-point calibration standard curve for EPA 8082, 8082A and SM 6431B or a 3-point calibration curve for EPA Method 608 (PCBs). The lowest level of the calibration curve must be at or below the RL. The lowest standard also serves as the MRL verification standard and must be re-analyzed or re-processed, using the new calibration curve, following each new initial calibration, to meet regulatory requirements. The MRL must be processed using the same calibration curve as is being utilized for client samples and must meet the requirements found in section 10.13. The same GC operating conditions used for the initial calibration must also be used for field sample analyses and QC samples.

8.4.1 When PCBs are quantitatively determined as Aroclors, the initial calibration consists of two parts.

8.4.1.1 A standard containing a mixture of Aroclor 1016 and 1260 includes many of the peaks represented in the other five Aroclor mixtures. Thus, such a standard is used to demonstrate the linearity of the detector. In addition, such a mixture is used to demonstrate that a sample does not contain peaks that are represented in any one of the Aroclors. This standard is

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also used to determine the concentrations of either Aroclor 1016 or 1260 are present in a sample. Therefore, an initial five-point calibration is performed using the mixture of Aroclors 1016 and 1260 and the response (RF) or calibration factor (CF) for each concentration level is calculated. See section 9.0 for calculations.

8.4.1.2 **Dual Column Confirmation:** Calibration criteria must be met on both columns for positive confirmation of target analytes.

8.4.1.3 Standards of the other five Aroclors are necessary for pattern recognition. These standards are also used to determine a single-point calibration factor for each Aroclor, assuming that the Aroclor 1016/1260 mixture has been used to describe the detector response. The standards for these seven Aroclors must be analyzed prior to the analysis of any samples and can be analyzed before or after the analysis of the 1016/1260 calibration standards.

8.4.1.4 Where only a few Aroclors are of interest for a specific project, the analyst can employ a 5-point initial calibration for Method 8082A (3 points for Method 608) of each of the Aroclors of interest and not use the 1016/1260 mixture or the pattern recognition standards.

STATE NOTE: For Arizona compliance samples, a full calibration curve for Aroclor 1016/1260 is analyzed while all other multi-peak components, including all other Aroclors, toxaphene, and chlordane, are injected at the reporting limit. If any of these compounds are detected in the sample, a five-point calibration of the detected Aroclor is performed with the lowest standard at or below the RL. The samples require dilution if high concentrations of these compounds are present. The area of 3-5 selected peaks is compared to the same peaks in the sample for the determination of concentration.

8.4.2 **Working Calibration Curve:** Inject the calibration standards to generate a working curve. HP Chemstation calculates the calibration factor or response factor for each compound in each standard according to the equations found in section 9.0. . If multi-point calibration is performed for individual Aroclors, use the calibration factors determined from those standards to evaluate linearity.

8.4.3 Initial Calibration Verification (ICV)/Continuing Calibration Verification (CCV): On days that the instrument does not require full calibration, the initial calibration of the analytical system must be verified once for every 12 hour analytical sequence or 20 samples. For calibration verification, the mixture of 1016/1260 is used, unless one of the other 5 Aroclors is the target of interest and the calibration curve has been performed using that Aroclor. The calibration verification process does not require analysis of the other Aroclors that are used for pattern recognition.

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8.4.3.1 The linear calibration or response factors for the (ICV/CCV) is determined using the calculations found in section 9.0 and then the percent difference or drift from the initial calibration curve is determined using the calculations in section 9.0.

8.4.3.2 The ICV/CCV is routinely at the mid-level concentration of the calibration standard; however other concentrations may be used to better meet client or regulatory requirements.

8.4.4 Second Source Calibration Verification (SSCV): The initial calibration curve generated must be verified using a source that is different from the stock solutions used to prepare the calibration curve. This source can be a separate manufacturer or separate lot number from the same manufacturer, if available. Routinely, the second source verification is performed at the mid-range of the calibration curve, but the concentration may be altered to better reflect client/project needs. The calibration factor for the SSCV is calculated using the equation found in section 9.0 and the difference from the initial calibration curve is determined using the equation also found in that section.

8.4.5 Method 608.3 Requirements

Injection of calibration solutions— Inject a constant volume of each calibration solution into the GC column/detector pairs. An alternative volume may be used provided all requirements in this method are met. Beginning with the lowest level mixture and proceeding to the highest level mixture may limit the risk of carryover from one standard to the next, but other sequences may be used. An instrument blank should be analyzed after the highest standard to demonstrate that there is no carry-over within the system for this calibration range.

8.5 Confirmations and Qualitative Identification. The identification of PCBs as Aroclors is based on agreement between the retention times of peaks in the sample chromatogram with the retention time windows established through the analysis of standards of the target analytes. Analyst judgment and experience also weigh heavily in the positive identification of potential Aroclors. Tentative identification of an analyte occurs when a peak from a sample extract falls within the established retention time window for a specific target analyte. Each tentative identification must be confirmed using a second GC column of dissimilar stationary phase (as in the dual-column analysis), based on a clearly identifiable Aroclor pattern, or using another technique such as GC/MS.

8.5.1 The results of a single column/single injection analysis MUST be confirmed on a second, dissimilar GC column. In order to be used for confirmation, retention time windows must be established for the second GC column.

8.5.1.1 Method 608.3 Requirements

8.5.1.1.1 Report the lower result from the two columns for each analyte in each sample or QC standard at or above the ML to 3 significant figures.

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8.5.1.1.2 Results for each analyte in MS/MSD samples should be reported from the same GC column as used to report the results for that analyte in the unspiked sample. If the MS/MSD recoveries and RPDs calculated in this manner do not meet applicable acceptance criteria, the analyst may use the results from the other GC column to determine if the MS/MSD results meet the acceptance criteria. If such a situation occurs, the results for the sample should be recalculated using the same GC column data as used for the MS/MSD samples, and reported with appropriate annotations that alert the data user of the issue.

8.5.1.1.3 In general, if the %D of the two results is less than 50% (e.g., a factor of 2), then the pesticide is present. This %D is generous and allows for the pesticide that has the largest measurement error.

8.5.2 Known Contaminants - When samples are analyzed from a source known to contain specific Aroclors, the results from a single-column analysis are confirmed on the basis of a clearly recognizable Aroclor pattern. This approach cannot be used for samples from unknown or unfamiliar sources or for samples that appear to contain mixtures of Aroclors. In order to employ this approach, the analyst must document:

- The peaks that were evaluated when comparing the sample chromatogram and the Aroclor standard.
- The absence of major peaks representing any other Aroclor.
- The source-specific information indicating that Aroclors are anticipated in the sample (e.g., historical data, client knowledge, etc.).

8.5.3 Quantitation of PCBs as Aroclors. The quantitation of PCB as Aroclors is accomplished by comparison of the peak pattern in the sample chromatogram to that of the most similar peak pattern from the Aroclor standard(s). A choice must be made as to which Aroclor pattern is most similar to that of the extract and whether the pattern in the standard is truly representative of the PCBs in the sample.

STATE NOTE: Once the correct Aroclor has been identified for Arizona compliance samples, a full calibration curve is prepared and analyzed followed by the samples of interest along with the appropriate QC samples.

STATE NOTE: Arizona compliance samples require that one Aroclor (1016/1260) has a full calibration curve and all other multi-peak components (i.e., Aroclors, toxaphene, and chlordane) must be injected at the laboratory reporting limit.

8.5.3.1 Use the chromatograms from the individual Aroclor standards (not the 1016/1260 mixtures) to determine the pattern of peaks for Aroclors 1221, 1232, 1242, 1248, and 1254 (1262 & 1268 by request). The patterns for Aroclors 1016 and 1260 are evident from the mixed calibration

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standards; however, an individual 1016 standard may be injected to help determine slight differences between 1016 and 1242.

8.5.3.1.1 Once the pattern of the Aroclor present in the field samples has been identified, compare the responses of the 3-5 major peaks in the single-point calibration standard for the appropriate Aroclor with the peaks observed in the sample extract. The amount of Aroclor is calculated using the individual calibration factor for each of the characteristic peaks chosen and the calibration model (linear or non-linear) established from the multi-point calibration of the 1016/1260 mixture. A final analyte concentration is determined by calculating a concentration from each of the characteristic peaks and averaging those concentrations to determine the reportable concentration of that Aroclor in each field sample.

8.5.4 Three to five peaks are used for Aroclor identification and quantitation. Five peaks are preferred; however as few as three can be used where there is obvious interference. The peaks must be characteristic of the Aroclor in question. Choose peaks in the Aroclor standards that are at least 25% of the height of the largest Aroclor peak. For each Aroclor, the set of 3 to 5 peaks must include at least one peak that is unique to that Aroclor. Use 5 peaks for the Aroclor 1016/1260 mixture, none of which are found in both of these Aroclors.

8.5.5 When determining PCBs as Aroclors by the internal or external standard technique, calculate the response factor (RF) or calibration factor (CF) for each characteristic Aroclor peak in each of the initial calibration standards. Five sets of response/calibration factors will be generated for the Aroclor 1016/1260 mixture, each set consisting of the response/calibration factors for each of the peaks chosen for this mixture. The single standard for each of the other Aroclors will generate 5 response/calibration factors, one for each selected peak. See section 9.0 for the equations to calculate the response or calibration factors for the calibration curve and for the calculation of the concentration of Aroclors in field samples.

8.5.5.1 Peak height measurements are recommended over peak area only when overlapping peaks can cause errors in area integration.

8.5.5.2 If the peak response is less than 2.5 times the baseline noise level, the validity of the quantitative result may be questionable. The analyst can consult with the source of the sample to determine whether further concentration of the sample is warranted.

8.5.5.3 If compound identification or quantitation is precluded due to interference (e.g., broad, rounded peaks or ill-defined baselines are present) cleanup of the extract or replacement of the capillary column or detector is warranted. Re-analyze the sample on another instrument to determine if

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the problem results from analytical hardware or the sample matrix. Refer to the ESC procedures to be followed if extract cleanup is required.

8.6 Weathering of PCBs in the environment and changes resulting from waste treatment processes may alter the PCBs to the point that the pattern of a specific Aroclor is no longer recognizable. Samples containing more than one Aroclor present similar problems. Identification and determination of PCBs in these circumstances rely heavily on the experience and discretion of the analyst. Alternative quantitative procedures for said circumstances such as described in section 8.6.1, are not routinely employed by ESC analytical staff but available per method.

8.6.1 If results in terms of Aroclors are required, then the quantitation as Aroclors can be performed by measuring the total area of the PCB pattern and quantitating on the basis of the Aroclor standard that is most similar to the sample. Any peaks that are not identifiable as PCBs on the basis of retention times are subtracted from the total area. If quantitation is performed in this manner, the problems are fully described for the data user and the specific procedures employed by the analyst are thoroughly documented.

8.7 Acceptance criteria for all calibration standards and QC (Method Blank/internal standards/LCS/LCSD/MS/MSD) are contained in section 10.0. Corrective actions for outliers are contained in section 11.0.

9.0 DATA ANALYSIS AND CALCULATIONS

9.1 Internal Calibration Equations (Response Factors):

$$RF = \frac{[A_s][C_{is}]}{[A_{is}][C_s]}$$

where:

- A_s = Peak area (or height) of the analyte or surrogate.
- A_{is} = Peak area (or height) of the internal standard.
- C_s = Concentration of the analyte or surrogate, in $\mu\text{g/L}$.
- C_{is} = Concentration of the internal standard, in $\mu\text{g/L}$.

- Percent Relative Standard Deviation (%RSD)

$$\overline{RF} = \frac{\sum_{i=1}^n RF_i}{n} \quad SD = \sqrt{\frac{\sum_{i=1}^n (RF_i - \overline{RF})^2}{n-1}} \quad RSD = \frac{SD}{\overline{RF}} \times 100\%$$

where:

- RSD = Relative standard deviation.
- \overline{RF} = Mean of 5 initial RFs for a compound.
- SD = Standard deviation of average RFs for a compound.

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- Concentration of an analyte in an extract using RF (on column):

$$X_s = \frac{(Conc_{std})(Area_{Analyte})}{(Average RF_{analyte})(Area_{std})}$$

where:

X_s = Calculated raw concentration of analyte (in ppb)

- Quantitation Report Multiplier"

$$M_a = \frac{(V_t)(D)}{(V_s)} \quad \text{or} \quad M_s = \frac{(V_t)(D)}{(W_s)}$$

where:

M_a = Quantitation Report Multiplier for Aqueous Samples

M_s = Quantitation Report Multiplier for Solid Samples

V_t = Total volume of concentrated extract (in mL)

D = Dilution factor. If no dilution, $D=1$. Always dimensionless

V_s = Volume of aqueous sample extracted (in mL)

W_s = Weight sample extracted (in grams)

- Sample concentration by volume (ug/L) for aqueous samples:

$$\text{Concentration in } \frac{mg}{L} = (X_s)(M_a)$$

- Sample concentration by weight (ug/kg) for solid samples and non-aqueous liquids:

$$\text{Concentration in } \frac{mg}{kg} = \frac{(X_s)(M_s)}{(\%S)}$$

where:

$\%S$ = Percent solids expressed as a decimal

9.2 Percent Error (%Error)

$$\%Error = \frac{x_i - x'_i}{x_i} * 100$$

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where:

x'_i = Measured amount of analyte at the calibration level i , in mass or concentration units

x_i = True amount of analyte at calibration level i , in mass or concentration units

- 9.3 Relative Standard Error (%RSE) – As an alternative to using the average response factor when using Method 608.3, the quality of the calibration may be evaluated using the Relative Standard Error (RSE). The acceptance criterion for the RSE is the same as the acceptance criterion for Relative Standard Deviation (RSD), in the method. RSE is calculated as:

$$\%RSE = 100 \times \frac{\sum_{i=1}^n \left[\frac{x'_i - x_i}{x_i} \right]^2}{(n - p)}$$

where:

x'_i = Calculated concentration at level i

x_i = Actual concentration of the calibration level i

n = number of calibration points

p = Number of terms in the fitting equation (average = 1; linear = 2; quadratic = 3)

- 9.4 See the current Quality Assurance Manual for equations associated with common calculations.

10.0 QUALITY CONTROL AND METHOD PERFORMANCE

- 10.1 All analysts must meet the qualifications specified in SOP #030205, *Technical Training and Personnel Qualifications* before approval to perform this method. Analysts must complete an initial demonstration of proficiency before being approved to perform this method. Continuing proficiency must be demonstrated using proficiency testing, laboratory control sample analysis and/or MDL studies. Method performance is assessed per analyst. Updated method performance records are filed and stored in a central location within the department.

10.1.1 Method 608.3 Demonstration of Capability (DOC) Requirements

- 10.1.1.1 For the DOC, a QC check sample concentrate containing each analyte of interest is prepared in a water miscible solvent using the solution in Section 7.9.1.
- 10.1.1.2 Prepare four QC check samples by adding an appropriate volume of the concentrate and of the surrogate(s) to each of four 1-L aliquots of reagent water. Swirl or stir to mix.
- 10.1.1.3 Extract and analyze the well-mixed QC check samples.
- 10.1.1.4 Calculate the average percent recovery (\bar{X}) and the standard deviation (s) of the percent recovery for each analyte using the four results.



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- 10.1.1.5 For each analyte, compare s and \bar{X} with the following acceptance criteria for precision and recovery. For analytes that are not listed, QC acceptance criteria must be developed by the laboratory.

Analyte	Limit for s (% SD)	Range for \bar{X} (%)
PCB-1016	24	61 – 103
PCB-1221	50	44 – 150
PCB-1232	32	28 – 197
PCB-1242	26	50 – 139
PCB-1248	32	58 – 140
PCB-1254	34	44 – 130
PCB-1260	28	37 – 130

If s and \bar{X} for all analytes of interest meet the acceptance criteria, system performance is acceptable and analysis of blanks and samples can begin. If any individual s exceeds the precision limit or any individual \bar{X} falls outside the range for recovery, system performance is unacceptable for that analyte.

- 10.1.1.6 When one or more of the analytes tested fail at least one of the acceptance criteria, repeat the test for only the analytes that failed. If results for these analytes pass, system performance is acceptable and analysis of samples and blanks may proceed. If one or more of the analytes again fail, system performance is unacceptable for the analytes that failed the acceptance criteria. Correct the problem and repeat the test.

- 10.2 Use the designated Run log to record batch order and standards/reagents used during analysis. See SOP #030201, *Data Handling and Reporting*.

- 10.3 Batches:

Batches are defined as sets of 1 - 20 samples. Batch analysis must include the following: 1 method blank, 1 Initial Calibration Verification (ICV), 1 Laboratory Control Sample/Laboratory Control Sample Duplicate pair (LCS/LCSD), 1 Matrix Spike/Spike Duplicate (MS/MSD) pair, Exceptions are made for waste dilution samples where the minimum batch QC must include a method blank. An LCS/LCSD pair may be extracted but is not required per waste dilution SOP. All batch information must be maintained in the preparation documentation assigned to the department.

- 10.4 Initial Calibration – If the percent relative standard deviation (% RSD) of the calibration factors for each analyte is <20% for EPA 8082 and 8082A and <10% for EPA method 608, the average calibration factor can be used for quantitation. If the %RSD exceeds the method defined acceptance criteria, a calibration curve using linear regression can be employed. The linear regression calibration curve must have a correlation factor of 0.990

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(USACE requires 0.995) or greater using equal or inverse weighting. The origin may not be used as a point in the calibration curve and the curve must not be forced through zero. The method blank is also not included as a point in the calibration curve for this method.

10.4.1 Method 608.3 Requirements

10.4.1.1 Internal Standard Calibration – If the RSD is less than 15%, linearity through the origin can be assumed and the average RF can be used for calculations. Alternatively, the results can be used to prepare a calibration curve of response ratios, A_s/A_{is} , vs. concentration ratios, C_s/C_{is} , for the analyte. A minimum of six concentration levels is required for a nonlinear (e.g., quadratic) regression. If used, the regression must be weighted inversely proportional to concentration, and the coefficient of determination of the weighted regression must be greater than 0.920. Alternatively, the relative standard error (Reference 10) may be used as an acceptance criterion. As with the RSD, the RSE must be less than 15%. If an RSE less than 15% cannot be achieved for a quadratic regression, system performance is unacceptable and the system must be adjusted and re-calibrated.

10.5 Method Blank – A method blank must be extracted and analyzed with each set of samples. The method blank must be carried through the same procedure as the samples and must not contain target analytes above the method detection limit.

10.6 Initial Calibration Verification (ICV)/Continuing Calibration Verification (CCV) – On days when a full calibration is not needed, an ICV must be analyzed once per 12hr analytical sequence prior to the analysis of any QC or field samples.

- The CF/RF must be within 15% of the initial calibration.

10.6.1 For Aroclor analyses, the routine CCV standard is a mixture of Aroclor 1016 and Aroclor 1260. The calibration verification process does not *require* analysis of the other Aroclor standards used for pattern recognition; however, if one of the other Aroclors is the analyte of interest and the component used for the initial calibration, the CCV will be a mid-level standard of the Aroclor of interest.

10.6.2 Method 608.3 Requirements

10.6.2.1 The working calibration curve, CF, or RF must be verified immediately after calibration and at the beginning and end of each 24-hour shift by the analysis of a midlevel calibration standard. The calibration verification standard(s) must be obtained from a second manufacturer or a manufacturer's batch prepared independently from the batch used for calibration. Alternatively, calibration verification may be performed after a set number of injections (e.g., every 20 injections), to include injection of extracts of field samples, QC samples, instrument blanks, etc. (i.e., it is based on the number of injections performed, not sample extracts). The time for the injections may not exceed 24 hours.

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NOTE: The 24-hour shift begins after analysis of the combined QC standard (calibration verification) and ends 24 hours later. The ending calibration verification standard is run immediately after the last sample run during the 24-hour shift, so the beginning and ending calibration verifications are outside of the 24-hour shift. If calibration verification is based on the number of injections instead of time, then the ending verification standard for one group of injections may be used as the beginning verification for the next group of injections.

- 10.7 Second Source Calibration Verification (SSCV) – A second source calibration verification standard (SSCV) is analyzed after each calibration and must meet criteria of $\pm 20\%$ of the expected concentration for each analyte.
- 10.8 Laboratory Control Sample (LCS)/Laboratory Control Sample Duplicate (LCSD) – must be extracted with each batch of samples.

- The LCS/LCSD must be within the acceptance criteria listed in Section 10.8.1. Section 10.8.1 represents QC acceptance criteria calculated from historical ESC values for the method. The acceptance criteria are more stringent than those of methods 608.

10.8.1 Current QC Acceptance Criteria are available in the LIMS.

STATE NOTE: For all 608 samples analyzed from South Carolina, the LCS/LCSD RPD must be $<20\%$ and recoveries must be within and the following limits in a water matrix:

Parameter	Recovery Limits
Aroclor 1016	70 – 114%
Aroclor 1260	70 – 127%

STATE NOTE: For South Carolina, marginal exceedances do not apply. All outliers in QC require corrective action when possible and the data must be flagged when necessary.

10.8.2 Method 608.3 Requirements

- 10.8.2.1 Prepare the LCS by adding QC check sample concentrate to reagent water. Include all analytes of interest in the LCS. The volume of reagent water must be the same as the nominal volume used for the sample, the DOC, the blank, and the MS/MSD.
- 10.8.2.2 Analyze the LCS prior to analysis of samples in the extraction batch.
- 10.8.2.3 For each analyte, compare the percent recovery (P) with its corresponding QC acceptance criterion in the following table:

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Analyte	Range for P (%)
PCB-1016	50 - 140
PCB-1221	15 - 178
PCB-1232	10 - 215
PCB-1242	39 - 150
PCB-1248	38 - 158
PCB-1254	29 - 140
PCB-1260	8 - 140

For analytes of interest not listed in the table, use the QC acceptance criteria developed for the MS/MSD or limits based on laboratory control charts. If the recoveries for all analytes of interest fall within the designated ranges, analysis of blanks and field samples may proceed.

10.9 Matrix Spike (MS)/Matrix Spike Duplicate (MSD) – must be analyzed with each batch of samples.

- Method 608 states that matrix spikes must be done at a rate of 10%.
- The spike and spike duplicate must meet the criteria listed in Section 10.8.1. Section 10.8.1 represents QC acceptance criteria calculated from historical ESC values for the method.

STATE NOTE: For all samples analyzed from South Carolina, the MS/MSD recoveries must be within the most stringent limits comparing in-house derived recovery limits and those given in Table 3 of Method 608. The following are the current limits:

Parameter	Recovery Limits
Aroclor 1016	70 – 114%
Aroclor 1260	46 – 126%

10.9.1 Method 608.3 Requirements

- 10.9.1.1 The laboratory must, on an ongoing basis, spike at least 5% of the samples in duplicate from each discharge being monitored to assess accuracy (recovery and precision). If direction cannot be obtained from the data user, the laboratory must spike at least one sample in duplicate per extraction batch of up to 20 samples. Spiked sample results should be reported only to the data user whose sample was spiked, or as requested or required by a regulatory/control authority, or in a permit.

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10.9.1.2 If, as in compliance monitoring, the concentration of a specific analyte will be checked against a regulatory concentration limit, the concentration of the spike should be at that limit; otherwise, the concentration of the spike should be one to five times higher than the background concentration, at or near the midpoint of the calibration range, or at the concentration in the LCS whichever concentration would be larger. When no information is available, the midpoint of the calibration may be used.

10.9.1.3 Compare the percent recoveries (P1 and P2) and the RPD for each analyte in the MS/MSD aliquots with the corresponding QC acceptance criteria for recovery (P) and RPD in the following table:

Analyte	Range for P (%)	Maximum MS/MSD RPD (%)
PCB-1016	50 - 140	36
PCB-1221	15 - 178	48
PCB-1232	10 - 215	25
PCB-1242	39 - 150	29
PCB-1248	38 - 158	35
PCB-1254	29 - 140	45
PCB-1260	8 - 140	38

If any individual P falls outside the designated range for recovery in either aliquot, or the RPD limit is exceeded, the result for the analyte in the unspiked sample is suspect and may not be reported or used for permitting or regulatory compliance.

For analytes not listed in the table, QC acceptance criteria must be developed by the laboratory.

10.9.1.4 After analysis of a minimum of 20 MS/MSD samples for each target analyte and surrogate, and if the laboratory chooses to develop and apply optional in-house QC limits, the laboratory should calculate and apply the optional in-house QC limits for recovery and RPD of future MS/MSD samples. The in-house QC limits must be updated at least every two years and reestablished after any major change in the analytical instrumentation or process. At least 80% of the analytes tested in the MS/ MSD must have in-house QC acceptance criteria that are tighter than those in the table presented in Section 10.9.1.3 and the remaining analytes (those not included in the 80%) must meet the acceptance criteria in the table.

If an in-house QC limit for the RPD is greater than the limit in the table, then the limit in the table must be used. Similarly, if an in-house lower

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limit for recovery is below the lower limit in the table, then the lower limit in the table must be used, and if an in-house upper limit for recovery is above the upper limit in the table, then the upper limit in the table must be used.

- 10.10 Confirmation - Any sample that shows a detectable concentration of any compound above the method detection limit must be confirmed on a second column or by GC/MS, except as noted in section 8.5.2. The result from the primary column and the confirmation column must agree within 40% RPD and acceptable calibration criteria must be met on both columns.
- 10.11 Surrogate – Calculate the surrogate recovery on all samples, method blanks, and spikes. Determine if the recovery is within the QC Acceptance criteria in section 10.8.1.
- 10.12 Internal Standards (internal calibration model) – For Method 8082, the internal standard area counts must be monitored for all CCVs. ISTDs must recover within 50-150% of the average of the most recent calibration. For Method 608.3, the ISTD should be verified within 50-200% of the mid-level ICAL standard.

The internal standard responses and retention times in the check calibration standard must be evaluated immediately after or during data acquisition. If the retention time for any internal standard changes by more than 30 seconds from the last calibration verification, the chromatographic system must be inspected for malfunctions and corrections must be made, as required.

Internal standards must be monitored for each sample. ISTDs in samples must meet the 50-150% of the average of the most recent calibration. For Method 608.3, the ISTD should be verified within 50-200% of the daily verification standard.

- 10.13 MRL – The reporting limit verification when analyzed must recover within $\pm 50\%$ of the target concentration for the standard.

STATE NOTE: For all samples analyzed from Minnesota, the reporting limit must be verified at least monthly, with each new initial calibration, or when there has been significant change to the instrument (column replacement, cleaning source, etc.) whichever is more frequent. The reporting limit verification can be performed by either re-injecting the low standard or by re-processing the low standard that was analyzed in the calibration curve. The reporting limit verification (MRL) must recovery within $\pm 40\%$ of the expected concentration. If this criterion is not met, the MRL may be re-analyzed once, instrument maintenance can be performed, a higher concentration standard can be injected, or a new calibration curve must be generated. If a higher concentration standard is utilized, the reporting limit must be raised to the higher level verified.

- 10.14 Any sample analyte responses that are beyond the linear range of the calibration curve must be diluted and re-analyzed.

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- 10.15 Manual Integration – All manual integrations must comply with the requirements found in ESC SOP #030215, *Manual Integration Procedure*. Before and after integrations must be available for review by the secondary data reviewer.
- 10.16 Second Source Calibration Verification (SSCV) - A second source calibration verification standard (SSCV) is analyzed after each calibration and must meet criteria of $\pm 20\%$ of the expected concentration for each analyte for 8082. Method 608.3 utilizes CCV limits for evaluation.
- 10.17 For sample analyzed per the requirements of Method 8000D, the LLOQ (see Section 1.8.2) must be verified at least annually, and whenever significant changes are made to the preparation and/or analytical procedure, to demonstrate quantitation capability at lower analyte concentration levels
- 10.17.1 The LLOQ verification (to be performed after the initial calibration) is prepared by spiking a clean control material with the analyte(s) of interest at 0.5-2 times the LLOQ concentration level(s).
- 10.17.2 The LLOQ check is carried through the same preparation and analytical procedures as environmental samples and other QC samples.
- 10.17.3 It is recommended to analyze the LLOQ verification on every instrument where data is reported; however, at a minimum, the lab must rotate the verification among similar analytical instruments such that all are included within 3 years.
- 10.17.4 Recovery of target analytes in the LLOQ verification must be within established in-house limits or within other such project-specific acceptance limits to demonstrate acceptable method performance at the LLOQ. Until the laboratory has sufficient data to determine acceptance limits, the LCS criteria $\pm 20\%$ (i.e., lower limit minus 20% and upper limit plus 20%) may be used for the LLOQ acceptance criteria.
- 10.18 For corrective actions, see section 11.0.
- 11.0 DATA VALIDATION AND CORRECTIVE ACTION
- 11.1 All data must undergo a primary review by the analyst. The analyst must check the performance of the initial calibration, mid-point check standard, and continuing calibrations to ensure that they meet the criteria of the method. The analyst must review any sample that has quantifiable compounds and make sure that they have been confirmed. The analyst must also verify that reported results are derived from quantitation between the RL and the highest standard of the initial calibration curve. All calculations must be checked (any dilutions, %solids, etc.). Data must be checked for the presence or absence of appropriate flags. Comments must be noted when data is flagged.
- 11.2 All data must undergo a second analyst review. The analyst checking the data must check the performance of the initial calibration, mid-point check standard, and continuing calibrations to ensure that they meet the criteria of the method.

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- 11.2.1 The analyst must review any sample that has quantifiable compounds and make sure that they have been confirmed.
- 11.2.2 All calculations must be checked.
- 11.2.3 All surrogate recoveries must be checked to ensure that they are within QC acceptance criteria or that corrective action has occurred.
- 11.2.4 Method blanks must be free of all interfering peaks.
- 11.2.5 Quality control criteria must be checked for the LCS, LCSD, MS, and MSD.
- 11.2.6 Data must be checked to determine the need for appropriate flags. Comments are noted when results are flagged.
- 11.2.7 The reviewer must verify all reported results are derived from analytical results that are above the reporting limit and below the highest standard of the initial calibration curve.
- 11.2.8 Reported sample hits must include an overlay of the identified analyte with the sample for the second analyst review.
- 11.2.9 All manual integrations must be verified through checking the before/after shot of the sample and/or QC.
- 11.2.10 All multipliers/dilutions must be verified on the quant report and must agree with the information provided on the injection log.
- 11.2.11 Retention times of the samples must be compared to that of the calibration standard.
- 11.2.12 Verify any linear regression by reviewing the calibration curve printout.
- 11.2.13 See SOP #030201, *Data Handling and Reporting*.
- 11.3 Initial Calibration – Corrective actions for failures in the initial calibration curve include: instrument maintenance and re-preparing the calibration standards.

Method 8000D: To determine calibration function acceptability, refit the initial calibration data back to the calibration model and calculate %Error (see Section 9.1). Percent error between the calculated and expected amounts of an analyte must be $\leq 30\%$ for all standards. For some data uses, $\leq 50\%$ may be acceptable for the lowest calibration point.
- 11.4 SSCV – If the acceptance criterion is not met, a new calibration curve or new SSCV must be prepared and analyzed, depending on the source of the discrepancy. An SSCV must pass the acceptance criteria prior to the analysis of field samples. If SSCV show a high bias, samples with concentrations below the RL may be reported. Any sample that contains a target analyte at a value above the RL must be reanalyzed with acceptable bracketing CCVs.

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- 11.5 ICV/CCV – When the initial or continuing calibration verification is out of the acceptance criteria and analysis of a second consecutive (immediate) calibration verification fails to produce results within acceptance criteria, corrective actions shall be performed. The laboratory shall demonstrate acceptable performance after the final round of corrective action with two consecutive calibration verifications or a new initial instrument calibration shall be performed.
- 11.6 Method Blank – If the method blank shows any detectable amount greater than the RL, the laboratory performance is assumed to be out of control and the problem must be corrected. Corrective actions include: re-analysis once. If the failure persists, re-extract the entire batch of samples, if submitted sample volume permits, or, if acceptable to the client, the data may be qualified.

General guidelines for qualifying sample results with regard to method blank quality are as follows:

- If the method blank concentration is less than the MDL and sample results are greater than the RL, then no qualification is required.
- No qualification is necessary when an analyte is detected in the method blank but not in the associated samples.
- If the concentration in a sample is more than ten times the concentration in the method blank, then no qualification is required.
- If the method blank concentration is greater than the MDL but less than the RL and sample results are greater than the MDL, then qualify associated sample results to indicate that analyte was detected in the method blank.
- If the method blank concentration is greater than the RL, further corrective action and qualification is required. An analyst should consult their supervisor for further instruction.

Method 8000D: When samples that are extracted together are analyzed on separate instruments or in separate analytical shifts, the method blank associated with those samples (e.g., extracted with the samples) must be analyzed on at least one of those instruments. A solvent blank must be analyzed on all other instruments on which the set of samples was analyzed to demonstrate the instrument is not contributing contaminants to the samples. At least one method blank or instrument blank must be analyzed on every instrument after calibration standard(s) and prior to the analysis of any samples.

When sample extracts are subjected to cleanup procedures, the associated method blank must also be subjected to the same cleanup procedures.

Results of the method blank should be less than the LLOQ for the analyte or less than the level of acceptable blank contamination specified in the approved QAPP or other appropriate systematic planning document. Blanks are generally considered to be acceptable if target analyte concentrations are less than one-half the LLOQ or are less than project-specific requirements.

When new reagents or chemicals are received, the lab should monitor the blanks associated with samples for any signs of contamination. It is not necessary to test every

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new batch of reagents or chemicals prior to sample preparation if the source shows no prior problems. However, if reagents are changed during a preparation batch, separate blanks need to be prepared for each set of reagents.

- 11.7 **MS/MSD** – If the spike and spike duplicate do not meet the criteria listed in section 10.8.1, or current ESC quality control acceptance criteria, the sample must be flagged as possible matrix interference.
- 11.7.1 Spike failure that result in the use of a "J" flag followed by the appropriate number, which further explains the failure concerning high or low bias
- 11.7.2 **Method 8000D:** If, as in compliance monitoring, the concentration of a specific analyte in the sample is being checked against a regulatory concentration limit or action level, the spike should be at or below the limit, or 1 - 5 times the background concentration (if historical data are available), whichever concentration is higher. If historical data are not available, a background sample of the same matrix from the site may be submitted for matrix spiking purposes to ensure that high concentrations of target analytes and/or interferences will not prevent calculation of recoveries. If the background sample concentration is very low or non-detect, a spike of greater than 5 times the background concentration is still acceptable. To assess data precision with duplicate analyses, it is preferable to use a low concentration field sample to prepare a MS/MSD for organic analyses. This spiking procedure will be performed when project-specific instructions are received from the client.

If the concentration of a specific analyte in a sample is not being checked against a limit specific to that analyte, then the analyst may spike the matrix spike or MS/MSD sample(s) at the same concentration as the reference sample at 20 times the estimated LLOQ in the matrix of interest, or at a concentration near the middle of the calibration range. It is suggested that a background sample of the same matrix from the site be submitted as a sample for matrix spiking purposes. NOTE: Preparing the spiking solution from the same source as the calibration standards helps minimize additional variability due to differences between sources. Typically, spiking concentrations are near the middle of the calibration range.

To develop precision and bias data for the spiked compounds, the analyst has two choices: analyze the original sample, and an MS/MSD pair; or analyze the original sample, a duplicate sample, and one spiked sample. If samples are not expected to contain the target analytes of concern, then the laboratory may use a MS/MSD pair. If samples are expected to contain the target analytes of concern, then the laboratory may use one matrix spike and a duplicate analysis of an unspiked field sample as an alternative to the MS/MSD pair.

The laboratory should use 70 - 130% as interim acceptance criteria for recoveries of spiked analytes, until in-house LCS limits are developed. Where in-house limits have been developed for matrix spike percent recoveries, the LCS

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results should be similar to or tighter than those limits, as the LCS is prepared in a clean matrix.

- 11.8 LCS/LCSD - If the LCS/LCSD does not perform within the ranges listed in Attachment II, or current ESC quality control acceptance criteria, the laboratory performance is assumed to be out of control and the problem must be corrected. Corrective action can include re-analysis, if instrument malfunction is suspected, or re-preparation and re-analysis of the entire batch, if the failure is suspected as either extraction or sample related.
- 11.9 Confirmation - If the relative percent difference of the results exceeds 40% and one result is significantly higher (e.g., >40%), check the chromatograms to see if an obviously overlapping peak is causing an erroneously high result. If no overlapping peaks are noted, examine the baseline parameters established by the instrument data system (or operator) during peak integration. If re-integration is necessary, ESC manual integration procedures must be followed and documented by printing a before and after shot of the chromatograms. When confirmation is not within the 40% criteria (or 50% for Method 608.3), unless otherwise specified in an approved project plan, the higher result should be reported, as this is a conservative approach relative to protection of the environment, unless obvious additive contamination present does not represent target analytes based on the experience of the analyst.
- 11.10 Surrogates - If the recovery is not within the quality control acceptance criteria stated in section 10.8.1, confirm that there are no errors in the calculations, surrogate solutions and standards. Check the instrument performance. Examine the chromatograms for interfering peaks and integrated areas. Re-calculate the data and/or re-analyze the extract if any of the above checks reveal a problem. Re-extract and re-analyze the sample if none of the above is determined to be the problem.
- 11.10.1 If a field sample exhibits poor surrogate recovery due to obvious matrix interferences, then qualify the sample with "J1" high or "J2" low to show that the surrogate quality control acceptance criteria were not met. Samples with unacceptable recoveries are re-extracted if there is sufficient field sample volume remaining.
- 11.10.2 If low surrogate recoveries are found throughout the analytical batch, including the QC samples, then the run must be re-extracted and re-analyzed, if sufficient volume was submitted by the client.
- 11.11 Internal Standards - If any internal standard response is beyond the acceptable recovery in the ICV/CCV, corrective action is required. Corrective action can take to form of checking the original calculations to ensure accuracy, re-analysis of the CCV to verify initial results, instrument maintenance (i.e. column clipping or changing, inlet liner cleaning/replacement, etc.) or re-calibration.

If the retention time for any internal standard changes by more than 30 seconds from the last calibration verification, the chromatographic system must be inspected for malfunctions and corrections must be made, as required. When corrections are made,

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re-analysis of the CCV or a complete re-calibration is necessary, depending on the impact of the correction on the analytical system.

Internal standards in the field samples must be monitored. If ISTD recovery does not meet the acceptance criteria, correction action is required. Possible corrective actions include: re-analysis, if instrument malfunction is suspected, or re-preparation and re-analysis, if the failure is suspected as either extraction or sample related. If the sample has an obvious matrix interferent and the internal standard recovery is greater than 150%, the sample can be diluted (if acceptable reporting limits can be achieved) to minimize the interference or the sample must be re-extracted and re-analyzed. If interference is not obviously the problem with the ISTD recovery, the sample must be re-analyzed undiluted to confirm the original failure.

- 11.12 MRL – If the MRL does not meet the acceptance criteria, the RLV may be re-analyzed once, instrument maintenance can be performed, a higher concentration standard can be injected, or a new calibration curve must be generated. If a higher concentration standard is utilized, the reporting limit for the field samples must be elevated to the higher level verified. High bias of MRL does not impact samples with target analyte concentrations lower than the reporting limit.
- 11.13 Instrument maintenance is performed as needed to optimize instrument performance and improve chromatography. Commonly performed maintenance includes changing of the injection port liner and clipping the column at the injection port end to eliminate active sites. A new calibration curve must be analyzed following any major maintenance performed on the analytical system if most recent calibration does not confirm with method limits.
- 11.14 Data that does not meet acceptable QC criteria may be acceptable for use in certain circumstances.
- 11.14.1 If a method blank contains an amount of target analyte, but all samples are non-detected, the data may be reported with a "B3" flag. If a method blank contains an amount of target analyte, but the samples contain analyte at a level that is 10 times the level present in the method blanks, the data may be reported with a "B" flag.
- 11.14.2 If the MS/MSD fails acceptance criteria in an initial analysis and again upon re-analysis, the data is released with an appropriate qualifier as the failure is accepted as matrix related.
- 11.14.3 If a calibration verification standard is above the acceptable QC criteria and all samples being bracketed are below the reporting limit, the data is acceptable based on a high calibration bias with undetectable levels in the field samples. Any positive samples require re-analysis.
- 11.14.4 If the surrogate exhibits high recovery in the field samples and the target analytes in the field samples are below the reporting limit, the data may be released with a J1 qualifier indicating the high bias. If the QC samples (LCS, LCSD, MS, MSD) exhibit a high bias in the surrogate and the field samples are below the reporting limit for the target analyte, the data may be released with a J1 qualifier.

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11.14.5 If the target analyte spiked in the quality control samples (LCS, LCSD, MS, MSD) exhibits high recovery and the target analytes in the field samples are below the reporting limit, the data may be released with a J4 or L1 qualifier indicating the high bias.

11.14.6 If the target analyte spiked into the QC pair (LCS/LCSD, MS/MSD) exhibit acceptable recoveries, but high calculated RPD values for precision, and the target analytes in the field sample are flagged with a J3 for the precision beyond acceptable quality control limits.

11.14.7 Sample results can be qualified and possible bias is narrated per the ESC SOP #030201, *Data Handling and Reporting*.

11.14.7.1 For samples analyzed per the requirements of Method 8000D, reported concentrations of target analytes between the MDL and the LLOQ must be qualified as estimated.

12.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

12.1 The EPA requires that laboratory waste management practice to be conducted consistent with all applicable federal and state laws and regulations. Excess reagents, samples and method process wastes must be characterized and disposed of in an acceptable manner. See *ESC Waste Management Plan*.

12.2 See SOP #030302, *Environmental Sustainability & Pollution Prevention*.

13.0 METHOD MODIFICATIONS/CLARIFICATIONS

13.1 Modifications to this method are noted in the body of the text as notes. Compliance analyses performed in conjunction with specific state and/or method requirements must be performed as noted.

13.2 Adjustments to the concentrations of standards/spiking solutions, standards providers, and quality control are subject to change to better meet client/project/regulatory needs or to improve laboratory method performance.

13.3 Additional Aroclors (i.e. Aroclor 1262 and 1268) are quantitated using this procedure than those specifically listed in EPA Method 8082 and/or 8082A

13.4 The reduced volume of field sample used in this procedure is performed in accordance with section 7.1 of the published EPA 3510C method. The reduction in volume extracted along with either sufficient sensitivity of detection and/or large volume injection technique (>5uL injected) on the GC allows for acceptable detection limits in line with those obtained using a 1L extraction. Complete method validation is performed for this process prior to utilizing the reduced volume extraction. This validation is maintained by the Regulatory Affairs Department and is regularly verified using LCS/LCSD, MDL studies and DOCs.

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- 13.5 Due to the laboratory information management systems (LIMS) requirements and in conjunction with the EPA Methods Update Rule, Table II (5/18/12), a sample holding time has been set for 365 days from collection to extraction.
- 13.6 Method 608.3 allowed method modifications:
- 13.6.1 If the underlying chemistry and determinative technique in a modified method are essentially the same as an approved Part 136 method, then the modified method is an equivalent and acceptable alternative to the approved method provided the requirements of this section are met.
 - 13.6.2 Those who develop or use a modification to an approved (Part 136) method must document that the performance of the modified method, in the matrix to which the modified method will be applied, is equivalent to the performance of the approved method. If such a demonstration cannot be made and documented, then the modified method is not an acceptable alternative to the approved method.
 - 13.6.3 Supporting documentation must, if applicable, include the routine initial demonstration of capability and ongoing QC including determination of precision and accuracy, detection limits, and matrix spike recoveries.
 - 13.6.3.1 Initial demonstration of capability typically includes analysis of four replicates of a mid-level standard and a method detection limit study.
 - 13.6.3.2 Ongoing quality control typically includes method blanks, mid-level laboratory control samples, and matrix spikes (QC is as specified in the method).
 - 13.6.3.3 The method is considered equivalent if the quality control requirements in the reference method are achieved.
 - 13.6.3.3.1 Where the laboratory is using a vendor-supplied method, it is the QC criteria in the reference method, not the vendor's method, that must be met to show equivalency.
 - 13.6.3.3.2 Where a sample preparation step is required (i.e., digestion, distillation), QC tests are to be run using standards treated in the same way as the samples.
 - 13.6.3.4 The method user's Standard Operating Procedure (SOP) must clearly document the modifications made to the reference method.
 - 13.6.4 If the method user is uncertain whether a method modification is allowed, the Regional ATP Coordinator or Director should be contacted for approval prior to implementing the modification
 - 13.6.5 The method user should also complete necessary performance checks to verify that acceptable performance is achieved with the method modification prior to analyses of compliance samples.

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- 13.6.6 The modified method must meet or exceed performance of the approved method(s) for the analyte(s) of interest, as documented by meeting the initial and ongoing quality control requirements in the method.
- 13.6.7 The permittee must notify their permitting authority of the intent to use a modified method. Such notification should be of the form "Method xxx has been modified within the flexibility allowed in 40 CFR 136.6." The permittee may indicate the specific paragraph of § 136.6 allowing the method modification. Specific details of the modification need not be provided, but must be documented in the Standard Operating Procedure (SOP) and maintained by the analytical laboratory that performs the analysis.

14.0 REFERENCES

- 14.1 *Polychlorinated Biphenyls (PCBs) by Gas Chromatography*, SW-846 Method 8082, Revision 0, December 1996.
- 14.2 *Polychlorinated Biphenyls (PCBs) by Gas Chromatography*, SW-846 Method 8082A, Revision 1, February 2007.
- 14.3 *The Determination of Polychlorinated Biphenyls in Transformer Fluid and Waste Oils*, EPA 600/4-81-045, Sept. 1982.
- 14.4 *Determinative Chromatographic Separations*, SW-846 Method 8000B, Revision 2, December 1996.
- 14.5 *Determinative Chromatographic Separations*, SW-846 Method 8000C, Revision 3, March 2003.
- 14.6 *Determinative Chromatographic Separations*, SW846 Method 8000D, Revision 4, July 2014.
- 14.7 *Polychlorinated Biphenyls (PCBs) by Liquid-Liquid Extraction Gas Chromatographic Method*, SM 6431B.
- 14.8 *Organochlorine Pesticides and PCBs*, EPA Method 608, 40CFR Part 136, Appendix A
- 14.9 *Organochlorine Pesticides and PCBs by GC/HSD*, EPA Method 608.3, Federal Register, Volume 82, Number 165, August 28, 2017.
- 14.10 EPA Method 608 ATP 3M0222, Federal Register, Volume 60, Number 148, August 2, 1995.
- 14.11 40 Code of Federal Regulations §136.6(b)(4)(j).

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Attachment I: Revision History

Current Version:

Version	Date	Description of Revisions
17	3/23/2018	Technical and quality review and update. Added Method 608.3 criteria. Revised Title and Sections 1.0, 1.1, 2.5, 7.3, 8.2, 8.4.3, 9.4, 10.8.1, 11.9, 11.14.7.1, 13.1, and 13.4. Added Sections 1.4.3, 4.7, 5.5, 7.8, 7.8.1, 7.9 and all subsections, 8.4.5, 8.5.4.4 and all subsections, 9.3, 10.1.1 and all subsections, 10.4.1, 10.4.1.1, 10.6.2 and all subsections, 10.8.2 and all subsections, 10.9.1 and all subsections, 10.12, 10.16, 13.6 and all subsections, 14.9, 14.10, 14.11, and re-numbered as necessary.

Superseded Versions:

This document supersedes the following:

Version	Date	Description of Revisions
0	2/11/00	Origination
1	8/21/00	
2	10/16/01	
3	7/9/03	
4	12/17/04	
5	2/23/09	Technical and Quality Review and update. Included state notes, included criteria for dual column analysis, clarified ICV/CCV use and criteria, included correlation coefficient and linear regression calculations, revised sections 12.0 & 13.0.
6	3/25/11	Technical and Quality Review and update. Revised sections 1.1, 2.0, 4.3, 4.4, 6.1, 7.1, 7.3, 7.4, 7.7, 8.1, 8.3, 8.4, 8.5.3.1, 8.5.5, 9.0, 10.0, 11.0, and 12.1; Added state notes in sections 1.0, 4.5, and 13.2
7	9/21/11	Technical and Quality Review and update. Revised sections 2.2, 8.5.1, 9.6, 10.10, 11.10, 11.11, and 11.14; Added state notes in sections 1.0, 8.0, 11.6, and 11.14; Added sections 8.7, 9.7, 10.16, 11.14.4 through 11.14.6 and 13.3
8	2/17/12	Technical and Quality Review and update. Revised sections 2.1, 2.2, 6.1, 7.3, 7.7, 8.1.1, and 10.8.1; Added state notes in sections 1.0 and 10.8.1; Added sections 1.4.1, 2.29, 2.30, and 13.4.
9	4/24/12	Technical and Quality Review and update. Revised sections 7.3, 7.4, 7.7, 8.7, 10.8.1, and 11.5; Added sections 4.6 and 11.14.7. Ohio VAP approved 4/24/12.
10	1/6/14	Technical and Quality Review and update. Revised sections 2.1, 2.3, 2.28, 4.3, 4.4, 7.1, 7.3, 7.4, 7.7, 8.1.1, 8.3, 8.4, 8.4.3, 10.3, 10.4, 10.6, 10.8.1, 10.12, 10.13, 11.4, 11.9, 11.12, 11.13, 11.14.2, and 11.14.5; Added state notes in sections 1.0 and 11.14.5 and added sections 2.31, 13.5 and 14.8.

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Version	Date	Description of Revisions
11	11/3/2015	Technical and quality review and update. Header and signature bar reformatting. Revised Sections 1.4.1, 10.5, 10.8.1, 11.6, and 13.4. Added Attachment II.
12	8/16/2016	Technical and quality review and update. Revised header and Sections 1.0, 1.4.1, 2.8, 4.6, 7.1, 8.3, 8.4.2, 8.4.3, 8.6, 10.3, 10.6, 11.5, 11.13, 11.14.7, 12.2, and Attachment II Table 2. Deleted Sections 2.9 through 2.31, 8.3.1, and 8.3.2, 9.1 through 9.10.
13	10/24/2016	Technical and quality review and update to comply with SC DHEC SOP requirements (see correspondence dated 6/24/16). Revised Sections 1.0, 11.3, 11.6, 14.1, 14.4, 14.5, 14.7, and 14.8. Added Sections 1.4.2, 2.8, 9.1, 10.16 and all subsections, 11.7.2, 11.14.7.1, 14.2, and 14.6.
14	6/19/2017	Technical and quality review and update. Revised Sections 1.4, 3.1, 9.1, 9.2, 9.3, 10.8, and 10.9.
15	7/10/2017	Technical and quality review and update. Revised Sections 2.9, 2.10, 9.1, 13.4
16	11/29/2017	Update in response to A2LA audit finding CAR2872. Revised Attachment II Table 5.

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Attachment II: DoD Requirements

1.0 Equipment/Instrument Maintenance

Instrument maintenance must be performed routinely to optimize instrument performance and improve chromatography. Commonly performed maintenance includes cleaning/repairing detector, column clipping/replacement, injector port cleaning/changing liner, etc. A new calibration curve must be analyzed following any major maintenance performed on the analytical system.

2.0 Computer Hardware and Software

Software name and version: HP Chemstation G1701DA or equivalent

3.0 Troubleshooting

Table 1. GC Troubleshooting Guide		
Problem	Cause	Treatment
No Peaks	Syringe clogged	Clean or replace syringe
	Detector/Software/Computer failure	Check cables. Restart computer.
	Column Leaks	Use new ferrules.
	Broken Column	If at ends, clip column. If in the middle or multiple sites, replace column.
Peaks too Small	Split too high	Reduce split
	Column connection leaks	Check column installation. Search for leaks. Replace ferrules.
	Injector temperature too low	Check temperature program. Increase injector temperature.
	Dirty ECD	Clean ECD.
Retention Times Change	Gas flow too low or too high	Replace septum. Check gas regulator.
	Oven temperature unstable	Check temperature program. Check temperature with external thermometer.
	Column blocked	Compare flow at column entrance to outlet. Replace column.
Constantly Rising Baseline	Leak at column entrance or injection septum.	Check column installation; search for leaks; replace ferrules.
	Injector contaminated.	Make a run at lower injector temperature; if the baseline improves, replace liner, use low bleed or high temperature septa.
	Column contaminated.	Cut two turns from column entrance; rinse column with solvent (only chemically bonded phases); otherwise replace column or use guard column.
	Detector contaminated.	Clean detector.
	Increase of temperature too fast.	Decrease temperature gradient and end temperature.

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Table 1. GC Troubleshooting Guide

Problem	Cause	Treatment
	Poor gas quality.	Use gas grades recommended for GC; for longer supply lines from gas source to GC use gas purification cartridges directly connected to the GC.
Increasing Baseline at High Temperatures	Decomposition of the stationary phase.	Check for leaks; matrix check for compatibility with the column.
	Column contaminated.	Cut two turns from column entrance; rinse column with solvent (only chemically bonded phases); otherwise replace column or use guard column.
	Increase of temperature too fast / end temperature too high.	Decrease temperature gradient and end temperature.
	Column not properly conditioned.	Condition column according to manufacturers' instructions (while column is not connected to the detector).
	Detector contaminated	Clean detector according to manufacturers' instructions.
	Poor gas quality.	Use gas grades recommended for GC; for longer supply lines from gas source to GC use gas purification cartridges directly connected to the GC.
Plateaus at Certain Temperatures	Steps in temperature program too drastic.	Avoid very short and strong heating periods.
Fronting	Column overload.	Decrease injection volume; dilute sample.
	Sample vaporizes too slowly, not evenly or condenses.	Increase injector temperature (consider max. temperature limits of the column).
	Analytes coelute.	Change temperature program or use column with different selectivity.
	Sample decomposes.	Check temperature program, oven temperature (external thermometer); if analytes are not temperature-stable, reduce injector temperature; replace liner.
	Column absorbs or decomposes analytes.	Check capillary ends; check intact deactivation using the test mixture; for poor results shorten both column ends by about 10 cm; or replace column; if column test does not show any defects: a) use a column with thicker film b) use phase with better deactivation c) use column with special selectivity.

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Table 1. GC Troubleshooting Guide

Problem	Cause	Treatment
Tailing	Sample vaporizes too slowly, not evenly or condenses.	Increase injector temperature (consider max. temperature limits of the column).
	System leaks.	Check column installation; search for leaks; replace ferrules.
	Analytes coelute.	Change temperature program or use column with different selectivity.
	Sample decomposes.	Check temperature program, oven temperature (external thermometer); if analytes are not temperature-stable, reduce injector temperature; replace liner by a deactivated one.
	Column absorbs or decomposes analytes.	Check capillary ends; check intact deactivation using the test mixture; for poor results shorten both column ends by about 10 cm; or replace column; if column test does not show any defects: a) use a column with thicker film b) use phase with better deactivation c) use column with special selectivity.
	Split rate too low.	Increase split rate.
	Column overload.	Decrease injection volume; dilute sample.
Split Peaks	Solvent and column not compatible.	Change solvent or use guard column.
	Solvent mixtures with large differences in boiling point and polarity.	Use just one solvent.
	Sample decomposes.	Check temperature program, oven temperature (external thermometer); if analytes are not temperature-stable, reduce injector temperature; replace liner by a deactivated one.
	Analytes coelute.	Modify temperature program or use longer column; possibly change column polarity.
	Detector overload.	Inject less; control make-up flow.

4.0 Other Requirements

- 4.1 All hardcopy laboratory notebooks must be reviewed by the Supervisor, or their designee, on a monthly basis.
- 4.2 If not self-explanatory (e.g., a typo or transposed number), corrections to technical and quality records shall also include a justification for the change.

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- 4.3 A person performing a manual integration must sign and date each manually integrated chromatogram and record the rationale for performing manual integration. Electronic signatures are acceptable.
- 4.4 The results of calibration and verification of support equipment must be within the specifications required of the application for which this equipment is used or the equipment must be removed from service until repaired. Calibration and verification records, including those of established correction factors, must be maintained. In the absence of method-specific requirements, the minimum requirements are as follows:

Table 2. Support Equipment Checks		
Performance Check	Frequency	Acceptance Criteria
Balance calibration check [Using two standard weights that bracket the expected mass]	Daily prior to use	Top-loading balance: $\pm 2\%$ or $\pm 0.02\text{g}$, whichever is greater Analytical balance: $\pm 0.1\%$ or $\pm 0.5\text{mg}$, whichever is greater
Verification of standard mass [Using weights traceable to the International System of Units (SI) through a NMI]	Every 5 years	Certificate of Calibration from ISO/IEC 17025 accredited calibration laboratory
Monitoring of refrigerator/freezer temperatures	Daily (i.e. 7 days per week) [use MIN/MAX thermometers or data loggers equipped with notification of out of control event capabilities if personnel not available to record daily]	Refrigerators: 0°C to 6°C Freezers: $\leq -10^{\circ}\text{C}$
Thermometer verification check [Using a thermometer traceable to the SI through an NMI] [Performed at two temperatures that bracket the target temperature(s). Assume linearity between the two bracketing temperatures.] [If only a single temperature is used, at the temperature of use]	Liquid in glass: Before first use and annually Electronic: Before first use and quarterly	Apply correction factors or replace thermometer
Volumetric labware	Class B: By lot before first use Class A and B: Upon evidence of deterioration	Bias: Mean within $\pm 2\%$ of nominal volume Precision: $\text{RSD} \leq 1\%$ of nominal volume (based on 10 replicate measurements)
Non-volumetric labware [Applicable only when used for measuring initial sample volume and final extract/ digestates volume]	By lot before first use or upon evidence of deterioration	Bias: Mean within $\pm 3\%$ of nominal volume Precision: $\text{RSD} \leq 3\%$ of nominal volume (based on 10 replicate measurements)

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Table 2. Support Equipment Checks

Performance Check	Frequency	Acceptance Criteria
Mechanical volumetric pipette	Quarterly	Bias: Mean within $\pm 2\%$ of nominal volume Precision: RSD $\leq 1\%$ of nominal volume (based on minimum of 3 replicate measurements) [Note: for variable volume pipettes, the nominal volume is the volume of use]
Glass microliter syringe	Upon receipt and upon evidence of deterioration	General Certificate of Bias & Precision upon receipt Replace if deterioration is evident
Drying oven temperature check	Daily prior to and after use	Within $\pm 5\%$ of set temperature
Water purification system	Daily prior to use	See method blank criteria given in Section 4.20 of this addendum

- 4.5 The expiration date of the prepared standard shall not exceed the expiration date of the primary standard. All containers must bear a preparation date.
- 4.6 To avoid preparing non-representative samples, the laboratory shall not “target” within a relatively small mass range (e.g., $1.00 \pm 0.01\text{g}$) because such targeting will produce non-representative subsamples if the sample has high heterogeneity. The laboratory shall not manipulate the sample material so the sample aliquot weighs exactly $1.00\text{g} \pm 0.01\text{g}$, as an example.
- 4.7 In the absence of project-specific requirements, the minimum standard data qualifiers to be used are:
- U Analyte was not detected and is reported as less than the LOD or as defined by the customer. The LOD has been adjusted for any dilution or concentration of the sample.
 - J The reported result is an estimated value (e.g., matrix interference was observed or the analyte was detected at a concentration outside the quantitation range).
 - B Blank contamination. The recorded result is associated with a contaminated blank.
 - N Non-target analyte. The analyte is a tentatively identified compound using mass spectrometry or any non-customer requested compounds that are tentatively identified.
 - Q One or more quality control criteria failed (e.g., LCS recovery, surrogate spike recovery, or CCV recovery).

Additional data qualifiers may be used, or different letters or symbols to denote the qualifiers listed above, as long as they are appropriately defined and their use is

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consistent with project-specific requirements (e.g., QSM 5.0, the contract, and project-planning documents).

- 4.8 If the time of the sample collection is not provided, assume the most conservative time of day. For the purpose of batch processing, the start and stop dates and times of the batch preparation shall be recorded.
- 4.9 Each preparation method listed on the scope of accreditation must have quarterly LOD/LOQ verifications. However, not all possible combinations of preparation and cleanup techniques are required to have LOD/LOQ verifications. If LOD/LOQ verifications are not performed on all combinations, the laboratory must base the LOD/LOQ verifications on the worst case basis (preparation method with all applicable cleanup steps).
- 4.10 After each MDL determination, the laboratory must establish the LOD by spiking a quality system matrix at a concentration of at least 2 times but no greater than four times the MDL. This spike concentration establishes the LOD and the concentration at which the LOD shall be verified. It is specific to each suite of analyte, matrix, and method (including sample preparation). The following requirements apply to the initial LOD establishment and to the LOD verifications:
- The apparent signal to noise (S/N) ratio at the LOD must be at least three and the results must meet all method requirements for analyte identification (e.g., ion abundance, second column confirmation, or pattern recognition). For data systems that do not provide a measure of noise, the signal produced by the verification sample must produce a result that is at least three standard deviations greater than the mean method blank concentration. This is initially estimated based on a minimum of four method blank analyses and later established with a minimum of 20 method blank results.
 - If the LOD verification fails, then the laboratory must repeat the MDL determination and LOD verification or perform and pass two consecutive LOD verifications at a higher spike concentration and set the LOD at the higher concentration.
 - The laboratory shall maintain documentation for all MDL determinations and LOD verifications.
 - The DL and LOD must be reported for all analyte-matrix-methods suites, unless it is not applicable to the test or specifically excluded by project requirements.
- 4.11 The LOD shall be verified quarterly. In situations where methods are setup and used on an infrequent basis, the laboratory may choose to perform LOD verifications on a one per batch basis. All verification data will be in compliance, reported, and available for review.
- 4.12 For DoD, at a minimum, the LOQ shall be verified quarterly. In situations where methods are setup and used on an infrequent basis, the laboratory may choose to perform LOQ verifications on a one per batch basis.
- 4.13 All initial instrument calibrations must be verified with a standard obtained from a second manufacturer prior to analyzing any samples. The use of a standard from a second lot obtained from the same manufacturer (independently prepared from different source

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materials) is acceptable for use as a second source standard. The concentration of the second source standard shall be at or near the midpoint of the calibration range. The acceptance criteria for the initial calibration verification must be at least as stringent as those for the continuing calibration verification.

- 4.14 Exclusion of calibration points without documented scientifically valid technical justification is not permitted.
- 4.15 The concentration of the CCV standard shall be greater than the low calibration standard and less than or equal to the midpoint of the calibration range.
- 4.16 All CCVs analyzed must be evaluated and reported. If a CCV fails, reanalysis or corrective actions must be taken.
- If a CCV fails, the laboratory can immediately analyze two additional consecutive CCVs (immediately is defined as starting a consecutive pair within one hour; no samples can be run between the failed CCV and the two additional CCVs). This approach allows for spurious failures of analytes to be reported without reanalysis of samples. Any corrective actions that change the dynamics of the system (e.g., clip column, clean injection port, run blanks) requires that all samples since the last acceptable CCV be reanalyzed.
 - Both of these CCVs must meet acceptance criteria in order for the samples to be reported without reanalysis.
 - If either of these two CCVs fail or if the laboratory cannot immediately analyze two CCVs, the associated samples cannot be reported and must be reanalyzed.
 - Corrective action(s) and recalibration must occur if the above scenario fails. All affected samples since the last acceptable CCV must be reanalyzed.
 - Flagging of data for a failed CCV is only appropriate when the affected samples cannot be reanalyzed. The laboratory must notify the client prior to reporting data associated with a failed CCV.
- 4.17 The results of all MS/MSDs must be evaluated using the same acceptance criteria used for the DoD LCS limits (see Addendum Tables 3 and 4) or project limits, if specified. If the specific analyte(s) are not available in the Addendum Tables 3 and 4, the laboratory shall use their LCS in-house limits (see the LIMS) as a means of evaluating MS/MSDs. The MS and MSD must be spiked with all reported analytes.
- 4.19 Surrogate spike results shall be compared with DoD LCS limits (see Addendum Tables 3 and 4) or acceptance criteria specified by the client. If these criteria are not available, the laboratory shall compare the results with its in-house statistically established LCS criteria (see the LIMS).
- 4.20 The method blank shall be considered to be contaminated if:
- The concentration of any target analyte (chemical of concern) in the blank exceeds 1/2 the LOQ and is greater than 1/10th the amount measured in any associated sample, or 1/10th the regulatory limit, whichever is greater;

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- The concentration of any common laboratory contaminant in the blank exceeds the LOQ;
 - If a method blank is contaminated as described above, then the laboratory shall reprocess affected samples in a subsequent preparation batch, except when sample results are below the LOD. If insufficient sample volume remains for reprocessing, the results shall be reported with appropriate data qualifiers.
- 4.21 Sporadic Marginal Exceedances are not allowed for target analytes (chemicals of concern as identified by a project) without project-specific approval. Target analytes are considered those few analytes that are critical for the success of a project (such as risk drivers) where sporadic marginal exceedances cannot be allowed. Laboratories should consult with clients whenever long lists of analytes are requested for analysis to determine if marginal exceedances will not be allowed.
- 4.22 DoD considers the same analyte exceeding the LCS control limit two (2) out of three (3) consecutive LCS to be indicative of non-random behavior, which requires corrective action and reanalysis of the LCS.

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Table 3. LCS Control Limits – Method 8082 Solid Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
12674-11-2	Aroclor 1016	6847	90.1	14.5	47	134
11097-69-1	Aroclor 1254	406	101.2	11.4	67	135
11096-82-5	Aroclor 1260	7975	96.6	14.4	53	140
877-09-8	Tetrachloro-m-xylene	2379	86.7	14.4	44	130

Table 4. LCS Control Limits – Method 8082 Water Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
12674-11-2	Aroclor 1016	3356	87.1	13.8	46	129
11097-69-1	Aroclor 1254	184	80.1	15.4	34	127
11096-82-5	Aroclor 1260	3538	89.4	14.8	45	134

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Table 5. Quality Control Requirements – Organic Analysis by Gas Chromatography

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Breakdown check (Endrin/DDT 8081 only)	Before sample analysis and at the beginning of each 12-hour shift.	Degradation of DDT and Endrin must each be $\leq 15\%$.	Correct problem, then repeat breakdown checks.	Flagging is not appropriate.	No samples shall be run until degradation of DDT and Endrin is each $\leq 15\%$.
Initial Calibration (ICAL) for all analytes (including surrogates)	At instrument set-up and after ICV or CCV failure, prior to sample analysis.	ICAL must meet one of the three options below: Option 1: RSD for each analyte $\leq 20\%$; Option 2: linear least squares regression for each analyte: $r^2 \geq 0.99$; Option 3: non-linear least squares regression (quadratic) for each analyte: $r^2 \geq 0.99$.	Correct problem then repeat ICAL.	Flagging is not appropriate.	Minimum 5 levels for linear and 6 levels for quadratic. Quantitation for multicomponent analytes such as chlordane, toxaphene, and Aroclors must be performed using a 5-point calibration. Results may not be quantitated using a single point. No samples shall be analyzed until ICAL has passed.
Retention Time window position establishment	Once per ICAL and at the beginning of the analytical sequence.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA	NA	Calculated for each analyte and surrogate.

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Table 5. Quality Control Requirements – Organic Analysis by Gas Chromatography

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Retention Time (RT) window width	At method set-up and after major maintenance (e.g., column change).	RT width is ± 3 times standard deviation for each analyte RT from the 72-hour study or 0.03 minutes, whichever is greater.	NA	NA	Calculated for each analyte and surrogate. Only applicable if internal standard calibration is not used.
Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All reported analytes within established RT windows. All reported analytes within $\pm 20\%$ of true value.	Correct problem, rerun ICV. If that fails, repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed until calibration has been verified with a second source.

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Table 5. Quality Control Requirements – Organic Analysis by Gas Chromatography

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Continuing Calibration Verification (CCV)	Before sample analysis, after every 10 field samples, and at the end of the analysis sequence with the exception of CCVs for Pesticides multi-component analytes (i.e., Toxaphene, Chlordane, and Aroclors other than 1016 and 1260), which are only required before sample analysis.	All reported analytes and surrogates within established RT windows. All reported analytes and surrogates within \pm 20% of true value.	<p>Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails or if two consecutive CCVs cannot be run, perform corrective action(s) and repeat CCV and all associated samples since the last successful CCV.</p> <p>Alternately, recalibrate if necessary; then reanalyze all associated samples since the last acceptable CCV.</p>	<p>If reanalysis cannot be performed, data must be qualified and explained in the case narrative.</p> <p>Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.</p>	<p>Results may not be reported without a valid CCV.</p> <p>Flagging is only appropriate in cases where the samples cannot be reanalyzed.</p>

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TITLE: POLYCHLORINATED BIPHENYLS (PCBS) BY GAS CHROMATOGRAPHY (SOIL, WATER & OIL) (EPA METHODS 608, 8082, & 8082A, SM 6431B)

Table 5. Quality Control Requirements – Organic Analysis by Gas Chromatography

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Internal Standards (IS)	If employed, every field sample, standard, and QC sample.	Retention time within ± 0.06 RRT UNITS from retention time of the midpoint standard in the ICAL; Internal standard signal (area or height) within -50% to +100% of ICAL midpoint standard. On days when ICAL is not performed, the daily initial CCV can be used.	Inspect GC for malfunctions and correct problem. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	If corrective action fails in field samples, data must be qualified and explained in the Case Narrative. Apply Q-flag to analytes associated with the non-compliant IS. Flagging is not appropriate for failed standards.	NA.
Method Blank (MB)	One per preparatory batch.	No analytes detected $>1/2$ LOQ or $> 1/10$ the amount measured in any sample or $1/10$ the regulatory limit, whichever is greater.	Correct problem. If required, reprep and reanalyze MB and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

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TITLE: POLYCHLORINATED BIPHENYLS (PCBS) BY GAS CHROMATOGRAPHY (SOIL, WATER & OIL) (EPA METHODS 608, 8082, & 8082A, SM 6431B)

Table 5. Quality Control Requirements – Organic Analysis by Gas Chromatography

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Laboratory Control Sample (LCS)	One per preparatory batch.	A laboratory must use Table 3 and 4 limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits (see the LIMS) if project limits are not specified.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per preparatory batch.	A laboratory must use Table 3 and 4 limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits (see the LIMS) if project limits are not specified.	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	For matrix evaluation only. If MS results are outside the limits, the data shall be evaluated to determine the source(s) of difference (i.e., matrix effect or analytical error).

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TITLE: POLYCHLORINATED BIPHENYLS (PCBS) BY GAS CHROMATOGRAPHY (SOIL, WATER & OIL) (EPA METHODS 608, 8082, & 8082A, SM 6431B)

Table 5. Quality Control Requirements – Organic Analysis by Gas Chromatography

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch.	A laboratory must use Table 3 and 4 limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits (see the LIMS) if project limits are not specified. $RPD \leq 30\%$ (between MS and MSD or sample and MD).	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	The data shall be evaluated to determine the source of difference. For Sample/MD: RPD criteria only apply to analytes whose concentration in the sample is greater than or equal to the LOQ.

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TITLE: POLYCHLORINATED BIPHENYLS (PCBS) BY GAS CHROMATOGRAPHY (SOIL, WATER & OIL) (EPA METHODS 608, 8082, & 8082A, SM 6431B)

Table 5. Quality Control Requirements – Organic Analysis by Gas Chromatography

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Surrogate Spike	All field and QC samples.	QC acceptance criteria specified by the project, if available; otherwise use Table 3 and 4 limits or in-house LCS limits (See the LIMS) if analyte(s) are not listed.	Correct problem, then reprep and reanalyze all failed samples for all surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary, but the client must be notified prior to reporting data, and the failures must be discussed in the Case Narrative.	Apply Q-flag to all associated analytes if acceptance criteria are not met and explain in the case narrative.	Alternative surrogates are recommended when there is obvious chromatographic interference.
Confirmation of positive results (second column)	All positive results must be confirmed (except for single column methods such as TPH by Method 8015 where confirmation is not an option or requirement).	Calibration and QC criteria for second column are the same as for initial or primary column analysis. Results between primary and secondary column RPD $\leq 40\%$.	NA	Apply J-flag if RPD $>40\%$. Discuss in the case narrative.	Use project-specific reporting requirements if available; otherwise, use method requirements if available; otherwise report the result from the primary column.

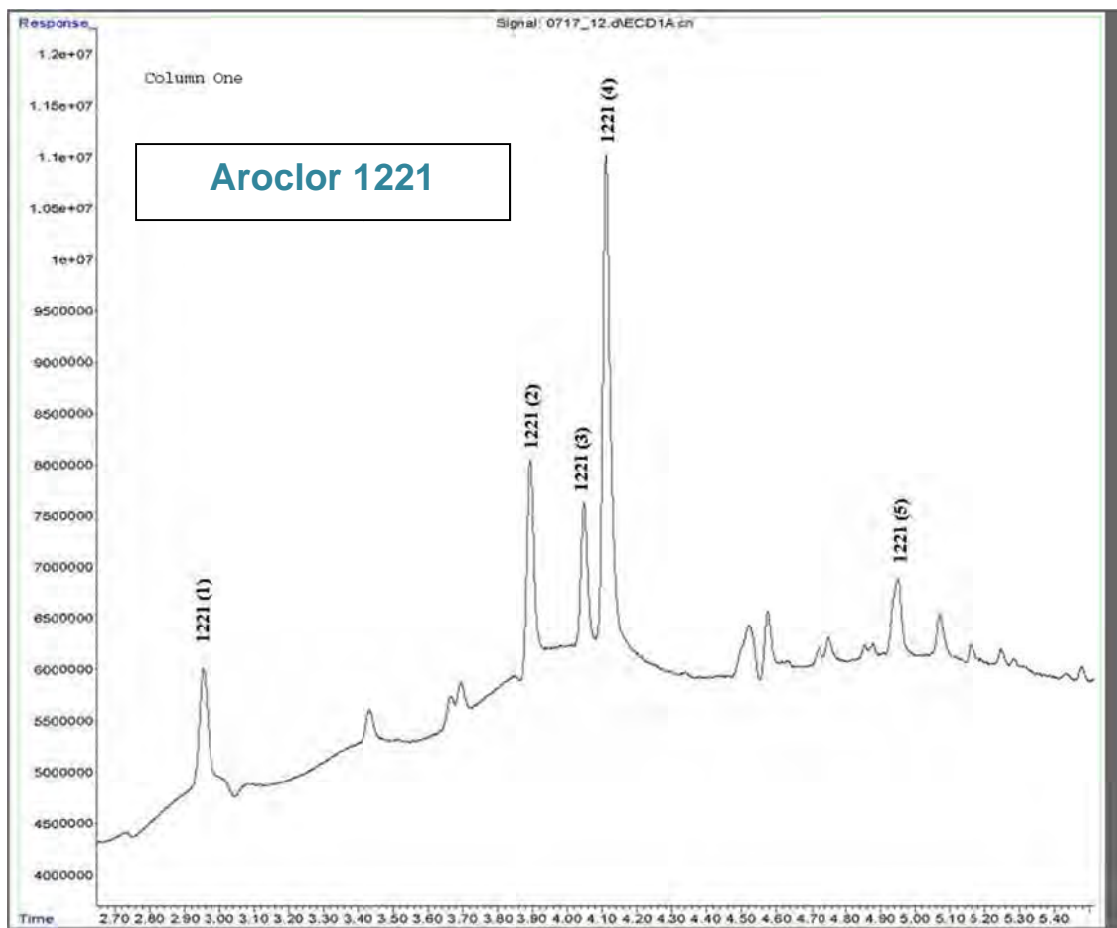
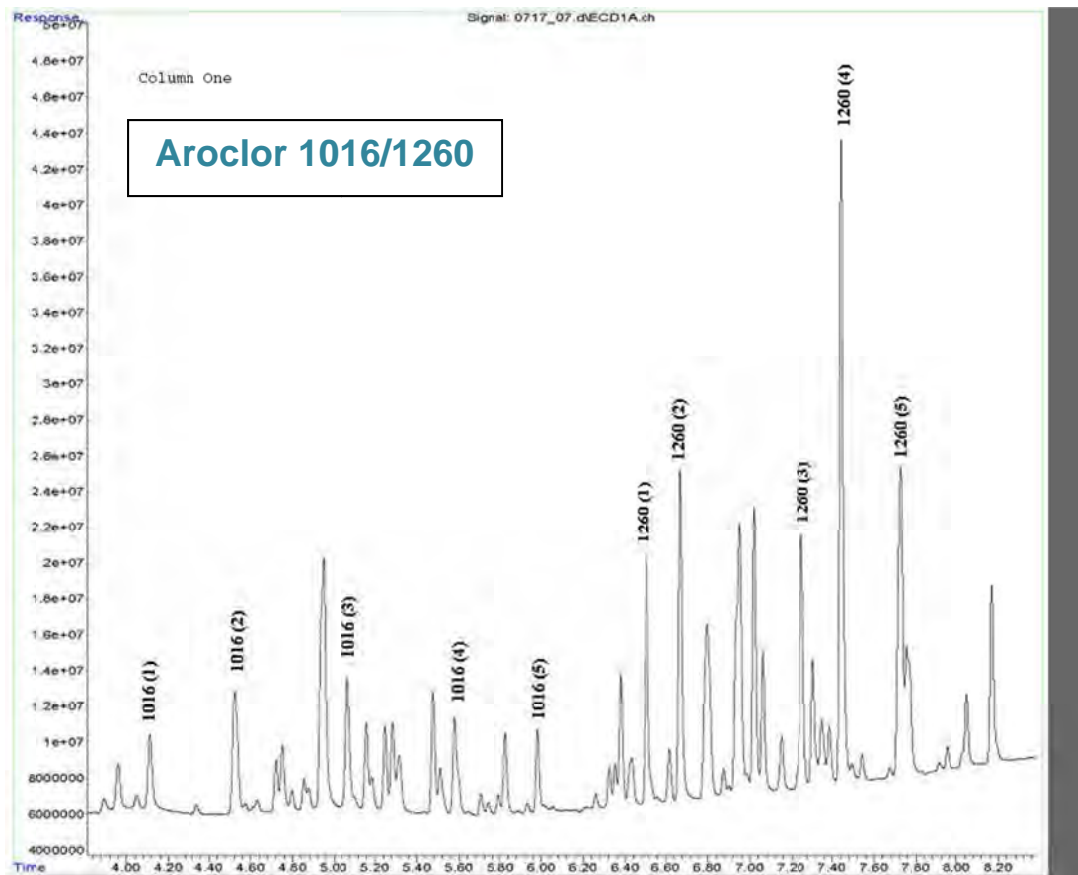
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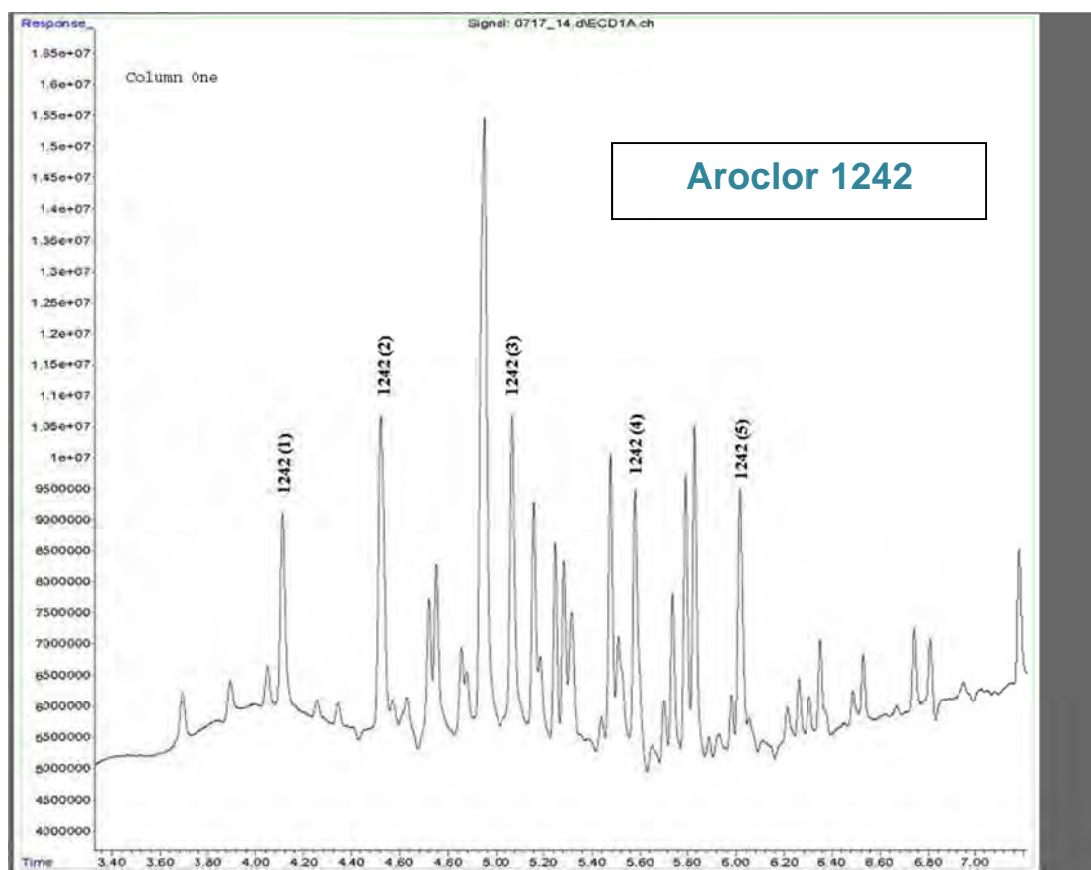
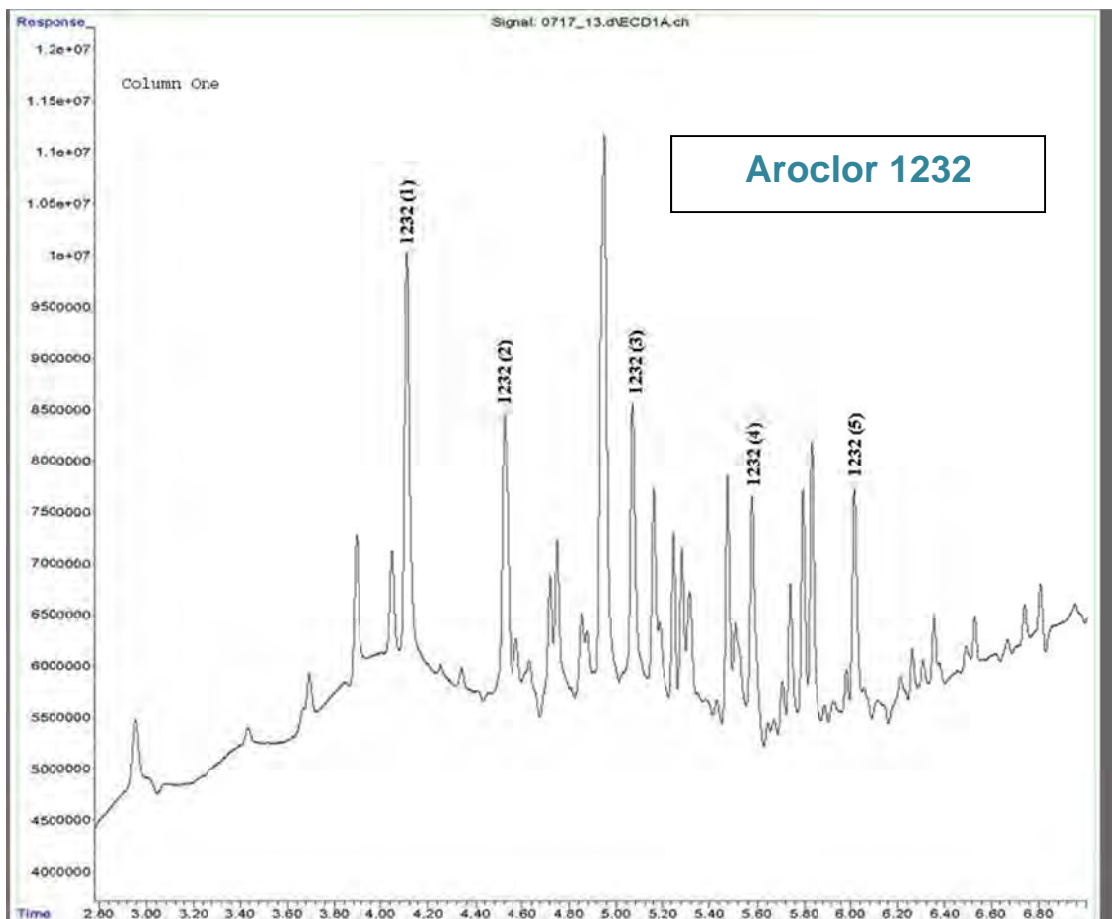
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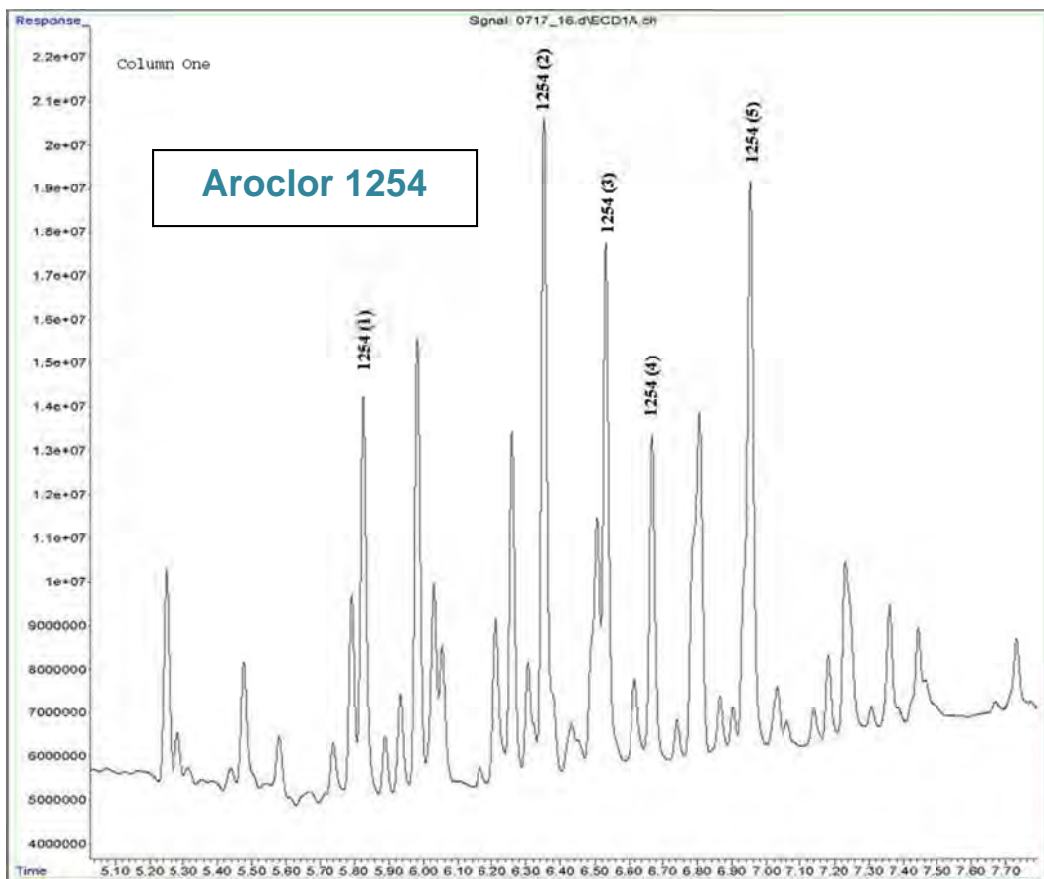
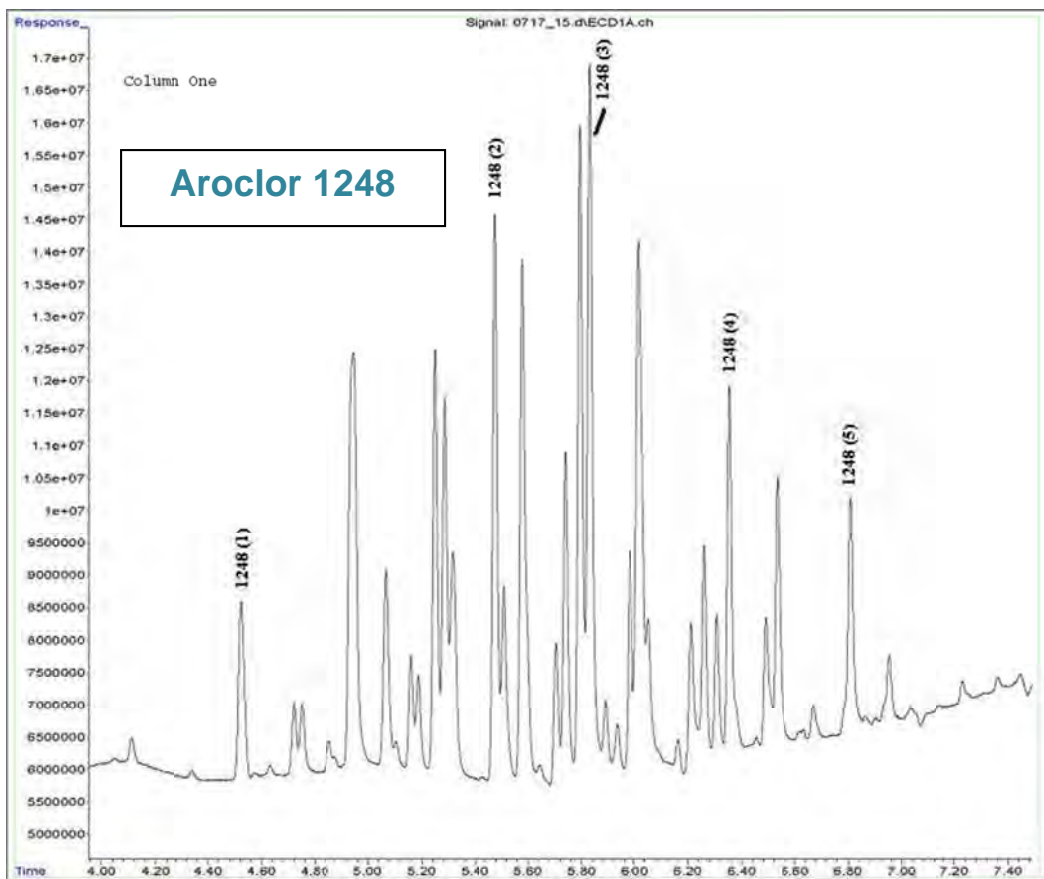
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Procedure/Method : POLYCHLORINATED BIPHENYLS (PCBS) BY GAS CHROMATOGRAPHY (SOIL, WATER & OIL) (EPA METHODS 608, 608.3, 8082, & 8082A, SM 6431B)	

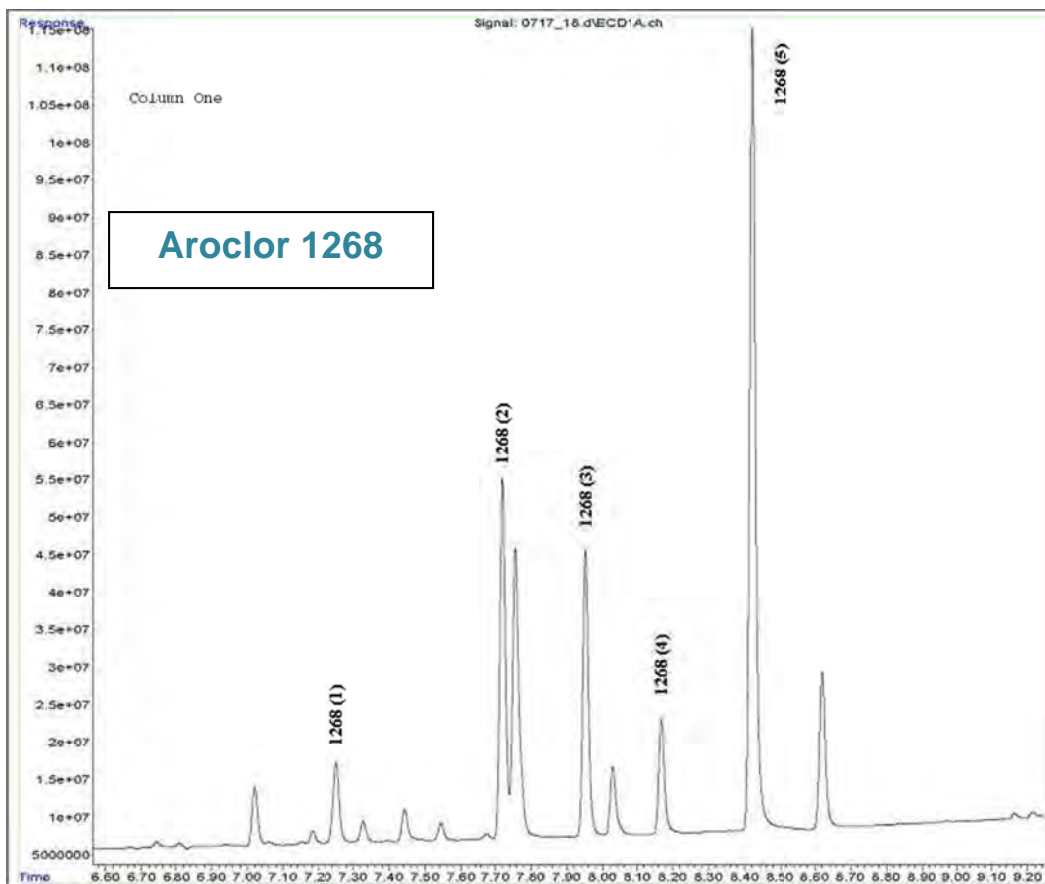
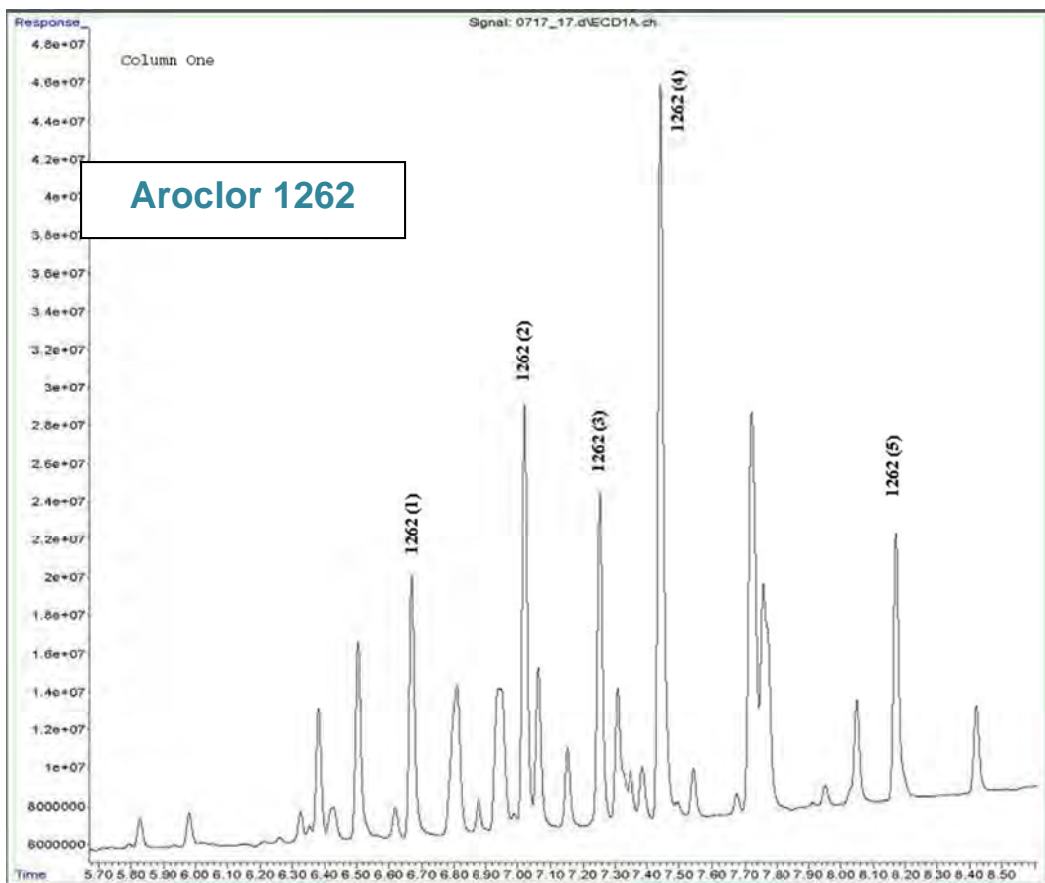
Date	Requested By	Section	Revision	Reason*	Approvals	
					Supervisor	QA
08/07/18	Steve Miller	8.5	<p>Add the following to Section 8.5:</p> <p>“In order to better facilitate consistency and reduce differences between different analysts, see below guidelines and Chromatograms of different Aroclors patterns as references and tools for the process of identification and quantitation of PCBs and multicomponent pesticides, Chlordane and Toxaphene.</p> <p>Aroclor 1016 has the same characteristic first three peak pattern as 1248, but the first peak is much more visible in 1016 when compared to 1242. Aroclor 1016 also does not contain the heavier components of either 1242 or 1248.</p> <p>When deciding between Aroclor 1242 and Aroclor 1248 look for the following characteristics. First, evaluate the ratios of the first three peaks in. both Aroclors. In Aroclor 1242 the peak heights for the first three peaks double from peak to peak. In Aroclor 1248 the peak heights triple from peak to peak.</p> <p>When comparing Aroclors 1254 and 1260, 1254 elutes earlier than 1260. Therefore, if both Aroclors are present, the front part of 1254 will be present ahead of 1260. See chromatograms for labeled peak references.</p> <p>In addition to the aforementioned guidelines chromatograms of each Arclor with the most abundant 5 peaks labeled with in each Aroclor, are now a part of the SOP for a visual reference. Shared peaks identified to be used as guides in determining which Aroclor they belong in cases where more than one multicomponent is detected in the same sample.”</p>	CAR3151	Shakir Wani	

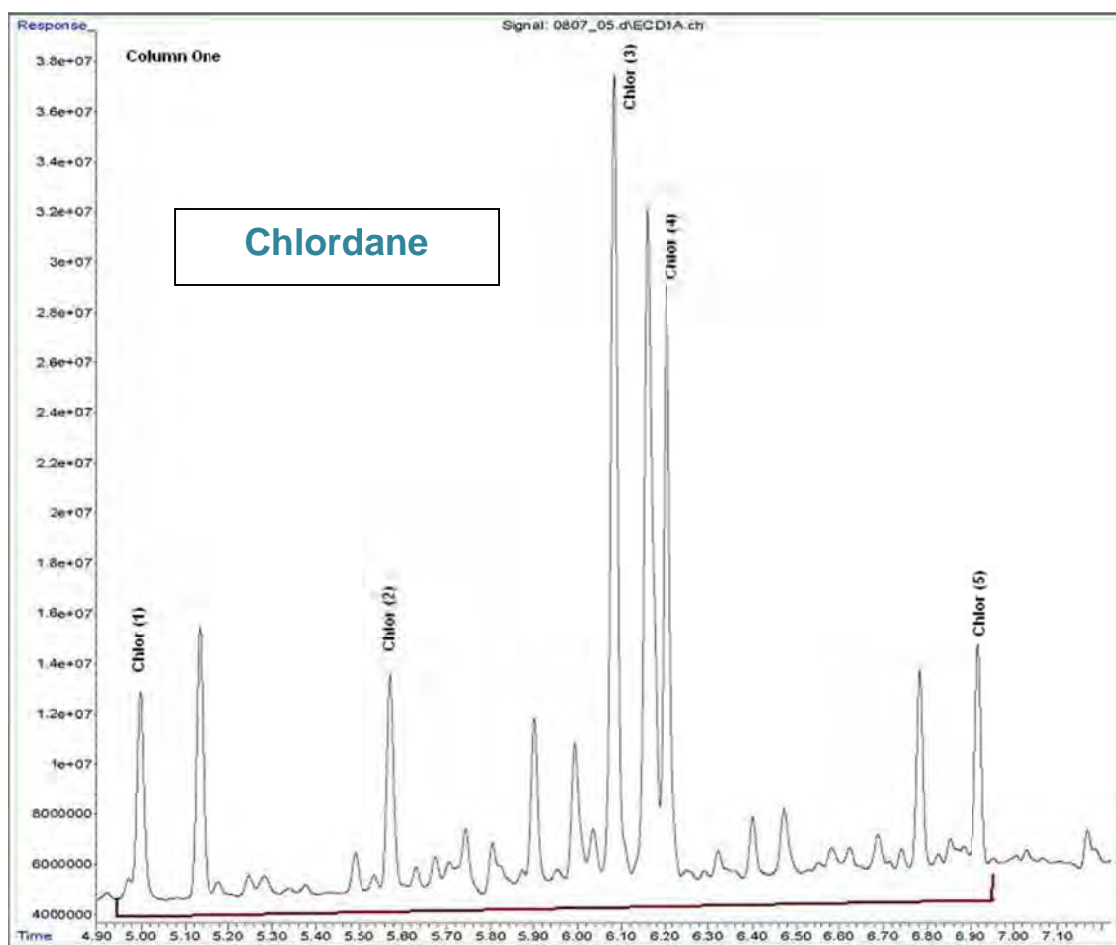
*Comments: See attached chromatograms to be added to the SOP for a visual comparing PCB patterns for identifications.











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**TITLE: PESTICIDES BY GAS CHROMATOGRAPHY
(EPA METHODS 608, 608.3, 8081A, 8081B, SM 6630C)**

Reviewed by: Chris Johnson, Blake Judge, Shakir Wani, Steve Miller

Shakir Wani

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1.0 SCOPE AND APPLICATION

STATE NOTE: For samples analyzed in conjunction with the Ohio Voluntary Action Program (VAP) please utilize SOP #330344OH.

NOTE: EPA Methods 608 and 608.3 include the analysis of Polychlorinated Biphenyls (PCBs). For direction regarding PCB analysis using these methods, see SOP #330343.

1.1 This standard operating procedure represents the following:

- Is designed to determine the amount of certain chlorine-containing pesticides per unit weight or volume in matrices such as waste samples, waters, soils and sludge.
- Capillary columns are employed with electron capture detectors (ECD). Additional procedures can be used for further clarifying highly-contaminated matrices.
- The compounds that are determined by this method are listed in the Table 1.1 with their reporting limits. Actual detection limits vary with the different matrices.

TABLE 1.1 Method Compounds and Reporting Limits (See section 13.2.)

COMPOUND	Pace National		Method 608.3	
	RL (Water mg/L)	RL (Soil mg/kg)	MDL (ng/L)	ML (ng/L)
alpha-BHC	0.0005	0.02	3	9
Aldrin	0.0005	0.02	4	12
beta-BHC	0.0005	0.02	6	18
Chlordane [†]	0.005	0.20		
delta-BHC	0.0005	0.02	9	27
Dieldrin	0.0005	0.02	2	6
Endosulfan I	0.0005	0.02	14	42
Endosulfan II	0.0005	0.02	4	12
Endosulfan sulfate	0.0005	0.02	66	198
Endrin	0.0005	0.02	6	18
Endrin Aldehyde	0.0005	0.02	23	70
Endrin Ketone [†]	0.0005	0.02		
Heptachlor	0.0005	0.02	3	9

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COMPOUND	Pace National		Method 608.3	
	RL (Water mg/L)	RL (Soil mg/kg)	MDL (ng/L)	ML (ng/L)
Heptachlor Epoxide	0.0005	0.02	83	249
Lindane (gamma-BHC)	0.0005	0.02	4	12
Methoxychlor	0.0005	0.02		
p,p'DDD	0.0005	0.02	11	33
p,p'DDE	0.0005	0.02	4	12
p,p'DDT	0.0005	0.02	12	36
Toxaphene	0.01	0.37		
Chlorpyrifos (Dursban)**	0.1	20.0		
Alpha Chlordane	0.5	20.0	14	42
Gamma Chlordane	0.5	20.0	14	42
Method 608.3 – Additional Analytes				
Acephate				
Alachlor				
Atrazine				
Benfluralin (Benefin)				
Bromacil				
Bromoxynil octanoate				
Butachlor				
Captafol				
Captan				
Carbophenothion (Trithion)				
Chlorobenzilate				
Chloroneb (Terraneb)				
Chloropropylate (Acaralate)				
Chlorothalonil				
Cyanazine				
DCPA (Dacthal)				
2,4'-DDD				
2,4'-DDE				
2,4'-DDT				
Diallate (Avadex)				
1,2-Dibromo-3-chloropropane (DBCP)				
Dichlone				
Dichloran				
Dicofol				
Endrin ketone				
Ethalfuralin (Sonalan)				
Etridiazole				
Fenarimol (Rubigan)				
Hexachlorobenzene				

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COMPOUND	Pace National		Method 608.3	
	RL (Water mg/L)	RL (Soil mg/kg)	MDL (ng/L)	ML (ng/L)
Hexachlorocyclopentadiene				
Isodrin				
Isopropalin (Paarlan)				
Kepone				
Methoxychlor				
Metolachlor				
Metribuzin				
Mirex				
Nitrofen (TOK)				
cis-Nonachlor				
trans-Nonachlor				
Norfluorazon				
Octachlorostyrene				
Oxychlordane				
PCNB (Pentachloronitrobenzene)				
Pendamethalin (Prowl)				
cis-Permethrin				
trans-Permethrin				
Perthane (Ethylan)				
Propachlor				
Propanil				
Propazine				
Quintozone				
Simazine				
Strobane				
Technazene				
Technical Chlordane				
Terbacil				
Terbutylazine				
Toxaphene			240	720
Trifluralin				

****** Chlorpyrifos (Dursban, CAS number 2921-88-2) can also be analyzed and reported by this method for special projects, if requested.

F Not listed as a primary target analyte in Method 608.3.

- 1.2 A Method Detection Limits (MDL) study must be completed at least annually or more frequently if major instrumentation changes occur. MDLs are performed based on SOP #030206. Updated MDL records are filed and stored on Pace National's intranet.

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- 1.2.1 Limit of Detection (LOD) and Limit of Quantitation (LOQ) studies are completed at the frequency required by the TNI standard per the procedure identified in the SOP #030206, *Method Detection Limits (MDL), Limits of Detection (LOD) and Limits of Quantitation (LOQ)*. Should the procedure be utilized for DOD support; then the frequency of these studies must meet the requirements of the current DOD QSM (see Attachment II).
- 1.2.2 Lower Limit of Quantitation (LOQ) – For analyses performed per the requirements of Method 8000D, the LLOQ is established at concentrations where both quantitative and qualitative requirements can consistently be met (see Sections 2.2 and 10.19).

2.0 METHOD SUMMARY AND DEFINITIONS

- 2.1 A measured volume or weight of sample is extracted using the appropriate extraction technique. Liquid samples are extracted at neutral pH with methylene chloride using a separatory funnel (SOP #330702) per EPA method 3510C. Reduced volume (RV) extraction using EPA method 3510C that requires a smaller volume (usually 100mL) of field sample is also available for use where applicable. EPA method 3511 that requires a smaller volume (usually 40mL) of field sample is also available for use where applicable. See section 13.3 of this procedure and SOP #330702B. Solid samples are extracted with hexane-methylene chloride using microwave (SOP #330707), where permitted. The extract is brought to a final volume of 30mL with hexane without concentration. Routinely, an internal standard is added to the sample extract then the extract is injected into a gas chromatograph equipped with a capillary column with an electron capture detector (ECD). In these cases, internal calibration is performed; however in cases where there is an obvious interferent co-eluting with the internal standard peak, extracts without internal standard are analyzed and quantitation using external calibration is performed.

METHOD NOTE: Samples may also be extracted using a disk-based solid-phase extraction (SPE) procedure which was approved by the U.S. EPA as an Alternate Test Procedure (ATP) for waste water analyses in 1995 (see Section 14.10).

- 2.2 Lower Limit of Quantitation (LLOQ) – For analyses performed according to the requirements of Method 8000D, the lowest concentration at which the laboratory has demonstrated target analytes can be reliably measured and reported with a certain degree of confidence, which must be greater than or equal to the lowest point in the calibration curve.
- 2.3 LVI: Large Volume Injection: any injection volume >5ul. Technique is dependent upon type of GC inlet used and sensitivity of detection.
- 2.4 See the current Quality Assurance Manual for definitions associated with terms found in this document.

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**TITLE: PESTICIDES BY GAS CHROMATOGRAPHY
(EPA METHODS 608, 608.3, 8081A, 8081B, SM 6630C)**

3.0 HEALTH AND SAFETY

- 3.1 The toxicity or carcinogenicity of each reagent used in the laboratory has not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds must be as low as reasonably achievable. A reference file of safety data sheets (SDSs) is made available on Pace National's intranet to all personnel. Use hazardous reagents in a fume hood whenever possible and if eye or skin contact occurs, flush with large volumes of water. Always wear safety glasses or a shield for eye protection, protective clothing and observe proper mixing protocols.
- 3.2 Many of the compounds determined by this methodology have been identified as known or putative carcinogens in man and/or animals. Exposure to these compounds must be reduced to a minimum. Neat standards should be handled in a fume hood.
- The analyst must use gloves to minimize the possibility of trans-dermal adsorption of these compounds.
- 3.3 Since the electron capture detector is a non-destructive detector, effluent from the gas chromatograph must be vented through an adsorption trap. Large quantities of the dichloromethane extraction solvent should be handled in the fume hood. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. This file is made available to all personnel involved in the chemical analysis.

4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

- 4.1 All samples must have been collected using a sampling plan that addresses the considerations of this method.
- 4.2 Preservation & Holding Time
- If residual chlorine is present, water samples are preserved with 3mL/1000mL sample of 10% sodium thiosulfate per gallon and cooled to $4 \pm 2^{\circ}\text{C}$.
 - The holding time for water samples begins at collection and ends with extraction that must be completed within 7 days. Extract holding time begins with extraction and ends with analysis that must be completed within 40 days.
 - Soils and sludge are cooled to $4 \pm 2^{\circ}\text{C}$ upon collection.
 - The holding time for soil samples begins at collection and ends with extraction that must be completed within 14 days. Extract holding time begins with extraction and ends with analysis that must be completed within 40 days.
- 4.3 Container
- Water samples are collected in a 1 Liter amber bottle with Teflon lined caps for traditional EPA 3510C extractions or in a 100mL amber bottle with Teflon lined caps for 3510RV extraction.
 - Soils are collected in wide-mouth jars with Teflon lined caps.

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- 4.4 Additional requirements for sample extraction are detailed in SOP numbers 330702, 330702B, 330705, 330706, 330707, and 330743.
- 4.5 Samples submitted for analysis that do not meet the requirements contained within this section must be addressed before performing the logging process within the laboratory. In some cases, exceeding the appropriate preservation and storage criteria can cause significant bias in the resulting data. Clients may need to resubmit samples where the conditions during shipment cause uncertainty regarding sample integrity. If samples do not meet the requirements for preservation, sampling, shipment and storage and the client approves the completion of the analytical process, sample results can be qualified and possible bias is narrated per the SOP #030201, *Data Handling and Reporting*.
- 4.6 Method 608.3 allows the use of hydrogen as a carrier gas in place of helium. If used, the laboratory should take the necessary precautions in dealing with hydrogen, and should limit hydrogen flow at the source to prevent buildup of an explosive mixture of hydrogen in air.
- 4.7 Method 608.3 requires that when Aldrin is to be determined and residual chlorine is present in the sample, 80mg/L of sodium thiosulfate must be added, but not to excess.

5.0 INTERFERENCES

5.1 Interferences can be caused by the following:

- Contaminated solvents or reagents
- Sample processing hardware or glassware
- Contaminated carrier gas
- GC parts, column surfaces or detectors
- Co-eluting compounds

5.2 Cleanup procedures are used to remove some of the interferences from sample matrix or interferences from sample matrix or interferences introduced during sample processing (phthalate esters). Cleanup procedures should not be performed on extracts that contain internal standards, as the effects of cleanup processes on these analytes are not fully known.

5.3 Glassware:

- Glassware must be scrupulously cleaned.
- Clean all glassware by detergent washing with hot water and rinsing with tap water and organic-free reagent water.
- Drain the glassware and rinse with acetone and hexane.
- Store dry glassware in a clean environment. See SOP #030701, *Glassware Cleaning*.

5.4 Sulfur:

- The presence of sulfur results in broadening of the peaks on the GC chromatogram.

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- Sulfur can be removed by using copper cleanup. See SOP #330741, *Sulfur Cleanup*.

5.5 Polar contaminants, phenols and unidentified co-extracts may be eliminated using silica gel cleanup. See SOP #330739.

5.6 If co-elutions occur in analysis of a sample, a co-elution on one column is acceptable so long as effective separation of the co-eluting compounds can be achieved on the second column.

6.0 EQUIPMENT AND SUPPLIES

6.1 Instrumentation (or equivalents):

Instrument name:	SVGC #24, #29
Use (method #'s):	608, 8081, 8082, SM 6630C
Model #:	HP 6890/7890 or equivalent
Column (type, brand, size):	Two of the following (or equivalent): STX-CLPesticides 30m x 0.32mm x 0.5um, STX-CLPesticides II 30m x 0.32mm x 0.25um
Detector:	Dual micro ECD
Software name and version:	Enviro Quant Chemstation G1701BA or equivalent
Software version:	D.01.00/E.02.00 or equivalent
Sample introduction system:	Agilent 7683/7693 AS or equivalent
Computer	HP Vectra or equivalent
Gases used (grade and supplier):	He, H ₂ & N ₂ – 4.8
Syringes used (brand, size, type):	Hamilton 250uL, 100uL, 10uL or equivalent

6.2 Class A Volumetric flasks, 10mL and 25mL, for preparation of standards.

6.3 9" VWR Disposable Pasteur pipette or equivalent

6.4 10mL Pyrex disposable pipette or equivalent

6.5 1.8mL Wheaton ABC vials with rubber, Teflon lined cap or equivalent.

7.0 REAGENTS AND STANDARDS

7.1 All reagents and standards must be recorded in the appropriate preparation log and assigned a unique number. See SOP #030230, *Standards Logger – Tree Operation*. Additional information regarding reagent preparation can be found in the Standards Logger (Tree) digital archive system. All spiking solutions and surrogate standard solutions should be replaced at least every 6 months or sooner if a problem is detected unless otherwise noted.

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- 7.2 TCL Pesticide mix (Restek) 10ug/mL, CAT # 563209 or equivalent
- 7.3 TCL Pesticide mix - (Restek) 200ug/L, CAT# 570337 or equivalent
- 7.4 Chlordane - 1000ug/mL (Restek), CAT 32021 or equivalent
- 7.5 Toxaphene - 1000ug/mL (Restek) CAT # 32005 or equivalent
- 7.6 Surrogate standards - 200ug/mL TCMX and Decachlorobiphenyl Ultra Scientific (Cat#ISM-320) or equivalent.
- 7.7 Degradation Check Solution – ULTRA Scientific Cat# ISM-450 or equivalent
- 7.8 SSCV Pest Extraction Spike – 2.0ug/mL – NSI Cat # Q3425 or equivalent

Dilute as follows:

Technique	Method	Matrix	Extracted Sample Amount (g or mL)	Amount Added to Sample (mL)	Final Solvent Volume (mL)
Microwave	3546	Soil	15	0.5	30
Sep Funnel	3510RV	Water	100	.05	5
Sep Funnel	3510C	Water	1000	0.10	10

- 7.9 Organic free reagent water – Prepared in the extraction lab by processing the laboratory DI water through a carbon filtration system.
- 7.10 Hexane VWR Cat# BJGC217-4 or equivalent – pesticide grade
- 7.11 LCS/Matrix Spike Solution - NSI Cat #Q3425 or equivalent. The concentration is 2.0 µg/mL.
 - Water - Measure 100uL of the LCS/MS solution and add to 1 liter of sample or laboratory reagent water for traditional 3510C extraction or 50uL into 100mL for 3510RV. The final concentration is 1.0ug/mL for 100ml and 0.2ug/ml for 1000ml. Soil - 0.5mL to 15g of sample (concentrated to 30mL for microwave extraction). The final concentration is 66.7ug/Kg
- 7.12 Stock Internal standard: 1-Bromo-2-nitrobenzene (5000 mg/L). Ultra Scientific Cat# PPS-351 or equivalent. Dilute stock standard 1-1000. Add 10uL to each 1mL of standards, field samples, method blank, and QC (LCS/LCSD/MS/MSD) extract.
- 7.13 Working Standards: The lowest level of the calibration curve must be at or below the RL Surrogates are included in the working standards at the same concentration as the target analytes. Dilute the certified stock solution in section 7.2 to a final volume of 1mL in hexane as follows to prepare the working calibration standards:

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- For RV and non-concentrated soil analysis from 50ppb intermediate:

Standard Concentration (ppb)	Intermediate Stock Used (uL) in 1mL
.4	2
1.0	5
5.0	25
10.0	50
20.0*	100
50.0	250
100	500
200	1000

* Levels also used for ICV/CCV.

METHOD NOTE: For Method 608.3, one of the calibration standards should be at a concentration at or below the method-defined minimum level (ML) specified in Table 1.1, as specified by a regulatory/control authority, or in a permit:

Alternatively, the laboratory may establish an ML for each analyte based on the concentration of the lowest calibration standard in a series of standards produced by the laboratory or obtained from a commercial vendor, again, provided that the ML does not exceed the method-defined ML, and provided that the resulting calibration meets the acceptance criteria in based on the RSD, RSE, or R^2 .

A separate standard near the MDL may be analyzed as a check on sensitivity, but should not be included in the linearity assessment. The solvent for the standards must match the final solvent for the sample extracts (e.g., isooctane or hexane).

7.14 Sodium sulfate, reagent grade, granular anhydrous, rinsed with methylene chloride, baked in a shallow tray at 450°C for 1 hour minimum, cooled in a desiccator, and stored in a pre-cleaned glass bottle with screw cap which prevents moisture from entering.

7.14.1 If, after heating, the sodium sulfate develops a noticeable grayish cast (due to the presence of carbon in the crystal matrix), that batch of reagent is not suitable for use and should be discarded. Extraction with methylene chloride (as opposed to simple rinsing) and baking at a lower temperature may produce sodium sulfate suitable for use.

7.15 Method 608.3 Standard Requirements

7.15.1 Quality Control (QC) Check Sample Concentrate—Prepare one or more mid-level standard mixtures (concentrates) in acetone (or other water miscible solvent). The concentrate is used as the spiking solution with which to prepare the Demonstration of Capabilities (DOC) samples, the Laboratory Control

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Sample (LCS), and Matrix Spike (MS) and Matrix Spike Duplicate (MSD) samples. If prepared by the laboratory (as opposed the purchasing it from a commercial supplier), the concentrate must be prepared independently from the standards used for calibration, but may be prepared from the same source as the second source standard used for calibration verification.

- 7.15.2 Calibration Verification Standards— In order to verify the results of the initial calibration standards, prepare one or more mid-level standard mixtures in isooctane or hexane, using standards obtained from a second source (different manufacturer or different certified lot from the calibration standards). These standards will be analyzed to verify the accuracy of the calibration. As with the QC sample concentrate, multiple solutions may be required to address coelutions among all of the analytes.
- 7.15.3 Internal standard solution—If the internal standard calibration technique is to be used, prepare Pentachloronitrobenzene (PCNB) at a concentration of 10 mg/mL in ethyl acetate. Alternative and multiple internal standards (e.g., tetrachloro-m-xylene, 4,4'-dibromobiphenyl, and/or decachlorobiphenyl) may be used provided that the laboratory performs all QC tests and meets all QC acceptance criteria with the alternative or additional internal standard(s) as an integral part of this method.
- 7.15.4 Surrogate solution—Prepare a solution containing one or more surrogates at a concentration of 2mg/mL in acetone. Potential surrogates include: dibutyl chlorendate (DBC), tetrachloro-m-xylene (TCMX), 4,4'-dibromobiphenyl, or decachlorobiphenyl. Alternative surrogates and concentrations may be used, provided the laboratory performs all QC tests and meets all QC acceptance criteria with the alternative surrogate(s) as an integral part of this method. If the internal standard calibration technique is used, do not use the internal standard as a surrogate.
- 7.15.5 DDT and endrin decomposition (breakdown) solution—Prepare a solution containing endrin at a concentration of 50ng/mL and 4,4'-DDT at a concentration of 100ng/mL, in isooctane or hexane. A 1-mL injection of this standard will contain 50 picograms (pg) of endrin and 100 pg of DDT. The concentration of the solution may be adjusted by the laboratory to accommodate other injection volumes such that the same masses of the two analytes are introduced into the instrument.

8.0 PROCEDURE

- 8.1 See instrument maintenance logs or Cyberlab for specific details to acquisition method
- 8.2 **Initial Calibration** – Due to the calibration verification and breakdown requirements for this method, the injection liner must be changed daily prior to calibration and/or verification. All other maintenance will be recorded in the specified instrument maintenance log.

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8.2.1 DDT and Endrin Degradation – For calculations, see section 9.1

- DDT and Endrin are easily degraded in the injection port.
- Breakdown occurs when the injection liner is contaminated with high boiling residue.
- Check for degradation by injecting up to 50uL of a solution containing 10/20ug/L of Endrin/DDT
- Acceptance criteria for this injection are found in section 10.9 with corrective actions for failures in section 11.8.

8.2.1.1 Method 608.3 requires that if DDT, Endrin, or their breakdown products are to be determined, then the degradation test must be performed prior to calibration verification. DDT decomposes to DDE and DDD. Endrin decomposes to Endrin aldehyde and Endrin ketone.

8.2.2 Initial Calibration Curve

- Prepare five or more concentrations of each single component pesticide, multicomponent pesticide, and surrogate (TCMX and DCBP) in 1mL of hexane as noted in section 7.13. A minimum of 5 standards for each analyte are required for EPA Method 8081 and 608.3. A minimum of 3 standards for each analyte are required for EPA Method 608.
- All target analytes are included in the calibration curve.
- Calibration standards must be replaced if comparison to a secondary check standard reveals a problem.
- Inject the calibration standards to generate a working curve. HP Chemstation calculates the calibration factor or response factor for each compound in each standard according to the equations found in section 9.2 & 9.3.
- Average Calibration/Response Factor is calculated for each target analyte by averaging each of the individual calibration factors. See equations in section 9.2 & 9.3 and method performance requirements in section 10.4.
- Linear Regression Option: A calibration curve can be used instead of average CF/RF for quantitation when the percent relative standard deviation %RSD exceeds acceptance criteria. See equations in sections 9.4 & 9.5.
- Method 608.3 requires that one of the calibration standards be at or below the ML (see Table 1.1).

When the appropriate number of calibration standards is used, all points must be considered in the average response factor calculation or linear regression calculation. The deletion of the highest point is acceptable when necessary, with the analyst noting that the high end of the calibration has been lowered. The deletion of the lowest calibration point is acceptable, when necessary, provided that the analyst notes the deletion on the injection log and raises the reporting

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limit, if necessary, for that compound. It is also necessary to print the calibration curve showing linearity and acceptable correlation coefficient.

8.2.3 **Dual Column Confirmation:** Calibration criteria must be met on both columns for positive confirmation of target analytes.

8.2.4 **Manual Integrations:** Manual integrations are performed, when needed, according to the SOP #030215, *Manual Integrations*.

8.2.5 Method 608.3 Requirements

Injection of calibration solutions— Inject a constant volume of each calibration solution into the GC column/detector pairs. An alternative volume may be used provided all requirements in this method are met. Beginning with the lowest level mixture and proceeding to the highest level mixture may limit the risk of carryover from one standard to the next, but other sequences may be used. An instrument blank should be analyzed after the highest standard to demonstrate that there is no carry-over within the system for this calibration range.

8.3 **Priming the Column:** The GC column may be primed (or deactivated) prior to calibration/degradation check by injecting a pesticide standard approximately 20 times the midpoint if the column has not been used for a day or more.

8.4 **Multicomponent Standard Check:** Each working day a mid-level ICV of Chlordane & Toxaphene is injected before analysis of samples at 200ug/L to verify calibration. If the ICV does not pass for these multicomponent targets, then the analytes in field samples are not reportable until a new calibration curve is analyzed.

STATE NOTE: For Arizona compliance samples multi-peak components, including Toxaphene, and Chlordane, are injected at the reporting level. If any of these compounds are detected in the sample, a five-point calibration is performed, with the lowest standard at or below the RL. The sample is diluted if high concentrations of these compounds are present. The area of 5 selected peaks is compared to the same peaks in the sample for quantitation for Chlordane.

STATE NOTE: For all samples analyzed from Minnesota, the reporting limit must be verified at least monthly, with each new initial calibration, or when there has been significant change to the instrument (column replacement, cleaning source, etc.) whichever is more frequent. The reporting limit verification can be performed by either re-injecting the low standard or by re-processing the low standard that was analyzed in the calibration curve. The reporting limit verification (RLV) must recovery within $\pm 40\%$ of the expected concentration. If this criterion is not met, the RLV may be re-analyzed once, instrument maintenance can be performed, a higher concentration standard can be injected, or a new calibration curve must be generated. If a higher concentration standard is utilized, the reporting limit must be raised to the higher level verified.

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8.5 Quantitation and Chromatogram Review:

Sample Analysis: Sample extracts, method blank and corresponding (LCS/LCSD/MS/MSD), in 1.8mL vials for soil, 16ml for 1L water extractions, or 8mL for RV waters, are retrieved from the extraction lab.

- The sample ID's must be checked, by the analyst, and must match the samples listed in the Prep Data program.
- The analyst must sign the extraction log as verification of receipt and correctness. A secondary signature is by another trained extractor denotes review of the Standards used
- The method blank, LCS/LCSD, MS/MSD and samples are loaded into the autosampler. All dilution, volume, and weight information are carefully recorded as part of the injection log entry.
- Appropriate multipliers are assigned based on the sample extraction information. Sample information on the quant report must match the information on the injection log.
- The samples are injected via autosampler into the instrument and the corresponding quantitation report and chromatograph are generated.

Identification: Tentative identification of an analyte occurs when a peak from a sample extract falls within the absolute retention time window.

- Each tentative identification must be confirmed using either a second GC column of dissimilar stationary phase or using another technique such as GC/MS.
- Results are routinely confirmed using a second GC column of dissimilar stationary phase followed by the use of a second detector.
- The analyst must check the agreement between the quantitative results on both columns/detectors once the identification has been confirmed.
- Detectable amounts on either column require the analyst to review the same compound on the corresponding column.
- If the compound is below the reporting limit on the corresponding column, the analyst must review the integration and verify that the compound is or is not valid.
- Detectable amounts between the MDL and RL require appropriate review and integration for verification.
- If chromatographic problems are evident, clean-up procedures must be considered. The silica Gel clean-up technique is usually appropriate for typical interferences.

Method 608.3 Identification:

- In order to identify a single component analyte from analysis of a sample, blank, or other QC sample, the peak representing the analyte must fall within its respective retention time windows on both column/detector systems.
- The relative agreement between the numerical results from the two GC columns may be used to support the identification of the target analyte by providing evidence that coeluting interferences are not present at the retention time of the

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target analyte. Calculate the percent difference (%D) between the results for the analyte from both columns. In general, if the %D of the two results is less than 50% (e.g., a factor of 2), then the pesticide is present. Note: Laboratories may employ metrics less than 50% for this comparison, including those specified in other analytical methods for these pesticides (e.g., CLP or SW-846).

- Report the lower result from the two columns for each analyte in each sample or QC standard at or above the ML to 3 significant figures.
- Report a result for each analyte in each sample or QC standard below the ML (see Table 1.1) as "<ML," where "ML" is the concentration of the analyte at the ML (e.g., if the ML is 10 mg/L, then report the result as <10 mg/L), or as required by the regulatory authority or permit.
- Report a result for each analyte in a blank at or above the MDL to 2 significant figures. Report a result for each analyte found in a blank below the MDL as "<MDL," where MDL is the concentration of the analyte at the MDL, or as required by the regulatory/control authority or permit.

8.6 Chlordane and Toxaphene

- ICV is run with every batch at 100ppb

Detection of Multi-Peak Compounds:

- When toxaphene, and/or chlordane are detected in field samples, a full calibration curve as noted in section 8.2.3 is utilized for quantitation. If a full calibration is not verified on the day of detection of these compounds in field samples, then the daily ICV and any CCVs must pass method continuing calibration criteria for these analytes to be able to report these targets.
- When reporting toxaphene or chlordane, the results from the total area is used unless background interference is present. Where there is interference, the five major peaks for chlordane are used over the total area, to calculate the final result.
- Pace National maintains current records of pattern recognition for all multi component compounds in the semi-volatile department.

STATE NOTE: Arizona compliance samples require that one multi-peak component target has a full calibration curve and all other multi-peak components (i.e., toxaphene, Aroclors, and chlordane) must be injected at the laboratory reporting level for DW samples.

8.7 Midpoint Check Standard – Initial/Continuing Calibration Verification (ICV/CCV)

- An initial calibration verification standard (ICV) must be analyzed to verify continuing acceptability of the most recent calibration curve on days that a full initial calibration curve is not required.
- For internal standard technique, a calibration check standard (CCV) must be analyzed once every 12hrs and/or prior to QC or 20 client sample batches.
- Acceptance criteria for the ICV/CCV can be found in section 10.7.

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- All CCVs must be checked against set retention time windows.
- If any CCV falls outside retention time windows, the GC system is out of control.
- Determine the cause of the problem and correct it.
- If the problem cannot be corrected, a new initial calibration must be performed.

STATE NOTE: For Arizona compliance samples and other states a complete calibration curve for each single-component pesticide is analyzed and quantitated before the analysis of samples.

8.7.1 Method 608.3 requires that the calibration curve be verified at the beginning and end of each 24-hour shift by the analysis of a mid-level calibration standard. The calibration verification standard(s) must be obtained from a second source.

8.8 Second Source Calibration Verification (SSCV) - The initial calibration curve generated must be verified using a source that is different from the stock solutions used to prepare the calibration curve. This source can be a separate manufacturer or separate lot number from the same manufacturer, if available. Routinely, the second source verification is performed at the mid-range of the calibration curve, but the concentration may be altered to better reflect client/project needs. The calibration factor for the SSCV is calculated using the equation found in section 9.0 and the difference from the initial calibration curve is determined using the equation also found in that section.

9.0 DATA ANALYSIS AND CALCULATIONS

9.1 DDT Breakdown:

$$\% \text{ Breakdown for DDT} = \frac{\text{Total DDT Degradation peak area (DDD + DDE)}}{\text{Total Peak Areas (DDT + DDD + DDE)}}$$

Endrin Breakdown:

$$\% \text{ Breakdown for Endrin} = \frac{\text{Total Endrin Degradation peak area (Endrin Aldehyde + Endrin ketone)}}{\text{Total Peak Areas Endrin + Endrin Aldehyde + Endrin ketone}}$$

9.2 The compounds detected are quantitated as follows (except for multi-component compounds):

$$\text{water mg/L} = \frac{\text{Area of Analyte}}{\text{Average CF}} \times \frac{\text{mL of extract}}{\text{mL of sample}} \times \text{Dilution Factor}$$

$$\text{soil mg/kg} = \frac{\text{Area of Analyte}}{\text{Average CF}} \times \frac{\text{mL of extract}}{\text{grams of sample}} \times \text{Dilution Factor}$$

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9.3 Multi-component compounds:

$$\text{soil mg/kg} = \frac{\text{Total Peak Area}}{\text{Average CF}} \times \frac{\text{mL of extract}}{\text{grams of sample}} \times \text{Dilution Factor}$$

$$\text{water mg/L} = \frac{\text{Total Peak Area}}{\text{Average CF}} \times \frac{\text{mL of extract}}{\text{mL of sample}} \times \text{Dilution Factor}$$

9.3.1 Total Peak Area:

NOTE: The "Total Peak Area" may be replaced by the "Total of selected major peaks" when the background interference is high, see 9.6.2.

9.3.2 Selected Individual Peaks

$$\text{Sample Concentration} = \left(\frac{\text{Sum of Selected Peaks}}{\text{\# of Peaks Selected}} \right)$$

9.4 Internal Calibration Equations (Response Factors):

$$RF = \frac{[A_s][C_{is}]}{[A_{is}][C_s]}$$

where:

- A_s = Peak area (or height) of the analyte or surrogate.
- A_{is} = Peak area (or height) of the internal standard.
- C_s = Concentration of the analyte or surrogate, in $\mu\text{g/L}$.
- C_{is} = Concentration of the internal standard, in $\mu\text{g/L}$.

- Percent Relative Standard Deviation (%RSD)

$$\overline{RF} = \frac{\sum_{i=1}^n RF_i}{n} \quad SD = \sqrt{\frac{\sum_{i=1}^n (RF_i - \overline{RF})^2}{n-1}} \quad RSD = \frac{SD}{\overline{RF}} \times 100\%$$

where:

- RSD = Relative standard deviation.
- RF = Mean of 5 initial RFs for a compound.
- SD = Standard deviation of average RFs for a compound.

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- Concentration of an analyte in an extract using RF (on column):

$$X_s = \frac{(Conc_{ISld})(Area_{Analyte})}{(Average RF_{analyte})(Area_{ISld})}$$

where:

X_s = Calculated raw concentration of analyte (in ppb)

- Quantitation Report Multiplier

$$M_a = \frac{(V_t)(D)}{(V_s)} \quad \text{or} \quad M_s = \frac{(V_t)(D)}{(W_s)}$$

where:

M_a = Quantitation Report Multiplier for Aqueous Samples

M_s = Quantitation Report Multiplier for Solid Samples

V_t = Total volume of concentrated extract (in mL)

D = Dilution factor. If no dilution, $D=1$. Always dimensionless

V_s = Volume of aqueous sample extracted (in mL)

W_s = Weight sample extracted (in grams)

- Sample concentration by volume (ug/L) for aqueous samples:

$$\text{Concentration in } \frac{\mu g}{L} = X_s * M_a$$

- Sample concentration by weight (ug/kg) for solid samples and non-aqueous liquids:

$$\text{Concentration in } \frac{\mu g}{kg} = \frac{(X_s)(M_s)}{(\%S)}$$

where:

$\%S$ = Percent solids expressed as a decimal

9.5 Percent Error (%Error)

$$\%Error = \frac{x_i - x'_i}{x_i} * 100$$

where:

x'_i = Measured amount of analyte at the calibration level i , in mass or concentration units

x_i = True amount of analyte at calibration level i , in mass or concentration units

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- 9.6 Relative Standard Error (%RSE) – As an alternative to using the average response factor when using Method 608.3, the quality of the calibration may be evaluated using the Relative Standard Error (RSE). The acceptance criterion for the RSE is the same as the acceptance criterion for Relative Standard Deviation (RSD), in the method. RSE is calculated as:

$$\%RSE = 100 \times \frac{\sum_{i=1}^n \left[\frac{x'_i - x_i}{x_i} \right]^2}{(n - p)}$$

where:

- x'_i = Calculated concentration at level i
- x_i = Actual concentration of the calibration level i
- n = number of calibration points
- p = Number of terms in the fitting equation (average = 1; linear = 2; quadratic = 3)

- 9.7 See the current Quality Assurance Manual for other equations associated with common calculations.

10.0 QUALITY CONTROL AND METHOD PERFORMANCE

- 10.1 All analysts must meet the qualifications specified in SOP #030205, *Technical Training and Personnel Qualifications* before approval to perform this method. Analysts must complete an initial demonstration of proficiency before being approved to perform this method. Continuing proficiency must be demonstrated using proficiency testing, laboratory control sample analysis and/or MDL studies. Method performance is assessed per analyst. Updated method performance records are filed and stored in a central location within the department.

10.1.1 Method 608.3 Demonstration of Capability (DOC) Requirements

- 10.1.1.1 For the DOC, a QC check sample concentrate containing each analyte of interest is prepared in a water miscible solvent using the solution in Section 7.15.1.
- 10.1.1.2 Prepare four QC check samples by adding an appropriate volume of the concentrate and of the surrogate(s) to each of four 1–L aliquots of reagent water. Swirl or stir to mix.
- 10.1.1.3 Extract and analyze the well-mixed QC check samples.
- 10.1.1.4 Calculate the average percent recovery (\bar{x}) and the standard deviation (s) of the percent recovery for each analyte using the four results.
- 10.1.1.5 For each analyte, compare s and \bar{x} with the following acceptance criteria for precision and recovery. For analytes that are not listed, QC acceptance criteria must be developed by the laboratory.

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Analyte	Limit for s (% SD)	Range for \bar{x} (%)
Aldrin	25	54-130
Alpha-BHC	28	49-130
Beta-BHC	38	39-130
Delta-BHC	43	51-130
Gamma-BHC	29	43-130
Alpha-Chlordane	24	55-130
Gamma-Chlordane	24	55-130
4,4'-DDD	32	48-130
4,4'-DDE	30	54-130
4,4'-DDT	39	46-137
Dieldrin	42	58-130
Endosulfan I	25	57-141
Endosulfan II	63	22-171
Endosulfan sulfate	32	38-132
Endrin	42	51-130
Heptachlor	28	43-130
Heptachlor epoxide	22	57-132
Toxaphene	30	56-130

If s and \bar{x} for all analytes of interest meet the acceptance criteria, system performance is acceptable and analysis of blanks and samples can begin. If any individual s exceeds the precision limit or any individual \bar{x} falls outside the range for recovery, system performance is unacceptable for that analyte.

- 10.1.1.6 When one or more of the analytes tested fail at least one of the acceptance criteria, repeat the test for only the analytes that failed. If results for these analytes pass, system performance is acceptable and analysis of samples and blanks may proceed. If one or more of the analytes again fail, system performance is unacceptable for the analytes that failed the acceptance criteria. Correct the problem and repeat the test.

- 10.2 Use the designated Run log to record batch order and standards/reagents used during analysis. See SOP #030201, *Data Handling and Reporting*.

- 10.3 Batches:

Batches are defined as sets of 1 - 20 samples. Batch analysis must include the following: 1 method blank, 1 Laboratory Control Sample/Laboratory Control Sample Duplicate (LCS/LCSD), 1 Initial Calibration Verification (ICV), 1 Matrix Spike/Spike Duplicate (MS/MSD), 1 Continuing Calibration Verification (CCV) before client samples/every 12hrs/prior to every 20 samples, Method 608 requires a matrix spike at

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the rate of 10%. All batch information must be maintained in the preparation documentation assigned to the department.

- 10.4 Initial Calibration – If the percent relative standard deviation (% RSD) of the calibration factors for each analyte is $\leq 20\%$ for EPA 8081A/8081B/SM6630C and $\leq 10\%$ for EPA method 608, the average calibration factor can be used for quantitation. If the %RSD exceeds the method defined acceptance criteria, a calibration curve using linear regression can be employed. The linear regression calibration curve must have a correlation factor of 0.990 (USACE requires 0.995) or greater using equal or inverse weighting. The origin may not be used as a point in the calibration curve and the curve must not be forced through zero.

10.4.1 Method 608.3 Requirements

10.4.1.1 External Standard Calibration

- 10.4.1.1.1 For multi-component analytes, choose a series of characteristic peaks for each analyte (3 to 5 for each Aroclor) and calculate individual calibration factors for each peak.
- 10.4.1.1.2 If the RSD is less than 20%, linearity through the origin can be assumed and the average CF can be used for calculations. Alternatively, the results can be used to fit a linear or quadratic regression of response. If used, the regression must be weighted inversely proportional to concentration. The coefficient of determination (R^2) of the weighted regression must be greater than 0.920. Alternatively, the relative standard error may be used as an acceptance criterion. As with the RSD, the RSE must be less than 20%. If an RSE less than 20% cannot be achieved for a quadratic regression, system performance is unacceptable and the system must be adjusted and re-calibrated.

10.4.1.2 Internal Standard Calibration

- 10.4.1.2.1 If the RSD is less than 15%, linearity through the origin can be assumed and the average RF can be used for calculations. Alternatively, the results can be used to prepare a calibration curve of response ratios, A_s/A_{is} , vs. concentration ratios, C_s/C_{is} , for the analyte. A minimum of six concentration levels is required for a nonlinear (e.g., quadratic) regression. If used, the regression must be weighted inversely proportional to concentration, and the coefficient of determination of the weighted regression must be greater than 0.920. Alternatively, the relative standard error (Reference 10) may be used as an

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acceptance criterion. As with the RSD, the RSE must be less than 15%. If an RSE less than 15% cannot be achieved for a quadratic regression, system performance is unacceptable and the system must be adjusted and re-calibrated.

- 10.5 Method Blank - A method blank must be extracted and analyzed with each set of samples. The method blank must be carried through the same procedure as the samples and must not contain target analytes above the method detection limit.
- 10.6 Matrix Spike (MS) And Matrix Spike Duplicate (MSD) - must be extracted and analyzed with each batch of samples when sufficient sample volume is provided by the client
- Method 608 states that matrix spikes must be done at a rate of 10%.
 - The spike and spike duplicate must meet current acceptance criteria. Attachment II represents QC acceptance criteria calculated from historical values for the method. The acceptance criteria are tighter than that of the 608 and 6630C methods.

STATE NOTE: For all samples analyzed from South Carolina, the MS/MSD recoveries must be within the most stringent limits comparing in-house derived recovery limits and those given in Table 3 of Method 608. The following are the current limits:

Parameter	Pace National Recovery Limits	Maximum MS/MSD %RPD
alpha-BHC	43 – 104%	36
Aldrin	42 – 113%	35
beta-BHC	35 – 120%	44
delta-BHC	32 – 113%	52
Dieldrin	45 – 109%	49
Endosulfan I	45 – 117%	28
Endosulfan II	40 – 125%	53
Endosulfan sulfate	37 – 126%	38
Endrin	36 – 135%	48
Endrin aldehyde	31 – 98%	
Endrin ketone	26 – 129%	
Heptachlor	34 – 111%	43
Heptachlor epoxide	44 – 107%	26
Lindane (gamma-BHC)	43 – 105%	39
Methoxychlor	10 – 147%	
p,p-DDD	47 – 117%	39
p,p-DDE	43 – 107%	35
p,p-DDT	25 – 136%	42
alpha-Chlordane	39 – 109%	35
gamma-Chlordane	27 – 131%	35
Toxaphene		41

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10.6.1 Method 608.3 Requirements

10.6.1.1 The laboratory must, on an ongoing basis, spike at least 5% of the samples in duplicate from each discharge being monitored to assess accuracy (recovery and precision). If direction cannot be obtained from the data user, the laboratory must spike at least one sample in duplicate per extraction batch of up to 20 samples. Spiked sample results should be reported only to the data user whose sample was spiked, or as requested or required by a regulatory/control authority, or in a permit.

10.6.1.2 If, as in compliance monitoring, the concentration of a specific analyte will be checked against a regulatory concentration limit, the concentration of the spike should be at that limit; otherwise, the concentration of the spike should be one to five times higher than the background concentration, at or near the midpoint of the calibration range, or at the concentration in the LCS whichever concentration would be larger. When no information is available, the midpoint of the calibration may be used.

10.6.1.3 Compare the percent recoveries (P1 and P2) and the RPD for each analyte in the MS/MSD aliquots with the corresponding QC acceptance criteria for recovery (P) and RPD in the tables in Sections 10.6 and 10.7 of this SOP.

If any individual P falls outside the designated range for recovery in either aliquot, or the RPD limit is exceeded, the result for the analyte in the unspiked sample is suspect and may not be reported or used for permitting or regulatory compliance.

For analytes not listed in the table, QC acceptance criteria must be developed by the laboratory.

10.6.1.4 After analysis of a minimum of 20 MS/MSD samples for each target analyte and surrogate, and if the laboratory chooses to develop and apply optional in-house QC limits, the laboratory should calculate and apply the optional in-house QC limits for recovery and RPD of future MS/MSD samples. The in-house QC limits must be updated at least every two years and reestablished after any major change in the analytical instrumentation or process. At least 80% of the analytes tested in the MS/MSD must have in-house QC acceptance criteria that are tighter than those in the table presented in Section 10.7 and the remaining analytes (those not included in the 80%) must meet the acceptance criteria in the table.

If an in-house QC limit for the RPD is greater than the limit in the table, then the limit in the table must be used. Similarly, if an in-house lower limit for recovery is below the lower limit in the table, then the lower

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limit in the table must be used, and if an in-house upper limit for recovery is above the upper limit in the table, then the upper limit in the table must be used.

10.7 Laboratory Control Sample/Laboratory Control Sample Duplicate (LCS/LCSD) - must be extracted and analyzed with each batch of samples.

- The control must be within current acceptance criteria.

STATE NOTE: For all 8081 samples analyzed from South Carolina, the LCS/LCSD RPD must be <20% and recoveries must be within and the following limits in a water matrix:

Parameter	Pace National Recovery Limits	Method 608.3 Range for P
alpha-BHC	70 – 130%	37-140
Aldrin	70 – 122%	42-140
beta-BHC	70 – 130%	17-147
delta-BHC	70 – 130%	19-140
Dieldrin	70 – 130%	36-146
Endosulfan I	70 – 130%	45-153
Endosulfan II	70 – 130%	D-202
Endosulfan sulfate	70 – 130%	26-144
Endrin	70 – 130%	30-147
Endrin aldehyde	70 – 130%	
Endrin ketone	70 – 130%	
Heptachlor	70 – 111%	34-140
Heptachlor epoxide	70 – 130%	37-142
Lindane (gamma-BHC)	70 – 127%	32-140
Methoxychlor	70 – 130%	
p,p-DDD	70 – 130%	31-141
p,p-DDE	70 – 130%	30-145
p,p-DDT	70 – 130%	25-160
alpha-Chlordane	70 – 130%	45-140
gamma-Chlordane	70 – 130%	45-140
Toxaphene		41-140

10.7.1 Method 608.3 Requirements

- 10.7.1.1 Prepare the LCS by adding QC check sample concentrate to reagent water. Include all analytes of interest in the LCS. The volume of reagent water must be the same as the nominal volume used for the sample, the DOC, the blank, and the MS/MSD.
- 10.7.1.2 Analyze the LCS prior to analysis of samples in the extraction batch.
- 10.7.1.3 For each analyte, compare the percent recovery (P) with its corresponding QC acceptance criterion in the table in Section 10.7.

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For analytes of interest not listed in the table, use the QC acceptance criteria developed for the MS/MSD or limits based on laboratory control charts. If the recoveries for all analytes of interest fall within the designated ranges, analysis of blanks and field samples may proceed.

10.8 Initial/Calibration Check Standard (ICV/CCV) - On days when a full calibration is not needed, an ICV/CCV must be analyzed prior to the analysis of any Method Blank, QC (LCS/LCSD/MS/MSD) and field samples, every 12 hours, and prior to every 20 samples. There is not an ending standard required for internal standard technique.

- The CF/RF must be within 15% of the initial calibration.

10.8.1 Method 608.3 Requirements

10.8.1.1 The working calibration curve, CF, or RF must be verified immediately after calibration and at the beginning and end of each 24-hour shift by the analysis of a midlevel calibration standard. The calibration verification standard(s) must be obtained from a second manufacturer or a manufacturer's batch prepared independently from the batch used for calibration. Alternatively, calibration verification may be performed after a set number of injections (e.g., every 20 injections), to include injection of extracts of field samples, QC samples, instrument blanks, etc. (i.e., it is based on the number of injections performed, not sample extracts). The time for the injections may not exceed 24 hours.

NOTE: The 24-hour shift begins after analysis of the combined QC standard (calibration verification) and ends 24 hours later. The ending calibration verification standard is run immediately after the last sample run during the 24-hour shift, so the beginning and ending calibration verifications are outside of the 24-hour shift. If calibration verification is based on the number of injections instead of time, then the ending verification standard for one group of injections may be used as the beginning verification for the next group of injections.

10.9 Endrin and DDT breakdown - must be determined before analysis begins and at the beginning of each 12 hour shift. The breakdown must not be greater than 15% for either compound. For Method 608.3, the percent breakdown must be less than 20%.

10.10 Confirmation - Any sample that shows a detectable concentration of any compound above the method detection limit must be confirmed on a second column or by GC/MS. The result from the primary column and the confirmation column should agree within 40% RPD. This also applies to results between MDL and RL.

For Method 608.3, if the %D of the two results is less than 50% (e.g., a factor of 2), then the pesticide is present. Note: Laboratories may employ metrics less than 50% for this comparison, including those specified in other analytical methods for these pesticides (e.g., CLP or SW-846).

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- 10.11 Surrogate Recovery - Calculate the surrogate recovery on all samples, method blanks, and spikes (MS/MSD/LCS/LCSD). Determine if the recovery is within the acceptance criteria.
- 10.12 Internal Standards (internal calibration model) – For method 8081: The internal standard area counts must be monitored for all ICVs/CCVs. ISTDs must recover within 50-150% of the average of all calibration points. For Method 608.3, the ISTD should be verified within 50-200% of the mid-level ICAL standard.

The internal standard responses and retention times in the check calibration standard must be evaluated immediately after or during data acquisition. If the retention time for any internal standard changes by more than 30 seconds from the last calibration verification, the chromatographic system must be inspected for malfunctions and corrections must be made, as required.

Internal standards must be monitored for each sample. For Method 8081; ISTDs in samples must meet the $\pm 50\%$ criteria when compared to the average ISTDs of the current calibration. For Method 608.3, the ISTD should be verified within 50-200% of the daily verification standard.

- 10.13 Reporting Limit Verification - The reporting limit verification standard is injected as needed and must recover within $\pm 50\%$, except as noted. This standard may also be referred to as the MRL on the instrument

STATE NOTE: For all samples analyzed from Minnesota, the reporting limit must be verified at least monthly, with each new initial calibration, or when there has been significant change to the instrument (column replacement, cleaning source, etc.) whichever is more frequent. The reporting limit verification can be performed by either re-injecting the low standard or by re-processing the low standard that was analyzed in the calibration curve. The reporting limit verification (RLV) must recovery within $\pm 40\%$ of the expected concentration. If this criterion is not met, the RLV may be re-analyzed once, instrument maintenance can be performed, a higher concentration standard can be injected, or a new calibration curve must be generated. If a higher concentration standard is utilized, the reporting limit must be raised to the higher level verified.

- 10.14 QC Acceptance Criteria – See the LIMS (criteria subject to change based on historical data)
- 10.15 Any sample analyte responses that are beyond the linear range of the calibration curve must be diluted and re-analyzed.
- 10.16 Manual Integration – All manual integrations must comply with the requirements found in SOP #030215, *Manual Integration Procedure*. Before and after integrations must be available for review by the secondary data reviewer.
- 10.17 Analyte Retention Time – Establish retention time windows for each compound in the calibration mix. To determine the retention time window, make three injections of a

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standard containing the compounds of interest over a 72 hour period. The retention time window shall be defined as plus or minus 3 standard deviations of the absolute retention time for each standard. The typical estimated retention times are set at +/- 0.05 minutes. For multi-component standards such as PCB's, the analyst should use pattern recognition because of retention time shifts. Retention time windows should be recalculated whenever a new column is installed or major instrument maintenance is performed.

Routine maintenance requires analysts to assess retention time windows daily. The calibration standard is used to set the retention time window for the analytical batch. Default windows of ± 0.05 minutes are set for each compound in the calibration standard. Where compounds closely elute, the analyst must determine if the default values are appropriate. For multi-component standards such as Chlordane and Toxaphene, the analyst uses pattern recognition due to possible retention time shifts. When internal standard calibration is used, the retention times of the internal standards and the area responses of the internal standards must be checked for each analysis. Retention time shifts of >30 sec from the retention of the most recent calibration standard and/or changes in the internal standard area of more than 50-150% are cause for concern and must be investigated.

- 10.18 Second Source Calibration Verification (SSCV) - A second source calibration verification standard (SSCV) is analyzed after each calibration and must meet criteria of $\pm 20\%$ of the expected concentration for each analyte for 8081. Method 608.3 utilizes CCV limits for evaluation.
- 10.19 For sample analyzed per the requirements of Method 8000D, the LLOQ (see Section 1.8.2) must be verified at least annually, and whenever significant changes are made to the preparation and/or analytical procedure, to demonstrate quantitation capability at lower analyte concentration levels
- 10.19.1 The LLOQ verification (to be performed after the initial calibration) is prepared by spiking a clean control material with the analyte(s) of interest at 0.5-2 times the LLOQ concentration level(s).
- 10.19.2 The LLOQ check is carried through the same preparation and analytical procedures as environmental samples and other QC samples.
- 10.19.3 It is recommended to analyze the LLOQ verification on every instrument where data is reported; however, at a minimum, the lab must rotate the verification among similar analytical instruments such that all are included within 3 years.
- 10.19.4 Recovery of target analytes in the LLOQ verification must be within established in-house limits or within other such project-specific acceptance limits to demonstrate acceptable method performance at the LLOQ. Until the laboratory has sufficient data to determine acceptance limits, the LCS criteria $\pm 20\%$ (i.e., lower limit minus 20% and upper limit plus 20%) may be used for the LLOQ acceptance criteria.

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10.20 For corrective actions, see section 11.0.

11.0 DATA VALIDATION AND CORRECTIVE ACTION

- 11.1 All data must undergo a primary review by the analyst. The analyst must check the performance of the initial calibration, mid-point check standard, and continuing calibrations to ensure that they meet the criteria of the method. The analyst must review any sample that has quantifiable compounds and make sure that they have been confirmed. The analyst must also verify that reported results are derived from quantitation between the RL and the highest standard of the initial calibration curve. All calculations must be checked (any dilutions, %solids, etc.). Data must be checked for the presence or absence of appropriate flags. Comments must be noted when data is flagged.
- 11.2 All data must undergo a second analyst review. The analyst checking the data must check the performance of the initial calibration, mid-point check standard, and continuing calibrations to ensure that they meet the criteria of the method.
- 11.2.1 The analyst should must review any sample that has quantifiable compounds and make sure that they have been confirmed.
- 11.2.2 All calculations must be checked.
- 11.2.3 All surrogate recoveries must be checked to ensure that they are within QC acceptance criteria or that corrective action has occurred.
- 11.2.4 Blanks must be free of all interfering peaks.
- 11.2.5 Quality control criteria must be checked for the LCS, LCSD, MS, and MSD.
- 11.2.6 Data must be checked to determine the need for appropriate flags. Comments are noted when results are flagged.
- 11.2.7 The reviewer must verify all reported results are derived from analytical results that are above the reporting limit and below the highest standard of the initial calibration curve.
- 11.2.8 All manual integrations must be available for review per SOP #030215, *Manual Integration Procedure*.
- 11.2.9 All multipliers/dilutions must be verified on the quant report and must agree with the information provided on the injection log.
- 11.2.10 Retention times of the samples must be compared to that of the calibration standard. Random spot checking of 10% of the data should be sufficient.
- 11.2.11 Verify linear regression by reviewing the calibration curve printout.
- 11.2.12 See SOP #030201, *Data Handling and Reporting* and SOP #030227, *Data Review*.

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- 11.3 Initial Calibration – If the calibration curve fit does not meet method requirements when using %RSD, then linear regression can be used when the minimum correlation coefficient is achieved. The deletion of the highest point is acceptable when necessary, with the analyst noting that the high end of the calibration has been lowered. The deletion of the lowest calibration point is acceptable, when necessary, provided that the analyst notes the deletion on the injection log and raises the reporting limit, if necessary, for that compound. The method blank is also not included as a point in the calibration curve for this method. If none of the mentioned factors produce an acceptable calibration curve, then the entire calibration curve must be re-analyzed. Instrument maintenance (cleaning/repairing detector, column clipping/replacement, injector port cleaning/changing liner, etc.) may be required prior to calibration standard reanalysis.

Method 8000D: To determine calibration function acceptability, refit the initial calibration data back to the calibration model and calculate %Error (see Section 9.4). Percent error between the calculated and expected amounts of an analyte must be $\leq 30\%$ for all standards. For some data uses, $\leq 50\%$ may be acceptable for the lowest calibration point.

- 11..4 Method Blank - If the method blank shows any detectable amount greater than the RL, the laboratory performance is assumed to be out of control and the problem must be corrected. Corrective actions include: re-analysis once or re-pour fresh extract if available.

General guidelines for qualifying sample results with regard to method blank quality are as follows:

- If the method blank concentration is less than the MDL and sample results are greater than the RL, then no qualification is required.
- No qualification is necessary when an analyte is detected in the method blank but not in the associated samples.
- If the concentration in a sample is more than ten times the concentration in the method blank, then no qualification is required.
- If the method blank concentration is greater than the MDL but less than the RL and sample results are greater than the MDL, then qualify associated sample results to indicate that analyte was detected in the method blank.
- If the method blank concentration is greater than the RL, further corrective action and qualification is required. An analyst should consult their supervisor for further instruction.

Method 8000D: When samples that are extracted together are analyzed on separate instruments or in separate analytical shifts, the method blank associated with those samples (e.g., extracted with the samples) must be analyzed on at least one of those instruments. A solvent blank must be analyzed on all other instruments on which the set of samples was analyzed to demonstrate the instrument is not contributing contaminants to the samples. At least one method blank or instrument blank must be analyzed on every instrument after calibration standard(s) and prior to the analysis of any samples.

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When sample extracts are subjected to cleanup procedures, the associated method blank must also be subjected to the same cleanup procedures.

Results of the method blank should be less than the LLOQ for the analyte or less than the level of acceptable blank contamination specified in the approved QAPP or other appropriate systematic planning document. Blanks are generally considered to be acceptable if target analyte concentrations are less than one-half the LLOQ or are less than project-specific requirements.

When new reagents or chemicals are received, the lab should monitor the blanks associated with samples for any signs of contamination. It is not necessary to test every new batch of reagents or chemicals prior to sample preparation if the source shows no prior problems. However, if reagents are changed during a preparation batch, separate blanks need to be prepared for each set of reagents.

11.5 Matrix Spike (MS) And Matrix Spike Duplicate (MSD) - If the spike and spike duplicate do not meet current acceptance criteria, the sample must be flagged as possible matrix interference.

11.5.1 Spike failure that result in the use of a "J" flag followed by the appropriate number, which further explains the failure concerning high or low bias

11.5.2 Method 8000D: If, as in compliance monitoring, the concentration of a specific analyte in the sample is being checked against a regulatory concentration limit or action level, the spike should be at or below the limit, or 1 - 5 times the background concentration (if historical data are available), whichever concentration is higher. If historical data are not available, a background sample of the same matrix from the site may be submitted for matrix spiking purposes to ensure that high concentrations of target analytes and/or interferences will not prevent calculation of recoveries. If the background sample concentration is very low or non-detect, a spike of greater than 5 times the background concentration is still acceptable. To assess data precision with duplicate analyses, it is preferable to use a low concentration field sample to prepare a MS/MSD for organic analyses. This spiking procedure will be performed when project-specific instructions are received from the client.

If the concentration of a specific analyte in a sample is not being checked against a limit specific to that analyte, then the analyst may spike the matrix spike or MS/MSD sample(s) at the same concentration as the reference sample at 20 times the estimated LLOQ in the matrix of interest, or at a concentration near the middle of the calibration range. It is suggested that a background sample of the same matrix from the site be submitted as a sample for matrix spiking purposes. NOTE: Preparing the spiking solution from the same source as the calibration standards helps minimize additional variability due to differences between sources. Typically, spiking concentrations are near the middle of the calibration range.

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To develop precision and bias data for the spiked compounds, the analyst has two choices: analyze the original sample, and an MS/MSD pair; or analyze the original sample, a duplicate sample, and one spiked sample. If samples are not expected to contain the target analytes of concern, then the laboratory may use a MS/MSD pair. If samples are expected to contain the target analytes of concern, then the laboratory may use one matrix spike and a duplicate analysis of an unspiked field sample as an alternative to the MS/MSD pair.

The laboratory should use 70 - 130% as interim acceptance criteria for recoveries of spiked analytes, until in-house LCS limits are developed. Where in-house limits have been developed for matrix spike percent recoveries, the LCS results should be similar to or tighter than those limits, as the LCS is prepared in a clean matrix.

- 11.6 Laboratory Control Sample/Laboratory Control Sample Duplicate (LCS/LCSD) - If the control does not perform within current acceptance criteria, the laboratory performance is assumed to be out of control and the problem must be corrected. Corrective action can include re-analysis, if instrument malfunction is suspected, or re-preparation and re-analysis of the entire batch, if the failure is suspected as either extraction or sample related.

STATE NOTE: For all 8081 samples analyzed from South Carolina, the LCS/LCSD recovery must be evaluated within the limits given in Section 10.7 for both soil and water matrices with an RPD of <20%.

- 11.7 Initial/Calibration Check Standard (ICV/CCV) - When the initial or continuing calibration verification is out of the acceptance criteria and analysis of a second consecutive (immediate) calibration verification fails to produce results within acceptance criteria, corrective actions shall be performed. The laboratory shall demonstrate acceptable performance after the final round of corrective action with two consecutive calibration verifications or a new initial instrument calibration shall be performed.
- 11.8 Endrin and DDT breakdown - Any breakdown check that exceeds the acceptance criteria (section 10.9) must be followed by corrective action. The breakdown check can be re-injected once. If the failure persists, additional corrective actions include: instrument maintenance, injection port cleaning/deactivation, and column clipping. A passing breakdown must be achieved prior to the analysis of any field samples and every 12hrs.
- 11.9 Confirmation - If the relative percent difference of the results exceeds 40% and one result is significantly higher (e.g., >40%), check the chromatograms to see if an obviously overlapping peak is causing an erroneously high result. If no overlapping peaks are noted, examine the baseline parameters established by the instrument data system (or operator) during peak integration. If re-integration is necessary, manual integration procedures must be followed and documented by printing a before and after shot of the chromatograms. When confirmation is not within the 40% criteria (50% for Method 608.3), analyst judgment weighs heavily in the interpretation of the data and the appropriate action. A conservative approach is the preferred course of action to protect the environment and public health and unless obvious interferent is present, the higher

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result is reported. Both columns must be calibrated and both columns must meet acceptance criteria. For a particular sample, if criteria are met on only one column and target analytes are not detected, then data may be reported; otherwise, the sample must be re-analyzed or qualified.

- 11.10 Surrogates - If the recovery is not within current acceptance criteria, confirm that there are no errors in the calculations, surrogate solutions and standards. Check the instrument performance. Examine the chromatograms for interfering peaks and integrated areas. Re-calculate the data and/or re-analyze the extract if any of the above checks reveal a problem. Re-extract and re-analyze the sample if none of the above is a problem or flag the data "J1" (surrogate high) or "J2" (surrogate low).
- 11.11 Internal Standards - If any internal standard response is beyond the acceptable recovery in the ICV/CCV, corrective action is required. Corrective action can take to form of checking the original calculations to ensure accuracy, re-analysis of the CCV to verify initial results, instrument maintenance (i.e. column clipping or changing, inlet liner cleaning/replacement, etc.) or re-calibration.

If the retention time for any internal standard changes by more than 30 seconds from the last calibration verification, the chromatographic system must be inspected for malfunctions and corrections must be made, as required. When corrections are made, re-analysis of the CCV or a complete re-calibration is necessary, depending on the impact of the correction on the analytical system.

Internal standards in the field samples must be monitored. If ISTD recovery does not meet the acceptance criteria, correction action is required. Possible corrective actions include: if instrument malfunction is suspected, or re-preparation and re-analysis, if the failure is suspected as either extraction or sample related. If the sample has an obvious matrix interferent and the internal standard recovery is greater than 150%, the sample can be diluted (if acceptable reporting limits can be achieved) to minimize the interference or the sample must be re-extracted and re-analyzed using an external calibration model.

- 11.12 Holding Time - If the samples are out of holding time, the data can be flagged with a "Q/T8" to show that the sample has exceeded the holding time. If this happens, the Technical Service Representative (TSR) must be notified of the situation so that the client can be contacted. It may be necessary to obtain a new sample from the client. See section 4.5 for additional information.
- 11.13 Marginal Exceedances - The laboratory control sample, laboratory control sample duplicate, matrix spike and matrix spike duplicate recoveries must be evaluated against the acceptance criteria listed in this procedure. The LCS/LCSD and MS/MSD are spiked with the same list of compounds for which the instrument is calibrated. Due to the large number of compounds analyzed using these methods, it is statistically likely that accuracy and precision failures will occur.

LCS or LCSD samples that do not pass the acceptable QC acceptance criteria must be re-analyzed. LCS/LCSD failures must meet the marginal exceedance criteria below.

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Allowable marginal exceedance outliers are based on the number of compounds being analyzed and must be random events.

Upper and lower marginal exceedance (ME) limits are established by ± 4 times the standard deviation of historical accuracy data.

Number of allowable marginal exceedances:

- 90+ analytes, 5 analytes allowed in the ME limit
- 71 – 90 analytes, 4 analytes allowed in the ME limit.
- 51 – 70 analytes, 3 analytes allowed in the ME limit.
- 31 – 50 analytes, 2 analytes allowed in the ME limit.
- 11 – 30 analytes, 1 analyte allowed in the ME limit.
- < 11 analytes, no analyte allowed in the ME limit.

- 11.14 SSCV – If the acceptance criteria are not met, a new calibration curve or new SSCV must be prepared and analyzed, depending on the source of the discrepancy. An SSCV must pass the acceptance criteria prior to the analysis of field samples.
- 11.15 Data that does not meet acceptable QC criteria may be acceptable for use in certain circumstances.
- 11.15.1 If a method blank contains an amount of target analyte, but all samples are non-detected, the data may be reported with a “B3” flag. If a method blank contains an amount of target analyte, but the samples contain analyte at a level that is 10 times the level present in the method blanks, the data may be reported with a “B” flag.
- 11.15.2 If the MS/MSD fails in an initial analysis and again upon re-analysis, the data is released with an appropriate qualifier as the failure is accepted as matrix related.
- 11.15.3 If a calibration verification standard is above the acceptable QC criteria and all samples being reported are below the reporting limit, the data is acceptable based on a high calibration bias with undetectable levels in the field samples. Any positive samples require re-analysis.
- 11.15.4 If the surrogate exhibits high recovery in the field samples and the target analytes in the field samples are below the reporting limit, the data may be released with a J1 qualifier indicating the high bias. If the QC samples (LCS, LCSD, MS, MSD) exhibit a high bias in the surrogate and the field samples are below the reporting limit for the target analyte, the data may be released with a J1 qualifier. Any failure of target analyte quantitation above 200% is suspect and should be re-extracted for confirmation if matrix impact is not apparent, or sufficient amount of sample and holding time remains.
- 11.15.5 If the target analyte spiked in the quality control samples (LCS, LCSD, MS, MSD) exhibits high recovery and the target analytes in the field samples are below the reporting limit, the data may be released with a J4 qualifier indicating the high bias.

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11.15.6 If the target analyte spiked into the QC pair (LCS/LCSD, MS/MSD) exhibit acceptable recoveries, but high calculated RPD values for precision, the target analytes in the field sample are flagged with a J3 for the precision beyond acceptable quality control limits.

11.15.7 Sample results can be qualified and possible bias is narrated per the SOP #030201, *Data Handling*.

11.15.7.1 For samples analyzed per the requirements of Method 8000D, reported concentrations of target analytes between the MDL and the LLOQ must be qualified as estimated.

12.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

12.1 The EPA requires that laboratory waste management practice to be conducted consistent with all applicable federal and state laws and regulations. Excess reagents, samples and method process wastes must be characterized and disposed of in an acceptable manner. See *Waste Management Plan*.

12.2 See SOP #030302, *Environmental Sustainability & Pollution Prevention*.

13.0 METHOD MODIFICATIONS/CLARIFICATIONS

13.1 Modifications to this method are noted in the body of the text as notes. Compliance analyses performed in conjunction with specific state and/or method requirements must be performed as noted.

13.2 Adjustments to the concentrations of standards/spiking solutions, standards providers, and quality control are subject to change to better meet client/project/regulatory needs or to improve laboratory method performance.

13.3 The reduction of the size of the field sample used in this procedure is performed in accordance with section 7.1 of the published EPA 3510C method. The reduction in volume extracted along with either sufficient sensitivity of detection and/or large volume injection technique (>5uL) on the GC allows for acceptable detection limits in line with those obtained using a 1L extraction. Complete method validation is performed for each method prior to utilizing the reduced volume extraction. This validation is maintained by the Regulatory Affairs Department and is regularly verified using LCS/LCSD, MDL studies and DOCs.

STATE NOTE: Pace National is not currently certified to perform the reduced volume extraction method using EPA 3510C in conjunction with South Carolina samples.

13.4 Method 608.3 allowed method modifications:

13.4.1 If the underlying chemistry and determinative technique in a modified method are essentially the same as an approved Part 136 method, then the modified method is an equivalent and acceptable alternative to the approved method provided the requirements of this section are met.

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- 13.4.2 Those who develop or use a modification to an approved (Part 136) method must document that the performance of the modified method, in the matrix to which the modified method will be applied, is equivalent to the performance of the approved method. If such a demonstration cannot be made and documented, then the modified method is not an acceptable alternative to the approved method.
- 13.4.3 Supporting documentation must, if applicable, include the routine initial demonstration of capability and ongoing QC including determination of precision and accuracy, detection limits, and matrix spike recoveries.
- 13.4.3.1 Initial demonstration of capability typically includes analysis of four replicates of a mid-level standard and a method detection limit study.
- 13.4.3.2 Ongoing quality control typically includes method blanks, mid-level laboratory control samples, and matrix spikes (QC is as specified in the method).
- 13.4.3.3 The method is considered equivalent if the quality control requirements in the reference method are achieved.
- 13.4.3.3.1 Where the laboratory is using a vendor-supplied method, it is the QC criteria in the reference method, not the vendor's method, that must be met to show equivalency.
- 13.4.3.3.2 Where a sample preparation step is required (i.e., digestion, distillation), QC tests are to be run using standards treated in the same way as the samples.
- 13.4.3.4 The method user's Standard Operating Procedure (SOP) must clearly document the modifications made to the reference method.
- 13.4.4 If the method user is uncertain whether a method modification is allowed, the Regional ATP Coordinator or Director should be contacted for approval prior to implementing the modification
- 13.4.5 The method user should also complete necessary performance checks to verify that acceptable performance is achieved with the method modification prior to analyses of compliance samples.
- 13.4.6 The modified method must meet or exceed performance of the approved method(s) for the analyte(s) of interest, as documented by meeting the initial and ongoing quality control requirements in the method.
- 13.4.7 The permittee must notify their permitting authority of the intent to use a modified method. Such notification should be of the form "Method xxx has been modified within the flexibility allowed in 40 CFR 136.6." The permittee may indicate the specific paragraph of § 136.6 allowing the method modification. Specific details of the modification need not be provided, but must be documented in the

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Standard Operating Procedure (SOP) and maintained by the analytical laboratory that performs the analysis.

14.0 REFERENCES

- 14.1 *Organochlorine Pesticides by Gas Chromatography*, SW-846 Method 8081A, Revision 1, December 1996.
- 14.2 *Organochlorine Pesticides by Gas Chromatography*, SW-846 Method 8081B, Revision 2, February 2007.
- 14.3 *Determinative Chromatographic Separations*, SW-846 Method 8000B, Revision 2, December 1996.
- 14.4 *Determinative Chromatographic Separations*, SW-846 Method 8000C, Revision 3, March 2003.
- 14.5 *Determinative Chromatographic Separations*, SW-846 Method 8000D, Revision 4, July 2014.
- 14.6 *Organochlorine Pesticides and PCBs*, EPA Method 608, 40 CFR Part 136, Appendix A.
- 14.7 *Organochlorine Pesticides by Liquid-Liquid Extraction Gas Chromatographic Method II*, SM 6630C, 20th edition.
- 14.8 *Organochlorine Pesticides by Liquid-Liquid Extraction Gas Chromatographic Method II*, SM 6630C, 2000.
- 14.9 *Organochlorine Pesticides by Liquid-Liquid Extraction Gas Chromatographic Method II*, SM 6630C, 2007.
- 14.10 *Organochlorine Pesticides and PCBs by GC/HSD*, EPA Method 608.3, Federal Register, Volume 82, Number 165, August 28, 2017.
- 14.11 EPA Method 608 ATP 3M0222, Federal Register, Volume 60, Number 148, August 2, 1995.
- 14.12 40 Code of Federal Regulations §136.6(b)(4)(j).

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Attachment I: Revision History

Current Version:

Version	Date	Description of Revisions
21	7/17/2018	Technical and quality review and update. Replaced logo and watermark. Added Method 608.3 requirements. Revised Sections 1.0, 1.1, 1.2, 1.2.1, 2.1, 3.1, 4.4, 5.3, 5.4, 5.5, 7.13, 8.2.2, 8.5, 8.6, 8.7, 9.6, 10.6, 10.7, 10.9, 10.10, 10.12, 10.16, 10.17, 10.18, 11.2.12, 11.5, 11.6, 11.9, 11.10, 11.12, 11.13, 11.15.1, 11.15.7, 12.1, and 13.1. Added Sections 4.6, 4.7, 5.6, 7.14, 7.15, 8.2.1.1, 8.2.5, 8.7.1, 9.7, 10.1.1, 10.4.1, 10.6.1, 10.7.1, 10.8.1, 13.4, 14.10, 14.11, and 14.12.

Superseded Versions:

This document supersedes the following:

Version	Date	Description of Revisions
0	8/23/94	Origination
1	7/12/95	
2	2/1/99	
3	2/11/00	
4	8/21/00	
5	10/16/01	
6	9/19/03	
7	12/14/04	
8	2/7/05	
9	2/17/09	Technical and Quality Review and update. Update to include internal standard use. Inclusion of sections 8.2.7, 9.3, State notes; corrective actions in sections 11.3 & 11.5. Ohio VAP approved 2/17/09.
10	7/28/11	Technical and Quality Review and update. Revised sections 1.1, 2.1 through 2.3, 5.2, 6.1, 7.1, 7.4, 7.5, 7.7, 7.9 through 7.11, 8.4, 8.6, 8.8, 9.4, 9.7 through 9.9, 11.8, and Attachment II; Added sections 2.20 through 2.23, 10.12, 10.14, 10.15, 11.12, 12.1, 13.2, and state notes following sections 8.4, 10.12, 10.13, 11.12.
11	2/22/12	Technical and Quality Review and update. Revised sections 2.1, 4.3, 4.4, 6.1, 7.8, 7.11, 7.12, 7.13, 8.1, 8.2, 8.4, 8.6, 8.7, and 14.6; Added sections 1.2.1, 2.25, 2.26, and 13.3.
12	6/12/12	Technical and Quality Review and update. Revised sections 1.1, 1.2, 2.1, 7.12, 7.13, 8.2, 8.5, 8.6, 8.8, 9.4, 9.7, 9.8, 9.9, 10.4, 10.7, 10.10, 11.1, 11.2, 11.3, 11.5, 11.9, and 11.12; Added sections 4.5, 8.9, 9.10, 10.12, 10.14 through 10.17 and 11.14 through 11.15.

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Version	Date	Description of Revisions
13	12/19/13	Technical and Quality Review and update. Revised Attachment II and sections 6.1, 7.1, 7.8, 7.13, 8.1, 8.2.3, 8.4, 8.7, 10.3, 10.4, 10.5, 10.7, 10.11, 10.12, 10.16, and 11.15.4; Added state note in section 1.0 and sections 14.7 and 14.8.
14	12/8/14	Technical and Quality Review and update. Revised Attachment II and sections 2.1, 7.8, 7.11 7.13, and 8.8. Removed Section 8.5
15	11/3/2015	Technical and quality review and update. Header and signature block reformatting. Revised Sections 1.2.1, 7.11, 7.12, 7.13, 8.2, 8.2.1, 8.4, 8.5, 8.6, 8.7, 10.3, 10.5, 10.5, 10.6, 10.7, 10.8, 10.11, 10.13, 10.14, 10.17, 11.2.8, 11.4, 11.5, 11.6, 11.10, 11.12, 10.15.2, 10.15.3, 10.15.4, 13.3, and Attachment II. Removed Section 8.5 and 11.2.8.
16	8/16/2016	Technical and quality review and update. Revised header and Sections 1.0, 1.2.1, 2.2, 4.5, 7.1, 8.1, 8.6, 10.7, 11.7, 11.13, 11.14, 12.2, and Attachment II Table 2. Deleted Sections 2.3 through 2.26, 9.2 through 9.4, 9.8, and 9.9.
17	10/24/16	Technical and quality review and update to satisfy the requirements of SC DHEC (see correspondence dated 6/24/16). Revised Sections 10.14, 11.2.7, 11.3, 11.4, 14.1, 14.3, 14.6, and 14.7. Added Sections 1.2.2, 2.2, 9.4, 10.19 and all subsections, 11.5.2, 11.15.7.1, 14.4, 14.5, 14.8, and 14.9. Deleted Sections 14.5 and 14.6.
18	6/19/2017	Technical and quality review and update. Revised Sections 1.2, 3.1, 7.13, 9.4, 9.5, 9.6, 10.6, and 10.7.
19	7/10/2017	Technical and quality review and update. Revised Sections 2.1, 2.3, 2.4, 9.4, 13.3
20	11/29/2017	Update in response to A2LA audit finding CAR2872. Revised Sections 8.7, 10.3, 10.8, 11.9, and Attachment II Table 5.

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Attachment II: DOD Requirements

1.0 Equipment/Instrument Maintenance

Instrument maintenance must be performed routinely to optimize instrument performance and improve chromatography. Commonly performed maintenance includes cleaning/repairing detector, column clipping/replacement, injector port cleaning/changing liner, etc. A new calibration curve must be analyzed following any major maintenance performed on the analytical system.

2.0 Computer Hardware and Software

Software name and version: HP Chemstation G1701BA Version C.00.00 or equivalent

3.0 Troubleshooting

Table 1. GC Troubleshooting Guide		
Problem	Cause	Treatment
No Peaks	Syringe clogged	Clean or replace syringe
	Detector/Software/Computer failure	Check cables. Restart computer.
	Column Leaks	Use new ferrules.
	Broken Column	If at ends, clip column. If in the middle or multiple sites, replace column.
Peaks too Small	Split too high	Reduce split
	Column connection leaks	Check column installation. Search for leaks. Replace ferrules.
	Injector temperature too low	Check temperature program. Increase injector temperature.
	Dirty ECD	Clean ECD.
Retention Times Change	Gas flow too low or too high	Replace septum. Check gas regulator.
	Oven temperature unstable	Check temperature program. Check temperature with external thermometer.
	Column blocked	Compare flow at column entrance to outlet. Replace column.
Constantly Rising Baseline	Leak at column entrance or injection septum.	Check column installation; search for leaks; replace ferrules.
	Injector contaminated.	Make a run at lower injector temperature; if the baseline improves, replace liner, use low bleed or high temperature septa.
	Column contaminated.	Cut two turns from column entrance; rinse column with solvent (only chemically bonded phases); otherwise replace column or use guard column.
	Detector contaminated.	Clean detector.
	Increase of temperature too fast.	Decrease temperature gradient and end temperature.

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Table 1. GC Troubleshooting Guide

Problem	Cause	Treatment
	Poor gas quality.	Use gas grades recommended for GC; for longer supply lines from gas source to GC use gas purification cartridges directly connected to the GC.
Increasing Baseline at High Temperatures	Decomposition of the stationary phase.	Check for leaks; matrix check for compatibility with the column.
	Column contaminated.	Cut two turns from column entrance; rinse column with solvent (only chemically bonded phases); otherwise replace column or use guard column.
	Increase of temperature too fast / end temperature too high.	Decrease temperature gradient and end temperature.
	Column not properly conditioned.	Condition column according to manufacturers' instructions (while column is not connected to the detector).
	Detector contaminated	Clean detector according to manufacturers' instructions.
	Poor gas quality.	Use gas grades recommended for GC; for longer supply lines from gas source to GC use gas purification cartridges directly connected to the GC.
Plateaus at Certain Temperatures	Steps in temperature program too drastic.	Avoid very short and strong heating periods.
Fronting	Column overload.	Decrease injection volume; dilute sample.
	Sample vaporizes too slowly, not evenly or condenses.	Increase injector temperature (consider max. temperature limits of the column).
	Analytes coelute.	Change temperature program or use column with different selectivity.
	Sample decomposes.	Check temperature program, oven temperature (external thermometer); if analytes are not temperature-stable, reduce injector temperature; replace liner.
	Column absorbs or decomposes analytes.	Check capillary ends; check intact deactivation using the test mixture; for poor results shorten both column ends by about 10 cm; or replace column; if column test does not show any defects: a) use a column with thicker film b) use phase with better deactivation c) use column with special selectivity.

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Table 1. GC Troubleshooting Guide

Problem	Cause	Treatment
Tailing	Sample vaporizes too slowly, not evenly or condenses.	Increase injector temperature (consider max. temperature limits of the column).
	System leaks.	Check column installation; search for leaks; replace ferrules.
	Analytes coelute.	Change temperature program or use column with different selectivity.
	Sample decomposes.	Check temperature program, oven temperature (external thermometer); if analytes are not temperature-stable, reduce injector temperature; replace liner by a deactivated one.
	Column absorbs or decomposes analytes.	Check capillary ends; check intact deactivation using the test mixture; for poor results shorten both column ends by about 10 cm; or replace column; if column test does not show any defects: a) use a column with thicker film b) use phase with better deactivation c) use column with special selectivity.
	Split rate too low.	Increase split rate.
	Column overload.	Decrease injection volume; dilute sample.
Split Peaks	Solvent and column not compatible.	Change solvent or use guard column.
	Solvent mixtures with large differences in boiling point and polarity.	Use just one solvent.
	Sample decomposes.	Check temperature program, oven temperature (external thermometer); if analytes are not temperature-stable, reduce injector temperature; replace liner by a deactivated one.
	Analytes coelute.	Modify temperature program or use longer column; possibly change column polarity.
	Detector overload.	Inject less; control make-up flow.

4.0 Other Requirements

- 4.1 All hardcopy laboratory notebooks must be reviewed by the Supervisor, or their designee, on a monthly basis.
- 4.2 If not self-explanatory (e.g., a typo or transposed number), corrections to technical and quality records shall also include a justification for the change.

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- 4.3 A person performing a manual integration must sign and date each manually integrated chromatogram and record the rationale for performing manual integration. Electronic signatures are acceptable.
- 4.4 The results of calibration and verification of support equipment must be within the specifications required of the application for which this equipment is used or the equipment must be removed from service until repaired. Calibration and verification records, including those of established correction factors, must be maintained. In the absence of method-specific requirements, the minimum requirements are as follows:

Table 2. Support Equipment Checks		
Performance Check	Frequency	Acceptance Criteria
Balance calibration check [Using two standard weights that bracket the expected mass]	Daily prior to use	Top-loading balance: $\pm 2\%$ or $\pm 0.02\text{g}$, whichever is greater Analytical balance: $\pm 0.1\%$ or $\pm 0.5\text{mg}$, whichever is greater
Verification of standard mass [Using weights traceable to the International System of Units (SI) through a NMI]	Every 5 years	Certificate of Calibration from ISO/IEC 17025 accredited calibration laboratory
Monitoring of refrigerator/freezer temperatures	Daily (i.e. 7 days per week) [use MIN/MAX thermometers or data loggers equipped with notification of out of control event capabilities if personnel not available to record daily]	Refrigerators: 0°C to 6°C Freezers: $\leq -10^{\circ}\text{C}$
Thermometer verification check [Using a thermometer traceable to the SI through an NMI] [Performed at two temperatures that bracket the target temperature(s). Assume linearity between the two bracketing temperatures.] [If only a single temperature is used, at the temperature of use]	Liquid in glass: Before first use and annually Electronic: Before first use and quarterly	Apply correction factors or replace thermometer
Volumetric labware	Class B: By lot before first use Class A and B: Upon evidence of deterioration	Bias: Mean within $\pm 2\%$ of nominal volume Precision: $\text{RSD} \leq 1\%$ of nominal volume (based on 10 replicate measurements)
Non-volumetric labware [Applicable only when used for measuring initial sample volume and final extract/ digestates volume]	By lot before first use or upon evidence of deterioration	Bias: Mean within $\pm 3\%$ of nominal volume Precision: $\text{RSD} \leq 3\%$ of nominal volume (based on 10 replicate measurements)

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Table 2. Support Equipment Checks

Performance Check	Frequency	Acceptance Criteria
Mechanical volumetric pipette	Quarterly	Bias: Mean within $\pm 2\%$ of nominal volume Precision: RSD $\leq 1\%$ of nominal volume (based on minimum of 3 replicate measurements) [Note: for variable volume pipettes, the nominal volume is the volume of use]
Glass microliter syringe	Upon receipt and upon evidence of deterioration	General Certificate of Bias & Precision upon receipt Replace if deterioration is evident
Drying oven temperature check	Daily prior to and after use	Within $\pm 5\%$ of set temperature
Water purification system	Daily prior to use	See method blank criteria given in Section 4.20 of this addendum

- 4.5 The expiration date of the prepared standard shall not exceed the expiration date of the primary standard. All containers must bear a preparation date.
- 4.6 To avoid preparing non-representative samples, the laboratory shall not “target” within a relatively small mass range (e.g., $1.00 \pm 0.01\text{g}$) because such targeting will produce non-representative subsamples if the sample has high heterogeneity. The laboratory shall not manipulate the sample material so the sample aliquot weighs exactly $1.00\text{g} \pm 0.01\text{g}$, as an example.
- 4.7 In the absence of project-specific requirements, the minimum standard data qualifiers to be used are:
- U Analyte was not detected and is reported as less than the LOD or as defined by the customer. The LOD has been adjusted for any dilution or concentration of the sample.
 - J The reported result is an estimated value (e.g., matrix interference was observed or the analyte was detected at a concentration outside the quantitation range).
 - B Blank contamination. The recorded result is associated with a contaminated blank.
 - N Non-target analyte. The analyte is a tentatively identified compound using mass spectrometry or any non-customer requested compounds that are tentatively identified.
 - Q One or more quality control criteria failed (e.g., LCS recovery, surrogate spike recovery, or CCV recovery).

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Additional data qualifiers may be used, or different letters or symbols to denote the qualifiers listed above, as long as they are appropriately defined and their use is consistent with project-specific requirements (e.g., QSM 5.0, the contract, and project-planning documents).

- 4.8 If the time of the sample collection is not provided, assume the most conservative time of day. For the purpose of batch processing, the start and stop dates and times of the batch preparation shall be recorded.
- 4.9 Each preparation method listed on the scope of accreditation must have quarterly LOD/LOQ verifications. However, not all possible combinations of preparation and cleanup techniques are required to have LOD/LOQ verifications. If LOD/LOQ verifications are not performed on all combinations, the laboratory must base the LOD/LOQ verifications on the worst case basis (preparation method with all applicable cleanup steps).
- 4.10 After each MDL determination, the laboratory must establish the LOD by spiking a quality system matrix at a concentration of at least 2 times but no greater than four times the MDL. This spike concentration establishes the LOD and the concentration at which the LOD shall be verified. It is specific to each suite of analyte, matrix, and method (including sample preparation). The following requirements apply to the initial LOD establishment and to the LOD verifications:
- The apparent signal to noise (S/N) ratio at the LOD must be at least three and the results must meet all method requirements for analyte identification (e.g., ion abundance, second column confirmation, or pattern recognition). For data systems that do not provide a measure of noise, the signal produced by the verification sample must produce a result that is at least three standard deviations greater than the mean method blank concentration. This is initially estimated based on a minimum of four method blank analyses and later established with a minimum of 20 method blank results.
 - If the LOD verification fails, then the laboratory must repeat the MDL determination and LOD verification or perform and pass two consecutive LOD verifications at a higher spike concentration and set the LOD at the higher concentration.
 - The laboratory shall maintain documentation for all MDL determinations and LOD verifications.
 - The DL and LOD must be reported for all analyte-matrix-methods suites, unless it is not applicable to the test or specifically excluded by project requirements.
- 4.11 The LOD shall be verified quarterly. In situations where methods are setup and used on an infrequent basis, the laboratory may choose to perform LOD verifications on a one per batch basis. All verification data will be in compliance, reported, and available for review.
- 4.12 For DoD, at a minimum, the LOQ shall be verified quarterly. In situations where methods are setup and used on an infrequent basis, the laboratory may choose to perform LOQ verifications on a one per batch basis.

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- 4.13 All initial instrument calibrations must be verified with a standard obtained from a second manufacturer prior to analyzing any samples. The use of a standard from a second lot obtained from the same manufacturer (independently prepared from different source materials) is acceptable for use as a second source standard. The concentration of the second source standard shall be at or near the midpoint of the calibration range. The acceptance criteria for the initial calibration verification must be at least as stringent as those for the continuing calibration verification.
- 4.14 Exclusion of calibration points without documented scientifically valid technical justification is not permitted.
- 4.15 The concentration of the CCV standard shall be greater than the low calibration standard and less than or equal to the midpoint of the calibration range.
- 4.16 All CCVs analyzed must be evaluated and reported. If a CCV fails, reanalysis or corrective actions must be taken.
- If a CCV fails, the laboratory can immediately analyze two additional consecutive CCVs (immediately is defined as starting a consecutive pair within one hour; no samples can be run between the failed CCV and the two additional CCVs). This approach allows for spurious failures of analytes to be reported without reanalysis of samples. Any corrective actions that change the dynamics of the system (e.g., clip column, clean injection port, run blanks) requires that all samples since the last acceptable CCV be reanalyzed.
 - Both of these CCVs must meet acceptance criteria in order for the samples to be reported without reanalysis.
 - If either of these two CCVs fail or if the laboratory cannot immediately analyze two CCVs, the associated samples cannot be reported and must be reanalyzed.
 - Corrective action(s) and recalibration must occur if the above scenario fails. All affected samples since the last acceptable CCV must be reanalyzed.
 - Flagging of data for a failed CCV is only appropriate when the affected samples cannot be reanalyzed. The laboratory must notify the client prior to reporting data associated with a failed CCV.
- 4.17 The results of all MS/MSDs must be evaluated using the same acceptance criteria used for the DoD LCS limits (see Addendum Tables 3 and 4) or project limits, if specified. If the specific analyte(s) are not available in the Addendum Tables 3 and 4, the laboratory shall use their LCS in-house limits (see the LIMS) as a means of evaluating MS/MSDs. The MS and MSD must be spiked with all reported analytes.
- 4.19 Surrogate spike results shall be compared with DoD LCS limits (see Addendum Tables 3 and 4) or acceptance criteria specified by the client. If these criteria are not available, the laboratory shall compare the results with its in-house statistically established LCS criteria (see the LIMS).
- 4.20 The method blank shall be considered to be contaminated if:

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- The concentration of any target analyte (chemical of concern) in the blank exceeds 1/2 the LOQ and is greater than 1/10th the amount measured in any associated sample, or 1/10th the regulatory limit, whichever is greater;
 - The concentration of any common laboratory contaminant in the blank exceeds the LOQ;
 - If a method blank is contaminated as described above, then the laboratory shall reprocess affected samples in a subsequent preparation batch, except when sample results are below the LOD. If insufficient sample volume remains for reprocessing, the results shall be reported with appropriate data qualifiers.
- 4.21 Sporadic Marginal Exceedances are not allowed for target analytes (chemicals of concern as identified by a project) without project-specific approval. Target analytes are considered those few analytes that are critical for the success of a project (such as risk drivers) where sporadic marginal exceedances cannot be allowed. Laboratories should consult with clients whenever long lists of analytes are requested for analysis to determine if marginal exceedances will not be allowed.
- 4.22 DoD considers the same analyte exceeding the LCS control limit two (2) out of three (3) consecutive LCS to be indicative of non-random behavior, which requires corrective action and reanalysis of the LCS.

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Table 3. LCS Control Limits – Method 8081 Solid Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
789-02-6	2,4'-DDT	110	100.1	11.9	64	136
53-19-0	2,4-DDD	111	102.8	9.2	75	130
3424-82-6	2,4-DDE	111	102.2	9.5	74	131
72-54-8	4,4'-DDD	2995	97.7	13.9	56	139
72-55-9	4,4'-DDE	2938	95.3	13	56	134
50-29-3	4,4'-DDT	2470	95.8	15.1	50	141
309-00-2	Aldrin	2985	90.5	15.2	45	136
319-84-6	alpha-BHC	3021	90.9	15.3	45	137
5103-71-9	alpha-Chlordane	2681	93.7	13.2	54	133
319-85-7	beta-BHC	2989	93.1	14.3	50	136
57-74-9	Chlordane	229	95.7	17.7	43	149
319-86-8	delta-BHC	2943	93.3	15.3	47	139
60-57-1	Dieldrin	2987	95.7	13.4	56	136
959-98-8	Endosulfan I	984	92.2	13.2	53	132
33213-65-9	Endosulfan II	2913	93.1	13.5	53	134
1031-07-8	Endosulfan sulfate	2954	95.9	13.5	55	136
72-20-8	Endrin	3076	98.1	13.9	57	140
7421-93-4	Endrin Aldehyde	3004	86	17	35	137
53494-70-5	Endrin Ketone	2953	95.5	13.5	55	136
58-89-9	gamma-BHC [Lindane]	3153	92.1	14.4	49	135
5103-74-2	gamma-Chlordane	2749	94.3	13.7	53	135
76-44-8	Heptachlor	3144	91.6	14.9	47	136
1024-57-3	Heptachlor Epoxide	3093	93.9	13.9	52	136
118-74-1	Hexachlorobenzene	319	91.6	11.4	57	126
72-43-5	Methoxychlor	3021	97.6	15.2	52	143
2385-85-5	Mirex	303	96.4	10.6	65	128
877-09-8	Tetrachloro-m-xylene	1482	85.3	14.6	42	129
8001-35-2	Toxaphene	532	86.7	17.9	33	141

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Table 4. LCS Control Limits – Method 8081 Water Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
72-54-8	4,4'-DDD	3112	99.6	14.4	56	143
72-55-9	4,4'-DDE	3062	96	12.9	57	135
50-29-3	4,4'-DDT	2681	97	15.3	51	143
309-00-2	Aldrin	3021	89.5	14.7	45	134
319-84-6	alpha-BHC	3070	95.8	13.9	54	138
5103-71-9	alpha-Chlordane	2736	94.3	11.6	60	129
319-85-7	beta-BHC	3068	96.3	13.3	56	136
57-74-9	Chlordane	150	101.2	13	62	140
319-86-8	delta-BHC	3035	97.2	15	52	142
60-57-1	Dieldrin	3078	98	12.6	60	136
959-98-8	Endosulfan I	968	93.8	10.7	62	126
33213-65-9	Endosulfan II	3047	93.4	13.7	52	135
1031-07-8	Endosulfan sulfate	3013	97.2	11.9	62	133
72-20-8	Endrin	3635	98.7	13	60	138
7421-93-4	Endrin aldehyde	3018	91.1	13.5	51	132
53494-70-5	Endrin Ketone	2908	95.9	12.6	58	134
58-89-9	gamma-BHC [Lindane]	3693	96.4	12.5	59	134
5103-74-2	gamma-Chlordane	3008	95.8	13.2	56	136
76-44-8	Heptachlor	3597	91.9	12.8	54	130
1024-57-3	Heptachlor Epoxide	3574	96.9	12.1	61	133
118-74-1	Hexachlorobenzene	134	82.1	18.1	27.8	136.5
72-43-5	Methoxychlor	3569	99	15.2	54	145
2385-85-5	Mirex	340	88.8	12.6	51	127
877-09-8	Tetrachloro-m-xylene	1510	84.1	13.3	44	124
8001-35-2	Toxaphene	421	83.9	16.8	33	134

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TITLE: PESTICIDES BY GAS CHROMATOGRAPHY (EPA METHODS 608, 8081A, 8081B, SM 6630C)

Table 5. Quality Control Requirements – Organic Analysis by Gas Chromatography

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Breakdown check (Endrin/DDT)	Before sample analysis and at the beginning of each 12-hour shift.	Degradation of DDT and Endrin must each be $\leq 15\%$.	Correct problem, then repeat breakdown checks.	Flagging is not appropriate.	No samples shall be run until degradation of DDT and Endrin is each $\leq 15\%$.
Initial Calibration (ICAL) for all analytes (including surrogates)	At instrument set-up and after ICV or CCV failure, prior to sample analysis.	ICAL must meet one of the three options below: Option 1: RSD for each analyte $\leq 20\%$; Option 2: linear least squares regression for each analyte: $r^2 \geq 0.99$; Option 3: non-linear least squares regression (quadratic) for each analyte: $r^2 \geq 0.99$.	Correct problem then repeat ICAL.	Flagging is not appropriate.	Minimum 5 levels for linear and 6 levels for quadratic. Quantitation for multicomponent analytes such as chlordane, toxaphene, and Aroclors must be performed using a 5-point calibration. Results may not be quantitated using a single point. No samples shall be analyzed until ICAL has passed.
Retention Time window position establishment	Once per ICAL and at the beginning of the analytical sequence.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA	NA	Calculated for each analyte and surrogate.

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TITLE: PESTICIDES BY GAS CHROMATOGRAPHY (EPA METHODS 608, 8081A, 8081B, SM 6630C)

Table 5. Quality Control Requirements – Organic Analysis by Gas Chromatography

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Retention Time (RT) window width	At method set-up and after major maintenance (e.g., column change).	RT width is ± 3 times standard deviation for each analyte RT from the 72-hour study or 0.03 minutes, whichever is greater.	NA	NA	Calculated for each analyte and surrogate. Only applicable if internal standard calibration is not used.
Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All reported analytes within established RT windows. All reported analytes within $\pm 20\%$ of true value.	Correct problem, rerun ICV. If that fails, repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed until calibration has been verified with a second source.

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TITLE: PESTICIDES BY GAS CHROMATOGRAPHY (EPA METHODS 608, 8081A, 8081B, SM 6630C)

Table 5. Quality Control Requirements – Organic Analysis by Gas Chromatography

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Continuing Calibration Verification (CCV)	Before sample analysis, after every 10 field samples, and at the end of the analysis sequence with the exception of CCVs for Pesticides multi-component analytes (i.e., Toxaphene, Chlordane and Aroclors other than 1016 and 1260), which are only required before sample analysis.	All reported analytes and surrogates within established RT windows. All reported analytes and surrogates within $\pm 20\%$ of true value.	Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails or if two consecutive CCVs cannot be run, perform corrective action(s) and repeat CCV and all associated samples since the last Successful CCV. Alternately, recalibrate if necessary; then reanalyze all associated samples since the last acceptable CCV.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.	Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

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Table 5. Quality Control Requirements – Organic Analysis by Gas Chromatography

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Internal Standards (IS)	If employed, every field sample, standard, and QC sample.	Retention time within ± 0.06 RRT UNITS from retention time of the midpoint standard in the ICAL; Internal standard signal (area or height) within -50% to +100% of ICAL midpoint standard. On days when ICAL is not performed, the daily initial CCV can be used.	Inspect GC for malfunctions and correct problem. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	If corrective action fails in field samples, data must be qualified and explained in the Case Narrative. Apply Q-flag to analytes associated with the non-compliant IS. Flagging is not appropriate for failed standards.	NA.
Method Blank (MB)	One per preparatory batch.	No analytes detected $>1/2$ LOQ or $> 1/10$ the amount measured in any sample or $1/10$ the regulatory limit, whichever is greater.	Correct problem. If required, reprep and reanalyze MB and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

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Table 5. Quality Control Requirements – Organic Analysis by Gas Chromatography

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Laboratory Control Sample (LCS)	One per preparatory batch.	A laboratory must use Table 3 and 4 limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits (see the LIMS) if project limits are not specified.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per preparatory batch.	A laboratory must use Table 3 and 4 limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits (see the LIMS) if project limits are not specified.	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	For matrix evaluation only. If MS results are outside the limits, the data shall be evaluated to determine the source(s) of difference (i.e., matrix effect or analytical error).

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Table 5. Quality Control Requirements – Organic Analysis by Gas Chromatography

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch.	A laboratory must use Table 3 and 4 limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits (see the LIMS) if project limits are not specified. RPD \leq 30% (between MS and MSD or sample and MD).	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	The data shall be evaluated to determine the source of difference. For Sample/MD: RPD criteria only apply to analytes whose concentration in the sample is greater than or equal to the LOQ.
Surrogate Spike	All field and QC samples.	QC acceptance criteria specified by the project, if available; otherwise use Table 3 and 4 limits or in-house LCS limits (see the LIMS) if analyte(s) are not listed.	Correct problem, then reprep and reanalyze all failed samples for all surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary, but the client must be notified prior to reporting data, and the failures must be discussed in the Case Narrative.	Apply Q-flag to all associated analytes if acceptance criteria are not met and explain in the case narrative.	Alternative surrogates are recommended when there is obvious chromatographic interference.

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Table 5. Quality Control Requirements – Organic Analysis by Gas Chromatography

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Confirmation of positive results (second column)	All results > the DL must be confirmed (except for single column methods such as TPH by Method 8015 where confirmation is not an option or requirement).	Calibration and QC criteria for second column are the same as for initial or primary column analysis. Results between primary and secondary column RPD \leq 40%.	NA	Apply J-flag if RPD >40%. Discuss in the case narrative.	Use project-specific reporting requirements if available; otherwise, use method requirements if available; otherwise, report the result from the primary column.

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SOP Minor Revision Summary

SOP:			
Title -	SEMIVOLATILE ORGANICS BY GC/MS (EPA METHODS 8270C, 8270D, 625, 625.1 AND SM 6410B), INCLUDING PROVISIONS FOR ANALYSIS IN SIM MODE		
Number -	330345	Department -	SVOA
Revision -	26	Rev. Date -	3/22/18

This Standard Operating Procedure has been amended to include changes required during normal business operations. These changes as defined by SOP 010103 (Document Control and Distribution) are routine modifications that will be incorporated into the SOP upon the next scheduled review.

Rev.	Date	Section	Brief Description
a	6/4/18	8.1.2	Added details about the use of peak detection thresholds
		7.15.1	Added details about spike verification



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1.0 SCOPE AND APPLICATION

STATE NOTE: For samples analyzed in conjunction with the Ohio Voluntary Action Program (VAP) please utilize SOP #330345OH.

- 1.1 This method is used to determine the concentration of semi-volatile organic compounds in extracts prepared from many types of solid waste matrices, soils, and water samples. The lists of compounds that are routinely determined by this method are listed in Attachment II. This table represents a default list to be used in the absence of a project-specific list, which would take precedence. See section 13.4.
- 1.2 This method is used to quantitate most neutral, acidic and/or basic organic compounds that are soluble in methylene chloride and capable of being eluted, without derivatization, from a gas chromatographic fused-silica column coated with a slightly polar methyl silicone phase. Such compounds include polynuclear aromatic hydrocarbons, chlorinated hydrocarbons and pesticides, phthalate esters, organophosphate esters, nitrosamines, haloethers, aldehydes, ethers, ketones, anilines, pyridines, quinolines, aromatic nitro compounds, and phenols, including nitrophenols.
- 1.3 In general, this method is not appropriate for the quantitation of multi-component analytes (i.e. Toxaphene, Chlordane, Aroclors, etc.) because of the limited sensitivity for those analytes; however when those analytes are identified using another analytical technique, this procedure is appropriate for confirmation pending sufficient analyte concentration is present in the extract.
- 1.4 Detection limits, sensitivity and optimum ranges of organic compounds vary with sample matrices, extraction technique, detector parameters, and model of GC/MS.
- 1.5 Qualifier ions are method specified and can be found in Attachment IV.
- 1.6 Use of this method is restricted to analysts who are knowledgeable in the interpretation of Mass Spectrometry and use of GC/MS systems.
- 1.7 The use of selected ion monitoring (SIM) is acceptable for applications requiring limits below the normal range of electron impact mass spectrometry. However, SIM may provide a lesser degree of confidence in the compound identification unless multiple ions are monitored for each compound.

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- 1.8 An MDL study must be completed at least annually or more frequently if major instrumentation changes occur. Method Detection Limits (MDLs) are performed based on ESC SOP #030206. Updated MDL records are filed and stored on ESC's intranet.
 - 1.8.1 Limit of Detection (LOD) and Limit of Quantitation (LOQ) studies are completed at the frequency required by the TNI standard per the procedure identified in the ESC SOP #030206, *Method Detection Limits (MDL), Limits of Detection (LOD) and Limits of Quantitation (LOQ)*. Should the procedure be utilized for DOD support; then the frequency of these studies must meet the requirements of the current DOD QSM (see Attachment IX).
 - 1.8.2 Lower Limit of Quantitation (LOQ) – For analyses performed per the requirements of Method 8000D, the LLOQ is established at concentrations where both quantitative and qualitative requirements can consistently be met (see Sections 2.10 and 10.4).

2.0 METHOD SUMMARY AND DEFINITIONS

- 2.1 Field samples are prepared for analysis by gas chromatography/mass spectrometry (GC/MS) using the appropriate sample extraction technique. See ESC SOPs 330702/330702A/330702B/330705/330707/330708/330709/330754 for extraction and extract concentration methods. A measured volume or weight of sample is extracted using the appropriate extraction technique. Liquid samples are extracted at neutral pH with methylene chloride using a separatory funnel (SOP #330702) per EPA method 3510C. Reduced volume (RV) extraction using EPA method 3510C that requires a smaller volume (usually 100mL) of field sample is also available for use where applicable. Large volume injection (LVI) extraction using EPA method 3511 that requires a smaller volume (usually 40mL) of field sample is also available for use where applicable. See section 13.5 of this procedure and ESC SOP #330702B. Soil analysis using the same technology can also be performed with extraction as noted in ESC SOP #330707 and no concentration performed on the extract. This process is termed throughout this SOP as non-concentrated soil. Solid samples can also be extracted traditionally using methylene chloride-acetone (1:1) and a sonication process (SOP #330705) or with methylene chloride using the microwave process (SOP #330707), where permitted. These extracts are denoted in this procedure using the terminology "concentrated soil" extracts.
- 2.2 The semi-volatile compounds are introduced into the GC/MS by directly injecting a volume of the sample extract into a gas chromatograph oven (GC) equipped with a narrow-bore fused-silica capillary column. The oven, containing the capillary column, is temperature and pressure programmed to separate the analytes by molecular composition. The capillary column transfers the eluting analytes to the detector (MS) connected to a computer that then collects and stores the information for each injection.
- 2.3 Identification of target analytes is accomplished by comparing the mass spectra of each peak with the reference spectra of authentic standards.

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- 2.4 Quantitation of the analytes of interest is accomplished by comparing the response of a major (quantitation) ion, present in the target analyte, relative to an internal standard in each extract, in conjunction with the response factor generated from a calibration curve.
- 2.5 Proper quantitation ions for each compound must be selected so that no interferences are present from adjoining (or co-eluting) analytes with common ions. Proper GC conditions must be used to resolve compounds with similar mass spectra. Background subtraction of mass spectra may be necessary when matrix interference is present.
- 2.6 Qualitative - The identification of compounds based on retention time and comparison of the sample mass spectra, after background correction, with characteristic ions in the reference mass spectra. The reference mass spectra must be generated by the laboratory using the same analytical conditions used for the analysis of field samples. The characteristic ions from the reference mass spectra are defined as the three ions of greatest relative intensity or any ions over 30% relative intensity if less than three such ions occur in the reference spectra.
- 2.7 Quantitative – Following qualitative identification, the quantitation of the identified compound is based on the integrated abundance of the primary characteristic ion from the Extracted Ion Current Profile (EICP).
- 2.8 Relative Retention Time (RRT) – The process of normalizing the response (peak area) of the target compound to the response of the internal standard.
- 2.9 Isotope dilution calibration - Isotope dilution calibration is essentially a special case of internal standard calibration. In isotope dilution, the internal standards are stable isotopically-labeled analogs of the target analytes *and* they are added to the sample prior to any sample handling steps, including sample extraction. Because the spiked compounds differ from the target compounds only in the presence of the stable isotopes, the physical and chemical behavior of each labeled compound is virtually the same as its unlabeled "native" analog. Thus, any losses of the target compound that may occur during any of the sample preparation, extraction, cleanup, or determinative steps will be mirrored by a similar loss of the labeled standard.
- 2.10 Lower Limit of Quantitation (LLOQ) – For analyses performed according to the requirements of Method 8000D, the lowest concentration at which the laboratory has demonstrated target analytes can be reliably measured and reported with a certain degree of confidence, which must be greater than or equal to the lowest point in the calibration curve.
- 2.11 Large Volume Injection (LVI): any injection volume >5ul. Technique is dependent upon type of GC inlet used and sensitivity of detection.
- 2.12 Minimum Level (ML): A term used in Method 625.1 which refers to either the sample concentration equivalent to the lowest calibration point in a method or a multiple of the MDL, whichever is higher. Minimum levels may be obtained in several ways: They may be published in a method; they may be based on the lowest acceptable calibration point used by a laboratory; or they may be calculated by multiplying the MDL in a method, or the MDL determined by a laboratory, by a factor of 3. For the purposes of NPDES

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compliance monitoring, EPA considers the following terms to be synonymous: "quantitation limit," "reporting limit," and "minimum level."

- 2.13 See the current Quality Assurance Manual for other definitions associated with terms found in this document.

3.0 HEALTH AND SAFETY

- 3.1 The toxicity or carcinogenicity of each reagent used in the laboratory has not been fully established. Each chemical must be regarded as a potential health hazard and exposure to these compounds must be as low as reasonably achievable. A reference file of safety data sheets (SDSs) is made available on ESC's intranet to all personnel. Use hazardous reagents in a fume hood whenever possible and if eye or skin contact occurs, flush with large volumes of water. Always wear safety glasses or a shield for eye protection, protective clothing and observe proper mixing protocols.
- 3.2 **CAUTION:** Be careful when diluting and mixing acids. ALWAYS pour acid into water when mixing. Gently heat acid mixtures (NEVER HEAT RAPIDLY), to prevent splatter from extremely exothermic reactions typical of acid-water mixtures, etc.
- 3.3 Prior to performing this procedure, the analyst should be familiar with the proper use of corrosive liquid spill kits and contaminant procedures.
- 3.4 Much of the instrumentation used in this procedure has heated zones that can cause severe burns. Always unplug all instruments before doing any maintenance that involves electrical parts.

4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

- 4.1 All samples must have been collected using a sampling plan that addresses the considerations of this method.
- 4.2 Requirements for sample extraction are detailed in SOP numbers 330702, 330702A, 330702B, 330705, 330707, 330708, 330709, and 330754.
- 4.3 The sample holding time for solid samples is 14 days to extraction and, for aqueous samples, the holding time is 7 days. Holding time begins when (date and time) the samples are collected and ends either 14 or 7 days following sampling, at the time sampled.
- 4.4 The holding time for each extract is 40 days from sample preparation to analysis.
- 4.5 The container for aqueous samples and liquid sludge being extracted using the traditional 1L EPA 3510 method are 1L amber glass bottles. For the reduced volume extraction process using the EPA 3510 method, 100mL amber glass bottles are utilized. The containers for aqueous samples being extracted using EPA Method 3511 are 40mL amber glass bottles. Add 0.008% $\text{Na}_2\text{S}_2\text{O}_3$ per liter, if residual chlorine is expected or present.

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- 4.6 Collect solid sample materials in 4 oz. jars or larger, depending on the weight and density of the sampled materials.
- 4.7 All samples and extracts must be shipped and stored at $<6^{\circ}\text{C}$.
- 4.8 Samples submitted for analysis that do not meet the requirements contained within this section must be addressed before performing the logging process within the laboratory. In some cases, exceeding the appropriate preservation and storage criteria can cause significant bias in the resulting data. Clients may need to resubmit samples where the conditions during shipment cause uncertainty regarding sample integrity. If samples do not meet the requirements for preservation, sampling, shipment and storage and the client approves the completion of the analytical process, sample results can be qualified and possible bias is narrated per the ESC SOP #030201, *Data Handling and Reporting*.

5.0 INTERFERENCES

- 5.1 Raw GC/MS data from all method blanks, samples, and spikes is evaluated for interferences. Determine if the source of interference is in the preparation and/or cleanup of samples and take corrective action to eliminate the problem.
- 5.2 Contamination by carryover can occur whenever high-concentration and low-concentration samples are sequentially analyzed. To reduce carryover, the sample syringe is rinsed between sample injections. Whenever an unusually concentrated sample is encountered, it should be followed by analysis of solvent to check for cross-contamination. Clean/replace injector liner or clip column, check with solvent blanks, and repeat samples if necessary.
- 5.3 Choice of quantitative ions and qualifier ions: Some compounds may co-elute, so the selection of quantitation ions and qualifier ions must be made carefully so these ions are specific to each of the compounds that co-elute. Qualifier ions that are most commonly used are listed in Attachment IV and are recommended from the published 8270 methods. There is no method stated ions for the following: Pyridine, 1-Methylnaphthalene, Biphenyl, Carbazole. Aniline and Bis (2-Chloroethyl)ether quantitation ions may vary due to chromatographic conditions causing co-elution of the shared primary ion. Targets have strongly-responding, analyte-specific secondary ions suitable for quantitative use. Refer to Attachment IV for ESC ions.
- 5.4 Problematic Compounds:
 - 5.4.1 Benzidine may be subject to oxidative losses during solvent concentration and exhibits poor chromatographic behavior.
 - 5.4.2 Hexachlorocyclopentadiene is subject to thermal decomposition in the GC inlet, as well as photochemical decomposition.
 - 5.4.3 N-nitrosodimethylamine may be difficult to separate from the solvent using the chromatographic conditions listed in this method.
 - 5.4.4 N-nitrosodiphenylamine decomposes in the GC inlet and can't be separated from diphenylamine.

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- 5.4.5 Pentachlorophenol, 2,4-Dinitrophenol, 4-Nitrophenol, Benzoic Acid, 4,6-Dinitro-2-methylphenol, 4-Chloro-3-methylphenol, 2-Nitroaniline, 3-Nitroaniline, 4-Chloroaniline, and Benzyl Alcohol are subject to erratic chromatographic behavior, especially when there is high boiling material contamination of the GC system.
- 5.4.6 Pyridine may perform poorly at the GC injection port temperatures listed in this method. The amount of degradation may be reduced by lowering the injection port temperature. Modification of the injection port temperature may adversely affect the performance of other target analytes.
- 5.4.7 Benzenethiol, or thiophenol, can be found in refinery wastes at caustic pH values. Benzenethiol is unstable in water/soils of neutral or acidic pH values. Benzenethiol rapidly degrades in organic solvents used to prepare the instrument calibration standards. Benzenethiol is part of Appendix VIII and the 1985 Skinner List, but was never included in Appendix IX to 40 CFR 264, due to its instability in the environment

6.0 EQUIPMENT AND SUPPLIES

- 6.1 Gas chromatograph/mass spectrometer system.
 - 6.1.1 Gas chromatograph (HP 6890/7890 or equivalent)- An analytical system complete with a temperature- programmable gas chromatograph suitable for split-less injection and all required accessories, including, auto sampler, syringes, analytical columns, and gases. The capillary column is directly coupled with the source.
 - 6.1.2 Column 1 - 30m x 0.25mm ID with a 0.25µm film thickness silicon-coated fused silica capillary column (Phenomonex ZB-5MS or equivalent).
 - 6.1.3 Column 2 – J&W 30m x 0.25mm x 0.5um film DB5MS or an equivalent is used. Ultrapure (99.999%) Helium gas is used for a mobile phase.
 - 6.1.4 Syringes: Agilent (or equivalent) syringes sizes 10µL, 25µL, 50µL, 100µL and 1.0mL.
- 6.2 Mass spectrometer (HP-5973/5975 or equivalent) capable of scanning from 35 to 550 amu every 1 second, using 70 volts (nominal) electron energy in the electron impact ionization mode. The mass spectrum for decafluorotriphenylphosphine (DFTPP) must meet the applicable criteria in method 8270C, 8270D or 525 when 50ng of DFTPP GC/MS tuning standard is injected.
- 6.3 GC/MS interface - The interface is capillary-direct into the mass spectrometer source.
- 6.4 Data system (HP Chemstation with Enviroquant) - A computer system is interfaced to the mass spectrometer. The system allows the continuous acquisition and storage of machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer has software that can search any GC/MS data file for ions of a specific mass and that can plot such ion abundances versus time or scan

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number. This type of plot is defined as Extracted Ion Current Profile (EICP). The most recent version of the EPA/NIST Mass Spectral Library is also available

6.5 Volumetric flasks, Class A - Appropriate sizes with ground-glass stoppers.

6.6 Balance - Analytical, capable of weighing 0.0001g

7.0 REAGENTS AND STANDARDS

7.1 All reagents and standards must be recorded in the appropriate preparation log and assigned a unique number. See SOP #030230, *Standard Logger – Tree Operation*. Additional information regarding reagent preparation can be found in the Standards Logger (Tree) digital archive system. All spiking solutions and surrogate standard solutions should be replaced at least every 6 months or sooner if a problem is detected unless otherwise noted.

7.2 Reagent grade inorganic chemicals are used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.3 Organic-free reagent water - all references to water in this method refer to organic-free reagent water (ASTM II or equivalent).

7.4 Burdick & Jackson Omni Solv Dichloromethane Dx0831-1 (or equivalent).

7.5 Stock standard solutions - Standard solutions are purchased as certified solutions. Commercially-prepared stock standards are used at concentrations that are certified by the manufacturer or by an independent source.

7.5.1 Restek, Custom 8270 Mix – 56321, or equivalent, at 200ppm

7.5.2 NSI, 8270 TCL Project Mix – Q4296, or equivalent, at 1000ppm

7.5.3 AccuStandard, Composite Mix #3 – Z-014E-R3, or equivalent, at 2000ppm

7.5.4 Restek, Benzoic Acid Mix – 31879, or equivalent, at 2000ppm

7.5.5 Restek, Benzidine Mix #2 – 31852, or equivalent, at 1000ppm

7.5.6 AccuStandard, 2-Nitrodiphenylamine – S-4829A, or equivalent, at 200ppm

7.5.7 B/N Surrogate Mix – C-376M-39, or equivalent, at 2000ppm

7.5.8 Organic Acid Surrogate Mix – C-131M-24, or equivalent, 4000ppm

7.5.9 Second Source: Restek, 8270 MegaMix – 31850, or equivalent, at 1000ppm

7.5.10 Benzenethiol Std: GCS011266-01-SS, or equivalent, at 1000ppm

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- 7.5.11 Indene Std: GCS011267-03, or equivalent, at 1000ppm
- 7.5.12 Quinoline Std: GCS011268-01-SS, or equivalent, at 1000ppm
- 7.5.13 Dibenz(a,h)acridine Std: GCS011269-05-SS, or equivalent, at 1000ppm
- 7.5.14 N-Nitrosodimethylamine (NDMA) STD: Restek, 521 Surr Std – 33910, or equivalent, at 1000ppm
- 7.5.15 Transfer the stock standard solutions into bottles with PTFE-lined screw caps. Store, protected from light, at -10°C or less or as recommended by the standard manufacturer. Stock standards should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them. Stock standards are assigned a 6 month expiration date from the day that a sealed ampoule is opened. Standards are discarded if signs of degradation are apparent when compared to a second source standard.
- 7.5.16 1,4-Dioxane ISTD: Restek; 1,4-Dioxane- d_8 ; #30614 2000ppm in methanol.
- 7.5.17 1,4-Dioxane ICAL: Restek; 1,4-Dioxane #31853 2000ppm in methylene chloride.
- 7.5.18 1,4-Dioxane SSCV/LCS: Restek; 1,4-Dioxane; #30287 2000ppm in methanol.
- 7.6 For PAHs by SIM, use a custom mix purchased from Ultra Scientific (Cat#: CUS-9356) with all required PAH targets. Each target compound is at concentration of 200ppm. Other concentrations may be acceptable with dilutions as appropriate for yielding the appropriate concentrations in the secondary source. The secondary source is also a custom mix from Ultra Scientific (Cat#: CUS-9345) at 200ppm.
- 7.7 For NDMA by EPA 8270C/D SIM, use the 8270 Mega Mix (sec. 7.5.1) and the SSCV (section 7.5.9) or equivalent.
- 7.8 For the Missouri Department of Natural Resources-specified Diesel Range Organics (DROMO) by GC/MS, use a custom mix purchased from Ultra Scientific (Cat#: CUS-8255), or equivalent, which is a neat solution of diesel and an Ultra Scientific custom mix (Cat#: CUS-8254) for the gasoline components at neat. Alternatively, calibration standards can be prepared using the TX TPH Calibration Mix from Restek (Cat#: 31483) at 10,000ppm each. The secondary source is an NSI, Diesel Range Organic Spike (Cat#: Q4394) at 2500ppm each.
- 7.9 Internal standards solutions- the internal standards are naphthalene- d_8 , acenaphthene- d_{10} , phenanthrene- d_{10} , chrysene- d_{12} , perylene- d_{12} and 1-4 dichlorobenzene- d_4 . Purchase from NSI (Cat # Q-6343-O) as certified stock solution at 800 $\mu\text{g/mL}$. Alternative internal standard concentrations may be used for LVI work. Internal standard intermediates at 16 $\mu\text{g/mL}$ and 4 $\mu\text{g/mL}$ are prepared for spiking, RV/LVI 8270PAHand RV/LVI 8270SIM analyses, respectively.
- 7.9.1 For all concentrated soil, 1000mL concentrated water, and 8270 full run water reduced volume extracts, use the 800 $\mu\text{g/mL}$ internal standard solution. Each sample extract undergoing analysis is spiked with 10 μL of internal standard

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intermediate solution, resulting in a concentration of 8µg/mL of each internal standard.

- 7.9.2 For non-concentrated soil, reduced volume water and 3511 water analyses, including PAH and DROMO, use the 16µg/mL ISTD intermediate. For non-concentrated soil, reduced volume and EPA 3511 water extracts being analyzed by the SIM process, use the 4µg/mL ISTD intermediate. Each sample extract undergoing analysis is spiked with 10µL of the appropriate internal standard intermediate solution, resulting in a concentration of 160µg/L and 40µg/L, respectively, for each internal standard.

7.10 Preparation of Intermediate Standard

Stock Mix	Section ID:	Amount Added (mL)	Concentration of Stock in ppm	Concentration in Intermediate (ppm)
Restek - Custom Mix with Surrogates	7.5.1	--	200	--
NSI - 8270 TCL Project Mix	7.5.2	2.0	1000	200
AccuStandard - Composite Mix #3	7.5.3	1.0	2000	200
Restek - Benzoic Acid Mix	7.5.4	1.0	2000	200
Restek - Benzidine Mix #2	7.5.5	1.0	2000	200
AccuStandard - 2-Nitrodiphenylamine	7.5.6	2.0	1000	200

Using a volumetric syringe, measure each of the solutions listed in Section 7.9 and place into a 10mL volumetric flask. The final concentration will be 200µg/mL of each component. Use this solution or the certified custom mix purchased from Restek in section 7.5.1 to prepare the working standards in the tables in section 7.10.

- 7.10.1 For 1L extractions or concentrated soil extracts using SIM, prepare a 5µg/mL intermediate by diluting the 10µg/mL PAH mix described in section 7.6.
- 7.10.2 For EPA Method 3511 extracts for 8270 analyses, PAH reduced volume, and non-concentrated soil, a 2µg/mL intermediate is prepared directly by diluting 100µL of the 200µg/mL stock to a final volume of 10mL using volumetric glassware.
- 7.10.3 For SIM analyses using reduced volume or EPA 3511 water extracts or non-concentrated soil extracts, a 200ug/L intermediate is prepared directly by diluting 10µL from the 200µg/mL stock to a final volume of 10mL using volumetric glassware.

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7.10.4 For DROMO analyses using extraction method 3511 for water samples or non-concentrated soil, prepare a 200ug/mL intermediate in 10mL of methylene chloride by adding 40uL of each Gasoline and Diesel at 50,000ug/mL.

7.11 Preparation of Working Standards

Standards must be stored at $4 \pm 2^{\circ}\text{C}$. The expiration date of any working standard will be 6 months unless the manufacturer's stock expires prior to that date or if the standard starts showing signs of degradation. See section 7.11.1 through 7.11.6 for preparation instructions. Concentrations of standards used are subject to change depending on instrument condition, client needs and sample preparation method of the variety of analysis being performed. A minimum of five calibration levels is required for Method 8270C and 8270D, while a minimum of 3 calibration levels is required for Method 625.

7.11.1 8270C/D Calibration standards for concentrated soil and 1L concentrated water extractions: A minimum of five calibration standards is prepared at different concentrations. At least one of the calibration standards must correspond to a sample concentration at or below the laboratory's reporting limit (RL). The remaining standards correspond to the working range of the GC/MS system. Each standard contains each analyte for detection. Working standards are made directly from the intermediate stock standard described in section 7.10 give solutions at concentrations of 0.2µg/mL up to 50µg/mL. Surrogates are included at the same concentrations. Internal standards are spiked at a constant concentration per extraction method for quantitation purposes.

SVOC mix (200ppm) µL	ISTD mix uL	Final volume	Final conc. ppm	Level
1	10	1.0mL	0.2	1
5	10	1.0mL	1	2
10	10	1.0mL	2	3
25	10	1.0mL	5	4
50	10	1.0mL	10	5
75	10	1.0mL	15	6
100	10	1.0mL	20	7
150	10	1.0mL	30	8
200	10	1.0mL	40	9
250	10	1.0mL	50	10

A minimum of 5 points are used to construct the calibration curve.

7.11.2 Calibration standards for 8270C/D reduced volume and EPA 3511 (soil and water) extracted samples: A minimum of five calibration standards is prepared at different concentrations. At least one of the calibration standards must correspond to a sample concentration at or below the laboratory-reporting limit (RL). The remaining standards correspond to the working range of the GC/MS system. Each standard contains each analyte for detection. Working standards are made directly from the intermediates described in section 7.9 to give solutions at concentrations of 0.01µg/mL up to 1µg/mL. Surrogates are included

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at the same concentrations. Internal standards are spiked at a constant of 160µg/L for quantitation purposes.

SVOC mix (2ppm) µL	ISTD mix uL	Final volume	Final conc. ppb	Level
5	10	1.0mL	10	1
25	10	1.0mL	50	2
50	10	1.0mL	100	3
100	10	1.0mL	200	4
200	10	1.0mL	400	5
300	10	1.0mL	600	6
400	10	1.0mL	800	7
500	10	1.0mL	1000	8

7.11.3 For SIM analyses concentrated soil and 1L water extractions, calibration standards are diluted from the intermediate standard solution (section 7.9.1) to give a calibration at the following concentrations: 20, 50, 100, 500, 1000, 2000, 4000, 10,000µg/L. A minimum of five calibration standards is prepared at different concentrations. At least one of the calibration standards must correspond to a sample concentration at or below the laboratory-reporting limit (RL). The calibration levels may change based on the working range of the GC/MS system. Surrogates are included at the same concentrations. The internal standards are at a constant 8µg/mL.

SIM Standard Concentration (ug/L)	Amount Added (uL) 5µg/mL Int.	Final Volume (mL)
20	2.0	1.0
50	5.0	1.0
100	10.0	1.0
500	50.0	1.0
1000	100.0	1.0
2000	200.0	1.0
4000	400.0	1.0
10000	1000.0	1.0

7.11.4 For SIM analyses using reduced volume, non-concentrated soil, or EPA 3511 extracts, calibration standards are diluted from the intermediate standard solution (section 7.9.3) to give a calibration at the following concentrations: 1, 5, 10, 20, 40, 80, 200µg/L. A minimum of five calibration standards is prepared at different concentrations. At least one of the calibration standards must correspond to a sample concentration at or below the laboratory-reporting limit (RL). The calibration levels may change based on the working range of the GC/MS system. Surrogates are included at the same concentrations. The internal standards are at a constant 40µg/L.

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SIM RV 3511 Standard Concentration (ug/L)	Amount Added (uL) 200µg/L Int.	Final Volume (mL)
1	5	1.0
5	25	1.0
10	50	1.0
20	100	1.0
40	200	1.0
80	400	1.0
200	1000	1.0

7.11.5 For Missouri DRO analysis by 3511 and non-concentrated soil, prepare the working calibration curve as reflected in the following table.

DROMO mix (200ppb) µL	Final Volume	Final conc. (ppb)
25	1.0 mL	5
50	1.0 mL	10
100	1.0 mL	20
200	1.0 mL	40
400	1.0 mL	80
600	1.0 mL	120
800	1.0 mL	160

7.12 DFTPP Standard Prep for 50ppm Solution – 50µL of 1000 ppm DFTPP (AccuStandard M-625-TS-20X) + 950µL of Methylene Chloride (final volume of 1mL).

7.13 DFTPP Standard Prep for 25ppm Solution – 25µL of 1000 ppm DFTPP (AccuStandard M-625-TS-20X) + 950µL of Methylene Chloride (final volume of 1mL).

7.14 DFTPP Standard Prep for 2ppm Solution – 2µL of 1000 ppm DFTPP (AccuStandard M-625-TS-20X) + 950µL of Methylene Chloride (final volume of 1mL).

7.15 Surrogates and Spike Solutions – Preparation techniques are detailed in SOP numbers 330702, 330702A, 330702B, 330705, 330707, 330708, 330709, and 330754.

7.16 See section 13.4 for additional information regarding standards and spiking solutions.

8.0 PROCEDURE

STATE NOTE: For samples analyzed in conjunction with the Ohio VAP program, the criteria found and itemized in this procedure for EPA method 8270C must be utilized. Alternative GCMS tuning criteria from that specified in EPA 8270C is acceptable as permitted in Section 7 of the published method.

8.1 GC Conditions: The GC conditions are listed in each instrument maintenance log and are updated as necessary.

8.1.1 Due to the tuning and calibration requirements outlined in this method, a liner change is necessary prior to beginning an analytical sequence. Any additional maintenance performed on the instrumentation will be documented as performed

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in the specific instrument maintenance log. (i.e., column clip/change, septa change, inlet cleaning, detector cleaning/maintenance, etc.)

- 8.2 Mass Spectrometer Tuning Criteria: The GC/MS is hardware-tuned using a 50ng (or less) injection of DFTPP. Analyses must not begin until the tuning criteria are met. It is recommended that each initial tune verification utilize the "Autofind" function and be set up to look at three scans (the apex & ± 1 scan) and average the three scans then perform background subtraction. Background subtraction is required prior to the start of the peak but no more than 20 scans prior. Background correction cannot include any parts of the target peak. The scans must be averaged and background corrected. Average scans 0.1 minute before to 0.1 minute after the target peak including 2 scans and the peak apex. The mass spectrometer must be tuned every 12 hours if samples, standards, etc. are to be analyzed for Methods 8270C, 8270D, and 625.1 or every 24 hours for Method 625. ESC uses 8270D evaluation criteria per method allowances.

TABLE 8.2
Method 8270D
DFTPP Key Ions And Ion Abundance Criteria^(a, b)

Mass Ion Abundance Criteria	
51	10-80% of mass 198
68	<2% of mass 69
70	<2% of mass 69
127	10-80% of mass 198
197	<2% of mass 198
198	Base peak, or >50% of mass 442
199	5-9% of mass 198
275	10-60% of mass 198
365	>1% of mass 198
441	Present, but <24% of mass 442
442	Base peak, or >50% of mass 198
443	15-24% of mass 442

(a) Data taken from Table 3 in SW-846 Method 8270D.

(b) Alternate tuning criteria may be used (e.g., CLP, Method 525, or manufacturers' instructions), providing that method performance is not adversely affected.

METHOD NOTE: Per Method 625.1 requirements, the 12-hour shift begins after the DFTPP and DDT/endrin tests (if DDT and endrin are to be determined), and after analysis of the calibration verification standard. The 12-hour shift ends 12 hours later. The DFTPP, DDT/endrin, and calibration verification tests are outside of the 12-hour shift.

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STATE NOTE: All South Carolina samples require a tune every 12 hours, regardless of which method is being utilized.

The GC/MS tuning standard solution must also be used to assess GC column performance and injection port inertness. Degradation of DDT to DDE and DDD is used to assess breakdown occurring in the injection port. The calculation for the determination of the breakdown occurring is found in section 9.1 and must include both DDD and DDE. Breakdown must not exceed 20%. Benidine and pentachlorophenol are used to assess tailing occurring within the analytical system and both analytes should be present at their normal responses with no obvious peak tailing. To determine the tailing factor for benidine and pentachlorophenol, use the calculation found in section 9.2. For EPA Methods 625 and 8270C, benidine must have a tailing ratio of <3 and pentachlorophenol must have a tailing ratio of <5. For EPA Method 8270D and 625.1, benidine and pentachlorophenol must have a tailing ratio of <2. The Missouri diesel method does not require tailing or degradation checks prior to or during analysis.

- 8.3 The use of selected ion monitoring (SIM) is acceptable for applications requiring quantitation limits below the normal range of electron impact mass spectrometry. However, SIM may provide a lesser degree of confidence in the compound identification since less mass spectral information is available. Using the primary ion for quantitation and the secondary ions for confirmation set up the collection groups based on their retention times. The selected ions are nominal ions and most compounds have small mass defect, usually less than 0.2 amu, in their spectra. These mass defects should be used in the acquisition table. The dwell time may be automatically calculated by the laboratory's GC/MS software or manually calculated using the following formula. The total scan time should be less than 1,000 msec and produce at least 5 to 10 scans per chromatographic peak. The start and stop times for the SIM groups are determined from the full scan analysis using the formula below: Additional guidance for performing SIM analyses, in particular for PAHs and phenol target analyte compounds, can be found in the most recent CLP semivolatile organic methods statement of work (SOW). See the SIM sections from the following CLP SOW for further details: EPA CLP Organics SOW. (Reference 14)

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SIM Groups for PAHs and including pentachlorophenol and hexachlorobenzene

SIM Group	1	2	3	4	5	6	7	8
RT start	Solvent delay	Before 2-Methyl naphthalene	Before Acenaphthalene	Before Fluorene	Before Fluoranthene	Before Benzo (a)-anthracene	Before Benzo (b)-fluoranthene	Before Dibenz (a,h)-anthracene
Ions	82, 128, 129, 136, 137	127, 141, 142, 162, 171, 172	139, 151, 152, 153, 154, 162, 164, 168	94, 165, 166, 176, 178, 179, 188, 264, 266, 268, 282, 284, 286	200, 202, 203, 244, 245	226, 228, 229, 240, 241	252, 253, 260, 264	138, 139, 276, 277, 278, 279
Dwell	40	35	25	30	40	50	75	50

8.4 Calibration

8.4.1 Initial Calibration

EPA Method 8270C: The working standards prepared in section 7.10 are injected and average response factors are calculated. The calibration curve is typically constructed of six to nine standards, however, this may change depending on instrument conditions and/or client needs (see Section 13.4). See section 8.3.2 for information regarding use and deletion of calibration points.

The calibration check compounds (CCCs) listed in Section 8.3.1a must have an average percent relative standard deviation (%RSD) of less than or equal to 30%. Any target analyte that has a %RSD >15% for the RF must be calculated by linear or quadratic regression instead of RF. If the RSD of any target analyte is ≤15%, the average response factor may be used for quantitation. When any compound does not meet the calibration criteria for RF, the analyst MUST use linear regression or quadratic curve fit. The calibration curve cannot be forced through zero and does not include a method blank. It must also meet a correlation coefficient of 0.990 or better. Analyses being generated for USACE projects must meet a correlation coefficient of 0.995 or better. If a quadratic curve fit is used, a minimum of 6 calibration standards must be utilized to obtain a working calibration curve.

The system performance check compounds (SPCCs) in Table 8.3.1b must have an average RF of ≥0.05. When these criteria are met, samples can be analyzed.

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Table 8.3.1a: Calibration Check Compounds (CCC)

Base/Neutral Fraction	Acid Fraction
Acenaphthene	4-Chloro-3-methylphenol
1,4-Dichlorobenzene	2,4-Dichlorophenol
Hexachlorobutadiene	2-Nitrophenol
n-Nitrosodiphenylamine	Phenol
Di-n-octyl phthalate	Pentachlorophenol
Fluoranthene	2,4,6-Trichlorophenol
Benzo(a)pyrene	

Table 8.3.1b: System Performance Check Compounds (SPCC)

Compound	Minimum Average Response Factor
n-Nitroso-di-n-propylamine	>0.05
Hexachlorocyclopentadiene	>0.05
2,4-Dinitrophenol	>0.05
4-Nitrophenol	>0.05

EPA Method 8270D: The working standards prepared in section 7.10 are injected and average response factors are calculated. The calibration curve is typically constructed of six to nine standards, however, this may change depending on instrument conditions and/or client needs (see section 13.4). At least five standards are required for Response Factor and linear regression calibration. If a quadratic curve fit is used, a minimum of 6 calibration standards must be utilized to obtain a working calibration curve. See section 8.3.2 for information regarding use and deletion of calibration points.

Target analytes must have an average RSD of $\leq 20\%$. Any target analyte that has a %RSD $> 20\%$ for the RF must be calculated by linear or quadratic regression instead of RF. If the RSD of any target analyte is $\leq 20\%$, the average response factor may be used for quantitation. When any compound does not meet the calibration criteria for RF, the analyst MUST use linear regression or, if permitted, quadratic curve fit. The calibration curve cannot be forced through zero. It must also meet a correlation coefficient of 0.990 or better. Analyses being generated for USACE projects must meet a correlation coefficient of 0.995 or better.

In addition to the minimum %RSD criteria, it is recommended that a minimum response factor for the most common target analytes be demonstrated for each individual calibration level to ensure that these compounds are performing as expected. See Table 8.3.1c. Meeting the minimum response factor criteria for the lowest calibration standard is critical in establishing and demonstrating the desired sensitivity.

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Table 8.3.1c: Recommended Minimum Response Factors for Each Calibration Level (Initial and Continuing Calibration)

<i>Compound</i>	<i>Minimum Response Factor</i>	<i>Compound</i>	<i>Minimum Response Factor</i>
Benaldehyde	0.010	4-Nitrophenol	0.010
Phenol	0.800	Dibenzofuran	0.800
Bis(2-chloroethyl)ether	0.700	2,4-Dinitrotoluene	0.200
2-Chlorophenol	0.800	Diethyl phthalate	0.010
2-Methylphenol	0.700	1,2,4,5-Tetrachlorobenzene	0.010
2,2-Oxybis-(1-chloropropane)	0.010	4-Chlorophenyl-phenyl ether	0.400
Acetophenone	0.010	Fluorene	0.900
4-Methylphenol	0.600	4-Nitroaniline	0.010
n-Nitroso-di-n-propylamine	0.500	4,6-Dinitro-2-methylphenol	0.010
Hexachloroethane	0.300	4-Bromophenyl-phenyl ether	0.100
Nitrobenzene	0.200	n-Nitrosodiphenylamine	0.010
Isophorone	0.400	Hexachlorobenzene	0.100
2-Nitrophenol	0.100	Atrazine	0.010
2,4-Dimethylphenol	0.200	Pentachlorophenol	0.050
Bis(2-chloroethoxy)methane	0.300	Phenanthrene	0.700
2,4-Dichlorophenol	0.200	Anthracene	0.700
Naphthalene	0.700	Carbazole	0.010
4-Chloroaniline	0.010	Di-n-butyl phthalate	0.010
Hexachlorobutadiene	0.010	Fluoranthene	0.600
Caprolactam	0.010	Pyrene	0.600
4-Chloro-3-methylphenol	0.200	Butyl Benzyl phthalate	0.010
2-Methylnaphthalene	0.400	3,3-Dichlorobenzidine	0.010
Hexachlorocyclopentadiene	0.050	Benzo(a)anthracene	0.800
2,4,6-Trichlorophenol	0.200	Chrysene	0.700
2,4,5-Trichlorophenol	0.200	Bis (2-ethylhexyl)phthalate	0.010
1,1-Biphenyl	0.010	Di-n-octyl phthalate	0.010
2-Chloronaphthalene	0.800	Benzo(b)fluoranthene	0.700
2-Nitroaniline	0.010	Benzo(k)fluoranthene	0.700
Dimethyl phthalate	0.010	Benzo(a)pyrene	0.700
2,6-Dinitrotoluene	0.200	Indeno(1,23-c,d)pyrene	0.500
Acenaphthylene	0.900	Dibenz(a,h)anthracene	0.400
3-Nitroaniline	0.010	Benzo(g,h,i)perylene	0.500
Acenaphthene	0.900	2,3,4,6-Tetrachlorophenol	0.010
2,4-Dinitrophenol	0.010		

EPA 8270C GC/MS SIM: When analyzing samples using SW-846 8270C SIM, all target compounds must be treated as CCCs and must have an average RSD of $\leq 30\%$.

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EPA 8270D GC/MS SIM: If analyzing samples by EPA 8270D SIM, follow the initial calibration criteria for the specified referenced method as found in section 8.3.1 (EPA Method 8270D).

EPA Method 625: The working standards prepared in section 7.10 are injected and average response factors are calculated. A minimum of 3 points calibration is required for method 625. The %RSD is calculated for the standards analyzed and must be $\leq 35\%$ for all compounds in order to assume linearity.

EPA Method 625.1: One of the calibration standards should be at a concentration at or below the minimum level (ML) specified in Attachment VII or as specified by a regulatory/control authority or in a permit. The ML value may be rounded to a whole number that is more convenient for preparing the standard, but must not exceed the ML in Attachment VII for those analytes which list ML values. Alternatively, the laboratory may establish a laboratory ML for each analyte based on the concentration in a nominal whole-volume sample that is equivalent to the concentration of the lowest calibration standard in a series of standards produced in the laboratory or obtained from a commercial vendor. The laboratory's ML must not exceed the ML in Attachment VII, and the resulting calibration must meet all applicable acceptance criteria in Section 10, based on the RSD, RSE, or r^2 . The concentrations of the other calibration standards should correspond to the expected range of concentrations found in real samples or should define the working range of the GC/MS system for full-scan and/ or SIM operation, as appropriate. A minimum of six concentration levels is required for a second order, non-linear (i.e., quadratic) calibration.

Calculate the mean (average) and relative standard deviation (RSD) of the responses factors. If the RSD is less than 35%, the RF can be assumed to be invariant and the average RF can be used for calculations. Alternatively, the results can be used to fit a linear or quadratic regression of response ratios, A_s/A_{is} , vs. concentration ratios C_s/C_{is} . If used, the regression must be weighted inversely proportional to concentration. The coefficient of determination (r^2) of the weighted regression must be greater than 0.920 (this value roughly corresponds to the RSD limit of 35%). Alternatively, the relative standard error (RSE) may be used as an acceptance criterion. As with the RSD, the RSE must be less than 35%. If an RSE less than 35% cannot be achieved for a quadratic regression, system performance is unacceptable and the system must be adjusted and re-calibrated.

All Published Methods: Reference spectra must be updated upon analysis of each new calibration curve.

Linear Regression Weighting: As an alternative to calculating mean response factors and applying the RSD test, use the GC/MS data system software or other available software to generate a linear or second order regression calibration curve, by plotting A/A_{is} vs. $Q(x)$ using the equations found in section 9.4. Either equal weighting factors or $1/x$ regressions may be used.

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STATE NOTE: For all Minnesota sample analyses, the RL level standard is re-injected and quantitated against the newly updated calibration curve or the applicable standards are reprocessed (re-quantitated) using the completed calibration curve and is evaluated for the $\pm 40\%$ deviation criterion with the exception of the listed poor performers in this procedure.

STATE NOTE: For all Wisconsin sample analyses, analysts must evaluate the %RSD of calibrations to ensure that they do not have unacceptable curvature. The %RSD limit criteria, as found in the specific methods listed above, applies to calibrations using average RF calibrations. For linear and quadratic curve fits, a limit of 40% RSD is used for normal target analytes and 50% RSD is utilized for known poor performing compounds.

STATE NOTE: When analyzing samples in conjunction with the Ohio VAP or South Carolina DHEC programs, the calibration model must be RSD or linear. Quadratic curve modeling is not permitted unless historical performance of analytes exhibited a nonlinear response (i.e., Benzoic Acid and problematic phenols). Quadratic models cannot be used to extend the calibration range or bypass instrument maintenance.

8.4.2 CALIBRATION POINTS – Usage and Deletion

When the appropriate number of calibration standards is used, all points must be considered in the average response factor calculation or linear regression calculation. The deletion of the highest point is acceptable when necessary, with the analyst noting that the high end of the calibration has been lowered. The deletion of the lowest calibration point is acceptable, when necessary, provided that the analyst notes the deletion on the injection log and raises the reporting limit, if necessary, for that compound.

8.4.3 EPA Method 8270D: LINEAR REGRESSION USE – The method of linear regression calibration has the potential for a significant bias to the lower portion of the calibration model. This bias is not normally seen in relative percent difference methods. When utilizing linear regression fits, a minimum quantitation check on the viability of the lowest calibration point should be performed by re-fitting the response from the lowest concentration standard back into the completed calibration curve. It is not necessary to re-analyze a low concentration standard, but using the analytical system software, the low standard can be re-quantitated as if it were a field sample. The recalculated concentrations of the analytes utilizing the linear regression curve fit must be within $\pm 30\%$ of the true standard concentration.

STATE NOTE: For the analysis of South Carolina samples, all target analytes, including Hexachlorophene, is required to utilize linear regression. Quadratic curve fit is not allowed. To achieve this,

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the calibration curve may be modified by the removal of the lowest two levels and will utilize calibration levels of 60, 80, 100, 120, and 140 for quantitation of this analyte in South Carolina samples. The reporting limit (RL) for South Carolina will routinely be 100ppb for water samples.

- 8.4.4 Quadratic Regression: Quadratic regression may be used for the following compounds: Pentachlorophenol, 4-Nitrophenol, 2,3,4,6-Tetrachlorophenol, 4,6-Dinitro-2-methylphenol, 2,4-Dinitrophenol. Plots must have a minimum of 6 points and a correlation coefficient of 0.995 or better.

STATE NOTE: Quadratic curve modeling is not permitted for samples originating in South Carolina or for samples reported for the Ohio VAP unless historical performance of analytes exhibited a nonlinear response (e.g., Benzoic Acid).

- 8.4.5 Second Source Calibration Verification – the initial calibration for each target analyte must be checked with a standard from a source that is different from those used for initial calibration.

8.4.6 **Daily Tuning and Continuing Calibration**

As with the initial calibration, the system must be tuned with 50ng of DFTPP or less to meet the acceptance criteria found in section 8.1. Following successful tuning, the midpoint level standard (CCV) is analyzed. Calibration verification for each method, as listed below, must be met prior to the analysis of field samples and every 12 hours for 8270C/D and every 24 hours for EPA 625 (see the method note in Section 8.2 for Method 625.1 requirements).

EPA Method 8270C: The percent difference of the CCCs (see Table 8.3.1a & b) in the mid-level standard must be $\leq 20\%$ and the SPCCs must have an RF ≥ 0.05 . The retention time of the internal standards must be within ± 30 seconds from the mid-point standard level of the last initial calibration curve and the area response must be within -50% to $+100\%$. Once these criteria are met, samples can be analyzed.

EPA Method 625: The calculated recovery for any parameter in the method from the mid-level standard must not vary by more than $\pm 20\%$ drift from the initial calibration curve.

EPA Method 625.1: The RF or calibration curve must be verified immediately after calibration and at the beginning of each 12-hour shift, by analysis of a standard at or near the concentration of the mid-point calibration standard. The standard(s) must be obtained from a second manufacturer or a manufacturer's batch prepared independently from the batch used for calibration. Include the surrogates in this solution. It is necessary to verify calibration for the analytes of interest only.

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Compare the recoveries for the analytes of interest against the acceptance criteria for recovery (Q) in Attachment VII and the recoveries for surrogates against the acceptance criteria in Attachment VIII. If recovery of the analytes of interest and surrogates meet acceptance criteria, system performance is acceptable and analysis of samples may continue. If any individual recovery is outside its limit, system performance is unacceptable for that analyte.

EPA Methods 8270C and 625 (analyzed concurrently): The CCV must be evaluated for CCC and SPCCs as per EPA Method 8270C requirements. All non-CCC and other target analytes must meet the criteria established in Method 625 for all analytes ($\pm 20\%$). For analytes not contained in the Method 625 analyte list, the analyst evaluates the CCV and the experience of the analyst weighs heavily in determining the usability of the data.

STATE NOTE: For all Wisconsin sample analyses, non-CCC compounds for 8270C requires a $\pm 50\%$ criteria for the CCV.

EPA 8270C GC/MS SIM: When analyzing samples using SW-846 EPA 8270C SIM, all compounds in the CCV must be treated as CCCs and must meet the minimum requirements of $\leq 20\%$ difference.

EPA 8270D GC/MS SIM: If analyzing samples by EPA 8270D SIM, follow the initial calibration criteria for the specified referenced method as found in section 8.3.6 (EPA Method 8270D below).

EPA Method 8270D: Each of the most common target analytes in the CCV must meet the minimum response factors in Table 8.3.1c. When using the average RF, the percent difference for each target compound in the CCV must be $\leq 20\%$. When using regression fit calibration, the percent drift of the CCV must be $\leq 20\%$. The retention time of the internal standards must be within ± 30 seconds from the mid-point standard level of the last initial calibration curve and the area response must be within -50% to $+100\%$.

8.4.7 For corrective action regarding tuning and calibration, see sections 11.1 and 11.2.

8.5 Method Blank Analysis – A method blank should be analyzed prior to any field sample analysis to verify that the analytical system is free from contaminants. If the method blank indicates that contamination may be present in the analytical system, it may be necessary to analyze a solvent blank to demonstrate the source of the contamination is not carryover from standards or lingering field sample artifacts.

8.6 GC/MS analysis of field samples and preparation QC.

8.6.1 It is highly recommended that the extracts be screened on a GC/FID or GC/PID using the same type of capillary column used in the GC/MS system. This will minimize contamination of the GC/MS system from unexpectedly high concentrations of organic compounds.

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- 8.6.2 Allow the extracts to warm to room temperature. Just prior to analysis, add 10 μ L of the internal standard solution to the 1mL concentrated extract or 5 μ L of the internal standard solution to the 0.5mL extract obtained from sample preparation.
- 8.6.3 If the response for any quantitation ion exceeds highest level of the initial calibration range, the extract must be diluted and re-analyzed. Additional internal standard must be added to the diluted extract to maintain the same concentration as in the calibration standards (0.04, 0.16 or 8ng/uL, unless a more sensitive GC/MS system is being used). For example, if performing a 1:10 dilution on a concentrated extract, take 100uL of the extract and dilute to a volume of 1mL with the appropriate solvent. Add 9uL of the appropriate internal standard solution to the diluted extract and inject on the analytical system. It can be assumed that 1uL of internal standard was contained in the 100uL extract used for the initial dilution.
- 8.6.4 Internal standard area counts and retention times must be monitored in all samples, spikes and method blanks to monitor system performance, check for drifting, ensure effective autosampler performance, etc. If the area of the Extracted Ion Current Profile (EICP) changes by a factor of 2 (-50% to +100%) from the areas in the daily CCV, corrective action is required. The RRT of the internal standard in the extract must be within ± 0.06 RRT units of the RRT of the daily CCV.

STATE NOTE: With each new calibration curve, a reporting limit verification (RLV) standard must be analyzed for samples analyzed from Minnesota. This standard consists of either re-injecting the low calibration standard(s) or re-processing the low standard(s) utilized in the construction of the calibration curve. The RLV must recover within $\pm 40\%$ of the expected concentration. See section 11.10 for additional information.

8.7 Qualitative Identification

- 8.7.1 The qualitative identification of compounds determined by this method is based on retention time and on comparison of the sample mass spectrum, after background correction, with characteristic ions in a reference mass spectrum. The reference mass spectrum must be generated by the laboratory using the conditions of this method. The characteristic ions from the reference mass spectrum are defined as the three ions of greatest relative intensity, or any ions over 30% relative intensity if less than three such ions occur in the reference spectrum. Retention time windows for internal standards and target compounds integrations are updated with each calibration curve and after any instrument maintenance occurs that causes a shift that may affect ChemStation integrations.
- 8.7.1.1 The intensities of the characteristic ions of a compound must maximize in the same scan or within one scan of each other. Selection of a peak by a data system target compound search routine where the search is based on the presence of a target chromatographic peak containing ions specific for the target compound at a compound-specific retention time will be accepted as meeting this criterion.

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- 8.7.1.2 The RRT of the sample component is within ± 0.06 RRT units of the RRT of the standard component.
- 8.7.1.3 The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum.
- (EXAMPLE: For an ion with an abundance of 50% in the reference spectrum, the corresponding abundance in a sample spectrum can range between 20% and 80%). Analyst experience is vital in this determination when interferences are present.
- 8.7.1.4 Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between the two isomers is <50% of the average of the two peak heights (for Method 8270D) and <25% of the sum of the two peak heights (for Methods 8270C & 625). Otherwise, structural isomers are identified as isomeric pairs.
- 8.7.1.5 Identification is hampered when sample components are not resolved chromatographically and produce mass spectra containing ions contributed by more than one analyte. When gas chromatographic peaks obviously represent more than one sample component (i.e., a broadened peak with shoulder(s) or a valley between two or more maxima), appropriate selection of analyte spectra and background spectra is important.
- 8.7.1.6 Examination of extracted ion current profiles of appropriate ions can aid in the selection of spectra, and in qualitative identification of compounds. When analytes co-elute (i.e., only one chromatographic peak is apparent), the identification criteria can be met, but each analyte spectrum will contain extraneous ions contributed by the co-eluting compound.
- 8.7.1.7 Absolute retention times are used for compound identification in all GC methods that do *not* employ internal standard calibration. Retention time windows are established to compensate for minor shifts in absolute retention times as a result of sample loadings and normal chromatographic variability. The width of the retention time window should be carefully established to minimize the occurrence of both false positive and false negative results. Tight retention time windows may result in false negatives and/or may cause unnecessary reanalysis of samples when surrogates or spiked compounds are erroneously not identified. Overly wide retention time windows may result in false positive results that may not be confirmed.
- 8.7.1.7.1 Before establishing retention time windows, make sure that the chromatographic system is operating reliably and that the system conditions are optimized for the target analytes and surrogates in the sample matrix to be analyzed. Make three injections of all

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standard mixtures over the course of a 72-hour period. Serial injections or injections over a period of less than 72 hours may result in retention time windows that are too tight.

- 8.7.1.7.2 Record the retention time (in minutes) for each single component analyte and surrogate to three decimal places. Calculate the mean and standard deviation of the three absolute retention times for each single component analyte and surrogate. For multi-component analytes, choose three to five major peaks (see the determinative methods for more details) and calculate the mean and standard deviation of those peaks.
- 8.7.1.7.3 If the standard deviation of the retention times for a target compound is 0.000 (i.e., no difference between the absolute retention times), then either collect data from additional injections of standards or use a default standard deviation of 0.01 minutes.
- 8.7.1.7.4 The width of the retention time window for each analyte, surrogate, and major constituent in multi-component analytes is defined as ± 3 times the standard deviation of the mean absolute retention time established during the 72-hour period or 0.03 minutes, whichever is greater.
- 8.7.1.7.5 Establish the center of the retention time window for each analyte and surrogate by using the absolute retention time for each analyte and surrogate from the calibration verification standard at the beginning of the analytical shift. For samples run during the same shift as an initial calibration, use the retention time of the mid-point standard of the initial calibration.
- 8.7.1.7.6 Calculate absolute retention time windows for each analyte and surrogate on each chromatographic column and instrument. New retention time windows must be established when a new GC column is installed or if a GC column has been shortened during maintenance.

8.8 TICs – Tentatively Identified Compounds

Periodically, clients may request the tentative identification of compounds present in the field sample that are not normal target compounds and are not normally calibrated. This identification is limited to the compounds in the current NBS (National Bureau of Standards) mass spectral library employed by ESC.

Library Search Identification – For samples containing components not associated with the calibration standards, a library search may be made for the purpose of a tentative identification. Data system library searches must not use normalization routines that would misrepresent the library or unknown spectra when making comparisons. For example, the RCRA permit or waste delisting requirements may require the reporting of

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non-target analytes. The analyst may only assign tentative identifications after visual comparison of sample spectra with the nearest library searches.

Guidelines for tentative identification are:

- Relative intensities of major ions in the reference spectrum (ions >10% of the most abundant ion) should be present in the sample spectrum.
- The relative intensities of the major ions should agree within $\pm 20\%$. (EXAMPLE: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30% and 70%).
- Molecular ions present in the reference spectrum should be present in the sample spectrum.
- Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of co-eluting compounds.
- Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or co-eluting peaks. Data system library reduction programs can sometimes create these discrepancies.

Routinely, ESC employs a minimum Q value of 80 for tentative identifications and a minimum concentration of 10ppb. Peaks below a Q value of 80 but above 10ppb are reported as "Unknown". Any identified peaks below 10ppb are removed as these could result from baseline noise or other interferences, not necessarily attributable to the field sample or reliably quantifiable using GCMS technology. Additionally, any peaks that are attributable to instrument contamination (i.e., siloxanes) are also removed.

8.9 Quantitative analysis

8.9.1 Once a compound has been identified, the quantitation of that compound will be based on the integrated abundance of the primary characteristic ion from the EICP.

8.9.1.1 It is recommended to use the integrations produced by the software if the integration is correct because the software will produce more consistent integrations of peaks in chromatograms. Manual integrations may be necessary in some cases and must be performed in conjunction with ESC SOP #030215, *Manual Integration*.

DOD samples must include a reason for each integration performed on the manual integration documentation.

8.9.2 If the RSD of a compound's response factor meets method requirements, then the concentration in the extract may be determined using the average response factor (average RF) from initial calibration data.

8.9.3 Where applicable, the concentration of any tentatively identified compounds in the sample should be estimated. The same formula as is used to calculate target analyte concentrations is used with the following modifications: The areas A_x and

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A_{is} must be from the total ion chromatograms and the RF for the compound is assumed at 1. See section 9.7 for calculation.

- 8.9.4 The resulting concentration must be reported indicating that the value is an estimate. Use the nearest internal standard free of interferences for estimated concentration calculations.
- 8.9.5 Quantitation of multi-component compounds (e.g., Toxaphene, Aroclors, etc.) is beyond the scope of Method 8270. Normally, quantitation is performed using a GC/ECD, by Methods 8081 or 8082. However, Method 8270 may be used to confirm the identification of these compounds, when the concentrations are at least 10 ng/ μ L in the concentrated sample extract.
- 8.9.6 **Peak Resolution:** Structural isomers that produce very similar spectra must be quantitated as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between the two isomers is <50% of the average of the two peak heights (for Method 8270D) and <25% of the sum of the two peak heights (for Methods 8270C & 625). Otherwise, structural isomers should be identified as isomeric pairs.

STATE NOTE: Minnesota MPCA requires that peak resolution of all co-eluters, analyzed using Method 8270C, must be resolved as close to 75% as possible, but not <70%. Resolution must be adequate at lower levels and not worsen as concentration increases.

- 8.9.7 Indeno(1,2,3-cd)pyrene and dibenz(a,h)anthracene share a similar structure and physical properties. Under routine analytical production conditions it is very difficult to achieve resolved chromatographic separation. The mass-spectra of these compounds exhibit base peaks separated by 2 AMUs (276 and 278 respectively) and these unique ions are used for quantitation of the respective compounds as defined by Method 8270. It has been found that the major base ion, 276, for indeno(1,2,3-cd)pyrene includes a significant contribution from dibenz(a,h)anthracene when the targets are present together at equal concentrations; however, indeno(1,2,3-cd)pyrene presence *does not* contribute significant ion 278 abundance to dibenz(a,h)anthracene quantitation at equal concentrations. For these reasons when dibenz(a,h)anthracene is found to be present at similar or lesser concentrations than indeno(1,2,3-cd)pyrene, the results are normalized by the calibration conditions and considered to be non-impacted. Alternatively, when dibenz(a,h)anthracene is found to be present at relatively greater concentrations than indeno(1,2,3-cd)pyrene, the indeno(1,2,3-cd)pyrene results are considered to be elevated and may be confirmed by a secondary acquisition and analysis utilizing a technique for chromatographic separation of the targets. Concentrations shall be considered similar up to a factor of two.

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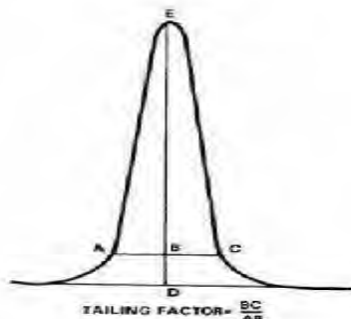
9.0 DATA ANALYSIS AND CALCULATIONS

9.1 GC/MS Tune: DDT Breakdown Determination during Tuning:

$$\% \text{ breakdown of DDT} = \frac{\text{sum of degradation peak areas (DDE + DDE)}}{\text{sum of all peak areas (DDT + DDE + DDD)}} \times 100$$

9.2 GC/MS Tune: Benzidine and Pentachlorophenol Tailing Factor

$$\text{Tailing Factor} = \frac{BC}{AB}$$



where: BC is the width of the back ½ of the peak at 10% of the peak height
 AB is the width of the front ½ of the peak at 10% of the peak height.

9.3 Internal Calibration Equations (Response Factors):

$$RF = \frac{[A_s][C_{is}]}{[A_{is}][C_s]}$$

where:

- A_s = Peak area (or height) of the analyte or surrogate.
- A_{is} = Peak area (or height) of the internal standard.
- C_s = Concentration of the analyte or surrogate, in µg/L.
- C_{is} = Concentration of the internal standard, in µg/L.

- Percent Relative Standard Deviation (%RSD)

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$$\overline{RF} = \frac{\sum_{i=1}^n RF_i}{n} \quad SD = \sqrt{\frac{\sum_{i=1}^n (RF_i - \overline{RF})^2}{n-1}} \quad RSD = \frac{SD}{\overline{RF}} \times 100\%$$

where:

RSD = Relative standard deviation.

RF = Mean of 5 initial RFs for a compound.

SD = Standard deviation of average RFs for a compound.

- Concentration of an analyte in an extract using RF (on column):

$$X_s = \frac{(Conc_{Std})(Area_{Analyte})}{(Average RF_{analyte})(Area_{Std})}$$

where:

X_s = Calculated raw concentration of analyte (in ppb)

- Quantitation Report Multiplier

$$M_a = \frac{(V_t)(D)}{(V_s)} \quad \text{or} \quad M_s = \frac{(V_t)(D)}{(W_s)}$$

where:

M_a = Quantitation Report Multiplier for Aqueous Samples

M_s = Quantitation Report Multiplier for Solid Samples

V_t = Total volume of concentrated extract (in mL)

D = Dilution factor. If no dilution, D=1. Always dimensionless

V_s = Volume of aqueous sample extracted (in mL)

W_s = Weight sample extracted (in grams)

- Sample concentration by volume (ug/L) for aqueous samples:

$$\text{Concentration in } \frac{mg}{L} = (X_s)(M_a)$$

- Sample concentration by weight (ug/kg) for solid samples and non-aqueous liquids:

$$\text{Concentration in } \frac{mg}{kg} = \frac{(X_s)(M_s)}{(\%S)}$$

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where:

%S = Percent solids expressed as a decimal

9.4 Relative Retention Time (RRT):

$$RRT = \frac{RT \text{ of Target Analyte}}{RT \text{ of Internal Standard}}$$

9.5 Percent Error (%Error)

$$\%Error = \frac{x_i - x'_i}{x_i} * 100$$

where:

x'_i = Measured amount of analyte at the calibration level i , in mass or concentration units

x_i = True amount of analyte at calibration level i , in mass or concentration units

9.6 Relative Standard Error (%RSE) – As an alternative to using the average response factor when using Method 625.1, the quality of the calibration may be evaluated using the Relative Standard Error (RSE). The acceptance criterion for the RSE is the same as the acceptance criterion for Relative Standard Deviation (RSD), in the method. RSE is calculated as:

$$\%RSE = 100 \times \frac{\sum_{i=1}^n \left[\frac{x'_i - x_i}{x_i} \right]^2}{(n - p)}$$

where:

x'_i = Calculated concentration at level i

x_i = Actual concentration of the calibration level i

n = Number of calibration points

p = Number of terms in the fitting equation (average = 1; linear = 2; quadratic = 3)

9.7 See the current Quality Assurance Manual for other equations associated with common calculations.

10.0 QUALITY CONTROL AND METHOD PERFORMANCE

10.1 All analysts must meet the qualifications specified in SOP #030205, *Technical Training and Personnel Qualifications* before approval to perform this method. Analysts must complete an initial demonstration of proficiency before being approved to perform this method. Continuing proficiency must be demonstrated using proficiency testing, laboratory control sample analysis and/or MDL studies. Method performance is assessed per analyst. Updated method performance records are filed and stored in a central location within the department.

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10.1.1 Method 625.1 Demonstration of Capability (DOC) Requirements

10.1.1.1 For the DOC, a QC check sample (LCS) concentrate containing each analyte of interest is prepared in a water miscible solvent. The QC check sample concentrate must be prepared independently from those used for calibration, but may be from the same source as the second-source standard used for calibration verification. The concentrate should produce concentrations of the analytes of interest in water at the midpoint of the calibration range, and may be at the same concentration as the LCS.

10.1.1.2 Prepare four QC check samples by adding an appropriate volume of the concentrate and of the surrogate(s) to each of four aliquots of reagent water and mix well. The volume of reagent water must be the same as the volume that will be used for the sample, blank, and MS/MSD.

10.1.1.3 Extract and analyze the four LCSs samples.

10.1.1.4 Calculate the average percent recovery (\bar{X}) and the standard deviation (s) of the percent recovery for each analyte using the four results.

10.1.1.5 For each analyte, compare s and \bar{X} with the acceptance criteria for precision and recovery presented in Attachment VII. For analytes that are not listed, QC acceptance criteria must be developed by the laboratory.

If s and \bar{X} for all analytes of interest meet the acceptance criteria, system performance is acceptable and analysis of blanks and samples can begin. If any individual s exceeds the precision limit or any individual \bar{X} falls outside the range for recovery, system performance is unacceptable for that analyte.

10.1.1.6 When one or more of the analytes tested fail at least one of the acceptance criteria, repeat the test for only the analytes that failed. If results for these analytes pass, system performance is acceptable and analysis of samples and blanks may proceed. If one or more of the analytes again fail, system performance is unacceptable for the analytes that failed the acceptance criteria. Correct the problem and repeat the test.

10.2 Use the designated Run log to record batch order and standards/reagents used during analysis. See SOP #030201, *Data Handling and Reporting*.

10.3 Batches:

Batches are defined as sets of 1 - 20 samples. Batch analysis must include the following: 1 method blank, 1 Laboratory Control Sample (LCS), 1 Laboratory Control Sample Duplicate (LCSD), 1 Matrix Spike/Spike Duplicate (MS/MSD) (if client has supplied

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sufficient sample volume). All batch information must be maintained in the preparation documentation assigned to the department.

- 10.4 For sample analyzed per the requirements of Method 8000D, the LLOQ (see Section 1.8.2) must be verified at least annually, and whenever significant changes are made to the preparation and/or analytical procedure, to demonstrate quantitation capability at lower analyte concentration levels
- 10.4.1 The LLOQ verification (to be performed after the initial calibration) is prepared by spiking a clean control material with the analyte(s) of interest at 0.5-2 times the LLOQ concentration level(s).
- 10.4.2 The LLOQ check is carried through the same preparation and analytical procedures as environmental samples and other QC samples.
- 10.4.3 It is recommended to analyze the LLOQ verification on every instrument where data is reported; however, at a minimum, the lab must rotate the verification among similar analytical instruments such that all are included within 3 years.
- 10.4.4 Recovery of target analytes in the LLOQ verification must be within established in-house limits or within other such project-specific acceptance limits to demonstrate acceptable method performance at the LLOQ. Until the laboratory has sufficient data to determine acceptance limits, the LCS criteria $\pm 20\%$ (i.e., lower limit minus 20% and upper limit plus 20%) may be used for the LLOQ acceptance criteria.
- 10.5 Method 625.1 Requirements
- 10.5.1 At the beginning of each 12-hour shift during which standards or extracts will be analyzed, perform the tests in this section to verify system performance. If an extract is concentrated for greater sensitivity (e.g., by SIM), all tests must be performed at levels consistent with the reduced extract volume.
- 10.5.2 Inject the DFTPP standard and verify that the criteria are met.
- 10.5.2.1 Analysis of DFTPP, the DDT/Endrin decomposition test (if used), the LCS, and the blank are outside of the 12-hour analysis shift. The total time for DFTPP, DDT/ Endrin, the LCS, the blank, and the 12-hour shift must not exceed 15 hours.
- 10.5.3 The resolution should be verified on the mid-point concentration of the initial calibration as well as the laboratory designated continuing calibration verification level if closely eluting isomers are to be reported (e.g., benzo(b)fluoranthene and benzo(k)fluoranthene). Sufficient gas chromatographic resolution is achieved if the height of the valley between two isomer peaks is less than 50% of the average of the two peak heights.
- 10.5.4 Verify calibration.
- 10.5.5 Verify tailing factor specifications.

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- 10.5.6 Analyze the extract of the LCS at the beginning of analyses of samples in the extraction batch. The LCS must meet the following requirements in section 8.4, and the blank must meet the following requirements before sample extracts may be analyzed.
- 10.5.6.1 Compare the percent recovery (PS) for each analyte with its corresponding QC acceptance criterion in Attachment VII. For analytes of interest not listed in Attachment VII, use the QC acceptance criteria developed for the LCS, or limits based on laboratory control charts. If the recoveries for all analytes of interest fall within their respective QC acceptance criteria, analysis of blanks and field samples may proceed
- 10.5.7 Analyze the extract of the blank at the beginning of analyses of samples in the extraction batch. The blank must meet the requirements in section 8.5 before sample extracts may be analyzed.
- 10.5.7.1 Analyze the blank immediately after analysis of the LCS and prior to analysis of the MS/MSD and samples to demonstrate freedom from contamination.
- 10.5.7.2 If an analyte of interest is found in the blank at a concentration greater than the MDL for the analyte, at a concentration greater than one-third the regulatory compliance limit, or at a concentration greater than one-tenth the concentration in a sample in the extraction batch, whichever is greater, analysis of samples must be halted, and the problem corrected. If the contamination is traceable to the extraction batch, samples affected by the blank must be re-extracted and the extracts re-analyzed. If, however, continued re-testing results in repeated blank contamination, the laboratory must document and report the failures (e.g., as qualifiers on results), unless the failures are not required to be reported as determined by the regulatory/control authority. Results associated with blank contamination for an analyte regulated in a discharge cannot be used to demonstrate regulatory compliance.
- 10.5.8 If DDT and/or endrin are to be determined, the breakdown test must be performed prior to calibration verification. The QC acceptance criteria must be met before analyzing samples for DDE and/ or Endrin. DDT decomposes to DDE and DDD. Endrin decomposes to endrin aldehyde and endrin ketone.
- 10.5.8.1 Both the % breakdown of DDT and of Endrin must be less than 20%, otherwise the system is not performing acceptably for DDT and endrin.
- 10.5.9 The data user should identify the sample and the analytes of interest to be spiked and provide sufficient sample volume to perform MS/MSD analyses. The laboratory must, on an ongoing basis, spike at least 5% of the samples in duplicate from each discharge being monitored to assess accuracy (recovery and precision). If direction cannot be obtained from the data user, the laboratory

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must spike at least one sample in duplicate per extraction batch of up to 20 samples with the analytes in Table 1. Spiked sample results should be reported only to the data user whose sample was spiked, or as requested or required by a regulatory/control authority, or in a permit.

10.5.9.1 If, as in compliance monitoring, the concentration of a specific analyte will be checked against a regulatory concentration limit, the concentration of the spike should be at that limit; otherwise, the concentration of the spike should be one to five times higher than the background concentration (determined below), at or near the midpoint of the calibration range, or at the concentration in the LCS whichever concentration would be larger.

10.5.9.2 Analyze one sample aliquot to determine the background concentration (B) of the each analyte of interest. If necessary, prepare a new check sample concentrate (section 8.2.1) appropriate for the background concentration. Spike and analyze two additional sample aliquots, and determine the concentration after spiking (A1 and A2) of each analyte. Calculate the percent recoveries (P1 and P2) as $100(A1 - B)/T$ and $100(A2 - B)/T$, where T is the known true value of the spike. Also calculate the relative percent difference (RPD) between the concentrations.

10.5.9.3 Compare the percent recoveries (P1 and P2) and the RPD for each analyte in the MS/MSD aliquots with the corresponding QC acceptance criteria in Attachment VII. The laboratory may develop and apply QC acceptance criteria more restrictive than the criteria in Attachment VII, if desired.

10.6 For acceptance criteria for calibration standards, QC samples and field samples and corrective actions, see section 11.0.

11.0 DATA VALIDATION AND CORRECTIVE ACTION

11.1 A successful DFTPP tune must be achieved prior to initial calibration or daily calibration verification. If a tune does not meet the acceptance criteria in section 8.2, then re-inject the tuning solution. If the failure persists, instrument maintenance or detector adjustment is required. The instrument is equipped with detector adjustments in routines called "Autotunes" that can make minor adjustments to m/z ratios and detector setting and can align the analytical system to return the system to peak performance. If after performing the Autotune routine, the injected tuning standard still fails, the system may require injector and/or detector cleaning, column cutting or replacement, injection liner cleaning or replacement, or other maintenance as specified by the manufacturer. Following successful tuning of the DFTPP solution, the DDT degradation and Benzidine/Pentachlorophenol tailing must be assessed. If either fail to meet the required acceptance criteria, instrument maintenance is required. The DDT degradation is most likely an inlet or column condition and corrective action entails clipping 6-12" from the injector end of the column, changing the injection port liner, possibly changing the gold

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inlet seal and re-injecting the tuning solution. The tailing issue is most likely caused by the same type of inlet issues and the same corrective action steps should occur when the tailing criteria is not met. Tailing may also be caused by incorrect column positioning in the inlet and the correct position of the column should be verified prior to performing more involved corrective action processes.

A successful instrument tune, including degradation and tailing acceptability, must be achieved prior to the analysis of calibration standards and sample extracts.

Method 625.1: The DFTPP spectrum may be evaluated by summing the intensities of the m/z's across the GC peak, subtracting the background at each m/z in a region of the chromatogram within 20 scans of but not including any part of, the DFTPP peak. The DFTPP spectrum may also be evaluated by fitting a Gaussian to each m/z and using the intensity at the maximum for each Gaussian or by integrating the area at each m/z and using the integrated areas. Other means may be used for evaluation of the DFTPP spectrum so long as the spectrum is not distorted to meet the criteria in Table 8.2d of this SOP.

The tailing factor for benzidine and pentachlorophenol must be <2; otherwise, adjust instrument conditions and either replace the column or break off a short section of the front end of the column, and repeat the test. Once the scan conditions are established, they must be used for analyses of all standards, blanks, and samples.

11.2 Initial or Continuing Calibration:

Method 8270C, SM 6410B & Methods 625 and 625.1: If the calibration curve or daily calibration verification fails to meet the applicable method verification criteria for RSD, the analyst MUST use linear regression or quadratic curve fit. Quadratic models cannot be used to extend the calibration range or bypass instrument maintenance. If the method criteria are still not met when using the alternate curve fits, samples may not be quantitated using the calibration curve and a new calibration curve must be analyzed. Instrument maintenance and/or new standard preparation may also be required prior to the analysis of the new calibration curve. Following maintenance, the new calibration curve can be generated. The system may require injector and/or detector cleaning, column cutting or replacement, injection liner cleaning or replacement, or other maintenance as specified by the manufacturer. Additional actions that can be taken to address failures in calibration are included in section 8.3.

Method 8270D: Due to the large number of compounds that may be analyzed by this method, some compounds in the initial and/or daily calibration verification will fail to meet the initial and continuing calibration acceptance criteria. For these instances, failing compounds may not be critical to specific project needs and therefore may be utilized as qualified data or estimated values for screening purposes. If more than 10% of the compounds in the initial or continuing calibration exceed the 20% RSD limit and/or do not meet the minimum correlation coefficient (0.990) for alternate curve fits, then the chromatographic system is considered too reactive for analysis. Instrument maintenance must be performed and the calibration process must be repeated. The system may require injector and/or detector cleaning, column cutting or replacement, injection liner cleaning or replacement, or other maintenance as specified by

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the manufacturer. Additional actions that can be taken to address failures in calibration are included in section 8.3.

TNI: If the ICV or CCV results obtained are outside the established acceptance criteria and analysis of a second consecutive (immediate) calibration verification fails to produce results within acceptance criteria, corrective actions shall be performed. The laboratory shall demonstrate acceptable performance after the final round of corrective action with two consecutive calibration verifications, or a new initial instrument calibration shall be performed.

Method 8000D: To determine calibration function acceptability, refit the initial calibration data back to the calibration model and calculate %Error (see Section 9.5). Percent error between the calculated and expected amounts of an analyte must be $\leq 30\%$ for all standards. For some data uses, $\leq 50\%$ may be acceptable for the lowest calibration point.

Method 625.1: The RF or calibration curve must be verified immediately after calibration and at the beginning of each 12-hour shift, by analysis of a standard at or near the concentration of the mid-point calibration standard. The standard(s) must be obtained from a second manufacturer or a manufacturer's batch prepared independently from the batch used for calibration.

When one or more analytes fail acceptance criteria, analyze a second aliquot of the calibration verification standard and compare ONLY those analytes that failed the first test with their respective acceptance criteria. If these analytes now pass, system performance is acceptable and analysis of samples may continue. A repeat failure of any analyte that failed the first test, however, will confirm a general problem with the measurement system. If this occurs, repair the system and repeat the test, or prepare a fresh calibration standard and repeat the test. If calibration cannot be verified after maintenance or injection of the fresh calibration standard, re-calibrate the instrument.

- 11.3 The method blank must be extracted and analyzed with each set of samples at a frequency of at least 5% and must be free of the analytes of interest at the method detection limit. If the method blank contains target analytes at a detectable concentration, it may be necessary to analyze a solvent blank to demonstrate the source of the contamination is not carryover from standards or lingering field sample artifacts. Following verification that the analytical system is free from interferences, the method blank can be re-analyzed once. A passing method blank must be analyzed before any samples are analyzed; otherwise corrective action is required. Corrective action can take the form of checking the original calculations to ensure accuracy or instrument maintenance (i.e. column clipping or changing, inlet liner cleaning/replacement, etc.) or re-calibration. The surrogate recoveries in the method blank must meet the established control criteria (see the LIMS). If not, the recovery demonstrates an analytical system that is in an out-of-control mode and the batch must be re-extracted/re-analysis unless directed otherwise by the client.

General guidelines for qualifying sample results with regard to method blank quality are as follows:

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- If the method blank concentration is less than the MDL and sample results are greater than the RL, then no qualification is required.
- No qualification is necessary when an analyte is detected in the method blank but not in the associated samples.
- If the concentration in a sample is more than ten times the concentration in the method blank, then no qualification is required.
- If the method blank concentration is greater than the MDL but less than the RL and sample results are greater than the MDL, then qualify associated sample results to indicate that analyte was detected in the method blank.
- If the method blank concentration is greater than the RL, further corrective action and qualification is required. An analyst should consult their supervisor for further instruction.

Method 8000D: When samples that are extracted together are analyzed on separate instruments or in separate analytical shifts, the method blank associated with those samples (e.g., extracted with the samples) must be analyzed on at least one of those instruments. A solvent blank must be analyzed on all other instruments on which the set of samples was analyzed to demonstrate the instrument is not contributing contaminants to the samples. At least one method blank or instrument blank must be analyzed on every instrument after calibration standard(s) and prior to the analysis of any samples.

When sample extracts are subjected to cleanup procedures, the associated method blank must also be subjected to the same cleanup procedures.

Results of the method blank should be less than the LLOQ for the analyte or less than the level of acceptable blank contamination specified in the approved QAPP or other appropriate systematic planning document. Blanks are generally considered to be acceptable if target analyte concentrations are less than one-half the LLOQ or are less than project-specific requirements.

When new reagents or chemicals are received, the lab should monitor the blanks associated with samples for any signs of contamination. It is not necessary to test every new batch of reagents or chemicals prior to sample preparation if the source shows no prior problems. However, if reagents are changed during a preparation batch, separate blanks need to be prepared for each set of reagents.

Method 625.1: If an analyte of interest is found in the blank at a concentration greater than the MDL for the analyte, at a concentration greater than one-third the regulatory compliance limit, or at a concentration greater than one-tenth the concentration in a sample in the extraction batch, whichever is greater, analysis of samples must be halted, and the problem corrected. If the contamination is traceable to the extraction batch, samples affected by the blank must be re-extracted and the extracts re-analyzed. If, however, continued re-testing results in repeated blank contamination, the laboratory must document and report the failures (e.g., as qualifiers on results), unless the failures are not required to be reported as determined by the regulatory/control authority. Results associated with blank contamination for an analyte regulated in a discharge cannot be used to demonstrate regulatory compliance. QC failures do not relieve a discharger or permittee of reporting timely results.

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11.4 Second Source Calibration Verification

Method 8270D: The value determined from the second source calibration verification (SSCV) should be within $\pm 30\%$ drift of the expected concentration. Alternative recovery limits may be appropriate based on analyte performance and project specific requirements. Quantitative analysis cannot proceed for analytes that fail the SSCV, except for screening purposes only.

Method 8270C/625/SM6410B: The value determined from the second source calibration verification (SSCV) must be $\leq 50\%$ drift for non-CCC compounds; $\leq 20\%$ drift for CCC compounds and meet the minimum response factor criteria for SPCC compounds as in the initial calibration construction. Historical performance weighs heavily in the acceptability of those analytes that are known to perform poorly. Corrective action can take the form of checking the original calculations to ensure accuracy, re-analysis of the SSCV to verify initial results, instrument maintenance (i.e. column clipping or changing, inlet liner cleaning/replacement, etc.) or re-calibration.

STATE NOTE: If the samples are analyzed in conjunction with South Carolina DHEC, alternate recovery limits can only be used if they are more stringent than method criteria.

Method 625.1: The RF or calibration curve must be verified immediately after calibration and at the beginning of each 12-hour shift, by analysis of a standard at or near the concentration of the mid-point calibration standard. The standard(s) must be obtained from a second manufacturer or a manufacturer's batch prepared independently from the batch used for calibration. Traceability must be to a national standard, when available. Include the surrogates in this solution. It is necessary to verify calibration for the analytes of interest only.

When one or more analytes fail acceptance criteria, analyze a second aliquot of the calibration verification standard and compare ONLY those analytes that failed the first test with their respective acceptance criteria. If these analytes now pass, system performance is acceptable and analysis of samples may continue. A repeat failure of any analyte that failed the first test, however, will confirm a general problem with the measurement system. If this occurs, repair the system and repeat the test, or prepare a fresh calibration standard and repeat the test. If calibration cannot be verified after maintenance or injection of the fresh calibration standard, re-calibrate the instrument.

- 11.5 Surrogates: If the surrogate recoveries in the samples do not fall within the appropriate acceptance criteria presented in the LIMS, ensure that there were no errors in calculations, internal standard, or instrument performance. If the recovery of any one surrogate is critically low ($<10\%$) or critically high ($>200\%$), then the sample must be re-extracted unless otherwise directed by the client or a clear, documented matrix interference is exhibited. If two of three acid and two of three base/neutral surrogates are within acceptance criteria, then the sample may be reported. If re-extraction is required and there is no more sample available or it has exceeded holding times, the data must be flagged with a "J1" (surrogate high) or a "J2" (surrogate low). See SOP #030201, *Data Handling and Reporting*, for more information on qualifying out of control data.

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STATE NOTE: If the sample is from North Carolina, two of the three acid and two of the three base/neutral surrogates must pass. If two of the three acid or base/neutral surrogates fail, the sample must be re-extracted. For all other samples, one of the three surrogates must pass from both the acid and base/neutral sides. If any surrogates have less than a 10% or greater than 200% recovery, and matrix interferences are not confirmed as the cause of the failure, the sample must be re-extracted.

STATE NOTE: If field samples are analyzed in conjunction with the Ohio VAP program, surrogate outliers in batch QC samples, including the method blank, LCS/LCSD, MS/MSD require re-extraction of the entire batch, if sufficient volume has been submitted by the client and an obvious matrix interferent is not present.

STATE NOTE: If the sample is analyzed in conjunction with the Ohio VAP, corrective action for failing QC (i.e. method blank, surrogate, MS/MSD, LCS/LCSD, ISTD, etc.) must be performed prior to flagging data, if sufficient sample volume was submitted by the client. Corrective action can include re-analysis, if instrument malfunction is suspected, or re-preparation and re-analysis, if the failure is suspected as either extraction or sample related.

Method 625.1: The laboratory must evaluate surrogate recovery data in each sample against its in-house surrogate recovery limits. The laboratory may use 60–140% as interim acceptance criteria for recoveries for surrogates not listed in Attachment VIII. At least 80% of the surrogates must meet the 60–140% interim criteria until in-house limits are developed. Alternatively, surrogate recovery limits may be developed from laboratory control charts, but such limits must be at least as restrictive as those in Attachment VIII. Spike the surrogates into all samples, blanks, LCSs, and MS/MSDs. Compare surrogate recoveries against the QC acceptance criteria in Attachment VIII and/or those developed in-house. If any recovery fails its criteria, attempt to find and correct the cause of the failure.

The large number of analytes tested in performance tests in this method present a substantial probability that one or more will fail acceptance criteria when many analytes are tested simultaneously, and a retest is allowed if this situation should occur. If, however, continued re-testing results in further repeated failures, the laboratory must document and report the failures (e.g., as qualifiers on results), unless the failures are not required to be reported as determined by the regulatory/control authority. Results associated with a QC failure for an analyte regulated in a discharge cannot be used to demonstrate regulatory compliance. QC failures do not relieve a discharger or permittee of reporting timely results.

- 11.6 Internal Standard: The internal standard area counts must be monitored for all ICVs. ISTDs must recover within –50% to +100% of the area counts from the internal standard area counts of the midpoint standard of the most recent initial calibration sequence. If any internal standard response is beyond the acceptable recovery, corrective action is required. Corrective action can take the form of checking the original calculations to

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ensure accuracy, re-analysis of the ICV to verify initial results, instrument maintenance (i.e. column clipping or changing, inlet liner cleaning/replacement, etc.) or re-calibration.

The internal standard responses and retention times in the check calibration standard must be evaluated immediately after or during data acquisition. If the retention time for any internal standard changes by more than 30 seconds from the last calibration verification, the chromatographic system must be inspected for malfunctions and corrections must be made, as required. When corrections are made, re-analysis of the CCV or a complete re-calibration is necessary, depending on the impact of the correction on the analytical system.

Internal standards must be monitored for each sample. ISTDs in samples must meet the -50% to +100% criteria when compared to the ISTDs in the daily CCV or mid-level of the calibration curve, on 12h shifts when full calibration is performed. Possible corrective actions include: re-analysis, if instrument malfunction is suspected, or re-preparation and re-analysis, if the failure is suspected as either extraction or sample related. If the sample has an obvious matrix interferent and the internal standard recovery is greater than +100%, the sample can be diluted (if acceptable reporting limits can be achieved) to minimize the interference or the sample must be re-extracted and re-analyzed to confirm the original results. ISTD failures <50% of daily ICV may be reported if all corresponding analytes are BDL as the high quantitation bias created by the reduced internal standard recovery has not adversely impacted the reported analyte results.

Method 625.1: The responses (GC peak heights or areas) of the internal standards in the calibration verification must be within 50% to 200% (1/2 to 2x) of their respective responses in the mid-point calibration standard. If they are not, repeat the calibration verification test or perform and document system repair. Subsequent to repair, repeat the calibration verification. If the responses are still not within 50% to 200%, re-calibrate the instrument and repeat the calibration verification test.

The responses (GC peak heights or areas) of each internal standard in each sample, blank, and MS/MSD must be within 50% to 200% (1/2 to 2x) of its respective response in the LCS for the extraction batch. If, as a group, all internal standards are not within this range, perform and document system repair, repeat the calibration verification, and re-analyze the affected samples. If a single internal standard is not within the 50% to 200% range, use an alternate internal standard for quantitation of the analyte referenced to the affected internal standard. It may be necessary to use the data system to calculate a new response factor from calibration data for the alternate internal standard/analyte pair. If an internal standard fails the 50–200% criteria and no analytes are detected in the sample, ignore the failure or report it if required by the regulatory/control authority.

- 11.7 LCS/LCSD and MS/MSD: The laboratory control sample, laboratory control sample duplicate, matrix spike and matrix spike duplicate recoveries must be evaluated against the acceptance criteria given in the LIMS. The LCS/LCSD and MS/MSD are spiked with the same list of compounds for which the instrument is calibrated. Due to the large number of compounds analyzed using these methods, it is statistically likely that accuracy and precision failures will occur.

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LCS or LCSD samples that do not pass the acceptable QC criteria must be re-analyzed. LCS/LCSD failures must meet the marginal exceedance criteria below. The normal compound list for 8270/625 typically contains 90 analytes; therefore only 5 analytes can be considered as marginally exceeding the acceptance criteria. If more than 5 failures occur or if the failures demonstrate a pattern that is causing the outliers, the entire sample batch with associated QC must be re-extracted and re-analyzed. Marginal exceedances must be random events.

Upper and lower marginal exceedance (ME) limits are established by +/- four times the standard deviation of historical accuracy data and the number of marginal exceedances allowed is based on the number of analytes spiked in the LCS.

Number of allowable marginal exceedances:

90 analytes, 5 analytes allowed in the ME limit
71 – 90 analytes, 4 analytes allowed in the ME limit.
51 – 70 analytes, 3 analytes allowed in the ME limit.
31 – 50 analytes, 2 analytes allowed in the ME limit.
11 – 30 analytes, 1 analyte allowed in the ME limit.
< 11 analytes, no analyte allowed in the ME limit.

If the MS/MSD fails to meet recovery limits listed in the LIMS, the data on the unspiked field sample for that compound must be flagged with a “J5” (high recovery) or a “J6” (low recovery). If the MS/MSD fail to pass precision limits (%RSD), the data on the unspiked field sample for that compound must be flagged with a “J3” qualifier.

Method 8000D: If, as in compliance monitoring, the concentration of a specific analyte in the sample is being checked against a regulatory concentration limit or action level, the spike should be at or below the limit, or 1 - 5 times the background concentration (if historical data are available), whichever concentration is higher. If historical data are not available, a background sample of the same matrix from the site may be submitted for matrix spiking purposes to ensure that high concentrations of target analytes and/or interferences will not prevent calculation of recoveries. If the background sample concentration is very low or non-detect, a spike of greater than 5 times the background concentration is still acceptable. To assess data precision with duplicate analyses, it is preferable to use a low concentration field sample to prepare a MS/MSD for organic analyses. This spiking procedure will be performed when project-specific instructions are received from the client.

If the concentration of a specific analyte in a sample is not being checked against a limit specific to that analyte, then the analyst may spike the matrix spike or MS/MSD sample(s) at the same concentration as the reference sample at 20 times the estimated LLOQ in the matrix of interest, or at a concentration near the middle of the calibration range. It is suggested that a background sample of the same matrix from the site be submitted as a sample for matrix spiking purposes. NOTE: Preparing the spiking solution from the same source as the calibration standards helps minimize additional variability due to differences between sources. Typically, spiking concentrations are near the middle of the calibration range.

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To develop precision and bias data for the spiked compounds, the analyst has two choices: analyze the original sample, and an MS/MSD pair; or analyze the original sample, a duplicate sample, and one spiked sample. If samples are not expected to contain the target analytes of concern, then the laboratory may use a MS/MSD pair. If samples are expected to contain the target analytes of concern, then the laboratory may use one matrix spike and a duplicate analysis of an unspiked field sample as an alternative to the MS/MSD pair.

The laboratory should use 70 - 130% as interim acceptance criteria for recoveries of spiked analytes, until in-house LCS limits are developed. Where in-house limits have been developed for matrix spike percent recoveries, the LCS results should be similar to or tighter than those limits, as the LCS is prepared in a clean matrix.

STATE NOTE: For South Carolina or Ohio VAP samples, marginal exceedances do not apply. All outliers in QC require corrective action when possible and the data must be flagged when necessary.

STATE NOTE: For all samples from South Carolina, the LCS/LCSD recovery must be evaluated within 70-130% and the MS/MSD recoveries must be within in-house derived recovery limits; however, if the limits given in Method 625 Table 6 are more stringent, then those limits must be used. The following are the current limits:

Parameter	LCS/LCSD	MS/MSD
1,2,4-TRICHLOROBENZENE	70 - 130%	44 - 104%
2,4,6-TRICHLOROPHENOL	70 - 130%	37 - 132%
2,4-DICHLOROPHENOL	70 - 130%	39 - 117%
2,4-DIMETHYLPHENOL	70 - 119%	32 - 119%
2,4-DINITROPHENOL	70 - 130%	10 - 141%
2,4-DINITROTOLUENE	70 - 130%	45.4 - 139%
2,6-DINITROTOLUENE	70 - 130%	50 - 134%
2-CHLORONAPHTHALENE	70 - 118%	60 - 118%
2-CHLOROPHENOL	70 - 130%	23 - 111%
2-NITROPHENOL	70 - 130%	29 - 135%
3,3-DICHLOROBENZIDINE	70 - 130%	10 - 143%
4,6-DINITRO-2-METHYLPHENOL	70 - 130%	10 - 143%
4-BROMOPHENYL-PHENYLETHER	70 - 127%	53 - 127%
4-CHLORO-3-METHYLPHENOL	70 - 130%	38.4 - 123%
4-CHLOROPHENYL-PHENYLETHER	70 - 130%	49.8 - 127%
4-NITROPHENOL	70 - 130%	10 - 52.8%
ACENAPHTHENE	70 - 130%	47 - 141%
ACENAPHTHYLENE	70 - 130%	40 - 132%
ANTHRACENE	70 - 130%	44.5 - 130%
BENZO(A)ANTHRACENE	70 - 130%	46.4 - 130%
BENZO(A)PYRENE	70 - 130%	34.6 - 129%
BENZO(B)FLUORANTHENE	70 - 130%	36.3 - 137%
BENZO(G,H,I)PERYLENE	70 - 130%	10 - 140%
BENZO(K)FLUORANTHENE	70 - 130%	30.3 - 136%
BENZYL BUTYL PHTHALATE	70 - 130%	44.8 - 152%

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Parameter	LCS/LCSD	MS/MSD
BIS(2-CHLORETHOXY)METHANE	70 - 130%	39.2 - 128%
BIS(2-CHLOROETHYL)ETHER	70 - 130%	14.8 - 131%
BIS(2-CHLOROISOPROPYL)ETHER	70 - 130%	36 - 117%
BIS(2-ETHYLHEXYL)PHTHALATE	70 - 130%	12.6 - 153%
CHRYSENE	70 - 130%	40 - 133%
DIBENZ(A,H)ANTHRACENE	70 - 130%	10 - 143%
DIETHYL PHTHALATE	70 - 114%	50.4 - 114%
DIMETHYL PHTHALATE	70 - 112%	9.1 - 112%
DI-N-BUTYL PHTHALATE	70 - 118%	53.3 - 118%
DI-N-OCTYL PHTHALATE	70 - 130%	13.3 - 146%
FLUORANTHENE	70 - 130%	42.9 - 137%
FLUORENE	70 - 121%	59 - 121%
HEXACHLORO-1,3-BUTADIENE	70 - 116%	28.9 - 116%
HEXACHLOROBENZENE	70 - 130%	47 - 121%
HEXACHLOROCYCLOPENTADIENE	70 - 130%	10 - 128%
HEXACHLOROETHANE	70 - 113%	40 - 109%
INDENO(1,2,3-CD)PYRENE	70 - 130%	10 - 141%
ISOPHORONE	70 - 130%	31.9 - 118%
NAPHTHALENE	70 - 130%	29 - 115%
NITROBENZENE	70 - 130%	35 - 118%
N-NITROSODI-N-PROPYLAMINE	70 - 130%	35.4 - 129%
PENTACHLOROPHENOL	70 - 130%	14 - 128%
PHENANTHRENE	70 - 130%	54 - 120%
PHENOL	70 - 112%	10 - 55.7%
PYRENE	70 - 115%	52 - 115%

Method 625.1: For LCS analyses, repeat the test only for those analytes that failed to meet the acceptance criteria (PS). If these analytes now pass, system performance is acceptable and analysis of blanks and samples may proceed. Repeated failure, however, will confirm a general problem with the measurement system. If this occurs, repeat the test using a fresh LCS or an LCS prepared with a fresh QC check sample concentrate, or perform and document system repair. Subsequent to analysis of the LCS prepared with a fresh sample concentrate, or to system repair, repeat the LCS test. If failure of the LCS indicates a systemic problem with samples in the batch, re-extract and re-analyze the samples in the batch.

The large number of analytes tested in performance tests in this method present a substantial probability that one or more will fail acceptance criteria when many analytes are tested simultaneously, and a retest is allowed if this situation should occur. If, however, continued re-testing results in further repeated failures, the laboratory must document and report the failures (e.g., as qualifiers on results), unless the failures are not required to be reported as determined by the regulatory/control authority. Results associated with a QC failure for an analyte regulated in a discharge cannot be used to demonstrate regulatory compliance. QC failures do not relieve a discharger or permittee of reporting timely results.

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NOTE: To maintain the validity of the test and re-test, system maintenance and/or adjustment is not permitted between the pair of tests.

For MS/MSD analyses, compare the percent recoveries (P1 and P2) and the RPD for each analyte in the MS/MSD aliquots with the corresponding QC acceptance criteria in Attachment VII. A laboratory may develop and apply QC acceptance criteria more restrictive than the criteria in Attachment VII, if desired.

If any individual P falls outside the designated range for recovery in either aliquot, or the RPD limit is exceeded, the result for the analyte in the unspiked sample is suspect. The large number of analytes tested in performance tests in this method present a substantial probability that one or more will fail acceptance criteria when many analytes are tested simultaneously, and a retest is allowed if this situation should occur. If, however, continued re-testing results in further repeated failures, the laboratory must document and report the failures (e.g., as qualifiers on results), unless the failures are not required to be reported as determined by the regulatory/control authority. Results associated with a QC failure for an analyte regulated in a discharge cannot be used to demonstrate regulatory compliance. QC failures do not relieve a discharger or permittee of reporting timely results.

- 11.8 Calibration Range: For any compound found in a sample at a level above the highest standard, the extract must be diluted and re-analyzed to allow quantitation within the range of instrument calibration. Whenever an extract dilution is made, the appropriate amount of internal standard must be added to bring the ISTD concentrations back to the concentrations consistent with the calibration standards.

STATE NOTE: For all samples analyzed from Minnesota, the reporting limit must be verified at least monthly, with each new initial calibration, or when there has been significant change to the instrument (column replacement, cleaning source, etc.) whichever is more frequent. The reporting limit verification can be performed by either re-injecting the low standard or by re-processing the low standard that was analyzed in the calibration curve. The reporting limit verification (RLV) must recovery within $\pm 40\%$ of the expected concentration. If this criterion is not met, the RLV may be re-analyzed once, instrument maintenance can be performed, a higher concentration standard can be injected, or a new calibration curve must be generated. If a higher concentration standard is utilized, the reporting limit must be raised to the higher level verified.

- 11.9 All data must undergo a primary review by the analyst. The analyst must check the performance of the initial calibration, check standard, and continuing calibrations to ensure that they meet the criteria of the method. The analyst must review any sample that has quantifiable compounds and make sure that they have been confirmed, if necessary. The analyst must also verify that reported results are derived from quantitation between the RL and the highest standard of the initial calibration curve. All calculations must be checked (any dilutions, %solids, etc.). Data must be checked for the presence or absence of appropriate flags. Comments must be noted when data is flagged.

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- 11.10 All data must undergo a second analyst review. The analyst checking the data must check the performance of the initial calibration, mid-point check standard, and continuing calibrations to ensure that they meet the criteria of the method.
 - 11.10.1 The analyst should must review any sample that has quantifiable compounds and make sure that they have been confirmed.
 - 11.10.2 All calculations must be checked.
 - 11.10.3 All surrogate recoveries must be checked to ensure that they are within QC acceptance criteria or that corrective action has occurred.
 - 11.10.4 Blanks must be free of all interfering peaks.
 - 11.10.5 Quality control criteria must be checked for the LCS, LCSD, MS, and MSD.
 - 11.10.6 Data must be checked to determine the need for appropriate flags. Comments are noted when results are flagged.
 - 11.10.7 The reviewer must verify all reported results are derived from analytical results that are either above the reporting limit/MDL, as applicable, and below the highest standard of the initial calibration curve.
 - 11.10.8 All manual integrations must be verified through checking the before/after shot of the sample, method blank, and/or QC (LCS/LCSD/MS/MSD).
 - 11.10.9 All multipliers/dilutions must be verified on the quant report and must agree with the information provided on the injection log.
 - 11.10.10 Retention times of the samples must be compared to that of the calibration standard. Random spot checking of 10% of the data should be sufficient.
 - 11.10.11 Verify linear regression by reviewing the calibration curve printout.
 - 11.10.12 See SOP #030201, *Data Handling and Reporting* and SOP #030227, *Data Review*.
- 11.11 Data that does not meet acceptable QC criteria may be acceptable for use in certain circumstances.
 - 11.11.1 If a method blank contains an amount of target analyte, but all samples are non-detected, the data may be reported with a "B3" flag. If a method blank contains an amount of target analyte, but the samples contain analyte at a level that is 10 times the level present in the method blanks, the data may be reported with a "B" flag.

STATE NOTE: The Ohio VAP program or South Carolina DHEC does not accept data released using the 10X criteria for method blank contamination as noted in section 11.11.1.

- 11.11.3 If a calibration verification standard is above the acceptable QC criteria and all samples being bracketed are below the reporting limit, the data is acceptable based

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on a high calibration bias with undetectable levels in the field samples. Any positive samples require re-analysis. If MDL reporting is required by the client, reported samples must calculate <MDL to be considered not impacted by the high bias.

11.11.4 If the surrogate exhibits high recovery in the field samples and the target analytes in the field samples are below the reporting limit, the data may be released with a J1 qualifier indicating the high bias. If the QC samples (LCS, LCSD, MS, MSD) exhibit a high bias in the surrogate and the field samples are below the reporting limit for the target analyte, the data may be released with a J1 qualifier.

11.11.5 If the target analyte spiked in the quality control samples (LCS, LCSD, MS, MSD) exhibits high recovery and the target analytes in the field samples are below the reporting limit, the data may be released with a J4 qualifier indicating the high bias.

11.11.6 If the target analyte spiked into the QC pair (LCS/LCSD, MS/MSD) exhibit acceptable recoveries, but high calculated RPD values for precision, target analytes in the field sample are flagged with a J3 for the precision beyond acceptable quality control limits.

11.11.7 Sample results can be qualified and possible bias is narrated per the ESC SOP #030201, *Data Handling and Reporting*.

11.11.7.1 For samples analyzed per the requirements of Method 8000D, reported concentrations of target analytes between the MDL and the LLOQ must be qualified as estimated.

12.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

12.1 The EPA requires that laboratory waste management practice to be conducted consistent with all applicable federal and state laws and regulations. Excess reagents, samples and method process wastes must be characterized and disposed of in an acceptable manner. See *ESC Waste Management Plan*.

12.2 See SOP #030302, *Environmental Sustainability & Pollution Prevention*.

13.0 METHOD MODIFICATIONS/CLARIFICATIONS

13.1 The **Missouri Department of Natural Resources** requires that **DRO** be analyzed by GC/MS. Tuning and frequency requirements are the same as 8270C, omitting DDT, pentachlorophenol, and benzidine assessments. Extract samples the same as 8270PAH using the appropriate extraction method. Only base/neutral surrogates are needed. GC/MS mass range should be 35-550amu. Prepare a five-point calibration curve with 1:1 unleaded gasoline and #2 diesel fuel at 10,000 µg/mL each in methylene chloride. Calibration standards range from 200 to 10,000ug/mL for concentrated soil or 1L water extractions and calibration levels for EPA 3511 extracted water samples and non-concentrated Soil range from 5-200ppm from a 200ppm intermediate. Retention time windows are set using C₁₀, C₂₁, and C₃₅. For DRO, set RT 0.1 minutes after C₁₀ to 0.1 minutes after C₂₁. For ORO, set RT 0.1 minutes after C₂₁ to 0.1 minutes after C₃₅. Verify RT windows daily (24 hours) by running component standard. Quantitate using baseline-

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to baseline, not valley-to-valley. The total ion chromatogram must be used to quantitate. DRO is quantitated using external standard method. The response factor determined for DRO (C₁₀-C₂₁) **must** be used for C₂₁-C₃₅. Subtract area for any internal standard and surrogates. %RSD <20. Run a CCV every 12 hours near mid-point of calibration, %D <20. Run a method blank, LCS and MS/MSD every extraction batch. May re-process files to quantitate PAH analytes, if needed. Quantitation of DRO must be performed using the external standard process.

- 13.2 EPA method 625 employs the use of 2 separate packed GC columns for base/neutral and acidic analyte separations. Modern capillary column technology employs a single column that provides sufficient separatory abilities for use in this analytical process as is demonstrated in EPA method 8270C.
- 13.3 Modifications to this method are noted in the body of the text as state notes. Compliance analyses performed in conjunction with specific state requirements must be performed as noted within the specific state(s) note listed.
- 13.4 Adjustments to the concentrations of standards/spiking solutions, standards providers, and quality control samples are subject to change to better meet client/project/regulatory needs or to improve laboratory method performance.
- 13.5 The reduction of the size of the field sample used in this procedure is performed in accordance with section 7.1 of the published EPA 3510C/625method. The reduction in volume extracted along with increased sensitivity at detection and/or analysis of the resulting extract using large volume injection (>5uL) on each GCMS allows for low detection limits typical of those obtained using a 1L extraction. Complete method validation is performed for each method prior to utilizing the reduced volume extraction. This validation is maintained by the Regulatory Affairs Department and is regularly verified using LCS/LCSD, MDL studies and DOCs.
- 13.6 **Low level NDMA and 1,4-Dioxane by SIM scan/isotope dilution.** Tuning and frequency requirements are the same as 8270C. Extract samples the same as 8270BNA using the appropriate extraction method. 250ng of N-nitrosodimethylamine-d₆ or 1,4Dioxane-d₈ is added to each sample per every 0.5mL of final extract volume prior to extraction resulting in a true value of 500ppb in extract. Only base/neutral surrogates monitoring is necessary. GC/MS is set to scan for masses 42, 43, 46, 48, 54, 74, 80, 82, 115, 128, 150 and 152 for NDMA-d₆ or masses 57, 58, 62, 64, and 88 for 1,4-Dioxane-d₈ in SIM mode. Calibrate at least 5 points using 8270BNA mega mix or 1,4-Dioxane ICAL standard. 500ng of N-nitrosodimethylamine-d₆ is added per every 1mL of calibration standard to each level of the calibration resulting in a true value of 500ppb Calibration standards range from 5ppb to 10,000ppb for 3510RV extracted water samples. Quantitate using Chemstation auto-integration software unless a significant discrepancy is noted in which case manually adjust integrations to best represent the calibration. Select ion monitoring should be used for acquisition and quantitation. NDMA and 1,4-Dioxane are quantitated using the isotope dilution method as described in 8000C. The %RSD determined for NDMA RFs **must** be <15% in order to use the average of response factors for quantitation, otherwise linear regression is to be used. Run a DFTPP tune and CCV every 12 hours near mid-point of calibration, %Diff must be <20%

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for the calibration to be deemed in control and sample analysis to proceed. Run a method blank, LCS and LCSD with every extraction batch. MS/MSDs will be processed with batches when requested by the client as matrix spiking and duplication does not yield reliable precision data when analyzed by the isotope dilution method. Quantitation of low level NDMA and 1,4-Dioxane should be performed using the isotope dilution process.

14.0 REFERENCES

- 14.1 *Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)*, SW846 Method 8270C, Revision 3, December 1996.
- 14.2 *Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)*, SW846 Method 8270D, Revision 4, February 2007.
- 14.3 *Determinative Chromatographic Separations*, SW846 Method 8000B, Revision 2, December 1996.
- 14.4 *Determinative Chromatographic Separations*, SW846 Method 8000C, Revision 3, March 2003.
- 14.5 *Determinative Chromatographic Separations*, SW846 Method 8000D, Revision 4, July 2014.
- 14.6 *Base/Neutrals and Acids*, 40 CFR Part 136, Appendix A, EPA Method 625, October 1991.
- 14.7 *Extractable Base/Neutrals and Acids*, Standard Methods for the Examination of Water and Wastewater, Method 6410B-2000.
- 14.8 *Extractable Base/Neutrals and Acids*, Standard Methods for the Examination of Water and Wastewater, Method 6410B-1997 (20th Ed).
- 14.9 *Base/Neutrals and Acids by GC/MS*, EPA Method 625.1, Federal Register, Volume 82, Number 165, August 28, 2017.

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Attachment I: Revision History

Current Version:

Version	Date	Description of Revisions
26	3/22/2018	Technical and quality review and update. Added 625.1 requirements. Revised SOP Title and Sections 1.8.1, 2.11, 8.2, 8.4.1, 8.4.6, 9.7, 10.6, 11.1, 11.2, 11.3, 11.4, 11.5, 11.6, and 11.7. Added Sections 2.12, 9.6, 10.1.1 and all subsections, 10.5 and all subsections, 14.9, Attachment VII, and Attachment VIII.

Superseded Versions:

This document supersedes the following:

Version	Date	Description of Revisions
0	4/27/95	Origination
1	7/13/95	
2	8/22/96	
3	8/20/99	
4	4/18/00	
5	8/21/00	
6	12/20/00	
7	9/3/01	
8	7/30/02	
9	7/9/03	
10	3/25/04	
11	8/7/06	Technical and Quality Review and update.
12	2/11/09	Addition of 8270D requirements; Addition of State Notes; Update of standards information; Technical and Quality Review and update. Ohio VAP approval 2/11/09.
13	11/23/10	Technical and Quality Review and update. Revised sections 2.1, 2.10, 4.2 through 4.6, 7.1, 7.6, 7.8, 7.10.2, 7.12, 7.13, 8.3, 8.6, 9.3, 9.4, 9.5, 9.10 through 9.13, 11.3, 11.6, 12.1; Added sections 2.27 through 2.30, 4.7, 7.14, state note following section 11.9, 11.10, and 13.4; Removed section 1.2.
14	2/24/12	Technical and Quality Review and update. Revised sections 2.1, 4.2, 4.5, 5.3, 6.1.4, 6.2, 7.8, 7.9, 7.10, 7.13, 8.2, 8.3, 8.5, 8.6, 8.8, 11.2, 11.10, 13.1 and Attachment IV; Added state note to section 1.0; Added sections 1.8.1, 2.31, 2.32, and 13.5.
15	6/12/12	Technical and Quality Review and update. Revised sections 2.1, 7.9, 7.10, 8.3.2, 9.9, 9.14, 11.2, 11.3, 11.4, and 11.11; Added sections 2.19, 2.34, 4.8, 11.9 through 11.10, and 11.11.4 through 11.11.7.

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Version	Date	Description of Revisions
16	3/26/13	Technical and Quality Review and update. Revised title, Attachment IV and sections 2.1, 7.8, 7.9, 7.10, 8.0 (state note), 8.3, 11.6, and 13.1; Added sections 7.13, 14.6 and state notes in sections 1.0 and 11.4; Removed sections 7.10.5 and 8.5.3.
17	6/10/14	Technical and Quality Review and update. Revised Attachments II, IVc through IVk, and V along with sections 5.3, 8.3.1, 8.3.6; 11.7; Added sections 5.4.7, 7.5.10 through 7.5.13, 7.7, 8.3.4; Removed sections 6.1.4 and 11.11.2.
18	8/14/14	Technical and Quality Review and update. Added sections 2.35, 7.5.14, and 13.6.
19	11/17/2015	Technical and quality review and update. Header and signature block re-formatting. Revised Sections 1.5, 1.8.1, 5.3, 7.9.1, 7.11.1, 8.1.1, 8.4.1, 8.4.4, 8.4.6, 8.7.1, 8.8, 8.9.1.1, 11.3, 11.5, 11.7, 11.11.4, 11.11.5, and 13.5. Added Section 8.6.1.7, 8.6.1.7.1 through 8.6.1.7.6, and Attachment VI. Deleted Attachment Iva, Attachment IVb, Attachment IVc, Attachment IVd, Attachment IVe, Attachment IVf, Attachment IVg, Attachment IVh, Attachment IVi, Attachment IVj, and Attachment IVk.
20	4/1/2016	Technical and quality review and update. Revised Sections 1.8.1, 2.8, 2.9, 2.10, 4.8, 7.1, 9.4, 9.5, 11.2, 11.4, 11.11.7, 12.2, and 13.6. Deleted Sections 2.11 through 2.35 and 9.6 through 9.13. Added Sections 7.5.16, 7.5.17, 7.5.18, and 8.9.7.
21	10/24/16	Technical and quality review and update to satisfy the requirements of SC DHEC (see correspondence dated 6/24/16) Header and signature block re-formatting. Revised Sections 11.2, 11.3, 11.4, 11.7, 14.1, 14.2, 14.3, 14.4, 14.6, 14.7, 14.8, and Attachment VII Table 2. Added Sections 1.8.2, 2.10, 9.5, 10.4 and all subsections, and 11.11.7.1.
22	6/19/2017	Technical and quality review and update. Revised Sections 1.8, 3.1, 7.5.14, 7.8, 7.11.1, 8.4.1, 8.8, 9.3, 10.3, 11.7, and Attachment III.
23	7/10/2017	Technical and quality review and update. Revised Sections 2.1, 2.11, 2.12, 7.9.2, 7.10.2, 7.10.3, 7.10.4, 7.11.4, 7.11.5, 9.3, 13.1, 13.5
24	10/6/2017	Technical and quality review and update. Revised Sections 7.9.2, 11.5, and Attachment IV. Added Table 8 in Attachment VII.
25	11/29/2017	Update in response to A2LA audit finding CAR2872. Revised Attachment IV.

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Attachment II: 8270/625 Common Calibration List & Reporting Limits *(may be updated without notice)**

Analyte	Water mg/L	Soil mg/Kg
Acenaphthene	0.001	0.033
Acenaphthylene	0.001	0.033
Acetophenone	0.01	0.33
Anthracene	0.001	0.033
Atrazine	0.01	0.33
Benzaldehyde	0.01	0.33
Benzidine	0.05	0.33
Benzo(a)anthracene	0.001	0.033
Benzo(b)fluoranthene	0.001	0.033
Benzo(k)fluoranthene	0.001	0.033
Benzo(g,h,i)perylene	0.001	0.033
Benzo(a)pyrene	0.001	0.033
Bis(2-chlorethoxy)methane	0.01	0.33
Bis(2-chloroethyl)ether	0.01	0.33
Bis(2-chloroisopropyl)ether	0.01	0.33
4-Bromophenyl-phenylether	0.01	0.33
Caprolactam	0.01	0.33
2-Chloronaphthalene	0.01	0.33
4-Chlorophenyl-phenylether	0.01	0.33
Chrysene	0.001	0.033
Dibenz(a,h)anthracene	0.001	0.033
3,3-Dichlorobenzidine	0.01	0.33
2,4-Dinitrotoluene	0.01	0.33
2,6-Dinitrotoluene	0.01	0.33
Fluoranthene	0.001	0.033
Fluorene	0.001	0.033
Hexachlorobenzene	0.01	0.33
Hexachloro-1,3-butadiene	0.01	0.33
Hexachlorocyclopentadiene	0.01	0.33
Hexachloroethane	0.01	0.33
Indeno(1,2,3-cd)pyrene	0.001	0.033
Isophorone	0.01	0.33
Naphthalene	0.001	0.033
Nitrobenzene	0.01	0.33

Analyte	Water mg/L	Soil mg/Kg
n-Nitrosodimethylamine	0.01	0.33
n-Nitrosodiphenylamine	0.01	0.33
n-Nitrosodi-n-propylamine	0.01	0.33
Phenanthrene	0.001	0.033
Benzylbutyl phthalate	0.003	0.033
Bis(2-ethylhexyl)phthalate	0.003	0.033
Di-n-butyl phthalate	0.003	0.033
Diethyl phthalate	0.003	0.033
Dimethyl phthalate	0.003	0.033
Di-n-octyl phthalate	0.003	0.033
Pyrene	0.001	0.033
1,2,4-Trichlorobenzene	0.01	0.33
4-Chloro-3-methylphenol	0.01	0.33
2-Chlorophenol	0.01	0.33
2,4-Dichlorophenol	0.01	0.33
2,4-Dimethylphenol	0.01	0.33
4,6-Dinitro-2-methylphenol	0.01	0.33
2,4-Dinitrophenol	0.01	0.33
2-Methylphenol	0.01	0.33
4-Methylphenol	0.01	0.33
2-Nitrophenol	0.01	0.33
4-Nitrophenol	0.01	0.33
Pentachlorophenol	0.01	0.33
Phenol	0.01	0.33
2,4,6-Trichlorophenol	0.01	0.33
1-Methylnapthalene	0.001	0.033
2-Methylnapthalene	0.001	0.033
4-Chloroaniline	0.01	0.33
2-Nitroaniline	0.01	0.33
3-Nitroaniline	0.01	0.33
4-Nitroaniline	0.01	0.33
1,2,3,4-Tetrachlorobenzene	0.05	1.65
1,2,3,5-Tetrachlorobenzene	0.05	1.65
1,2,4,5-Tetrachlorobenzene	0.05	1.65

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Analyte	Water mg/L	Soil mg/Kg
1,2,4,5-Tetrachlorobenzene	0.05	1.65
1,2-diphenylhydrazine	0.01	0.33
1,3-Dinitrobenzene	0.05	1.65
1,4-Naphthoquinone	0.05	1.65
1-Chloronaphthalene	0.05	1.65
1-Naphthylamine	0.05	1.65
2,3,4,6-Tetrachlorophenol	0.05	1.65
2,3-Dichloroaniline	0.01	0.33
2,6-Dichlorophenol	0.05	1.65
2-Acetylaminofluorene	0.05	1.65
2-Naphthylamine	0.05	1.65
2-Picoline	0.05	1.65
3,3'-Dimethylbenzidine	0.05	1.65
3-Methylcholanthrene	0.05	1.65
4-Aminobiphenyl	0.05	1.65
4-Nitroquinoline-1-oxide	0.05	1.65
5-Nitro-o-toluidine	0.05	1.65
7,12-Dimethylbenz(a)anthracene	0.05	1.65
7H-Dibenzo (c,g) carbazole	0.05	1.65
a,a-Dimethylphenethylamine	0.05	1.65
Acetophenone	0.01	0.33
Alpha-terpineol	0.01	0.33
Aniline	0.01	0.33
Aramite	0.05	1.65
Benzal Chloride	0.05	1.65
Benzo (j) fluoranthene	0.05	1.65
Benzotrichloride	0.05	1.65
Benzyl Chloride	0.05	1.65
Chlorobenzilate	0.05	1.65
Diallate (cis)	0.05	1.65
Diallate (trans)	0.05	1.65
Dibenz (a,e) pyrene	0.05	1.65
Dibenz (a,h) acridine	0.05	0.33
Dibenz (a,h) pyrene	0.05	1.65

Analyte	Water mg/L	Soil mg/Kg
Dibenz (a,i) pyrene	0.05	1.65
Dimethoate	0.05	1.65
Dinoseb	0.05	1.65
Diphenylamine	0.05	1.65
Disulfoton	0.05	1.65
Ethyl methanesulfonate	0.05	1.65
Famphur	0.05	1.65
Hexachlorophene	0.05	1.65
Hexachloropropene	0.05	1.65
Isodrin	0.05	1.65
Isosafrole (cis)	0.05	1.65
Isosafrole (trans)	0.05	1.65
Kepone	0.05	1.65
Methapyriline	0.05	1.65
Methyl methanesulfonate	0.05	1.65
Methyl parathion	0.05	1.65
N-Nitrosodiethylamine	0.05	1.65
n-nitrosodi-n-butylamine	0.01	0.33
N-Nitrosodi-n-butylamine	0.05	1.65
N-Nitrosomethylethylamine	0.05	1.65
N-Nitrosomorpholine	0.05	1.65
N-Nitrosopiperidine	0.05	1.65
N-Nitrosopyrrolidine	0.05	1.65
o,o,o-Triethylphosphorothioate	0.05	1.65
o-cresol	0.01	0.33
o-Toluidine	0.05	1.65
Parathion	0.05	1.65
p-cresol	0.01	0.33
p-Dimethylaminoazobenzene	0.05	1.65
Pentachlorobenzene	0.05	1.65
Pentachloroethane	0.05	1.65
Pentachloronitrobenzene	0.05	1.65
Phenacetin	0.05	1.65
Phorate	0.05	1.65

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Analyte	Water mg/L	Soil mg/Kg
p-Phenyleneamine	0.05	1.65
Pronamide	0.05	1.65
Safrole	0.05	1.65
Sulfotepp	0.05	1.65
sym-Trinitrobenzene	0.05	1.65
Thionazin	0.05	1.65
2-nitrodiphenylamine	0.01	0.33
n-decane	0.01	0.33
n-octadecane	0.01	0.33
Pentachlorophenol (SIM)	0.001	-
Sulfolane	0.0002	0.33
Mirex	0.02	NA
Dicofol	0.02	NA
Quinoline	0.05	0.33
Indene	NA	0.33
Benzenethiol	0.02	3.3

*Alternate reporting levels may be possible using different technologies (i.e. SIM, LVI, etc.). Please contact the laboratory for additional information.

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Attachment III – Appropriate Extraction Methods by Analyte (printed from SW-846 Method 8270C)

ANALYTE:	3510*	3520	3540/3541	3550*	3580*	CAS #(a)
Acenaphthene	X	X	X	X	X	83-32-9
Acenaphthylene	X	X	X	X	X	208-96-8
Acetophenone	X	ND	ND	ND	X	98-86-2
2-Acetylaminofluorene	X	ND	ND	ND	X	53-96-3
1-Acetyl-2-thiourea	LR	ND	ND	ND	LR	591-08-2
Aldrin	X	X	X	X	X	309-00-2
2-Aminoanthraquinone	X	ND	ND	ND	X	117-79-3
Aminoazobenzene	X	ND	ND	ND	X	60-09-3
4-Aminobiphenyl	X	ND	ND	ND	X	92-67-1
3-Amino-9-ethylcarbazole	X	X	ND	ND	ND	132-32-1
Anilazine	X	ND	ND	ND	X	101-05-3
Aniline	X	X	ND	X	X	62-53-3
Ortho-anisidine	X	ND	ND	ND	X	90-04-0
Anthracene	X	X	X	X	X	120-12-7
Aramite HS	(43)	ND	ND	ND	X	140-57-8
Aroclor 1016	X	X	X	X	X	12674-11-2
Aroclor 1221	X	X	X	X	X	11104-28-2
Aroclor 1232	X	X	X	X	X	11141-16-5
Aroclor 1242	X	X	X	X	X	53469-21-9
Aroclor 1248	X	X	X	X	X	12672-29-6
Aroclor 1254	X	X	X	X	X	11097-69-1
Aroclor 1260	X	X	X	X	X	11096-82-5
Azinphos-methyl HS	(62)	ND	ND	ND	X	86-50-0
Barban	LR	ND	ND	ND	LR	101-27-9
Benzidine	CP	CP	CP	CP	CP	92-87-5
Benzoic Acid	X	X	ND	X	X	65-85-0
Benz(a)anthracene	X	X	X	X	X	56-55-3
Benzo(b)fluoranthene	X	X	X	X	X	205-99-2
Benzo(k)fluoranthene	X	X	X	X	X	207-08-9
Benzo(g,h,i)perylene	X	X	X	X	X	191-24-2
Benzo(a)pyrene	X	X	X	X	X	50-32-8
Para-benzoquinone	OE	ND	ND	ND	X	106-51-4
Benzyl Alcohol	X	X	ND	X	X	100-51-6
Alpha-BHC	X	X	X	X	X	319-84-6
Beta-BHC	X	X	X	X	X	319-85-7
Delta-BHC	X	X	X	X	X	319-86-8
Gamma-BHC	X	X	X	X		58-89-9
Lindane	X	X	X	X	X	58-89-9
Bis(2-chloroethoxy)methane	X	X	X	X	X	111-91-1
Bis(2-chloroethyl) Ether	X	X	X	X	X	111-44-4

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ANALYTE:	3510*	3520	3540/3541	3550*	3580*	CAS #(a)
Bis(2-chloroisopropyl) Ether	X	X	X	X	X	108-60-1
Bis(2-ethylhexyl) Phthalate	X	X	X	X	X	117-81-7
4-Bromophenyl Phenyl Ether	X	X	X	X	X	101-55-3
Bromoxynil	X	ND	ND	ND	X	1689-84-5
Butyl Benzyl Phthalate	X	X	X	X	X	85-68-7
Captafol HS	(55)	ND	ND	ND	X	6/1/2425
Captan HS	(40)	ND	ND	ND	X	133-06-2
Carbaryl	X	ND	ND	ND	X	63-25-2
Carbofuran	X	ND	ND	ND	X	1563-66-2
Carbophenothion	X	ND	ND	ND	X	786-19-6
Chlordane	X	X	X	X	X	57-74-9
Chlorfenvinphos	X	ND	ND	ND	X	470-90-6
4-Chloroaniline	X	ND	ND	ND	X	106-47-8
Chlorobenzilate	X	ND	ND	ND	X	510-15-6
5-Chloro-2-methylaniline	X	ND	ND	ND	X	95-79-4
4-Chloro-3-methylphenol	X	X	X	X	X	59-50-7
hydrochloride	X	ND	ND	ND	X	6959-48-4
1-Chloronaphthalene	X	X	X	X	X	90-13-1
2-Chloronaphthalene	X	X	X	X	X	91-58-7
2-Chlorophenol	X	X	X	X	X	95-57-8
4-Chloro-1,2-phenylenediamine	X	X	ND	ND	ND	95-83-0
4-Chloro-1,3-phenylenediamine	X	X	ND	ND	ND	5131-60-2
4-Chlorophenyl Phenyl Ether	X	X	X	X	X	7005-72-3
Chrysene	X	X	X	X	X	218-01-9
Coumaphos	X	ND	ND	ND	X	56-72-4
Para-cresidine	X	ND	ND	ND	X	120-71-8
Crotoxypfos	X	ND	ND	ND	X	7700-17-6
2-Cyclohexyl-4,6-dinitrophenol	X	ND	ND	ND	LR	131-89-5
4,"-DDD	X	X	X	X	X	72-54-8
4,"-DDE	X	X	X	X	X	72-55-9
4,"-DDT	X	X	X	X	X	50-29-3
Demeton-O HS	(68)	ND	ND	ND	X	298-03-3
Demeton-S	X	ND	ND	ND	X	126-75-0
Diallate (cis or trans)	X	ND	ND	ND	X	2303-16-4
2,4-Diaminotoluene DC,	OE(42) ND	ND	ND	ND	X	95-80-7
Dibenz(a,j)acridine	X	ND	ND	ND	X	224-42-0
Dibenz(a,h)anthracene	X	X	X	X	X	53-70-3
Dibenzofuran	X	X	ND	X	X	132-64-9
Dibenzo(a,e)pyrene	ND	ND	ND	ND	X	192-65-4
1,2-Dibromo-3-chloropropane	X	X	ND	ND	ND	96-12-8
Di-n-butyl Phthalate	X	X	X	X	X	84-74-2

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ANALYTE:	3510*	3520	3540/3541	3550*	3580*	CAS #(a)
Dichlone	OE	ND	ND	ND	X	117-80-6
1,2-Dichlorobenzene	X	X	X	X	X	95-50-1
1,3-Dichlorobenzene	X	X	X	X	X	541-73-1
1,4-Dichlorobenzene	X	X	X	X	X	106-46-7
3,3'-Dichlorobenzidine	X	X	X	X	X	91-94-1
2,4-Dichlorophenol	X	X	X	X	X	120-83-2
2,6-Dichlorophenol	X	ND	ND	ND	X	87-65-0
Dichlorovos	X	ND	ND	ND	X	62-73-7
Dicrotophos	X	ND	ND	ND	X	141-66-2
Dieldrin	X	X	X	X	X	60-57-1
Diethyl Phthalate	X	X	X	X	X	84-66-2
Diethylstilbestrol	AW,OS(67)	ND	ND	ND	X	56-53-1
Diethyl Sulfate	LR	ND	ND	ND	LR	64-67-5
Dihydrosaffrole	ND	ND	ND	ND	ND	56312-13-1
Dimethoate	HE,HS	ND	ND	ND	X	60-51-5
3,'-Dimethoxybenzidine	X	ND	ND	ND	LR	119-90-4
Dimethylaminoazobenzene	X	ND	ND	ND	X	60-11-7
7,12-Dimethylbenz(a)-anthracene	CP(45)	ND	ND	ND	CP	57-97-6
3,'-Dimethylbenzidine	X	ND	ND	ND	X	119-93-7
α,α-Dimethylphenethylamine	ND	ND	ND	ND	X	122-09-8
2,4-Dimethylphenol	X	X	X	X	X	105-67-9
Dimethyl Phthalate	X	X	X	X	X	131-11-3
1,2-Dinitrobenzene	X	ND	ND	ND	X	528-29-0
1,3-Dinitrobenzene	X	ND	ND	ND	X	99-65-0
1,4-Dinitrobenzene	HE(14)	ND	ND	ND	X	100-25-4
4,6-Dinitro-2-methylphenol	X	X	X	X	X	534-52-1
2,4-Dinitrophenol	X	X	X	X	X	51-28-5
2,4-Dinitrotoluene	X	X	X	X	X	121-14-2
2,6-Dinitrotoluene	X	X	X	X	X	606-20-2
Dinocap	CP,HS(28)	ND	ND	ND	CP	39300-45-3
Dinoseb	X	ND	ND	ND	X	88-85-7
Dioxathion	ND	ND	ND	ND	ND	78-34-2
Diphenylamine	X	X	X	X	X	122-39-4
5,5-Diphenylhydantoin	X	ND	ND	ND	X	57-41-0
1,2-Diphenylhydrazine	X	X	X	X	X	122-66-7
Di-n-octyl Phthalate	X	X	X	X	X	117-84-0
Disulfoton	X	ND	ND	ND	X	298-04-4
Endosulfan I	X	X	X	X	X	959-98-8
Endosulfan II	X	X	X	X	X	33212-65-9
Endosulfan Sulfate	X	X	X	X	X	1031-07-8
Endrin	X	X	X	X	X	72-20-8

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ANALYTE:	3510*	3520	3540/3541	3550*	3580*	CAS #(a)
Endrin Aldehyde	X	X	X	X	X	7421-93-4
Endrin Ketone	X	X	ND	X	X	53494-70-5
EPN	X	ND	ND	ND	X	2104-64-5
Ethion	X	ND	ND	ND	X	563-12-2
Ethyl Carbamate	DC(28)	ND	ND	ND	X	51-79-6
Ethyl Methanesulfonate	X	ND	ND	ND	X	62-50-0
Famphur	X	ND	ND	ND	X	52-85-7
Fensulfothion	X	ND	ND	ND	X	115-90-2
Fenthion	X	ND	ND	ND	X	55-38-9
Fluchloralin	X	ND	ND	ND	X	33245-39-5
Fluoranthene	X	X	X	X	X	206-44-0
Fluorene	X	X	X	X	X	86-73-7
2-Fluorobiphenyl (Surr)	X	X	X	X	X	321-60-8
2-Fluorophenol (Surr)	X	X	X	X	X	367-12-4
Heptachlor	X	X	X	X	X	76-44-8
Heptachlor Epoxide	X	X	X	X	X	1024-57-3
Hexachlorobenzene	X	X	X	X	X	118-74-1
Hexachlorobutadiene	X	X	X	X	X	87-68-3
Hexachlorocyclopentadiene	X	X	X	X	X	77-47-4
Hexachloroethane	X	X	X	X	X	67-72-1
Hexachlorophene	AW,CP(62)	ND	ND	ND	CP	70-30-4
Hexachloropropene	X	ND	ND	ND	X	1888-71-7
Hexamethylphosphoramide	X	ND	ND	ND	X	680-31-9
Hydroquinone	ND	ND	ND	ND	X	123-31-9
Indeno(1,2,3-cd)pyrene	X	X	X	X	X	193-39-5
Isodrin	X	ND	ND	ND	X	465-73-6
Isophorone	X	X	X	X	X	78-59-1
Isosafrole	DC(46) ND	ND	ND	ND	X	120-58-1
Kepone	X	ND	ND	ND	X	143-50-0
Leptophos	X	ND	ND	ND	X	21609-90-5
Malathion	HS(5)	ND	ND	ND	X	121-75-5
Maleic Anhydride	HE	ND	ND	ND	X	108-31-6
Mestranol	X	ND	ND	ND	X	72-33-3
Methapyrilene	X	ND	ND	ND	X	91-80-5
Methoxychlor	X	ND	ND	ND	X	72-43-5
3-Methylcholanthrene	X	ND	ND	ND	X	56-49-5
4,"-Methylenebis (2-chloroaniline)	OE,OS(0)	ND	ND	ND	LR	101-14-4
4,"-Methylenebis-(N-n-dimethylaniline)	X	X	ND	ND	ND	101-61-1
Methyl methanesulfonate	X	ND	ND	ND	X	66-27-3
2-Methylnaphthalene	X	X	ND	X	X	91-57-6

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ANALYTE:	3510*	3520	3540/3541	3550*	3580*	CAS #(a)
Methyl Parathion	X	ND	ND	ND	X	298-00-0
2-Methylphenol	X	ND	ND	ND	X	95-48-7
3-Methylphenol	X	ND	ND	ND	X	108-39-4
4-Methylphenol	X	ND	ND	ND	X	106-44-5
2-Methylpyridine	X	X	ND	ND	ND	109-06-8
Mevinphos	X	ND	ND	ND	X	7786-34-7
Mexacarbate	HE,HS(68)	ND	ND	ND	X	315-18-4
Mirex	X	ND	ND	ND	X	2385-85-5
Monocrotophos	HE	ND	ND	ND	X	6923-22-4
Naled	X	ND	ND	ND	X	300-76-5
Naphthalene	X	X	X	X	X	91-20-3
1,4-Naphthoquinone	X	ND	ND	ND	X	130-15-4
1-Naphthylamine	OS(44)	ND	ND	ND	X	134-32-7
2-Naphthylamine	X	ND	ND	ND	X	91-59-8
Nicotine	DE(67)	ND	ND	ND	X	54-11-5
5-Nitroacenaphthene	X	ND	ND	ND	X	602-87-9
2-Nitroaniline	X	X	ND	X	X	88-74-4
3-Nitroaniline	X	X	ND	X	X	99-09-2
4-Nitroaniline	X	X	ND	X	X	100-01-6
5-Nitro-o-anisidine	X	ND	ND	ND	X	99-59-2
Nitrobenzene	X	X	X	X	X	98-95-3
4-Nitrobiphenyl	X	ND	ND	ND	X	92-93-3
Nitrofen	X	ND	ND	ND	X	1836-75-5
2-Nitrophenol	X	X	X	X	X	88-75-5
4-Nitrophenol	X	X	X	X	X	100-02-7
5-Nitro-o-toluidine	X	ND	ND	ND	X	99-55-8
Nitroquinoline-1-oxide	X	ND	ND	ND	X	56-57-5
N-nitrosodi-n-butylamine	X	ND	ND	ND	X	924-16-3
N-nitrosodiethylamine	X	ND	ND	ND	X	55-18-5
N-nitrosodimethylamine	X	X	X	X	X	62-75-9
N-nitrosomethylethylamine	X	ND	ND	ND	X	10595-95-6
N-nitrosodiphenylamine	X	X	X	X	X	86-30-6
N-nitrosodi-n-propylamine	X	X	X	X	X	621-64-7
N-nitrosomorpholine	ND	ND	ND	ND	X	59-89-2
N-nitrosopiperidine	X	ND	ND	ND	X	100-75-4
N-nitrosopyrrolidine	X	ND	ND	ND	X	930-55-2
Octamethyl Pyrophosphoramidate	LR	ND	ND	ND	LR	152-16-9
Parathion	X	ND	ND	ND	X	56-38-2
Pentachlorobenzene	X	ND	ND	ND	X	608-93-5
Pentachloronitrobenzene	X	ND	ND	ND	X	82-68-8
Pentachlorophenol	X	X	X	X	X	87-86-5

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ANALYTE:	3510*	3520	3540/3541	3550*	3580*	CAS #(a)
Phenacetin	X	ND	ND	ND	X	62-44-2
Phenanthrene	X	X	X	X	X	85-01-8
Phenobarbital	X	ND	ND	ND	X	50-06-6
Phenol	DC(28)	X	X	X	X	108-95-2
1,4-Phenylenediamine	X	ND	ND	ND	X	106-50-3
Phorate	X	ND	ND	ND	X	298-02-2
Phosalone	HS(65)	ND	ND	ND	X	2310-17-0
Phosmet	HS(15)	ND	ND	ND	X	732-11-6
Phosphamidon	HE(63)	ND	ND	ND	X	13171-21-6
Phthalic Anhydride	CP,HE(1)	ND	ND	ND	CP	85-44-9
2-Picoline	X	X	ND	ND	ND	109-06-8
Piperonyl Sulfoxide	X	ND	ND	ND	X	120-62-7
Pronamide	X	ND	ND	ND	X	23950-58-5
Pyrene	X	X	X	X	X	129-00-0
Pyridine	ND	ND	ND	ND	ND	110-86-1
Resorcinol	DC, OE(10)	ND	ND	ND	X	94-59-7
Safrole	X	ND	ND	ND	X	60-41-3
Sulfallate	X	ND	ND	ND	X	95-06-7
Terbufos	X	ND	ND	ND	X	13071-79-9
Terphenyl d(l4)(surr)	X	X	ND	X	X	1718-51-0
1,2,4,5-Tetrachlorobenzene	X	ND	ND	ND	X	95-94-3
2,3,4,6-Tetrachlorophenol	X	ND	ND	ND	X	58-90-2
Tetrachlorvinphos	X	ND	ND	ND	X	961-11-5
Tetraethyl Dithiopyrophosphate	X	X	ND	ND	ND	3689-24-5
Tetraethyl Pyrophosphate	X	ND	ND	ND	X	107-49-3
Thionazine	X	ND	ND	ND	X	297-97-2
Thiophenol	X	ND	ND	ND	X	108-98-5
Benzenethiol	X	ND	ND	ND	X	108-98-5
Toluene Diisocyanate	HE(6)	ND	ND	ND	X	584-84-9
Ortho-toluidine	X	ND	ND	ND	X	95-53-4
Toxaphene	X	X	X	X	X	8001-35-2
1,2,4-Trichlorobenzene	X	X	X	X	X	120-82-1
2,4,5-Trichlorophenol	X	X	ND	X	X	95-95-4
2,4,6-Trichlorophenol	X	X	X	X	X	88-06-2
Trifluralin	X	ND	ND	ND	X	1582-09-8
2,4,5-Trimethylaniline	X	ND	ND	ND	X	137-17-7
Trimethyl Phosphate	HE(60)	ND	ND	ND	X	512-56-1
1,3,5-Trinitrobenzene	X	ND	ND	ND	X	99-35-4
Tris(2,3-dibromopropyl) phosphate	X	ND	ND	ND	LR	126-72-7
O,O,O-Triethyl Phosphorothioate	X	ND	ND	ND	X	126-68-1

KEY TO ANALYTE LIST ESC extraction technique Chemical Abstract Service Registry Number



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- (b) See Sec. 1.2 for other acceptable preparation methods.
- (IS) This compound may be used as an internal standard.
- (surr) This compound may be used as a surrogate.
- (AW) Adsorption to walls of glassware during extraction and storage.
- (CP) Non-reproducible chromatographic performance.
- (DC) Unfavorable distribution coefficient (number in parenthesis is percent recovery).
- (HE) Hydrolysis during extraction accelerated by acidic or basic conditions (number in parenthesis is percent recovery).
- (HS) Hydrolysis during storage (number in parenthesis is percent stability).
- (LR) Low response.
- (ND) Not determined.
- (OE) Oxidation during extraction accelerated by basic conditions (number in parenthesis is percent recovery).
- (OS) Oxidation during storage (number in parenthesis is percent stability).
- (X) Greater than 70 percent recovery by this technique.

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Attachment IV: Characteristic Masses (m/z) for Extractable Organic Compounds

(Reprinted from SW-846 Method 8270C /Dec. 1996)

Compound	Primary Characteristic Ion	Secondary Characteristic Ion(s)
Pyridine	79	52,78,51
N-Nitrosodimethylamine	42	74,44
2-Picoline	93	66,92
Aniline	93	66,65
Phenol	94	65,66
Benzaldehyde	105	106,77,51
Bis(2-chloroethyl) ether	93	63,95
2-Chlorophenol	128	64,130
1,3-Dichlorobenzene	146	148,111
1,4-Dichlorobenzene-d4 (ISTD)	152	150,115
1,4-Dichlorobenzene	146	148,111
Benzyl alcohol	108	79,77
1,2-Dichlorobenzene	146	148,111
N-Nitrosomethylethylamine	88	42,43,56
Bis(2-chloroisopropyl) ether	45	77,121
Methyl methanesulfonate	80	79,65,95
N-Nitrosodi-n-propylamine	70	42,101,130
Hexachloroethane	117	201,199
Nitrobenzene	77	123,65
Isophorone	82	95,138
N-Nitrosodiethylamine	102	42,57,44,56
2-Nitrophenol	139	109,65
2,4-Dimethylphenol	122	107,121
Bis(2-chloroethoxy)methane	93	95,123
Benzoic acid	122	105,77
2,4-Dichlorophenol	162	164,98
Ethyl methanesulfonate	79	109,97,45,65
1,2,4-Trichlorobenzene	180	182,145
Naphthalene-d8 (ISTD)	136	68
Naphthalene	128	129,127
Hexachlorobutadiene	225	223,227
Caprolactam	113	55,56,42
4-Chloro-3-methylphenol	107	144,142
2-Methylnaphthalene	142	141
1-Methylnaphthalene	142	141
2-Methylphenol	107	108,77,79,90
Hexachloropropene	213	211,215,117,106,141
Hexachlorocyclopentadiene	237	235,272
N-Nitrosopyrrolidine	100	41,42,68,69

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Compound	Primary Characteristic Ion	Secondary Characteristic Ion(s)
Acetophenone	105	71,51,120
4-Methylphenol	107	108,77,79,90
2,4,6-Trichlorophenol	196	198,200
2,4,5-Trichlorophenol	196	198,200
o-Toluidine	106	107,77,51,79
3-Methylphenol	107	108,77,79,90
2-Chloronaphthalene	162	127,164
N-Nitrosopiperidine	114	42,55,56,41
1-Chloronaphthalene	162	127,164
2-Nitroaniline	65	92,138
Dimethyl phthalate	163	194,164
Acenaphthylene	152	151,153
2,6-Dinitrotoluene	165	63,89
3-Nitroaniline	138	108,92
Acenaphthene-d10 (ISTD)	164	162,160
Acenaphthene	154	153,152
2,4-Dinitrophenol	184	63,154
2,6-Dinitrophenol	162	164,126,98,63
4-Chloroaniline	127	129,65,92
Isosafrole	162	131,104,77,51
Dibenzofuran	168	139
2,4-Dinitrotoluene	165	63,89
4-Nitrophenol	139	109,65
2-Naphthylamine	143	115,116
1,4-Naphthoquinone	158	104,102,76,50,130
Diethyl phthalate	149	177,150
Fluorene	166	165,167
N-Nitrosodi-n-butylamine	84	57,41,116,158
4-Chlorophenyl phenyl ether	204	206,141
Atrazine	200	215,58
4,6-Dinitro-2-methylphenol	198	51,105
N-Nitrosodiphenylamine	169	168,167
Safrole	162	104,77,103,135
Diphenylamine	169	168,167
1,2,4,5-Tetrachlorobenzene	216	214,179,108,143,218
1-Naphthylamine	143	115,89,63
4-Bromophenyl phenyl ether	248	250,141
2,4,5-Trichlorophenol	196	198,97,132,99
Hexachlorobenzene	284	142,249
Pentachlorophenol	266	264,268
5-Nitro-o-toluidine	152	77,79,106,94

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Compound	Primary Characteristic Ion	Secondary Characteristic Ion(s)
Thionazine	107	96,97,143,79,68
4-Nitroaniline	138	65,108,92,80,39
Phenanthrene-d10 (ISTD)	188	94,80
Phenanthrene	178	179,176
Anthracene	178	176,179
Carbazole	167	166,168,139
1,3-Dinitrobenzene	168	76,50,75,92,122
Diallate (cis or trans)	86	234,43,70
Pentachlorobenzene	250	252,108,248,215,254
Pentachloronitrobenzene	237	142,214,249,295,265
4-Nitroquinoline-1-oxide	174	101,128,75,116
Di-n-butyl phthalate	149	150,104
2,3,4,6-Tetrachlorophenol	232	131,230,166,234,168
Demeton-O	88	89,60,61,115,171
Fluoranthene	202	101,203
1,3,5-Trinitrobenzene	75	74,213,120,91,63
Benzidine	184	92,185
Pyrene	202	200,203
Phorate	75	121,97,93,260
Demeton-S	88	60,81,89,114,115
Phenacetin	108	180,179,109,137,80
Dimethoate	87	93,125,143,229
4-Aminobiphenyl	169	168,170,115
Dimethylphenylamine	58	91,65,134,42
Pronamide	173	175,145,109,147
Dinoseb	211	163,147,117,240
Disulfoton	88	97,89,142,186
Butyl benzyl phthalate	149	91,206
Methyl parathion	109	125,263,79,93
Dimethylaminoazobenzene	225	120,77,105,148,42
Benz(a)anthracene	228	229,226
Chrysene-d12 (ISTD)	240	120,236
3,3'-Dichlorobenzidine	252	254,126
Chrysene	228	226,229
Kepone	272	274,237,178,143,270
Parathion	109	97,291,139,155
Bis(2-ethylhexyl) phthalate	149	167,279
3,3'-Dimethylbenzidine	212	106,196,180
Methapyrilene	97	50,191,71
Isodrin	193	66,195,263,265,147

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Compound	Primary Characteristic Ion	Secondary Characteristic Ion(s)
Di-n-octyl phthalate	149	167,43
Aramite	185	191,319,334,197,321
Benzo(b)fluoranthene	252	253,125
Benzo(k)fluoranthene	252	253,125
Famphur	218	125,93,109,217
Benzo(a)pyrene	252	253,125
Perylene-d12 (ISTD)	264	260,265
7,12-Dimethylbenz(a)anthracene	256	241,239,120
2-Acetylaminofluorene	181	180,223,152
3-Methylcholanthrene	268	252,253,126,134,113
Dibenz(a,j)acridine	279	280,277,250
Indeno(1,2,3-cd)pyrene	276	138,227
Dibenz(a,h)anthracene	278	139,279
Benzo(g,h,i)perylene	276	138,277
Hexachlorophene	196	198,209,211,406,408
1,2-Diphenylhydrazine/Azobenzene	77	105,182
Mirex	272	274, 237,270
Kelthane (Dicofol)	251	139.111.253
Indene	115	116,117
Quinoline	129	128,130,102
Benzenethiol	110	109,66
Diphenyl Disulfide	218	109,65
Surrogates		
2-Fluorobiphenyl (surr)	172	171
2-Fluorophenol (surr)	112	64
Nitrobenzene-d5 (surr)	82	128,54
Phenol-d6 (surr)	99	42,71
Terphenyl-d14 (surr)	244	122,212
2,4,6-Tribromophenol (surr)	330	332,141
2-Methylnaphthalene-d10 (surr)	152	150, 122, 151
Fluoranthene-d10 (surr)	212	208, 313, 210
PAH by SIM		
Naphthalene	128	129
2-Methylnaphthalene	142	141
1-Methylnaphthalene	142	141
2-Chloronaphthalene	162	127
Acenaphthylene	152	153, 151
Acenaphthene	153	154, 152, 151
Dibenzofuran	168	139
Fluorene	166	165
Phenanthrene	178	179

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Compound	Primary Characteristic Ion	Secondary Characteristic Ion(s)
Anthracene	178	179, 176
Fluoranthene	202	203, 200
Pyrene	202	203, 200
Benzo(a)anthracene	228	226
Chrysene	228	226, 229
Benzo(b)fluoranthene	252	253
Benzo(k)fluoranthene	252	253
Benzo(a)pyrene	252	253
Indeno(1,2,3-cd)pyrene	276	277, 138
Dibenz(a,h)anthracene	278	279, 139, 138
Benzo(g,h,i)perylene	276	138

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TITLE: SEMIVOLATILE ORGANICS BY GC/MS (EPA METHODS 8270C, 8270D, 625, 625.1 AND SM 6410B), INCLUDING PROVISIONS FOR ANALYSIS IN SIM MODE

Attachment V - QC Acceptance Criteria for Method 625

Compound	Test conc. (µg/L)	Limit for s (µg/L)	Range for x (µg/L)	Range p, p(s) (%)
Acenaphthene	100	27.6	60.1-132.3	47-145
Acenaphthylene	100	40.2	53.5-126.0	33-145
Aldrin	100	39	7.2-152.2	D-166
Anthracene	100	32	43.4-118.0	27-133
Benz(a)anthracene	100	27.6	41.8-133.0	33-143
Benzo(b)fluoranthene	100	38.8	42.0-140.4	24-159
Benzo(k)fluoranthene	100	32.3	25.2-145.7	11-162
Benzo(a)pyrene	100	39	31.7-148.0	17-163
Benzo(g,h,i)perylene	100	58.9	D-195.0	D-219
Benzyl butyl phthalate	100	23.4	D-139.9	D-152
beta-BHC	100	31.5	41.5-130.6	24-149
delta-BHC	100	21.6	D-100.0	D-110
Bis(2-chloroethyl) ether	100	55	42.9-126.0	12-158
Bis(2-chloroethoxy)methane	100	34.5	49.2-164.7	33-184
Bis(2-chloroisopropyl) ether	100	46.3	62.8-138.6	36-166
Bis(2-ethylhexyl) phthalate	100	41.1	28.9-136.8	8-158
4-Bromophenyl phenyl ether	100	23	64.9-114.4	53-127
2-Chloronaphthalene	100	13	64.5-113.5	60-118
4-Chlorophenyl phenyl ether	100	33.4	38.4-144.7	25-158
Chrysene	100	48.3	44.1-139.9	17-168
4,4'-DDD	100	31	D-134.5	D-145
4,4'-DDE	100	32	19.2-119.7	4-136
4,4'-DDT	100	61.6	D-170.6	D-203
Dibenzo(a,h)anthracene	100	70	D-199.7	D-227
Di-n-butyl phthalate	100	16.7	8.4-111.0	1-118
1,2-Dichlorobenzene	100	30.9	48.6-112.0	32-129
1,3-Dichlorobenzene	100	41.7	16.7-153.9	D-172
1,4-Dichlorobenzene	100	32.1	37.3-105.7	20-124
3,3'-Dichlorobenzidine	100	71.4	8.2-212.5	D-262
Dieldrin	100	30.7	44.3-119.3	29-136
Diethyl phthalate	100	26.5	D-100.0	D-114
Dimethyl phthalate	100	23.2	D-100.0	D-112
2,4-Dinitrotoluene	100	21.8	47.5-126.9	39-139
2,6-Dinitrotoluene	100	29.6	68.1-136.7	50-158
Di-n-octyl phthalate	100	31.4	18.6-131.8	4-146
Endosulfan sulfate	100	16.7	D-103.5	D-107
Endrin aldehyde	100	32.5	D-188.8	D-209
Fluoranthene	100	32.8	42.9-121.3	26-137
Fluorene	100	20.7	71.6-108.4	59-121
Heptachlor	100	37.2	D-172.2	D-192
Heptachlor epoxide	100	54.7	70.9-109.4	26-155

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Compound	Test conc. (µg/L)	Limit for s (µg/L)	Range for x (µg/L)	Range p, p(s) (%)
Hexachlorobenzene	100	24.9	7.8-141.5	D-152
Hexachlorobutadiene	100	26.3	37.8-102.2	24-116
Hexachloroethane	100	24.5	55.2-100.0	40-113
Indeno(1,2,3-cd)pyrene	100	44.6	D-150.9	D-171
Isophorone	100	63.3	46.6-180.2	21-196
Naphthalene	100	30.1	35.6-119.6	21-133
Nitrobenzene	100	39.3	54.3-157.6	35-180
N-Nitrosodi-n-propylamine	100	55.4	13.6-197.9	D-230
Aroclor 1260	100	54.2	19.3-121.0	D-164
Phenanthrene	100	20.6	65.2-108.7	54-120
Pyrene	100	25.2	69.6-100.0	52-115
1,2,4-Trichlorobenzene	100	28.1	57.3-129.2	44-142
4-Chloro-3-methylphenol	100	37.2	40.8-127.9	22-147
2-Chlorophenol	100	28.7	36.2-120.4	23-134
2,4-Chlorophenol	100	26.4	52.5-121.7	39-135
2,4-Dimethylphenol	100	26.1	41.8-109.0	32-119
2,4-Dinitrophenol	100	49.8	D-172.9	D-191
2-Methyl-4,6-dinitrophenol	100	93.2	53.0-100.0	D-181
2-Nitrophenol	100	35.2	45.0-166.7	29-182
4-Nitrophenol	100	47.2	13.0-106.5	D-132
Pentachlorophenol	100	48.9	38.1-151.8	14-176
Phenol	100	22.6	16.6-100.0	5-112
2,4,6-Trichlorophenol	100	31.7	52.4-129.2	37-144

(s) = Standard deviation of four recovery measurements, in µg/L

(x) = Average recovery for four recovery measurements, in µg/L

(p, p(s)) = Measured percent recovery

(D) = Detected; result must be greater than zero

(a) = Criteria from 40 CFR Part 136 for Method 625, using a packed GC column. These criteria are based directly on the method performance data. Where necessary, the limits for recovery have been broadened to assure applicability of the limits to concentrations below those used to develop method performance data. These values are for guidance only. Appropriate derivation of acceptance criteria for capillary columns should result in much narrower ranges. See Method 8000 for information on developing and updating acceptance criteria for method performance.

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Attachment VI - BNA Poor Performing Compounds

The following compounds are considered to be poor performing compounds.

Pyridine
Aniline
Benzoic Acid
n-Nitrosodimethylamine
Hexachlorocyclopentadiene
4-Chloroaniline
2-Nitroaniline
3-Nitroaniline
4-Nitroaniline
2,4-Dinitro-2-methylphenol
Pentachlorophenol
Carbazole
Benzidine
Atrazine
Acetophenone
Caprolactam
Benzaldehyde
1,2,4,5-Tetrachlorobenzene
Hexachlorophene

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Attachment VII – Method 625.1 Criteria

Analyte	ML (ug/L)	Range for Q (%)	Limit for s (%)	Range for \bar{X} (%)	Range for P1, P2 (%)	Limit for RPD (%)
Acenaphthene*	5.7	70-130	29	60-132	47-145	48
Acenaphthylene*	10.5	60-130	45	54-126	33-145	74
Anthracene*	5.7	58-130	40	43-120	27-133	66
Benzidine*	132					
Benzo(a)anthracene*	23.4	42-133	32	42-133	33-143	53
Benzo(a)pyrene*	7.5	32-148	43	32-148	17-163	72
Benzo(b)fluoranthene*	14.4	42-140	43	42-140	24-159	71
Benzo(k)fluoranthene*	7.5	25-146	38	25-146	11-162	63
Benzo(ghi)perylene*	12.3	13-195	61	D-195	D-219	97
Benzyl butyl phthalate*	7.5	43-140	36	D-140	D-152	60
bis(2-Chloroethoxy)methane	15.9	52-164	32	49-165	33-184	54
bis(2-Ethylhexyl)phthalate*	7.5	43-137	50	29-137	8-158	82
bis(2-Chloroisopropyl) ether (2,2'-Oxybis[1-chloropropane])*	17.1	63-139	46	63-139	36-166	76
4-Bromophenyl phenyl ether*	5.7	70-130	26	65-120	53-127	43
2-Chloronaphthalene*	5.7	70-130	15	65-120	60-120	24
4-Chlorophenyl phenyl ether*	12.6	57-145	36	38-145	25-158	61
Chrysene*	7.5	44-140	53	44-140	17-168	87
Dibenz(a,h)anthracene*	7.5	13-200	75	D-200	D-227	126
Di-n-butylphthalate*	7.5	52-130	28	8-120	1-120	47
3,3'-Dichlorobenzidine*	49.5	18-213	65	8-213	D-262	108
Diethyl phthalate*	5.7	47-130	60	D-120	D-120	100
Dimethyl phthalate*	4.8	50-130	110	D-120	D-120	183
2,4-Dinitrotoluene*	17.1	53-130	25	48-127	39-139	42
2,6-Dinitrotoluene*	5.7	68-137	29	68-137	50-158	48
Di-n-octylphthalate*	7.5	21-132	42	19-132	4-146	69
Fluoranthene*	6.6	47-130	40	43-121	26-137	66
Fluorene*	5.7	70-130	23	70-120	59-121	38
Hexachlorobenzene*	5.7	38-142	33	8-142	D-152	55
Hexachlorobutadiene*	2.7	68-130	38	38-120	24-120	62
Hexachloroethane*	4.8	55-130	32	55-120	40-120	52
Indeno(1,2,3-cd)pyrene*	11.1	13-151	60	D-151	D-171	99
Isophorone*	6.6	52-180	56	47-180	21-196	93
Naphthalene*	4.8	70-130	39	36-120	21-133	65
Nitrobenzene*	5.7	54-158	37	54-158	35-180	62
N-Nitrosodi-n-propylamine*	—	59-170	52	14-198	D-230	
Phenanthrene*	16.2	67-130	24	65-120	54-120	39
Pyrene*	5.7	70-130	30	70-120	52-120	49
1,2,4-Trichlorobenzene*	5.7	61-130	30	57-130	44-142	50
4-Chloro-3-methylphenol	9.0	68-130	44	41-128	22-147	73
2-Chlorophenol	9.9	55-130	37	36-120	23-134	61

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Analyte	ML (ug/L)	Range for Q (%)	Limit for s (%)	Range for \bar{X} (%)	Range for P1, P2 (%)	Limit for RPD (%)
2,4-Dichlorophenol	8.1	64-130	30	53-122	39-155	50
2,4-Dimethylphenol	8.1	58-130	35	42-120	32-120	58
2,4-Dinitrophenol	126	39-173	79	D-173	D-191	132
2-Methyl-4,6-dinitrophenol	72	56-130	122	53-130	D-181	203
2-Nitrophenol	10.8	61-163	33	45-167	29-182	55
4-Nitrophenol	7.2	35-130	79	13-129	D-132	131
Pentachlorophenol	10.8	42-152	52	38-152	14-176	86
Phenol	4.5	48-130	39	17-120	5-120	64
2,4,6-Trichlorophenol	8.1	69-130	35	52-129	37-144	58
4-Chloro-3-methylphenol	9.0					
2-Chlorophenol	9.9					
2,4-Dichlorophenol	8.1					
2,4-Dimethylphenol	8.1					
2,4-Dinitrophenol	126					
2-Methyl-4,6-dinitrophenol	72					
2-Nitrophenol	10.8					
4-Nitrophenol	7.2					
Acetophenone						
2-Acetylaminofluorene						
1-Acetyl-2-thiourea						
Alachlor						
Aldrin	5.7	7-152	39	7-152	D-166	81
Ametryn						
2-Aminoanthraquinone						
Aminoazobenzene						
4-Aminobiphenyl						
3-Amino-9-ethylcarbazole						
Anilazine						
Aniline						
o-Anisidine						
Aramite						
Atraton						
Atrazine						
Azinphos-methyl						
Barban						
Benzanthrone						
Benzenethiol						
Benzoic acid						
2,3-Benzofluorene						
p-Benzoquinone						
Benzyl alcohol						
alpha-BHC						

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Analyte	ML (ug/L)	Range for Q (%)	Limit for s (%)	Range for \bar{X} (%)	Range for P1, P2 (%)	Limit for RPD (%)
beta-BHC	9.3	42-131	37	42-131	24-149	61
gamma-BHC (Lindane)	12.6					
delta-BHC		D-130	77	D-120	D-120	129
Biphenyl						
Bromacil						
2-Bromochlorobenzene						
3-Bromochlorobenzene						
Bromoxynil						
Butachlor						
Butylate						
n-C10 (n-decane)						
n-C12 (n-undecane)						
n-C14 (n-tetradecane)						
n-C16 (n-hexadecane)						
n-C18 (n-octadecane)						
n-C20 (n-eicosane)						
n-C22 (n-docosane)						
n-C24 (n-tetracosane)						
n-C26 (n-hexacosane)						
n-C28 (n-octacosane)						
n-C30 (n-triacontane)						
Captafol						
Captan						
Carbaryl						
Carbazole						
Carbofuran						
Carboxin						
Carbophenothion						
Chlordane 3,5						
bis(2-Chloroethyl) ether	17.1	52-130	65	43-126	12-158	108
Chloroneb						
4-Chloroaniline						
Chlorobenzilate						
Chlorfenvinphos						
4-Chloro-2-methylaniline						
3-(Chloromethyl)pyridine hydrochloride						
4-Chloro-2-nitroaniline						
Chlorpropham						
Chlorothalonil						
1-Chloronaphthalene						
3-Chloronitrobenzene						

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Analyte	ML (ug/L)	Range for Q (%)	Limit for s (%)	Range for \bar{X} (%)	Range for P1, P2 (%)	Limit for RPD (%)
4-Chloro-1,2-phenylenediamine						
4-Chloro-1,3-phenylenediamine						
2-Chlorobiphenyl						
Chlorpyrifos						
Coumaphos						
m + p-Cresol						
o-Cresol						
p-Cresidine						
Crotoxyphos						
2-Cyclohexyl-4,6-dinitro-phenol						
Cyanazine						
Cycloate						
p-Cymene						
Dacthal (DCPA)						
4,4'-DDD	8.4	D-135	56	D-135	D-145	93
4,4'-DDE	16.8	19-130	46	19-120	4-136	77
4,4'-DDT	14.1	D-171	81	D-171	D-203	135
Demeton-O						
Demeton-S						
Diallate (cis or trans)						
2,4-Diaminotoluene						
Diazinon						
Dibenz(a,j)acridine						
Dibenzofuran						
Dibenzo(a,e)pyrene						
Dibenzothiophene						
1,2-Dibromo-3-chloropropane						
3,5-Dibromo-4-hydroxybenzonitrile						
2,6-Di-tert-butyl-p-benzoquinone						
Dichlone						
2,3-Dichloroaniline						
2,3-Dichlorobiphenyl						
2,6-Dichloro-4-nitroaniline						
2,3-Dichloronitrobenzene						
1,3-Dichloro-2-propanol						
2,6-Dichlorophenol						
Dichlorvos						
Dicrotophos						
Dieldrin 3	7.5	70-130	38	44-119	29-136	62
1,2:3,4-Diepoxybutane						
Di(2-ethylhexyl) adipate						

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Analyte	ML (ug/L)	Range for Q (%)	Limit for s (%)	Range for \bar{X} (%)	Range for P1, P2 (%)	Limit for RPD (%)
Diethylstilbestrol						
Diethyl sulfate						
Dilantin (5,5-Diphenylhydantoin)						
Dimethoate						
3,3'-Dimethoxybenzidine						
Dimethylaminoazobenzene						
7,12-Dimethylbenz(a)anthracene						
3,3'-Dimethylbenzidine						
N,N-Dimethylformamide						
3,6-Dimethylphenathrene						
alpha, alpha-Dimethylphenethylamine						
Dimethyl sulfone						
1,2-Dinitrobenzene						
1,3-Dinitrobenzene						
1,4-Dinitrobenzene						
Dinocap						
Dinoseb						
Diphenylamine						
Diphenyl ether						
1,2-Diphenylhydrazine						
Diphenamid						
Diphenyldisulfide						
Disulfoton						
Disulfoton sulfoxide						
Disulfoton sulfone						
Endosulfan I						
Endosulfan II						
Endosulfan sulfate	16.8	D-130	42	D-120	D-120	70
Endrin						
Endrin aldehyde		D-189	45	D-189	D-209	75
Endrin ketone						
EPN						
EPTC						
Ethion						
Ethoprop						
Ethyl carbamate						
Ethyl methanesulfonate						
Ethylenethiourea						
Etridiazole						
Ethynylestradiol-3-methyl ether						
Famphur						

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Analyte	ML (ug/L)	Range for Q (%)	Limit for s (%)	Range for \bar{X} (%)	Range for P1, P2 (%)	Limit for RPD (%)
Fenamiphos						
Fenarimol						
Fensulfothion						
Fenthion						
Fluchloralin						
Fluridone						
Heptachlor	5.7	D-172	44	D-172	D-192	74
Heptachlor epoxide	6.6	70-130	61	71-120	26-155	101
2,2',3,3',4,4',6- Heptachlorobiphenyl						
2,2',4,4',5',6-Hexachlorobiphenyl						
Hexachlorocyclopentadiene						
Hexachlorophene						
Hexachloropropene						
Hexamethylphosphoramide						
Hexanoic acid						
Hexazinone						
Hydroquinone						
Isodrin						
2-Isopropyl naphthalene						
Isosafrole						
Kepone						
Leptophos						
Longifolene						
Malachite green						
Malathion						
Maleic anhydride						
Merphos						
Mestranol						
Methapyrilene						
Methoxychlor						
2-Methylbenzothioazole						
3-Methylcholanthrene						
4,4'-Methylenebis(2-chloroaniline)						
4,4'-Methylenebis(N,N- dimethylaniline)						
4,5-Methylenephenanthrene						
1-Methylfluorene						
Methyl methanesulfonate						
2-Methylnaphthalene						
Methylparaoxon						
Methyl parathion						

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Analyte	ML (ug/L)	Range for Q (%)	Limit for s (%)	Range for \bar{X} (%)	Range for P1, P2 (%)	Limit for RPD (%)
1-Methylphenanthrene						
2-(Methylthio)benzothiazole						
Metolachlor						
Metribuzin						
Mevinphos						
Mexacarbate						
MGK 264						
Mirex						
Molinate						
Monocrotophos						
Naled						
Napropamide						
1,4-Naphthoquinone						
1-Naphthylamine						
2-Naphthylamine						
1,5-Naphthalenediamine						
Nicotine						
5-Nitroacenaphthene						
2-Nitroaniline						
3-Nitroaniline.						
4-Nitroaniline.						
5-Nitro-o-anisidine						
4-Nitrobiphenyl						
Nitrofen						
5-Nitro-o-toluidine						
Nitroquinoline-1-oxide						
N-Nitrosodi-n-butylamine						
N-Nitrosodiethylamine						
N-Nitrosodimethylamine						
N-Nitrosodiphenylamine						
N-Nitrosomethylethylamine						
N-Nitrosomethylphenylamine						
N-Nitrosomorpholine						
N-Nitrosopiperidine						
N-Nitrosopyrrolidine						
trans-Nonachlor						
Norflurazon						
2,2',3,3',4,5',6,6'- Octachlorobiphenyl						
Octamethyl pyrophosphoramide						
4,4'-Oxydianiline						
Parathion						

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Analyte	ML (ug/L)	Range for Q (%)	Limit for s (%)	Range for \bar{X} (%)	Range for P1, P2 (%)	Limit for RPD (%)
PCB-1016						
PCB-1221	90					
PCB-1232						
PCB-1242						
PCB-1248						
PCB-1254	108					
PCB-1260		19-130	77	19-130	D-164	128
PCB-1268						
Pebulate						
Pentachlorobenzene						
Pentachloronitrobenzene						
2,2',3,4',6-Pentachlorobiphenyl						
Pentachloroethane						
Pentamethylbenzene						
Perylene						
Phenacetin						
cis-Permethrin						
trans-Permethrin						
Phenobarbital						
Phenothiazene						
1,4-Phenylenediamine						
1-Phenylnaphthalene						
2-Phenylnaphthalene						
Phorate						
Phosalone						
Phosmet						
Phosphamidon						
Phthalic anhydride						
alpha-Picoline (2-Methylpyridine)						
Piperonyl sulfoxide						
Prometon						
Prometryn						
Pronamide						
Propachlor						
Propazine						
Propylthiouracil						
Pyridine						
Resorcinol (1,3-Benzenediol)						
Safrole						
Simazine						
Simetryn						
Squalene						

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Analyte	ML (ug/L)	Range for Q (%)	Limit for s (%)	Range for \bar{X} (%)	Range for P1, P2 (%)	Limit for RPD (%)
Stirofos						
Strychnine						
Styrene 9						
Sulfallate						
Tebuthiuron						
Terbacil..						
Terbufos						
Terbutryn						
alpha-Terpineol						
1,2,4,5-Tetrachlorobenzene						
2,2',4,4'-Tetrachlorobiphenyl						
2,3,7,8-Tetrachlorodibenzo-p-dioxin						
2,3,4,6-Tetrachlorophenol						
Tetrachlorvinphos						
Tetraethyl dithiopyrophosphate						
Tetraethyl pyrophosphate						
Thianaphthene (2,3-Benzothiophene)						
Thioacetamide						
Thionazin						
Thiophenol (Benzenethiol)						
Thioxanthone						
Toluene-1,3-diisocyanate						
Toluene-2,4-diisocyanate						
o-Toluidine						
Toxaphene 3,5						
Triadimefon						
1,2,3-Trichlorobenzene						
2,4,5-Trichlorobiphenyl						
2,3,6-Trichlorophenol						
2,4,5-Trichlorophenol						
Tricyclazole						
Trifluralin						
1,2,3-Trimethoxybenzene						
2,4,5-Trimethylaniline						
Trimethyl phosphate						
Triphenylene						
Tripropyleneglycolmethyl ether						
1,3,5-Trinitrobenzene						
Tris(2,3-dibromopropyl) phosphate						

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Analyte	ML (ug/L)	Range for Q (%)	Limit for s (%)	Range for \bar{X} (%)	Range for P1, P2 (%)	Limit for RPD (%)
Tri-p-tolyl phosphate						
O,O,O-Triethyl phosphorothioate.						
Trithiane						
Vernolate						

Many of the analytes in this table do not have QC acceptance criteria. If calibration is to be verified and other QC tests are to be performed for these analytes, acceptance criteria must be developed and applied. EPA has provided guidance for development of QC acceptance criteria (see 40 CFR 136.6(b)(2)(i) and *Protocol for EPA Approval of New Methods for Organic and Inorganic Analytes in Wastewater and Drinking Water* (EPA-821-B-98-003) March 1999). Alternatively, analytes that do not have acceptance criteria may be based on laboratory control charts, or 60 to 140% may be used.

* At a minimum, these compounds must be spiked into the MS/MSD analyses when direction cannot be obtained from the data user.

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Attachment VIII – Method 625.1 Suggested Internal Standards and Surrogates

Compound	Range for Surrogate Recovery	
	Calibration Verification	Recovery from Samples
Base/Neutral Fraction		
Acenaphthalene-d ₈	66-152	33-168
Acenaphthene-d ₁₀	71-141	30-180
Aniline-d ₅		
Anthracene-d ₁₀	58-171	53-142
Benzo(a)anthracene-d ₁₂	28-357	22-329
Benzo(a)pyrene-d ₁₂	32-194	32-194
4-Chloroaniline-d ₄	1-145	1-145
bis(2-Chloroethyl)ether-d ₈	52-194	25-222
Chrysene-d ₁₂	23-290	23-290
Decafluorobiphenyl		
4,4'-Dibromobiphenyl		
4,4'-Dibromooctafluorobiphenyl		
1,4-Dichlorobenzene-d ₄	65-153	11-245
2,2'-Difluorobiphenyl		
Dimethyl phthalate-d ₆	47-211	1-500
Fluoranthene-d ₁₀	61-164	38-172
4-Fluoroaniline		
1-Fluoronaphthalene		
2-Fluoronaphthalene		
2-Methylnaphthalene-d ₁₀	50-150	50-150
Naphthalene-d ₈	71-141	22-192
Nitrobenzene-d ₅	46-219	15-314
2,3,4,5,6-Pentafluorobiphenyl		
Perylene-d ₁₂		
Phenanthrene-d ₁₀	67-149	34-168
Pyrene-d ₁₀	48-210	28-196
Pyridine-d ₅		
Acid Fraction		
2-Chlorophenol-d ₄	55-180	33-180
2,4-Dichlorophenol-d ₃	64-157	34-182
4,6-Dinitro-2-methylphenol-d ₂	56-177	22-307
2-Fluorophenol		
4-Methylphenol-d ₈	25-111	25-111
2-Nitrophenol-d ₄	61-163	37-163
4-Nitrophenol-d ₄	35-287	6-500

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Compound	Range for Surrogate Recovery	
	Calibration Verification	Recovery from Samples
Pentafluorophenol		
2-Perfluoromethylphenol		
Phenol-d ₅	48-208	8-424

Many of the surrogates in this table do not have QC acceptance criteria. If calibration is to be verified and other QC tests are to be performed for these surrogates, acceptance criteria must be developed and applied. EPA has provided guidance for development of QC acceptance criteria (see 40 CFR 136.6(b)(2)(i) and *Protocol for EPA Approval of New Methods for Organic and Inorganic Analytes in Wastewater and Drinking Water* (EPA-821-B-98-003) March 1999). Alternatively, surrogates that do not have acceptance criteria may be based on laboratory control charts, or 60 to 140% may be used.

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Attachment IX – DoD Requirements

1.0 Equipment/Instrument Maintenance

Instrument maintenance must be performed routinely to optimize instrument performance and improve chromatography. Commonly performed maintenance includes baking columns, changing injection port liners, changing pump oil, etc. A new calibration curve must be analyzed following any major maintenance performed on the analytical system.

2.0 Computer Hardware and Software

Software name and version: HP Chemstation G1701CA Version C.00.00 or equivalent

3.0 Troubleshooting

Table 1. GCMS Troubleshooting Guide		
Problem	Cause	Treatment
Peaks broaden and tail	Poor column installation causing dead volume in the injector	Reinstall column in injector. Check seal at ferrule. Check insertion depth. Ensure a good column cut.
	Solvent flashing in hot injector	Reduce injection speed on hot injectors and if possible reduce injector temperature
	Injector not being purged properly after splitless injection	For splitless injection, the vent flow should be 70 ml/min, and the injector should be switched to the split mode 0.5_1.5 min after injection.
Tailing sample peaks for active components	Active sites in the injector insert or liner	Change or clean the injector insert
	Active sites or degraded phase in column	Remove the front 15 cm of the column and reinstall. If retention times are changing or cutting the column does not help, replace the column.
	Injector not hot enough for higher boiling compounds	Increase the injector temperature and lower the injection speed. Check that the graphite ferrule is free of cracks and the septum support is tight.
Low response and tailing of high boiling point compounds	Injector is not hot enough to vaporize high boilers	Increase injector temperature
	Interface/ion source not getting to adequate temperature	Change the manifold heater
Leading sample peaks	Column overload due to excess amount of component injected	Dilute the sample or do split injection
	Degradation of stationary phase	Change the column

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Table 1. GCMS Troubleshooting Guide

Problem	Cause	Treatment
	Carrier gas velocity too low	Increase carrier gas flow rate
Poor chromatographic resolution	Column temperature or program not optimized	Modify method by changing temperature ramp segment slopes
	Carrier gas flow rate not optimized	Decrease carrier gas linear velocity
	Stationary phase has degraded	Replace the column
Peak splitting, especially low boilers	Sample is flashing in the injector simulating two injections	Lower injector temperature
Retention times shift in chromatogram	Unstable carrier gas flow controller/regulator	Check pneumatics for leaks. Replace flow controller/ regulator if necessary.
	Column contamination or degradation	Condition or replace column
	Leaks at septum or column to injector connection	Replace septum regularly and check that the septum nut and the capillary column nut are tight
Cannot reach operating vacuum	Analyzer contaminated by diffusion pump oil	Shut down and clean mass spec
	Major air leak around column fitting into interface	Replace column ferrule and reseal compression fitting
No calibration gas peaks	Cal gas valve not open	Open cal gas valve
	Calibration gas solenoid valve stuck open. All calibration gas evaporated.	Have solenoid replaced. Put fresh PFBTA in the cal gas vial.
Analysis sensitivity has decreased	Background has increased	Check column bleed, septum bleed, pump oil, and ion source contamination
	Detector needs replacement	Replace detector
	Defective syringe	Try a new or proven syringe
	"Blown" septum or other massive leaks at the inlet or with carrier gas flow. Poor peak shapes usually result from bad leaks.	Find and fix leaks and adjust gas flow.
	Purge flow or split ratio too high	Adjust gas flow rates

4.0 Other Requirements

- 4.1 All hardcopy laboratory notebooks must be reviewed by the Supervisor, or their designee, on a monthly basis.

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- 4.2 If not self-explanatory (e.g., a typo or transposed number), corrections to technical and quality records shall also include a justification for the change.
- 4.3 A storage blank must be stored with all volatile organic samples, regardless of suspected concentration levels.
- 4.4 A person performing a manual integration must sign and date each manually integrated chromatogram and record the rationale for performing manual integration. Electronic signatures are acceptable.
- 4.5 The results of calibration and verification of support equipment must be within the specifications required of the application for which this equipment is used or the equipment must be removed from service until repaired. Calibration and verification records, including those of established correction factors, must be maintained. In the absence of method-specific requirements, the minimum requirements are as follows:

Table 2. Support Equipment Checks		
Performance Check	Frequency	Acceptance Criteria
Balance calibration check [Using two standard weights that bracket the expected mass]	Daily prior to use	Top-loading balance: $\pm 2\%$ or $\pm 0.02\text{g}$, whichever is greater Analytical balance: $\pm 0.1\%$ or $\pm 0.5\text{mg}$, whichever is greater
Verification of standard mass [Using weights traceable to the International System of Units (SI) through a NMI]	Every 5 years	Certificate of Calibration from ISO/IEC 17025 accredited calibration laboratory
Monitoring of refrigerator/freezer temperatures	Daily (i.e. 7 days per week) [use MIN/MAX thermometers or data loggers equipped with notification of out of control event capabilities if personnel not available to record daily]	Refrigerators: 0°C to 6°C Freezers: $\leq -10^{\circ}\text{C}$
Thermometer verification check [Using a thermometer traceable to the SI through an NMI] [Performed at two temperatures that bracket the target temperature(s). Assume linearity between the two bracketing temperatures.] [If only a single temperature is used, at the temperature of use]	Liquid in glass: Before first use and annually Electronic: Before first use and quarterly	Apply correction factors or replace thermometer
Volumetric labware	Class B: By lot before first use Class A and B: Upon evidence of deterioration	Bias: Mean within $\pm 2\%$ of nominal volume Precision: $\text{RSD} \leq 1\%$ of nominal volume (based on 10 replicate measurements)

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Table 2. Support Equipment Checks

Performance Check	Frequency	Acceptance Criteria
Non-volumetric labware [Applicable only when used for measuring initial sample volume and final extract/ digestates volume]	By lot before first use or upon evidence of deterioration	Bias: Mean within $\pm 3\%$ of nominal volume Precision: RSD $\leq 3\%$ of nominal volume (based on 10 replicate measurements)
Mechanical volumetric pipette	Quarterly	Bias: Mean within $\pm 2\%$ of nominal volume Precision: RSD $\leq 1\%$ of nominal volume (based on minimum of 3 replicate measurements) [Note: for variable volume pipettes, the nominal volume is the volume of use]
Glass microliter syringe	Upon receipt and upon evidence of deterioration	General Certificate of Bias & Precision upon receipt Replace if deterioration is evident
Drying oven temperature check	Daily prior to and after use	Within $\pm 5\%$ of set temperature
Water purification system	Daily prior to use	See method blank criteria given in Section 4.20 of this addendum

- 4.6 The expiration date of the prepared standard shall not exceed the expiration date of the primary standard. All containers must bear a preparation date.
- 4.7 To avoid preparing non-representative samples, the laboratory shall not “target” within a relatively small mass range (e.g., $1.00 \pm 0.01\text{g}$) because such targeting will produce non-representative subsamples if the sample has high heterogeneity. The laboratory shall not manipulate the sample material so the sample aliquot weighs exactly $1.00\text{g} \pm 0.01\text{g}$, as an example.
- 4.8 In the absence of project-specific requirements, the minimum standard data qualifiers to be used are:
- U Analyte was not detected and is reported as less than the LOD or as defined by the customer. The LOD has been adjusted for any dilution or concentration of the sample.
 - J The reported result is an estimated value (e.g., matrix interference was observed or the analyte was detected at a concentration outside the quantitation range).
 - B Blank contamination. The recorded result is associated with a contaminated blank.
 - N Non-target analyte. The analyte is a tentatively identified compound using mass spectrometry or any non-customer requested compounds that are tentatively identified.

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Q One or more quality control criteria failed (e.g., LCS recovery, surrogate spike recovery, or CCV recovery).

Additional data qualifiers may be used, or different letters or symbols to denote the qualifiers listed above, as long as they are appropriately defined and their use is consistent with project-specific requirements (e.g., QSM 5.0, the contract, and project-planning documents).

4.9 If the time of the sample collection is not provided, assume the most conservative time of day. For the purpose of batch processing, the start and stop dates and times of the batch preparation shall be recorded.

4.10 Each preparation method listed on the scope of accreditation must have quarterly LOD/LOQ verifications. However, not all possible combinations of preparation and cleanup techniques are required to have LOD/LOQ verifications. If LOD/LOQ verifications are not performed on all combinations, the laboratory must base the LOD/LOQ verifications on the worst case basis (preparation method with all applicable cleanup steps).

4.11 After each MDL determination, the laboratory must establish the LOD by spiking a quality system matrix at a concentration of at least 2 times but no greater than four times the MDL. This spike concentration establishes the LOD and the concentration at which the LOD shall be verified. It is specific to each suite of analyte, matrix, and method (including sample preparation). The following requirements apply to the initial LOD establishment and to the LOD verifications:

- The apparent signal to noise (S/N) ratio at the LOD must be at least three and the results must meet all method requirements for analyte identification (e.g., ion abundance, second column confirmation, or pattern recognition). For data systems that do not provide a measure of noise, the signal produced by the verification sample must produce a result that is at least three standard deviations greater than the mean method blank concentration. This is initially estimated based on a minimum of four method blank analyses and later established with a minimum of 20 method blank results.
- If the LOD verification fails, then the laboratory must repeat the MDL determination and LOD verification or perform and pass two consecutive LOD verifications at a higher spike concentration and set the LOD at the higher concentration.
- The laboratory shall maintain documentation for all MDL determinations and LOD verifications.
- The DL and LOD must be reported for all analyte-matrix-methods suites, unless it is not applicable to the test or specifically excluded by project requirements.

4.12 The LOD shall be verified quarterly. In situations where methods are setup and used on an infrequent basis, the laboratory may choose to perform LOD verifications on a one per batch basis. All verification data will be in compliance, reported, and available for review.

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- 4.13 For DoD, at a minimum, the LOQ shall be verified quarterly. In situations where methods are setup and used on an infrequent basis, the laboratory may choose to perform LOQ verifications on a one per batch basis.
- 4.14 All initial instrument calibrations must be verified with a standard obtained from a second manufacturer prior to analyzing any samples. The use of a standard from a second lot obtained from the same manufacturer (independently prepared from different source materials) is acceptable for use as a second source standard. The concentration of the second source standard shall be at or near the midpoint of the calibration range. The acceptance criteria for the initial calibration verification must be at least as stringent as those for the continuing calibration verification.
- 4.15 Exclusion of calibration points without documented scientifically valid technical justification is not permitted.
- 4.16 The concentration of the CCV standard shall be greater than the low calibration standard and less than or equal to the midpoint of the calibration range.
- 4.17 All CCVs analyzed must be evaluated and reported. If a CCV fails, reanalysis or corrective actions must be taken.
- If a CCV fails, the laboratory can immediately analyze two additional consecutive CCVs (immediately is defined as starting a consecutive pair within one hour; no samples can be run between the failed CCV and the two additional CCVs). This approach allows for spurious failures of analytes to be reported without reanalysis of samples. Any corrective actions that change the dynamics of the system (e.g., clip column, clean injection port, run blanks) requires that all samples since the last acceptable CCV be reanalyzed.
 - Both of these CCVs must meet acceptance criteria in order for the samples to be reported without reanalysis.
 - If either of these two CCVs fail or if the laboratory cannot immediately analyze two CCVs, the associated samples cannot be reported and must be reanalyzed.
 - Corrective action(s) and recalibration must occur if the above scenario fails. All affected samples since the last acceptable CCV must be reanalyzed.
 - Flagging of data for a failed CCV is only appropriate when the affected samples cannot be reanalyzed. The laboratory must notify the client prior to reporting data associated with a failed CCV.
- 4.18 The results of all MS/MSDs must be evaluated using the same acceptance criteria used for the DoD LCS limits (see Addendum Tables 3 through 6) or project limits, if specified. If the specific analyte(s) are not available in the Addendum Tables 3 through 6, the laboratory shall use their LCS in-house limits (see the LIMS) as a means of evaluating MS/MSDs. The MS and MSD must be spiked with all reported analytes.
- 4.19 Surrogate spike results shall be compared with DoD LCS limits (see Addendum Tables 3 through 6) or acceptance criteria specified by the client. If these criteria are not available, the laboratory shall compare the results with its in-house statistically established LCS criteria (see the LIMS).

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- 4.20 The method blank shall be considered to be contaminated if:
- The concentration of any target analyte (chemical of concern) in the blank exceeds 1/2 the LOQ and is greater than 1/10th the amount measured in any associated sample, or 1/10th the regulatory limit, whichever is greater;
 - The concentration of any common laboratory contaminant in the blank exceeds the LOQ;
 - If a method blank is contaminated as described above, then the laboratory shall reprocess affected samples in a subsequent preparation batch, except when sample results are below the LOD. If insufficient sample volume remains for reprocessing, the results shall be reported with appropriate data qualifiers.
- 4.21 Sporadic Marginal Exceedances are not allowed for target analytes (chemicals of concern as identified by a project) without project-specific approval. Target analytes are considered those few analytes that are critical for the success of a project (such as risk drivers) where sporadic marginal exceedances cannot be allowed. Laboratories should consult with clients whenever long lists of analytes are requested for analysis to determine if marginal exceedances will not be allowed.
- 4.22 DoD considers the same analyte exceeding the LCS control limit two (2) out of three (3) consecutive LCS to be indicative of non-random behavior, which requires corrective action and reanalysis of the LCS.

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TITLE: SEMIVOLATILE ORGANICS BY GC/MS (EPA METHODS 8270C, 8270D, 625, 625.1 AND SM 6410B), INCLUDING PROVISIONS FOR ANALYSIS IN SIM MODE

Table 3. LCS Control Limits – Method 8270 Solid Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
92-52-4	1,1-Biphenyl	1645	78.5	13	40	117
95-94-3	1,2,4,5-Tetrachlorobenzene	1810	77.8	13.7	37	119
120-82-1	1,2,4-Trichlorobenzene	3577	75.7	13.9	34	118
95-50-1	1,2-Dichlorobenzene	3352	74.6	14	33	117
528-29-0	1,2-Dinitrobenzene [1,2-DNB]	203	79.4	11.9	44	115
122-66-7	1,2-Diphenylhydrazine [Azobenzene]	2039	83	13.9	41	125
99-35-4	1,3,5-Trinitrobenzene [1,3,5-TNB]	154	89.2	10.7	57	121
541-73-1	1,3-Dichlorobenzene	3288	72.6	14.1	30	115
99-65-0	1,3-Dinitrobenzene [1,3-DNB]	598	84.6	14	43	127
106-46-7	1,4-Dichlorobenzene	3793	73.1	13.9	31	115
100-25-4	1,4-Dinitrobenzene	248	84.4	15.7	37	132
130-15-4	1,4-Naphthoquinone	150	81.2	8.8	55	108
90-13-1	1-Chloronaphthalene	119	81.1	11.1	48	115
90-12-0	1-Methylnaphthalene	3004	79.2	13.2	40	119
58-90-2	2,3,4,6-Tetrachlorophenol	1724	84.7	13.6	44	125
935-95-5	2,3,5,6-Tetrachlorophenol	227	75.9	11.9	40	112
608-27-5	2,3-Dichloroaniline	108	82.4	13	44	121
95-95-4	2,4,5-Trichlorophenol	4014	82.6	13.7	41	124
118-79-6	2,4,6-Tribromophenol	2930	85.7	15.4	39	132
88-06-2	2,4,6-Trichlorophenol	4183	82.1	14.5	39	126
120-83-2	2,4-Dichlorophenol	3794	80.9	13.7	40	122
105-67-9	2,4-Dimethylphenol	3886	78.4	16.2	30	127
121-14-2	2,4-Dinitrotoluene	4075	86.8	12.9	48	126
87-65-0	2,6-Dichlorophenol	1364	79.2	12.6	41	117
606-20-2	2,6-Dinitrotoluene	3706	85	13	46	124
53-96-3	2-Acetylaminofluorene	175	94	13.3	54	134
91-58-7	2-Chloronaphthalene	3569	77.5	12.1	41	114
95-57-8	2-Chlorophenol	3977	77.3	14.5	34	121
321-60-8	2-Fluorobiphenyl	3191	79.5	11.8	44	115
367-12-4	2-Fluorophenol	3008	75.2	13.3	35	115
91-57-6	2-Methylnaphthalene	5059	80.1	14	38	122
95-48-7	2-Methylphenol (o-Cresol)	4016	77	14.9	32	122
88-74-4	2-Nitroaniline	3639	85.4	13.8	44	127
119-75-5	2-Nitrodiphenylamine	279	88.1	11.6	53	123
88-75-5	2-Nitrophenol	3804	79.6	14.5	36	123
109-06-8	2-Picoline [2-Methylpyridine]	181	64.5	12.7	27	103
91-94-1	3,3'-Dichlorobenzidine	3521	71.3	16.5	22	121
56-49-5	3-Methylcholanthrene	188	95.1	13	56	134

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Table 3. LCS Control Limits – Method 8270 Solid Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
99-09-2	3-Nitroaniline	3454	75.9	14.3	33	119
65794-96-9	3/4-Methylphenol [m/p-Cresol]	2900	76.5	14.1	34	119
534-52-1	4,6-Dinitro-2-methylphenol	3739	80.7	17.2	29	132
101-55-3	4-Bromophenyl phenyl ether	3708	85.1	13	46	124
59-50-7	4-Chloro-3-methylphenol	3880	83.3	12.9	45	122
106-47-8	4-Chloroaniline [p-Chloroaniline]	3435	61.3	14.9	17	106
7005-72-3	4-Chlorophenyl phenyl ether	3673	83	12.7	45	121
106-44-5	4-Methylphenol [p-Cresol]	1555	84.1	14.1	42	126
100-02-7	4-Nitrophenol	3976	80.6	17	30	132
99-55-8	5-Nitro-o-toluidine [2-Amino-4-nitrotoluene]	187	69.8	15.8	23	117
57-97-6	7,12-Dimethylbenz(a)-anthracene	338	96.2	15.3	50	142
83-32-9	Acenaphthene	5300	81.3	13.7	40	123
208-96-8	Acenaphthylene	5194	81.8	16.8	32	132
98-86-2	Acetophenone	2101	73.9	13.6	33	115
120-12-7	Anthracene	5250	85.2	12.7	47	123
1912-24-9	Atrazine	1428	87.1	13.4	47	127
103-33-3	Azobenzene	378	82.1	14.2	39	125
56-55-3	Benz(a)anthracene	5385	87.4	12.9	49	126
50-32-8	Benzo(a)pyrene	5500	86.9	13.9	45	129
205-99-2	Benzo(b)fluoranthene	5323	88.3	14.5	45	132
191-24-2	Benzo(g,h,i)perylene	5263	88.5	15.1	43	134
207-08-9	Benzo(k)fluoranthene	5386	89.6	14.2	47	132
100-51-6	Benzyl alcohol	2895	75.7	15.6	29	122
111-91-1	bis(2-Chloroethoxy)methane	3705	78.4	14.2	36	121
111-44-4	Bis(2-chloroethyl) ether	3711	75.4	14.9	31	120
39638-32-9	bis(2-Chloroisopropyl) ether	769	82	16.3	33	131
117-81-7	Bis(2-ethylhexyl) phthalate	4018	91.9	13.7	51	133
103-23-1	bis(2-Ethylhexyl)adipate	156	90.8	10.1	61	121
85-68-7	Butyl benzyl phthalate	3956	90.3	14	48	132
105-60-2	Caprolactam	1203	81.3	11.9	46	117
86-74-8	Carbazole	3095	86.3	12	50	123
510-15-6	Chlorobenzilate	172	99.7	16.9	49	150
218-01-9	Chrysene	5395	87.1	12.2	50	124
84-74-2	Di-n-butyl phthalate	4041	89.4	12.8	51	128
117-84-0	Di-n-octyl phthalate	3985	92.4	16	45	140
2303-16-4	Diallate [cis or trans]	173	93.7	12.7	56	132
53-70-3	Dibenzo(a,h)anthracene	5393	89.5	14.7	45	134

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Table 3. LCS Control Limits – Method 8270 Solid Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
132-64-9	Dibenzofuran	3749	81.5	12.7	44	120
84-66-2	Diethyl phthalate	4012	87.2	12.3	50	124
60-51-5	Dimethoate	137	68	13.3	28	108
131-11-3	Dimethyl phthalate	4023	85.9	12.6	48	124
60-11-7	Dimethylaminoazobenzene	177	98.7	11.6	64	134
88-85-7	Dinoseb	123	67.3	17.1	16	119
101-84-8	Diphenyl ether	114	95.6	6	78	114
122-39-4	Diphenylamine	854	79.5	10.6	48	111
62-50-0	Ethyl methanesulfonate	174	85.1	16.9	34	136
206-44-0	Fluoranthene	5340	88.3	12.9	50	127
86-73-7	Fluorene	5150	84.2	13.8	43	125
118-74-1	Hexachlorobenzene	4138	83.5	13	45	122
87-68-3	Hexachlorobutadiene	4003	77.3	15.3	32	123
67-72-1	Hexachloroethane	4049	72.2	14.9	28	117
1888-71-7	Hexachloropropene	259	81.9	16.7	32	132
95-13-6	Indene	188	85.3	8.9	59	112
193-39-5	Indeno(1,2,3-cd)pyrene	5367	89.3	14.7	45	133
465-73-6	isodrin	167	93.8	12.8	56	132
78-59-1	Isophorone	3787	75.9	15.2	30	122
120-58-1	Isosafrole	174	89.5	15.4	43	136
66-27-3	Methyl methanesulfonate	150	77.9	13.1	38	117
100-75-4	N-Nitrosopiperidine	232	89.4	9.8	60	119
924-16-3	N-Nitrosodi-n-butylamine	236	91.7	10.8	59	124
621-64-7	N-Nitrosodi-n-propylamine	3857	78.2	13.9	36	120
55-18-5	N-nitrosodiethylamine	421	82.1	13.8	41	124
62-75-9	N-Nitrosodimethylamine	3170	71.6	16.2	23	120
86-30-6	N-Nitrosodiphenylamine	2968	82.7	14.8	38	127
10595-95-6	n-Nitrosomethylethylamine	265	78.7	14.9	34	123
59-89-2	n-Nitrosomorpholine	172	91.3	13.8	50	133
930-55-2	n-Nitrosopyrrolidine	326	85.5	13.6	45	126
91-20-3	Naphthalene	5342	78.8	14.7	35	123
98-95-3	Nitrobenzene	4103	77.8	14.7	34	122
4165-60-0	Nitrobenzene-d5	3226	79.3	14.2	37	122
56-57-5	Nitroquinoline-1-oxide	177	91.3	24.5	18	165
126-68-1	O,O,O-Triethyl phosphorothioate	138	91.6	10.8	59	124
593-45-3	Octadecane	113	87.4	14.5	44	131
608-93-5	Pentachlorobenzene	346	89.7	11.8	54	125
76-01-7	Pentachloroethane	131	70.4	10.6	39	102
87-86-5	Pentachlorophenol	4161	78.7	18	25	133

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Table 3. LCS Control Limits – Method 8270 Solid Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
82-68-8	Pentachloronitrobenzene	579	86.1	16	38	134
62-44-2	Phenacetin	185	95	12.5	57	133
85-01-8	Phenanthrene	5259	85.4	12	50	121
108-95-2	Phenol	4029	77.3	14.4	34	121
4165-62-2	Phenol-d5	1016	77.4	14.9	33	122
23950-58-5	Pronamide	179	93	12.4	56	130
129-00-0	Pyrene	5518	87.2	13.3	47	127
91-22-5	Quinoline	219	90	11.9	54	126
94-59-7	Safrole	176	87.8	13.6	47	129
1718-51-0	Terphenyl-d14	3111	90.5	12.3	54	127
3689-24-5	Tetraethyl dithiopyrophosphate [Sulfotep]	136	94.4	14	52	137
297-97-2	Thionazine	139	94.6	10.7	62	127

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Table 4. LCS Control Limits – Method 8270 Water Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
92-52-4	1,1-Biphenyl	2247	82.1	11.1	49	115
95-94-3	1,2,4,5-Tetrachlorobenzene	2326	77.9	14.5	35	121
120-82-1	1,2,4-Trichlorobenzene	4716	72.6	14.5	29	116
95-50-1	1,2-Dichlorobenzene	4442	71.4	13.3	32	111
528-29-0	1,2-Dinitrobenzene [1,2-DNB]	112	83.9	8.3	59	109
122-66-7	1,2-Diphenylhydrazine [Azobenzene]	2244	85.4	12.2	49	122
99-35-4	1,3,5-Trinitrobenzene [1,3,5-TNB]	241	89.1	16	41	137
541-73-1	1,3-Dichlorobenzene	4375	68.6	13.6	28	110
99-65-0	1,3-Dinitrobenzene [1,3-DNB]	601	88.2	13.1	49	128
106-46-7	1,4-Dichlorobenzene	5433	70.4	13.9	29	112
90-13-1	1-Chloronaphthalene	211	84.5	8.8	58	111
90-12-0	1-Methylnaphthalene	3742	80	13.1	41	119
134-32-7	1-Naphthylamine	258	73.7	16.6	24	124
58-90-2	2,3,4,6-Tetrachlorophenol	2293	89	13	50	128
935-95-5	2,3,5,6-Tetrachlorophenol	266	85.6	11.7	50	121
608-27-5	2,3-Dichloroaniline	150	99.2	9.8	70	129
95-95-4	2,4,5-Trichlorophenol	5707	88.1	11.8	53	123
118-79-6	2,4,6-Tribromophenol	2059	91.5	16	43	140
88-06-2	2,4,6-Trichlorophenol	6136	87.2	12.4	50	125
120-83-2	2,4-Dichlorophenol	5330	84	12.2	47	121
105-67-9	2,4-Dimethylphenol	5298	77.5	15.6	31	124
51-28-5	2,4-Dinitrophenol	5127	82.9	20	23	143
121-14-2	2,4-Dinitrotoluene	6032	92.3	11.8	57	128
87-65-0	2,6-Dichlorophenol	1583	84	11.4	50	118
606-20-2	2,6-Dinitrotoluene	5107	90.7	11.2	57	124
53-96-3	2-Acetylaminofluorene	228	98.9	12.9	60	138
91-58-7	2-Chloronaphthalene	5084	78	12.8	40	116
95-57-8	2-Chlorophenol	5571	77.5	13.2	38	117
93951-73-6	2-Chlorophenol-d4	119	79.9	8.7	54	106
321-60-8	2-Fluorobiphenyl	2263	81.2	12.4	44	119
367-12-4	2-Fluorophenol	2022	68.8	16.6	19	119
91-57-6	2-Methylnaphthalene	6330	80.7	13.6	40	121
95-48-7	2-Methylphenol (o-Cresol)	5800	73	14.5	30	117
88-74-4	2-Nitroaniline	4855	90.8	12.1	55	127
119-75-5	2-Nitrodiphenylamine	272	97.3	11.3	64	131
88-75-5	2-Nitrophenol	5097	84.6	12.7	47	123
109-06-8	2-Picoline [2-Methylpyridine]	195	71.6	12.6	34	109
91-94-1	3,3'-Dichlorobenzidine	4815	77.9	16.9	27	129

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Table 4. LCS Control Limits – Method 8270 Water Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
56-49-5	3-Methylcholanthrene	237	94	12.8	56	133
99-09-2	3-Nitroaniline	4808	84.4	14.5	41	128
65794-96-9	3/4-Methylphenol [m/p-Cresol]	3472	69.7	13.6	29	110
534-52-1	4,6-Dinitro-2-methylphenol	5097	90.1	15.5	44	137
101-55-3	4-Bromophenyl phenyl ether	5074	89.1	11.5	55	124
59-50-7	4-Chloro-3-methylphenol	5338	85.5	11.3	52	119
106-47-8	4-Chloroaniline [p-Chloroaniline]	4687	75.3	14	33	117
7005-72-3	4-Chlorophenyl phenyl ether	5071	86.7	11.3	53	121
106-44-5	4-Methylphenol [p-Cresol]	2798	72.5	15.8	25	120
99-55-8	5-Nitro-o-toluidine [2-amino-4-nitrotoluene]	260	82.1	14.6	38	126
57-97-6	7,12-Dimethylbenz(a)-anthracene	373	97.1	11.9	61	133
83-32-9	Acenaphthene	6952	84.5	12.3	47	122
208-96-8	Acenaphthylene	6662	85.3	14.7	41	130
98-86-2	Acetophenone	2877	82.1	12	46	118
120-12-7	Anthracene	6792	89.6	11	57	123
140-57-8	Aramite	100	82.8	16.3	34	132
1912-24-9	Atrazine	2328	92.8	16.4	44	142
103-33-3	Azobenzene	578	88.5	9.3	61	116
56-55-3	Benz(a)anthracene	6867	91.6	11.1	58	125
50-32-8	Benzo(a)pyrene	7045	90.8	12.4	54	128
205-99-2	Benzo(b)fluoranthene	6767	92	12.9	53	131
191-24-2	Benzo(g,h,i)perylene	6624	92	13.9	50	134
207-08-9	Benzo(k)fluoranthene	6803	93.2	12.1	57	129
100-51-6	Benzyl alcohol	3349	71.2	13.5	31	112
111-91-1	bis(2-Chloroethoxy)methane	5094	83.9	11.9	48	120
111-44-4	Bis(2-chloroethyl) ether	5139	80.8	12.6	43	118
39638-32-9	bis(2-Chloroisopropyl) ether	1140	83.4	15.4	37	130
117-81-7	Bis(2-ethylhexyl) phthalate	5288	95.2	13.3	55	135
85-68-7	Butyl benzyl phthalate	5173	93.3	13.5	53	134
86-74-8	Carbazole	4187	91.1	10.4	60	122
510-15-6	Chlorobenzilate	226	104.3	15.4	58	150
218-01-9	Chrysene	6779	91.3	10.7	59	123
124-18-5	Decane	126	66.9	12.8	29	105
84-74-2	Di-n-butyl phthalate	5329	93	11.4	59	127
117-84-0	Di-n-octyl phthalate	5222	95.5	15	51	140
2303-16-4	Diallate [cis or trans]	249	95.3	9.6	67	124
226-36-8	Dibenz(a,h)acridine	136	104.4	9.7	75	134

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Table 4. LCS Control Limits – Method 8270 Water Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
53-70-3	Dibenzo(a,h)anthracene	6840	92.7	13.8	51	134
132-64-9	Dibenzofuran	4963	85.3	10.8	53	118
84-66-2	Diethyl phthalate	5207	90.1	11.5	56	125
131-11-3	Dimethyl phthalate	4977	86	13.7	45	127
60-11-7	Dimethylaminoazobenzene	238	97.1	11.6	62	132
88-85-7	Dinoseb	144	93.4	10.8	61	126
101-84-8	Diphenyl ether	142	91.7	7.8	68	115
122-39-4	Diphenylamine	754	83	9.2	55	111
298-04-4	Disulfoton	122	92.5	12.5	55	130
62-50-0	Ethyl methanesulfonate	215	90.1	9.4	62	118
206-44-0	Fluoranthene	6826	92.6	11.9	57	128
86-73-7	Fluorene	6786	88.1	12	52	124
118-74-1	Hexachlorobenzene	6263	88.7	12.1	53	125
87-68-3	Hexachlorobutadiene	5878	73.1	16.9	22	124
39638-32-9	bis(2-Chloroisopropyl) ether	1140	83.4	15.4	37	130
117-81-7	Bis(2-ethylhexyl) phthalate	5288	95.2	13.3	55	135
85-68-7	Butyl benzyl phthalate	5173	93.3	13.5	53	134
86-74-8	Carbazole	4187	91.1	10.4	60	122
510-15-6	Chlorobenzilate	226	104.3	15.4	58	150
218-01-9	Chrysene	6779	91.3	10.7	59	123
124-18-5	Decane	126	66.9	12.8	29	105
84-74-2	Di-n-butyl phthalate	5329	93	11.4	59	127
117-84-0	Di-n-octyl phthalate	5222	95.5	15	51	140
2303-16-4	Diallate [cis or trans]	249	95.3	9.6	67	124
226-36-8	Dibenz(a,h)acridine	136	104.4	9.7	75	134
53-70-3	Dibenzo(a,h)anthracene	6840	92.7	13.8	51	134
132-64-9	Dibenzofuran	4963	85.3	10.8	53	118
84-66-2	Diethyl phthalate	5207	90.1	11.5	56	125
131-11-3	Dimethyl phthalate	4977	86	13.7	45	127
60-11-7	Dimethylaminoazobenzene	238	97.1	11.6	62	132
88-85-7	Dinoseb	144	93.4	10.8	61	126
101-84-8	Diphenyl ether	142	91.7	7.8	68	115
122-39-4	Diphenylamine	754	83	9.2	55	111
298-04-4	Disulfoton	122	92.5	12.5	55	130
62-50-0	Ethyl methanesulfonate	215	90.1	9.4	62	118
206-44-0	Fluoranthene	6826	92.6	11.9	57	128
86-73-7	Fluorene	6786	88.1	12	52	124
118-74-1	Hexachlorobenzene	6263	88.7	12.1	53	125
87-68-3	Hexachlorobutadiene	5878	73.1	16.9	22	124
67-72-1	Hexachloroethane	5904	68	15.7	21	115

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Table 4. LCS Control Limits – Method 8270 Water Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
95-13-6	Indene	253	93.8	13.7	53	135
193-39-5	Indeno(1,2,3-cd)pyrene	6880	92.6	13.6	52	134
465-73-6	isodrin	212	97.6	10	68	128
78-59-1	Isophorone	5190	83.3	13.7	42	124
120-58-1	Isosafrole	230	91.1	11.8	56	126
66-27-3	Methyl methanesulfonate	237	70.1	12.3	33	107
298-00-0	Methyl parathion	121	101.6	19	45	159
100-75-4	N-Nitrosopiperidine	299	88.6	10.8	56	121
924-16-3	N-Nitrosodi-n-butylamine	322	90.4	10.3	60	121
621-64-7	N-Nitrosodi-n-propylamine	5145	84	11.7	49	119
55-18-5	N-nitrosodiethylamine	488	81.8	12.9	43	121
86-30-6	N-Nitrosodiphenylamine	3743	86.8	11.9	51	123
10595-95-6	n-Nitrosomethylethylamine	311	78.7	12.7	41	117
59-89-2	n-Nitrosomorpholine	214	86.2	10.3	55	117
930-55-2	n-Nitrosopyrrolidine	716	80.8	10.8	48	113
91-20-3	Naphthalene	6953	80	13.5	40	121
98-95-3	Nitrobenzene	5955	83	12.8	45	121
4165-60-0	Nitrobenzene-d5	2223	82.1	12.6	44	120
126-68-1	O,O,O-Triethyl phosphorothioate	212	92.6	8.8	66	119
95-53-4	o-Toluidine	296	69.9	13.2	30	110
593-45-3	Octadecane	151	89	13.1	50	128
56-38-2	Parathion	152	102.6	12.3	66	140
608-93-5	Pentachlorobenzene	401	91.1	10.7	59	123
76-01-7	Pentachloroethane	139	60.9	10.4	30	92
87-86-5	Pentachlorophenol	6083	86.4	17.1	35	138
82-68-8	Pentchloronitrobenzene	618	94.5	13.4	54	135
62-44-2	Phenacetin	241	97.9	8.9	71	124
85-01-8	Phenanthrene	6822	89.6	10.2	59	120
298-02-2	Phorate	126	88.6	16.8	38	139
23950-58-5	Pronamide	249	97	10.5	65	129
129-00-0	Pyrene	7013	91.1	11.5	57	126
91-22-5	Quinoline	249	100.1	10.5	69	132
94-59-7	Safrole	233	90	9.7	61	119
1718-51-0	Terphenyl-d14	1893	91.7	13.9	50	134
3689-24-5	Tetraethyl dithiopyrophosphate [Sulfotep]	200	96.7	11.9	61	133
297-97-2	Thionazine	196	102	10.1	72	132

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Table 5. LCS Control Limits – Method 8270 SIM Solid Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
90-12-0	1-Methylnaphthalene	2267	76.6	11.3	43	111
95-95-4	2,4,5-Trichlorophenol	169	79.9	14.9	35	125
91-58-7	2-Chloronaphthalene	615	76.7	10.5	45	108
321-60-8	2-Fluorobiphenyl	1961	80.6	11.6	46	115
91-57-6	2-Methylnaphthalene	2535	76.8	12.5	39	114
83-32-9	Acenaphthene	2813	77.7	11.2	44	111
208-96-8	Acenaphthylene	2761	77.1	12.8	39	116
120-12-7	Anthracene	2812	82.1	10.7	50	114
56-55-3	Benz(a)anthracene	2827	88	11.4	54	122
50-32-8	Benzo(a)pyrene	2789	87.3	12.5	50	125
205-99-2	Benzo(b)fluoranthene	2790	90.3	12.6	53	128
191-24-2	Benzo(g,h,i)perylene	2739	87.8	13	49	127
207-08-9	Benzo(k)fluoranthene	2761	89.3	11.2	56	123
111-44-4	Bis(2-chloroethyl) ether	192	65.4	15.8	18	113
117-81-7	Bis(2-ethylhexyl) phthalate	181	108.9	13.9	67	150
85-68-7	Butyl benzyl phthalate	144	103.5	10.6	72	135
86-74-8	Carbazole	183	79.3	14.6	36	123
218-01-9	Chrysene	2812	87.5	10.2	57	118
84-74-2	Di-n-butyl phthalate	150	106.5	12.9	68	145
117-84-0	Di-n-octyl phthalate	144	105.5	16.8	55	156
53-70-3	Dibenzo(a,h)anthracene	2778	89.2	13.2	50	129
132-64-9	Dibenzofuran	282	71.9	12.2	35	108
84-66-2	Diethyl phthalate	147	99.3	10.9	67	132
131-11-3	Dimethyl phthalate	149	99.3	9.3	71	127
206-44-0	Fluoranthene	2782	87.3	10.7	55	119
86-73-7	Fluorene	2795	80.6	11.2	47	114
118-74-1	Hexachlorobenzene	201	81.9	14.2	39	125
193-39-5	Indeno(1,2,3-cd)pyrene	2812	89.6	13.5	49	130
62-75-9	N-Nitrosodimethylamine	117	90.7	10.9	58	124
91-20-3	Naphthalene	2823	74.7	12.2	38	111
4165-60-0	Nitrobenzene-d5	531	84.7	13.6	44	125
87-86-5	Pentachlorophenol	259	82.4	15.5	36	129
85-01-8	Phenanthrene	2792	80.8	10.6	49	113
129-00-0	Pyrene	2792	85.8	10.2	55	117
1718-51-0	Terphenyl-d14	1864	95.3	12.6	58	133

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Table 6. LCS Control Limits – Method 8270 SIM Water Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
92-52-4	1,1-Biphenyl	106	77.3	7.3	56	99
90-12-0	1-Methylnaphthalene	2566	77.9	12.5	41	115
95-95-4	2,4,5-Trichlorophenol	488	84.1	13.4	44	124
118-79-6	2,4,6-Tribromophenol	164	83.7	12.7	46	122
606-20-2	2,6-Dinitrotoluene	118	67.2	15.8	20	115
91-58-7	2-Chloronaphthalene	717	72.4	12.7	34	111
321-60-8	2-Fluorobiphenyl	747	79.2	8.8	53	106
91-57-6	2-Methylnaphthalene	2984	76.5	12.6	39	114
83-32-9	Acenaphthene	3241	80.9	11.1	48	114
208-96-8	Acenaphthylene	3234	77.8	14.4	35	121
120-12-7	Anthracene	3224	85.8	11	53	119
56-55-3	Benz(a)anthracene	3277	89.3	10.1	59	120
50-32-8	Benzo(a)pyrene	3284	86.4	11.2	53	120
205-99-2	Benzo(b)fluoranthene	3248	89.7	12.3	53	126
191-24-2	Benzo(g,h,i)perylene	3178	86	14.1	44	128
207-08-9	Benzo(k)fluoranthene	3167	89.3	11.9	54	125
111-44-4	Bis(2-chloroethyl) ether	775	77.8	12.6	40	116
117-81-7	Bis(2-ethylhexyl) phthalate	275	114.1	19.6	55	173
85-68-7	Butyl benzyl phthalate	159	90.7	17.3	39	143
86-74-8	Carbazole	631	84	13.1	45	123
218-01-9	Chrysene	3215	88.3	10.4	57	120
84-74-2	Di-n-butyl phthalate	153	102.5	14.2	60	145
117-84-0	Di-n-octyl phthalate	157	103.3	19	46	160
53-70-3	Dibenzo(a,h)anthracene	3233	87.2	14.5	44	131
132-64-9	Dibenzofuran	864	77.5	14.1	35	120
84-66-2	Diethyl phthalate	142	94.5	13.5	54	135
206-44-0	Fluoranthene	3242	89.1	10.4	58	120
86-73-7	Fluorene	3232	84.1	11.3	50	118
118-74-1	Hexachlorobenzene	947	84.8	13	46	124
87-68-3	Hexachlorobutadiene	187	84.5	14.7	40	129
193-39-5	Indeno(1,2,3-cd)pyrene	3244	88.7	13.7	48	130
62-75-9	N-Nitrosodimethylamine	162	62.5	10	33	92
91-20-3	Naphthalene	3277	78.8	11.9	43	114
4165-60-0	Nitrobenzene-d5	444	83.1	9.2	55	111
87-86-5	Pentachlorophenol	808	88.4	17.6	36	141
85-01-8	Phenanthrene	3240	83.6	10.3	53	115
129-00-0	Pyrene	3252	87.1	11.3	53	121
1718-51-0	Terphenyl-d14	642	95.1	12.4	58	132

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Table 7. Quality Control Requirements – Organic Analysis by Gas Chromatography/Mass Spectrometry

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Tune Check	Prior to ICAL and prior to each 12-hour period of sample analysis.	Specific ion abundance criteria of DFTPP from method.	Retune instrument and verify.	Flagging is not appropriate.	No samples shall be analyzed without a valid tune.
Performance Check	At the beginning of each 12-hour period, prior to analysis of samples.	Degradation $\leq 20\%$ for DDT. Benzidine and penta-chlorophenol shall be present at their normal responses, and shall not exceed a tailing factor of 2.	Correct problem, then repeat performance checks.	Flagging is not appropriate.	No samples shall be analyzed until performance check is within criteria. The DDT breakdown and Benzidine/Pentachlorophenol tailing factors are considered overall system checks to evaluate injector port inertness and column performance and are required regardless of the reported analyte list.
Initial calibration (ICAL) for all analytes (including surrogates)	At instrument set-up, prior to sample analysis	Each analyte must meet one of the three options below: Option 1: RSD for each analyte $\leq 15\%$; Option 2: linear least squares regression for each analyte: $r^2 \geq 0.99$; Option 3: non-linear least squares regression (quadratic) for each analyte: $r^2 \geq 0.99$.	Correct problem then repeat ICAL.	Flagging is not appropriate.	Minimum 5 levels for linear and 6 levels for quadratic. No samples shall be analyzed until ICAL has passed. If the specific version of a method requires additional evaluation (e.g., RFs or low calibration standard analysis and recovery criteria) these additional requirements must also be met.

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Table 7. Quality Control Requirements – Organic Analysis by Gas Chromatography/Mass Spectrometry

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Retention Time window position establishment	Once per ICAL and at the beginning of the analytical sequence.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA.	NA.	Required for each analyte and surrogate.
Evaluation of Relative Retention Times(RRT)	With each sample.	RRT of each reported analyte within ± 0.06 RRT units.	Correct problem, then rerun ICAL.	NA	RRTs may be updated based on the daily CCV. RRTs shall be compared with the most recently updated RRTs.
Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis	All reported analytes within $\pm 20\%$ of true value.	Correct problem. Rerun ICV. If that fails, repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed until calibration has been verified with a second source.

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Table 7. Quality Control Requirements – Organic Analysis by Gas Chromatography/Mass Spectrometry

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Continuing Calibration Verification (CCV)	Daily before sample analysis; after every 12 hours of analysis time; and at the end of the analytical batch run.	All reported analytes and surrogates within $\pm 20\%$ of true value. All reported analytes and surrogates within $\pm 50\%$ for end of analytical batch CCV.	Recalibrate, and reanalyze all affected samples since the last acceptable CCV; or Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected samples since the	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since last acceptable calibration verification.	Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed. If the specific version of a method requires additional evaluation (e.g., average RFs) these additional requirements must also be met.
Internal standards (IS)	Every field sample, standard and QC sample.	Retention time within ± 10 seconds from retention time of the midpoint standard in the ICAL; EICP area within - 50% to +100% of ICAL midpoint standard.	Inspect mass spectrometer and GC for malfunctions and correct problem. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	If corrective action fails in field samples, data must be qualified and explained in the case narrative. Apply Q-flag to analytes associated with the non-compliant IS. Flagging is not appropriate for failed standards.	

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Table 7. Quality Control Requirements – Organic Analysis by Gas Chromatography/Mass Spectrometry

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Method Blank (MB)	One per preparatory batch.	No analytes detected > ½ LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater. Common contaminants must not be detected > LOQ.	Correct problem. If required, reprep and reanalyze MB and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Laboratory Control Sample (LCS)	One per preparatory batch.	A laboratory must use the limits in Tables 3 through 6 for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits (see the LIMS) if project limits are not specified.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Must contain all surrogates and all analytes to be reported. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

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Table 7. Quality Control Requirements – Organic Analysis by Gas Chromatography/Mass Spectrometry

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Matrix Spike (MS)	One per preparatory batch.	A laboratory must use the limits in Tables 3 through 6 for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits (see the LIMS) if project limits are not specified.	Examine the project specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	Must contain all surrogates and all analytes to be reported. If MS results are outside the limits, the data shall be evaluated to determine the source(s) of difference, i.e., matrix effect or analytical error.
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch.	A laboratory must use the limits in Tables 3 through 6 for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits (see the LIMS) if project limits are not specified. MSD or MD: RPD of all analytes $\leq 20\%$ (between MS and MSD or sample and MD).	Examine the project specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	MSD: Must contain all surrogates and all analytes to be reported. The data shall be evaluated to determine the source of difference.

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Table 7. Quality Control Requirements – Organic Analysis by Gas Chromatography/Mass Spectrometry

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Surrogate Spike	All field and QC samples.	QC acceptance criteria specified by the project, if available; otherwise use limits in Tables 3 through 6 or in-house LCS limits (see the LIMS) if analyte(s) are not listed.	Correct problem, then reprep and reanalyze all failed samples for all surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary.	Apply Q-flag to all associated analytes if acceptance criteria are not met and explain in the case narrative.	Alternative surrogates are recommended when there is obvious chromatographic interference.

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Table 8. Quality Control Requirements – Organic Semi-Volatile Analysis by GC/MS in SIM Mode

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Tune Check	Prior to ICAL and prior to each 12-hour period of sample analysis.	Specific ion abundance criteria of DFTPP from method 8270. Tune check can be acquired as a full scan.	Retune instrument and verify.	Flagging is not appropriate.	No samples shall be analyzed without a valid tune. In addition to the full scan tune check, optimization for the analytes of interest is recommended.
Deuterated Monitoring Compounds (DMCs) (surrogates)	All field and QC samples.	PAH analysis: DMCs required for polyaromatic hydrocarbon (PAH) target analytes: fluoranthene-d10 and 2-methylnaphthalene-d10. Minimum RRF for PAH DMCs: 0.40. All DMCs: Requires 50-150% recovery until in-house limits can be established.	Correct problem, and then reprep and reanalyze all samples with failing DMCs if sufficient sample material is available. If obvious chromatographic interference is present, reanalysis may not be necessary, but the client must be notified prior to reporting data and the failures must be discussed in the Case Narrative.	Apply Q-flag to all associated samples and analytes if acceptance criteria are not met and explain in the Case Narrative.	For non-PAH target analytes, other DMCs with similar chemistry must be assigned. Laboratories may use the same extract for full scan and SIM analysis if the SIM-specific DMCs are added prior to extraction.

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TITLE: SEMI-VOLATILE ORGANICS BY GC/MS (EPA METHODS 8270C, 8270D, 625, 625.1 AND SM 6410B), INCLUDING PROVISIONS FOR ANALYSIS IN SIM MODE

Table 8. Quality Control Requirements – Organic Semi-Volatile Analysis by GC/MS in SIM Mode

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Performance Checks	At the beginning of each 12-hour period, prior to analysis of samples.	Degradation \leq 20% for DDT.	Correct problem, then repeat performance checks.	Flagging is not appropriate.	No samples shall be analyzed until the performance checks are within criteria. DDT breakdown and tailing factors are considered overall measures of port inertness and column performance and are required checks for SIM operation. DDT breakdown and tailing factor checks can be acquired as a full scan.
Initial Calibration (ICAL) for all analytes	At instrument set-up, prior to sample analysis.	Each analyte must meet one of the following options: RSD for each analyte \leq 20% [If pentachlorophenol is a target analyte, an RSD of \leq 40% allowed] Or Linear least squares regression for each analyte: $r^2 \geq 0.99$.	Correct problem then repeat ICAL.	Flagging is not appropriate.	Minimum 5 levels required for ICAL with one calibration point at the same concentration as the daily CCV. No samples shall be analyzed until ICAL has passed.

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TITLE: SEMI-VOLATILE ORGANICS BY GC/MS (EPA METHODS 8270C, 8270D, 625, 625.1 AND SM 6410B), INCLUDING PROVISIONS FOR ANALYSIS IN SIM MODE

Table 8. Quality Control Requirements – Organic Semi-Volatile Analysis by GC/MS in SIM Mode

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Retention Time window position establishment	Once per ICAL and at the beginning of the analytical sequence.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA.	NA.	Calculated for each analyte.
Evaluation of Relative Retention Times (RRT)	With each sample.	RRT of each reported analyte within ± 0.06 RRT units of the mean RRT of the calibration standards. RRTs may be updated based on the daily CCV.	Correct problem, then rerun ICAL.	NA.	RRTs shall be compared with the most recently updated RRTs. Characteristic ions must maximize in the same scan or within one scan of each other. After any maintenance is performed which could affect retention times, RRTs may be updated based on the daily CCV.
Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All reported analytes within $\pm 20\%$ of true value. If pentachlorophenol is a target analyte, a %D from the true value of $\pm 50\%$ is allowed.	Correct problem. Rerun ICV. If that fails, repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed until calibration has been verified with a second source.

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TITLE: SEMI-VOLATILE ORGANICS BY GC/MS (EPA METHODS 8270C, 8270D, 625, 625.1 AND SM 6410B), INCLUDING PROVISIONS FOR ANALYSIS IN SIM MODE

Table 8. Quality Control Requirements – Organic Semi-Volatile Analysis by GC/MS in SIM Mode

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Continuing Calibration Verification (CCV)	Daily before sample analysis; after every 12 hours of analysis time; and at the end of the analytical batch run.	Concentration the same as the mid-point calibration standard (or lower). All reported analytes within $\pm 20\%$ of true value. If pentachlorophenol is a target analyte, a %D from true value of $\pm 50\%$ is allowed. All reported analytes within $\pm 50\%$ for end of analytical batch within $\pm 50\%$ for end of analytical batch CCV.	Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails or if two consecutive CCVs cannot be run, perform corrective action(s) until a passing CCV is attained, and then reanalyze all associated samples since last acceptable CCV. Alternatively, perform an ICAL (including appropriate instrument QC) if necessary; then reanalyze all associated samples since the last acceptable CCV	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since last acceptable calibration verification.	Results may not be reported without valid CCVs. Flagging is only appropriate in cases where the samples cannot be reanalyzed. If the specific version of a method requires additional evaluation (e.g., average RFs), these additional requirements must also be met.

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Table 8. Quality Control Requirements – Organic Semi-Volatile Analysis by GC/MS in SIM Mode

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Internal Standards (IS)	Every field sample, Standards, blanks, and QC sample.	Retention time within ± 10 seconds from retention time of the midpoint standard in the ICAL; EICP area within 50% to +100% of ICAL midpoint standard. On days when ICAL is not performed, the initial CCV is used.	Inspect mass spectrometer and GC for malfunctions and correct problem. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	If corrective action fails in field samples, data must be qualified and explained in the Case Narrative. Apply Q-flag to analytes associated with the non-compliant IS. Flagging is not appropriate for failed standards.	Internal Standard is spiked no greater than 0.40 ng/ μ L concentration. According to the EPA Contract Laboratory Program Statement of Work (CLP SOW), this is the concentration of internal standard specified for SIM analysis. The SOW indicates calibration standards range from 0.10 to 1.0 ng/ μ L, so 0.40 ng/ μ L is mid-range. 1, 4-dichlorobenzene-d4 is ignored for SIM
Method Blank (MB)	One per preparation batch, prior to analysis of any field samples.	No analytes detected $> \frac{1}{2}$ LOQ or $> \frac{1}{10}$ th the amount measured in any sample or $\frac{1}{10}$ th the regulatory limit, whichever is greater.	Conduct investigation to determine the source of the contamination and take appropriate corrective actions. Correct problem. If required, reprep and reanalyze MB and all QC samples and field samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated analytical batch.	Laboratories may use the same extract for full scan and SIM analysis provided the applicable DMCs and IS are spiked at the appropriate concentrations. Results may not be reported without a valid Method Blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

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Table 8. Quality Control Requirements – Organic Semi-Volatile Analysis by GC/MS in SIM Mode

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Laboratory Control Sample (LCS)	One per preparation batch.	A laboratory must use Table 3 through Table 6 Limits (8270 SIM) for batch control if project specific limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Correct problem, and then reanalyze the LCS and all samples in the associated analytical batch for failed analytes if sufficient sample material is available.	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply Q-flag to specific analyte(s) in all samples in the associated analytical batch.	Must contain all analytes to be reported. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per preparation batch.	A laboratory must use the QSM Appendix C Limits (8270 SIM) for batch control if project specific limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply the J-flag if acceptance criteria are not met and explain in the Case Narrative.	Must contain all analytes to be reported spiked at concentrations appropriate for SIM analysis. For matrix evaluation only. If MS results are outside the limits, the data shall be evaluated to determine the source(s) of difference (i.e., matrix effect or analytical error).

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Table 8. Quality Control Requirements – Organic Semi-Volatile Analysis by GC/MS in SIM Mode

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparation batch.	A laboratory must use the QSM Appendix C Limits (8270 SIM) for batch control if project specific limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified. MSD or MD: RPD of all analytes $\leq 40\%$ (between MS and MSD or sample and MD).	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply the J-flag if acceptance criteria are not met and explain in the Case Narrative.	The MSD must contain all analytes to be reported spiked at concentrations appropriate for SIM analysis. All data must be evaluated to determine the source of difference. For Sample/MD: RPD criteria only apply to analytes whose concentration in the sample is greater than or equal to the LOQ.
Characteristic ions for MS confirmation	Minimum 3 ions.	The relative intensities of the characteristic ions of target analytes agree within 30% of the relative intensities in the reference spectrum and the relative intensities must be > 0 . Confirmation requires S/N ratio of ≥ 3 for each quant and confirmation ion.	No data can be reported without MS confirmation.	NA.	Need 3 structurally significant ions that are logical fragments – not isotopic clusters. Internal standard and DMC can use fewer than 3 ions.

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Environmental Science Corporation
SOP MINOR REVISION FORM

SOP/DOC#	330345	Current revision date & number:	03/22/18 R26
Procedure/Method : SEMIVOLATILE ORGANICS BY GC/MS (EPA METHODS 8270C, 8270D, 625, 625.1 AND SM 6410B), INCLUDING PROVISIONS FOR ANALYSIS IN SIM MODE			

Date	Requested By	Section	Revision	Reason*	Approvals	
					Supervisor	QA
06/01/18	Shakir Wani	8.1.2	Add Section 8.1.2 - Peak detection thresholds will be set in the data acquisition software based on the lowest MDL. MDLs are posted in LIMS and are readily accessible. Current settings are 4.00 ppb for soils and 0.08 ppb for waters; these are based on our current lowest MDLs, however are subject to change with each MDL update. Global detection settings in data analysis software are mainly used for soil and water analysis for full BNA analysis. Detection thresholds settings for all SIM analysis must always be set to zero due to extreme low level reporting.	Internal Audit	Shakir Wani 6/1/18	Jim Brownfield 6/4/18
6/4/18	Shakir Wani	7.15.1	Add Section 7.15.1 – Spike solutions are verified before use. Guideline for spike verification acceptance criteria: Typical acceptance criterion for spike verification is $\pm 20\%$ ($\pm 15\%$ for ESI). Due to large number of compounds in the BNA list, it is expected that some poor performers will not meet this criteria. Analyst should use experience & judgment in evaluating these based on current instrument performance. Outliers may be discussed with department leads. If the spike fails to meet criteria for target analytes, corrective action is taken; the spike is re-prepped and reanalyzed.	Internal Audit	Shakir Wani 6/4/18	Jim Brownfield 6/4/18

*Comments:

SOP Minor Revision Summary

SOP:			
Title -	pH, MANUAL OR AUTOMATED (EPA METHODS 150.1, 9040B, 9040C, 9045C AND 9045D; SM 4500H+ B) INCLUDING CORROSIVITY FOR SOLIDS AND LIQUIDS USING THESE METHODS		
Number -	340335	Department -	Wet Chemistry
Revision -	16	Rev. Date -	4/9/2018

This Standard Operating Procedure has been amended to include changes required during normal business operations. These changes as defined by SOP 010103 (Document Control and Distribution) are routine modifications that will be incorporated into the SOP upon the next scheduled review.

Rev.	Date	Section	Brief Description
a	8/9/2018	Add Section 8.2.6	Require documentation of sample temperature when pH > 12su.

Standard Operating Procedure

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TITLE: pH, MANUAL OR AUTOMATED (EPA METHODS 150.1, 9040B, 9040C, 9045C AND 9045D; SM 4500H⁺ B) INCLUDING CORROSIVITY FOR SOLIDS AND LIQUIDS USING THESE METHODS

Reviewed by: Mary Garrett, Chris Unterstein, Steve Miller, Johnny Davis, Kandy Kaul



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Department Manager

QA Department

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1.0 SCOPE AND APPLICATION

- 1.1 EPA Methods 9040B and 9040C are used to measure the pH of aqueous wastes and those multiphase wastes with an aqueous phase that is at least 20% of the total volume of waste.
- 1.2 EPA Methods 9045C and 9045D are used to measure the pH of solids, sludge, or non-aqueous liquids. If water is present, its volume must be less than 20% of the total sample volume.
- 1.3 SM4500H⁺ B is used to measure the pH of drinking, surface, and saline waters, acid rain, and domestic and industrial wastes. EPA 150.1 can be used to measure the pH of drinking water. See section 13.5 for limitations.
- 1.4 This method is recommended for utilization only by, or under the supervision of, trained analysts. Each analyst must demonstrate the ability to generate acceptable results with this method.
- 1.5 Corrosivity is designed to identify wastes posing hazards to human health or environment.
 - 1.5.1 The corrosivity characteristic is designed to identify wastes that might pose a hazard to human health or the environment due to their ability to:
 - 1.5.1.1 Mobilize toxic metals if discharged in a landfill environment.
 - 1.5.1.2 Corrode handling storage, transportation, and management equipment.
 - 1.5.1.3 Destroy human or animal tissue in the event of inadvertent contact.
 - 1.5.2 EPA has selected two properties upon which to base the definition of a corrosive waste. These properties are pH and the corrosivity toward Type SAE 1020 steel^{14.6}.

2.0 METHOD SUMMARY AND DEFINITIONS

- 2.1 The pH of field samples is determined electrometrically using a combination electrode that incorporates both measuring and reference electrode functions. An automatic temperature compensation probe is also utilized to compensate for temperature variations during analysis. Both electrodes, in combination with a pH meter that can

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interpret the electrode responses, are calibrated using a series of standards having known pH values that bracket the typical range of field samples. Aqueous samples are measured directly. Non-aqueous samples require extraction with water prior to analysis. Corrosivity determination is made using the pH value obtained. A pH value of <2, or ≥ 12.5 is determined to be corrosive^{14,6}. Values >2 and <12.5 are determined to be non-corrosive.

- 2.2 pH - the inverse logarithm (base 10) of the hydrogen ion activity (in moles/L) in solution.
- 2.3 Corrosivity – The ability of a substance to destroy and/or damage other substances with which it comes into contact. Routinely, common corrosives are either strong acids/bases or concentrated solutions of certain weak acids/bases. They can exist as any state of matter, including liquids, solids, gases, mists or vapors.
- 2.4 See the current Quality Assurance Manual for other definitions associated with terms found in this document.

3.0 HEALTH AND SAFETY

The toxicity or carcinogenicity of each reagent used in the laboratory has not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds should be as low as reasonably achievable. A reference file of safety data sheets (SDSs) is made available on ESC's intranet to all personnel. Use hazardous reagents in a fume hood whenever possible and if eye or skin contact occurs, flush with large volumes of water. Always wear safety glasses or a shield for eye protection, protective clothing and observe proper mixing protocols.

4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

- 4.1 All samples must have been collected using a sampling plan that addresses the considerations of this method.
- 4.2 Aqueous Samples should be analyzed as soon as possible, preferably in the field at time of sampling. The Federal Register citation 40 CFR 136.3 (Table II) specifies a maximum holding time of 15 minutes. Samples that are shipped to the laboratory are reported with a T8 qualifier. Non-aqueous samples should be analyzed as soon as the preparation is completed. The results should also be reported with a T8 qualifier.
- 4.3 Some samples are subject to changes in pH when exposed to the atmosphere. Sample containers should be filled completely and kept sealed prior to analysis.
- 4.4 Store samples during shipping and prior to analysis at $4^{\circ} \pm 2^{\circ}\text{C}$. Allow samples to reach ambient temperature immediately prior to analysis.

5.0 INTERFERENCES

- 5.1 Oily material or particulate matter may coat electrodes and impair responses. Remove these interferents by gently washing with detergent, rinsing with DI water, and wiping with a soft cloth.

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- 5.2 Electrode response can differ at varying temperatures. Using a temperature-compensating electrode and a pH meter with temperature compensation capability controls fluctuations of this kind.
 - 5.3 In some samples, pH change is inherent at different temperatures. This error is sample dependant and cannot be controlled. Both pH and temperature, at the time of analysis, should be noted on the report.
 - 5.4 Acid rain samples must not be analyzed using a magnetic stirrer because pH values will change if the sample is not in equilibrium with the air. Instead, swirl the sample gently.
 - 5.5 Sodium concentration in samples at a pH value greater than 10 can cause a bias in pH measurements. If necessary, a "low sodium error" electrode can be purchased to reduce the error of measurements in this pH range.
 - 5.6 Scratches, deterioration, and accumulation of debris on the glass surface are the most common causes of electrode failure. Use, store and maintain electrodes according to manufacturer's instructions.
 - 5.7 Organic solvents are damaging to the epoxy composition of the electrodes. If a sample is an organic solvent, do not allow it to contact the electrodes. Perform an estimated pH, using a wide-range (1-14) pH strip. See section 11.4 for more details.
 - 5.7.1 Test strips must never be inserted directly into a sample undergoing preparation prior to analysis. Instead, remove sample material from the original container with an appropriate non-contaminated implement (e.g., disposable pipette, glass rod) and place on the test strip.
- 6.0 EQUIPMENT AND SUPPLIES
- 6.1 Instruments:
 - 6.1.1 Manual: pH meter with temperature compensation capability (Thermo Scientific Orion Versa Star or equivalent).
 - Combination pH electrode (Orion 8157BNUMD pH/ATC Triode or equivalent)
 - Temperature-compensating electrode (Thermo Scientific ATC or equivalent)
 - Magnetic stir plate (Thermo SP-131015Q or equivalent)
 - Magnetic stir bars (Fisher 14-511-58 or equivalent)
 - 6.2 Vortex Mixer (Vortex Genie 2 G560 or equivalent)
 - 6.3 Stirring Hot Plate (Thermo SP-131015Q or equivalent)
 - 6.4 Centrifuge tubes (Corning 430829 or equivalent)

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6.5 Top loading analytical balance capable of weighing to 0.01g (Ohaus Scout Pro or equivalent)

6.6 Disposable Beakers (Fisher 01-291-10 or equivalent)

6.7 Centrifuge (Thermo Fisher Sorvall ST40 or equivalent)

7.0 REAGENTS AND STANDARDS

7.1 All reagents and standards must be recorded in the appropriate preparation log and assigned a unique number. See SOP #030230, *Standards Logger – Tree Operation*. Additional information regarding reagent preparation can be found in the Standards Logger (Tree) digital archive system. All spiking solutions and surrogate standard solutions should be replaced at least every six months or sooner if a problem is detected unless otherwise noted.

7.2 pH 1 buffer (Ricca 1489-32 or equivalent)

7.3 pH 4 buffer (Inorganic Ventures PHRED- 4 or equivalent)

7.4 pH 7 buffer (Inorganic Ventures PHYellow-7 or equivalent). Used for ICV and CCV, but must be from a different vendor or lot.

7.5 pH 10 buffer (Inorganic Ventures PHBLUE-10 or equivalent). Used for LCS/LCSD.

7.6 pH 13 buffer (Ricca 1625-16 or equivalent)

NOTE: Buffers can be used only once. Use the smallest possible aliquots of each buffer that provides sufficient probe immersion and discard remainders of the aliquots when completed.

8.0 PROCEDURE

8.1 Preparation of non-aqueous samples.

8.1.1 Tare a disposable beaker on the top-loader balance.

8.1.2 Weigh up to 20 grams of sample into a centrifuge tube.

8.1.3 Add an equal weight of deionized water.

8.1.3.1 If the sample is hygroscopic, further dilution is allowed.

8.1.4 Mix sample and water by vortex for 10 seconds.

8.1.5 Allow solids to settle for one hour to reach equilibrium or centrifuge for quicker separation.

8.1.6 Decant the aqueous layer and proceed to 8.4 or 8.5 as appropriate.

8.2 Manual Sample Analysis

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8.2.1 Place a stir bar into a disposable beaker and add sufficient sample, buffer, or water extract to cover the sensing unit on the probe and to allow for adequate clearance between the probe and the rotating stir bar.

8.2.1.1 For preparation of the LCS/LCSD pair, use aliquots of a second source pH buffer or certified reference material.

8.2.2 Place the sample on the stir plate and begin stirring gently in order to generate as little agitation as possible at the air/sample interface. See 5.4 for special handling of acid rain samples. See 11.4 for special handling of organic solvent samples.

8.2.3 Immerse the electrodes in the sample.

8.2.4 When the reading is stable (less than 0.1 pH unit drift), the "Stable" prompt will appear on the meter display.

8.2.5 Record the result, rinse the electrodes with deionized water, and continue analyzing the remainder of samples and buffers in the Workgroup.

9.0 DATA ANALYSIS AND CALCULATIONS

9.1 Due to the interrelationship between temperature and pH, both values should be noted on the test report.

9.2 For Corrosivity: Report results as either corrosive ($\text{pH} \leq 2.0$ or ≥ 12.5) or not corrosive ($\text{pH} > 2.0 - < 12.5$)^{14.6}. Report actual pH values for QC samples (blank, LCS/LCSD) to allow the review for acceptability.

9.3 Calculation of the slope of the calibrated pH meter when pH readings can be provided in millivolts (mV) and slope is not automatically provided by the meter^{14.7}:

$$\text{Slope (as \%)} = [\text{mV at pH 7} - \text{mV at pH 4}] \times 100/177$$

9.4 See the current Quality Assurance Manual for other equations associated with common calculations.

10.0 QUALITY CONTROL AND METHOD PERFORMANCE

10.1 All analysts must meet the qualifications specified in SOP #030205, *Technical Training and Personnel Qualifications* before approval to perform this method. Analysts must complete an initial demonstration of proficiency before being approved to perform this method. Continuing proficiency must be demonstrated using proficiency testing and/or laboratory control sample analysis. Method performance is assessed per analyst. Updated method performance records are filed and stored in a central location within the department.

10.2 Use the designated Run log to record batch order and standards/reagents used during analysis. See SOP #030201, *Data Handling and Reporting*.

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- 10.3 Batches are defined as sets of 1 - 20 samples. Batch analysis must include the following: 1 Initial Calibration Verification (ICV), 1 Laboratory Control Sample/Laboratory Control Sample Duplicate pair (LCS/LCSD), 1 Sample Duplicate for every 10 samples, 1 Continuing Calibration Verification (CCV) every 10 analyses and at the end of the sequence.
- 10.4 Initial Calibration Verification (ICV)/Continuing Calibration Verification (CCV): A CCV standard must be analyzed at a frequency of every ten injections (including LCS, LCSD, MS, MSD, duplicates, and blanks in the count) during an analytical sequence. The CCV standard must also be analyzed after the last analytical sample. The measured concentration must be within 0.1su of the true value.
- 10.5 Laboratory Control Sample and Duplicate (LCS/LCSD): One LCS/ LCSD pair must be analyzed with every batch of 20 samples. The measured concentration of the soil LCS/LCSD must fall within the acceptable range provided by the standard manufacturer. The difference between the LCS/LCSD must not be more than 0.1su for aqueous matrices and $\leq 1\%$ RPD for a solid matrix.
- 10.6 Sample Duplicate: One sample duplicate must be analyzed with every set of 10 samples. A sample duplicate is an additional aliquot of the original sample that has been taken through the entire procedure. The results must be within 0.1su of each other for water matrices and the calculated RPD should be $\leq 1\%$ for solid samples.
- 10.7 Calibration Curve: A calibration curve must be performed daily. The slope must be 95-105%^{14.7}.
- 10.8 The requirements of ESC SOP #030229, Verification of Automatic Temperature Compensation Probes on Laboratory pH Meters, must be performed quarterly for ATC-equipped pH meters. Results of this verification must be documented on controlled document GEN-01 and archived at H:\QAQC\Public\ATC verifications.
- 11.0 DATA VALIDATION AND CORRECTIVE ACTION
- 11.1 All data must undergo a primary review by the analyst. The analyst should review sample results and make sure that they have been confirmed. All calculations must be checked. Data must be checked for the presence or absence of appropriate flags. Comments should be noted when data is flagged.
- 11.2 All data must then undergo a second analyst review. This review must be performed according to SOP #030201, *Data Handling and Reporting*.
- 11.3 Data that does not meet acceptable QC criteria may be acceptable for use in certain cases.
- 11.3.1 Due to the "As Soon As Possible" method specified holding time, results for samples analyzed in the laboratory should be reported with a T8 (Sample received out of hold time) qualifier.

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TITLE: pH, MANUAL OR AUTOMATED (EPA METHODS 150.1, 9040B, 9040C, 9045C AND 9045D; SM 4500H⁺ B) INCLUDING CORROSIVITY FOR SOLIDS AND LIQUIDS USING THESE METHODS

11.3.2 Sample duplicate – If the difference between the sample and sample duplicate is greater than ± 0.1 su, the sample should be reported with a J3 qualifier for precision beyond the range for the method.

11.4 If a sample is an organic solvent that has been analyzed using wide range pH paper. Record results with a T6 qualifier (method used is an alternative to approved methodology). Be sure to include an explanation on the bench sheet.

11.5 LCS/LCSD: If the LCS/LCSD are not within acceptance criteria, the sample(s) may be re-analyzed once. If the failure of the LCS/LCSD persists, the instrument must be re-calibrated and any needed maintenance performed. All samples analyzed in conjunction with the failure must be re-analyzed.

11.6 ICV/CCV: If the ICV/CCV are not within acceptance criteria and analysis of a second consecutive (immediate) calibration verification fails to produce results within acceptance criteria, corrective actions shall be performed. The laboratory shall demonstrate acceptable performance after corrective action with two consecutive calibration verifications, or a new initial instrument calibration shall be performed.

11.7 Sample Duplicate: If the results of the sample duplicates exceed the acceptance criteria, prepare another aliquot of the sample and analyze to confirm the initial readings. If the results do not confirm, flag the results with a J3 qualifier.

11.8 Calibration Curve: If the calibration curve does not meet the acceptance criteria, it must be re-analyzed. A calibration curve must be successfully run before sample analysis can be performed.

STATE NOTE: Drinking water samples analyzed using this procedure for compliance cannot be qualified.

12.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

12.1 The EPA requires that laboratory waste management practice to be conducted consistent with all applicable federal and state laws and regulations. Excess reagents, samples and method process wastes must be characterized and disposed of in an acceptable manner. See *ESC Waste Management Plan*.

12.2 See SOP #030302, *Environmental Sustainability & Pollution Prevention*.

13.0 METHOD MODIFICATIONS/CLARIFICATIONS

13.1 Replicate analyses of each sample are not performed per method suggestions due to the added cost associated with the additional time and the revenue generated by this analysis^{14.8}. Replicate analyses, on samples at a frequency greater than standard QC requirements, may be performed by the analyst if there is reason to believe that initial analyses may vary throughout the sample.

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- 13.2 Modifications to this method are noted in the body of the text as state notes. Compliance analyses performed in conjunction with specific state requirements must be performed as noted within the specific state(s) note listed.
- 13.3 Superscripts are provided where necessary to indicate the reference in Section 14.0 where the requirement/information can be found. Subscripts noted identify the most frequent/restrictive cases, but requirements may also be included at different frequencies/conditions in other references noted in section 14.0.
- 13.4 EPA Method 150.1 was removed from use in the 2007 EPA MUR for CWA compliance and replaced by SM 4500H⁺ B. EPA Method 150.1 remains an approved analysis under the SDWA. This procedure can also be utilized for other regulatory programs or where specific NPDES permits still identify this method as required for analyses.

14.0 REFERENCES

- 14.1 *pH Value, Electrometric Method*, Standard Methods 4500H⁺ B, 20th Edition.
- 14.2 *pH Electrometric Measurement*, SW-846 9040C, Revision 3, November 2004.
- 14.3 *Soil and Waste pH*, SW-846 Method 9045C, Revision 3, January 1995.
- 14.4 *Soil and Waste pH*, SW-846 Method 9045D, Revision 4, November 2004.
- 14.5 Accumet AB15 Plus instrument manual.
- 14.6 *pH Value, Electrometric Method*, Standard Methods 4500H⁺ B-2000.
- 14.7 *pH Value, Electrometric Method*, Standard Methods 4500H⁺ B-2011.
- 14.8 Code of Federal Regulations, Title 40, Part 261.22.
- 14.9 *EPA Manual for the Certification of Laboratories Analyzing Drinking Water*, EPA 815-R-05-004, 5th Edition, January 2005.
- 14.10 *pH (Electrometric)*, EPA Method 150.1, Approved for NPDES, Editorial Revision 1978, 1982.

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TITLE: pH, MANUAL OR AUTOMATED (EPA METHODS 150.1, 9040B, 9040C, 9045C AND 9045D; SM 4500H⁺ B) INCLUDING CORROSIVITY FOR SOLIDS AND LIQUIDS USING THESE METHODS

Attachment I: Revision History

Current Version:

Version	Date	Description of Revisions
16	4/9/2018	Technical and quality review and revision. Changed ESC logo. Revised sections 3.1, 4.2, 4.4, 6.1.1, 6.2, 6.3, 6.4, 6.5, 6.6, 7.1, 7.2, 7.3, 7.4, 7.5, 7.6, 8.3.2, 8.3.4, 10.3, 13.4, 14.1, 14.3, 14.4, 14.6, 14.7, 14.8 and 14.9. Deleted sections 6.1.2, 7.7, 8.5 (and subsections). Deleted sections 8.1 and 8.2 (and subsections) and renumbered as necessary. Deleted section 8.4.1.1 and renumbered next section. Added section 6.7. Revised Attachment II sections 3c and 3e.

Superseded Versions:

This document supersedes the following:

Version	Date	Description of Revisions
0	11/13/92	Origination
1	8/3/00	
2	8/22/00	
3	1/16/02	
4	7/23/03	
5	1/27/04	
6	1/19/06	
7	9/7/07	Complete Revision for Format, Technical Content and Clarity.
8	5/21/09	Annual Technical and Quality Review. Removed EPA Method 150.1 (MUR); update EPA methods and references; Revised sections 10.4 – 10.8 & 11.0
9	10/01/10	Annual Technical and Quality Review. Added sections 2.8 and 10.4; Revised sections 4.2, 5.7, 7.1, 8.1.3, 8.1.8, 8.2.6, 10.6, 10.7, and 12.1.
10	11/3/11	Annual Technical and Quality Review. Added sections 13.3 and Attachment II; Revised sections 2.8 and 12.1.
11	2/28/12	Annual Technical and Quality Review. Added Attachment III; Revised sections 7.4, 8.1 and Attachment II; Added sections 7.7.
12	5/24/13	Annual Technical and Quality Review. Revised Attachment II and sections 4.2, 6.1, 8.4, and 10.5; Added sections 8.2, 8.5, 9.2, 10.7, 11.8, 14.4 and 14.5; Removed sections 2.4, 6.3 through 6.5 and 10.5. Consolidated 340335 and 340335A into single procedure
13	8/8/14	Annual Technical and Quality Review. Revised Attachment II & III and sections 1.3, 2.1, 6.1, 8.1, 8.3.2, 8.3.5, 8.3.6 and 10.5; Added note in section 7.0 and added sections 1.5, 2.3, 9.3, 9.4, 13.4, 13.5 and 14.6 through 14.8. Consolidated 340335 and 340306 into single procedure

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TITLE: pH, MANUAL OR AUTOMATED (EPA METHODS 150.1, 9040B, 9040C, 9045C AND 9045D; SM 4500H⁺ B) INCLUDING CORROSIVITY FOR SOLIDS AND LIQUIDS USING THESE METHODS

Version	Date	Description of Revisions
14	10/20/2015	Technical and Quality review and revision. Revised header and signature block. Revised Title and Sections 1.1, 1.2, 5.7.1, 12.2, 13.5, and 14.2.
15	11/4/2016	Technical and quality review and update. Update per SC DHEC correspondence of 10/19/2016. Header and signature bar re-formatting. Revised Sections 2.4, 7.1, 8.3.4, 10.4, 11.6, 14.1, 14.2, 14.6, 14.7, and 14.10. Deleted Sections 2.5 through 2.8, 9.2, and 13.2. Added Sections 6.3, 9.4, 10.8, 14.3, and 14.4.

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Attachment II: Analytical Tech Notes

1. Calibrate (Attachment III)
2. Prepare soil samples (8.3)
3. Run Samples and QC
 - a. ICV (6.9-7.1)
 - b. LCS/LCSD (± 0.1 su)
 - c. 7 samples plus 1 duplicate (± 0.1 su)
 - d. CCV (6.9-7.1)
 - e. 9 samples plus 1 duplicate (± 0.1 su)
 - f. CCV (6.9-7.1)
4. Do not put probes into organic solvent (5.7 and 11.4)
5. For Corrosivity Determination: If pH is ≤ 2 or ≥ 12.5 , report as corrosive. If pH is > 2 and < 12.5 , report as noncorrosive.

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TITLE: pH, MANUAL OR AUTOMATED (EPA METHODS 150.1, 9040B, 9040C, 9045C AND 9045D; SM 4500H⁺ B) INCLUDING CORROSIVITY FOR SOLIDS AND LIQUIDS USING THESE METHODS

Attachment III: Manual Technical Notes:

1. Calibration – pH calibration procedures vary from meter to meter. The following instructions are used with the Thermo Scientific Orion Versa Star pH meter and are provided as general direction. Use of other pH meters in conjunction with this procedure may cause slight variation from the process that follows. Refer to the manufacturer's instructions on calibration and operation of the pH meter in use.
 - 1.1 Press Cal (F1).
 - 1.2 Select desired channel (1 or 2)
 - 1.3 Pour pH 4 buffer into a disposable beaker with a stir bar and place the beaker on a stir plate. Use sufficient volume of solution to cover the sensing unit on the probe and to allow for adequate clearance between the probe and the rotating stir bar. Rinse the electrodes and place them in the buffer solution. When the display prompts that the reading is stable, press [accept] (F2) once. Record the mV and temperature value that is then displayed in the pH calibration log and then proceed with other buffers by selecting [next].
 - 1.4 Place pH 7 buffer on stir plate and repeat instructions from 1.3.
 - 1.5 Place pH 10 buffer on stir plate and repeat instructions from 1.3.
 - 1.6 Place the pH 13 buffer on the stir plate and repeat instructions from 1.3.
 - 1.7 Place the pH 1 buffer on the stir plate and repeat instructions from 1.3.
 - 1.8 When finished select [CAL DONE] button. Record slope in the pH calibration log. Then select [Measure] to begin analyzing samples.
 - 1.9 The slope must be between 95% and 105% for analysis to proceed. If the slope is not within this range, repeat calibration with fresh buffers.
 - 1.10 Buffers can be used only once. Use the smallest possible aliquots and discard.

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Environmental Science Corporation
SOP MINOR REVISION FORM

SOP/DOC# <u>340335</u>	Current revision date & number: <u>4/9/2018 R16</u>
Procedure/Method : pH, MANUAL OR AUTOMATED (EPA METHODS 150.1, 9040B, 9040C, 9045C AND 9045D; SM 4500H+ B) INCLUDING CORROSIVITY FOR SOLIDS AND LIQUIDS USING THESE METHODS	

Date	Requested By	Section	Revision	Reason*	Approvals	
					Supervisor	QA
8/9/18	SCM	8.2.6	Add new Section 8.2.6: "The temperature of the sample must be recorded if the pH is >12su. Additionally, if the temperature is not within 24-26°C, then the sample must be re-prepped."	CAR3174		

*Comments:

Standard Operating Procedure

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TITLE: MERCURY IN AQUEOUS/LIQUID SAMPLES (COLD-VAPOR TECHNIQUE)
(EPA METHODS 7470A AND 245.1)

Reviewed by: Jeremy Gupton, Jim Brownfield

Jeremy Gupton

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QA Department

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STATE NOTE: For samples analyzed in conjunction with the Ohio Voluntary Action Program (VAP), please utilize the current revision of SOP #340384A_OH.

1.0 SCOPE AND APPLICATION

- 1.1 This cold vapor atomic absorption procedure is for determining the concentration of mercury in aqueous wastes, ground waters, wastewaters, drinking waters, saline waters, domestic and industrial wastes. The laboratory routine reporting limit is 0.2µg/L; however reporting limits are subject to change to better meet client/project/regulatory needs or to improve laboratory method performance.
- 1.2 An MDL study must be completed at least annually or more frequently if major instrumentation changes occur. Method Detection Limits (MDLs) are performed based on Pace National SOP #030206. Updated MDL records are filed and stored in a central location within the department.
 - 1.2.1 Limit of Detection (LOD) and Limit of Quantitation (LOQ) studies are completed at the frequency required by the TNI standard per the procedure identified in the Pace National SOP #030206, *Method Detection Limits (MDL), Limits of Detection (LOD) and Limits of Quantitation (LOQ)*. Should the procedure be utilized for a U.S. Department of Defense (DoD) project; then the frequency of these studies must meet the requirements of the current DoD QSM (see Attachment II).

2.0 METHOD SUMMARY AND DEFINITIONS

- 2.1 The goals of the mercury analytical system are to convert all Hg species to Hg⁺⁺ ions and break down all organic molecules in each sample. Organic molecules must be broken down to prevent their interference with mercury sample analysis. By adding an appropriate sequence of reagents to each sample then heating in a hot block for 2 hours, all organic molecules are broken down and all Hg species are completely oxidized to Hg⁺⁺ ions, used for cold vapor atomic absorption analysis.
- 2.2 A digested liquid sample with mercury in the divalent form enters the system and is mixed with a reducing agent (SnCl₂) to form elemental mercury vapor.
- 2.3 The dry vapor then enters the optical cell that has been optimized for fast response time and sensitivity. A mercury light source emits a stable source of light at 253.7nm wavelength. The intensity of the light source passing through the mercury cold vapor cell is measured using a solid-state detector with a wide dynamic range. To measure the mercury concentration, the

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TITLE: MERCURY IN SOLID WASTE (COLD-VAPOR TECHNIQUE)
(EPA METHODS 7471A & 7471B)

resulting sample absorbance signal is compared to the absorbance of the pure carrier gas flowing through the optical path under identical conditions.

- 2.4 Instrument Detection Limit (IDL) - The smallest signal above background noise that an instrument can reliably detect.
- 2.5 Linear Dynamic Range (LDR) – The concentration range where absorbance and concentration remain directly proportional to each other. A wide linear dynamic range permits the analysis of a wide range of sample concentrations (optical densities) and reduces sample preparation (dilution) requirements.
- 2.6 Serial Dilution (SD) – A subsequent dilution of a high concentration field sample that should agree within acceptance criteria of the original undiluted analysis. This is generally used as a test for matrix interferences or matrix effects.
- 2.7 Post Spike (PS) – A standard prepared from a previously analyzed spiked sample digestate that yielded reduced recovery for the target analyte due to a suspected matrix interferent.
- 2.8 See the current Quality Assurance Manual for other definitions associated with terms found in this document.

3.0 HEALTH AND SAFETY

- 3.1 The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds should be as low as reasonably achievable. A reference file of safety data sheets (SDSs) are made available on Pace National's intranet to all personnel. Use hazardous reagents in a fume hood whenever possible and if eye or skin contact occurs, flush with large volumes of water. Always wear safety glasses or a shield for eye protection, protective clothing and observe proper mixing protocols.
- 3.2 Use of this procedure requires handling acid preserved samples, standards and concentrated acids. Always wear safety glasses, protective gloves and laboratory coat. Also, fume hoods must be used for both the automated and manual preparation of mercury samples. Mercury vapor is toxic: precaution must be taken to avoid inhalation.
- 3.3 Before preparing any samples, analyst must be familiar with all safety techniques involving strong acids and any of the other chemicals or reagents used. Refer to the appropriate Material Safety Data Sheets (MSDS) for all pertinent information.
- 3.4 Before starting a sample preparation run, check to ensure that the vent hood connection is drawing at the required flow rate. The exhaust may contain minute amounts of mercury or other potentially dangerous chemicals.
- 3.5 Many mercury-containing compounds are highly toxic, if swallowed, inhaled or absorbed through the skin. Extreme care must be exercised in the handling of concentrated mercury reagents. These reagents should only be handled by analysts knowledgeable of their risks and of safe handling procedures.

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TITLE: MERCURY IN SOLID WASTE (COLD-VAPOR TECHNIQUE)
(EPA METHODS 7471A & 7471B)

4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

- 4.1 All samples must have been collected using a sampling plan that addresses the considerations of this method.
- 4.2 Soil samples are stored at $4^{\circ} \pm 2^{\circ}\text{C}$.
- 4.3 Holding times for field samples are 28 days from sample collection to preparation.
- 4.4 All glassware must be washed with a laboratory detergent (e.g., Alconox), tap water rinsed, nitric acid rinsed and then rinsed with DI water. See Pace National SOP #030701, *Glassware Cleaning*.
- 4.5 Samples can be collected in either plastic or glass containers.
- 4.6 Samples submitted for analysis that do not meet the requirements contained within this section must be addressed before performing the logging process within the laboratory. In some cases, exceeding the appropriate preservation and storage criteria can cause significant bias in the resulting data. Clients may need to resubmit samples where the conditions during shipment cause uncertainty regarding sample integrity. If samples do not meet the requirements for preservation, sampling, shipment and storage and the client approves the completion of the analytical process, sample results can be qualified per the Pace National SOP #030201, *Data Handling and Reporting*.

5.0 INTERFERENCES

- 5.1 Potassium permanganate is added to eliminate possible interference from sulfide. The KMnO_4 oxidizes the sulfides.
- 5.2 It has been reported that copper could cause interference though the laboratory has not detected this effect.
- 5.3 Chlorides can also cause interferences; so additional potassium permanganate is added to oxidize chloride to free chlorine. Free chlorine absorbs radiation at 254nm, so an excess of hydroxylamine sulfate is added to remove free chlorine.
- 5.4 Some volatile organic materials that absorb at 253.7nm could cause interference. If interference is a problem during a sample run, a preliminary run without reagents determine if this type of interference is present.

6.0 EQUIPMENT AND SUPPLIES

- 6.1 Perkin Elmer FIMS400 Mercury Analyzer, Leeman Hydra II or equivalent.
- 6.2 Polypropylene culture test tubes, 15mL and 50mL capacity.
- 6.3 10mL graduated glass disposable serological pipettes. (Pyrex or equivalent)
- 6.4 Class A 100mL volumetric flasks (Pyrex or equivalent).
- 6.5 Class A volumetric pipettes, 1mL and 5mL.

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TITLE: MERCURY IN SOLID WASTE (COLD-VAPOR TECHNIQUE)
(EPA METHODS 7471A & 7471B)

- 6.6 8 cup capacity standards rack
- 6.7 98 cup capacity sample rack
- 6.8 50mL disposable polypropylene sample containers
- 6.9 Mettler analytical balance or equivalent
- 6.10 Bulk Liquid Compressed Argon gas, pre-purified, used as instrument carrier gas.
- 6.11 Precision Hot Block (Brand name - MOD Blocks or equivalent).
- 6.12 Computer software used: (AA WINLAB, version 2.50 or equivalent).
- 6.13 Sample introduction system (auto sampler) - (Model, instrument #1 & #3 is a Perkin Elmer AS91, and for instrument #2 a Perkin Elmer AS90, or equivalent).
- 6.14 Peristaltic pump, to pump reagents and samples through the detector.
- 6.15 Computer used is a COMPAQ or equivalent.
- 6.16 Adjustable-volume pipetter (Eppendorf, or equivalent)

7.0 REAGENTS AND STANDARDS

- 7.1 All reagents and standards must be recorded in the appropriate preparation log and assigned a unique number. See SOP #030230, *Standards Logger – Tree Operation*. Additional information regarding reagent preparation can be found in the Standards Logger (Tree) digital archive system. All spiking solutions and surrogate standard solutions should be replaced at least every 6 months or sooner if a problem is detected, unless otherwise noted.
- 7.2 ASTM Type 1 water - Water must be free of mercury or anything that may interfere with the mercury analysis.
- 7.3 Stock standards expiration date is one year from the date received or the expiration date assigned by the manufacturer whichever is sooner.
- 7.4 Concentrated hydrochloric acid (HCl) – Concentrated, trace metal grade (OMNI TRACE, catalog# HX0607-2 or equivalent). NOTE: manufacturer shelf life/expiration date.
- 7.5 Concentrated nitric acid (HNO₃) – Concentrated, trace metal grade (VWR EM-Nx0407-2 or equivalent). NOTE: manufacturer shelf life/expiration date.
- 7.6 5% Potassium Permanganate (KMnO₄) solution – JT Baker 3227-01 or equivalent. Weigh 500 ± 0.01g of potassium permanganate per 10L reagent water.
- 7.7 Sodium Chloride-Hydroxylamine Sulfate – prepared by placing 240 ± 0.01g of Hydroxylamine sulfate, JT Baker N646-07, or equivalent, and 240 ± 0.01g of sodium chloride (EM Science SX0420-1 or equivalent) into 2L reagent water.

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TITLE: MERCURY IN SOLID WASTE (COLD-VAPOR TECHNIQUE)
(EPA METHODS 7471A & 7471B)

- 7.8 1.1% Stannous Chloride – JT Baker 3980-11 or equivalent. Weigh 22.0 ± 0.01 g SnCl₂ and 60mL conc. HCl (3%) in a 2L volumetric flask and dilute to 2L with DI water. See section 13.2. **Made fresh DAILY. The reagents used to make this are recorded separately on the Prep sheets.**
- 7.9 10% Stannous Chloride – JT Baker 3980-11 or equivalent. Weigh 100.0 g SnCl₂ and 100mL conc. HCl (3%) in a 1L volumetric flask and dilute to 1L with DI water. See section 13.2. **Made fresh DAILY. The reagents used to make this are recorded separately on the Prep sheets.**
- 7.10 Aqua Regia – Prepare immediately before use, by carefully adding three volumes of concentrated HCl to one volume of concentrated HNO₃. **The reagents used to make this are recorded separately on the Prep sheets.**
- 7.11 Primary Stock Mercury Solution: 1000ppm, Ultra Scientific, Cat # ICP-080, Inorganic Ventures, cat# AAHG1-5 or equivalent. This primary stock solution is used for calibration standards, continuing calibration verification (CCV), Low Level Calibration Verification (ICVLL/CCVLL), Matrix Spike (MS), Matrix Spike Duplicate (MSD), (see section 13.4).
- 7.12 Secondary Source Stock Mercury Solution: Must be a stock solution equivalent to the Primary stock solution but MUST be from a different vendor. This stock solution is used for initial calibration verification (ICV), Laboratory control sample (LCS), and Laboratory control sample duplicate (LCSD) (see section 13.4).
- 7.13 1PPM Intermediary Standards: Make up this concentration from both sources: the Primary 1000PPM source and the Secondary 1000PPM source. For the 1PPM Primary intermediate solution, spike 0.5mL of the 1000PPM stock solution into 50mL Class A volumetric flask with approximately 10mL of DI water and 5mL of concentrated nitric acid and bring up to volume with DI water. For the 1PPM Secondary Intermediate solution, spike 0.5mL of the 1000PPM secondary stock solution into 50mL Class A volumetric flask with approximately 10mL of DI water and 5mL of concentrated nitric acid and bring up to volume with DI water. These intermediate standards are prepared fresh weekly. The 1PPM secondary stock solution is used for the ICV, LCS, LCSD. See section 13.4.

8.0 PROCEDURE

8.1 Digestion

- 8.1.1 For the calibration curve and instrument QC (ICVs and CCVs), using a continuously adjustable-volume pipetter, add 5mL of DI water to the digestion tubes and spike each level with the appropriate amount of intermediate standard, and continue prepping per method. Typical spiking levels are as follows; however see Section 13.4 for more information:

- 0.0ppb - No spike
- 0.2ppb - 6uL of 1PPM Primary Intermediate
- 0.4ppb - 12uL of 1PPM Primary Intermediate
- 1.0ppb - 30uL of 1PPM Primary Intermediate
- 2.0ppb - 60uL of 1PPM Primary Intermediate

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TITLE: MERCURY IN SOLID WASTE (COLD-VAPOR TECHNIQUE)
(EPA METHODS 7471A & 7471B)

- 5.0ppb - 150uL of 1PPM Primary Intermediate
 - 10.0ppb - 300uL of 1PPM Primary Intermediate
 - ICV-3.0ppb - 90uL of 1PPM Secondary Intermediate
 - CCV-2.5ppb - 75uL of 1PPM Primary Intermediate
 - ICVLL/CCVLL --0.2ppb --6uL of 1PPM Primary Intermediate
- 8.1.2 For the method blank, weigh out 0.3 ± 0.05 g of Chemware Teflon chips into the digestion tube. Using an adjustable volume pipetter, add 2.5mL of DI water to the digestion tube. Aliquots of this blank will serve as the method blank, the ICB, and the CCB during analysis.
- 8.1.3 For the LCS/LCSD, weigh out 0.3g of Chemware Teflon chips into the digestion tube, spike 90uL of 1PPM Secondary Intermediate standard into the digestive tube, then using adjustable volume pipetter add 2.5mL of DI water to the digestion tube. This weight may vary, depending on the concentration of the Hg standard from one lot# to the next.
- 8.1.4 For the samples, weigh out 0.3 ± 0.05 g of each field sample and record the weight. Prior to weighing, mix the sample to ensure that it is adequately homogenized. If there are large solid artifacts such as stones and concrete, these can be broken up with a mortar and pestle before the sample is weighed. Note the final weight in the prep log. With the adjustable volume pipetter, add 2.5mL of DI water into each of the digestive tubes. See section 13.5.
- 8.1.5 For the MS/MSD, weigh out 0.3 ± 0.05 g of a specified client sample, or a randomly selected client sample if none are specified, into the digestive tube. Spike 90uL of 1PPM Primary Intermediate standard into the digestive tube then using adjustable volume pipetter, add 2.5mL of DI water to the digestive tube.
- 8.1.6 Add 2.5mL of Aqua Regia to all samples, standards and QC samples and cap tightly.
- 8.1.7 Heat for two minutes in a hot block at $95 \pm 3^{\circ}\text{C}$.
- 8.1.8 Cool for a minimum of five minutes. Add 25mL of DI water, swirl to mix, then add 7.5mL of 5% potassium permanganate solution. Swirl to mix. Allow purple color to persist for at least fifteen minutes. If the purple color persists move to step 8.1.9. If the purple color does not persist, add additional amounts of potassium permanganate crystals until the sample stays purple. Add the same amount of additional crystals to the LCS/LCSD and blanks as well.
- 8.1.9 Heat in a hot block for at least thirty minutes at $95 \pm 3^{\circ}\text{C}$. Record the time-in and temperature-in of the hot block on the Hg bench sheet.
- 8.1.10 After thirty minutes of digestion, take the sample out of the hot block and allow them to cool for a minimum of ten minutes. Record the time-out and temperature-out of the hot block. If any sample is colorless, it must be re-digested since the sample may have been lost due to insufficient amount of potassium permanganate added prior to digestion, or due to the matrix of the

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sample. If the temperature-out is outside the $95 \pm 3^{\circ}\text{C}$ range, the samples must be re-digested.

- 8.1.11 After cooling, add 3mL of sodium chloride-hydroxylamine sulfate to reduce the excess potassium permanganate. Swirl until purple color is gone. If the purple color persists, extra hydroxylamine sulfate crystals can be added. Allow sample to cool further, approximately 10-15 minutes, or to room temperature, and then adjust the volume to 50mL with DI water, as needed, using the graduations on the digestion vessel that are certified volumetrically by the manufacturer. The samples are ready for analysis.

NOTE: Final volume is recorded as 27.5mL for calculation purposes.

- 8.1.12 Stannous Chloride-HCl (SnCl_2) mix is not added during the digestion process, but is added during analysis through the instrument peristaltic pump. It is mixed with each sample, QC sample and all standards during the analysis.

8.2 Mercury Calibration and Analysis:

- 8.2.1 Daily maintenance- For FIMS100 instruments change tubing, change membrane, and flush system daily. For Leeman Hydra II AA instrument the system is flushed daily.
- 8.2.2 Allow the detector to warm up at least thirty minutes before analyzing samples.
- 8.2.3 Turn on the auto sampler and pump. Clamp pump tubing down. Make sure the argon gas is turned on and that argon gas flow is present. Ensure that the SnCl_2 and DI water are flowing. If the pump is not running correctly, re-adjust the tubing on the pump.
- 8.2.4 Turn on the Flow Injection Automated System (FIAS) Mercury unit and prime the instrument with reagents for thirty minutes.
- 8.2.5 Enter in the computer, the sample run (standards, QC samples and then the samples).
- 8.2.6 After all the information for the sample run has been entered in the computer, and the instrument has been primed, load the digested calibration standards, QC samples, and then the samples into the auto sampler rack. The peristaltic pump mixes the SnCl_2 -HCl solution into the field samples and transfers the standards and samples into the analyzer. Begin the analytical sequence.

- 8.3 Method performance criteria and corrective action procedures are found in Sections 10 & 11 of this procedure.

9.0 DATA ANALYSIS AND CALCULATIONS

See the current Quality Assurance Manual for equations associated with common calculations.

10.0 QUALITY CONTROL AND METHOD PERFORMANCE

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- 10.1 All analysts must meet the qualifications specified in SOP #030205, *Technical Training and Personnel Qualifications* before approval to perform this method. Analysts must complete an initial demonstration of proficiency before being approved to perform this method. Continuing proficiency must be demonstrated using proficiency testing, laboratory control sample analysis and/or MDL studies. Method performance is assessed per analyst. Updated method performance records are filed and stored in a central location within the department.
- 10.2 Use the designated Run log to record batch order and standards/reagents used during analysis. See SOP #030201, *Data Handling and Reporting*.
- 10.3 Batches:
- Preparation batches are defined as sets of 1 – 20 samples. Preparation batch analysis must include the following: 1 method blank, 1 Laboratory Control Sample (LCS), 1 Laboratory Control Sample Duplicate (LCSD), 1 Matrix Spike/Spike Duplicate (MS/MSD) pair. All batch information must be maintained in the preparation documentation assigned to the department.
- 10.4 Analytical batches require all the samples contained within the previous preparation batches and additional instrument operating requirements. Analytical batches include: 1 Initial Calibration Verification (ICV) sample, 1 Low Level Calibration Verification (ICVLL/CCVLL) sample, 1 Initial Calibration Blank (ICB, 1 Continuing Calibration Verification (CCV) sample following every 10th sample and at the conclusion of the sequence, 1 Continuing Calibration Blank following each CCV, Initial Calibration - The curve is prepared daily and must consist of at least five standards and a blank. The calibration is acceptable when the correlation coefficient is ≥ 0.998 . The concentrations of the curve analyzed are as follows, 0.0, 0.2, 0.4, 1.0, 2.0, 5.0, and 10.0ppb. See section 13.4. The calibration curve must contain a standard at or below the reporting limit. The blank is included as a point in the calibration curve to account for any background interferences that may be present in the digestion solutions. Do not force the curve regression fit through zero.
- 10.5 Initial Calibration Verification (ICV)/Low Level Calibration Verification (ICVLL/CCVLL) - After the passing standardization is achieved, analyze the calibration verification standard (ICV). The ICV recovery must be within $\pm 10\%$ of the true value. Following the ICB, a Low Level calibration verification (ICVLL/CCVLL) is analyzed, recovery must be within $\pm 30\%$ of the true value. CCVLL must be run at the end of the analysis.
- 10.6 Continuing Calibration Verification (CCV) - After every ten samples and at the end of the analysis, analyze the mid-range CCV. The mid-range CCV must be within $\pm 10\%$ of the true value.
- 10.7 Method Blank/Initial & Continuing Calibration Blank (ICB/CCB) - One method blank must be analyzed for every twenty samples. The ICB is analyzed following every Initial Calibration Verification (ICV) standard and the CCB is analyzed following each CCV and at the conclusion of the sequence. Mercury should not be detected in the blank $> \frac{1}{2}$ reporting limit (RL).

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- 10.8 Laboratory Control Sample/ Laboratory Control Sample Duplicate (LCS/LCSD) – An LCS/LCSD must be performed with each batch of twenty field samples. The LCS/LCSD recoveries must be $\pm 20\%$. The RPD of the LCS/LCSD must be $< 20\%$.
- 10.9 Matrix Spike/Matrix Spike Duplicate (MS/MSD) - An MS & MSD must be analyzed with every batch of samples. The MS/MSD are prepared by spiking two separate aliquots of a field sample with a known amount of standard. The MS and MSD are prepped and analyzed in the same manner as the samples. Recovery must be within $\pm 25\%$ of the true value for accuracy and the relative percent difference (RPD) must be $< 20\%$.
- 10.10 Serial Dilution - A serial dilution is analyzed if a sample is $\times 25$ the MDL or higher. A serial dilution is, at a minimum, a 5 times dilution of the sample, and must agree within 10% of the original value.
- 10.11 Post Spike - If the MS/MSD recovery fails, or if an MS/MSD is not analyzed, due to insufficient sample, a post spike must be analyzed when the sample concentration is less than $\times 25$ the MDL. If the sample concentration is less than the MDL, spike the sample at $\times 20$ the MDL. The criteria for the post spike must be within $\pm 15\%$ of the true value. **If the MS/MSD recovery passes, a post spike need not be analyzed.**
- 10.12 Dilutions - Any sample, with a concentration over the high standard in the curve, must be diluted within the range of the calibration. This dilution must be at the lowest dilution possible, to keep reporting limits as low as possible.
- 10.13 Digestion Temperature - The hot block's temperature is monitored using a temperature blank containing a thermometer that is calibrated against a NIST traceable reference thermometer. The temperature blank is moved to a different position in the hot block each time samples are digested. The daily temperature blank is documented each day in the appropriate logbook.
- 10.14 Sample Analysis - During the analysis of the samples, the instrument supplies triplicate readings. The responses must have a %RSD of $< 20\%$ for all results $> RL$. The instrument automatically reports the mean of triplicate scans using a simple average calculation: $A+B+C/3 =$ reported concentration for the sample.

Do not report data below the determined analyte reporting limit concentration or below an adjusted detection limit reflecting smaller sample aliquots used in processing or additional dilutions required by the analysis.

Sample data should be reported in units of mg/Kg for soil samples on a dry weight basis. For soil samples report the data generated directly from the instrument with allowance for any sample dilution.

When reporting, round the data values to the tenth place and report analyte concentrations up to two significant figures. Extract concentrations for solids data are rounded in a similar manner before calculations in Section 9.2 are performed.

- 10.15 Instrument Detection Limit (IDL) - The instrument detection limit is calculated by performing ten sequential replicate measurements of a method blank. The standard

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deviation of these measurements is calculated and the IDL is equal to three times the standard deviation of the measurements. The IDL assures with 99% certainty that a value is above the instrument noise level. This IDL study is done on a quarterly basis.

NOTE: An IDL is a statistical determination without analytes present and an MDL is determined with low levels of analytes present.

10.16 For corrective actions, see section 11.0.

11.0 DATA VALIDATION AND CORRECTIVE ACTION

11.1 All sample data must undergo a QC review.

11.1.1 The reviewer must verify that all reportable results are derived from data that are within the calibration range.

11.1.2 Analysts signs and dates the appropriate raw data, printouts and bench sheets

11.1.3 All calculations must be checked.

11.1.4 Data must be checked to confirm that all required QC checks have been analyzed and that they are within acceptable limits.

11.1.5 Data must be checked for the presence or absence of appropriate flags. Comments must be noted when data is flagged.

11.1.6 See SOP #030201, *Data Handling and Reporting*.

11.2 Initial Calibration – Since calibration curves are prepared and digested with field samples, any curve that is prepared and analyzed that does not meet the acceptance criteria must be re-prepared, along with all the relevant field samples and QC that are prepared in conjunction with the calibration curve.

11.3 ICV – If the first run of the mid-range ICV does not pass the $\pm 10\%$ criteria and/or the Low Level does not pass the $\pm 30\%$ criteria, rinse and rerun the standard once. If this fails, corrective action must be taken. The corrective action includes re-calibration and re-analysis, using the same ICV/LL standard. If acceptance criteria are still not met, re-check the standard curve and ICV/LL preparation and/or perform instrument maintenance. If still does not pass, refer to manufacturer's instruction manual, or call a service representative. Since the standards are digested in conjunction with the field sample, if failures result from the standards, all samples within the preparation batch must be re-digested.

11.4 CCV – If the first run of the CCV or the Low Level does not pass and analysis of a second consecutive (immediate) calibration verification fails to produce results within acceptance criteria, corrective actions shall be performed. The laboratory shall demonstrate acceptable performance after the final round of corrective action with two consecutive calibration verifications, or a new initial instrument calibration shall be performed.

11.5 Blanks (Method/ICB/CCB) - If the Method Blank, ICB, or CCB fails the acceptance criteria, stop the run and re-analyze once. . If the contamination still occurs, corrective actions

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can include instrument maintenance, reviewing data for errors and review of the calibration curve. If the contamination still occurs after maintenance has been performed the batch must be re-distilled. A passing method blank must be analyzed before any samples are analyzed.

State Note: For Wisconsin samples, the method blank must not contain analytes more negative than the MDL value. If target analytes are more negative than the MDL, the instrument must be recalibrated or a new LOD study performed.

General guidelines for qualifying sample results with regard to method blank quality are as follows:

- If the method blank concentration is less than the MDL and sample results are greater than the RL, then no qualification is required.
- No qualification is necessary when an analyte is detected in the method blank but not in the associated samples.
- If the concentration in a sample is more than ten times the concentration in the method blank, then no qualification is required.
- If the method blank concentration is greater than the MDL but less than the RL and sample results are greater than the MDL, then qualify associated sample results to indicate that analyte was detected in the method blank.

If the method blank concentration is greater than the RL, further corrective action and qualification is required. An analyst should consult their supervisor for further instruction.

- 11.6 LCS/LCSD – If the LCS fails the criteria, stop the run and re-analyze one more time. If the LCS still fails, re-calibrate the instrument. After recalibration, if the LCS fails again, all samples within the prep batch must be re-digested. If an LCSD is performed, it must also meet the LCS acceptance criteria provided by the manufacturer. If a LCS/LCSD are outside the control limits (>20%), results are flagged with a “J3” (the associated batch QC was outside the established quality control range for precision), re-digestion may be necessary. Consult your supervisor.
- 11.7 MS/MSD – If the MS/MSD fails the criteria, stop the sequence and re-analyze once. If the MS/MSD failure persists, run a post spike. The post spike must pass within the acceptance criteria listed in section 10.12. If the post fails and the LCS was within method limits, then the recovery problem with the MS/MSD is judged to matrix-related. If the LCS is within method control limits, this demonstrates the laboratory performance was in control in a clean matrix. Re-digestion may still be necessary however, if the supervisor or the client requests a re-digestion. If re-digestion is not performed, the failures must be flagged with a “J5” (the sample matrix interfered with the ability to make any accurate determination; spike value is high), or “J6” (the sample matrix interfered with the ability to make any accurate determination; spike value is low). If there is a RPD failure, a re-prep is be used to confirm results, when possible.
- 11.8 Sample Duplicate - If a duplicate is analyzed and the duplicate RPD is outside the control limits (>20%), results are flagged with a “J3” (the associated batch QC was outside the

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established quality control range for precision), re-digestion may be necessary. Consult your supervisor.

- 11.9 Serial Dilution – If the dilution does not meet the required acceptance criteria, re-dilute the sample and re-analyze. If the failure persists, re-analyze both the parent sample and the dilution. If failure still occurs, re-calibrate and re-analyze both the parent and the dilution.
- 11.10 Post Spike – If the MS/MSD recoveries pass the acceptance criteria, a post spike is not required. If performed and the post spike does not meet the acceptance criteria, re-analyze once. If the failure persists, re-analyze both the parent sample and the spike. If failure still occurs, dilute the parent sample and re-spike and re-analyze. If the re-analysis passes, the confirmation of a matrix effect is confirmed and must be noted on the final client report.
- 11.11 Digestion Temperature - If the temperature-out is outside $95 \pm 3^{\circ}\text{C}$ range after the thirty minutes of digestion, re-digest the entire batch.
- 11.12 Digestate - If any sample is colorless after digestion, the sample must be re-digested since the sample may have been lost due to insufficient amount of potassium permanganate added prior to the digestion or due to the matrix of the sample.
- 11.13 Sample Concentrations - The analyst must verify all reported results are derived from the analytical results that are both above the MDL and below the high standard used in the curve.

Sample concentrations that have been analyzed using the extracts in its most concentrated form, and are <RL, report the result as <RL.

For sample results that are above the MDL, but below, the reporting limit, these results must be flagged as estimated values (J flag).

- 11.14 CCVLL - The %R for the CCVLL must be within 30% of the expected concentration. If the recovery does not meet this criterion, re-analyze once. If the failure persists, the instrument should be checked, the reagents and standards should be checked, and the instrument must be re-calibrated. If no obvious cause is identified for the failure, re-prepare and re-analyze the workgroup

STATE NOTE: If the sample is analyzed in conjunction with the Ohio VAP, corrective action for failing QC (i.e. blank, spike, etc.) must be performed prior to flagging data, if sufficient sample volume was submitted by the client. Corrective action can include re-analysis, if instrument malfunction is suspected, or re-preparation and re-analysis, if the failure is suspected as either extraction or sample related.

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12.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

12.1 The EPA requires that laboratory waste management practice to be conducted consistent with all applicable federal and state laws and regulations. Excess reagents, samples and method process wastes must be characterized and disposed of in an acceptable manner. See *Pace National Waste Management Plan*.

12.2 See SOP #030302, *Environmental Sustainability & Pollution Prevention*.

13.0 METHOD MODIFICATIONS/CLARIFICATIONS

13.1 HCl is used in the SnCl₂ mixture per the instrument manufacturer's instruction manual that specifies HCl in the SnCl₂ instead of sulfuric acid.

13.2 The stannous chloride solution concentration has been modified in this procedure to reflect the instrument manufacturer's recommendation (Mercury Hydride Analysis Flow Injection (pg. 2/13).

13.3 Modifications to this method are noted in the body of the text as state notes. Compliance analyses performed in conjunction with specific state requirements must be performed as noted within the specific state(s) listed.

13.4 Adjustments to the concentrations of standards/spiking solutions, standards providers, and quality control are subject to change to better meet client/project/regulatory needs or to improve laboratory method performance.

13.5 The sample amount utilized by this procedure has been modified from the method due to the capacity of the digestion containers. Digestion solutions have been reduced proportionally with this reduction in sample used to remain as consistent as possible with method requirements.

14.0 REFERENCES

14.1 *Mercury in Solid or Semisolid Waste (Manual Cold-Vapor Technique)*, SW-846 Method 7471A, Revision 1, September 1994.

14.2 Perkin Elmer FIMS 400 Instruction Manual.

14.3 *Mercury in Solid or Semisolid Waste (Manual Cold-Vapor Technique)*, SW-846 Method 7471B, Revision 2, February 2007.

14.4 *Flame Atomic Absorption Spectrophotometry*, SW-846 Method 7000B, Revision 2, February 2007.

14.5 *Atomic Absorption Methods*, SW-846 Method 7000A, Revision1, July 1992.

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Attachment I: Revision History

Current Version:

Version	Date	Description of Revisions
13	7/27/18	Update in response to WI audit. Changed logo. Added state note to 11.5

Superseded Versions:

This document supersedes the following:

Version	Date	Description of Revisions
0	7/30/04	Origination
1	12/5/05	
2	11/3/06	
3	1/23/09	
4	1/29/09	
5	9/20/10	
6	5/4/12	Technical and Quality Review and update. Revised sections 8.1.1 through 8.1.5, 12.1 and 13.4; Added state notes prior to section 1.0 and following section 10.17; Added sections 1.2.1, 2.20 through 2.27, 4.6, 10.17 and 11.15.
7	10/15/13	Technical and Quality Review and update. Revised sections 1.1, 7.1, 7.8, 8.1.2 through 8.1.5, 8.1.8 and 8.1.11; Added section 13.5.
8	8/17/2015	Technical and Quality Review and update. Revised Sections 2.10, 2.13, 2.14, 6.1, 6.8, 7.8, 7.10 through 7.12, 8.1.1 through 8.1.3, 8.1.5, 8.1.8, 10.3, 10.5 through 10.10, 11.3, 11.4, and 11.6. Revised State Note Prior to Section 1.0. Deleted State Note in Section 10.7. Deleted Sections 2.10, 7.13, 10.10, and 11.11.
9	9/9/2015	Header and signature block formatting. Technical and quality review and update. Revised Sections 1.2.1 and 8.1.5. Added Attachment II.
10	10/20/2015	Technical review and update. Added Section 8.2.1.
11	10/14/2016	Technical and quality review and update. Header and signature block re-formatting. Revised Sections 2.4, 2.5, 2.6, 2.7, 2.8, 7.1, 7.8, 7.9, 7.10, 7.11, 7.12, 7.13, 8.1.3, 8.1.11, 9.0, 10.16, 11.4, 11.5, 11.14, 14.1, 14.2, 14.3, 14.4, 14.5, and Attachment II Table 2. Deleted Sections 2.4, 2.6, 2.8 through 2.16, 2.19 through 2.26, 9.1 through 9.6, 10.5, and 10.16.
12	11/30/2017	Update in response to A2LA audit finding CAR2872. Changed ESC logo. Updated Section 3.1 and Attachment II Table 4.

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Attachment II: DoD Requirements

1.0 Equipment/Instrument Maintenance

Instrument maintenance must be performed routinely to optimize instrument performance and improve chromatography. Commonly performed maintenance includes lamp replacement, optical cell and UV window cleaning, mirror cleaning, tubing replacement, etc. A new calibration curve must be analyzed following any major maintenance performed on the analytical system.

2.0 Computer Hardware and Software

Software name and version: AA WINLAB, version 2.50 or equivalent

3.0 Troubleshooting

Table 1. Mercury Troubleshooting Guide		
Problem	Cause	Treatment
Hydra II		
No or Low Signal for Standards	Stannous Chloride	Check tubing. Replace stannous chloride solution.
	Carrier Gas	Check liquid/gas separator to ensure proper gas flow. Check exhaust of the CVAAS module by placing tubing into container of water and observe bubbling.
	Leak in the sample vapor path	Check all tubing and connections. Ensure that the absorption cell windows are in place.
	Blockage in the sample vapor path	Submerge the CVAAS exhaust line in water to confirm that the carrier gas is flowing throughout the system.
No or Low Signal for Samples	Incomplete Digestion	Samples high in organic content may require additional oxidant or heating during the digestion to complete oxidation.
	Sample Interferences	Samples with high iodide (or other interferent) content will cause low signal. Dilute sample.
Poor Precision	Signal has not reached plateau	Increase the uptake time or gas flow
	Integration	Longer integration times should result in better precision.
	Low light intensity	The signal value displayed on the Method/Instrument Control should be 250000 or greater. If low, make sure cells are properly held in clamps and clean optics if necessary.
Lamp does not light	Instrument not powered	Check electrical connections. Ensure instrument is powered up.

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Table 1. Mercury Troubleshooting Guide

Problem	Cause	Treatment
	Instrument status is idle	Restart instrument by completely powering down. If problem persists, schedule outside maintenance.
	Lamp status is off	Change status to on in instrument menu bar.
	Lamp assembly is not fully inserted into control board	Power down the instrument before touching the lamp. Release lamp contacts then re-insert lamp.
	Defective lamp	Replace lamp.
FIMS		
Poor Sensitivity	Reagents	Prepare fresh solutions. Install new tubes and manifold.
	Gas/Liquid Separator	Adjust the flow. Install a new dry membrane. Clean the gas/liquid separator.
	Carrier Gas Flow	Optimize the carrier gas flow.
	Leaks, Blockages, Contamination	Ensure the carrier and reductant tubes are in the correct containers. Replace worn pump tubes. Clean the fluid system. Clean the manifold.
	FIMS-Cell	Clean the cell or windows. Reduce the extraction rate of the fumes from the exhaust outlet of the cell.
All replicates following the first reading give low absorbance readings	Fill Step in FIAS program too short	Increase the time for the Fill step on the FIAS page of the method editor
First replicate of a series gives low absorbance reading	Prefill step in FIAS program too short	Increase the time for the prefill step on the FIAS page of the method editor or shorten the tube between the sample container and the FIAS valve.
Double peak in peak display	Concentration of samples too high	Dilute the samples
	Air trapped in the fluid system	Check all tubing connections. Tighten by hand. Replace damaged connectors
	Carrier or reductant flows are incorrect	Ensure the carrier and reductant tubes are in the correct containers. Set the flows correctly.
	No acid in sample solutions or acid too weak	Add acid to the samples
Baseline shift in Peak Profile	Air trapped in the fluid system	Check all tubing connections. Tighten by hand. Replace damaged connectors
Pump heads stop rotating	Pressure on the pump tube magazines is too high	Reduce the pressure with the pressure adjustment screws

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4.0 Other Requirements

- 4.1 All hardcopy laboratory notebooks must be reviewed by the Supervisor, or their designee, on a monthly basis.
- 4.2 If not self-explanatory (e.g., a typo or transposed number), corrections to technical and quality records shall also include a justification for the change.
- 4.3 A person performing a manual integration must sign and date each manually integrated chromatogram and record the rationale for performing manual integration. Electronic signatures are acceptable.
- 4.4 The results of calibration and verification of support equipment must be within the specifications required of the application for which this equipment is used or the equipment must be removed from service until repaired. Calibration and verification records, including those of established correction factors, must be maintained. In the absence of method-specific requirements, the minimum requirements are as follows:

Table 2. Support Equipment Checks		
Performance Check	Frequency	Acceptance Criteria
Balance calibration check [Using two standard weights that bracket the expected mass]	Daily prior to use	Top-loading balance: $\pm 2\%$ or $\pm 0.02\text{g}$, whichever is greater Analytical balance: $\pm 0.1\%$ or $\pm 0.5\text{mg}$, whichever is greater
Verification of standard mass [Using weights traceable to the International System of Units (SI) through a NMI]	Every 5 years	Certificate of Calibration from ISO/IEC 17025 accredited calibration laboratory
Monitoring of refrigerator/freezer temperatures	Daily (i.e. 7 days per week) [use MIN/MAX thermometers or data loggers equipped with notification of out of control event capabilities if personnel not available to record daily]	Refrigerators: 0°C to 6°C Freezers: $\leq -10^{\circ}\text{C}$
Thermometer verification check [Using a thermometer traceable to the SI through an NMI] [Performed at two temperatures that bracket the target temperature(s). Assume linearity between the two bracketing temperatures.] [If only a single temperature is used, at the temperature of use]	Liquid in glass: Before first use and annually Electronic: Before first use and quarterly	Apply correction factors or replace thermometer

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**TITLE: MERCURY IN SOLID WASTE (COLD-VAPOR TECHNIQUE)
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Table 2. Support Equipment Checks

Performance Check	Frequency	Acceptance Criteria
Volumetric labware	Class B: By lot before first use Class A and B: Upon evidence of deterioration	Bias: Mean within $\pm 2\%$ of nominal volume Precision: RSD $\leq 1\%$ of nominal volume (based on 10 replicate measurements)
Non-volumetric labware [Applicable only when used for measuring initial sample volume and final extract/ digestates volume]	By lot before first use or upon evidence of deterioration	Bias: Mean within $\pm 3\%$ of nominal volume Precision: RSD $\leq 3\%$ of nominal volume (based on 10 replicate measurements)
Mechanical volumetric pipette	Quarterly	Bias: Mean within $\pm 2\%$ of nominal volume Precision: RSD $\leq 1\%$ of nominal volume (based on minimum of 3 replicate measurements) [Note: for variable volume pipettes, the nominal volume is the volume of use]
Glass microliter syringe	Upon receipt and upon evidence of deterioration	General Certificate of Bias & Precision upon receipt Replace if deterioration is evident
Drying oven temperature check	Daily prior to and after use	Within $\pm 5\%$ of set temperature
Water purification system	Daily prior to use	See method blank criteria given in Section 4.20 of this addendum

- 4.5 The expiration date of the prepared standard shall not exceed the expiration date of the primary standard. All containers must bear a preparation date.
- 4.6 To avoid preparing non-representative samples, the laboratory shall not “target” within a relatively small mass range (e.g., $1.00 \pm 0.01\text{g}$) because such targeting will produce non-representative subsamples if the sample has high heterogeneity. The laboratory shall not manipulate the sample material so the sample aliquot weighs exactly $1.00\text{g} \pm 0.01\text{g}$, as an example.
- 4.7 In the absence of project-specific requirements, the minimum standard data qualifiers to be used are:
- U Analyte was not detected and is reported as less than the LOD or as defined by the customer. The LOD has been adjusted for any dilution or concentration of the sample.
 - J The reported result is an estimated value (e.g., matrix interference was observed or the analyte was detected at a concentration outside the quantitation range).

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- B Blank contamination. The recorded result is associated with a contaminated blank.
- N Non-target analyte. The analyte is a tentatively identified compound using mass spectrometry or any non-customer requested compounds that are tentatively identified.
- Q One or more quality control criteria failed (e.g., LCS recovery, surrogate spike recovery, or CCV recovery).

Additional data qualifiers may be used, or different letters or symbols to denote the qualifiers listed above, as long as they are appropriately defined and their use is consistent with project-specific requirements (e.g., QSM 5.0, the contract, and project-planning documents).

- 4.8 If the time of the sample collection is not provided, assume the most conservative time of day. For the purpose of batch processing, the start and stop dates and times of the batch preparation shall be recorded.
- 4.9 Each preparation method listed on the scope of accreditation must have quarterly LOD/LOQ verifications. However, not all possible combinations of preparation and cleanup techniques are required to have LOD/LOQ verifications. If LOD/LOQ verifications are not performed on all combinations, the laboratory must base the LOD/LOQ verifications on the worst case basis (preparation method with all applicable cleanup steps).
- 4.10 After each MDL determination, the laboratory must establish the LOD by spiking a quality system matrix at a concentration of at least 2 times but no greater than four times the MDL. This spike concentration establishes the LOD and the concentration at which the LOD shall be verified. It is specific to each suite of analyte, matrix, and method (including sample preparation). The following requirements apply to the initial LOD establishment and to the LOD verifications:
- The apparent signal to noise (S/N) ratio at the LOD must be at least three and the results must meet all method requirements for analyte identification (e.g., ion abundance, second column confirmation, or pattern recognition). For data systems that do not provide a measure of noise, the signal produced by the verification sample must produce a result that is at least three standard deviations greater than the mean method blank concentration. This is initially estimated based on a minimum of four method blank analyses and later established with a minimum of 20 method blank results.
 - If the LOD verification fails, then the laboratory must repeat the MDL determination and LOD verification or perform and pass two consecutive LOD verifications at a higher spike concentration and set the LOD at the higher concentration.
 - The laboratory shall maintain documentation for all MDL determinations and LOD verifications.

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- The DL and LOD must be reported for all analyte-matrix-methods suites, unless it is not applicable to the test or specifically excluded by project requirements.
- 4.11 The LOD shall be verified quarterly. In situations where methods are setup and used on an infrequent basis, the laboratory may choose to perform LOD verifications on a one per batch basis. All verification data will be in compliance, reported, and available for review.
- 4.12 For DoD, at a minimum, the LOQ shall be verified quarterly. In situations where methods are setup and used on an infrequent basis, the laboratory may choose to perform LOQ verifications on a one per batch basis.
- 4.13 All initial instrument calibrations must be verified with a standard obtained from a second manufacturer prior to analyzing any samples. The use of a standard from a second lot obtained from the same manufacturer (independently prepared from different source materials) is acceptable for use as a second source standard. The concentration of the second source standard shall be at or near the midpoint of the calibration range. The acceptance criteria for the initial calibration verification must be at least as stringent as those for the continuing calibration verification.
- 4.14 Exclusion of calibration points without documented scientifically valid technical justification is not permitted.
- 4.15 The concentration of the CCV standard shall be greater than the low calibration standard and less than or equal to the midpoint of the calibration range.
- 4.16 All CCVs analyzed must be evaluated and reported. If a CCV fails, reanalysis or corrective actions must be taken.
 - If a CCV fails, the laboratory can immediately analyze two additional consecutive CCVs (immediately is defined as starting a consecutive pair within one hour; no samples can be run between the failed CCV and the two additional CCVs). This approach allows for spurious failures of analytes to be reported without reanalysis of samples. Any corrective actions that change the dynamics of the system (e.g., clip column, clean injection port, run blanks) requires that all samples since the last acceptable CCV be reanalyzed.
 - Both of these CCVs must meet acceptance criteria in order for the samples to be reported without reanalysis.
 - If either of these two CCVs fail or if the laboratory cannot immediately analyze two CCVs, the associated samples cannot be reported and must be reanalyzed.
 - Corrective action(s) and recalibration must occur if the above scenario fails. All affected samples since the last acceptable CCV must be reanalyzed.
 - Flagging of data for a failed CCV is only appropriate when the affected samples cannot be reanalyzed. The laboratory must notify the client prior to reporting data associated with a failed CCV.

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**TITLE: MERCURY IN SOLID WASTE (COLD-VAPOR TECHNIQUE)
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- 4.17 The results of all MS/MSDs must be evaluated using the same acceptance criteria used for the DoD LCS limits (see Addendum Table 3) or project limits, if specified. If the specific analyte(s) are not available in the Addendum Table 3, the laboratory shall use their LCS in-house limits (see the LIMS) as a means of evaluating MS/MSDs. The MS and MSD must be spiked with all reported analytes.
- 4.19 Surrogate spike results shall be compared with DoD LCS limits (see Addendum Table 3) or acceptance criteria specified by the client. If these criteria are not available, the laboratory shall compare the results with its in-house statistically established LCS criteria (see the LIMS).
- 4.20 The method blank shall be considered to be contaminated if:
- The concentration of any target analyte (chemical of concern) in the blank exceeds 1/2 the LOQ and is greater than 1/10th the amount measured in any associated sample, or 1/10th the regulatory limit, whichever is greater;
 - The concentration of any common laboratory contaminant in the blank exceeds the LOQ;
 - If a method blank is contaminated as described above, then the laboratory shall reprocess affected samples in a subsequent preparation batch, except when sample results are below the LOD. If insufficient sample volume remains for reprocessing, the results shall be reported with appropriate data qualifiers.
- 4.21 Sporadic Marginal Exceedances are not allowed for target analytes (chemicals of concern as identified by a project) without project-specific approval. Target analytes are considered those few analytes that are critical for the success of a project (such as risk drivers) where sporadic marginal exceedances cannot be allowed. Laboratories should consult with clients whenever long lists of analytes are requested for analysis to determine if marginal exceedances will not be allowed.
- 4.22 DoD considers the same analyte exceeding the LCS control limit two (2) out of three (3) consecutive LCS to be indicative of non-random behavior, which requires corrective action and reanalysis of the LCS.

Table 3. LCS Control Limits – Method 7470 – 7471 Series Solid Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
7439-97-6	Mercury	6471	102	7.5	80	124

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TITLE: MERCURY IN SOLID WASTE (COLD-VAPOR TECHNIQUE) (EPA METHODS 7471A & 7471B)

Table 4. Quality Control Requirements – Inorganic Analysis by Atomic Absorption Spectrophotometry (AA)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Initial Calibration (ICAL) for all analytes	Daily ICAL prior to sample analysis.	$r^2 \geq 0.99$.	Correct problem, then repeat ICAL.	Flagging is not appropriate.	FLAA and GFAA: minimum three standards and a calibration blank. CVAA/Mercury: minimum 5 standards and a calibration blank. No samples shall be analyzed until ICAL has passed.
Initial Calibration Verification(ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All reported analytes within $\pm 10\%$ of the true value.	Correct problem. Rerun ICV. If that fails, rerun ICAL.	Flagging is not appropriate.	No samples shall be analyzed until calibration has been verified with a second source.

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TITLE: MERCURY IN SOLID WASTE (COLD-VAPOR TECHNIQUE) (EPA METHODS 7471A & 7471B)

Table 4. Quality Control Requirements – Inorganic Analysis by Atomic Absorption Spectrophotometry (AA)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Low-level Calibration Check Standard (LLCCV)	Daily.	All reported analytes within $\pm 20\%$ of the true value.	Correct problem and repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed without a valid Low-Level Calibration Check Standard (LLCCV). LLCCV should be less than or equal to the LOQ. If the concentration of the lowest calibration standard is less than or equal to the LOQ, the lowest standard may be re-quantified against the calibration curve as a LLCCV. Otherwise, a separate standard must be analyzed as the LLCCV prior to the analysis of any samples.

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TITLE: MERCURY IN SOLID WASTE (COLD-VAPOR TECHNIQUE) (EPA METHODS 7471A & 7471B)

Table 4. Quality Control Requirements – Inorganic Analysis by Atomic Absorption Spectrophotometry (AA)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Continuing Calibration Verification (CCV)	After every 10 field samples and at the end of the analysis sequence.	All reported analytes within $\pm 10\%$ of the true value.	Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails or if two consecutive CCVs cannot be run, take perform corrective action(s) and repeat CCV and all affected samples since the last successful CCV. Alternately, recalibrate if necessary; then reanalyze all associated samples since the last acceptable CCV.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable CCV.	Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

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TITLE: MERCURY IN SOLID WASTE (COLD-VAPOR TECHNIQUE) (EPA METHODS 7471A & 7471B)

Table 4. Quality Control Requirements – Inorganic Analysis by Atomic Absorption Spectrophotometry (AA)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Method Blank (MB)	One per preparatory batch.	The absolute values of all analytes must be < ½ LOQ or < 1/10th the amount measured in any sample or 1/10 the regulatory limit, whichever is greater.	Correct problem. If required, reprep and reanalyze MB and all QC samples and field samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be re-prepped or reanalyzed. Non-detects associated with positive blank infractions may be reported. Sample results > 10X the LOQ associated with negative blanks may be reported.
Initial and Continuing Calibration Blank (ICB/CCB)	Immediately after the ICB and immediately after every CCV.	The absolute values of all analytes must be < ½ LOQ or < 1/10th the amount measured in any sample or 1/10th the regulatory limit, whichever is greater.	ICB: Correct problem and repeat ICB/ICB analysis. If that fails, rerun ICAL. All samples following the last acceptable Calibration Blank must be reanalyzed. CCBs may not be reanalyzed without reanalysis of the associated samples and CCV(s).	Flagging is not appropriate.	Results may not be reported without a valid calibration blank. Non-detects associated with positive blank infractions may be reported. Sample results > 10X the LOQ associated with negative blanks may be reported. For CCB, failures due to carryover may not require an ICAL.

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TITLE: MERCURY IN SOLID WASTE (COLD-VAPOR TECHNIQUE) (EPA METHODS 7471A & 7471B)

Table 4. Quality Control Requirements – Inorganic Analysis by Atomic Absorption Spectrophotometry (AA)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Laboratory Control Sample(LCS)	One per preparatory batch.	A laboratory must use Table 3 limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per preparatory batch.	A laboratory must use Table 3 limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	For matrix evaluation only. If MS results are outside the limits, the data shall be evaluated to the source of difference (i.e., matrix effect or analytical error).
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch.	A laboratory must use Table 3 limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified. MSD or MD: RPD of all analytes \leq 20% (between MS and MSD or sample and MD).	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J- flag if acceptance criteria are not met and explain in the case narrative.	The data shall be evaluated to determine the source of difference. For Sample/MD: %Recovery and RPD criteria only apply to analytes whose concentration in the sample is greater than or equal to the LOQ.

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TITLE: MERCURY IN SOLID WASTE (COLD-VAPOR TECHNIQUE) (EPA METHODS 7471A & 7471B)

Table 4. Quality Control Requirements – Inorganic Analysis by Atomic Absorption Spectrophotometry (AA)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Dilution Test (Flame AA and GFAA only)	One per preparatory batch if MS or MSD fails.	Five-fold dilution must agree within $\pm 10\%$ of the original measurement.	No specific CA, unless required by the project.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	Only applicable for samples with concentrations $> 50 \times$ LOQ (prior to dilution). Use along with MS/MSD or PDS data to confirm matrix effects.
Post-Digestion Spike (PDS) Addition (Flame AA and GFAA only)	One per preparatory batch if MS or MSD fails.	Recovery within 80-120%.	No specific CA, unless required by the project.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	Criteria apply for samples with concentrations $< 50 \times$ LOQ prior to dilution.
Method of Standard Additions (MSA)	When dilution or post digestion spike fails and if the required by project.	NA.	NA.	NA.	Document use of MSA in the case narrative.

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TITLE: DETERMINATION OF METALS AND TRACE ELEMENTS IN VARIOUS MATRICES BY ICP-AES (EPA METHODS 6010B, 6010C, 6010D [ICP-OES], AND 200.7) INCLUDING HARDNESS (EPA METHODS 200.7 AND 6010B/C/D AND SM 2340B)

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1.0 SCOPE AND APPLICATION

STATE NOTE: For samples analyzed in conjunction with the Ohio Voluntary Action Program (VAP) please utilize SOP #340386OH.

- 1.1 Inductively coupled plasma-atomic emission spectrometry (ICP-AES) determines trace elements, including metals and some non-metals in solution. This procedure follows the guidelines established in EPA method 200.7 and SW-846 Method 6010B, 6010C, and 6010D for drinking water, waste water, ground water, TCLP, SPLP, and STLC leachates, soils, sludge, sediments, solid wastes, oils, and other digestates after appropriate preparatory procedure is performed.

This procedure is also applicable to reporting calculated values for Calcium, Magnesium, and Total Hardness from values determined using EPA methods 200.7 or 6010B/C/D from groundwater, wastewater and drinking waters. Reporting limits for Hardness are derived from the annual MDL studies for Calcium and Magnesium of the appropriate determinative EPA method. The routine reporting limits for each category of hardness are listed in Table 1.2c.

- 1.2 This method is applicable for the analytes listed in Table 1.2a, b and c. Detection limits, sensitivity, and the optimum and linear concentration ranges of the elements can vary with the wavelength, spectrometer, matrix, and instrument operating conditions. Table 1.2 also lists the Reporting Limits (RLs), used routinely by Pace National.

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**DETERMINATION OF METALS AND TRACE ELEMENTS IN VARIOUS MATRICES BY
TITLE: ICP-AES (EPA METHODS 6010B, 6010C, 6010D [ICP-OES], AND 200.7) INCLUDING
HARDNESS (EPA METHODS 200.7 AND 6010B/C/D AND SM 2340B)**

Table 1.2a: Environmental Analytes and Reporting Limits *(Subject to change, see section 13.1)*

Analyte	Aqueous				Sediment		
	Ground Water/ Wastewater 6010B/C/D/200 .7	Drinking Water 200.7*	RL	Units	Solids 6010B/C/ D	RL	Units
Aluminum	✓	✓	00.200	mg/L	✓	2.00	mg/Kg
Antimony	✓	✓	0.010	mg/L	✓	1.00	mg/Kg
Arsenic	✓	✓	00.010	mg/L	✓	1.00	mg/Kg
Barium	✓	✓	0.005	mg/L	✓	0.50	mg/Kg
Beryllium	✓	✓	0.002	mg/L	✓	0.20	mg/Kg
Boron	✓	✓	0.050	mg/L	✓	5.0	mg/Kg
Cadmium	✓	✓	0.002	mg/L	✓	0.20	mg/Kg
Calcium	✓	✓	1.000	mg/L	✓	100	mg/Kg
Chromium	✓	✓	0.010	mg/L	✓	1.00	mg/Kg
Cobalt	✓	✓	0.010	mg/L	✓	1.00	mg/Kg
Copper	✓	✓	0.010	mg/L	✓	1.00	mg/Kg
Iron	✓	✓	0.100	mg/L	✓	10.0	mg/Kg
Lead	✓	✓	0.005	mg/L	✓	0.50	mg/Kg
Lithium	✓		0.015	mg/L	✓	1.50	mg/Kg
Magnesium	✓	✓	1.000	mg/L	✓	100	mg/Kg
Manganese	✓	✓	0.010	mg/L	✓	1.00	mg/Kg
Molybdenum	✓	✓	0.005	mg/L	✓	0.50	mg/Kg
Nickel	✓	✓	0.010	mg/L	✓	1.00	mg/Kg
Potassium	✓	✓	1.000	mg/L	✓	100	mg/Kg
Selenium	✓	✓	0.010	mg/L	✓	1.00	mg/Kg
Silicon	✓	✓	0.050	mg/L	✓	5.00	mg/Kg
Silver	✓	✓	0.005	mg/L	✓	5.00	mg/Kg
Sulfur	✓	✓	1.0	mg/L	✓		mg/Kg
Sodium	✓	✓	1.000	mg/L	✓	100	mg/Kg
Strontium	✓	✓	0.010	mg/L	✓	1.00	mg/Kg
Thallium	✓	✓	0.010	mg/L	✓	1.00	mg/Kg
Tin	✓	✓	0.050	mg/L	✓	5.00	mg/Kg
Titanium	✓	✓	0.050	mg/L	✓	5.00	mg/Kg
Vanadium	✓	✓	0.010	mg/L	✓	1.00	mg/Kg
Zinc	✓	✓	0.050	mg/L	✓	5.00	mg/Kg

*May not meet required Drinking Water Maximum Contamination Levels (MCLs) using this methodology.

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TITLE: DETERMINATION OF METALS AND TRACE ELEMENTS IN VARIOUS MATRICES BY ICP-AES (EPA METHODS 6010B, 6010C, 6010D [ICP-OES], AND 200.7) INCLUDING HARDNESS (EPA METHODS 200.7 AND 6010B/C/D AND SM 2340B)

Table 1.2b: Hardness Categories and Reporting Limits

(Subject to change, see section 13.1)

Hardness:	RL (mg/L)
Calcium Hardness	1.25
Magnesium Hardness	0.41
Total Hardness	1.6

- 1.3 For the determination of total recoverable analytes in aqueous and solid samples, an acid digestion process is required. Environmental samples for analysis by Method 6010B, 6010C, or 6010D including, TCLP or EP leachates, soils, sludge, sediments, and other solid wastes require an acid digestion prior to analysis. Samples are digested by SW-846 methods 3005 (Acid Digestion of Waters for Total Recoverable Metals), 3010 (Acid Digestion of Aqueous Samples), 3015 (Microwave Digestion of Aqueous Samples), 3050 (Acid Digestion of Sediments, Sludge, Soil, and Oils) and 3051 (Microwave Assisted Digestion of Sediments, Sludge, Soil, and Oils). Digestion methods are found in Pace National SOPs 340388 and 340389.
- 1.4 The Clean Water Act has approved EPA Method 200.7 for demonstrating compliance on discharge monitoring for NPDES (National Pollution Discharge Elimination System) permits. 40 CFR136.3 has Guidelines for Establishing Test Procedures for Analysis of Pollutants. The National Primary Drinking Water Regulations for inorganic chemical sampling and analytical requirements can be found in 40 CFR141.23. Updates to these regulations can be found in the current Code of the Federal Register.
- 1.5 To determine dissolved analytes in aqueous samples, a 0.45µm filtration method is employed then the filtered samples are acidified. To reduce potential interferences, dissolved solids must be < 0.2% (w/v).
- 1.6 Analysis without acid digestion can be used for drinking water samples if the samples have been properly preserved with acid and have turbidity of < 1 NTU at the time of analysis. These samples must be acidified to match the acid matrix of the calibration standards and analyzed directly. This total recoverable determination procedure is referred to as "direct analysis". Silver concentration cannot be determined from direct analysis when chloride ions are present as a silver chloride precipitate may be formed. The sample must be acid digested to form a soluble silver chloride complex. Some primary drinking water metal contaminants may require sample concentration to meet regulatory drinking water reporting limits criteria^{14.2}.

Method 6010D – Samples that are not digested necessitate the use of either an internal standard or should be matrix-matched with the standards. If using the former option, the instrument software should be programmed to correct for the intensity differences of the internal standard between samples and standards. NOTE: All samples analyzed by Method 6010 are typically digested.

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- 1.7 When determining boron and silicon in aqueous samples, only plastic, PTFE (Teflon®) sample containers and laboratory glassware must be used. For accurate determination of boron in solid samples, only quartz or PTFE tubes must be used during acid digestion with immediate transfer of an aliquot of the final volume of digestate to a plastic centrifuge tube^{14.2}.
- 1.8 For the determination of titanium, white plastic and white printed containers must be avoided as titanium dioxide is used as a white pigment.
- 1.9 The total recoverable sample digestion procedure dissolves and maintains in solution only minimal concentrations of barium in the presence of free sulfate. For the analysis of barium in samples having varying and unknown concentrations of sulfate, analysis must be completed as soon as possible following sample preparation^{14.2}.
- 1.10 Detection limits and linear ranges for the elements vary with the wavelength selected, the spectrometer, and the matrix. Table 1.11 provides a list of routinely used wavelengths and the type of spectrometer view used.

Method 6010D – IDLs are necessarily instrument-specific. Therefore, if needed, an IDL must be determined through a separate experimental study for each instrument. IDLs should be established, at a minimum, on an annual basis for each matrix and for each preparatory/determinative method combination used.

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TABLE 1.10: WAVELENGTHS
(exact wavelengths vary slightly depending on the instrument)

Analyte	Wavelength (nm)	Type of View
Aluminum	308.215	Radial
Antimony	206.836	Axial
Arsenic	188.979	Axial
Barium	233.527	Axial
Beryllium	313.107	Radial
Boron	249.772	Radial
Cadmium	214.440	Axial
Calcium	317.933 373.690	Radial
Chromium	205.560	Axial
Cobalt	228.616	Axial
Copper	324.752	Radial
Iron	259.940 271.441	Radial
Lead	220.353	Axial
Lithium	670.784	Radial
Magnesium	279.077	Radial
Manganese	257.610	Axial
Molybdenum	202.031	Axial
Nickel	232.003	Axial
Potassium	766.490	Radial
Selenium	196.026	Axial
Silicon	251.611	Axial
Silver	328.068	Axial
Sodium	589.592 818.326	Radial
Strontium	407.771	Radial
Sulfur	181.972	Axial
Thallium	190.801	Axial
Tin	189.927	Axial
Titanium	334.940	Radial
Vanadium	292.402	Radial
Zinc	213.857	Axial

- 1.11 Users of the data generated using this method must state the data-quality objectives (DQOs) prior to analysis.

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- 1.12 Any deviations from this SOP must be documented. Deviations are reflected in a case narrative and the method is reported as modified. Per customer requirement, the procedure and QC criteria described in this SOP can be changed/modified. Authorization from the Operations Manager and Project Manager is required for each modification and Regulatory Affairs approval must also be secured for any deviation.
- 1.13 An MDL study must be completed at least annually or more frequently if major instrumentation changes occur. Method Detection Limits (MDLs) are performed based on Pace National SOP #030206. Updated MDL records are filed and stored in a central location within the department.
- 1.13.1 Limit of Detection (LOD) and Limit of Quantitation (LOQ) studies are completed at the frequency required by the TNI standard per the procedure identified in the Pace National SOP #030206, *Method Detection Limits (MDL), Limits of Detection (LOD), and Limits of Quantitation (LOQ)*. Should the procedure be utilized for DOD support; then the frequency of these studies must meet the requirements of the current DOD QSM.
- 1.14 Linear Dynamic Range (LDR) and Inter-element correction factor (IEC) studies must be analyzed semi-annually for each analytical instrument or when there are major changes/repairs to the instrument^{14.5, 14.1}. Instrument Detection Limit studies must be analyzed at least quarterly for each analytical instrument^{14.5}.

2.0 METHOD SUMMARY AND DEFINITIONS

- 2.1 The analysis described in this method involves multi-elemental determinations by ICP-AES using sequential or simultaneous instruments. The instrument measures characteristic atomic-line emission spectra by optical spectrometry. Samples are aspirated into the nebulizer and the resulting aerosol is transported to the plasma torch. The emission spectra are dispersed by a grating spectrometer separating the light emitted into the distinct wavelengths generated by each element in the sample. A photosensitive device monitors the intensities of each wavelength line in the spectra. The intensity of light on the photosensitive device produces a signal that is measured and processed by a computer system. Due to the many possible wavelengths of light generated by each element and possible overlapping of high intensity peaks, a background correction technique is required for trace element determination. Background intensities must be measured adjacent to the analyte spectra lines during analysis. The position selected for background intensity measurement can be selected on either or both sides of the analyte wavelength line and must be determined by the complexity of the spectrum adjacent to the analyte line. The position used for background correction must be as free from spectral interference as possible and must reflect the same change in background intensity as occurs at the analyte wavelength. Background correction is not required in cases of line broadening where the background correction measurement would actually degrade the analytical result. The possibility of additional interferences should also be recognized and appropriate corrections made.

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- 2.2 Dissolved Analyte - The concentration of analyte in an aqueous sample that has been passed through a 0.45µm membrane filter assembly prior to sample acidification and digestion.
- 2.3 Total (Total Recoverable) Analyte – The concentration of analyte determined either by “direct analysis” of an unfiltered acid preserved drinking water sample with turbidity of <1 NTU or by analysis of the solution extract of a solid sample or an unfiltered aqueous sample following digestion by refluxing with hot dilute mineral acid(s) as specified in the method
- 2.4 Instrument Detection Limit (IDL) - The concentration equivalent to the analyte signal which is equal to three times the standard deviation of a series of 10 replicate measurements of the calibration blank signal at the same wavelength. The IDL assures with 99% certainty that a value is above the instrument noise level.
- Note:** An IDL is a statistical determination without analytes present used to assess background correction protocols and an MDL is determined with low levels of analytes present to determine instrument sensitivity for each analyte.
- 2.5 Linear Dynamic Range (LDR) - The range over which the instrument response to analyte concentration remains linear.
- 2.6 Plasma Solution - A solution that is used to determine the optimum torch height relative to the radio frequency (RF) coil for viewing the spectrum.
- 2.7 Interference Check Sample (ICS) – A series of two solutions (ICSA & ICSAB) to verify that inter-element interferences are correctly compensated. The ICSA and ICSAB provide an adequate on-going test of inter-element correction (IEC) factors. These standards are referred to the Spectra Interference Check (SIC) in EPA Method 200.7
- 2.8.1 ICSA – A solution containing only the interfering analytes at high concentrations.
- 2.8.2 ICSAB – A solution containing interferents plus other method analytes at the level of concern, which corresponds to the project specific action limits.
- 2.8 Water Sample - For the purpose of this method, a sample taken from one of the following sources: drinking water, surface water, ground water, storm water, industrial or domestic wastewater.
- 2.9 Preparation Batch - For method 6010B/C/D/ EPA 200.7 (WW only): A group of samples (not to exceed twenty) of a similar matrix, which have been digested at the same time using the same digestion process and have all necessary QC associated with them. For method 200.7 (DW only): A group of samples (not to exceed ten) of similar matrix, which have been digested at the same time using the same digestion process and have all necessary associated QC.
- 2.10 Analytical batch - A group of samples that are analyzed in the same sequence with all appropriate preparation and analytical QC.

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- 2.11 Inter-element correction (IEC) coefficient - analyte concentration equivalent arising from a given interferent's concentration.
- 2.12 Serial Dilution - a dilution and reanalysis of a field sample that is performed once per batch of samples. One sample is diluted 5X and reanalyzed.
- 2.13 Post Spike – A second aliquot of a field sample that is spiked with known concentrations of target analytes and analyzed to assess recovery of the spike. A post spike must be analyzed when the MS and/or MSD fail due to a suspected matrix effect. One sample is spiked after digestion and analyzed per batch.
- 2.14 Lower Limit of Quantitation (LLOQ) - A term associated with analysis per the requirements of Method 6010D. The lowest point of quantitation which, in most cases, is the lowest concentration in the calibration curve.
- 2.15 See the current Quality Assurance Manual for other definitions associated with terms found in this document.

3.0 HEALTH AND SAFETY

- 3.1 The toxicity or carcinogenicity of each reagent used in the laboratory has not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds should be as low as reasonably achievable. A reference file of safety data sheets (SDSs) are made available on Pace National's intranet to all personnel. Use hazardous reagents in a fume hood whenever possible and if eye or skin contact occurs, flush with large volumes of water. Always wear safety glasses or a shield for eye protection, protective clothing and observe proper mixing protocols.
- 3.2 The acidification of samples containing reactive materials may result in the release of toxic gases, such as cyanides or sulfides. Acidification of samples should be done in a fume hood.
- 3.3 All personnel handling environmental samples known to contain or to have been in contact with human waste should be immunized against known disease causative agents.
- 3.4 All ICP instruments provide protection from ultraviolet light emission. These shielding screens cannot be removed.

4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

- 4.1 All samples must have been collected using a sampling plan that addresses the considerations of this method.
- 4.2 Samples submitted for analysis that do not meet the requirements contained within this section must be addressed before performing the logging process within the laboratory. In some cases, exceeding the appropriate preservation and storage criteria can cause significant bias in the resulting data. Clients may need to resubmit samples where the conditions during shipment cause uncertainty regarding sample integrity. If samples do not meet the requirements for preservation, sampling, shipment and storage and the

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client approves the completion of the analytical process, sample results can be qualified per the Pace National SOP #030201, *Data Handling and Reporting*.

- 4.3 Prior to the collection of an aqueous sample, consideration must be given to the type of data required, (i.e., dissolved or total recoverable), so that appropriate preservation and pre-treatment steps can be taken. The pH of all aqueous samples must be assessed immediately prior to sample digestion or "direct analysis" to ensure the sample has been properly preserved. If the field sample is properly preserved, the sample can be held up to 6 months prior to analysis.
- 4.4 For the determination of dissolved elements, the sample must be filtered through a 0.45µm pore diameter membrane filter to remove the suspended elements or particles. This filtration must take place at the time of collection or as soon thereafter as practically possible. Glass or plastic filtering apparatus are recommended to avoid possible contamination. Only plastic apparatus must be used when the determinations of boron and silica are critical. Use a portion of the filtered sample to rinse the filter flask, discard this portion and collect the required volume of filtrate. Acidify the filtrate with (1:1) nitric acid: water immediately following filtration to pH < 2.
- 4.5 For the determination of total recoverable elements in aqueous samples, samples must not be filtered, but acidified with (1:1) nitric acid: water to pH <2. Preservation may be done at the time of collection; however, to avoid the hazards of strong acid use in the field, possible transport restrictions, or possible contamination, it is recommended that the samples be returned to the laboratory within two weeks of collection and acid preserved upon receipt in the laboratory. Following acidification, the sample must be mixed and equilibrated for 24 hours. The pH is verified at <2 prior to withdrawing an aliquot for acid digestion or "direct analysis". If, for reasons such as high alkalinity, the sample pH is verified to be >2, more acid must be added and the sample equilibrated for another sixteen hours until verified to be pH <2.
- 4.6 Solid samples require no preservation prior to analysis. Solid samples can be held up to six months from the time of sample collection until preparation and analysis.
- 4.7 For aqueous samples, a field blank must be prepared and analyzed as required by the data user. Use the same container and preservative as is used in field sample collection. The sample holding time is 6 months from the date and time of collection until analysis. Samples are preserved to pH <2 with nitric acid.

5.0 INTERFERENCES

- 5.1 Spectral interferences are caused by background emission from continuous or recombination phenomena, stray light from the line emission of high concentration elements, overlap of a spectral line from another element, or unresolved overlap of molecular band spectra.
- 5.1.1 Subtracting the background emission determined by measurement(s) adjacent to the analyte wavelength peak can usually compensate for background emission and stray light. The location(s) selected for the measurement of background

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intensity is determined by the complexity of the spectrum adjacent to the wavelength peak. The location(s) used for routine measurement must be free of off-line spectral interference (inter-element or molecular) or adequately corrected to reflect the same change in background intensity as occurs at the wavelength peak. Changes in background correction must be saved in the instrument method. Background correction can be established by scanning the following three solutions: 1) blank (same as calibration blank); 2) solution, containing analytes at significant concentration to raise a signal above background signal (CCV solution may be used) at mid-range of the curve; 3) solution(s) containing most common interfering elements at high concentration and other interferents as well (ICSAB solution may be used).

- 5.1.2 Spectral overlaps can be compensated for by equations that correct for inter-element contributions, which involve measuring the interfering elements. When operative and uncorrected, these interferences produce false-positive determinations and are reported as analyte concentrations. Users may apply inter-element correction factors determined on their instruments within tested concentration ranges to compensate (offline or online) for the effects of interfering elements. Consult the method for specific identified interferences.
- 5.1.3 When inter-element corrections are applied, there is a need to verify their accuracy by analyzing spectral interference check solutions. The IEC's are established by analyzing a solution of the interfering element at a high concentration within the LDR limit, measuring the analyte concentration equivalents arising from the interfering element, calculating the interference factor as analyte reading in mg/L, then dividing by the interfering element concentration. The IEC's are changed in the stored ICP instrument method. Inter-element corrections vary for the same emission line among instruments because of differences in resolution, as determined by the grating plus the entrance and exit slit widths, and by the order of dispersion. Inter-element corrections also vary depending upon the choice of background correction points. Selecting a background correction point where an interfering emission line may appear should be avoided. Inter-element corrections that constitute a major portion of an emission signal may not yield accurate data. Users must not forget that some samples might contain uncommon elements that could contribute spectral interferences.
- 5.1.4 Interference effects must be evaluated for each individual instrument. For each instrument, intensities vary not only with optical resolution but also with operating conditions (such as power, viewing height and argon flow rate). To determine the appropriate location for offline background correction, the user must scan the area on either side of the peak adjacent to the wavelength and record the apparent emission intensity from all other method analytes. The location selected for background correction must be either free from offline inter-element spectral interference or a computer routine must be used for their automatic correction on all determinations.

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- 5.2 Physical interferences are effects associated with the sample nebulization and transport processes. Changes in viscosity and surface tension can cause significant inaccuracies, especially in samples containing high dissolved solids or high acid concentrations. If physical interferences are present, they must be reduced by such means as using a high-solids nebulizer, diluting the sample, using a peristaltic pump, or using an appropriate internal standard element. Another problem that can occur with high dissolved solids is salt buildup at the tip of the nebulizer, which affects aerosol flow rate and causes instrumental drift. This can be controlled using a high-solids nebulizer, wetting the argon prior to nebulization, using a tip washer, or diluting the sample. Also, it has been reported that better control of the argon flow rates, especially for the nebulizer, improves instrument stability and precision. This is accomplished with the use of mass flow controllers.
- 5.3 Chemical interferences include molecular-compound formation, ionization effects, and solute-vaporization effects. Normally, these effects are not significant with the ICP-AES technique. If observed, they can be minimized by careful selection of operating conditions (such as incident power and observation height), by buffering of the sample, by matrix matching, and by standards addition procedures. Chemical interferences are highly dependent on matrix type.
- 5.4 Memory interferences result when analytes in a previous sample contribute to the signals measured in a new sample. Memory effects can result from sample deposition on the uptake tubing to the nebulizer and from the buildup of sample material in the plasma torch and spray chamber. The site where these effects occur is dependent on the element and can be minimized by flushing the system with a rinse blank between samples. The possibility of memory interferences must be recognized within an analytical run and suitable rinse times must be used to reduce them.
- 5.5 Linear Dynamic Range (LDR) study is performed by analyzing a solution of each element at maximal concentration unless the result falls outside 10% RPD. The highest calibration standard for each analyte cannot be greater than the LDR for that analyte. If an interferent is found greater than the LDR and an IEC factor is established between the interferent and analyte of interest, the sample must be diluted for proper correction of inter-element interferences. Instrument methods with different calibration standard concentrations require separate LDR studies.
- 5.6 Background correction is performed as needed and LDR and IEC studies are completed as required by each published analytical method and whenever significant changes to instrumentation are made. Background, or blank matrix, subtraction is not performed for environmental samples.
- 6.0 EQUIPMENT AND SUPPLIES
- 6.1 Inductively coupled plasma emission spectrometer:
- 6.1.1 Perkin Elmer Model 5300 or Thermo Model 7000 series ICP, or equivalent, with background correction and computer control.
- 6.1.2 Cetac Autosampler or ESI autosampler

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- 6.1.3 Argon gas supply - High purity grade (99.99%). When analyses are conducted frequently, liquid argon is more economical and requires less frequent replacement of tanks than compressed argon in conventional cylinders.
- 6.2 Narrow-mouth storage bottles, FEP (fluorinated ethylene propylene) with screw closure, 125mL to 1L capacities.
- 6.3 One-piece stem FEP wash bottle with screw closure, 125mL capacity.
- 6.4 Adjustable pipettes (Eppendorf or equivalent), ranges from 2 μ L to 5000 μ L.
- 6.5 Class A volumetric flasks for standards preparations.
- 6.6 Polypropylene (PP) conical tubes.
- 6.7 Peristaltic pump.
- 7.0 REAGENTS AND STANDARDS
 - 7.1 All reagents and standards must be recorded in the appropriate preparation log and assigned a unique number. See Pace National SOP #030230, *Standard Logger – Tree Operation*. Additional information regarding reagent preparation can be found in the Standards Logger (Tree) digital archive system. All spiking solutions and surrogate standard solutions should be replaced at least every 6 months or sooner if a problem is detected unless otherwise noted.
 - 7.2 Hydrochloric acid, concentrated (sp. gr. 1.19) - HCl.
 - 7.2.1 Hydrochloric acid (1+1) - Add 500mL concentrated HCl to 400mL reagent water and dilute to 1L with reagent water.
 - 7.3 Nitric acid, concentrated (sp. gr. 1.41) - HNO₃.
 - 7.3.1 Nitric acid (1+1) - Add 500mL concentrated HNO₃ to 400mL reagent water and dilute to 1L with reagent water.
 - 7.4 Reagent water. All references to water in this method refer to ASTM Type I grade water.
 - 7.5 Blanks - Three types of blanks are required for ICP-AES analysis.
 - 7.5.1 The calibration blank is used to establish the baseline for the instrument prior to the analysis of the analytical curve. The calibration blank is prepared by acidifying reagent water to the same acid concentration as used for the standards.

NOTE: The calibration blank must be stored in a FEP bottle to minimize leaching from other container materials that can cause an elevation in the target analytes leached causing an inherent bias in the calibration and quantitation of field samples when baselines are established prior to calibration of the ICP-AES.

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7.5.1.1 Following calibration, the Initial Calibration Blank (ICB) is analyzed prior to field sample analyses. A Continuing Calibration Blank (CCB) is analyzed following the CCV after every ten samples and at the end of the analytical sequence to verify on-going acceptable instrument conditions.

7.5.2 The method blank is used to assess possible contamination from the sample preparation procedure. The method blank must contain all the reagents in the same volumes as used in sample preparation. The method blank must be prepared in the same manner as the samples including sample digestion, when applicable

7.5.3 The rinse blank is prepared by acidifying reagent water to the same concentrations as the acids as used in the calibration blank. This solution is stored in a convenient manner. The rinse blank is used for equipment "wash out" to flush the sample delivery system and eliminate memory effects (carryover) from previous samples or standards.

7.6 Mixed Calibration Standard Solutions – Environmental Express Custom Mixes, or equivalent, is used to make the following calibration solutions. All standards are prepared in Class A volumetric flasks using adjustable pipettes. The final acid concentration is matrix matched to digested field sample concentrations. Use the calculation in section 9.14 to calculate the volume of the stock needed to produce each standard.

Note: **Environmental Express Custom Mix #HP 6373-1L** contains the following elements and concentrations in **mg/L**: Ag-20, Al-200, As-40, Ba-10, Be-4.0, B-40, Cd-10, Ca-1000, Cr-20, Co-20, Cu-40, Fe-200, Pb-10, Li-30, Mg-200, Mn-20, Mo-10, Ni-40, K-1000, Sb-40, Se-40, Si- 40, Sn-40, Na-1000, Sr-20, Ti-20, Tl-40, V-20, Zn-60, S-100.

Concentration of Target Analytes in Calibration Standards in mg/L.

Analyte	STD 1	STD 2	STD 3	STD 4	STD 5	STD 6	STD 7
Silver	0.005	0.5	1.0	2.0			
Aluminum	0.2				10	250	500
Arsenic	0.1	0.5	1.0				
Boron	0.05		1.0	2.0			
Barium	0.005	0.5	1.0	2.0	10		
Beryllium	0.002	0.5	1.0	2.0			
Calcium	1.0		1.0	2.0	10	250	500
Cadmium	0.002	0.5	1.0				
Cobalt	0.01	0.5	1.0	2.0			
Chromium	0.01	0.5	1.0	2.0			
Copper	0.01	0.5	1.0	2.0			
Iron	0.10	0.5	1.0	2.0	10	100	200
Potassium	1.0				10	50	100

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Analyte	STD 1	STD 2	STD 3	STD 4	STD 5	STD 6	STD 7
Lithium	0.015	0.5	1.0				
Magnesium	1.0				10	250	500
Manganese	0.01	0.5	1.0	2.0			
Molybdenum	0.005	0.5	1.0	2.0			
Sodium	1.0		1.0	2.0	10	250	500
Nickel	0.01	0.5	1.0	2.0			
Lead	0.005	0.5	1.0				
Antimony	1.0	0.5	1.0	2.0			
Selenium	0.01	0.5	1.0				
Silicon	0.01	0.5	1.0	2.0	10		
Strontium	0.05	0.5	1.0	2.0			
Sulfur	0.05		1.0	2.0	10	50	100
Tin	0.01	0.5	1.0	2.0			
Thallium	0.05	0.5	1.0				
Vanadium	0.01	0.5	1.0	2.0			
Zinc	0.02	0.5	1.0	2.0			
Titanium	0.02	0.5	1.0	2.0			

NOTE: If the addition of silver to the recommended mixed-acid calibration standard results in an initial precipitation, add 15mL of reagent water and warm the flask until the solution clears. For this acid combination, the silver concentration should be limited to 0.5mg/L.

7.7 Initial Calibration Verification (ICV) – The ICV is an analytical standard solution from a second source different from the calibration and CCV standards. The ICV is prepared at a mid-range concentration within the linear working range of the instrument. The ICV must have the same acid matrix as the Calibration Standards, CCV, blanks and the field sample digestates.

The ICV solutions are purchased from High Purity Standard SP2762-3227HPZ-A, High Purity Standard SP2762-3227HPZ-B, and Ultra Scientific ICP-016, or equivalents.

Custom Mix # SM-2552-008 Solution A, SM-2552-008 Solution B-1L (stock) contains the following element and concentrations in µg/mL: Ag-(B) 50, Al- 500, Sb- 50, As- 50, Ba- 50, Be- 50, B- 50, Cd- 50, Ca- 500, Cr- 50, Co- 50, Cu- 50, Fe- 500, Pb- 50, Li- 50, Mg- 500, Mo(B)- 50, Mn- 50, Na- 500, Ni- 50, K- 500, Se- 50, Si(B)- 50, Sn(B)- 50, (listed 2x), Sr- 20, Tl- 50, Ti(B)- 50, V- 50, Zn- 50., S(B)-500

This solution is prepared, by spiking 10mL of the custom stock solution A and 10mL of the custom stock solution B into a 100mL volumetric flask, then diluting to 100mL using 10% Nitric Acid.

All analytes are present in the mid-level ICV solution at the following concentrations (mg/L):

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Analyte	Concentration
Silver	1
Aluminum	10.0
Arsenic	1.0
Boron	1.0
Barium	1.0
Beryllium	1.0
Calcium	10.0
Cadmium	1.0
Cobalt	1.0
Chromium	1.0
Copper	1.0
Iron	10.
Potassium	10.
Lithium	1.0
Magnesium	10.

Analyte	Concentration
Manganese	1.0
Molybdenum	1.0
Sodium	10.
Nickel	1.0
Lead	1.0
Antimony	1.0
Selenium	1.0
Silicon	1.0
Strontium	0.4
Tin	1.0
Thallium	1.0
Vanadium	1.0
Zinc	1.0
Titanium	1.0
Sulfur	10.0

- 7.8 Continuing Calibration Verification (CCV) – The CCV is the mid-range calibration standard prepared from the same source as the initial calibration curve. The CCV is used to verify the regression of the initial calibration of the instrument and must be repeated following every ten samples and at the conclusion of the sequence. EPA Method 200.7 refers to this standard as the Instrument Performance Check (IPC) standard.

All analytes are present in the mid-level CCV solution at the following concentrations (mg/L):

Analyte	Concentration
Silver	.5
Aluminum	10.
Arsenic	1.0
Boron	1.0
Barium	0.50
Beryllium	0.20
Calcium	50.0
Cadmium	0.50
Cobalt	1.0
Chromium	1.0
Copper	1.0
Iron	10.
Potassium	50.
Lithium	1.0
Magnesium	10.

Analyte	Concentration
Manganese	1.0
Molybdenum	.25
Sodium	50.
Nickel	1.0
Lead	0.50
Antimony	.5
Selenium	1.0
Silicon	2.0
Strontium	1.0
Tin	.5
Thallium	1.0
Vanadium	1.0
Zinc	1.0
Titanium	1.0
Sulfur*	5

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- 7.9 Low Level Initial/Continuing Calibration Verification for EPA 6010C (ICVLL/CCVLL) – The ICVLL/CCVLL HP6590-500. See section 7.6. The ICVLL/CCVLL is prepared at a low concentration within the linear working range of the instrument and defines the lowest level of quantitation/reporting. The ICVLL/CCVLL must have the same acid matrix as the calibration standards, CCV, blanks and the field samples.

The working standard for the ICVLL/CCVLL is prepared by diluting 50mL of the solution(s) above into 1000mL of DI water that has been acidified to 10% Nitric Acid solution to matrix match the solution with the calibration standards and samples.

The concentration of the low-level ICVLL/CCVLL solution is listed in the table below:

Analyte	Concentration (mg/L)
Silver	0.005
Aluminum	0.2
Arsenic	0.01
Boron	0.05
Barium	0.005
Beryllium	0.002
Calcium	1
Cadmium	0.002
Cobalt	0.01
Chromium	0.01
Copper	0.01
Iron	0.10
Potassium	1
Lithium	0.015
Magnesium	1

Analyte	Concentration (mg/L)
Manganese	0.01
Molybdenum	0.005
Sodium	1
Nickel	0.01
Lead	0.005
Antimony	0.01
Selenium	0.01
Silicon	0.05
Strontium	0.01
Tin	0.02
Thallium	0.01
Vanadium	0.02
Zinc	0.02
Titanium	0.05
Sulfur	1.00

- 7.10 Interference Check Solutions (ICSA and ICSAB) – The ICSA and ICSAB are prepared to contain known concentrations of interfering elements that provides a test of the correction factors. The ICSA solution contains the interfering elements at a high concentration and the ICSAB contains both the interfering analytes at a high concentration and the analytes of interest at 0.5 to 1.0mg/L. EPA Method 200.7 refers to this standard as the Spectral Interference Check (SIC) standard.

7.10.1 The ICSA solution contains 5000mg/L of each Al, Ca, Mg and 2000 mg/L Fe. This solution is prepared from a 1:10 dilution of purchased stock from Environmental Express, Catalog No. ICL500-6.

7.10.2 The ICSAB solution contains all the components at the same concentrations of the ICSA and other target analytes of interest spiked. The solution is prepared from a 1:10 dilution of the stock from Section 7.10.1 (Environmental Express,

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Catalog No. HP 2739-1L) and a 1:100 dilution of the purchased stock from Environmental Express, Catalog No. HP2739-1L.

In the working solution, silver, boron, cadmium, nickel, lead, silica, and zinc are present at 1.0mg/L. All other analytes (arsenic, barium, beryllium, cobalt, chromium, copper, manganese, molybdenum, antimony, selenium, tin, thallium, vanadium and titanium are present at 0.5mg/L.

7.10.3 Method 6010D refers to the use of spectral interference check (SIC) solutions.

7.10.3.1 Individual element SIC solutions - Individual element SIC solutions are used to evaluate possible spectral interferences and to set interelement corrections if necessary. A solution of each element is prepared at the highest concentration in the linear range likely to be observed in samples. The acid strength should be equivalent to that of the calibration standards. See Section 10.11.1.1 for use of the individual element SIC solutions. SIC solutions should be tested to verify that they are not contaminated with elements of interest. The verification of purity can be done by analysis using an alternate technology, such as ICP-MS. For ICP-OES instruments with solid-state detectors, the verification might also be done by examining alternate wavelengths. If the SIC solutions are purchased ready-made, the vendor should provide details of any contaminants. In some cases it may not be possible to obtain solutions completely free of contaminants, in which case the known, verified concentration can be subtracted from the instrument result before assessing any interferences.

7.10.3.2 Mixed element SIC solution - The mixed element SIC solution is used as an ongoing daily check of freedom from spectral interferences. The mixed element SIC solution contains the following elements and is made up in an acid solution equivalent to the calibration standards. See Section 10.11.1.2 for use of the mixed element SIC solution. As for the single element solutions described in 7.10.3.1, known and documented contaminants are subtracted from the observed values in the mixed element SIC check. Mixed element SIC solution: Aluminum, 500mg/L; Calcium, 500mg/L; Iron, 200mg/L; Magnesium, 500mg/L.

7.11 The aqueous laboratory control standard (LCSW) is purchased with all analytes at a concentration of 100µg/mL except calcium, magnesium, potassium, and sodium, which are at 1000µg/mL. The LCSW is purchased from Ultra Scientific, Number ICUS-3490 or equivalent. For 6010/200.7 .45mL of ICUS-3490 is used for spiking LCS's for waters and .5mLs for soil LCS's.

7.12 The aqueous and solid matrix spike and matrix spike duplicate are prepared from the purchased standard specified in Section 7.11.

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7.13 Internal Standard (10,000µg/mL) – Yttrium is used as an internal standard. The yttrium is prepared from a 10.00µg/mL purchased standard (Environmental Express or equivalent) and diluted to a final concentration of 5ppm. The internal standard response is used to measure the relative responses of other method analytes in each sample. Indium (1000 ug/ml) is used as an internal standard purchased from Environmental Express and diluted to a final volume of 30 ppm. See the acceptance criteria in Section 10.14 of this procedure.

7.14 Lower limit of quantitation check sample when analyzing samples by EPA method 6010C (LLOQ): The sample should be analyzed after calibration curve to establish the lower laboratory reporting limits and on an as needed basis to demonstrate the desired detection capability. Ideally, this check sample and the low-level calibration verification standard will be prepared at the same concentrations with the only difference being the LLOQ sample is carried through the entire preparation and analytical procedure. Lower limits of quantitation are verified when all analytes in the LLOQ sample are detected within historical laboratory accuracy limits of their true value. This check should be used to both establish and confirm the lowest quantitation limit.

The lower limits of quantitation determination using reagent water represents a best case situation and does not represent possible matrix effects of real-world samples. For the application of lower limits of quantitation on a project-specific basis with established data quality objectives, low-level matrix specific spike studies may provide data users with a more reliable indication of the actual method sensitivity and minimum detection capabilities.

8.0 PROCEDURE

8.1 Sample Analysis

8.1.1 **Initializing the Instrument:** Prior to daily calibration of the instrument, inspect the sample introduction system including the nebulizer, torch, injector tube for salt deposits, dirt, and debris that would restrict solution flow and affect instrument performance.

8.1.1.1 Replace the uptake tubing daily.

8.1.1.2 If any of the sample introduction parts appear soiled, first remove the part from the instrument by following the maintenance procedure in the instrument manual. Once removed, attempt to clean the part with a dilute solution of 5% nitric acid. Cleaning may be performed using a cotton swab or by submersing the part in the acid solution for no longer than 5 minutes. If cleaning is successful, dry the part using compressed air or argon and replace it in the instrument. If cleaning does not adequately remove the residue, the part must be replaced with a new one in accordance with the manufacturer's directions. Replacement parts are kept in the cabinet in the instrument lab.

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- 8.1.2 **Instrument Stability:** The instrument must be allowed to become thermally stable before calibration and analyses. This usually requires at least 30 minutes of operation.
- 8.1.3 **Instrument Calibration:** For initial and daily operation, calibrate the instrument according to the instrument manufacturer's recommended procedures using mixed calibration standard solutions and the calibration blank. A peristaltic pump is used to introduce all solutions, samples, and the internal standard to the nebulizer. To allow adequate time for equilibrium to be reached in the plasma, aspirate all solutions for at least 30 seconds after the solution reaches the plasma before obtaining the sample analyte response.
- 8.1.3.1 Use the average value from three replicate analyte responses per sample to be correlated to the overall analyte concentration in the solution being sampled. Flush the system with the rinse blank for a minimum of 60 seconds between each standard.
- 8.1.3.2 The calibration regression is generated using first order linear regression of a calibration blank and three calibration standards where each element is present. The blank is included as a point in the calibration curve to determine the baseline correction needed for the instrument to effectively quantitate target analyte concentrations.
- 8.1.3.3 Calibration acceptance criteria are described in section 10.4.
- 8.1.4 **Internal Standard:** All standards/samples/QC etc. contain yttrium and indium as the internal standards. The instrument adds the internal standards automatically. The instrument injects a constant volume into each solution being analyzed (i.e. standard, blank, field sample, LCS/LCSD/MS/MSD/DUP) and monitors the intensity at the sample level. An internal standard is the chosen alternative to the method of standard additions (MSA). If signal variation results from the sample introduction system (samples of different viscosity, matrix constitution), all the elements are corrected in the same way by an internal standard. If variation results from a variation of the energy transfer, the internal standard most accurately corrects elements of similar energy. Internal standard acceptance criteria are described in section 10.14.
- 8.1.5 **Calibration Accuracy:** Verify the acceptable initial calibration of the instrument using a standard source that is either an independent lot or entirely different manufacturer to ensure calibration accuracy. After calibrating and rinsing the instrument, analyze the ICPV and, if analyzing samples using EPA 6010C, analyze the ICPVLL standards. These standards are prepared as directed in section 7.7 and 7.9. Acceptance criteria are described in section 10.5.
- 8.1.6 **On-going Calibration Stability:** Verify the acceptable on-going instrument calibration by analyzing appropriate check standards during the sequence. Instrument calibration acceptability is demonstrated after every 10 samples and at

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the end of the analytical run using the CCV, CCVLL (if analyzing EPA 6010C samples), and CCB that must meet the criteria described in sections 10.6 & 10.14.

- 8.1.7 **Accurate Background Corrections:** The interference check standards (ICSA and ICSAB) are used to verify the inter-element and background correction factors at the beginning and end of an analytical sequence or twice during every 8-hour work shift, whichever is more frequent. The interference check standards must meet the criteria found in section 10.10.
- 8.1.8 An Initial Calibration Blank (Section 7.5.1) is analyzed before sample analysis is initiated to verify the cleanliness of the analytical system. Acceptance criteria are described in section 10.14.
- 8.1.9 **Field Sample Analysis:** After completion of the above calibration requirements, samples must be analyzed in the same operational manner used in the calibration routine with the rinse blank also being used between all sample solutions, method blanks, Laboratory Control Standards, matrix spike, matrix spike duplicates, and check solutions.
- 8.1.10 **Dilutions:** If a sample analyte concentration is quantitated within 90% or greater of the upper limit of the analyte's determined Linear Dynamic Range (LDR), see section 10.4.2 for further guidance.

9.0 DATA ANALYSIS AND CALCULATIONS

- 9.1 Sample data should be reported in units of mg/L for aqueous samples and mg/kg dry weight corrected for solid samples.
- 9.2 For dissolved aqueous analytes, report the data generated directly from the instrument with compensation for sample dilution. Never report analyte concentrations below the MDL and if reporting between the MDL and the routine RL, results should be qualified with the appropriate indicator for estimated target analyte concentrations.
- 9.3 For total recoverable aqueous analytes, multiply solution analyte concentrations by the dilution factor 0.5 when a 100mL aliquot is used to produce the 50mL final digestate volume, and report data. If a different aliquot volume other than 100mL is used for sample preparation, adjust the dilution factor accordingly. Account for any additional dilution of the prepared sample digestate required to complete the determination of any analytes exceeding 90% or greater of the LDR upper limit. Never report analyte concentrations below the MDL and if reporting between the MDL and the routine RL, results should be qualified with the appropriate indicator for estimated target analyte concentration. Routine reporting limits are adjusted for any dilution required by the sample analysis.
- 9.4 Results are reported to Three significant figures by the laboratory LIMS. Analyte concentrations for solids data should be rounded in a similar manner following dry weight corrections.

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- 9.5 For total recoverable analytes in solid samples, calculate the target analyte concentration using the equation below and do not report analyte data below the estimated solids RL or an adjusted RL based on additional dilutions required to complete the analysis:

$$\text{Sample Conc. (mg/Kg) = dry-weight basis} = \frac{C \times V \times D}{W}$$

where: C = Concentration in extract (mg/L)
V = Volume of extract (L, 100 mL = 0. 1L)
D = Dilution factor (undiluted = 1)
W = Weight in Kg of sample aliquot extracted (g x 0.001 = Kg)

- 9.6 Soil samples are routinely reported on a dry weight basis. Soil samples must be processed using the Pace National SOP #340326, *Percent Moisture*. After a dry weight for each sample has been obtained, the calculations are performed automatically by the laboratory LIMS as follows:

$$\% \text{ solids (S)} = \frac{DW}{WW} \times 100$$

where: DW = Sample weight (g) dried
WW = Sample weight (g) before drying

- 9.7 Hardness calculations:

Total Hardness, mg equivalent CaCO₃/L = 2.497 [Ca, mg/L] + 4.118 [Mg, mg/L]

Calcium Hardness = 2.497 [Ca, mg/L]

Magnesium Hardness = 4.118 [Mg, mg/L]

- 9.8 To calculate the silica concentration from silicon analysis:

$$\text{Silica (mg/L)} = 2.14 \times [\text{Silicon, mg/L}]$$

- 9.9 Formula needed to calculate dilution of stock standards of known concentration to a known final volume, using the basic chemistry formula, $C_1 \times V_1 = C_2 \times V_2$:

$$V_{\text{stock}} = V_{\text{std}} \times C_{\text{std}} / C_{\text{stock}}$$

where: V_{stock} = volume of stock standard required (mL)
 V_{std} = final volume of diluted standard required (mL)
 C_{stock} = concentration of stock standard required (ug/mL or ug/L)
 C_{std} = final concentration of diluted standard required (ug/mL or ug/L)

NOTE: Be sure to maintain consistent units for both concentration and volume during the use of the calculation and keep in mind that (1ug/mL = 1mg/L=1000ug/L, 1L=1000mL=1000000uL, and 1mL=1000uL)

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9.10 Percent Relative Intensity (%RI) for internal standard assessment (ISTD):

$$\%RI = \text{Intensity of ISTD}_{\text{sample}} / \text{Intensity of ISTD}_{\text{CalBlk}} * 100\%$$

9.11 Relative Standard Error (RSE – expressed as a percentage)

$$RSE = 100 \times \sqrt{\frac{\sum_{i=1}^n \left[\frac{x'_i - x_i}{x_i} \right]^2}{(n - p)}}$$

where:

- x'_i = Measured amount of analyte at the calibration level i , in mass or concentration units
- x_i = True amount of analyte at calibration level i , in mass or concentration units
- p = Number of terms in the fitting equation (average – 1, linear = 2, quadratic = 3)

9.12 See the current Quality Assurance Manual for other equations associated with common calculations.

10.0 QUALITY CONTROL AND METHOD PERFORMANCE

10.1 All analysts must meet the qualifications specified in SOP #030205, *Technical Training and Personnel Qualifications*, before approval to perform this method. Analysts must complete an initial demonstration of proficiency before being approved to perform this method. Continuing proficiency must be demonstrated using proficiency testing, laboratory control sample analysis and/or MDL studies. Method performance is assessed per analyst. Updated method performance records are filed and stored in a central location within the department.

10.1.1 Prior to using Method 6010D for quantitation of samples, an initial demonstration of performance packet must be completed. This packet must document:

- The selection criteria for background correction points
- Analytical dynamic ranges including the applicable equations and upper limits of ranges
- IDLs and Method LLOQs
- The determination and verification of interelement correction equations or other routines for correcting spectral interferences. These data must be generated using the same instrument, operating conditions, and calibration routine to be used for sample analysis. The data must be kept on file and available for review by the data user or auditor.

10.2 Use Prep Data to record batch order and standards/reagents used during analysis. See SOP #030201, *Data Handling and Reporting*.

10.3 Batch Analyses:

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10.3.1 Environmental Preparation Batches: Preparation batches are defined as sets of 1 - 20 samples as defined in Chapter 1 of SW-846 and in section 9.3.1 of EPA 200.7. Preparation batch analysis must include the following: 1 Method Blank, 1 Laboratory Control Sample (LCS), 1 Laboratory Control Sample Duplicate (LCSD), 1 Serial Dilution, 1 Sample Post Digestion Spike, 1 Matrix Spike/Spike Duplicate (MS/MSD) pair. All batch information is maintained in Prep Data computer program.

10.3.2 Analytical Batches: Analytical batches are defined as a sequence of samples analyzed concurrently using the same calibrated instrument. Analytical batches include the QC samples produced in the Preparation Batches, in addition to: 1 Serial Dilution, 1 Sample Post Digestion Spike, 1 Initial Calibration Verification (ICV) following initial calibration, 1 Initial Calibration Verification-Low Level (ICVLL) following initial calibration (when analyzing EPA 6010C only) 1 Initial Calibration Blank following the ICVLL, 1 Continuing Calibration Verification (CCV) following each 10 samples and at the conclusion of the sequence, 1 Continuing Calibration Verification-Low Level (CCVLL) following each 10 samples and at the conclusion of the sequence (when analyzing EPA 6010C only), 1 Continuing Calibration Blank (CCB) following each CCV/CCVLL pair, 1 Interference Check Sample A (ICSA) and 1 Interference Check Sample AB (ICSAB) following each initial calibration and at the end of the sequence or at least twice per each 8 hour shift. All batch information is maintained in Prep Data computer program.

10.4 Supporting Analytical Studies:

10.4.1 Instrument Detection Limits (IDL) Studies - IDLs in µg/L can be determined as the mean of the calibration blank results plus three times the standard deviation of 10 replicate analyses of the solution. Use zero for the mean if the mean is determined to be a negative value.

IDLs must be verified quarterly^{14,13} or when major instrumentation change occurs.

10.4.2 Linear Dynamic Range (LDR) Studies – Linear dynamic ranges are established for each instrument to allow for quantitation above the highest level of calibration without qualification. ICP instruments are known to remain linear at high levels, but each upper limit of linearity is based on the target analyte being measured and the routine instrument operating conditions.

To perform a linear dynamic range study, the instrument must be calibrated normally as used with client field samples. The LDR is determined by the analysis of a minimum of three, but preferably five, different increasing concentrations of standards containing each target analyte across a range. One concentration should be near the expected upper linear range for each analyte. The highest concentration, where the instrument calibration remains linear, is determined when the observed concentration of the increasing standards is no

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more than 10% below the expected concentration of the analyte. If more than a 10% deviation exists, the instrument is proven to no longer be linear at that value for that analyte. The upper linear range is therefore the next lower concentration of standards used in the determination. Samples quantitated above that upper determined LDR require dilution to quantitate within the proven linear range of the instrument.

LDR studies must be verified semi-annually^{14,1} or when major instrumentation change occurs.

Method 6010D - LDR standards must be ran daily within ten percent of true value, or dilute all samples above the high standard in the curve.

STATE NOTE: For work performed in support of the NC Department of Natural Resources (15A NCAC 02H.0805(a)(7)(I)) for target analytes quantitated by ICP or ICPMS, a series of at least three standards must be analyzed along with each group of samples. The concentrations of these standards must bracket the concentration of the analytes in the field samples analyzed. Samples with target analyte concentrations above the highest level of calibration must be diluted to quantitate analytes within the calibration range. The use of the dynamic linear range studies to validate analyte/instrument calibration linearity must not be used for NC sample analysis

- 10.4.3 Method Detection Limits – See also Pace National SOP #030206, *Method Detection Limits (MDL), Limits of Detection (LOD) and Limits of Quantitation (LOQ)*.

MDL studies are required annually or when instrumentation change occurs. Method detection limit studies are performed on blank matrices most closely matching field sample matrices.

- 10.4.4 Inter-element Correction Factors – All inter-element spectral correction factors must be verified and updated every six months or when major instrumentation change occurs.^{14,1,14.5,}

Criteria for determining an inter-element spectral interference is an apparent positive or negative concentration of an analyte that is outside the 3-sigma control limits of the calibration blank for the analyte. See Attachment II for a listing of potential interfering analytes and their contributions from SW-846 method EPA 6010B. Testing is performed using 100 mg/L single element solutions; however, for analytes such as iron that may be found at high concentration, a more appropriate test would be to use a concentration near the upper analytical range limit.

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Suggested analytes that are known to commonly interfere include: Ag, Al, As, B, Ba, Be, Ca, Cd, Ce, Co, Cr, Cu, Fe, K, Li, Mg, Mn, Mo, Na, Ni, P, Pb, Sb, Se, Si, Sn, Sr, Ti, Tl, V, and Zn.

10.4.5 Proficiency Testing (PT) – See also Pace National SOP #030212, *Proficiency Testing Program*. Proficiency testing is performed in the metals department in support of both environmental and industrial hygiene analyses. Environmental PTs are performed semi-annually for Water Supply (Safe Drinking Water Act), Water Pollution (Clean Water Act), and soils (RCRA) testing.

10.5 Initial Calibration - Run a calibration curve on a daily basis that employs a minimum of a calibration blank and three standards for each target analyte. If the correlation coefficient does not meet the acceptance criteria, see the corrective action guidance listed in Section 11.1.

NOTE: For EPA Methods 200.7 & 6010B/D, the linear regression correlation coefficient for the each analyte in the calibration curve lines must be ≥ 0.995 .

NOTE: For Method 6010D - Relative Standard Error (see Section 9.11) may be used as an alternative to r or r^2 , and should be $< 20\%$. If a multipoint calibration is used the low standard must be at or below the LLOQ. Inversely weighted linear regressions are recommended in order to minimize curve fitting errors at the low end of the calibration curve.

NOTE: For EPA Method 6010C, the regression correlation coefficient must be ≥ 0.998 .

CLIENT NOTE: For Marathon sample analysis, simple linear curve fitting is identified in the instrument regression choices will be utilized when possible. When utilizing calibration curve fits identified by PE regression as “linear forced through 0” accuracy within 70-130% must be demonstrated at the reporting limit utilizing the most recent MDL study.

10.6 Initial Calibration Verification (ICV/ICVLL) - Verify the accuracy of the initial instrument standardization by analyzing appropriate check standards following calibration. The routine mid-level ICV must be prepared from a source that is independent of the stock standard used for the preparation of the initial calibration curve. For EPA Method 6010C, a low-level ICV (ICVLL) is also performed as required; however the low level ICV is not required to be from a second source. It may be made from the same stock standard as the calibration standards as long as the initial calibration is verified by a second source in the mid-level ICV.

10.6.1 **EPA Method 6010B/D** - The routine ICV standard recovery results must be $\pm 10\%$ of the true value for EPA method 6010B/D. The RSD must be $< 5\%$ for the triplicate passes of the spectrometer. If the RSD exceeds 5% and/or the recovery exceeds 10%, locate and correct the cause of the problem and do not proceed until this criterion is met. Corrective actions for failures can be found in section 11.2.

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- 10.6.2 **EPA Method 200.7** - The routine ICV standard recovery results must be $\pm 5\%$ of the true value for EPA method 200.7. The RSD must be within 3% for the four replicate passes of the spectrometer. If the RSD exceeds 3% and/or the recovery exceeds 5%, locate and correct the cause of the problem and do not proceed until this criterion is met. Corrective actions for failures can be found in section 11.2.
- 10.6.3 **EPA Method 6010C** - The routine ICV standard recovery results must be $\pm 10\%$ of the true value for EPA methods 6010C. The RSD must be $< 5\%$ for the triplicate passes of the spectrometer. The ICVLL standard recovery results should be within historical laboratory accuracy limits. If the recovery does not meet the criteria for either level of ICV and/or the %RSD is exceeded, locate and correct the cause of the problem and do not proceed until this criterion is met. Corrective actions for failures can be found in section 11.2.
- 10.6.4 **Method 6010D Low-level Readback or Verification** - For a multi-point calibration, the low level standard should quantitate to within 80-120% of the true value. For a single point calibration, a standard from the same source as the calibration standard and at the LLOQ is analyzed and should recover within 80-120% of the true value.
- 10.6.5 **Method 6010D Mid-level Readback or Verification** - For a multi-point calibration, the midlevel standard should quantitate to within 90-110% of the true value. For a single point calibration, a standard from the same source as the calibration standard and at the midpoint of the linear range is analyzed and should recover within 90-110% of the true value.
- 10.7 Continuing Calibration Verification (CCV/CCVLL) - Verify the on-going instrument standardization by analyzing appropriate check standards during the sequence. Verification is achieved by analyzing both a CCV standard and a CCB (instrument blank). Continuing calibration verification standards can be created from either primary or secondary source standards from those used in instrument calibration. Continuing instrument calibration acceptability is demonstrated after every 10 samples and at the end of the analytical run using the CCV that must meet the following criteria per the method being analyzed:
- 10.7.1 **For SW-846 Method 6010B/D** – Continuing calibration verification (CCV) analyzed after every 10 samples and at the conclusion of the sequence must have a recovery within $\pm 10\%$. The RSD must be within 5% for the triplicate passes of the spectrometer.
- 10.7.2 **For EPA Method 200.7** - Continuing calibration verification (CCV) analyzed after every 10 samples and at the conclusion of the sequence must have a recovery within $\pm 10\%$. The RSD must be within 3% for the triplicate passes of the spectrometer.
- 10.7.3 **For SW-846 Method 6010C** - Continuing calibration verification (CCV) analyzed after every 10 samples and at the conclusion of the sequence must have a

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recovery within $\pm 10\%$. The RSD must be within 5% for the triplicate passes of the spectrometer. The CCVLL recovery should be within $\pm 30\%$.

10.8 Method/Calibration/Rinse Blanks:

10.8.1 Method Blank:

10.8.1.1 A method blank is generated for each analytical batch during sample preparation to determine if any contamination is introduced during sample processing. A method blank is routinely a volume of reagent water that is carried through the entire digestion and analysis procedure with the samples.

10.8.1.2 The method blank must not contain analytes >MDL or, in the case of common laboratory contaminants, the concentrations should not exceed the RL. Common laboratory contaminants include: Calcium, Potassium, Magnesium, Zinc, Iron and Sodium. If target analytes are present in the method blank, corrective action must be taken. See section 11.5 for corrective actions.

NOTE: Per DOD QSM, version 5.0, Section 1.7.4.1, DoD/DOE require that method blanks be evaluated to $\frac{1}{2}$ RL (LOQ) for target analytes and RL (LOQ) for common laboratory contaminants. If contaminants are present in the blank above this level, samples must be re-prepared and re-analyzed or reported with appropriate qualification.

NOTE: Method 6010D – The method blank is considered to be acceptable if target analyte concentrations are less than $\frac{1}{2}$ the LLOQ or are less than project-specific requirements.

State Note: For Wisconsin samples, the method blank must not contain analytes more negative than the MDL value. If target analytes are more negative than the MDL, the instrument must be recalibrated or a new LOD study performed.

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10.8.2 Calibration Blank:

10.8.2.1 An initial calibration blank is generated for each analytical sequence using acidified reagent water. The CALBLK analyzed prior to the initial calibration standards is used to establish a baseline for the instrument prior to calibration. Great care is required during this analysis to ensure that the baseline is correctly established prior to the calibration of the instrument and the analysis of field samples. Inaccurate baselines established with contaminated calibration blanks degrade precision and accuracy of the analyses performed by creating biases in target analyte calibration.

If the initial calibration blank is grossly negative for a target analyte, then the quantitation of that target analyte in the calibration standards will be biased high due to over compensation by the instrument. This will lead to low recovery issues with the CCV, ICV, field samples, and batch QC samples. If target analytes are present in the CALBLK leading the instrument to make an over correction of the baseline for these targets, then the calibration curve will be biased high yielding a low bias for those target analytes in the CCV, ICV, field samples, and batch QC.

10.8.2.2 Continuing calibration blank (ICB/CCB) is also analyzed following each initial and continuing calibration verification standard within an instrument sequence to verify instrument stability and system cleanliness. This does not baseline correct the instrument for possible contaminants in the background and must be evaluated to ensure that background corrections are appropriate and consistently applied throughout the sequence.

The ICB must not contain analytes > ½ RL, for CCB must not contain analytes > RL for all 6010 methods or, in the case of common laboratory contaminants, the concentrations should not exceed the RL. Common laboratory contaminants include: Calcium, Potassium, Magnesium, Zinc, Iron and Sodium.

10.8.3 Rinse Blank:

10.8.3.1 A rinse blank is utilized by the ICP to cleanse the system following the intake of each digestate analyzed. No data is obtained during this rinse and no applicable controls are required for this type of blank. This is merely cleansing the lines throughout the analytical system.

10.9 Matrix Spike/Matrix Spike Duplicate (MS/MSD) - A matrix/matrix spike duplicate must be prepared for each matrix for each batch of 10 samples for method 200.7 or 20 samples for method 6010B/C/D, where sufficient sample volume was submitted by the client. Matrix spike and matrix spike duplicate are prepared from a sample aliquot spiked with the known concentration of analytes.

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The matrix spike recoveries must meet the criteria in the table below unless the analyte concentration in the sample is at least 4 times greater than the spike concentration.

Method	Acceptance Criteria	
	Water	Soil
6010B, C, and D	75 – 125%	75 – 125%
200.7	70-130%	NA

Assess that the matrix spike duplicate precision (%RPD) results meet project-established goals acceptance criteria. If no project goals are specified, then results for the RPD must be less than 20%.

10.9.1 For Method 6010D, if less than acceptable bias and precision data are generated for the matrix spike(s), the additional QC protocols in Sections 10.9.1.1 and/or 10.9.1.2 should be performed prior to reporting concentration data for the elements in this method. At a minimum these tests should be performed with each batch of samples prepared/analyzed with corresponding unacceptable data quality results. If matrix interference effects are confirmed, then an alternative test method should be considered or the current test method modified, so that the analysis is not affected by the same interference. The use of a standard-addition analysis procedure may also be used to compensate for this effect.

10.9.1.1 Dilution Test - If the analyte concentration is within the linear range of the instrument and sufficiently high (minimally, a factor of 25 times greater than the LLOQ), an analysis of a 1:5 dilution should agree to within $\pm 20\%$ of the original determination. If not, then a chemical or physical interference effect should be suspected. The matrix spike is often a good choice of sample for the dilution test, since reasonable concentrations of most analytes are present. Elements that fail the dilution test are reported as estimated values.

CAUTION: If spectral overlap is suspected, then the use of computerized compensation, an alternate wavelength, or comparison with an alternate method is recommended.

10.9.1.2 Post-Digestion MS - If a high concentration sample is not available for performing the dilution test, then a post-digestion MS should be performed. The test only needs to be performed for the specific elements that failed original matrix spike limits, and only if the spike concentration added was greater than the concentration determined in the unspiked sample. Following preparation, which may include, but is not limited to, pre-filtration, digestion, dilution and filtration, an aliquot, or dilution thereof, should be obtained from the final aqueous, unspiked-analytical sample, and spiked with a known quantity of target

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elements. The spike addition should be based on the indigenous concentration of each element of interest in the sample. The recovery of the post-digestion MS should fall within a $\pm 20\%$ acceptance range, relative to the known true value, or otherwise within the laboratory-derived acceptance limits. If the post-digestion MS recovery fails to meet the acceptance criteria, the sample results must be reported as estimated values.

- 10.10 Laboratory Control Sample/Laboratory Control Sample Duplicate (LCS/LCSD) - An LCS/LCSD pair must be digested and analyzed with each batch of 20 samples.

10.10.1 **For SW-846 Method 6010B, 6010C, and 6010D** – The LCS recovery must be 80-120%. The RPD must be less than 20%. When using a certified solid reference material for soils, the manufacturer's established limits are used for control limits

10.10.2 **For EPA Method 200.7** – The LCS recovery must be 85-115%. The RPD must be less than 20%.

- 10.11 Interference Check Standards (ICSA/ICSAB) – The ICSA and ICSAB must be analyzed at the beginning and ending of each sequence or twice within each 8 hour shift, whichever is more frequent. The recovery of the ICSA and ICSAB elements must be 80-120%. If the results are unsatisfactory, see section 11.10 for further guidance. Do not proceed until this criterion is met.

NOTE: The unspiked elements in the ICSA are not evaluated by the data capture software. The instrument analyst evaluates whether the unspiked elements are <2 times the LLOQ for the ICSA.

10.11.1 For Method 6010D two types of SIC checks are used. Individual element SIC checks are performed when the instrument is initially setup, and periodically (at least once every 6 months) thereafter. The mixed element SIC solution is used daily to check that the instrument is free from interference from elements typically observed in high concentration and to check that and interference corrections applied are still valid.

10.11.1.1 Single element interference checks - At a minimum, single element SIC checks must be performed for the following elements: Aluminum 500mg/L; Boron 50mg/L; Barium, 50mg/L; Calcium 500mg/L; Copper 20mg/L; Iron 200mg/L; Magnesium 500mg/L; Manganese 10mg/L; Molybdenum 20mg/L; Sodium 1000mg/L; Nickel 20mg/L; Selenium 20mg/L; Silicon 100mg/L; Tin 20mg/L; Vanadium 20mg/L; Zinc 10mg/L.

The absolute value of the concentration observed for any unspiked analyte in the single element SIC checks must be less than two times the analytes LLOQ. The concentration of the SIC checks are suggested, but become the highest concentration allowed in a

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sample analysis, and cannot be higher than the highest established linear range. Samples with concentrations of elements higher than the SIC check must be diluted until the concentration is less than the SIC check solution. Note that reanalysis of a diluted sample is required even if the high concentration element is not required to be reported for the specific sample, since the function of the SIC check is to evaluate spectral interferences on other elements.

The single element SIC checks are performed when the instrument is setup and periodically (at least once every 6 months) thereafter.

- 10.11.1.2 Mixed element interference check - The mixed element SIC solution (see section 7.10.3.2) is analyzed at least once per day, immediately after the initial calibration. The concentration measured for any target analytes must be less than +/- two times the LLOQ. If this criterion is not met then sample analysis may not proceed until the problem is corrected, or alternatively the LLOQ may be raised to twice the concentration observed in the SIC solution. The only exceptions are those elements that have been demonstrated to be contaminants in the SIC solutions (see Section 7.10.3.1). These may be present up to the concentration documented plus the LLOQ.

State Note: For Wisconsin samples, the ICS is evaluated to the LOQ.

- 10.12 Serial Dilution (SD) - If the analyte concentration is sufficiently high (minimally 10X the IDL), an analysis of a 1:4 dilution must agree within 10% of the original determination. If not, see section 11.11 for further guidance.
- 10.13 Post digestion Spike (PS) - An analyte spike added to a portion of a prepared sample, or its dilution and must be recovered to within 75% to 125% of the known value. The spike addition should produce a minimum level of 10 times and a maximum of 100 times the IDL. If the spike is not recovered within the specified limits, see section 11.11 for further guidance.
- For SW-846 Method 6010C** – The Post Digestion Spike must recover within $\pm 20\%$ of the known value.
- 10.14 Internal Standard (ISTD) – Verify the internal standard responses. The intensity of the internal standard response in a sample is monitored and compared to the intensity of the response for that internal standard in the calibration blank. The Percent Relative Intensity (%RI) in the sample must fall within 60-140% of the response in the calibration blank. If the %RI of the response in the sample falls outside of these limits, see sections 9.15 & 11.9 for further guidance.
- 10.15 Sample Dilution - If a sample analyte concentration is quantitated within 90% or greater of the upper limit of the analyte Linear Dynamic Range (LDR), the sample must be diluted with acidified reagent water and re-analyzed.

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10.16 Lower Limit of Quantitation (LLOQ) – When analyzing samples according to Method 6010D, the LLOQ is initially verified by the analysis of at least 7 replicate samples, spiked at the LLOQ and processed through all preparation and analysis steps of the method. The mean recovery and relative standard deviation of these samples provide an initial statement of precision and accuracy at the LLOQ. In most cases the mean recovery should be +/- 35% of the true value and RSD should be < 20%. In-house limits may be calculated when sufficient data points exist. Monitoring recovery of LLOQ over time is useful for assessing precision and bias.

10.16.1 Ongoing LLOQ verification, at a minimum, is on a quarterly basis to validate quantitation capability at low analyte concentration levels. This verification may be accomplished either with clean control material (e.g., reagent water, method blanks, Ottawa sand, diatomaceous earth, etc.) or a representative sample matrix (free of target compounds). Optimally, the LLOQ should be less than the desired regulatory action levels based on the stated project-specific requirements.

11.0 DATA VALIDATION AND CORRECTIVE ACTION

- 11.1 All data must undergo a primary review by the analyst. The analyst must check the performance of the initial calibration, mid-point check standard, and continuing calibrations to ensure that they meet the criteria of the method. The analyst should review any sample that has quantifiable compounds and make sure that they have been confirmed, if needed. The analyst must also verify that reported results are derived from quantitation between the RL and the highest standard of the initial calibration curve. All calculations must be checked (any dilutions, %solids, etc.). Data must be checked for the presence or absence of appropriate flags. Comments should be noted when data is flagged.
- 11.2 All data must then undergo a second analyst review. This review must be performed according to Pace National SOPs #030201, *Data Handling and Reporting* and #030227, *Data Review*.
- 11.3 Initial Calibration – After analyzing the calibration standards, the curve is reviewed to ensure the acceptance criteria described in section 10.5 are met. If analytes do not meet this requirement, corrective action must be taken. Corrective actions may include re-calibrating the instrument, replacing the tubing on the peristaltic pump, examining blanks and standards for degradation/contamination, or performing instrument maintenance. If the internal standard responses in the calibration standards do not meet the criteria in section 10.14, re-calibrate the instrument.
- 11.4 Initial Calibration Verification (ICV) – If the criteria described in section 10.6 are not met for a target analyte, re-analyze the ICV/ICVLL. If this fails a second time, corrective action must be taken. Re-calibrate and re-analyze the ICV/ICVLL using the same standard. If acceptance criteria are still not met, re-check standard curve and ICV/ICVLL preparation and/or perform routine instrument maintenance. If still not acceptable, refer to manufacturer's instruction or call service representative.

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- 11.5 Continuing Calibration Verification (CCV) – The continuing calibration verification standard must agree with the criteria in section 10.7 or the CCV must be re-analyzed. If the recovery fails a second time, corrective action must be taken. The corrective action may require re-calibrate the instrument and re-analyze the last 10 samples, using the same CCV standard. If acceptance criteria are still not met, re-check the standard curve and CCV preparation and/or perform instrument maintenance. If the CCV still does not pass, refer to the manufacturer's instruction or call a service representative.
- 11.6 Blanks – Evaluate the blanks. The analyst must confirm that both the method blanks and the continuing calibration blanks were analyzed at the required frequency. Other items to check are as follows:
- 11.6.1 The instrument blank or continuing calibration blank (ICB/CCB) must meet the criteria in section 10.8. Corrective actions for method blank contamination include re-prepping the entire batch of samples, or if the site-specific requirements can be met, an elevated detection limit may be used, or the sample data qualified with a "B" qualifier and footnoted.
- NOTE:** Method 6010D - If the method blank fails to meet the necessary acceptance criteria, it should be reanalyzed once. If still unacceptable, then all samples associated with the method blank must be re-prepared and re-analyzed along with all other appropriate analysis batch QC samples. If the method blank results do not meet the acceptance criteria and reanalysis is not practical, then the laboratory should report the sample results along with the method blank results and provide a discussion of the potential impact of the contamination on the sample results. However, if an analyte of interest is found in a sample in the batch near its concentration confirmed in the blank, the presence and/or concentration of that analyte should be considered suspect and may require qualification.
- 11.7 Laboratory Control Sample (LCS)/Laboratory Control Sample Duplicate (LCSD) – Assess that LCS pairs were prepared at the required frequency. If all target analyte recoveries are not within the criteria described in section 10.10, rinse the instrument and re-analyze. If the LCS/LCSD fails for a second time, re-prepared all samples prepared in conjunction with the failing LCS. The affected samples must be re-digested and re-analyzed along with a new LCS. If there is insufficient volume submitted to re-prepare the field samples, notify the project manager to contact the client for further instruction. Reporting with a qualifier may be performed if acceptable to the client.
- 11.8 Matrix Spike (MS)/Matrix Spike Duplicate (MSD) – Assess that MS pairs were prepared at the required frequency. If all target analyte recoveries are not within the criteria described in section 10.9, review the post digestion spike results for similar failures. If the post spike confirms the matrix interferent, qualify the data using an O1 and J4. If the post spike does not confirm the interferent, rinse the instrument and re-analyze. If the MS/MSD fails for a second time, review the data to see if similar results are also present in the LCS/LCSD. If the LCS/LCSD are acceptable, the MS/MSD failures can be

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attributed to matrix interferences and the data can be qualified with a J4 and reported. If similar results are seen in the LCS, then re-prepared all samples prepared in conjunction with the failing MS/MSD. In this case, the affected samples must be re-digested and re-analyzed along with a new MS/MSD pair. If there is insufficient volume submitted to re-prepare the field samples, notify the project manager to contact the client for further instruction. Reporting with a qualifier may be performed if acceptable to the client. If insufficient field sample remains for re-analysis, report results with a J3 and an L3.

NOTE: If the sample concentration for an analyte is greater than 4 times the spike concentration, a "V" qualifier is used. The "V" qualifier indicates that the high concentration of analyte in the sample interfered with the ability to make an accurate spike recovery determination.

- 11.9 ISTD – The intensity of the Yttrium internal standard response in each sample is monitored and compared to the intensity of the response for that internal standard in the calibration blank. The Percent Relative Intensity (%RI) in each sample must meet the criteria in section 10.14. If the %RI of the response in the sample falls outside of these limits, the laboratory must immediately re-analyze the calibration blank and monitor the internal standard intensities. If the %RI for that calibration blank is within the limits, the laboratory must re-analyze the original sample at a two-fold dilution due to a possible interference from the matrix on the ISTD. If the %RI for the re-analyzed calibration blank is outside the limits, the analysis must be terminated, the problem corrected, the instrument recalibrated, the new calibration verified, and the samples reanalyzed.
- 11.10 Interference Check Standards (ICSA/ICSAB) - Evaluate the ICSA and ICSAB. The analyst must verify that the ICS and ICSAB have been analyzed at the required frequency. If the criteria in section 10.11 are not met, check the background correction protocols currently in place for appropriateness. If this is the initial ICS and/or ICSAB run after daily calibration, re-analyze the CALBLANK and re-calibrate the instrument. If the ICSA and/or ICSAB did not agree at the end of an 8-hour shift, re-analyze the ICSA and ICSAB. If failure persists, perform instrument maintenance as needed, recalibrate and re-analyze any samples in the previous run that may have been affected.
- 11.11 Serial Dilution/Post-digestion Spike – The analyst must verify that the SD and PS have been analyzed at the required frequency. If either of these tests fails to meet the required criteria in sections 10.12 & 10.13, the possibility of a matrix interferent should be suspected. An O1 qualifier is used when either sample type fails due to matrix interferences.
- 11.11.1 Serial Dilution - An analysis of a 1:4 dilution must agree within 10% of the original determination. If not, a chemical or physical interference effect is suspected.
- 11.11.2 Post-digestion Spike - The spike addition should produce a minimum level of 10 times and a maximum of 100 times the instrumental detection limit. If the spike is not recovered within the specified limits, a matrix effect is suspected.

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CAUTION: If spectral overlap is suspected, use of computerized compensation, an alternate wavelength, or comparison with an alternate method is recommended.

- 11.12 Data that does not meet acceptable QC criteria may be acceptable for use in certain circumstances.
- 11.12.1 If a method blank contains an amount of target analyte, but all samples are non-detected, the data may be reported with a "B3" flag. If a method blank contains an amount of target analyte, but the samples contain analyte at a level that is 10 times the level present in the method blanks, the data may be reported with a "B" flag.
- 11.12.2 If the MS/MSD fails (recovery less than 30% or greater than 150% and/or RPD greater than 30%) in an initial analysis and again upon re-analysis, the data is released with an appropriate qualifier as the failure is accepted as matrix related.
- 11.12.3 If a calibration verification standard is above the acceptable QC criteria and all samples being bracketed are below the reporting limit, the data is acceptable based on a high calibration bias with undetectable levels in the field samples. Any positive samples require re-analysis.
- 11.12.4 If the target analyte spiked in the quality control samples (LCS, LCSD, MS, MSD) exhibits high recovery and the target analytes in the field samples are below the reporting limit, the data may be released with a J+ qualifier indicating the high bias with no impact on the field sample analysis due to the bias present.
- 11.12.5 If the target analyte spiked into the QC pair (LCS/LCSD, MS/MSD) exhibit acceptable recoveries, but high calculated RPD values for precision, and the target analytes in the field sample are flagged with a J3 for the precision beyond acceptable quality control limits.
- 11.12.6 Sample results can be qualified and possible bias is narrated per the Pace National SOP #030201, *Data Handling and Reporting*.

STATE NOTE: Drinking water samples analyzed using this procedure for compliance cannot be qualified.

12.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

- 12.1 The EPA requires that laboratory waste management practice to be conducted consistent with all applicable federal and state laws and regulations. Excess reagents, samples and method process wastes must be characterized and disposed of in an acceptable manner. See *Pace National Waste Management Plan*.
- 12.2 See SOP #030302, *Environmental Sustainability & Pollution Prevention*.

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13.0 METHOD MODIFICATIONS/CLARIFICATIONS

- 13.1 Adjustments to the concentrations of standards/spiking solutions, standards providers, and quality control are subject to change to better meet client/project/regulatory needs or to improve laboratory method performance.
- 13.2 Modifications to this method are noted in the body of the text as state notes. Compliance analyses performed in conjunction with specific state requirements must be performed as noted within the specific state(s) note listed.
- 13.3 Superscripts are provided where necessary to indicate the reference in Section 14.0 where the requirement/information can be found. Subscripts noted identify the most frequent/restrictive cases, but requirements may also be included at different frequencies/conditions in other references noted in section 14.0.
- 13.4 In the May 2012 Methods Update Rule, the EPA revised the previous interpretation of EPA 200.7 to include the use of axial torch orientation in the published method. Either axial or radial orientation is acceptable.

14.0 REFERENCES

- 14.1 *Inductively Coupled Plasma-Atomic Emission Spectrometry*, SW-846 Method 6010B, Revision 2, December 1996.
- 14.2 *Determination of Metals and Trace Elements in Water and Wastes by Inductively Coupled Plasma-Atomic Emission Spectrometry*, EPA Method 200.7, Revision 4.4, May 1994
- 14.3 *Identification of Test Procedures*, 40 CFR §136.3
- 14.4 *Inorganic Chemical Sampling and Analytical Requirements*, 40 CFR §141.23
- 14.5 *Inductively Coupled Plasma-Atomic Emission Spectrometry*, SW-846 Method 6010C, Revision 3, February, 2007.
- 14.6 *Hardness by Calculation*, Standard Methods (SM) 2340B, 20th Edition.
- 14.7 *Hardness by Calculation*, SM 2340B-2011.
- 14.8 *Hardness by Calculation*, SM 2340B-1997.
- 14.9 *Inorganic Analytes*, SW-846 Chapter 3, Revision 4, February, 2007.

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Attachment I: Revision History

Current Version:

Version	Date	Description of Revisions
21	9/10/18	Update in response AZ audit finding CAR3296. Revised Post-Spike acceptance criteria to $\pm 20\%$ in Section 10.9.1.2

Superseded Versions:

This document supersedes the following:

Version	Date	Description of Revisions
0	5/1/95	Origination
1	7/25/95	
2	3/11/97	
3	8/18/99	
4	2/11/00	
5	8/21/00	
6	3/28/01	
7	12/14/01	
8	4/11/03	
9	1/26/04	
10	8/2/04	
11	10/15/05	Corrected CCV criteria for EPA 200.7
12	10/29/08	Technical and Quality Review and update. Corrected acceptance criteria in Section 10.6. Updated format and re-organized sections 8.0, 10.0 and 11.0 based on new format.
13	1/23/09	Technical and Quality Review and update.
14	2/2/09	Technical and Quality Review and update. Clarification of holding times, Inclusion of cross-references. Inclusion of section 13.1 and section 7.1.
15	4/15/11	Technical and Quality Review and update. Added state notes where applicable; Added Tables 1.2b & 1.2c; Revised Table 1.2a and Sections 1.1, 1.3, 1.6, 1.11, 2.18, 2.22, 5.6, 7.1, 7.6.2, 7.9, 8.1.3, 8.1.5, through 8.1.10, 9.1, 9.7 through 9.12, 10.3, 10.0 & 11.0, 12.1; Added Sections 2.13.1, 2.14.1, 2.31 through 2.35, 3.1.1, 4.1, 4.7, 7.17, 13.2, 13.3, 14.5 through 14.10.
16	6/27/14	Complete Rewrite and update.
17	12/7/2015	Technical and quality review and update. Header and signature block re-formatting. Revised Sections 1.13.1, 2.13.1, 2.23, 5.1.3, 6.1.1, 6.1.2, 7.1, 7.6, 7.7, 7.8, 7.9, 7.10.1, 7.10.2, 7.11, 7.12, 7.13, 7.14, 7.16, 8.1.3, 8.1.5, 8.1.11, 9.4, 10.2, 10.3.1, 10.3.2, 10.4.2, 10.4.4, 10.9, 10.10, 10.14, 11.12.6, and 12.2. Revised Tables 1.2a, and 1.10. Deleted Sections 2.22 and 7.12. Added Attachment III.

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Version	Date	Description of Revisions
18	10/28/2016	Technical and quality review and update. Update per South Carolina DHEC correspondence of 6/24/16. Header and signature block re-formatting. Revised SOP title. Revised Sections 1.1, Table 1.2a, 1.3, 1.6, 1.10, 2.12, 2.15, 5.6, 7.5.2, 7.10.1, 9.1, 10.2, 10.3.1, 10.4.2, 10.4.3, 10.4.5, 10.5, 10.6.1, 10.6.2, 10.7.1, 10.8.1.2, 10.8.2.2, 10.10.1, 10.16, and Attachment III Table 2. Deleted Table 1.2b. Deleted Sections 2.2, 2.12, 2.13, 2.14, 2.15, 2.17, 2.20, through 2.33, 3.5, 4.8, 7.14, 7.16, 8.1.3, 9.7 through 9.10, 9.13, 10.4.5.1, 10.4.5.2, 10.4.5.3, 10.10.3, 13.5, 14.7, 14.8, 14.9, and 14.10. Added Sections 2.14, 7.10.3 and all subsections, 9.11, 9.12, 10.1.1, 10.6.4, 10.6.5, 10.8.1.2, 10.9.1 and all subsections, 10.11.1 and all subsections, and 11.6.1.
19	11/30/2017	Update in response to A2LA audit finding CAR2872. Changed ESC logo. Updated Sections 1.5, 3.1, 7.9, 10.11, 14.1, 14.2, 14.3, 14.4, 14.5, 14.6, 14.7, 14.8, 14.9, and Attachment III Table 5.
20	7/27/18	Update in response to WI and AZ audit findings. Changed logo and references to "ESC" to "Pace National". Revised Section 7.14, 10.4.4, and 10.7.3. Added State Note to Sections 10.8.1.2 and 10.11. Also added 6010C note to Section 10.13

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Attachment II: Potential ICP interferences arising from analytes present in field samples at concentrations of 100mg/L

Analyte	Wavelength (nm)	Interferant ^{a,b}									
		Al	Ca	Cr	Cu	Fe	Mg	Mn	Ni	Ti	V
Aluminum	308.215	--	--	--	--	--	--	0.21	--	--	1.4
Antimony	206.833	0.47	--	2.9	--	0.08	--	--	--	0.25	0.45
Arsenic	193.696	1.3	--	0.44	--	--	--	--	--	--	1.1
Barium	455.403	--	--	--	--	--	--	--	--	--	--
Beryllium	313.042	--	--	--	--	--	--	--	--	0.04	0.05
Cadmium	226.502	--	--	--	--	0.03	--	--	0.02	--	--
Calcium	317.933	--	--	0.08	--	0.01	0.01	0.04	--	0.03	0.03
Chromium	267.716	--	--	--	--	0.003	--	0.04	--	--	0.04
Cobalt	228.616	--	--	0.03	--	0.005	--	--	0.03	0.15	--
Copper	324.754	--	--	--	--	0.003	--	--	--	0.05	0.02
Iron	259.940	--	--	--	--	--	--	0.12	--	--	--
Lead	220.353	0.17	--	--	--	--	--	--	--	--	--
Magnesium	279.079	--	0.02	0.11	--	0.13	--	0.25	--	0.07	0.12
Manganese	257.610	0.005	--	0.01	--	0.002	0.002	--	--	--	--
Molybdenum	202.030	0.05	--	--	--	0.03	--	--	--	--	--
Nickel	231.804	--	--	--	--	--	--	--	--	--	--
Selenium	196.026	0.23	--	--	--	0.09	--	--	--	--	--
Sodium	588.995	--	--	--	--	--	--	--	--	0.08	--
Thallium	190.864	0.30	--	--	--	--	--	--	--	--	--
Vanadium	292.402	--	--	0.05	--	0.005	--	--	--	0.02	--
Zinc	213.856	--	--	--	0.14	--	--	--	0.29	--	--

^a Dashes indicate that no interference was observed even when interferents were introduced at the following levels:

Al - 1000 mg/L	Mg - 1000 mg/L
Ca - 1000 mg/L	Mn - 200 mg/L
Cr - 200 mg/L	Ti - 200 mg/L
Cu - 200 mg/L	V - 200 mg/L
Fe - 1000 mg/L	

^b The figures recorded as analyte concentrations are not the actual observed concentrations; to obtain those figures, add the listed concentration to the interferant figure.

^c Interferences will be affected by background choice and other interferences may be present.

NOTE: Using the above table, if analyzing for Lead in a sample containing 1000mg/L Aluminum, the lead results could demonstrate a high bias of 0.17mg/L. (If the sample contained 10000mg/L of Al, the bias in lead could be 1.7mg/L), if background corrections are not accurately applied by the instrument.

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Attachment III: DoD Requirements

1.0 Equipment/Instrument Maintenance

Instrument maintenance must be performed routinely to optimize instrument performance and improve chromatography. Commonly performed maintenance includes changing pump tubing, replacing the torch, cleaning the nebulizer, etc. A new calibration curve must be analyzed following any major maintenance performed on the analytical system.

2.0 Computer Hardware and Software

QTegra, Version 2.4

3.0 Troubleshooting

Table 1. GC Troubleshooting Guide		
Problem	Cause	Treatment
Poor Precision	Nebulizer Pressure	Pressure should be about 0.15 mPa for aqueous solutions. If pressure is substantially higher, clean the nebulizer orifice or replace it entirely.
	Pooling in Spray Chamber	Usually caused by an oily film in the spray chamber. Aspirate 0.1% HF solution for about 20 seconds or 0.01% Triton X-100 solution.
	Center Tube	Replace the tube.
	Capillary Tubing	Air bubble migration through tubing should be smooth and consistent. Replace kinked/ pinched tubing.
	Peristaltic Pump	Adjust platen pressure. Check for leaks. Replace damaged pump.
Poor Accuracy	Pump Rate	Ensure the flush pump rate is the same as the analysis pump rate.
	Flush Time	Ensure proper time set for adequate rinse (typically 30 seconds).
Poor Detection Limits	Dirty Window or Mirror	Clean or replace dirty components.

4.0 Other Requirements

- 4.1 All hardcopy laboratory notebooks must be reviewed by the Supervisor, or their designee, on a monthly basis.
- 4.2 If not self-explanatory (e.g., a typo or transposed number), corrections to technical and quality records shall also include a justification for the change.
- 4.3 A person performing a manual integration must sign and date each manually integrated chromatogram and record the rationale for performing manual integration. Electronic signatures are acceptable.

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- 4.4 The results of calibration and verification of support equipment must be within the specifications required of the application for which this equipment is used or the equipment must be removed from service until repaired. Calibration and verification records, including those of established correction factors, must be maintained. In the absence of method-specific requirements, the minimum requirements are as follows:

Table 2. Support Equipment Checks		
Performance Check	Frequency	Acceptance Criteria
Balance calibration check [Using two standard weights that bracket the expected mass]	Daily prior to use	Top-loading balance: $\pm 2\%$ or $\pm 0.02\text{g}$, whichever is greater Analytical balance: $\pm 0.1\%$ or $\pm 0.5\text{mg}$, whichever is greater
Verification of standard mass [Using weights traceable to the International System of Units (SI) through a NMI]	Every 5 years	Certificate of Calibration from ISO/IEC 17025 accredited calibration laboratory
Monitoring of refrigerator/freezer temperatures	Daily (i.e. 7 days per week) [use MIN/MAX thermometers or data loggers equipped with notification of out of control event capabilities if personnel not available to record daily]	Refrigerators: 0°C to 6°C Freezers: $\leq -10^{\circ}\text{C}$
Thermometer verification check [Using a thermometer traceable to the SI through an NMI] [Performed at two temperatures that bracket the target temperature(s). Assume linearity between the two bracketing temperatures.] [If only a single temperature is used, at the temperature of use]	Liquid in glass: Before first use and annually Electronic: Before first use and quarterly	Apply correction factors or replace thermometer
Volumetric labware	Class B: By lot before first use Class A and B: Upon evidence of deterioration	Bias: Mean within $\pm 2\%$ of nominal volume Precision: $\text{RSD} \leq 1\%$ of nominal volume (based on 10 replicate measurements)
Non-volumetric labware [Applicable only when used for measuring initial sample volume and final extract/ digestates volume]	By lot before first use or upon evidence of deterioration	Bias: Mean within $\pm 3\%$ of nominal volume Precision: $\text{RSD} \leq 3\%$ of nominal volume (based on 10 replicate measurements)

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Table 2. Support Equipment Checks

Performance Check	Frequency	Acceptance Criteria
Mechanical volumetric pipette	Quarterly	Bias: Mean within $\pm 2\%$ of nominal volume Precision: RSD $\leq 1\%$ of nominal volume (based on minimum of 3 replicate measurements) [Note: for variable volume pipettes, the nominal volume is the volume of use]
Glass microliter syringe	Upon receipt and upon evidence of deterioration	General Certificate of Bias & Precision upon receipt Replace if deterioration is evident
Drying oven temperature check	Daily prior to and after use	Within $\pm 5\%$ of set temperature
Water purification system	Daily prior to use	See method blank criteria given in Section 4.20 of this addendum

- 4.5 The expiration date of the prepared standard shall not exceed the expiration date of the primary standard. All containers must bear a preparation date.
- 4.6 To avoid preparing non-representative samples, the laboratory shall not “target” within a relatively small mass range (e.g., $1.00 \pm 0.01\text{g}$) because such targeting will produce non-representative subsamples if the sample has high heterogeneity. The laboratory shall not manipulate the sample material so the sample aliquot weighs exactly $1.00\text{g} \pm 0.01\text{g}$, as an example.
- 4.7 In the absence of project-specific requirements, the minimum standard data qualifiers to be used are:
- U Analyte was not detected and is reported as less than the LOD or as defined by the customer. The LOD has been adjusted for any dilution or concentration of the sample.
 - J The reported result is an estimated value (e.g., matrix interference was observed or the analyte was detected at a concentration outside the quantitation range).
 - B Blank contamination. The recorded result is associated with a contaminated blank.
 - N Non-target analyte. The analyte is a tentatively identified compound using mass spectrometry or any non-customer requested compounds that are tentatively identified.
 - Q One or more quality control criteria failed (e.g., LCS recovery, surrogate spike recovery, or CCV recovery).

Additional data qualifiers may be used, or different letters or symbols to denote the qualifiers listed above, as long as they are appropriately defined and their use is consistent with project-specific requirements (e.g., QSM 5.0, the contract, and project-planning documents).

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- 4.8 If the time of the sample collection is not provided, assume the most conservative time of day. For the purpose of batch processing, the start and stop dates and times of the batch preparation shall be recorded.
- 4.9 Each preparation method listed on the scope of accreditation must have quarterly LOD/LOQ verifications. However, not all possible combinations of preparation and cleanup techniques are required to have LOD/LOQ verifications. If LOD/LOQ verifications are not performed on all combinations, the laboratory must base the LOD/LOQ verifications on the worst case basis (preparation method with all applicable cleanup steps).
- 4.10 After each MDL determination, the laboratory must establish the LOD by spiking a quality system matrix at a concentration of at least 2 times but no greater than four times the MDL. This spike concentration establishes the LOD and the concentration at which the LOD shall be verified. It is specific to each suite of analyte, matrix, and method (including sample preparation). The following requirements apply to the initial LOD establishment and to the LOD verifications:
- The apparent signal to noise (S/N) ratio at the LOD must be at least three and the results must meet all method requirements for analyte identification (e.g., ion abundance, second column confirmation, or pattern recognition). For data systems that do not provide a measure of noise, the signal produced by the verification sample must produce a result that is at least three standard deviations greater than the mean method blank concentration. This is initially estimated based on a minimum of four method blank analyses and later established with a minimum of 20 method blank results.
 - If the LOD verification fails, then the laboratory must repeat the MDL determination and LOD verification or perform and pass two consecutive LOD verifications at a higher spike concentration and set the LOD at the higher concentration.
 - The laboratory shall maintain documentation for all MDL determinations and LOD verifications.
 - The DL and LOD must be reported for all analyte-matrix-methods suites, unless it is not applicable to the test or specifically excluded by project requirements.
- 4.11 The LOD shall be verified quarterly. In situations where methods are setup and used on an infrequent basis, the laboratory may choose to perform LOD verifications on a one per batch basis. All verification data will be in compliance, reported, and available for review.
- 4.12 For DoD, at a minimum, the LOQ shall be verified quarterly. In situations where methods are setup and used on an infrequent basis, the laboratory may choose to perform LOQ verifications on a one per batch basis.
- 4.13 All initial instrument calibrations must be verified with a standard obtained from a second manufacturer prior to analyzing any samples. The use of a standard from a second lot obtained from the same manufacturer (independently prepared from different source materials) is acceptable for use as a second source standard. The concentration of the second source standard shall be at or near the midpoint of the calibration range. The acceptance criteria for the initial calibration verification must be at least as stringent as those for the continuing calibration verification.

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- 4.14 Exclusion of calibration points without documented scientifically valid technical justification is not permitted.
- 4.15 The concentration of the CCV standard shall be greater than the low calibration standard and less than or equal to the midpoint of the calibration range.
- 4.16 All CCVs analyzed must be evaluated and reported. If a CCV fails, reanalysis or corrective actions must be taken.
- If a CCV fails, the laboratory can immediately analyze two additional consecutive CCVs (immediately is defined as starting a consecutive pair within one hour; no samples can be run between the failed CCV and the two additional CCVs). This approach allows for spurious failures of analytes to be reported without reanalysis of samples. Any corrective actions that change the dynamics of the system (e.g., clip column, clean injection port, run blanks) requires that all samples since the last acceptable CCV be reanalyzed.
 - Both of these CCVs must meet acceptance criteria in order for the samples to be reported without reanalysis.
 - If either of these two CCVs fail or if the laboratory cannot immediately analyze two CCVs, the associated samples cannot be reported and must be reanalyzed.
 - Corrective action(s) and recalibration must occur if the above scenario fails. All affected samples since the last acceptable CCV must be reanalyzed.
 - Flagging of data for a failed CCV is only appropriate when the affected samples cannot be reanalyzed. The laboratory must notify the client prior to reporting data associated with a failed CCV.
- 4.17 The results of all MS/MSDs must be evaluated using the same acceptance criteria used for the DoD LCS limits (see Addendum Tables 3 and 4) or project limits, if specified. If the specific analyte(s) are not available in the Addendum Tables 3 and 4, the laboratory shall use their LCS in-house limits (see the LIMS) as a means of evaluating MS/MSDs. The MS and MSD must be spiked with all reported analytes.
- 4.18 Surrogate spike results shall be compared with DoD LCS limits (see Addendum Tables 3 and 4) or acceptance criteria specified by the client. If these criteria are not available, the laboratory shall compare the results with its in-house statistically established LCS criteria (see the LIMS).
- 4.19 The method blank shall be considered to be contaminated if:
- The concentration of any target analyte (chemical of concern) in the blank exceeds 1/2 the LOQ and is greater than 1/10th the amount measured in any associated sample, or 1/0th the regulatory limit, whichever is greater;
 - The concentration of any common laboratory contaminant in the blank exceeds the LOQ;
 - If a method blank is contaminated as described above, then the laboratory shall reprocess affected samples in a subsequent preparation batch, except when sample results are below



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the LOD. If insufficient sample volume remains for reprocessing, the results shall be reported with appropriate data qualifiers.

- 4.20 Sporadic Marginal Exceedances are not allowed for target analytes (chemicals of concern as identified by a project) without project-specific approval. Target analytes are considered those few analytes that are critical for the success of a project (such as risk drivers) where sporadic marginal exceedances cannot be allowed. Laboratories should consult with clients whenever long lists of analytes are requested for analysis to determine if marginal exceedances will not be allowed.
- 4.21 DoD considers the same analyte exceeding the LCS control limit two (2) out of three (3) consecutive LCS to be indicative of non-random behavior, which requires corrective action and reanalysis of the LCS.

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Table 3. LCS Control Limits – Method 6010 Solid Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
7429-90-5	Aluminum	6258	96.7	7.5	74	119
7440-36-0	Antimony	5997	96.4	5.7	79	114
7440-38-2	Arsenic	9530	96.2	4.9	82	111
7440-39-3	Barium	9236	98.3	5	83	113
7440-41-7	Beryllium	6799	97.8	5.1	83	113
7440-42-8	Boron	2312	93	7.1	72	114
7440-43-9	Cadmium	9466	97.5	5.3	82	113
7440-70-2	Calcium	6347	98.1	5.8	81	116
7440-47-3	Chromium	9598	98.9	4.6	85	113
7440-48-4	Cobalt	6725	98.7	4.5	85	112
7440-50-8	Copper	7839	99.1	6	81	117
7439-89-6	Iron	5746	99.7	6.1	81	118
7439-92-1	Lead	10160	96.8	5.1	81	112
7439-93-2	Lithium	551	98.8	4.5	85	112
7439-95-4	Magnesium	6283	96.1	6.1	78	115
7439-96-5	Manganese	6732	99.1	4.9	84	114
7439-98-7	Molybdenum	4424	98.7	5.7	82	116
7440-02-0	Nickel	7412	98.1	4.9	83	113
7723-14-0	Phosphorus	189	103.1	3.8	92	114
7440-09-7	Potassium	6574	98.3	5.8	81	116
7782-49-2	Selenium	8862	94.5	5.6	78	111
7440-22-4	Silver	9105	97.3	5	82	112
7440-23-5	Sodium	5825	100.1	5.8	83	118
7440-24-6	Strontium	2573	98.5	5	83	114
7440-28-0	Thallium	6416	96.8	4.6	83	111
7440-31-5	Tin	2780	100.1	6.6	80	120
7440-32-6	Titanium	2107	98.2	5.2	83	114
7440-61-1	Uranium	109	97.4	5.2	82	113
7440-62-2	Vanadium	6934	98.3	5.4	82	114
7440-66-6	Zinc	7882	97.4	5	82	113

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**DETERMINATION OF METALS AND TRACE ELEMENTS IN VARIOUS MATRICES BY
TITLE: ICP-AES (EPA METHODS 6010B, 6010C, 6010D [ICP-OES], AND 200.7) INCLUDING
HARDNESS (EPA METHODS 200.7 AND 6010B/C/D AND SM 2340B)**

Table 4. LCS Control Limits – Method 6010 Water Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
7429-90-5	Aluminum	11532	100	4.8	86	115
7440-36-0	Antimony	10737	100.2	4.2	88	113
7440-38-2	Arsenic	14123	99.9	4.3	87	113
7440-39-3	Barium	14476	100.3	4.1	88	113
7440-41-7	Beryllium	11552	100.4	4	89	112
7440-69-9	Bismuth	147	95.8	3.2	86	105
7440-42-8	Boron	3871	98.8	4.8	85	113
7440-43-9	Cadmium	13922	100.8	4.1	88	113
7440-70-2	Calcium	11382	100	4.2	87	113
7440-47-3	Chromium	15027	101.1	3.9	90	113
7440-48-4	Cobalt	11824	101.2	4.2	89	114
7440-50-8	Copper	12910	100.2	4.6	86	114
7439-89-6	Iron	13797	100.7	4.7	87	115
7439-92-1	Lead	14391	99.3	4.4	86	113
7439-93-2	Lithium	938	100.7	5.3	85	117
7439-95-4	Magnesium	11423	98.8	4.8	85	113
7439-96-5	Manganese	12767	101.9	4.1	90	114
7439-98-7	Molybdenum	8251	101.1	4	89	113
7440-02-0	Nickel	12699	100.5	4.1	88	113
7440-05-3	Palladium	492	99.8	4	88	112
7723-14-0	Phosphorus	203	100.5	4.2	88	113
7440-09-7	Potassium	11006	99.9	4.7	86	114
7782-49-2	Selenium	13264	98.5	5.2	83	114
7440-21-3	Silicon	1525	100.6	6.1	82	119
7440-22-4	Silver	13770	99.1	5.1	84	115
7440-23-5	Sodium	10893	100.9	4.7	87	115
7440-24-6	Strontium	3782	101.3	3.8	90	113
7704-34-9	Sulfur	145	100.7	3.9	89	112
7440-28-0	Thallium	10063	99.5	4.7	85	114
7440-31-5	Tin	4502	101.3	4.4	88	115
7440-32-6	Titanium	5625	101.1	3.4	91	111
7440-61-1	Uranium	223	101.3	5.8	84	119
7440-62-2	Vanadium	12032	100.2	3.6	90	111
7440-66-6	Zinc	13549	100.6	4.6	87	115

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TITLE: DETERMINATION OF METALS AND TRACE ELEMENTS IN VARIOUS MATRICES BY ICP-AES (EPA METHODS 6010B, 6010C, 6010D [ICP-OES], AND 200.7) INCLUDING HARDNESS (EPA METHODS 200.7 AND 6010B/C/D AND SM 2340B)

Table 5. Quality Control Requirements – Inorganic Analysis by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP/AES)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Linear Dynamic Range (LDR) or high-level check standard	At initial set up and checked every 6 months with a high standard at the upper limit of the range.	Within $\pm 10\%$ of true value.	Dilute samples within the calibration range, or re-establish/ verify the LDR.	Flagging is not appropriate.	Data cannot be reported above the high calibration range without an established/passing high-level check standard.
Initial Calibration (ICAL) for all analytes	Daily ICAL prior to sample analysis.	If more than one calibration standard is used, $r^2 \geq 0.99$.	Correct problem, then repeat ICAL.	Flagging is not appropriate.	Minimum one high standard and a calibration blank. No samples shall be analyzed until ICAL has passed.
Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All reported analytes within $\pm 10\%$ of true value.	Correct problem. Rerun ICV. If that fails, repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed until calibration has been verified with a second source.

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Table 5. Quality Control Requirements – Inorganic Analysis by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP/AES)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Continuing Calibration Verification (CCV)	After every 10 field samples, and at the end of the analysis sequence.	All reported analytes within $\pm 10\%$ of the true value.	Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails or if two consecutive CCVs cannot be run, perform corrective action(s) and repeat CCV and all associated samples since the last successful CCV. Alternately, recalibrate if necessary; then reanalyze all associated samples since the last acceptable CCV	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.	Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

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Table 5. Quality Control Requirements – Inorganic Analysis by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP/AES)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Low-Level Calibration Check Standard (LLCCV)	Daily	All reported analytes within $\pm 20\%$ of true value.	Correct problem and repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed without a valid low-level calibration check standard (LLCCV). Low-level calibration check standard should be less than or equal to the LOQ. If the concentration of the lowest calibration standard is less than or equal to the LOQ, the lowest standard may be re-quantified against the calibration curve as a LLCCV. Otherwise, a separate standard must be analyzed as LLCCV prior to the analysis of any samples.

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Table 5. Quality Control Requirements – Inorganic Analysis by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP/AES)					
QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Method Blank (MB)	One per preparatory batch.	The absolute values of all analytes must be $< \frac{1}{2}$ LOQ or $< 1/10^{\text{th}}$ the amount measured in any sample or $1/10$ the regulatory limit, whichever is greater.	Correct problem. If required, reprep and reanalyze MB and all QC and field samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid method blank. Non-detects associated with positive blank infractions may be reported. Sample results $>10X$ the LOQ associated with negative blanks may be reported. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

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Table 5. Quality Control Requirements – Inorganic Analysis by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP/AES)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Initial and Continuing Calibration Blank (ICB/CCB)	Immediately after the ICV and immediately after every CCV.	The absolute values of all analytes must be < ½ LOQ or < 1/10th the amount measured in any sample.	ICB: Correct problem and repeat ICV/ICB analysis. If that fails, rerun ICAL. All samples following the last acceptable Calibration Blank must be reanalyzed. CCBs may not be reanalyzed without reanalysis of the associated samples and CCV(s).	Flagging is not appropriate.	Results may not be reported without a valid calibration blank. Non-detects associated with positive blank infractions may be reported. Sample results >10X the LOQ associated with negative blanks may be reported. For CCB, failures due to carryover may not require an ICAL.
Interference Check Solutions (ICS) (also called Spectral Interference Checks)	After ICAL and prior to sample analysis.	ICS-A: Absolute value of concentration for all non-spiked project analytes <1/2 LOQ (unless they are a verified trace impurity from one of the spiked analytes); ICS-AB: Within ± 20% of true value.	Terminate analysis; locate and correct problem; reanalyze ICS, reanalyze all samples.	If corrective action fails, apply Q-flag to all results for specific analyte(s) in all samples associated with the failed ICS.	All analytes must be within the LDR. ICS-AB is not needed if instrument can read negative responses.

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Table 5. Quality Control Requirements – Inorganic Analysis by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP/AES)					
QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Laboratory Control Sample (LCS)	One per preparatory batch.	A laboratory must use Table 3 and 4 limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Must contain all reported analytes. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per preparatory batch.	A laboratory must use Table 3 and 4 limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J flag if acceptance criteria are not met and explain in the case narrative.	For matrix evaluation only. If MS results are outside the limits, the data shall be evaluated to the source(s) of difference (i.e., matrix effect or analytical error).

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QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch.	A laboratory must use Table 3 and 4 limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified. MSD or MD: RPD of all analytes $\leq 20\%$ (between MS and MSD or sample and MD).	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J flag if acceptance criteria are not met and explain in the case narrative.	The data shall be evaluated to determine the source of difference. For Sample/MD: RPD criteria only apply to analytes whose concentration in the sample is greater than or equal to the LOQ.
Dilution Test	One per preparatory batch if MS or MSD fails.	Five-fold dilution must agree within $\pm 10\%$ of the original measurement.	No specific CA, unless required by the project.	For the specific analyte(s) in the parent sample, apply J flag if acceptance criteria are not met and explain in the case narrative.	Only applicable for samples with concentrations $> 50 \times$ LOQ (prior to dilution). Use along with MS/MSD and PDS data to confirm matrix effects.

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Table 5. Quality Control Requirements – Inorganic Analysis by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP/AES)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Post-Digestion Spike (PDS) Addition (ICP only)	Perform if MS/MSD fails. One per preparatory batch (using the same sample as used for the MS/MSD if possible).	Recovery within 80-120%.	No specific CA, unless required by the project.	For the specific analyte(s) in the parent sample, apply J flag if acceptance criteria are not met and explain in the case narrative.	Criteria applies for samples with concentrations <50 X LOQ prior to dilution.
Method of Standard Additions (MSA)	When dilution test or post digestion spike fails and if required by project.	NA	NA	NA	Document use of MSA in the case narrative.

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TITLE: DETERMINATION OF METALS BY INDUCTIVELY COUPLED PLASMA MASS SPECTROSCOPY (ICP-MS) (EPA METHODS 6020, 6020A, 6020B & 200.8)

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1.0 SCOPE AND APPLICATION

STATE NOTE: For samples analyzed in conjunction with the Ohio Voluntary Action Program (VAP) please utilize SOP #340390OH.

- 1.1 This procedure is applicable to drinking water (DW), ground water (GW), wastewater (WW), waste and soil sample digestates are analyzed for trace metals utilizing an Agilent 7700x ICP-MS, and Agilent 7900 ICP-MS.
- 1.2 Pace National maintains approval for the analysis of up to 29 elements by the EPA Methods 200.8, 6020A and 6020 for water and soil matrices in standard mode. All target analytes are analyzed either in Helium mode (Collision Cell) or No gas mode on the Agilent instruments depending on the sample matrix type. The following Table (1.2) contains a list of the analytes that are currently analyzed along with routine reporting limits per matrix. See also section 13.3.

Table 1.2 Analytes and Reporting Limits**

Analyte	Aqueous Samples				Solid Samples	
	DW RL	GW RL	WW RL	Units	SS RL	Units
Aluminum	0.100	0.100	0.100	mg/L	10.0	mg/Kg
Antimony	0.001	0.002	0.002	mg/L	0.100	mg/Kg
Arsenic	0.001	0.002	0.001	mg/L	0.100	mg/Kg
Barium	0.002	0.005	0.005	mg/L	0.200	mg/Kg
Beryllium	0.001	0.002	0.001	mg/L	0.100	mg/Kg
Boron	0.020	0.020	0.020	mg/L	2.00	mg/Kg
Cadmium	0.001	0.001	0.001	mg/L	0.100	mg/Kg
Calcium	1.00	1.00	1.00	mg/L	100.	mg/Kg
Chromium	0.001	0.002	0.001	mg/L	0.100	mg/Kg
Cobalt	0.001	0.002	0.002	mg/L	0.100	mg/Kg
Copper	0.002	0.005	0.001	mg/L	0.200	mg/Kg
Iron	0.100	0.100	0.100	mg/L	10.0	mg/Kg
Lead	0.001	0.002	0.001	mg/L	0.100	mg/Kg
Magnesium	1.00	1.00	1.00	mg/L	100.	mg/Kg
Manganese	0.002	0.005	0.005	mg/L	0.200	mg/Kg
Molybdenum	0.002	0.005	0.005	mg/L	0.200	mg/Kg
Nickel	0.001	0.002	0.001	mg/L	0.100	mg/Kg

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TITLE: DETERMINATION OF METALS BY INDUCTIVELY COUPLED PLASMA MASS SPECTROSCOPY (ICP-MS) (EPA METHODS 6020, 6020A, 6020B & 200.8)

Analyte	Aqueous Samples				Solid Samples	
	DW RL	GW RL	WW RL	Units	SS RL	Units
Potassium	1.00	1.00	1.00	mg/L	100.	mg/Kg
Selenium	0.001	0.002	0.002	mg/L	0.100	mg/Kg
Silver	0.001	0.002	0.001	mg/L	0.200	mg/Kg
Sodium	1.00	1.00	1.00	mg/L	100.	mg/Kg
Strontium	0.010	0.010	0.010	mg/L	1.00	mg/Kg
Thallium	0.001	0.002	0.001	mg/L	0.100	mg/Kg
Thorium	0.010	0.010	0.010	mg/L	1.00	mg/Kg
Tin	0.001	0.002	0.002	mg/L	0.100	mg/Kg
Titanium	0.010	0.010	0.010	mg/L	1.00	mg/Kg
Uranium	0.010	0.010	0.010	mg/L	1.00	mg/Kg
Vanadium	0.002	0.005	0.005	mg/L	0.200	mg/Kg
Zinc	0.010	0.025	0.010	mg/L	1.00	mg/Kg

****See section 13.1 & 13.3.**

- 1.3 An MDL study must be completed at least annually or more frequently if major instrumentation changes occur. Method Detection Limits (MDLs) are performed based on Pace National SOP #030206. Updated MDL records are filed and stored in a central location within the department.
 - 1.3.1 Limit of Detection (LOD) and Limit of Quantitation (LOQ) studies are completed at the frequency required by the TNI standard per the procedure identified in the Pace National SOP #030206, *Method Detection Limits (MDL), Limits of Detection (LOD) and Limits of Quantitation (LOQ)*. Should the procedure be utilized for DOD support; then the frequency of these studies must meet the requirements of the current DOD QSM (see Attachment III).
- 1.4 Instrument Detection Limit studies must be analyzed at least quarterly for each analytical instrument.^{14.2, 14.4} Linear dynamic ranges must be established for each wavelength from a minimum of three different concentration standard across the range of expected use for each instrument. These ranges should be rechecked for accuracy every 6 month.^{14.4}

2.0 METHOD SUMMARY AND DEFINITIONS

- 2.1 The ICP-MS uses inductively coupled plasma as the source of metal cations for the quadrupole mass spectrometer detector. This results in an instrument capable of detecting a wide variety of metals. This method measures ions produced by a radio frequency inductively coupled plasma. Analytes originating in a liquid are nebulized and the resulting aerosol transported by argon gas into the plasma torch. The ions produced are introduced in the plasma gas, by means of an interface, into a mass spectrometer. The ions produced are sorted according to their mass-to-charge ratios and are quantified with a channel electron multiplier.
- 2.2 The detection limits for ICP-MS are comparable to those of graphite furnace atomic absorption spectroscopy (GFAAS). Although ICP-MS data is actually acquired

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sequentially, ICP-MS has the capability of determining all the metals of interest in the time it would take GFAAS methods to determine one analyte. Internal standards are used to compensate for changes in instrumental sensitivity and mass transport phenomena.

- 2.3 Tuning Solution – A solution that is used to determine acceptable instrument performance prior to calibration and sample analyses.
- 2.4 Linear Dynamic Range (LDR) - The range over which the instrument response to analyte concentration is linear.
- 2.5 Serial Dilution (SD) – When the concentration of an element of interest is at least X25 the MDL, the sample must be diluted by a minimum of X5 dilution. One serial dilution must be prepared and analyzed per batch of samples, except for drinking water samples by EPA Method 200.8.
- 2.6 Post Spike (PS) – A second aliquot of a field sample that is spiked with known concentrations of target analytes and analyzed to assess recovery of the spike. A post spike must be analyzed when the MS and/or MSD fail due to a suspected matrix effect. One sample is spiked after digestion and analyzed per batch. Post spikes are not performed for drinking water samples EPA Method 200.8.
- 2.7 Interference Check Sample (ICS) – A series of two solutions (ICSA & ICSAB) to verify that inter-element interferences are correctly compensated. The ICSA and ICSAB provide an adequate on-going test of inter-element correction (IEC) factors.
- 2.8 Lower Limit of Quantitation (LLOQ) - A term associated with analysis per the requirements of Method 6010D. The lowest point of quantitation which, in most cases, is the lowest concentration in the calibration curve.
- 2.9 See the current Quality Assurance Manual for other definitions associated with terms found in this document.

3.0 HEALTH AND SAFETY

- 3.1 The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds should be as low as reasonably achievable. A reference file of safety data sheets (SDSs) is made available on Pace National's intranet to all personnel. Use hazardous reagents in a fume hood whenever possible and if eye or skin contact occurs, flush with large volumes of water. Always wear safety glasses or a shield for eye protection, protective clothing and observe proper mixing protocols.
- 3.2 Use CAUTION with strong irritants such as acids and bases. Avoid breathing the fumes of these irritants by using them in a hood when possible. Avoid contact of these irritants with skin and clothing by appropriate use of gloves, apron, facemask, safety glasses, hood shield, etc.
- 3.3 All waste is emptied into 10 or 20L carboys. The carboys are then emptied into a waste drum. The contents of the drum are neutralized with sodium hydroxide until the pH is

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between 6 and 10. The neutralized liquid is then disposed of through conventional municipal waste.

4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

- 4.1 All samples must have been collected using a sampling plan that addresses the considerations of this method.
- 4.2 Prior to collection of an aqueous sample, consideration should be given to the type of data required (i.e., dissolved or total recoverable), so that appropriate preservation and pretreatment steps can be taken. The pH of all aqueous samples must be tested immediately prior to taking an aliquot for processing or "direct analysis" to ensure the sample has been properly preserved.
- 4.3 For the determination of dissolved elements, the sample must be filtered through a 0.45-um pore diameter membrane filter at the time of collection or as soon thereafter as practically possible. Use a portion of the sample to rinse the filter flask, discard this portion and collect the required volume of filtrate. Acidify the filtrate with nitric acid immediately following filtration to pH < 2. The sample is now ready for digestion.
- The hold time begins from the time of collection and ends with digestion within 180 days of collection.
- 4.4 For the determination of total recoverable elements in aqueous samples, samples are not filtered, but acidified with (1+1) nitric acid to pH < 2 (approximately, 3mL of (1+1) acid per liter of sample is sufficient for most ambient and drinking water samples). Preservation may be done at the time of collection; however, to avoid the hazards of strong acids in the field, transport restrictions, and possible contamination, it is recommended that the sample be returned to the laboratory within two weeks of collection and acid preserved upon receipt in the laboratory. Following acidification, the sample should be mixed, held for 24 hours, and then verified to be pH < 2 just prior to withdrawing an aliquot for processing or "direct analysis". If for some reason, such as high alkalinity, the sample pH is verified to be > 2, a T2 qualifier is applied in Prep Data. If the sample is reactive with Nitric acid, the volume of sample is reduced to 5mL for digestion. .
- The holding times for total recoverable elements begins from the time of collection and ends with digestion within 180 days of collection.
- 4.5 Solid samples require no preservation prior to preparation/analysis unless mercury or hexavalent chromium analyses will be performed from the same container. If so, then storage at 4° ± 2°C is required^{14.5}. The holding times for total recoverable elements begins from the time of collection and ends with digestion within 180 days of collection.
- 4.6 For aqueous samples, a field blank should be prepared and analyzed as required by the data user. Use the same container and acid as used in sample collection.
- 4.7 Samples submitted for analysis that do not meet the requirements contained within this section must be addressed before performing the logging process within the laboratory. In some cases, exceeding the appropriate preservation and storage criteria can cause

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significant bias in the resulting data. Clients may need to resubmit samples where the conditions during shipment cause uncertainty regarding sample integrity. If samples do not meet the requirements for preservation, sampling, shipment and storage and the client approves the completion of the analytical process, sample results can be qualified per the Pace National SOP #030201, *Data Handling and Reporting*.

5.0 INTERFERENCES

5.1 Several interference sources may cause inaccuracies in the determination of trace elements by ICP-MS. These are:

- 5.1.1 Isobaric elemental interferences - Are caused by isotopes of different elements which form singly or doubly charged ions of the same nominal mass-to-charge ratio and which cannot be resolved by the mass spectrometer in use. All elements determined by this method have, at a minimum, one isotope free of isobaric elemental interference. Of the analytical isotopes recommended for use with this method, only molybdenum-98 (ruthenium) and selenium-82 (krypton) have isobaric elemental interferences. If alternative analytical isotopes having higher natural abundance are selected in order to achieve greater sensitivity, an isobaric interference may occur. All data obtained under such conditions must be corrected by measuring the signal from another isotope of the interfering element and subtracting the appropriate signal ratio from the isotope of interest. A record of this correction process must be included with the report and data. It should be noted that such corrections would only be as accurate as the accuracy of the isotope ratio used in the elemental equation for data calculations. Relevant isotope ratios should be established prior to the application of any corrections.
- 5.1.2 Abundance sensitivity - A property defining the degree to which the wings of a mass peak contribute to adjacent masses. The abundance sensitivity is affected by ion energy and quadrupole operating pressure. Wing overlap interferences may result when a small ion peak is being measured adjacent to a large one. The potential for these interferences must be recognized and the spectrometer resolution adjusted to minimize them.
- 5.1.3 Isobaric polyatomic ion interferences - Caused by ions consisting of more than one atom which have the same nominal mass-to-charge ratio as the isotope of interest, and which cannot be resolved by the mass spectrometer in use. These ions are commonly formed in the plasma or interface system from support gases or sample components. Most of the common interferences have been identified with the method elements affected. Such interferences must be recognized, and when they cannot be avoided by the selection of alternative analytical isotopes, appropriate corrections must be made to the data. Equations for the correction of data must be established at the time of the analytical sequence as the polyatomic ion interferences are highly dependent on the sample matrix and chosen instrument conditions. In particular, the common ^{82}Kr interference that affects the determination of both arsenic and selenium can be greatly reduced with the use of high purity krypton free argon.

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5.1.4 Physical interferences - Are associated with the physical processes that govern the transport of sample into the plasma, sample conversion processes in the plasma, and the transmission of ions through the plasma-mass spectrometer interface. These interferences may result in differences between instrument responses for the sample and the calibration standards. Physical interferences may occur in the transfer of solution to the nebulizer (e.g., viscosity effects), at the point of aerosol formation and transport to the plasma (e.g., surface tension), or during excitation and ionization processes within the plasma itself. High levels of dissolved solids in the sample may contribute deposits of material on the extraction and/or skimmer cones reducing the effective diameter of the orifices and therefore ion transmission. Dissolved solids levels not exceeding 0.2% (w/v) have been recommended to reduce such effects. Internal standardization may be effectively used to compensate for many physical interference effects. Ideally internal standards ideally should have similar analytical behavior to the elements being determined.

5.1.5 Memory interferences - Result when isotopes of elements in a previous sample contribute to the signals measured in a new sample. Memory effects can result from sample deposition on the sampler and skimmer cones and from the buildup of sample material in the plasma torch and spray chamber. The site where these effects occur is dependent on the element and can be minimized by flushing the system with a rinse blank between samples. The possibility of memory interferences must be recognized within an analytical sequence and suitable rinse times should be used to reduce them. The rinse times necessary for a particular element must be estimated prior to analysis. This may be achieved by aspirating a standard containing elements corresponding to ten times the upper end of the linear range for a normal sample analysis period, followed by analysis of the rinse blank at designated intervals. The length of time required to reduce analyte signals to within a factor of ten of the method detection limit must be noted. Memory interferences may also be assessed within an analytical sequence by using a minimum of three replicate integrations for data acquisition. If the integrated signal values drop consecutively, the analyst must be alerted to the possibility of a memory effect, and should examine the analyte concentration in the previous sample to identify if this was high. If memory interference is suspected, the sample should be reanalyzed after a long rinse period. In the determination of mercury, which suffers from severe memory effects, the addition of 100 ug/L gold effectively rinses 5ug/L mercury in approximately 2 minutes. Higher concentrations require a longer rinse time.

5.2 Instrument Interferences: The primary interferences in ICPMS are molecular ions such as oxides, doubly-charged ions, and isobaric interferences. For the few target isotopes that isobaric interferences affect (Se), a correction is entered into the parameter set to compensate for each one. The interference check SRM solution, IC6020 is used to assess the effects of a variety of molecular interferences. This solution should pass for all components to be reported. In addition, the data for samples with requested target analytes above the MDL should be examined for evidence of interfering molecular ions or doubly-charged ions. A list of the peaks that need to be monitored follows here.

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Target Analyte	Common Interference	Monitor	Correction Based On
$^{111}\text{Cd}^+$	$^{95}\text{Mo}^{16}\text{O}^+$	$^{95}\text{Mo}^+$	$^{95}\text{Mo}^+$
114Cd	95Mo16o+, 50Sn	118Sn	NA
$^{65}\text{Cu}^+$	$^{25}\text{Mg}^{40}\text{Ar}^+$	$^{25}\text{Mg}^+$	NA
$^{65}\text{Cu}^+$	$^{32}\text{S}^{33}\text{S}^+$	$^{32}\text{S}^+$	NA
$^{60}\text{Ni}^+$	$^{44}\text{Ca}^{16}\text{O}^+$	$^{44}\text{Ca}^+$	NA
$^{75}\text{As}^+$	$^{40}\text{Ar}^{35}\text{Cl}^+$	$^{77}\text{Se}^+$	See 11.5.
$^{40}\text{Ar}^{37}\text{Cl}^+$	$^{77}\text{Se}^+$	$^{82}\text{Se}^+$	See 11.5.
$^{77,78,82}\text{Se}^+$	$^{82}\text{Kr}^+, ^1\text{H}^{81}\text{Br}^+$	$^{83}\text{Kr}^+, ^{79}\text{Br}^+$	$^{83}\text{Kr}^+$
$^{98}\text{Mo}^+$	$^{98}\text{Ru}^+$	$^{101}\text{Ru}^+$	$^{101}\text{Ru}^+$
$^{115}\text{In}^+$	$^{115}\text{Sn}^+$	$^{118}\text{Sn}^+$	$^{118}\text{Sn}^+$
$^{66}\text{Zn}^+$	$^{50}\text{Ti}^{16}\text{O}^+$	$^{50}\text{Ti}^+$	$^{50}\text{Ti}^+$

NA - Not Applicable

NOTE: 40 Ar 37 Cl + is not the target analyte monitored; rather any ion or combination thereof possessing a mass of 77amu is monitored.

5.3 Representative Correction Equations

V51 -0.0108*Cl37
As75 -3.127*[Arcl77-(.873*Se82)]
Se82, 77, 78 -0.978*Kr83
Mo98 -0.110588*Ru101
Cd111 -1.073*[Mo0108-(.712*Pb106)]
In115 -0.014032*Sn118
Ba137 -0.000903*La139 - 0.002825*Ce140
Cd114 -0.026826*Sn118
Sn119 -0.013447*Te125
Sn120 -0.013447*Te125
Sb123 -0.127189*Te125
Pb208 +1*Pb206+1*Pb207
Cd114 -0.027250*Sn118
Fe54 -0.028226*Cr52

6.0 EQUIPMENT AND SUPPLIES

6.1 The inductively coupled plasma mass spectrometer used is an Agilent 7700x ICP-MS, or Agilent 7900 ICP-MS.

6.2 Each ICP-MS is coupled to a Cetac ASX-500 series Autosampler or Agilent SPS 4 series Autosampler. A modified pumped rinse station was added to maintain fresh rinse solution at a constant level that ensures a clean Autosampler probe.

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6.3 The pump tubing used is as follows:

Agilent Tubing:

Sample white-white 1.02mm ID

Internal standard orange-blue 0.25mm ID

Drain yellow-blue 1.52mm ID

6.4 Samples and Standards are aspirated from polypropylene centrifuge tubes arranged in the Autosampler.

6.5 Argon gas supply: High purity grade (99.99%).

6.6 Adjustable pipettes (Eppendorf or equivalent).

6.7 Class A volumetric flasks.

6.8 Ultra high purity Helium gas supply for Agilent collision cell.

7.0 REAGENTS AND STANDARDS

7.1 All reagents and standards must be recorded in the appropriate preparation log and assigned a unique number. See SOP #030230, *Standards Logger – Tree Operation*. Additional information regarding reagent preparation can be found in the Standards Logger (Tree) digital archive system. All spiking solutions and surrogate standard solutions should be replaced at least every 6 months or sooner if a problem is detected unless otherwise noted.

7.2 Be careful not to contaminate solutions with metals or minerals during preparation and use. Be sure to mix all solutions thoroughly after they are prepared. Store standard solutions in Teflon bottles if possible (polyethylene is acceptable for standards containing analytes at high concentration. Label all bottles containing solutions giving the following information: name of solution (as given below), analytes, concentrations, instructions for use (if any), date prepared, expiration date and initials of preparer. Record the details of preparation in the LIMS.

7.3 Wash Solution -2% HNO₃, 0.5% HCl: Dilute 80mL of Optima Nitric Acid, 20mL of HCL to 4L with nanopure water. .

7.4 Internal Standard Solution –. Agilent Internal standard prepared using 50uL Ge, 500uL Sc, 50uL Rh, 500uL Bi, and 500uL Te, (prepare from single element solutions) to 500mL with 2%HNO₃/5%HCL.

10,000ppm Germanium – Environmental Express – Catalog # 10M20-3

10,000ppm Rhodium – Environmental Express – Catalog # HP10M44-2

1,000ppm Scandium- Environmental Express- Catalog#HP100048-1

1,000ppm Tellurium- Environmental Express- Catalog#HP100056-2

1,000ppm Bismuth- Environmental Express- Catalog#HP10006-1

TritonX-100 – VWR – Catalog#AAA16046-AP

Methanol – VWR – Catalog#BDH1135-4LG

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7.5 Calibration Standards and Standard Reference Materials (SRMs) – Standards are prepared pipetting specific amounts of concentrated stock standards into volumetric flasks and diluting to volume with 2% HNO₃ and .5%HCl. The solution names as they appear in the analysis are in parenthesis. The name in parenthesis must be used exactly as it appears (including correct capitalization) in order to be recognized as a standard by the system software in the Sample ID in the Autosampler table.

7.5.1 Calibration Blank (Blank). 2%HNO₃/0.5%HCl.

7.5.2 Calibration standards for Agilent are prepared from a custom stock containing 10ppm(As, B, Ba, Be, Cd, Cr, Co, Cu, Li, Pb, Mn, Mo, Ni, Se, Sr, Ti, Th TI, U, V, Zn), 100ppm(Al/Fe) and 1000ppm (Mg, K, Na, Ca)) They are added to the calibration using stock standard Environmental Express Cat# HP6388-500 or equivalent.

See table below for the volume of stock used in the preparation of the calibration standards. All calibration standards have a final volume of 50mL.

Agilent

Calibration Standard Concentration in ppb	Amount of Stock used per 50mL of 2% nitric acid/.5%HCL
200/2ppmAl Fe/20ppm Ca, Mg, K, Na	1mL
100/1ppmAl, Fe/10ppm Ca, Mg, K, Na	.5mL
50/.5ppm Al, Fe/5ppm Ca, Mg, K, Na	.25mL
10/.1ppmAl, Fe/ 1ppm Ca, Mg, K, Na	50uL
2ppm 1ppm	1mL of 100ppb cal. Std. 0.5mL if 100ppb cal Std.
Optional high std.1 5ppm/50ppm Al Fe/500ppm Ca, Mg, K, Na	25mL
Optional High std. 2 10ppm/100ppm Al Fe/1000ppm Ca, Mg, K, Na	50mL

7.5.3 100ppb Initial Calibration Verification Solution (ICV): 0.5mL of 5ppm ICV solution diluted to 50mL with 2% nitric acid, 0.5%HCL solution. The ICV solution is prepared from one solution that is purchased through SCP Science Catalog#AQ0-010-382 or equivalent. . ICV must be prepared in the same acid matrix as the calibration standards. This solution is prepared from a source different than the calibration standards. The concentration should be near the midpoint of the curve.

7.5.4 Instrument Blank (BLK). Prepared in2% nitric acid/.5%HCL and laboratory DI water and used to flush the system between all samples and standards.

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- 7.5.5 Method Blank – an aliquot of deionized water that is subjected to the same preparation procedure as the samples including sample digestion if the sample matrix requires digestion. One method blank is assigned to each analytic batch. This blank is used to monitor possible contamination resulting from the preparation procedures.
- 7.5.6 Calibration Blank (ICB/CCB) - Prepared in 2% nitric acid/.5%HCL and laboratory DI water and used to flush verify the absence of carryover following each ICB/CCV.
- 7.5.7 100ppb Trace Metals CCV: 0.5mL of 10ppm standard solution diluted to 50mL with 2% nitric acid/.5%HCL solution.
- 7.5.8 Interference Check Solution A (ICSA): Prepared by adding 5mL of Inorganic Ventures (or equivalent) Custom Mix A to 50mL with 2% HNO₃/.5%HCL.
- 7.5.9 Interference Check Solution B (ICSB): Prepared by adding 5mL of Inorganic Ventures Custom Mix A and 0.5mL of Inorganic Ventures (or equivalent) Custom Mix B and C to a 50mL flask. The solution is then diluted with 2% HNO₃/.5%HCL to 50mL.
- 7.5.9.1 Method 6020B Spectral interference check (SIC) solutions - Prepare so as to contain known concentrations of interfering elements that will demonstrate the appropriate magnitude of interferences and provide an adequate test of any corrections. Chloride in the SIC solution provides a means to evaluate software corrections for chloride-related interferences such as ³⁵Cl¹⁶O⁺ on ⁵¹V⁺ and ⁴⁰Ar³⁵Cl⁺ on ⁷⁵As⁺. Iron is used to demonstrate adequate resolution of the spectrometer for the determination of manganese. Molybdenum serves to indicate oxide effects on cadmium isotopes. The other components are present to evaluate the ability of the measurement system to correct for various molecular-ion isobaric interferences. The SIC is used to verify that the interference levels are corrected by the data system within appropriate QC limits.

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Recommended SIC Solution Components and Concentrations

Component	Concentration (mg/L)
Al	100.0
Ca	100.0
Fe	100.0
Mg	100.0
Na	100.0
P	100.0
K	100.0
S	100.0
C	200.0
Cl	1000.0
Mo	2.0
Ti	2.0

7.5.9.1.1 Mixed Stock SIC Solutions - Prepare the SIC stock solutions using only ultra-pure reagents. They can be obtained commercially or prepared using the following procedures:

- Mixed SIC stock solution I - Prepare by adding 13.903g $\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$, 2.498g CaCO_3 (previously dried at 180°C for 1 hr), 1.000g Fe, 1.658g MgO , 2.305g Na_2CO_3 and 1.767g K_2CO_3 to 25 mL of reagent water. Slowly add 40mL of (50%) HNO_3 . After dissolution is complete, warm the solution to degas. Cool and dilute to 1-Liter with reagent water.
- Mixed SIC stock solution II - Prepare by slowly adding 7.444g 85% H_3PO_4 , 6.373g 96% H_2SO_4 , 40.024g 37% HCl , and 10.664g citric acid ($\text{C}_6\text{O}_7\text{H}_8$) to 100 mL of reagent water. Dilute to 1-Liter with reagent water.

7.5.9.1.2 Mixed Working SIC Solution - Prepare by combining 10.0mL of SIC stock solution I, 2.0 mL each of 100- $\mu\text{g}/\text{mL}$ titanium stock solution and 100- $\mu\text{g}/\text{mL}$ molybdenum stock solution, and 5.0mL of SIC stock solution II. Dilute to 100mL with reagent water. Prepare fresh weekly.

7.5.10 Tune/Optimization/Autolens Solutions: A custom stock of 1000ppb is diluted 5mL to 50mL to make a 100ppb solution of Ba, Co, Be, Cu, In, Mg, Rh, Ce, Pb, and U. Not all elements are used for each procedure, but the mix contains all necessary elements. The stock solution is purchased through Environmental Express, Catalog No., HP2047-A-250 or equivalent.

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- 7.6 The aqueous laboratory control standard (LCSW) or Laboratory Fortified Blank (LFB) is purchased with all analytes at a concentration of 100µg/mL. The LCSW is purchased from Ultra Scientific, Catalog Number ICUS-726 or equivalent.
 - 7.7 The solid laboratory controls standard (LCSS) is purchased through Inorganic Ventures ESC-6A and ESC-6B Inorganic Ventures or equivalent. ESC-6B contains Al, Ca, Fe, Mg, Na, and S at 1000ug/mL, , As, B, Ba, Be, Cd, Co, Cr, Cu, Li, Mn, Ni, P, Pb, Se, Sr, Th, Tl, U, V, and Zn at 100ug/mL. Ag at 20ug/mL. ESC-6A contains Mo, Sb, Si, Sn, and Ti at 100ug/mL.
 - 7.8 The aqueous and solid matrix spike and matrix spike duplicate are prepared from the standard in Section 7.6 and Section 7.7
 - 7.9 Deionized Water
 - 7.10 Method 6020B – If the determination of one or more metals using a non-aqueous solvent is required, then all standards and quality control samples must be prepared on a weight/weight basis in the non-aqueous solvent since the density of non-aqueous solvents is not uniform. Standards and quality control materials containing organometallic materials that are soluble in non-aqueous solvents are available from a variety of vendors.
- 8.0 PROCEDURE
- 8.1 Sample Preparation
 - 8.1.1 Digestion. All wastewater and drinking water samples and spikes that require a digestion follow SOP #340380, *Digestion of Metals and Trace Elements in Drinking Water and Wastes for ICP-AES and ICPMS by Microwave and Hot Block Digestion*. All RCRA samples must be digested using one of the following SOPs: #340388 for 3050B/3051 and #340389 for 3005A, 3010A and 3015.
 - 8.1.2 Internal Standards. The internal standard solution (100ppb each Sc, Te, Ge, Rh, and Bi) and sample solution are mixed in a mixing block prior to nebulization. The ratio of sample to internal standard is 4:1.
 - 8.2 Sample Analysis – Instructions vary slightly depending on the specific instrument being used for analysis. Consult the instrument operating guides for each instrument for the exact directions for each instrument.
 - 8.2.1 Pre-Start Checks: Turn on the computer and load the software. Initiate appropriate operating configuration of the instrument's computer according to the instrument manufacturer's instructions. Check the following:
 - 8.2.2 Vacuum pump oil - Examine the sight glasses of the vacuum pump. Oil should be no darker than a light brown color. If it is, change the oil in the pump according to the directions in the manufacturer's guide.

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- 8.2.3 Peristaltic pump tubing - Change the sample and internal standard tubing, spray chamber drain tubing and the rinse station tubing as needed. . Allow at least 1 hour for break-in period.
- 8.2.4 Wash solution level - The wash solution supply is maintained in a 4-liter carboy. (See 7.3). Ensure that there is sufficient volume present for the analytical sequence.
- 8.2.5 Nebulizer waste and rinse waste flow into one waste container.
- 8.2.6 Ar/O pressure - The argon supply pressure should be set at about 100psi. If the supply argon pressure falls below about 45psi, a safety interlock automatically shuts off the torch.
- 8.2.7 Liquid Ar level - Liquid argon is maintained in a bulk supply tank. This tank is refilled on a regular basis before the argon supply is exhausted and bleeding of the argon lines is not necessary.
- 8.2.8 Torch alignment – Torch alignment is done automatically during performance check.
- 8.2.9 Interface cones - Remove and inspect the outside of the sampling and skimmer cones around the orifice. Install a new set of cones if needed or clean the existing cones using the following procedure:

Carefully polish each cone with silver polish and cotton swabs dampened with deionized water. Rinse cones with deionized water and blow-dry with house air supply, being careful not to damage the cones. After the cones are fully dry, replace them in the instrument. Allow for conditioning of the cones with a solution containing all analytes needed.

The orifice should be circular and about 1mm in diameter. Examine the orifice periodically with a magnifier to determine if there are irregularities that may impair instrument performance. DO NOT use a cone with a significantly damaged tip.
- 8.2.10 Chiller temperature, pressure and water level - The temperature should be regulated at $15 \pm 1^{\circ}\text{C}$. Check the current temperature on the chiller to ensure it is within this range. Check the inlet cooling water pressure that must be between 55 and 60psi. Check to ensure that chiller water level is full. If it is not, fill with Polyclear 30.
- 8.2.11 Nebulizer - Check the nebulizer to be sure it is clean, the capillary is not clogged and it produces a smooth mist.
- 8.3 Lighting Torch and Warm-Up: After all pre-start checks pass inspection, perform the following steps:
 - 8.3.1 Torch Ignition - Click on the *Plasma* icon to open the *Instrument* window, and then click on the plasma on button to light the plasma. This takes a little over a minute to complete. (See instrument software guide)

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- 8.3.2 Warm-up: Instrument is allowed to warm-up 30minutes. Instrument has a timer to let you know when it is ready to move on to the next step.
- 8.3.3 Start-up Configuration- Once the analysis tubing is placed in the Agilent tune solution the Start-up configuration can be started. The instrument will then go through Torch Alignment, Axis/Resolution, EM settings, Plasma Correction, Standard Lenses tune, and Performance report. _.
- 8.4 Running Samples: After Warm-Up is complete, perform the following steps to collect a dataset.
- 8.4.1 Tune - Open Tune tab. Switch ISIS valve to tune. Monitor signal to ensure sample is reaching the detector. When the instrument achieves proper counts, stop the monitor and select the "semi auto tune: tab. (The instrument performs five replicates of tuning solution). The measured mass must be as follows:

Agilent:

Agilent monitored masses (No Gas)	Requirement
7	CPS RSD<5%
89	CPS RSD<5%
205	CPS RSD<5%
156/140	CPS Ratio<1.5%
70/140	CPS Ratio<3.0%

Agilent monitored masses (He)	Requirement
59	CPS RSD<5%
89	CPS RSD<5%
51/59	CPS Ratio<3%
75	CPS ~5 or less
78	CPS ~5 or less

- 8.4.2 Create New Dataset – Open template from the drive. Apply the proper run name for the day. (MMDDYYICPMS#).
- 8.4.3 Enter Sample ID's: Open the routine analysis workspace previously saved. Enter the sample ID's in the sample template previously created. Enter any necessary dilution factors in the column provided along with both beginning and ending volumes. Place all calibration standards, calibration check solutions, samples and QC in the Autosampler according to the template.
- 8.4.4 For initial and daily operation, calibrate the instrument according to the instrument manufacturer's recommended procedures using mixed calibration standard solutions and the calibration blank (see section 7.5). The concentration of the lowest standard must be at or below the reporting limit. A peristaltic pump

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must be used to introduce all solutions, samples, and internal standard to the nebulizer. (Agilent 7900 uses ISIS-3 pump) To allow adequate time for equilibrium to be reached in the plasma, aspirate all solutions for at least 30 seconds after the solution reaches the plasma before accumulating data. Use the average value from three replicate integration periods of the signal to be correlated to the analyte concentration. Flush the system with the rinse blank for a minimum of 60 seconds between each standard. The calibration line is generated by first order linear regression from a calibration blank and six calibration standards where each element is present in at least three concentration levels. Calibration acceptance criteria are described in section 10.5. For ICV/CCV/CCB/ICB/ICS requirements, frequency, see section 10.0.

NOTE: For Marathon sample analysis, simple linear curve fitting as identified in the instrument regression choices will be utilized when possible.

- 8.4.5 Begin Analyzing Samples: For Agilent instruments, in the mass hunter software select "sample list" and then choose the "unknown samples" tab. Once here, select "Start editing mode" and begin to add samples + instrument QC in the order desired for analysis. Assign appropriate vial location numbers as well as dilution factors to each sample in the table. Once completed, select "End editing mode" and analysis will begin.

The instrument performs sample analysis by executing 50 to 100 mass sweeps per replicate. Three replicates are utilized for an average result which must fall within a 20% RSD for the replicate values. If any sample or QC is found to have a concentration of >5x the RL and >20% RSD it must be evaluated for interference. If a matrix interferent is determined to be the cause, dilute the sample by 5x and re-analyze. Perform further dilutions if necessary.

- 8.4.6 Monitor Initial QC. Check the calibration blank Summary Report for signs of contamination and the rest of the calibration and initial QC Summary Reports for signs of problems.

NOTE: If analysis will not be complete until after hours, set to shutoff automatically when analysis is complete (Enable automatic shutoff in *Instrument* window. This can be done while the instrument is analyzing samples).

- 8.4.7 Instrument Shutdown - When batch is complete, MassHunter will auto export all data to the dataset and shut off plasma if selected. Otherwise, select turn off plasma to stop a run immediately. Turn off the computer monitor if it will not be used further that day.

9.0 DATA ANALYSIS AND CALCULATIONS

- 9.1 Sample data should be reported in units of mg/L for aqueous samples and mg/Kg dry weight for solid samples. The mean of three "reads" from the spectrometer is used to derive sample concentrations.

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- 9.2 For dissolved aqueous analytes, report the data generated directly from the instrument with compensation for sample dilution. Do not report analyte concentrations below the MDL.
- 9.3 For total recoverable aqueous analytes, multiply solution analyte concentrations by the dilution factor 0.5 when a 100mL aliquot is used to produce the 50mL final digestate volume, and report data. If a different aliquot volume other than 100mL is used for sample preparation, adjust the dilution factor accordingly. Account for any additional dilution of the prepared sample digestate required to complete the determination of any analytes exceeding 90% or greater of the LDR upper limit. Do not report data below the determined analyte MDL concentration or below an adjusted detection limit reflecting smaller sample aliquots used in processing or additional dilutions required by the analysis.
- 9.4 For analytes with RLs < 0.01mg/L, round the data values to the thousandth place and report analyte concentrations up to three significant figures. For analytes with RLs >/- 0.01mg/L round the data values to the hundredth place and report analyte concentrations up to three significant figures. Analyte concentrations for solids data should be rounded in a similar manner before dry weight corrections in Section 9.5 are performed.
- 9.5 For total recoverable analytes in solid samples, round the solution analyte concentrations (mg/L) as instructed in Section 9.4. Report the data up to three significant figures as mg/Kg dry-weight basis unless specified otherwise by the program or data user. Calculate the concentration using the equation below:

$$\text{Sample Conc. (mg/Kg) = dry-weight basis} = \frac{C \times V \times D}{W}$$

where: C = Concentration in extract (mg/L)
V = Volume of extract (L, 100mL = 0. 1L)
D = Dilution factor (undiluted = 1)
W = Weight in Kg of sample aliquot extracted (g x 0.001 = Kg)

- 9.6 Soil samples are reported on a dry weight basis. Soil samples must be processed using the Pace National SOP #340349, *Total Solids and/or Percent Moisture*. After a dry weight for each sample has been obtained, the calculations are performed as follows:

$$\% \text{ solids (S)} = \frac{DW}{WW} \times 100$$

where: DW = Sample weight (g) dried
WW = Sample weight (g) before drying

At Pace National, dry weight reporting conversions are performed by the LIMS data system.

- 9.7 Relative Standard Error (RSE – expressed as a percentage)

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$$RSE = 100 \times \sqrt{\frac{\sum_{i=1}^n \left[\frac{x'_i - x_i}{x_i} \right]^2}{(n - p)}}$$

where:

- x'_i = Measured amount of analyte at the calibration level i , in mass or concentration units
- x_i = True amount of analyte at calibration level i , in mass or concentration units
- p = Number of terms in the fitting equation (average = 1, linear = 2, quadratic = 3)

- 9.8 See the current Quality Assurance Manual for equations associated with common calculations.

10.0 QUALITY CONTROL AND METHOD PERFORMANCE

- 10.1 All analysts must meet the qualifications specified in SOP #030205, *Technical Training and Personnel Qualifications* before approval to perform this method. Analysts must complete an initial demonstration of proficiency before being approved to perform this method. Continuing proficiency must be demonstrated using proficiency testing, laboratory control sample analysis and/or MDL studies. Method performance is assessed per analyst. Updated method performance records are filed and stored in a central location within the department.

- 10.2 Use the designated run log to record batch order and standards/reagents used during analysis. See SOP #030201, *Data Handling and Reporting*.

- 10.3 Preparation Batches:

Batches are defined as sets of 1 - 20 samples for EPA 6020/6020A/6020B and 1 – 10 samples for EPA 200.8. Preparation batch analysis must include the following: 1 method blank, 1 Laboratory Control Sample (LCS), 1 Laboratory Control Sample Duplicate (LCSD), 1 Matrix Spike/Spike Duplicate (MS/MSD) pair. All batch information must be maintained in the preparation documentation assigned to the department.

Note: EPA 200.8 Wastewater samples may be batched in sets of 1-20. If >10 samples are prepared for EPA 200.8 Wastewater, they must include two sets of MS/MSD.

- 10.4 Supporting Analytical Studies:

- 10.4.1 Instrument Detection Limits (IDL) Studies - IDLs in µg/L can be determined as the mean of the calibration blank results plus three times the standard deviation of 7 replicate analyses of the solution. Use zero for the mean if the mean is determined to be a negative value.

IDLs must be verified quarterly^{14.2, 14.3} or when major instrumentation change occurs.

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10.4.1.1 Method 6020B - IDLs in µg/L can be estimated as the mean of the blank result plus three times the standard deviation of 10 replicate analyses of the reagent blank solution. Use zero for the mean if the mean is negative. Each measurement should be performed as though it were a separate analytical sample (i.e., each measurement must be followed by a rinse and/or any other procedure normally performed between the analysis of separate samples). IDLs should be determined at least once using new equipment, after major instrument maintenance such as changing the detector, and/or at a frequency designated by the project. An instrument log book should be kept with the dates and information pertaining to each IDL performed.

10.4.2 Linear Dynamic Range (LDR) Studies – Linear dynamic ranges are established for each instrument to allow for quantitation above the highest level of calibration without qualification. ICPMS instruments are known to remain linear at high levels, but each upper limit of linearity is based on the target analyte being measured and the routine instrument operating conditions.

To perform a linear dynamic range study, the instrument must be calibrated normally as used with client field samples. The LDR is determined by the analysis of a minimum of three, but preferably five, different increasing concentrations of standards containing each target analyte across a range. One concentration should be near the expected upper linear range for each analyte. The highest concentration, where the instrument calibration remains linear, is determined when the observed concentration of the increasing standards is no more than 10% below the expected concentration of the analyte. If more than a 10% deviation exists, the instrument is proven to no longer be linear at that value for that analyte. The upper linear range is therefore the next lower concentration of standards used in the determination. Samples quantitated above that upper determined LDR require dilution to quantitate within the proven linear range of the instrument.

LDR studies must be determined initially then verified semi-annually^{14.4} or when major instrumentation change occurs.

Method 6020B – The linear range must be analyzed in the same instrument run as the calibration they are associated with, but may be analyzed anywhere within that run. If a linear range standard is not analyzed for any specific element, the highest standard in the calibration becomes the linear range.

10.4.3 Method Detection Limits – See also Pace National SOP #030206, *Method Detection Limits (MDL), Limits of Detection (LOD) and Limits of Quantitation (LOQ)*.

MDL studies are required annually or when instrumentation change occurs, such as one in the torch, nebulizer, injector, or plasma conditions. Method detection

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limit studies are performed on blank matrices most closely matching field sample matrices.

10.4.4 Proficiency Testing (PT) – See also Pace National SOP #030212, *Proficiency Testing Program*. Proficiency testing is performed in the metals department for ICPMS in support of environmental analyses. Environmental PTs are performed semi-annually for Water Supply (Safe Drinking Water Act), Water Pollution (Clean Water Act), and soils (RCRA) testing.

10.5 Instrument Tune –The amu of each measured mass must be as follows:

All ICP-MS	Low limit	High limit
Mg 24	23.9	24.1
Mg 25	24.9	25.1
Mg 26	25.9	26.1
In 115	114.9	115.1
Pb 206	205.9	206.1
Pb 207	206.9	207.1
Pb 208	207.9	208.1
Be 9	8.9	9.1
Co 59	58.9	59.1

10.5.1 EPA 200.8 - EPA Method 200.8 requires a minimum of 5 replicates of the tuning solution yielding a relative standard deviation of <5% of the absolute signal^{14.3}. EPA tune check should be less than 0.75amu at 5% peak height

10.5.2 **SW-846 6020 & 6020A** - EPA Methods 6020 and 6020A require a minimum of 4 replicates of the tuning solution yielding a relative standard deviation of <5% of the absolute signal^{14.2, 14.4}. EPA tune check should be less than 0.9amu at 10% peak height.

10.6 Initial Calibration - Run a calibration curve on a daily basis that employs a minimum of a calibration blank and six standards where each element is present in at least three of the standard levels. The concentration of the lowest standard must be at or below the reporting limit. Corrective actions for failures can be found in section 11.4.

10.6.1 EPA 200.8 & 6020/6020B - The correlation coefficient for the each analyte in the calibration curve lines must be 0.995 or better for EPA Methods 200.8 and 6020^{14.2, 14.3}.

NOTE: For Method 6010D - Relative Standard Error (see Section 9.7) may be used as an alternative to r or r^2 , and should be < 20%. If a multipoint calibration is used the low standard must be at or below the LLOQ. Inversely weighted linear regressions are recommended in order to minimize curve fitting errors at the low end of the calibration curve.

10.6.2 **SW-846 6020A** - The correlation coefficient for each analyte in the calibration curve must be 0.998 for EPA 6020A^{14.4}.

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NOTE: For Wisconsin, the unspiked ICSEA elements are evaluated to LOQ.

- 10.7 Method Blank – A method blank is analyzed per each batch of field samples. The results obtained for the method blank must be less than the method detection limit ($<1/2$ RL for DOD), and not less than 2X the negative RL for all analytes. Corrective actions for failures can be found in section 11.5.
- 10.7.1 Method 6020B – The method blank is considered to be acceptable if target analyte concentrations are less than $\frac{1}{2}$ the LLOQ or are less than project-specific requirements.
- 10.8 Initial Calibration Verification (ICV/ICVLL) - Verify the initial instrument standardization by analyzing appropriate check standards following calibration.
- 10.8.1 **EPA Methods 6020 & 200.8** - The mid-level ICV standard recovery results must be $\pm 10\%$ of the true value for EPA methods 6020 and 200.8.
- 10.8.2 EPA Method 6020A only - The mid-level ICV standard recovery results must be $\pm 10\%$ of the true value for EPA methods 6020A. The low-level ICV standard recovery results should be within historical laboratory accuracy limits.
- 10.8.3 Method 6020B Low-level Readback or Verification - For a multi-point calibration, the low level standard should quantitate to within 80-120% of the true value. For a single point calibration, a standard from the same source as the calibration standard and at the LLOQ is analyzed and should recover within 80-120% of the true value.
- 10.8.4 Method 6010D Mid-level Readback or Verification - For a multi-point calibration, the midlevel standard should quantitate to within 90-110% of the true value. For a single point calibration, a standard from the same source as the calibration standard and at the midpoint of the linear range is analyzed and should recover within 90-110% of the true value.
- 10.8.5 Corrective actions for failures can be found in section 11.6.
- 10.9 Laboratory Control Sample (LCS)/Laboratory Control Sample Duplicate (LCSD) – An LCS/LCSD is analyzed with each batch of samples. The acceptance range for water samples is $\pm 15\%$ of the target concentration for 200.8 and $\pm 20\%$ for 6020. If an LCS/LCSD pair is analyzed, the RPD of each analyte must be $\leq 20\%$.
- 10.9.1 **For EPA Method 200.8** – The acceptance range for water samples is $\pm 15\%$ of the target concentration. The RPD for each analyte in the pair must be $\leq 20\%$.
- 10.9.2 **For SW-846 Method 6020/6020A/6020B** – The acceptance range for water samples is $\pm 20\%$ of the target concentration. The RPD for each analyte in the pair must be $\leq 20\%$.
- 10.10 Matrix Spike (MS)/Matrix Spike Duplicate (MSD) – An MS/MSD pair is analyzed with each batch of samples. Acceptance ranges for accuracy of the MS and MSD are $\pm 30\%$ of the target analyte concentration. The RPD of the pair must be $\leq 20\%$.

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- 10.10.1 **For EPA Method 200.8** – The acceptance range for water samples is $\pm 30\%$ of the target concentration. The RPD for each analyte in the pair must be $\leq 20\%$.
- 10.10.2 **For SW-846 Method 6020/6020A/6020B** – The acceptance range for water samples is $\pm 25\%$ of the target concentration. The RPD for each analyte in the pair must be $\leq 20\%$.
- 10.11 Continuing Calibration Verification (CCV) – Verify the on-going instrument standardization by analyzing appropriate check standards during the sequence. Instrument calibration acceptability is demonstrated after every 10 samples and at the end of the analytical run using the CCV that must meet the following criteria:
- 10.11.1 **For SW-846 Method 6020/6020B and EPA Method 200.8** – Continuing Calibration Verification (CCV) recovery must be within $\pm 10\%$ of the target concentration. Instrument calibration verification is achieved by analyzing both a CCV standard and a CCB (inst. blank).
- 10.11.2 **For SW-846 Method 6020A** - Instrument calibration verification for method 6020A is achieved by analyzing both a mid-level CCV standard, a low-level CCV standard, and a CCB (instrument blank). The mid-level Continuing Calibration Verification (CCV) recovery must be within $\pm 10\%$ of the target analyte concentration. The low-level Continuing Calibration Verification (LLCCV) recovery should be within historical laboratory accuracy limits.
- 10.12 Initial/Continuing Calibration Blank (ICB/CCB) – An ICB/CCB is analyzed following each ICV/CCV. The results of each ICB/CCB must be $< \text{RL}$ for each analyte being reported or $< 1/2 \text{ RL}$ for DOD.
- NOTE:** Calibration blanks analyzed in conjunction with DOD QSM analyses for compliance must be assessed at the established LOD. If the concentration of target analytes is above the LOD, then re-analysis is required or the data must be qualified as contained a possible high bias (see Attachment III).
- NOTE:** Method 6020B - If a multi-level calibration is used, an ICB is analyzed immediately after the calibration (or after the ICV) and must not contain target analytes above half the LLOQ. If a single point calibration is used, the calibration is forced through the ICB, but a second ICB is analyzed as a check and must not contain target analytes above half the LLOQ. If the ICB consistently has target analyte concentrations greater than half the LLOQ, the LLOQ should be re-evaluated.
- Note:** West Virginia requires all Calibration Blank results to be $< \text{MDL}$. If the concentration of target analytes is above the MDL, then re-analysis is required or the data must be qualified as contained a possible high bias
- 10.13 Interference Check Standard (ICSA/ICSAB) – Analyzed at the beginning of the analytical sequence or at least once every 12 hours^{14.2, 14.4}. Acceptance criteria is determined using historical data.

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10.13.1 Method 6020B - Results for the unspiked elements in the SIC solution should be less than 2 times the LLOQ. Note that it may not be possible to obtain SIC spiking solutions that are completely free of the unspiked elements. If the presence and concentration of an unspiked element can be confirmed via vendor documentation and/or determination of multiple isotopes of the element in the correct ratios, the concentration actually present may be subtracted from the determined value prior to comparing to the LLOQ limits.

Note: The unspiked elements in the ICSA are not evaluated by the data capture software. The instrument analyst evaluates if the unspiked elements are <2 times the LLOQ for the ICSA

10.14 Post Digestion Spike – One post digestion spike must be analyzed for each batch of samples analyzed per the definition of a batch found in section 10.3. The recovery for the target analyte should be $\pm 25\%$ of true value.

10.14.1 **For SW-846 Method 6020A** - The recovery for the target analyte should be $\pm 20\%$ of true value.

10.14.2 **For SW-846 Method 6020 & 6020B** - The recovery for the target analyte should be $\pm 25\%$ of true value.

10.14.3 **For EPA Method 200.8** – Post digestion spikes are not required by the published method.

10.15 Serial Dilution - One serial dilution must be analyzed for each batch of samples analyzed per the definition of a batch found in section 10.3.

10.15.1 **For SW-846 Method 6020 & 6020A** - The serial dilution should recover with <10% difference from the parent sample.

10.15.2 **For SW-846 Method 6020** - The recovery for the target analyte should be $\pm 25\%$ of true value.

10.15.3 **For EPA Method 200.8** – Serial Dilutions are not required by the published method.

10.15.4 Method 6020B – If the analyte concentration is within the linear range of the instrument and sufficiently high (minimally, a factor of 25 times greater than the LLOQ), an analysis of a 1:5 dilution should agree to within $\pm 20\%$ of the original determination. If not, then a chemical or physical interference effect must be suspected. The matrix spike is often a good choice of sample for the dilution test, since reasonable concentrations of most analytes are present. Elements that fail the dilution test are reported as estimated values.

10.16 Internal Standards -

For SW-846 Method 6020A/6020B - If the intensity of any internal standard in a sample falls to below 70% of the intensity of that internal standard in the initial calibration standard, a significant matrix effect must be suspected. Ensure that the instrument has

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not drifted by observing the internal standard intensities in the nearest clean matrix (i.e. calibration blank, method blank, etc.). If the low internal standard intensities are also seen in the nearest blank, terminate the analysis, correct the problem, recalibrate, verify the new calibration and re-analyze the affected samples. If drift has not occurred, matrix effects need to be removed by dilution of the affected sample. The sample must be diluted fivefold (1+4) and re-analyzed with the addition of appropriate amounts of internal standards. If the first dilution does not eliminate the problem, this procedure must be repeated until the internal standard intensities in the sample rise to the minimum 70% limit. Reported results must be corrected for all dilutions.

For SW-846 Method 6020 - If the intensity of any internal standard in a sample fails to fall between 60% to 120% of the intensity of that internal standard in the initial calibration standard, a significant matrix effect must be suspected. Ensure that the instrument has not drifted by observing the internal standard intensities in the nearest clean matrix (i.e. calibration blank, method blank, etc.). If the low internal standard intensities are also seen in the nearest blank, terminate the analysis, correct the problem, recalibrate, verify the new calibration and re-analyze the affected samples. If drift has not occurred, matrix effects need to be removed by dilution of the affected sample. The sample must be diluted fivefold (1+4) and re-analyzed with the addition of appropriate amounts of internal standards. If the first dilution does not eliminate the problem, this procedure must be repeated until the internal standard intensities in the sample rise to the acceptable range. Reported results must be corrected for all dilutions.

The intensity levels of the internal standards for the calibration blank and the interference check standards must agree within 20% of the intensity level of the internal standard of the original calibration solution. If they do not, terminate the analysis, correct the problem, re-calibrate the instrument, verify the new calibration and re-analyze the affected samples.

For EPA Method 200.8 - All internal standard responses must be within 60- 120% of the original response of the calibration blank. If the internal standard response is not within this window, dilute the sample 2X and re-analyze.

- 10.17 Replicate Criteria - The instrument performs sample analysis by executing 10 mass sweeps per replicate. Three replicates are utilized for an average result. The RSD for the replicates must be reviewed to ensure consistency of the scans and to ensure that the sample digestate was accurately sampled. High RSD values can result from low target analyte concentrations and from insufficient uptake of the digestate during the autosampler cycle. If an interference is determined due to the viscosity of the digestate, dilute the digestate by 2X and re-analyze. Perform further dilutions, as necessary.
- 10.18 Reporting Limit Verification (RLV) – The reporting limit verification when analyzed must recover within $\pm 50\%$ of the target concentration for the standard.

STATE NOTE: For all samples analyzed from Minnesota, the reporting limit must be verified at least monthly, with each new initial calibration, or when there has been significant change to the instrument (column replacement, cleaning source, etc.) whichever is more frequent. The reporting limit

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verification can be performed by either re-injecting the low standard or by re-processing the low standard that was analyzed in the calibration curve. The reporting limit verification (RLV) must recovery within $\pm 40\%$ of the expected concentration. If this criterion is not met, the RLV may be re-analyzed once, instrument maintenance can be performed, a higher concentration standard can be injected, or a new calibration curve must be generated. If a higher concentration standard is utilized, the reporting limit must be raised to the higher level verified.

- 10.19 Any sample analyte responses that are beyond 90% of the linear dynamic range of the instrument must be diluted and re-analyzed.
- 10.20 Lower Limit of Quantitation (LLOQ) – When analyzing samples according to Method 6020B, the LLOQ is initially verified by the analysis of at least 7 replicate samples, spiked at the LLOQ and processed through all preparation and analysis steps of the method. The mean recovery and relative standard deviation of these samples provide an initial statement of precision and accuracy at the LLOQ. In most cases the mean recovery should be $\pm 35\%$ of the true value and RSD should be $< 20\%$. In-house limits may be calculated when sufficient data points exist. Monitoring recovery of LLOQ over time is useful for assessing precision and bias.
- 10.20.1 Ongoing LLOQ verification, at a minimum, is on a quarterly basis to validate quantitation capability at low analyte concentration levels. This verification may be accomplished either with clean control material (e.g., reagent water, method blanks, Ottawa sand, diatomaceous earth, etc.) or a representative sample matrix (free of target compounds). Optimally, the LLOQ should be less than the desired regulatory action levels based on the stated project-specific requirements.
- 10.21 If these criteria are not met, corrective actions are found in section 11.0.

11.0 DATA VALIDATION AND CORRECTIVE ACTION

- 11.1 All data must undergo a primary review by the analyst. The analyst must check the performance of the initial calibration, interference checks, mid-point check standard, continuing calibrations, and blanks to ensure that they meet the criteria of the method. The analyst should review any sample that has quantifiable compounds and make sure that they have been confirmed. The analyst must also verify that reported results are derived from quantitation between the MDL and 90% of the LDR. All calculations must be checked (any dilutions, %solids, etc.). Data must be checked for the presence or absence of appropriate flags. Comments should be noted when data is flagged. Check for carryover (see Section 11.2.8 of this SOP).
- 11.2 All data must undergo a second analyst review. The analyst checking the data must check the performance of the initial calibration, interference checks, mid-point check standard, continuing calibrations, and blanks to ensure that they meet the criteria of the method.

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- 11.2.1 The analyst should look at any sample that has quantifiable compounds and check the RSD of triplicate spectrometer reads.
- 11.2.2 All calculations must be checked.
- 11.2.3 Blanks must meet SOP requirements.
- 11.2.4 Quality control criteria should be checked for the LCS, LCSD, MS, and MSD.
- 11.2.5 Data must be checked for the presence or absence of appropriate flags. Comments should be noted when data is flagged.
- 11.2.6 See SOP #030201, *Data Handling and Reporting*.
- 11.2.7 See SOP #030208, *Corrective and Preventive Action*.

STATE NOTE: For work performed in support of the NC Department of Environment and Natural Resources (15A NCAC 02H .0805 (a) (7) (I)). For target analytes quantitated by ICP or ICPMS, a series of at least three standards must be analyzed along with each group of samples. The concentrations of these standards must bracket the concentration of the analytes in the field samples analyzed. Samples with target analyte concentrations above the highest level of calibration must be diluted to quantitate analytes within the calibration range. The use of the dynamic linear range studies to validate analyte/instrument calibration linearity must not be used for NC sample analysis.

- 11.2.8 Assess carryover in each analysis by evaluating each run for large concentrations (i.e., exceeding the calibration range) of target analytes. If a large concentration detection is identified, then carefully scrutinize sequential analyses for any detected concentrations of this analyte (i.e., potential carryover). When carryover is suspected, a confirmation analysis is required.
- 11.3 Instrument Tune – The tuning criteria must be met before calibration/verification and any field samples are analyzed. If the tuning criteria fail, re-analyze the tuning sequence once. If the failure persists, prepare a fresh tuning solution, perform instrument adjustments as needed, and re-analyze the solution. If the tuning solution continues to fail, see the department supervisor for assistance.
- 11.4 Initial Calibration – If the acceptance criteria in section 10.5 are not met, locate and correct the cause and re-analyze the standards until the requirement is met. Corrections can include: re-preparation of the standards, instrument maintenance, manufacturer service, etc. Consult the department supervisor, if needed.
- 11.5 Method Blank – In most cases where the method blank contains target analyte, the entire sample batch requires re-prep and re-analysis.

State Note: For Wisconsin samples, the method blank must not contain analytes more negative than the MDL value. If target analytes are more negative than the MDL, the instrument must be recalibrated or a new LOD study performed.

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General guidelines for qualifying sample results with regard to method blank quality are as follows:

- If the method blank concentration is less than the MDL and sample results are greater than the RL, then no qualification is required.
- No qualification is necessary when an analyte is detected in the method blank but not in the associated samples.
- If the concentration in a sample is more than ten times the concentration in the method blank, then no qualification is required.
- If the method blank concentration is greater than the MDL but less than the RL and sample results are greater than the MDL, then qualify associated sample results to indicate that analyte was detected in the method blank.
- If the method blank concentration is greater than the RL, further corrective action and qualification is required. An analyst should consult their supervisor for further instruction.

NOTE: Method 6020B - If the method blank fails to meet the necessary acceptance criteria, it should be reanalyzed once. If still unacceptable, then all samples associated with the method blank must be re-prepared and re-analyzed along with all other appropriate analysis batch QC samples. If the method blank results do not meet the acceptance criteria and reanalysis is not practical, then the laboratory should report the sample results along with the method blank results and provide a discussion of the potential impact of the contamination on the sample results. However, if an analyte of interest is found in a sample in the batch near its concentration confirmed in the blank, the presence and/or concentration of that analyte should be considered suspect and may require qualification.

- 11.6 Initial Calibration Verification (ICV) – If any analyte is outside of stated criteria, re-analyze the ICV once. If the ICV is still beyond acceptance ranges, corrective action must be taken. Recalibrate the instrument and re-analyze the ICV.
- 11.7 Laboratory Control Sample (LCS)/Laboratory Control Sample Duplicate (LCSD) – Failures are indicative of analytical problems. If failures occur, halt analysis, and begin corrective action. Rejected LCS and/or LCSD result in a re-prep and re-analysis of the entire batch.
- 11.8 Matrix Spike (MS)/Matrix Spike Duplicate (MSD) – Any analyte outside of stated criteria are flagged with J5 or J6.
- 11.9 Continuing Calibration Verification (CCV) – If a calibration verification standard is not within the acceptable QC criteria and analysis of a second consecutive (immediate) calibration verification fails to produce results within acceptance criteria, corrective actions shall be performed. The laboratory shall demonstrate acceptable performance after the final round of corrective action with two consecutive calibration verifications, or a new initial instrument calibration shall be performed.

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- 11.10 Initial/Continuing Calibration Blank (ICB/CCB) – If an instrument blank contains an amount of target analyte, but all samples are non-detected, the data may be reported with a “B4” flag. If an instrument blank contains an amount of target analyte, but the samples contain analyte at a level that is 10 times the level present in the instrument blank, the data may be reported with a “B5” flag.
- 11.11 Interference Check Standard (ICSA/ICSAB) – Failures in the ICSA/ICSAB require reanalysis. If the failure persists, review the data for possible interferences, review the background corrections, recalibrate if necessary and reanalyze all samples associated with the initial failure.
- 11.12 Serial Dilution - If the post spike result is acceptable, no action is taken. If both the post spike and the serial dilution are out, the results are flagged with O1.
- 11.13 Internal Standards -
- 11.13.1 **For SW-846 Method 6020A/6020B:** Internal standard intensities in the ICV, CCV, and CCB must be within $\pm 20\%$ of the original calibration. Internal Standard intensities in the samples must be between 70-120% of the original calibration solution. If the internal standard intensity is not within the 70-120% range, dilute the sample 5X and re-analyze. Repeat the procedure until the sample internal standard intensities fall within the 70-120% window.
- For EPA Methods 6020/200.8:** All internal standard responses must be within 60-120% of the original response of the calibration blank. If the internal standard response is not within this window the data must be rejected.
- 11.13.2 Changes in internal standard intensity should not be more than 10% from one sample to the next unless the change is an attenuation that appears to be due to matrix.
- 11.13.2.1 Samples containing a significant amount of dissolved solids depress the instrument sensitivity. Although the constituents of a sample also play a role, generally the higher the dissolved solids the more the signal is depressed. Samples containing 0.5% solids attenuate the signal by about half. This effect should be approximately the same for internal standards as well. It is normal to observe a sudden decrease in the internal standard intensity for samples known to contain or are suspected of containing significant dissolved solids.
- 11.13.2.2 Generally, sudden increases in internal standard intensity are observed only after the signal was attenuated by a matrix effect. Should other increases in internal standard intensity be observed, it is likely because the samples are contaminated with the internal standards. This can be recognized because the intensities of the different internal standards do not move proportionately.
- 11.13.2.3 The internal standard intensities must be checked to see that the above criteria are met. If these criteria are not met, the samples must be reanalyzed. If the reason a sample does not meet these criteria

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appears to be excessive attenuation due to the matrix, the sample must be diluted and reanalyze.

- 11.14 Reporting Limit Verification (RLV) – When required, the reporting limit verification must be analyzed. If the acceptance criteria are not met, the RLV may be re-analyzed once, instrument maintenance can be performed, a higher concentration standard can be injected, or a new calibration curve must be generated. If a higher concentration standard is utilized, the reporting limit must be raised to the higher level verified.

METHOD NOTE: For EPA Method 6020A analyses, an RLV must be performed periodically. This RLV must be carried through the entire preparation and analytical procedure. Recoveries must be within $\pm 30\%$ of the expected true value for each analyte being reported using that method.

STATE NOTE: Drinking water samples analyzed using this procedure for compliance cannot be qualified.

12.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

- 12.1 The EPA requires that laboratory waste management practice to be conducted consistent with all applicable federal and state laws and regulations. Excess reagents, samples and method process wastes must be characterized and disposed of in an acceptable manner. See *Pace National Waste Management Plan*.
- 12.2 See SOP #030302, *Environmental Sustainability & Pollution Prevention*.

13.0 METHOD MODIFICATIONS/CLARIFICATIONS

- 13.1 Not all analytes listed as analyzed in this procedure are approved within the published EPA methods; however sufficient information is present within the laboratory to demonstrate acceptable performance of these analytes using this procedure.
- 13.2 Modifications to this method are noted in the body of the text as state notes. Compliance analyses performed in conjunction with specific state requirements must be performed as noted within the specific state(s) note listed.
- 13.3 Adjustments to the concentrations of standards/spiking solutions, standards providers, and quality control are subject to change to better meet client/project/regulatory needs or to improve laboratory method performance.
- 13.4 If a modification to the procedure is required to analyze a sample beyond those specified in the procedure as state notes, it must be properly documented. Any modification to the method is listed in the test report's case narrative, and the method is reported as modified.
- 13.5 Superscripts are provided where necessary to indicate the reference in Section 14.0 where the requirement/information can be found. Subscripts noted identify the most frequent/restrictive cases, but requirements may also be included at different frequencies/conditions in other references noted in section 14.0.

14.0 REFERENCES

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- 14.1 Agilent ICP/MS User's Guides.
- 14.2 *Inductively Coupled Plasma – Mass Spectrometry*, SW-846 Method 6020, Revision 0, September 1994.
- 14.3 *Inductively Coupled Plasma – Mass Spectrometry*, SW-846 Method 6020A, Revision 1, February 2007.
- 14.4 *Inductively Coupled Plasma – Mass Spectrometry*, SW-846 Method 6020B, Revision 2, July 2014.
- 14.5 *Determination of Trace Elements in Waters and Wastes by Inductively Coupled Plasma – Mass Spectrometry*, EPA Method 200.8, Supplement I, Revision 5.4, May 1994.
- 14.6 *Determination of Trace Elements in Waters and Wastes by Inductively Coupled Plasma – Mass Spectrometry*, EPA Method 200.8, Revision 5.5, October 1999, EPA-821-R-99-017.
- 14.7 *Chapter 3: Inorganic Analytes*, SW-846, Revision 4, February 2007.
- 14.8 *Identification of Test Procedures*, 40 CFR Part 136.3
- 14.9 *Inorganic Chemical Sampling and Analytical Requirements*, 40 CFR Part 141.23
- 14.10 *Department of Defense (DOD) Quality Systems Manual (QSM) for Environmental Laboratories*, Version 5.0, July 2013.

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Attachment I: Revision History

Current Version:

Version	Date	Description of Revisions
19	7/27/18	Update in response to WI audit. Updated logo. Added State note to section 11.5

Superseded Versions:

This document supersedes the following:

Version	Date	Description of Revisions
0	1/18/00	Origination
1	8/22/00	
2	9/10/01	
3	12/6/01	
4	4/11/03	
5	2/9/04	
6	4/16/04	
7	8/3/04	
8	10/15/05	Added batch criteria for 6020 & 200.8 to prep. blank. Deleted definition for QC std. #1 and QC Std. #4. Added criteria for sample duplicate, post spike and serial dilution.
9	11/15/06	
10	10/29/08	Technical and Quality Review and update. Clarified QC corrective actions and standardized waste management language.
11	2/11/09	Technical and Quality Review and update. Clarified QC corrective actions and standardized waste management language. Added state notes. Ohio VAP approved 2/11/09.
12	7/13/11	Technical and Quality Review and update. Revised sections 1.2, 2.6, 2.8, 2.10, 2.11, 2.12, 2.13, 2.16, 2.18, 2.20, 2.22, 5.2, 5.3, 7.1, 7.4, 7.5.7, 8.4.1.2, 8.4.8, 9.7 through 9.10, 11.1 through 11.16, and 12.1; Added sections 2.23 through 2.28, 7.10, 10.4 through 10.19, 13.3 through 13.4 and 14.5; Changed all instrument references from ELAN 6100 to ELAN 9000 per instrument update.
13	5/10/12	Technical and Quality Review and update. Revised sections 1.2, 5.2, 5.3, 7.5.8, 7.5.9, and 11.11; Added sections 1.3.1, 2.29, 2.30, 4.7, 6.6, and 8.2.9; Removed sections 2.26.1, 2.26.2 and state note following section 11.16.
14	11/11/14	Technical and Quality Review and update. Revised sections 1.1, 1.2, 2.5, 2.6, 2.6.1, 2.8.1, 4.5, 5.3, 6.1, 6.2, 6.3, 7.1, 7.3, 7.4, 7.5, 8.2, 8.3.1, 8.3.6, 8.4.2, 8.4.8, 8.4.9, 10.3, 11.11, 11.16, 13.1 and 14.1; Added sections 1.4, 5.2.1, 5.2.2, 6.9, 10.4, 13.5 and 14.5 through 14.8; Removed section 2.17.

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Version	Date	Description of Revisions
15	2/4/2016	Technical and quality review and update. Header and signature block re-formatting. Revised Sections 1.0, 1.1, 1.2, 1.3.1, 2.8, 3.3, 4.7, 5.2, 6.1, 6.2, 6.3, 7.1, 7.3, 7.4, 7.5, 7.5.1, 7.5.2, 7.5.3, 7.5.4, 7.5.6, 7.5.7, 7.5.8, 7.5.9, 7.5.10, 7.7, 8.1.1, 8.1.2, 8.2.2, 8.2.3, 8.2.4, 8.2.6, 8.2.8, 8.2.9, 8.2.10, 8.3.1, 8.3.2, 8.3.3, 8.4, 8.4.2 through 8.4.7, 9.6, 9.7, 10.3, 10.7, 10.9, 10.9.2, 10.10.2, 10.12, 10.16, 11.2.7, 11.5, 11.13.1, 11.13.2.2, 11.14, 12.2, and Attachment II. Deleted Sections 2.3 through 2.14, 2.17 through 2.19, 2.22 through 2.24, 2.26 through 2.29, 5.2.1, 5.2.2, 6.6, 7.10, 8.2.3, 8.2.9, 8.2.11, 8.3.4 through 8.3.7, 8.4.1, 8.4.1.1, 8.4.1.2, 8.4.4 through 8.4.6, 9.8 through 9.11, 10.14, 11.11, 11.12, Attachment II,
16	10/27/2016	Technical and Quality review and update. Header and signature block re-formatting. Also, update per SC DHEC correspondence of 6/24/16. Revised Sections 6.2, 7.3, 7.4, 7.5.2, 7.5.3, 7.5.9, 7.6, 7.7, 8.1.1, 10.3, 10.4.2, 10.4.3, 10.5, 10.5.1, 10.5.2, 10.6.1, 10.6.2, 10.7, 10.8.3, 10.9.1, 10.9.2, 10.10.1, 10.10.2, 10.11.1, 10.12, 10.14.2, 10.16, 11.5, 11.9, 11.13.1, 14.1, 14.2, 14.5, 14.6, 14.7, 14.8, 14.9, 14.10, and Attachment II Table 2. Added Sections 2.8, 7.5.9.1, 7.5.9.1.1, 7.5.9.1.2, 7.10, 9.7, 10.4.1.1, 10.7.1, 10.8.4, 10.13.1, 10.15.4, 10.20, 10.20.1, 14.3, and 14.4.
17	4/10/2017	Technical and quality review and update. Revised Sections 3.1, 4.4, 7.3, 7.4, 7.5.1, 7.5.3, 7.5.9, 7.7, 8.4.5, 10.3, 10.12, and 10.13.1.
18	11/30/2017	Update in response to A2LA audit finding CAR2872. Updated Section 10.6.2 and Attachment II Table 5. Update in response to ESI audit finding CAR2851. Revised Sections 11.2.1 and added Section 11.2.8.

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Attachment III: DoD Requirements

1.0 Equipment/Instrument Maintenance

Instrument maintenance must be performed routinely to optimize instrument performance and improve chromatography. Commonly performed maintenance includes changing tubing, changing cones, changing nebulizer tips, changing oil, etc. A new calibration curve must be analyzed following any major maintenance performed on the analytical system.

2.0 Computer Hardware and Software

MassHunter 4.1, Version C.01.01, Agilent Technologies, Inc., 2014.

3.0 Troubleshooting

Table 1. ICP-MS Troubleshooting Guide		
Problem	Cause	Treatment
Low Sensitivity	Peristaltic Pump Issues	Adjust pumping rate
	Nebulizer Clogged	Clean or replace nebulizer
	Leaks	Check tube connector and sample lines for leaks
	Cone Changes	Ensure that the diameters and shapes of the holes in cones have not changed.
	Torch	Ensure the torch is positioned correctly
	Mass Axis	Ensure the mass axis is adjusted correctly
	Lens	Make sure the signal changes when the lens voltage is varied.
	Gas Purity	If the sensitivity is acceptable in the no gas mode and is too low in He or H2 mode, the problem may be caused by the purity level of the cell gas
Plasma Does Not Ignite	Spray Chamber	Ensure that samples do not collect in the spray chamber
	Air	Ensure that air is not mixed from connection points.

4.0 Other Requirements

- 4.1 All hardcopy laboratory notebooks must be reviewed by the Supervisor, or their designee, on a monthly basis.
- 4.2 If not self-explanatory (e.g., a typo or transposed number), corrections to technical and quality records shall also include a justification for the change.
- 4.3 A person performing a manual integration must sign and date each manually integrated chromatogram and record the rationale for performing manual integration. Electronic signatures are acceptable.

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- 4.4 The results of calibration and verification of support equipment must be within the specifications required of the application for which this equipment is used or the equipment must be removed from service until repaired. Calibration and verification records, including those of established correction factors, must be maintained. In the absence of method-specific requirements, the minimum requirements are as follows:

Table 2. Support Equipment Checks		
Performance Check	Frequency	Acceptance Criteria
Balance calibration check [Using two standard weights that bracket the expected mass]	Daily prior to use	Top-loading balance: $\pm 2\%$ or $\pm 0.02\text{g}$, whichever is greater Analytical balance: $\pm 0.1\%$ or $\pm 0.5\text{mg}$, whichever is greater
Verification of standard mass [Using weights traceable to the International System of Units (SI) through a NMI]	Every 5 years	Certificate of Calibration from ISO/IEC 17025 accredited calibration laboratory
Monitoring of refrigerator/freezer temperatures	Daily (i.e. 7 days per week) [use MIN/MAX thermometers or data loggers equipped with notification of out of control event capabilities if personnel not available to record daily]	Refrigerators: 0°C to 6°C Freezers: $\leq -10^{\circ}\text{C}$
Thermometer verification check [Using a thermometer traceable to the SI through an NMI] [Performed at two temperatures that bracket the target temperature(s). Assume linearity between the two bracketing temperatures.] [If only a single temperature is used, at the temperature of use]	Liquid in glass: Before first use and annually Electronic: Before first use and quarterly	Apply correction factors or replace thermometer
Volumetric labware	Class B: By lot before first use Class A and B: Upon evidence of deterioration	Bias: Mean within $\pm 2\%$ of nominal volume Precision: $\text{RSD} \leq 1\%$ of nominal volume (based on 10 replicate measurements)
Non-volumetric labware [Applicable only when used for measuring initial sample volume and final extract/ digestates volume]	By lot before first use or upon evidence of deterioration	Bias: Mean within $\pm 3\%$ of nominal volume Precision: $\text{RSD} \leq 3\%$ of nominal volume (based on 10 replicate measurements)

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Table 2. Support Equipment Checks

Performance Check	Frequency	Acceptance Criteria
Mechanical volumetric pipette	Quarterly	Bias: Mean within $\pm 2\%$ of nominal volume Precision: $RSD \leq 1\%$ of nominal volume (based on minimum of 3 replicate measurements) [Note: for variable volume pipettes, the nominal volume is the volume of use]
Glass microliter syringe	Upon receipt and upon evidence of deterioration	General Certificate of Bias & Precision upon receipt Replace if deterioration is evident
Drying oven temperature check	Daily prior to and after use	Within $\pm 5\%$ of set temperature
Water purification system	Daily prior to use	See method blank criteria given in Section 4.20 of this addendum

- 4.5 The expiration date of the prepared standard shall not exceed the expiration date of the primary standard. All containers must bear a preparation date.
- 4.6 To avoid preparing non-representative samples, the laboratory shall not “target” within a relatively small mass range (e.g., $1.00 \pm 0.01\text{g}$) because such targeting will produce non-representative subsamples if the sample has high heterogeneity. The laboratory shall not manipulate the sample material so the sample aliquot weighs exactly $1.00\text{g} \pm 0.01\text{g}$, as an example.
- 4.7 In the absence of project-specific requirements, the minimum standard data qualifiers to be used are:
- U Analyte was not detected and is reported as less than the LOD or as defined by the customer. The LOD has been adjusted for any dilution or concentration of the sample.
 - J The reported result is an estimated value (e.g., matrix interference was observed or the analyte was detected at a concentration outside the quantitation range).
 - B Blank contamination. The recorded result is associated with a contaminated blank.
 - N Non-target analyte. The analyte is a tentatively identified compound using mass spectrometry or any non-customer requested compounds that are tentatively identified.
 - Q One or more quality control criteria failed (e.g., LCS recovery, surrogate spike recovery, or CCV recovery).

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Additional data qualifiers may be used, or different letters or symbols to denote the qualifiers listed above, as long as they are appropriately defined and their use is consistent with project-specific requirements (e.g., QSM 5.0, the contract, and project-planning documents).

- 4.8 If the time of the sample collection is not provided, assume the most conservative time of day. For the purpose of batch processing, the start and stop dates and times of the batch preparation shall be recorded.
- 4.9 Each preparation method listed on the scope of accreditation must have quarterly LOD/LOQ verifications. However, not all possible combinations of preparation and cleanup techniques are required to have LOD/LOQ verifications. If LOD/LOQ verifications are not performed on all combinations, the laboratory must base the LOD/LOQ verifications on the worst case basis (preparation method with all applicable cleanup steps).
- 4.10 After each MDL determination, the laboratory must establish the LOD by spiking a quality system matrix at a concentration of at least 2 times but no greater than four times the MDL. This spike concentration establishes the LOD and the concentration at which the LOD shall be verified. It is specific to each suite of analyte, matrix, and method (including sample preparation). The following requirements apply to the initial LOD establishment and to the LOD verifications:
- The apparent signal to noise (S/N) ratio at the LOD must be at least three and the results must meet all method requirements for analyte identification (e.g., ion abundance, second column confirmation, or pattern recognition). For data systems that do not provide a measure of noise, the signal produced by the verification sample must produce a result that is at least three standard deviations greater than the mean method blank concentration. This is initially estimated based on a minimum of four method blank analyses and later established with a minimum of 20 method blank results.
 - If the LOD verification fails, then the laboratory must repeat the MDL determination and LOD verification or perform and pass two consecutive LOD verifications at a higher spike concentration and set the LOD at the higher concentration.
 - The laboratory shall maintain documentation for all MDL determinations and LOD verifications.
 - The DL and LOD must be reported for all analyte-matrix-methods suites, unless it is not applicable to the test or specifically excluded by project requirements.
- 4.11 The LOD shall be verified quarterly. In situations where methods are setup and used on an infrequent basis, the laboratory may choose to perform LOD verifications on a one per batch basis. All verification data will be in compliance, reported, and available for review.
- 4.12 For DoD, at a minimum, the LOQ shall be verified quarterly. In situations where methods are setup and used on an infrequent basis, the laboratory may choose to perform LOQ verifications on a one per batch basis.

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- 4.13 All initial instrument calibrations must be verified with a standard obtained from a second manufacturer prior to analyzing any samples. The use of a standard from a second lot obtained from the same manufacturer (independently prepared from different source materials) is acceptable for use as a second source standard. The concentration of the second source standard shall be at or near the midpoint of the calibration range. The acceptance criteria for the initial calibration verification must be at least as stringent as those for the continuing calibration verification.
- 4.14 Exclusion of calibration points without documented scientifically valid technical justification is not permitted.
- 4.15 The concentration of the CCV standard shall be greater than the low calibration standard and less than or equal to the midpoint of the calibration range.
- 4.16 All CCVs analyzed must be evaluated and reported. If a CCV fails, reanalysis or corrective actions must be taken.
- If a CCV fails, the laboratory can immediately analyze two additional consecutive CCVs (immediately is defined as starting a consecutive pair within one hour; no samples can be run between the failed CCV and the two additional CCVs). This approach allows for spurious failures of analytes to be reported without reanalysis of samples. Any corrective actions that change the dynamics of the system (e.g., clip column, clean injection port, run blanks) requires that all samples since the last acceptable CCV be reanalyzed.
 - Both of these CCVs must meet acceptance criteria in order for the samples to be reported without reanalysis.
 - If either of these two CCVs fail or if the laboratory cannot immediately analyze two CCVs, the associated samples cannot be reported and must be reanalyzed.
 - Corrective action(s) and recalibration must occur if the above scenario fails. All affected samples since the last acceptable CCV must be reanalyzed.
 - Flagging of data for a failed CCV is only appropriate when the affected samples cannot be reanalyzed. The laboratory must notify the client prior to reporting data associated with a failed CCV.
- 4.17 The results of all MS/MSDs must be evaluated using the same acceptance criteria used for the DoD LCS limits (see Addendum Tables 3 and 4) or project limits, if specified. If the specific analyte(s) are not available in the Addendum Tables 3 and 4, the laboratory shall use their LCS in-house limits (see the LIMS) as a means of evaluating MS/MSDs. The MS and MSD must be spiked with all reported analytes.
- 4.19 Surrogate spike results shall be compared with DoD LCS limits (see Addendum Tables 3 and 4) or acceptance criteria specified by the client. If these criteria are not available, the laboratory shall compare the results with its in-house statistically established LCS criteria (see the LIMS).
- 4.20 The method blank shall be considered to be contaminated if:

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- The concentration of any target analyte (chemical of concern) in the blank exceeds 1/2 the LOQ and is greater than 1/10th the amount measured in any associated sample, or 1/10th the regulatory limit, whichever is greater;
 - The concentration of any common laboratory contaminant in the blank exceeds the LOQ;
 - If a method blank is contaminated as described above, then the laboratory shall reprocess affected samples in a subsequent preparation batch, except when sample results are below the LOD. If insufficient sample volume remains for reprocessing, the results shall be reported with appropriate data qualifiers.
- 4.21 Sporadic Marginal Exceedances are not allowed for target analytes (chemicals of concern as identified by a project) without project-specific approval. Target analytes are considered those few analytes that are critical for the success of a project (such as risk drivers) where sporadic marginal exceedances cannot be allowed. Laboratories should consult with clients whenever long lists of analytes are requested for analysis to determine if marginal exceedances will not be allowed.
- 4.22 DoD considers the same analyte exceeding the LCS control limit two (2) out of three (3) consecutive LCS to be indicative of non-random behavior, which requires corrective action and reanalysis of the LCS.

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Table 3. LCS Control Limits – Method 6020 Solid Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
7429-90-5	Aluminum	919	101	7.7	78	124
7440-36-0	Antimony	1911	98.2	8.7	72	124
7440-38-2	Arsenic	3686	99.8	6	82	118
7440-39-3	Barium	2598	100.6	5	86	116
7440-41-7	Beryllium	2457	100.3	6.6	80	120
7440-42-8	Boron	581	101.1	9	74	128
7440-43-9	Cadmium	2893	99.6	5.4	84	116
7440-70-2	Calcium	835	102.2	5.4	86	118
7440-47-3	Chromium	2420	100.8	6	83	119
7440-48-4	Cobalt	2005	99.7	5.1	84	115
7440-50-8	Copper	2548	101.3	5.8	84	119
7439-89-6	Iron	1131	102.7	7.1	81	124
7439-92-1	Lead	3228	101	5.7	84	118
7439-93-2	Lithium	162	97.8	7.5	75	120
7439-95-4	Magnesium	868	101.6	7.1	80	123
7439-96-5	Manganese	1830	100.3	5.1	85	116
7439-97-6	Mercury	226	99.9	8.8	74	126
7439-98-7	Molybdenum	1188	98.1	5.1	83	114
7440-02-0	Nickel	2617	101.4	5.8	84	119
7440-09-7	Potassium	803	102.3	5.7	85	119
7782-49-2	Selenium	3104	99.2	6.6	80	119
7440-22-4	Silver	2488	100.1	5.9	83	118
7440-23-5	Sodium	818	102.2	7.7	79	125
7440-24-6	Strontium	676	101.7	8.9	75	129
7440-28-0	Thallium	2589	100.1	5.9	83	118
7440-29-1	Thorium	341	98.4	5.7	81	116
7440-31-5	Tin	886	101.3	6.6	82	121
7440-32-6	Titanium	512	100.2	5.7	83	117
7440-61-1	Uranium	833	101.1	6.1	83	120
7440-62-2	Vanadium	1677	99.1	5.7	82	116
7440-66-6	Zinc	2352	100.1	6.2	82	119

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TITLE: DETERMINATION OF METALS BY INDUCTIVELY COUPLED PLASMA MASS SPECTROSCOPY (ICP-MS) (EPA METHODS 6020, 6020A, 6020B & 200.8)

Table 4. LCS Control Limits – Method 6020 Water Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
7429-90-5	Aluminum	3145	100.6	5.4	84	117
7440-36-0	Antimony	5172	100.9	5.3	85	117
7440-38-2	Arsenic	6404	100.1	5.3	84	116
7440-39-3	Barium	4452	99.9	4.8	86	114
7440-41-7	Beryllium	4297	102	6.3	83	121
7440-42-8	Boron	1460	101.5	9.6	73	130
7440-43-9	Cadmium	5699	100.8	4.7	87	115
7440-70-2	Calcium	2085	102.3	5.2	87	118
7440-47-3	Chromium	5569	100.6	5.1	85	116
7440-48-4	Cobalt	3885	100.7	4.7	86	115
7440-50-8	Copper	5092	101.4	5.4	85	118
7439-89-6	Iron	3135	102.4	5.2	87	118
7439-92-1	Lead	6868	101.7	4.5	88	115
7439-93-2	Lithium	461	102.3	8	78	126
7439-95-4	Magnesium	2399	100.4	5.9	83	118
7439-96-5	Manganese	4330	101.1	4.7	87	115
7439-97-6	Mercury	328	97.2	9	70	124
7439-98-7	Molybdenum	2908	99.3	5.4	83	115
7440-02-0	Nickel	5095	100.8	5.3	85	117
7440-09-7	Potassium	2154	101.2	4.7	87	115
7782-49-2	Selenium	5797	100.1	6.7	80	120
7440-22-4	Silver	4956	100.8	5.1	85	116
7440-23-5	Sodium	2313	100.7	5.3	85	117
7440-24-6	Strontium	1170	99.9	5.9	82	118
7440-28-0	Thallium	5352	99.3	5.6	82	116
7440-29-1	Thorium	313	103.7	5.7	87	121
7440-31-5	Tin	1509	100.6	4.8	86	115
7440-32-6	Titanium	1538	98.6	5.3	83	115
7440-33-7	Tungsten	130	103.5	6.2	85	122
7440-61-1	Uranium	1860	103.3	5.4	87	120
7440-62-2	Vanadium	3375	100.5	5	86	115
7440-66-6	Zinc	4253	101	6	83	119

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TITLE: DETERMINATION OF METALS BY INDUCTIVELY COUPLED PLASMA MASS SPECTROSCOPY (ICP-MS) (EPA METHODS 6020, 6020A, 6020B & 200.8)

Table 5. Quality Control Requirements – Inorganic Analysis by Inductively Coupled Plasma/Mass Spectrometry (ICP-MS)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Linear Dynamic Range (LDR) or High-level Check Standard	At initial set-up and checked every 6 months with a high standard at the upper limit of the range.	Within $\pm 10\%$ of true value.	Dilute samples within the calibration range, or re-establish/verify the LDR.	Flagging is not appropriate.	Data cannot be reported above the calibration range without an established/passing high-level check standard.
Tuning	Prior to ICAL.	Mass calibration ≤ 0.1 amu from the true value; Resolution < 0.9 amu full width at 10% peak height.	Retune instrument and verify.	Flagging is not appropriate.	No samples shall be analyzed without a valid tune.
Initial Calibration (ICAL) for All Analytes	Daily ICAL prior to sample analysis.	If more than one calibration standard is used, $r^2 \geq 0.99$.	Correct problem, then repeat ICAL.	Flagging is not appropriate.	Minimum one high standard and a calibration blank. No samples shall be analyzed until ICAL has passed.
Initial Calibration Verification(ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All reported analytes, within $\pm 10\%$ of true value.	Correct problem. Rerun ICV. If that fails, repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed until calibration has been verified with a second source.

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TITLE: DETERMINATION OF METALS BY INDUCTIVELY COUPLED PLASMA MASS SPECTROSCOPY (ICP-MS) (EPA METHODS 6020, 6020A, 6020B & 200.8)

Table 5. Quality Control Requirements – Inorganic Analysis by Inductively Coupled Plasma/Mass Spectrometry (ICP-MS)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Continuing Calibration Verification (CCV)	After every 10 field samples and at the end of the analysis sequence.	All reported analytes within $\pm 10\%$ of the true value.	Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails or if two consecutive CCVs cannot be run, perform corrective action(s) and repeat CCV and all associated samples since the last successful CCV. Alternately, recalibrate if necessary; then reanalyze all associated samples since the last acceptable CCV	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable CCV.	Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

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Table 5. Quality Control Requirements – Inorganic Analysis by Inductively Coupled Plasma/Mass Spectrometry (ICP-MS)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Low-level Calibration Check Standard (Low Level ICV)	Daily.	All reported analytes within $\pm 20\%$ of the true value.	Correct problem and repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed without a valid low-level calibration check standard (LLCCV). LLCCV standard should be less than or equal to the LOQ. If the concentration of the lowest calibration standard is less than or equal to the LOQ, the lowest standard may be re-quantified against the calibration curve as LLCCV. Otherwise, a separate standard must be analyzed as the LLCCV prior to the analysis of any samples.

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Table 5. Quality Control Requirements – Inorganic Analysis by Inductively Coupled Plasma/Mass Spectrometry (ICP-MS)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Internal Standards (IS)	Every field sample, standard and QC sample.	IS intensity in the samples within 30-120% of intensity of the IS in the ICAL blank.	If recoveries are acceptable for QC samples, but not field samples, the field samples may be considered to suffer from a matrix effect. Reanalyze sample at 5-fold dilutions until criteria is met. For failed QC samples, correct problem, and rerun all associated failed field samples.	Flagging is not appropriate.	Samples suffering from matrix effect should be diluted until criteria are met, or an alternate IS should be selected.
Method Blank (MB)	One per preparatory batch.	The absolute values of all analytes must be < ½ LOQ or < 1/10th the amount measured in any sample.	Correct problem. If required, reprep and reanalyze MB and all QC samples and field samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid method blank. Non-detects associated with positive blank infractions may be reported. Sample results > 10X the LOQ associated with negative blanks may be reported. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

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Table 5. Quality Control Requirements – Inorganic Analysis by Inductively Coupled Plasma/Mass Spectrometry (ICP-MS)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Initial and Continuing Calibration Blank (ICB/CCB)	Immediately after the ICV and immediately after every CCV.	The absolute values of all analytes must be $< \frac{1}{2}$ LOQ or $< 1/10$ th the amount measured in any sample.	ICB: Correct problem and repeat ICV/ICB analysis. If that fails, rerun ICAL. All samples following the last acceptable Calibration Blank must be reanalyzed. CCBs may not be reanalyzed without reanalysis of the associated samples and CCV(s).	Flagging is not appropriate.	Results may not be reported without a valid calibration blank. Non-detects associated with positive blank infractions may be reported. Sample results $> 10X$ the LOQ associated with negative blanks may be reported. For CCB, failures due to carryover may not require an ICAL.
Interference Check Solutions(ICS) (also called Spectral Interference Checks)	After ICAL and prior to sample analysis.	ICS-A: Absolute value of concentration for all non-spiked project analytes $< 1/2$ LOQ (unless they are a verified trace impurity from one of the spiked analytes); ICS-AB: Within $\pm 20\%$ of true value.	Terminate analysis, locate and correct problem, reanalyze ICS, reanalyze all samples.	If corrective action fails, apply Q-flag to all results for specific analyte(s) in all samples associated with the failed ICS.	All analytes must be within the LDR. ICS-AB is not needed if instrument can read negative responses.

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Table 5. Quality Control Requirements – Inorganic Analysis by Inductively Coupled Plasma/Mass Spectrometry (ICP-MS)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Laboratory Control Sample(LCS)	One per preparatory batch.	A laboratory must use Table 3 and 4 limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Correct problem, then re-prep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Must contain all reported analytes. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per preparatory batch.	A laboratory must use Table 3 and 4 limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	For matrix evaluation only. If MS results are outside the limits, the data shall be evaluated to determine the source(s) of difference (i.e., matrix effect or analytical error).

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Table 5. Quality Control Requirements – Inorganic Analysis by Inductively Coupled Plasma/Mass Spectrometry (ICP-MS)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch.	A laboratory must use Table 3 and 4 limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified. MSD or MD: RPD of all analytes $\leq 20\%$ (between MS and MSD or sample and MD).	Examine the project-specific requirements. Contact the client as to additional measure be taken. s to	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	The data shall be evaluated to determine the source of difference. For Sample/MD: RPD criteria only apply to analytes whose concentration in the sample is greater than or equal to the LOQ.
Dilution Test	One per preparatory batch if MS or MSD fails.	Five-fold dilution must agree within $\pm 10\%$ of the original measurement.	No specific CA, unless required by the project.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	Only applicable for samples with concentrations $> 50 \times$ LOQ (prior to dilution). Use along with MS/MSD or PDS data to confirm matrix effects.
Post Digestion Spike (PDS) Addition	One per preparatory batch if MS or MSD fails (using the same sample as used for the MS/MSD if possible).	Recovery within 80-120%.	No specific CA, unless required by the project.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	Criteria apply for samples with concentrations $< 50 \times$ LOQ prior to dilution.

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TITLE: DETERMINATION OF METALS BY INDUCTIVELY COUPLED PLASMA MASS SPECTROSCOPY (ICP-MS) (EPA METHODS 6020, 6020A, 6020B & 200.8)

Table 5. Quality Control Requirements – Inorganic Analysis by Inductively Coupled Plasma/Mass Spectrometry (ICP-MS)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Method of Standard Additions (MSA)	When dilution or post digestion spike fails and if the required by project.	NA.	NA.	NA.	Document use of MSA in the case narrative.

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Document Information

Document Number: ENV-SOP-MTJL-0087	Revision: 00
Document Title: BTEX (Method 8021B, 602, SM6200C 2th) and Gasoline Range Organics (Methdo 8015B, 8015C, 8015D) by GC (with provisions for Calif-Lo, NWTPH-Gx, OA1, WI GRO (synthetic), Wyoming LAUST Req., GRO Luoisiana, AK101 GRO)	
Department(s): VOA	
Previous Document Number: 330351 rev.22	

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Effective Date: 02 Mar 2018	
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Notes

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All Dates and Times are listed in: Central Time Zone

Signature Manifest

Document Number: ENV-SOP-MTJL-0087

Revision: 00

Title: BTEX (Method 8021B, 602, SM6200C 2th) and Gasoline Range Organics (Methdo 8015B, 8015C, 8015D) by GC (with provisions for Calif-Lo, NWTPH-Gx, OA1, WI GRO (synthetic), Wyoming LAUST Req., GRO Luoisiana, AK101 GRO)

All dates and times are in Central Time Zone.

Review: ENV-SOP-MTJL-0087 00 BTEX (Method 8021B, 602, SM6200C 2th) and Gasoline Range Organics (Methdo 8015B, 8015C, 8015D) by GC (with provisions for Calif-Lo, NWTPH-Gx, OA1, WI GRO (synthetic), Wyoming LAUST Req., GRO Luoisiana, AK101 GRO)

Review

Name/Signature	Title	Date	Meaning/Reason
Steven Miller (006597)	QA Director	01 Jan 2019, 11:19:46 AM	Reviewed



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TITLE: BTEX (Method 8021B, 602, SM6200C 20th) AND GASOLINE RANGE ORGANICS (Method 8015B, 8015C, 8015D) by GC (With provisions for Calif-Lo, NWTPH-Gx, OA1, WI GRO (synthetic), Wyoming LAUST Req., GRO Louisiana, AK101 GRO)

Reviewed by: Heidi Ferrell, Steve Miller, Kandy Kaul

Department Manager

QA Department

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1.0 SCOPE AND APPLICATION

STATE NOTE: For samples analyzed in conjunction with the Ohio Voluntary Action Program (VAP) please utilize SOP #330351OH.

- 1.1 BTEXM/GRO by gas chromatograph determines the concentration of benzene, toluene, ethylbenzene, m, p & o-xylene, MTBE, and gasoline range organics (C₆ - C₁₀) (range defined in Method 8015B) in solution. All matrices, including groundwater, aqueous samples, TCLP extracts, wastewater, soils, sludge, sediments, and other solid wastes, can be analyzed by this method. Wisconsin GRO and AK101 GRO are determined by this method. Samples analyzed by the GRO-Louisiana method are quantitated using a carbon range of C₆ - C₁₀.

NOTE: Quantitation for samples from Alaska (AK101) must use GRO range from the beginning of the C₆ peak to the beginning of the C₁₀ peak.

- 1.2 The data shown in Attachment II provides the reporting limits for analytes in clean aqueous samples for each instrument currently running this method; however reporting limits are subject to change to address matrix issues, to better meet client/project/regulatory needs or to improve laboratory method performance.
- 1.3 Method Detection Limits (MDLs) are performed and evaluated based on ESC SOP #030206. Updated MDL records are filed and stored in a central location within the department.
- 1.3.1 Limit of Detection (LOD) and Limit of Quantitation (LOQ) studies are completed at the frequency required by the TNI standard per the procedure identified in the ESC SOP #030206, *Method Detection Limits (MDL), Limits of Detection (LOD) and Limits of Quantitation (LOQ)*. Should the procedure be utilized for DOD support; then the frequency of these studies must meet the requirements of the current DOD QSM.

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TITLE: BTEX (Method 8021B, 602, SM6200C 20th) AND GASOLINE RANGE ORGANICS (Method 8015B, 8015C, 8015D) by GC (With provisions for Calif-Lo, NWTPH-Gx, OA1, WI GRO (synthetic), Wyoming LAUST Req., GRO Louisiana, AK101 GRO)

2.0 METHOD SUMMARY AND DEFINITIONS

- 2.1 Samples (except for those to be prepared by 5035A) require no preparation before analysis unless the concentration of the analyte is great enough to require a waste or serial dilution.

In samples being analyzed by 5035A, an aliquot of the methanol extract is used to prepare the necessary dilution in 5mL of DI water.

- 2.2 This SOP describes the determination of concentrations of benzene, toluene, ethylbenzene, MTBE, and m, p, & o-Xylene by PID and gasoline range organics by FID. This method uses purge and trap to determine these concentrations. The BTEXM compounds and GRO concentrations are determined by internal standard calibration using fluorobenzene as the internal standard. Sample Introduction Method: The volatile compounds are introduced into the gas chromatograph by the EPA Purge-and-Trap Method 5030, SW-846.

2.2.1 Samples are placed in vials and purged with helium gas. The purged volatile compounds are transported to a trap (Supelco Purge Trap G) that is at 40°C. The trap is rapidly heated at the end of the purge cycle to 200°C and the volatile compounds desorb to the capillary column. After passing through this column, the compounds first pass by the PID, which detects double bonds, and then by the FID, which detects compounds that burn. As they pass by these detectors, an electrical signal is transmitted to a computer or integrator and causes an electrical peak to be recorded. The area underneath these peaks can be compared to known concentrations to determine the concentrations of BTEXM compounds in the sample.

- 2.3 Gasoline Range Organics (GROs) - Correspond to the range of alkanes from C₆ to C₁₀ and covering a boiling point range of approximately 60°C – 170°C.
- 2.4 See the current Quality Assurance Manual for other definitions associated with terms found in this document.

3.0 HEALTH AND SAFETY

- 3.1 The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds must be as low as reasonably achievable. A reference file of safety data sheets (SDSs) is made available on ESC's intranet to all personnel. Use hazardous reagents in a fume hood whenever possible and if eye or skin contact occurs, flush with large volumes of water. Always wear safety glasses or a shield for eye protection, protective clothing and observe proper mixing protocols.
- 3.2 Use of this procedure requires the handling of samples and standards containing volatile organic compounds. Use of laboratory safety glasses and protective gloves are required.

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TITLE: BTEX (Method 8021B, 602, SM6200C 20th) AND GASOLINE RANGE ORGANICS (Method 8015B, 8015C, 8015D) by GC (With provisions for Calif-Lo, NWTPH-Gx, OA1, WI GRO (synthetic), Wyoming LAUST Req., GRO Louisiana, AK101 GRO)

- 3.3 For specific information regarding the toxicity of the compounds used in this procedure and other related health and safety issues including the proper storage and handling of reagents and chemicals, the analyst should consult the appropriate Safety Data Sheets (SDS). These are located in a notebook at the Safety Station located in each building.

4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

- 4.1 All samples must have been collected using a sampling plan that addresses the considerations of this method.

4.2 Holding Times, Storage and Container Requirements

4.2.1 Water (EPA 5030)

Aqueous samples must be collected in triplicate in 40mL vials with the pH adjusted to <2 with HCl and stored at $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$. Sample must be analyzed within 14 days of collection. Samples not preserved with HCl must be analyzed within seven days of collection.

4.2.1.1 Samples that do not have a pH <2 are qualified with a G1 if analyzed past seven days of collection.

4.2.2 Soil (EPA 5035A)

High Concentration - Collect an additional sample using the Encore sampler or equivalent.

4.2.2.1 Eject the sample into a 40mL pre-weighed vial containing 5mL of methanol. This vial can be supplied pre-weighed by the laboratory. The vial is sealed and placed in cold storage at $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$.

If a vial with 5mL of methanol is not available, the soil sampler is capped and placed in a foil bag. This sample must arrive at the laboratory within 48 hours. When the Encore arrives at the lab, the sample is ejected from the Encore into a pre-weighed (to 0.01g) vial containing 5mL of methanol then placed in cold storage of $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$.

If specially prepared vials were not available at the sampling site, a soil sample packed into a 125mL jar or brass core sample tube can be used. At the analysis site, approximately 5g of soil is removed from the sample and placed in a tared 40mL vial. 5mL of reagent water and a stir bar are added.

4.2.2.2 Collect additional duplicate aliquots of each sample in 40mL glass vials (septum sealed), 125mL glass container, brass capped core sample tube or equivalent for sample screening, dry weight determination, and any additional high concentration evaluation required.

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TITLE: BTEX (Method 8021B, 602, SM6200C 20th) AND GASOLINE RANGE ORGANICS (Method 8015B, 8015C, 8015D) by GC (With provisions for Calif-Lo, NWTPH-Gx, OA1, WI GRO (synthetic), Wyoming LAUST Req., GRO Louisiana, AK101 GRO)

STATE NOTE: Soil and Water samples received from the states of Missouri or Kansas may be preserved with tri-sodium phosphate and have a resulting pH > 12.

- 4.3 Glassware: All glassware must be pre-washed with detergent and rinsed with deionized water. Refer to SOP #030701, *Glassware Cleaning*.
- 4.4 **STATE NOTE:** AK101 Soil/Sediment Collection Procedure - Soils and Sediments: Soil and sediment samples require special procedures to minimize the loss of volatile organic compounds during transit from the field to laboratory.
Please note that this sample preservation is different from SW-846 Method 8021B. The use of sodium bisulfate as a preservative is not acceptable.
 - 4.4.1 Soil or sediment samples must be collected into appropriately sized containers and submerged in surrogate methanol.
 - 4.4.2 Solid samples must be collected with minimum disturbance into tared jars with a Teflon-lined septum fused to the lid. Jars should be 4oz. or larger. 25mL aliquots of methanol (includes 1.25µL of a surrogate solution at 50µg/mL) are carefully added to the undisturbed soil until the sample is submerged.
 - 4.4.3 It is extremely important that the weight of the jar, the weight of the methanol/surrogate solution, and the weight of the sample collected be known. These must either be measured directly, or sufficient information documented so that these weights can be calculated.
 - 4.4.4 The ratio of soil to methanol used to calculate the MDL and PQL offered in the AK101 method was 1:1 (w:w). However, absorbent, organic soils such as muskeg and tundra require a higher methanol-to-sample ratio, while beach sand may tolerate a lower ratio.
 - 4.4.5 Soil for volatiles analysis can be collected using any coring device that minimizes soil disturbance. Any scraping, stirring, or similar activity results in a loss of volatiles during sampling. A sufficient number of samples must be collected to provide for backup in case of breakage.
 - 4.4.6 Although it is not necessary to refrigerate all methanol preserved samples at 4° ± 2°C after collection and until analysis is complete, collected samples must be kept below 25°C.
 - 4.4.7 A second surrogate, added to the methanol and soil mixture after sample collection, may be used in addition to, but not in place of, the surrogate with which the field methanol preservative was prepared.

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- 4.4.8 A reagent methanol trip blank must be prepared in the same manner as the sample vials, and must contain surrogate methanol. One trip blank must be included with each shipping container and must be stored and analyzed with the field samples. Trip blank analysis is not required if all samples in a shipping container are less than the project specific cleanup level.
- 4.4.9 Field blanks may be added to the sampling protocol and are prepared in the field by addition of surrogate methanol to the prepared container, as required by the Assessment Firm or the Project Manager.
- 4.4.10 A sample of the same soil to be analyzed for GRO must be collected into a moisture-proof container for per cent moisture determination. This sample is processed as soon as possible upon arrival at the laboratory to assure that the resulting moisture determination is representative of the preserved sample as surveyed.
- 4.4.11 Trip blanks, field blanks, method blanks, etc. are prepared from the same batch of solvent, reagents and vials as are used for sample preservation.
- 4.4.12 Twenty-eight days is the maximum holding time for soil and sediment samples collected under this section.
- 4.4.13 Because the jars are pre-weighed, it is extremely important that the sampler put evidence tape on the kit ONLY, or the bubble bags in which the sample bottles are shipped, and not on the individual bottles. Removal of evidence tape is extremely difficult and the additional weight biases final results. Also, the glue on the evidence tape can contribute to the volatiles concentration in the sample.
- 4.4.14 Trip blanks, field blanks, and bottle blanks are prepared as appropriate to meet the quality assurance goals of the project plan.
- 4.4.15 28 days is the maximum holding time for AK101 soil samples preserved with MeOH. 14 days is the holding time if BTEX is included.
- 4.5 Samples submitted for analysis that do not meet the requirements contained within this section must be addressed before performing the logging process within the laboratory. In some cases, exceeding the appropriate preservation and storage criteria can cause significant bias in the resulting data. Clients may need to resubmit samples where the conditions during shipment cause uncertainty regarding sample integrity. If samples do not meet the requirements for preservation, sampling, shipment and storage and the client approves the completion of the analytical process, sample results can be qualified per the ESC SOP #030201, *Data Handling and Reporting*.

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TITLE: BTEX (Method 8021B, 602, SM6200C 20th) AND GASOLINE RANGE ORGANICS (Method 8015B, 8015C, 8015D) by GC (With provisions for Calif-Lo, NWTPH-Gx, OA1, WI GRO (synthetic), Wyoming LAUST Req., GRO Louisiana, AK101 GRO)

5.0 INTERFERENCES

- 5.1 Matrix interference can result in samples with high concentrations of volatile organic compounds (VOC). This interference can cause the resulting peaks to not be clear and concise. This can lead to misidentification of compounds and/or poor quantitation of those compounds. This problem can be solved by diluting the sample.
- 5.2 Carryover from previous samples must be monitored through the use of sample blanks. When a sample with a high concentration of VOC's is followed by a low level sample, false peaks may result from carryover. Sample blanks are used to clean the instrument.

6.0 EQUIPMENT AND SUPPLIES

The operation, cleaning and scheduled maintenance procedures prescribed by the equipment manufacturer are followed as provided in the Operator's Manuals. Documentation of maintenance or system modifications is recorded in a maintenance logbook which accompanies each instrument.

6.1 Instrumentation

- Designated Instruments: VOCGC #1, #3, #4, #5, #6, #7, #10, #12, #14, #15
- Use (method #'s): 8021B; 8015B, 8015C & 8015D
- Model #: HP 5890 or equivalent
- Column (type, brand, size): J & W Scientific DB VRX 75m x 0.450mm, 2.55um or equiv.
- Detector: GC FID, PID
- Software name and version: HP Chemstation G1701BA B.01.00, or equivalent
- Sample introduction system: Archon Autosampler, Encon P & T, or equivalent

6.2 Glassware

Volumetric – glassware equipped with penny head ground glass stopper. The volumetric flasks and graduated cylinders are cleaned by rinsing with methanol and laboratory reagent water. The volumetrics are dried in a low temperature oven at less than 120°C. Never use a brush or strong alkali solution to clean the volumetrics.

6.3 Glass Sample (VOA) and Standard Vials:

- 6.3.1 "42.5mL" VOA vials with a Teflon™/silicone septa and polypropylene open-top cap.
- 6.3.2 8mL vials with Teflon™/silicone/Teflon™ septa and polypropylene open-top cap. (Used to store unused standards.)
- 6.3.3 2mL vials with Teflon™ lined screw caps.

6.4 Miscellaneous:

- 6.4.1 Stainless Steel Spatula, tongue depressors

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6.4.2 Teflon™-coated stir bars, 8mm x 16mm

6.4.3 Laboratory Blank Matrix: Sand, glass beads, etc. is prepared by rinsing clean with methanol and laboratory reagent water several times. The matrix is baked in an oven at 175°C overnight to remove any volatiles and is then stored in the sealed container used for baking. The clean laboratory matrix may be purged with carrier grade helium or nitrogen to remove trapped volatiles.

6.5 Oven: Fisher IsoTemp Forced-Air Oven with capabilities of 100°C, or equivalent

6.6 Top-loading Balance, capable of weighing to 0.01g, or equivalent

7.0 REAGENTS AND STANDARDS

7.1 All reagents and standards must be recorded in the appropriate preparation log and assigned a unique number. See SOP #030230, *Standards Logger – Tree Operation*. Additional information regarding reagent preparation can be found in the Standards Logger (Tree) digital archive system. All spiking solutions and surrogate standard solutions should be replaced at least every 6 months, or sooner, if a problem is detected unless otherwise noted.

7.2 Reagents

7.2.1 Nanopure water or equivalent: Nanopure water is used in all blanks to assure that it contains less than the method detection limit (MDL) of all compounds of interest. The blank must be assessed to ensure that the water does not show any detection of any VOC compounds.

7.2.2 Methanol, CH₃OH (VWR #EM-MX0480-1 or equivalent) - purge and trap grade, demonstrated to be free of target analytes. Store isolated from other solvents in the designated flammables cabinet.

7.2.3 Sodium Bisulfate, Na₂S₂O₃ from QEC, Level 3 certified in 40mL vials, or equiv.

7.2.4 Sodium Bisulfate monohydrate, 99% for analysis, ACROS Organics or equivalent.

7.3 Stock Standards: Stock solutions may be prepared from pure standard materials or purchased as certified solutions. These standards are prepared in methanol. Store stock standards in vials at ≤10°C.

7.3.1 BTEXM/GRO Calibration Standard – NSI PVOC/GRO mixture UST-360, or equivalent is used for the BTEXM compounds, and Restek certified BTEX in unleaded gas composite Cat # 30237, or equivalent, is used for GRO. The stock standards are prepared from standards with the following components and approximate concentrations:

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TITLE: BTEX (Method 8021B, 602, SM6200C 20th) AND GASOLINE RANGE ORGANICS (Method 8015B, 8015C, 8015D) by GC (With provisions for Calif-Lo, NWTPH-Gx, OA1, WI GRO (synthetic), Wyoming LAUST Req., GRO Louisiana, AK101 GRO)

Benzene	1000ug/mL
Toluene	1000ug/mL
Ethylbenzene	1000ug/mL
M & P-xylene	2000ug/mL
o-xylene	1000ug/mL
MTBE	1000ug/mL
n-Pentane	1000000ug/mL
GRO	5500ug/mL

- 7.3.2 Synthetic WISGRO Calibration Standard – NSI PVOC/GRO mixture UST-360, or equivalent, while the LCS is from Restek Cat # 30095 revised WISC PVOC/GRO mixture or equivalent. The stock standard is prepared from a standard with the following components and concentrations:

MTBE	1000ug/mL
Benzene	1000ug/mL
Toluene	1000ug/mL
Ethylbenzene	1000ug/mL
m-xylene	1000ug/mL
p-xylene	1000ug/mL
o-xylene	1000ug/mL
1,2,4-trimethylbenzene	1000ug/mL
GRO (sum of rep. comp.)	10000ug/mL
1,3,5-TMB	1000ug/mL
Naphthalene	1000ug/mL

- 7.3.3 BTEXM/GRO Laboratory Control Standard - Restek revised WISC PVOC/GRO mix cat# 30095 or equivalent for BTEXM and NSI Gas composite Q-4643 or equivalent for GRO: Concentrations as stated in 7.3.2.
- 7.3.4 GRO Retention Time Marker: Restek revised WISC PVOC/GRO mix cat#30095 or equivalent for BTEXM and NSI Gas composite Q4643. n-Pentane standard – Fox Scientific, Inc. pure grade or equivalent. Concentrations as stated in 7.3.1

7.4 Intermediate ICV/CCV/LCS Standards

- 7.4.1 BTEX ICV from NSI (UST-360) is certified BTEX in unleaded gas. The LCS is from Restek Cat#30095. Secondary dilution standards of BTEXM/GRO Standard: This intermediate standard is stored with minimal headspace in the same manner as the stock standard. 2.5mL of BTEXM solution from 7.3.1 in 50mL of methanol has the following concentrations. The GRO standard is not diluted.

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TITLE: BTEX (Method 8021B, 602, SM6200C 20th) AND GASOLINE RANGE ORGANICS (Method 8015B, 8015C, 8015D) by GC (With provisions for Calif-Lo, NWTPH-Gx, OA1, WI GRO (synthetic), Wyoming LAUST Req., GRO Louisiana, AK101 GRO)

Benzene	50ug/mL
Toluene	50ug/mL
Ethylbenzene	50ug/mL
M & P-xylene	100ug/mL
O-xylene	50ug/mL
MTBE	50ug/mL
n-Pentane	50ug/mL
GRO	5500ug/mL

(Syringe sizes needed: 5mL, 25µL, 10µL, 1 µL & 0.5 µL)

7.4.2 WISGRO's working standard is prepared by mixing 2.5mL of synthetic WISGRO standard into 47.5mL of methanol to make a 50ppm working standard which is then used to prepare all calibration standards.

7.5 Calibration standards:

Calibration standards are prepared in reagent water at a minimum of five concentration levels. The lowest standard must be at or below the RL. The calibration standards are prepared from the primary source (which is a different Lot # than the LCS) according to the instructions in 7.3. This is the intermediate stock. Use the measurements listed below and dilute each to 5mL to produce each calibration point. The concentration varies slightly with lot number.

BTEXM/GRO

	Conc. ppm	Amt.uL	Amt.uL	Amt.uL	Amt.uL	Amt.uL	Amt.uL	Amt.uL	Amt.uL	Amt.uL
	Stock listed in 7.3.1	0.05	.1	.5	1	2.5	5	10	20	25
GRO ppm	5500	0.055 µg/mL	0.11 µg/mL	0.55 µg/mL	1.1 µg/mL	2.75 µg/mL	5.5 µg/mL	11 µg/mL	-	-
GRO Surrogate - a,a,a-TFT		200ug/L	202ug/L	204ug/L	206ug/L	208ug/L	210ug/L	212ug/L	-	-
Benzene ppb	50	0.5ug/L	1ug/L	5ug/L	10ug/L	25ug/L	50ug/L	100ug/L	200ug/L	250ug/L
Toluene ppb	50	0.5ug/L	1ug/L	5ug/L	10ug/L	25ug/L	50ug/L	100ug/L	200ug/L	250ug/L
Ethylbenzene ppb	50	0.5ug/L	1ug/L	5ug/L	10ug/L	25ug/L	50ug/L	100ug/L	200ug/L	250ug/L
m&p Xylene ppb	50	1ug/L	2ug/L	10ug/L	20ug/L	50ug/L	100ug/L	200ug/L	400ug/L	500ug/L
o Xylene ppb	50	0.5ug/L	1ug/L	5ug/L	10ug/L	25ug/L	50ug/L	100ug/L	200ug/L	250ug/L
MTBE ppb	50	0.5ug/L	1ug/L	5ug/L	10ug/L	25ug/L	50ug/L	100ug/L	200ug/L	250ug/L
BTEX Surrogate - a,a,a-TFT		200ug/L	202ug/L	204ug/L	206ug/L	208ug/L	210ug/L	212ug/L	216ug/L	218ug/L

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Synthetic GRO

	Conc. ppm	Amt.uL	Amt.uL	Amt.uL	Amt.uL	Amt.uL	Amt.uL	Amt.uL	Amt.uL	Amt.uL
	Stock listed in 7.3.1	.05	.1	.5	1	2.5	5	10	20	25
GRO ppm	500			0.05 µg/mL	0.1 µg/mL	0.25 µg/mL	0.5 µg/mL	1.0 µg/mL	2.0 µg/mL	2.5 µg/mL
Benzene ppb	50	0.5ug/L	1ug/L	5ug/L	10ug/L	25ug/L	50ug/L	100ug/L	200ug/L	250ug/L
Toluene ppb	50	0.5ug/L	1ug/L	5ug/L	10ug/L	25ug/L	50ug/L	100ug/L	200ug/L	250ug/L
Ethylbenzene ppb	50	0.5ug/L	1ug/L	5ug/L	10ug/L	25ug/L	50ug/L	100ug/L	200ug/L	250ug/L
m&p Xylene ppb	50	1ug/L	2ug/L	10ug/L	20ug/L	50ug/L	100ug/L	200ug/L	400ug/L	500ug/L
o Xylene ppb	50	0.5ug/L	1ug/L	5ug/L	10ug/L	25ug/L	50ug/L	100ug/L	200ug/L	250ug/L
MTBE ppb	50	0.5ug/L	1ug/L	5ug/L	10ug/L	25ug/L	50ug/L	100ug/L	200ug/L	250ug/L

7.6 **Internal standard Fluorobenzene** 100,000µg/mL - NSI Cat # Q-4187 or equivalent.

7.7 **Surrogate** $\alpha\alpha\alpha$ -trifluorotoluene 100,000µg/mL - NSI Cat # Q-4187 or equivalent.

7.8 **Surrogate/Internal standard preparation:** Commercially-prepared $\alpha\alpha\alpha$ -TFT at 100,000 µg/mL and Fluorobenzene at 100,000µg/mL are purchased from NSI for use in making an internal standard/surrogate mixture. This mixture is prepared by diluting 1mL of the NSI mixture into 99mL of methanol (100mL total volume). It is injected automatically by the instrument at a rate of 1µL per 5mL purge volume. This results in a 200µg/L solution of internal standard/surrogate. Check daily to make sure that the instrument reservoir has adequate IS/Surr solution.

STATE NOTE: For Wisconsin GRO/PVOC and AK101 samples, the internal standard/surrogate mix is prepared as described in section 7.8 except the concentration of the surrogate ($\alpha\alpha\alpha$ -TFT) is at 20,000µg/mL instead of 100,000 µg/mL.

7.9 **Solvent:** Methanol Fisher GC Resolve A457-4 or equivalent: High res.- GC grade.

7.10 **Spike Solution (LCS/LCSD/MS/MSD):** For the LCS/LCSD, spike the LCS spike solution prepared in section 7.3.1 into duplicate aliquots of a clean matrix. For the MS/MSD, prepare the spikes using the same as LCS solution except, introduce 5µl of the solution prepared in 7.3.1 directly into separate aliquots of the selected field sample.

8.0 PROCEDURE

8.1 **Analysis Summary:** Volatile compounds are introduced into the gas chromatograph by purge and trap, via the Archon autosampler. If soil samples are high in contamination, a methanolic extraction, as described section 8.4.4 and SOP #330760, may be necessary prior to purge and trap analysis. Soils require method 5035A for sample preparation, See SOP #330751.

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8.2 Gas Chromatography Conditions:

The particular settings for all instruments are subject to change at any time. See each individual instrument for the current autosampler, purge-and-trap, and gas chromatograph settings. Typical conditions are given in the following table:

Item	Setting
Autosampler and Purge-and-Trap	
Trap	G trap
Valve Oven Temperature	110°C
Transfer Line Temperature	110°C
Sample Mount	110°C
Purge Ready Temperature	45°C
Sample Heater Temperature	40°C
Sample Preheat Time	0.5 minutes
Purge Time	11 minutes
Purge Flow	40mL/minute
Dry Purge Time	1 minute
Desorb Preheat Temperature	220°C
Desorb Temperature	230°C
Desorb Time	1 minute
Bake Temperature	230°C
Bake Time	2 minutes
Gas Chromatograph	
Inlet Temperature	250°C
Split Ratio	0.8:1
Column	Rtx-VRX 75m x .45mm x 2.55µm
Column Flow	10mL/minute; Hold 6 minutes Ramp 2mL/minute to 22mL/minute
Oven Program	45°C Ramp 10°C/minute to 75°C Hold 1 minute Ramp 15°C/minute to 80°C Ramp 30°C/minute to 230°C Hold 5.67 minutes
Detector Temperature	300°C

8.3 **Calibration:** Method 8015, 8021B BTEXM, WI GRO, and GROMAR require a five-point calibration curve. This curve must have a % RSD of <20% for each of the BTEXM/GRO compounds. In the event that RF criteria are not met, linear regression may be used. In order to use this option, the correlation coefficient of the calibration curve must be a minimum of 0.990 or better. Equal weighting factors or 1/x regressions may be used.

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STATE NOTE: Linear regression is required for quantitation of **WI GRO** samples. PVOC is acceptable on average response.

STATE NOTE: AK101 GRO must have %RSD < 25%.

8.3.1 Retention Time Marker: GRO by 8015, WI GRO, and Synthetic GRO quantitation is performed using "baseline to baseline" integration. The area is summed from the marker compounds of MTBE to 1,2,4-trimethylbenzene, representing C₆ to C₁₀. These markers are evaluated and RT's changed when appropriate. The instrument response attributed to the surrogate and internal standard is not included.

STATE NOTE: For NWTPH-Gx, the retention time range for gasoline integration must, at a minimum, include toluene through naphthalene. For surrogates that elute within the retention time range used for TPH integration, the analyst must subtract the area of the surrogate(s) from the total area of the TPH peak to yield the appropriate area of the petroleum product.

STATE NOTE: For AK101, the retention time range for gasoline integration must include the resolved and unresolved components that elute between and including C₆ (hexane) and C₉ (nonane) to end at the peak start time of C₁₀ (decane). Quantitation must be performed using "baseline to baseline" integration.

8.3.2 ICV: A mid-point check standard is analyzed first. All BTEX, MTBE, compounds must be within 15% of the actual value and WI GRO, and GRO by Method 8015 must be within 20% of the actual value. When this criterion is met, a CCB is run to be sure there is no carryover or instrument contamination.

STATE NOTE: AK101 GRO CCV limits are $\pm 25\%$ for GRO and 60-120% for surrogates.

8.3.3 A Laboratory Control Standard (LCS) is evaluated by the QC limits in LIMS for BTEXM/GRO by Method 8015. WI GRO LCS must be within 80-120% and AK101 GRO must be within 60-120% for both soil and water. An LCS and LCSD are required with each batch. The RPD cannot exceed 20% for either matrix.

8.3.4 A Matrix Spike/Matrix Spike Duplicate must be evaluated for each matrix type. Evaluate the matrix spike and matrix spike duplicate recovery based on the QC limits in LIMS. If for any analyte, the laboratory control standard, matrix spike, and the matrix spike duplicate are all outside the QC limits, the entire analytical batch must be reanalyzed or QC can be reanalyzed.

8.3.5 A secondary source Calibration Verification Standard is required for AK101 GRO calibrations. This standard must be within $\pm 25\%$ with surrogates at 60-120%. See Section 10.4 for further information.

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STATE NOTE: For MN/WI GRO samples a reporting level check standard must be analyzed after each calibration or monthly, whichever is more often, and must recover within 60-140%.

CLIENT NOTE: For Marathon/MPC LLC/SSA samples a reporting level check standard must be analyzed after each calibration and must recover within 60-140%. If recovery of 60-140% is not met, the reporting limit must be raised, and a back calculation performed at that level. This process must be repeated until an acceptable RL recovery is achieved.

8.4 Gas chromatographic analysis:

Typical Batch order for loading the autosampler when a calibration is run:

Sample/QC Type	Use
Cleanup Blank	Verify system is contamination free
Retention Marker	Verify windows for gasoline ranges. Also required to be analyzed every 24 hours for AK101 and GROMAR. And every 12 hours for GROWY.
Calibration standard(s)	Initial 5-point calibration or single-point calibration verification. MUST be mid-point standard.
Second Source Cal. Verification (SSCV)	Second Source verification of initial calibration.
Laboratory Control Sample(s)	Laboratory blank, spiked with known amount(s) of analyte of interest
Matrix Spike/Matrix Spike Dup.	Sample spiked with known amount(s) of analytes of interest
Method blank	Ensure that carry over has not occurred from the calibration standard, and that the analytical system does not show contamination above the established detection limits
1 to 20 samples	Client samples

Typical Batch order for loading the autosampler when a calibration is not run:

Sample/QC Type	Use
Cleanup Blank	Verify system is contamination free
Retention Marker	Verify windows for gasoline ranges. Required to be analyzed every 24 hours for AK101 and GROMAR. And every 12 hours for GROWY.
Initial Calibration Verification (ICV)	Verify initial 5-point calibration.
Laboratory Control Sample(s)	Laboratory blank, spiked with known amount(s) of analyte of interest
Matrix Spike/Matrix Spike Dup.	Sample spiked with known amount(s) of analytes of interest

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Sample/QC Type	Use
Method blank	Ensure that carry over has not occurred from the calibration standard, and that the analytical system does not show contamination above the established detection limits
1 to 20 samples	Client samples

CLIENT NOTE: For Marathon/Speedway (MPC LLC/SSA), CCVs must be analyzed every 10 samples (including laboratory and quality control samples).

8.4.1 Water/Liquid Samples

Samples are received in 40mL vials containing HCl as a preservative. The autosampler removes 5mL of sample, mixes the sample with 1µL of internal standard/surrogate mix, and heats the sample for 30 seconds at 40°C. The sample then purges for 11min at 40°C to drive off all VOC's to the trap. The trap is desorbed for one minute at 175°C before entering the column for analysis.

8.4.2 Soil/Sediment Samples (Water Purge) - Collected in soil Jar

Weigh 5 grams of soil into a 40mL vial containing a stir bar. Add 5mL of Nanopure water and tighten cap. The autosampler injects the sample with 1µL of internal standard/surrogate mix. The autosampler moves the vial into a heating chamber and heats the sample for 1 minute at 40°C. The sample then purges for 11min at 40°C to drive off all VOC's to the trap. The trap is desorbed for one minute at 175°C before entering the column for analysis.

8.4.3 Soil/Sediment Samples - Collected in Encore (Encore "like") Sampling Device

The sample is collected using an Encore or Encore "like" sampling device. The device is designed to sample soil at approximately 5g. The sample is placed into a pre-weighed 40mL vial containing a stir bar and 5mL of Sodium Bisulfate, as a preservative. Weigh the vial to determine the weight of the soil. WISGRO is 25g of soil into 25mL of methanol.

Soil Sample Weight (g) = Total weight of Vial and Soil (g) - Pre - weigh value (g)

Record the determined weight of the sample and load onto the autosampler. The autosampler injects the sample with 1µL of internal standard/surrogate mix. The autosampler moves the vial into a heating chamber and heats the sample for 30 seconds at 40°C. The sample then purges for 11min at 40°C to drive off all VOC's to the trap. The trap is desorbed for one minute at 175°C before entering the column for analysis.

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8.4.5 High Level Soil/Sediment Sample (Methanol Extraction) – Collected in Soil Jar

NOTE: Samples known to have high concentrations greater than 200ppb may be collected in a 2oz. Sample jar with no headspace.

Weigh 5g of soil sample and place in vial. Add 5mL of Methanol and vortex for 30 seconds. Using a gas tight microsyringe, measure a maximum of 200µL of methanol extract and inject into a vial containing 5mL of water. Enter the sample multiplier as 25X. The autosampler injects the sample with 1µL of internal standard/surrogate mix. The autosampler moves the vial into a heating chamber and heats the sample for 30 seconds at 40°C. The sample then purges for 11min at 40°C to drive off all VOC's to the trap. The trap is desorbed for 1 minute at 175°C before entering the column for analysis. For PVOC/GRO soil samples 25g of soil is placed into a vial with 25mL of methanol. If the weight of the soil exceeds 35g the sample is discarded. If the weight of the sample is >26g but <35g methanol is added until the volume of methanol in mL is equal to the weight of the soil in g.

STATE NOTE: For WI PVOC/GRO samples, a maximum of 100µL may be injected into 5mL for a multiplier of 50x.

8.5 Quantitation:

8.5.1 Quantitation of GRO is performed by the internal standard method. The concentration of Gasoline Range Organics in the sample is determined from a summation of the total response within the range of the elution of MTBE and 1,2,4-Trimethylbenzene, using the calibration curve. No area other than that relating to the internal standard or surrogates may be subtracted from the GRO retention time window in calculating GRO results. WISGRO is evaluated by the external standard method. WISGRO no IS/SURR areas are subtracted and range is from the beginning of MTBE and to the conclusion of Naphthalene.

8.5.2 Integration must be "baseline to baseline" as opposed to a "valley to valley". Baseline to baseline is defined here as a flat baseline drawn parallel to the x-axis of the chromatogram that includes all responses within the retention time window. The correct baseline coincides with a horizontal line drawn through the lowest point in the chromatogram before the end of the window. The lowest point may be within the window, before the window, or before the solvent front. Baseline to baseline integration does not include the solvent peak. Placement of the baseline is determined for each sample.

CLIENT NOTE: When EPA 8021B analysis is performed for samples analyzed in conjunction with Marathon/MPC LLC/SSA, confirmation is performed using GC/MS by EPA 8260B.

8.5.3 BTEX/MTBE quantitation is performed using "total area vs selected peak".

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8.6 For acceptance criteria and corrective actions, see sections 10.0 & 11.0.

9.0 DATA ANALYSIS AND CALCULATIONS

9.1 AK101 Moisture Correction: In order to report results for volatiles analysis of samples containing significant moisture (>10%) content on an "as received" basis, the calculated concentration needs to be corrected using the total solvent/water mixture volume represented as V_t . This total solvent/water volume is calculated as follows:

$$\mu\text{L solvent/water } V_t = \left[\frac{\text{mL of solvent} + (\% \text{ moisture} \times \text{g of sample})}{100} \right] \times 1000 \mu\text{L/mL}$$

9.2 See the current Quality Assurance Manual for equations associated with common calculations.

10.0 QUALITY CONTROL AND METHOD PERFORMANCE

10.1 All analysts must meet the qualifications specified in SOP #030205, *Technical Training and Personnel Qualifications* before approval to perform this method. Analysts must complete an initial demonstration of proficiency before being approved to perform this method. Continuing proficiency must be demonstrated using proficiency testing, laboratory control sample analysis and/or MDL studies. Method performance is assessed per analyst. Updated method performance records are filed and stored in a central location within the department.

10.2 Use the designated Run log to record batch order and standards/reagents used during analysis. See SOP #030201, *Data Handling and Reporting*.

10.3 Initial Calibration – Initial calibration curves must meet the criteria found in the following table. One concentration of the calibration standards must be at or below the RL. The remaining concentration should encompass the linear working range of the instrument. In most cases, %RSD or linear regression is acceptable. When using linear regression, equal weighting factors or 1/x regressions may be used.

Analytical Method	Min. # of Calibration Standards Required	Initial Calibration Acceptance Criteria	
		%RSD	Linear Correlation Coefficient
EPA 8015B, 8015C, 8015D	5	≤20%	≥0.990
EPA 8021B	5	≤20%*	≥0.990*
WI PVOC	5	≤20%*	≥0.990*
WI GRO	5	NA	≥0.990
SM 6200	5	≤20%	≥0.994
NWTPH-Gx	5	≤20%	≥0.990
EPA 602	3	≤10%	≥0.990



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Analytical Method	Min. # of Calibration Standards Required	Initial Calibration Acceptance Criteria	
		%RSD	Linear Correlation Coefficient
OA1	3	≤20%	≥0.995
AK101	3	<25%	NA
GROMAR	5	<20%	>0.990

* required for each target analyte being reported for this method.

NA indicates that this process cannot be used for this method.

NOTE: For USACE samples the correlation coefficient must be ≥0.995.

- 10.4 Second Source Calibration Verification (SSCV) – Initial calibration curves must be verified with a second source prior to analyzing any samples. Recoveries from a mid-point standard made from a secondary source not used to generate the calibration curve must be within ±20% of the true value before sample analysis may begin.

STATE NOTE: For samples analyzed in conjunction with AK101, recoveries from a mid-point standard made from a secondary source not used to generate the calibration curve must be within ±25%

- 10.5 Initial Calibration Verification (ICV)/Continuing Calibration Verification (CCV) – Before beginning a sample run, a midpoint check standard (ICV) is analyzed initially to ensure accurate instrument calibration. Continuing calibration verification (CCV) must be checked after every 20 samples. Acceptance criteria for the specific methods are listed in the table below.

Analytical Method	Continuing Calibration Acceptance Criteria
EPA 8015B, 8015C, 8015D	+ 20% of Expected Value
EPA 8021B	+ 20% of Expected Value
WI PVOC	+ 15% of Expected Value
WI GRO	+ 20% of Expected Value
SM 6200	+ 30% of Expected Value
NWTPH-Gx	+ 20% of Expected Value
EPA 602	+ 30% of Expected Value
OA1	+ 20% of Expected Value
AK101	+ 25% of Expected Value
GROMAR	+ 20% of Expected Value

CLIENT NOTE: For Marathon/Speedway (MPC LLC/SSA), CCVs must be analyzed every 10 samples (including laboratory and quality control samples). %Difference or %Drift must be ≤20% for all target compounds and surrogates.

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10.6 Method Blank – Method Blanks contain reagent water that are analyzed following successful calibration and/or verification to ensure that the analytical system is free from interferences prior to the analysis of field samples. The acceptance criterion for all method blanks is less than the method detection limit.

10.6.1 If more than one instrument blank or method blanks are analyzed, evaluate and assess the blank and field samples under the same conditions for possible mid-level standard carryover using the subsequent blank after the mid-level standard on a per analyte basis.

STATE NOTE: For samples analyzed in conjunction with AK101, blank subtraction is not permitted. Blanks should be reported by value for data quality assessment.

NOTE: Additional blanks may be submitted with client batches to verify that no cross contamination occurs during shipping of samples and there is no contamination contributed from the sampling equipment. Additional blanks may also be analyzed to ensure that the analytical system remains clean following the analysis of highly contaminated samples.

10.7 Matrix Spike/Matrix Spike Duplicate - are run every 20 samples. The analyst also verifies that the spikes are at the appropriate levels. Spiking levels correspond to the midpoint of the calibration curve. Acceptance criteria are available in the LIMS. If the spike recovery does not meet criteria, verify matrix interference and apply qualifiers.

10.8 Laboratory Control Sample (LCS)/Laboratory Control Sample Duplicate (LCSD) – An LCS and LCSD are required with each batch and evaluated using the QC limits in LIMS for BTEXM/GRO by Method 8015. Levels correspond to the midpoint of the calibration curve.

STATE NOTE: WI GRO LCS must be within 80-120% for both soil and water. The RPD cannot exceed 20% for either matrix.

STATE NOTE: AK101 GRO LCS must be within 60-120% for both soil and water. An LCS and LCSD are required with each batch. The RPD cannot exceed 20% for either matrix. Surrogates in AK101 must meet 60% to 120% recovery in Blanks, LCS, and LCSD.

10.9 Surrogates - must be assessed for all samples and QC in the batch. Alpha, Alpha, Alpha - TFT recovery must be within acceptance criteria listed in LIMS.

STATE NOTE: The WI PVOC Surrogate must be within >80% for both soil and water and are analyzed from the PID only.

STATE NOTE: Surrogates in AK101 must meet 60% to 120% recovery in Blanks, LCS, and LCSD. Surrogates in field samples must meet 50-150% for both soil and water. For ease of analysis, the control limits used by the laboratory as found in LIMS, exceed the method required limits, but allow for running these samples in conjunction with other TPH analyses.

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STATE NOTE: Surrogates in NWTPH-Gx must meet 50-150% for both soil and water. For ease of analysis, the control limits used by the laboratory as found in LIMS, exceed the method required limits, but allow for running these samples in conjunction with other TPH analyses.

- 10.10 Internal standard - IS fluorobenzene, response must be within acceptance limits for all samples and quality control samples. The internal standard response must be within 50% - 200% of the response of the calibration verification standard.

CLIENT NOTE: For Marathon/Speedway (MPC LLC/SSA), recoveries based on internal standard area counts for all calibration standards, QC samples and field samples for quantitation must be within 70% to 130%.

- 10.11 Dilutions - All sample analytical results must be below the high standard of the calibration curve.
- 10.12 IDOC's - The analyst must demonstrate proficiency in performing the analysis as outlined in SOP #030205, *Technical Training and Personnel Qualifications*. Method proficiency must be re-demonstrated anytime a major method modification is made, a major software revision is added, or a major instrument modification is made.

STATE NOTE: Wisconsin GRO requires analysis of five replicates for initial demonstration of capability. Waters must be analyzed at a concentration of 100µg/L, with recoveries falling between 80-120% of the known concentration and the RSD must be <20% to be acceptable. Soils must be analyzed at a concentration of 10mg/kg, with recoveries falling between 75-120% of the known concentration and the RSD must be <20% to be acceptable.

- 10.13 Retention time windows - are calculated over a 72-hr period by taking the average RT of each compound in the ICV and calculating ± 3 SD from this average. This is the retention time window. Retention time windows can vary between instruments.

STATE NOTE: Wisconsin GRO requires verification of the retention time window at the beginning of each data and whenever a new GC column is installed. This can be accomplished as part of the calibration verification.

- 10.14 Manual Integration - All manual integrations must comply with the requirements found in ESC SOP #030215, *Manual Integration Procedure*. Before and after integrations must be available for review by the secondary data reviewer.

CLIENT NOTE: For Marathon/MPC LLC/SSA samples Manual integrations must be reviewed and approved by a supervisor or data review team.

- 10.15 RLV - The reporting limit verification when analyzed must recover within $\pm 50\%$ of the target concentration for the standard.

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STATE NOTE: For all samples analyzed from Minnesota, the reporting limit must be verified at least monthly, with each new initial calibration, or when there has been significant change to the instrument (column replacement, cleaning source, etc.) whichever is more frequent. The reporting limit verification can be performed by either re-injecting the low standard or by re-processing the low standard that was analyzed in the calibration curve. The reporting limit verification (RLV) must recovery within $\pm 40\%$ of the expected concentration. If this criterion is not met, the RLV may be re-analyzed once, instrument maintenance can be performed, a higher concentration standard can be injected, or a new calibration curve must be generated. If a higher concentration standard is utilized, the reporting limit must be raised to the higher level verified.

CLIENT NOTE: For Marathon/MPC LLC/SSA samples a reporting level check standard must be analyzed after each calibration and must recover within 60-140%. If recovery of 60-140% is not met, the reporting limit must be raised, and a back calculation performed at that level. This process must be repeated until an acceptable RL recovery is achieved.

11.0 DATA VALIDATION AND CORRECTIVE ACTION

11.1 All data must undergo a primary review by the analyst. The analyst must check the performance of the initial calibration, mid-point check standard and continuing calibrations to ensure that they meet the criteria of the method. The analyst should review any sample that has quantifiable compounds and make sure that they have been confirmed, if needed. The analyst must also verify that reported results are derived from quantitation between the required RL and the highest standard of the initial calibration curve. All calculations must be checked (any dilutions, %solids, etc.). Data must be checked for the presence or absence of appropriate flags. Comments should be noted when data is flagged.

11.2 All data must undergo a second analyst review. The analyst checking the data must check the performance of the initial calibration, mid-point check standard and continuing calibrations to ensure that they meet the criteria of the method.

11.2.1 The analyst should look at any sample that has quantifiable compounds and check the integration.

11.2.2 All calculations must be checked.

11.2.3 All surrogate recoveries must be checked to see if they are within limits.

11.2.4 Blanks must be clean of all interfering peaks.

11.2.5 Quality control criteria should be checked for the LCS, LCSD, MS, and MSD.

11.2.6 Data must be checked for the presence or absence of appropriate flags. Comments should be noted when data is flagged.

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11.2.7 See SOP #030201, *Data Handling and Reporting*.

11.2.8 See SOP #030208, *Corrective and Preventive Action*.

11.3 Initial calibration - If the initial calibration does not meet the criteria for acceptance using response/calibration factors, then linear regression can be utilized, as long as the correlation coefficient meets the necessary criteria. If the linear regression criteria cannot be met, additional corrective actions are required. Standards must be reviewed and re-prepared, if necessary. Instrument maintenance may also be required, including column clipping/replacement, source cleaning, etc. When corrective actions have been completed, the instrument must be re-calibrated and the acceptance criteria must be met for the analytes of interest prior to the analysis of any field samples.

11.4 Initial/Calibration Check Standard (ICV/CCV) - When the initial or continuing calibration verification is beyond the acceptance criteria and analysis of a second consecutive (immediate) calibration verification fails to produce results within acceptance criteria, corrective actions shall be performed. The laboratory shall demonstrate acceptable performance after the final round of corrective action with two consecutive calibration verifications, or a new initial instrument calibration shall be performed.

11.5 Method Blank – If the method blank shows any detectable amount greater than the MDL, the laboratory performance is assumed to be out of control and the problem must be corrected. Corrective actions include: re-analysis twice. If the failure persists, re-extract the entire batch of samples, if submitted sample volume permits.

General guidelines for qualifying sample results with regard to method blank quality are as follows:

- If the method blank concentration is less than the MDL and sample results are greater than the RL, then no qualification is required.
- No qualification is necessary when an analyte is detected in the method blank but not in the associated samples.
- If the concentration in a sample is more than ten times the concentration in the method blank, then no qualification is required.
- If the method blank concentration is greater than the MDL but less than the RL and sample results are greater than the MDL, then qualify associated sample results to indicate that analyte was detected in the method blank.
- If the method blank concentration is greater than the RL, further corrective action and qualification is required. An analyst should consult their supervisor for further instruction.

11.6 Matrix Spike/Matrix Spike Duplicate - Assess that matrix spike/matrix spike duplicates were analyzed at required frequency, and that results are within acceptance criteria. Spike failure results in the use of a "J" or "V" flag. If a "J" flag is used, it is followed by the appropriate number, which further explains the failure concerning high or low response. The "V" flag is used to indicate that the sample concentration was too high to accurately evaluate the spike recovery.

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- 11.7 Laboratory Control Sample (LCS)/Laboratory Control Sample Duplicate (LCSD) – A Laboratory Control Sample (LCS) is run every 20 samples. Levels correspond to the midpoint of the calibration curve.

STATE NOTE: WI GRO LCS must be within 80 - 120% for both soil and water. An LCS and LCSD are required with each batch. The RPD cannot exceed 20% for either matrix. Failure of the LCS results in a required of all samples within the batch.

STATE NOTE: AK101 GRO LCS must be within 60-120% for both soil and water with RPD not exceeding 20%. Surrogates in AK101 must meet 60% to 120% recovery in laboratory control samples – Blanks, LCS, and LCSD.

If the control does not perform within the ranges listed in LIMS, the laboratory performance is assumed to be out of control and the problem must be corrected. Corrective action can include re-analysis, if instrument malfunction is suspected, or re-preparation and re-analysis of the entire batch, if the failure is suspected as either extraction or sample related.

- 11.8 Surrogates - If the recovery is not within limits stated in LIMS, confirm that there are no errors in the calculations, surrogate solutions and standards. Check the instrument performance. Examine the chromatograms for interfering peaks and integrated areas. Re-calculate the data and/or re-analyze the field sample if any of the above checks reveal a problem. When permitted, flag the data "J1" (surrogate high) or "J2" (surrogate low).

11.8.1 High recoveries may be due to co-eluting matrix interference: examine the sample chromatogram.

11.8.2 Low recoveries may be due to the sample matrix.

STATE NOTE: The surrogate for WI PVOC must recovery >80% for both matrices.

- 11.9 Internal standard - The internal standard area counts must be monitored for all CCVs. ISTDs must recover within 50% to 200% of the area counts from the internal standard area counts of the midpoint standard of the most recent initial calibration sequence. If any internal standard response is beyond the acceptable recovery, corrective action is required. Corrective action can take to form of checking the original calculations to ensure accuracy, re-analysis of the CCV to verify initial results, instrument maintenance (i.e. column clipping or changing, inlet liner cleaning/replacement, etc.) or re-calibration.

The internal standard responses and retention times in the check calibration standard must be evaluated immediately after or during data acquisition. If the retention time for any internal standard changes by more than 30 seconds from the last calibration verification, the chromatographic system must be inspected for malfunctions and corrections must be made, as required. When corrections are made, re-analysis of the CCV or a complete re-calibration is necessary, depending on the impact of the correction on the analytical system.

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Internal standards must be monitored for each sample. ISTDs in samples must meet the -50% to +200% criteria when compared to the ISTDs in the daily CCV or mid-level of the calibration curve, on 12h shifts when full calibration is performed. Possible corrective actions include: if instrument malfunction is suspected, or re-preparation and re-analysis, if the failure is suspected as either extraction or sample related. If the sample has an obvious matrix interferent and the internal standard recovery is greater than 200%, the sample can be diluted (if acceptable reporting limits can be achieved) to minimize the interference or the sample must be re-extracted and re-analyzed to confirm the original results.

11.10 RLV - If the acceptance criteria are not met, the RLV may be re-analyzed once, instrument maintenance can be performed, a higher concentration standard can be injected, or a new calibration curve must be generated. If a higher concentration standard is utilized, the reporting limit must be raised to the higher level verified.

11.11 Data that does not meet acceptable QC criteria may be acceptable for use in certain circumstances.

11.11.1 If the MS/MSD fails in an initial analysis and again upon re-analysis, the data is released with an appropriate qualifier as the failure is accepted as matrix related.

11.11.2 If a calibration verification standard is above the acceptable QC criteria and all samples being bracketed are below the reporting limit, the data is acceptable based on a high calibration bias with undetectable levels in the field samples. Any positive samples require re-analysis.

11.11.3 If a sample duplicate is above the acceptable range for the RPD and the sample concentration is <5X the RL, then the value can be flagged with a "P1" qualifier indicating that the RPD calculation is not applicable at that concentration.

12.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

12.1 The EPA requires that laboratory waste management practice to be conducted consistent with all applicable federal and state laws and regulations. Excess reagents, samples and method process wastes must be characterized and disposed of in an acceptable manner. See *ESC Waste Management Plan*.

12.2 See SOP #030302, *Environmental Sustainability & Pollution Prevention*.

13.0 METHOD MODIFICATIONS/CLARIFICATIONS

13.1 Provisions for additional QC and specific variations have been added.

13.2 Modifications to this method are noted in the body of the text as state notes. Compliance analyses performed in conjunction with specific state requirements must be performed as noted within the specific state(s) note listed.

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- 13.3 Adjustments to the concentrations of standards/spiking solutions, standards providers, and quality control are subject to change to better meet client/project/regulatory needs or to improve laboratory method performance.
- 13.4 With the exception of samples for WI PVOC/GRO, a maximum of 200µL of methanol extract is injected into 5mL of water for a multiplier of 25X. For WI PVOC/GRO samples, a maximum of 100µL is injected into 5mL for a multiplier of 50X.
- 14.0 REFERENCES
- 14.1 *Nonhalogenated Organics Using GC/FID*, SW-846 Method 8015B, Revision 2, December 1996.
- 14.2 *Aromatic and Halogenated Volatiles by Gas Chromatography using Photoionization and/or Electrolytic Conductivity Detectors*, SW-846 Method 8021B, Revision 2, December 1996.
- 14.3 *Modified GRO Method for Determining Gasoline Range Organics*, Wisconsin DNR, September 1995.
- 14.4 *R211 – Specific Requirements: Wyoming Storage Tank Remediation Testing Laboratory Accreditation Program*, A2LA Document R211, June 30, 2015.
- 14.5 *Method 602 – Purgeable Aromatics*, 40 CFR Part 136 Appendix A.
- 14.6 *Volatile Organic Compounds Purge and Trap Capillary-Column Gas Chromatographic Method*, SM 6200C, Standard Methods for the Examination of Water and Wastewater.
- 14.7 *Method for Determination of Volatile Petroleum Hydrocarbons (Gasoline)*, Iowa Method OA-1 Revision 7/27/93, The University of Iowa, Hygienic Laboratory.
- 14.8 *Leaking Underground Fuel Tank Guidance Manual*, California State Water Resources Control Board, September 2012.
- 14.9 State of Alaska, Dept. of Env. Conservation, Contaminated Sites Laboratory Approval Memorandum, Soil Moisture Corrected Reporting by EPA Method 8000C, February, 2008.
- 14.10 *NWTPH-Gx Volatile Petroleum Products Method for Soil and Water*, Oregon Department of Environmental Quality.
- 14.11 *Method AK101 for the Determination of Gasoline Range Organics*, Version 4/08/02.
- 14.12 *Nonhalogenated Organics by Gas Chromatography*, SW-846 Method 8015C, Revision 3, February 2007.
- 14.13 *Nonhalogenated Organics Using GC/FID*, SW-846 Method 8015D, Revision 4, June 2003.
- 14.14 *Determinative Chromatographic Separations*, SW846 Method 8000B, Revision 2, September 1996

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- 14.15 *Determinative Chromatographic Separations*, SW846 Method 8000C, Revision 3, March 2003
- 14.16 *Louisiana Department of Environmental Quality Leaking Underground Storage Tank Program Quality Assurance Project Plan*, Louisiana DEQ, Revision 10, 5/6/2008.

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Attachment I: Revision History

Current Version:

Version	Date	Description of Revisions
22	3/2/2018	Update as corrective action for 2017 A2LA audit. Technical and Quality review and update. Revised sections 2.3, 3.3, 4.2.1, 4.2.1.1, 6.1, 6.3.2, 6.4.1, 7.2.1, 7.7, 7.8, 8.4.1, 8.4.2, 8.4.3, 8.4.5, 10.5, 14.1 through 14.10, and 14.12 through 14.16. Deleted state note in section 4.2.1. Added section 10.6.1.

Superseded Versions:

This document supersedes the following:

Version	Date	Description of Revisions
0	8/94	Origination
1	7/95	
2	12/28/98	
3	9/1/99	
4	8/22/00	
5	11/1/01	
6	4/29/02	
7	4/23/03	
8	11/3/03	
9	4/14/04	
10	8/15/06	
11	11/30/07	Technical and Quality Review and update.
12	1/10/08	Addition of Section 4.5.4 - AK101 requirements.
13	2/23/09	Clarification of spike solutions in section 7.9; addition of state notes; inclusion of calculations for average response factors, linear calibration and correlation coefficient; addition of corrective actions in section 11.3 through 11.8; Clarifications in sections 12.0 & 13.0. Ohio VAP approved 2/23/09.
14	3/23/12	Technical and Quality Review and update. Added sections 1.3.1, 2.14 through 2.28, 10.13, and state/client notes in sections 1.0, 8.3.2, 8.5.2, and 11.10; Revised Attachments II and III and sections 1.2, 2.4, 7.1, 7.5, 7.8, 8.3, 9.1 through 9.8, 10.3 through 10.12, 11.1 through 11.9, 12.1, 14.7, 14.9, and 14.10; Incorporated previous minor revisions.
15	6/10/13	Technical and Quality Review and update. Added sections 4.5, 10.14 and 11.10, ; Deleted WY note in sections 8.0 and MN note in section 11.11.4, Revised Attachment III and sections 1.2, 6.1, 7.1, 7.4.1, 7.8, 8.3, 8.4 and 14.4.
16	10/24/14	Technical and Quality Review and update. Deleted state note in sections 2.1, 4.2.2.2, 8.0, 8.3.2, 10.5, 11.5 and 11.8; Deleted sections 7.3.3, 7.4.3 and 8.4; Revised sections 1.1, 2.4, 6.1, 7.5, 10.4 and 11.4.
17	8/5/2015	Technical and Quality Review and update. Revised Sections 8.4.4.1, 12.2, and 13.1. Added Section 13.4. Added State Note in Sections 4.2.1 and 8.4.4.1.

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Version	Date	Description of Revisions
18	9/2/2015	Header and signature block formatting update. Added Attachment IV.
19	8/31/2016	Technical and quality review and update. Header and signature block re-formatting. Revised Sections 1.0, 1.3.1, 2.4, 4.2.2.2.2, 4.5, 6.1, 6.3.1, 6.4.3, 7.3.1, 7.3.2, 7.3.4, 7.4.1, 7.7, 8.0, 8.2, 8.3.2, 8.3.3, 8.3.4, 8.3.5, 8.3.6, 8.4, 8.4.1, 8.4.2, 8.4.3, 8.4.1, 8.5.1, 8.5.2, 9.1, 9.210.3, 10.4, 10.5, 10.6, 10.7, 10.8, 10.13, 10.14, 11.2.8, 11.4, 11.5, 11.6, 11.7, 11.8, and Attachment 2 Section 4.5. Deleted Sections 2.5 through 2.28, 6.4.2, 7.1, 9.3 through 9.8, and 11.11.1. Added Sections 6.3.3 and 7.2.4.
20	2/7/2017	Technical and quality review and update. Revised Sections 1.0, 1.3, 4.4.2, 8.0, 8.3, 8.3.5, 8.4, 8.4.1, 8.4.3, 10.5, and 14.1 through 14.16. Deleted Section 8.3.1. Added Sections 4.2.1.1, 8.5.3, and 10.4.
21	11/28/2017	Update as corrective action for 2017 A2LA audit. Changed ESC logo. Revised Section 3.1 and Attachment III Table 5.

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Attachment II: Routine Reporting Limits*

Compound	RL SOIL (mg/Kg) 1g sample size	RL Water (mg/L)
Benzene	0.0025	0.0005
Toluene	0.025	0.005
Ethylbenzene	0.0025	0.0005
M & P Xylenes	0.0050	0.0010
O Xylenes	0.0025	0.0005
MTBE	0.0250	0.005
GRO	0.5	0.10
Compound	RL methanol (mg/Kg) extract by 5035A	RL Sodium bisulfate (mg/Kg)
Benzene	0.025 (AK101 0.020)	0.0005
Toluene	0.25	0.005
Ethylbenzene	0.025	0.0005
M & P Xylenes	0.050	0.0010
O Xylenes	0.025	0.0005
MTBE	0.250	0.005
GRO	5.0	0.10

*See section 13.3.

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Attachment III; DOD Requirements

1.0 Equipment/Instrument Maintenance

Instrument maintenance must be performed routinely to optimize instrument performance and improve chromatography. Commonly performed maintenance includes baking traps and columns, changing injection port liners, etc. A new calibration curve must be analyzed following any major maintenance performed on the analytical system.

2.0 Computer Hardware and Software

Software name and version: HP Chemstation G1701BA Version B.01.00 or equivalent

3.0 Troubleshooting

Table 1. GC Troubleshooting Guide		
Problem	Cause	Treatment
No Peaks	Syringe clogged	Clean or replace syringe
	Detector/Software/Computer failure	Check cables. Restart computer.
	Column Leaks	Use new ferrules.
	Broken Column	If at ends, clip column. If in the middle or multiple sites, replace column.
Peaks too Small	Split too high	Reduce split
	Column connection leaks	Check column installation. Search for leaks. Replace ferrules.
	Injector temperature too low	Check temperature program. Increase injector temperature.
	Dirty PID	Clean PID.
Retention Times Change	Gas flow too low or too high	Replace septum. Check gas regulator.
	Oven temperature unstable	Check temperature program. Check temperature with external thermometer.
	Column blocked	Compare flow at column entrance to outlet. Replace column.
Constantly Rising Baseline	Leak at column entrance or injection septum.	Check column installation; search for leaks; replace ferrules.
	Injector contaminated.	Make a run at lower injector temperature; if the baseline improves, replace liner, use low bleed or high temperature septa.
	Column contaminated.	Cut two turns from column entrance; rinse column with solvent (only chemically bonded phases); otherwise replace column or use guard column.
	Detector contaminated.	Clean detector.
	Increase of temperature too fast.	Decrease temperature gradient and end temperature.



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Table 1. GC Troubleshooting Guide

Problem	Cause	Treatment
	Poor gas quality.	Use gas grades recommended for GC; for longer supply lines from gas source to GC use gas purification cartridges directly connected to the GC.
Increasing Baseline at High Temperatures	Decomposition of the stationary phase.	Check for leaks; matrix check for compatibility with the column.
	Column contaminated.	Cut two turns from column entrance; rinse column with solvent (only chemically bonded phases); otherwise replace column or use guard column.
	Increase of temperature too fast / end temperature too high.	Decrease temperature gradient and end temperature.
	Column not properly conditioned.	Condition column according to manufacturers' instructions (while column is not connected to the detector).
	Detector contaminated	Clean detector according to manufacturers' instructions.
	Poor gas quality.	Use gas grades recommended for GC; for longer supply lines from gas source to GC use gas purification cartridges directly connected to the GC.
Plateaus at Certain Temperatures	Steps in temperature program too drastic.	Avoid very short and strong heating periods.
Fronting	Column overload.	Decrease injection volume; dilute sample.
	Sample vaporizes too slowly, not evenly or condenses.	Increase injector temperature (consider max. temperature limits of the column).
	Analytes coelute.	Change temperature program or use column with different selectivity.
	Sample decomposes.	Check temperature program, oven temperature (external thermometer); if analytes are not temperature-stable, reduce injector temperature; replace liner.
	Column absorbs or decomposes analytes.	Check capillary ends; check intact deactivation using the test mixture; for poor results shorten both column ends by about 10 cm; or replace column; if column test does not show any defects: a) use a column with thicker film b) use phase with better deactivation c) use column with special selectivity.

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Table 1. GC Troubleshooting Guide

Problem	Cause	Treatment
Tailing	Sample vaporizes too slowly, not evenly or condenses.	Increase injector temperature (consider max. temperature limits of the column).
	System leaks.	Check column installation; search for leaks; replace ferrules.
	Analytes coelute.	Change temperature program or use column with different selectivity.
	Sample decomposes.	Check temperature program, oven temperature (external thermometer); if analytes are not temperature-stable, reduce injector temperature; replace liner by a deactivated one.
	Column absorbs or decomposes analytes.	Check capillary ends; check intact deactivation using the test mixture; for poor results shorten both column ends by about 10 cm; or replace column; if column test does not show any defects: a) use a column with thicker film b) use phase with better deactivation c) use column with special selectivity.
	Split rate too low.	Increase split rate.
	Column overload.	Decrease injection volume; dilute sample.
Split Peaks	Solvent and column not compatible.	Change solvent or use guard column.
	Solvent mixtures with large differences in boiling point and polarity.	Use just one solvent.
	Sample decomposes.	Check temperature program, oven temperature (external thermometer); if analytes are not temperature-stable, reduce injector temperature; replace liner by a deactivated one.
	Analytes coelute.	Modify temperature program or use longer column; possibly change column polarity.
	Detector overload.	Inject less; control make-up flow.

4.0 Other Requirements

- 4.1 All hardcopy laboratory notebooks must be reviewed by the Supervisor, or their designee, on a monthly basis.
- 4.2 If not self-explanatory (e.g., a typo or transposed number), corrections to technical and quality records shall also include a justification for the change.

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- 4.3 A storage blank must be stored with all volatile organic samples, regardless of suspected concentration levels.
- 4.4 A person performing a manual integration must sign and date each manually integrated chromatogram and record the rationale for performing manual integration. Electronic signatures are acceptable.
- 4.5 The results of calibration and verification of support equipment must be within the specifications required of the application for which this equipment is used or the equipment must be removed from service until repaired. Calibration and verification records, including those of established correction factors, must be maintained. In the absence of method-specific requirements, the minimum requirements are as follows:

Table 2. Support Equipment Checks		
Performance Check	Frequency	Acceptance Criteria
Balance calibration check [Using two standard weights that bracket the expected mass]	Daily prior to use	Top-loading balance: $\pm 2\%$ or $\pm 0.02\text{g}$, whichever is greater Analytical balance: $\pm 0.1\%$ or $\pm 0.5\text{mg}$, whichever is greater
Verification of standard mass [Using weights traceable to the International System of Units (SI) through a NMI]	Every 5 years	Certificate of Calibration from ISO/IEC 17025 accredited calibration laboratory
Monitoring of refrigerator/freezer temperatures	Daily (i.e. 7 days per week) [use MIN/MAX thermometers or data loggers equipped with notification of out of control event capabilities if personnel not available to record daily]	Refrigerators: 0°C to 6°C Freezers: $\leq -10^{\circ}\text{C}$
Thermometer verification check [Using a thermometer traceable to the SI through an NMI] [Performed at two temperatures that bracket the target temperature(s). Assume linearity between the two bracketing temperatures.] [If only a single temperature is used, at the temperature of use]	Liquid in glass: Before first use and annually Electronic: Before first use and quarterly	Apply correction factors or replace thermometer
Volumetric labware	Class B: By lot before first use Class A and B: Upon evidence of deterioration	Bias: Mean within $\pm 2\%$ of nominal volume Precision: RSD $\leq 1\%$ of nominal volume (based on 10 replicate measurements)



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Table 2. Support Equipment Checks

Performance Check	Frequency	Acceptance Criteria
Non-volumetric labware [Applicable only when used for measuring initial sample volume and final extract/ digestates volume]	By lot before first use or upon evidence of deterioration	Bias: Mean within $\pm 3\%$ of nominal volume Precision: RSD $\leq 3\%$ of nominal volume (based on 10 replicate measurements)
Mechanical volumetric pipette	Quarterly	Bias: Mean within $\pm 2\%$ of nominal volume Precision: RSD $\leq 1\%$ of nominal volume (based on minimum of 3 replicate measurements) [Note: for variable volume pipettes, the nominal volume is the volume of use]
Glass microliter syringe	Upon receipt and upon evidence of deterioration	General Certificate of Bias & Precision upon receipt Replace if deterioration is evident
Drying oven temperature check	Daily prior to and after use	Within $\pm 5\%$ of set temperature
Water purification system	Daily prior to use	See method blank criteria given in Section 4.20 of this addendum

- 4.6 The expiration date of the prepared standard shall not exceed the expiration date of the primary standard. All containers must bear a preparation date.
- 4.7 To avoid preparing non-representative samples, the laboratory shall not "target" within a relatively small mass range (e.g., $1.00 \pm 0.01\text{g}$) because such targeting will produce non-representative subsamples if the sample has high heterogeneity. The laboratory shall not manipulate the sample material so the sample aliquot weighs exactly $1.00\text{g} \pm 0.01\text{g}$, as an example.
- 4.8 In the absence of project-specific requirements, the minimum standard data qualifiers to be used are:
- U Analyte was not detected and is reported as less than the LOD or as defined by the customer. The LOD has been adjusted for any dilution or concentration of the sample.
 - J The reported result is an estimated value (e.g., matrix interference was observed or the analyte was detected at a concentration outside the quantitation range).
 - B Blank contamination. The recorded result is associated with a contaminated blank.

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N Non-target analyte. The analyte is a tentatively identified compound using mass spectrometry or any non-customer requested compounds that are tentatively identified.

Q One or more quality control criteria failed (e.g., LCS recovery, surrogate spike recovery, or CCV recovery).

Additional data qualifiers may be used, or different letters or symbols to denote the qualifiers listed above, as long as they are appropriately defined and their use is consistent with project-specific requirements (e.g., QSM 5.0, the contract, and project-planning documents).

4.9 If the time of the sample collection is not provided, assume the most conservative time of day. For the purpose of batch processing, the start and stop dates and times of the batch preparation shall be recorded.

4.10 Each preparation method listed on the scope of accreditation must have quarterly LOD/LOQ verifications. However, not all possible combinations of preparation and cleanup techniques are required to have LOD/LOQ verifications. If LOD/LOQ verifications are not performed on all combinations, the laboratory must base the LOD/LOQ verifications on the worst case basis (preparation method with all applicable cleanup steps).

4.11 After each MDL determination, the laboratory must establish the LOD by spiking a quality system matrix at a concentration of at least 2 times but no greater than four times the MDL. This spike concentration establishes the LOD and the concentration at which the LOD shall be verified. It is specific to each suite of analyte, matrix, and method (including sample preparation). The following requirements apply to the initial LOD establishment and to the LOD verifications:

- The apparent signal to noise (S/N) ratio at the LOD must be at least three and the results must meet all method requirements for analyte identification (e.g., ion abundance, second column confirmation, or pattern recognition). For data systems that do not provide a measure of noise, the signal produced by the verification sample must produce a result that is at least three standard deviations greater than the mean method blank concentration. This is initially estimated based on a minimum of four method blank analyses and later established with a minimum of 20 method blank results.
- If the LOD verification fails, then the laboratory must repeat the MDL determination and LOD verification or perform and pass two consecutive LOD verifications at a higher spike concentration and set the LOD at the higher concentration.
- The laboratory shall maintain documentation for all MDL determinations and LOD verifications.

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- The DL and LOD must be reported for all analyte-matrix-methods suites, unless it is not applicable to the test or specifically excluded by project requirements.
- 4.12 The LOD shall be verified quarterly. In situations where methods are setup and used on an infrequent basis, the laboratory may choose to perform LOD verifications on a one per batch basis. All verification data will be in compliance, reported, and available for review.
- 4.13 For DoD, at a minimum, the LOQ shall be verified quarterly. In situations where methods are setup and used on an infrequent basis, the laboratory may choose to perform LOQ verifications on a one per batch basis.
- 4.14 All initial instrument calibrations must be verified with a standard obtained from a second manufacturer prior to analyzing any samples. The use of a standard from a second lot obtained from the same manufacturer (independently prepared from different source materials) is acceptable for use as a second source standard. The concentration of the second source standard shall be at or near the midpoint of the calibration range. The acceptance criteria for the initial calibration verification must be at least as stringent as those for the continuing calibration verification.
- 4.15 Exclusion of calibration points without documented scientifically valid technical justification is not permitted.
- 4.16 The concentration of the CCV standard shall be greater than the low calibration standard and less than or equal to the midpoint of the calibration range.
- 4.17 All CCVs analyzed must be evaluated and reported. If a CCV fails, reanalysis or corrective actions must be taken.
 - If a CCV fails, the laboratory can immediately analyze two additional consecutive CCVs (immediately is defined as starting a consecutive pair within one hour; no samples can be run between the failed CCV and the two additional CCVs). This approach allows for spurious failures of analytes to be reported without reanalysis of samples. Any corrective actions that change the dynamics of the system (e.g., clip column, clean injection port, run blanks) requires that all samples since the last acceptable CCV be reanalyzed.
 - Both of these CCVs must meet acceptance criteria in order for the samples to be reported without reanalysis.
 - If either of these two CCVs fail or if the laboratory cannot immediately analyze two CCVs, the associated samples cannot be reported and must be reanalyzed.
 - Corrective action(s) and recalibration must occur if the above scenario fails. All affected samples since the last acceptable CCV must be reanalyzed.
 - Flagging of data for a failed CCV is only appropriate when the affected samples cannot be reanalyzed. The laboratory must notify the client prior to reporting data associated with a failed CCV.

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- 4.18 The results of all MS/MSDs must be evaluated using the same acceptance criteria used for the DoD LCS limits (see Addendum Tables 3 and 4) or project limits, if specified. If the specific analyte(s) are not available in the Addendum Tables 3 and 4, the laboratory shall use their LCS in-house limits (see the LIMS) as a means of evaluating MS/MSDs. The MS and MSD must be spiked with all reported analytes.
- 4.19 Surrogate spike results shall be compared with DoD LCS limits (see Addendum Tables 3 and 4) or acceptance criteria specified by the client. If these criteria are not available, the laboratory shall compare the results with its in-house statistically established LCS criteria (see the LIMS).
- 4.20 The method blank shall be considered to be contaminated if:
 - The concentration of any target analyte (chemical of concern) in the blank exceeds 1/2 the LOQ and is greater than 1/10th the amount measured in any associated sample, or 1/10th the regulatory limit, whichever is greater;
 - The concentration of any common laboratory contaminant in the blank exceeds the LOQ;
 - If a method blank is contaminated as described above, then the laboratory shall reprocess affected samples in a subsequent preparation batch, except when sample results are below the LOD. If insufficient sample volume remains for reprocessing, the results shall be reported with appropriate data qualifiers.
- 4.21 Sporadic Marginal Exceedances are not allowed for target analytes (chemicals of concern as identified by a project) without project-specific approval. Target analytes are considered those few analytes that are critical for the success of a project (such as risk drivers) where sporadic marginal exceedances cannot be allowed. Laboratories should consult with clients whenever long lists of analytes are requested for analysis to determine if marginal exceedances will not be allowed.
- 4.22 DoD considers the same analyte exceeding the LCS control limit two (2) out of three (3) consecutive LCS to be indicative of non-random behavior, which requires corrective action and reanalysis of the LCS.

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Table 3. LCS Control Limits – Method 8015 (MOD) Solid Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
460-00-4	4-Bromofluorobenzene	1263	100.7	11.1	67	134
303-04	Diesel Range Organics (DRO)	2184	85.2	15.7	38	132
307-27	Gasoline Range Organics (GRO)	1134	100.3	7.2	79	122
307-51	Motor Oil	658	72.2	11.2	39	106
84-15-1	o-Terphenyl	314	87.4	14.1	45	130

Table 4. LCS Control Limits – Method 8015 (MOD) Water Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
460-00-4	4-Bromofluorobenzene	756	101	10.8	69	133
303-04	Diesel Range Organics (DRO)	1757	83.7	16	36	132
307-27	Gasoline Range Organics (GRO)	971	99.9	7.3	78	122
307-51	Motor Oil	573	76.9	12.1	41	113
84-15-1	o-Terphenyl	299	90.5	11.4	56	125
630-02-4	Octacosane	130	101.1	13.8	60	142

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Table 5. Quality Control Requirements – Organic Analysis by Gas Chromatography

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Initial Calibration (ICAL) for all analytes (including surrogates)	At instrument set-up and after ICV or CCV failure, prior to sample analysis.	ICAL must meet one of the three options below: Option 1: RSD for each analyte $\leq 20\%$; Option 2: linear least squares regression for each analyte: $r^2 \geq 0.99$; Option 3: non-linear least squares regression (quadratic) for each analyte: $r^2 \geq 0.99$.	Correct problem then repeat ICAL.	Flagging is not appropriate.	Minimum 5 levels for linear and 6 levels for quadratic. Quantitation for multicomponent analytes such as chlordane, toxaphene, and Aroclors must be performed using a 5-point calibration. Results may not be quantitated using a single point. No samples shall be analyzed until ICAL has passed.
Retention Time window position establishment	Once per ICAL and at the beginning of the analytical sequence.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA	NA	Calculated for each analyte and surrogate.
Retention Time (RT) window width	At method set-up and after major maintenance (e.g., column change).	RT width is ± 3 times standard deviation for each analyte RT from the 72-hour study.	NA	NA	Calculated for each analyte and surrogate. Only applicable if internal standard calibration is not used.

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Table 5. Quality Control Requirements – Organic Analysis by Gas Chromatography

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All reported analytes within established RT windows. All reported analytes within $\pm 20\%$ of true value.	Correct problem, rerun ICV. If that fails, repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed until calibration has been verified with a second source.
Continuing Calibration Verification (CCV)	Before sample analysis, after every 10 field samples, and at the end of the analysis sequence with the exception of CCVs for Pesticides multi-component analytes (i.e., Toxaphene, Chlordane), which are only required before sample analysis.	All reported analytes and surrogates within established RT windows. All reported analytes and surrogates within $\pm 20\%$ of true value.	Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails or if two consecutive CCVs cannot be run, perform corrective action(s) and repeat CCV and all associated samples since the last successful CCV; Alternately, recalibrate if necessary; then reanalyze all associated samples since the last acceptable CCV.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.	Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

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Table 5. Quality Control Requirements – Organic Analysis by Gas Chromatography

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Internal Standards (IS)	If employed, every field sample, standard, and QC sample.	Retention time within ± 0.06 RRT UNITS from retention time of the midpoint standard in the ICAL; Internal standard signal (area or height) within -50% to +100% of ICAL midpoint standard. On days when ICAL is not performed, the daily initial CCV can be used.	Inspect GC for malfunctions and correct problem. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	If corrective action fails in field samples, data must be qualified and explained in the Case Narrative. Apply Q-flag to analytes associated with the non-compliant IS. Flagging is not appropriate for failed standards.	NA
Method Blank (MB)	One per preparatory batch.	No analytes detected $>1/2$ LOQ or $> 1/10$ the amount measured in any sample or $1/10$ the regulatory limit, whichever is greater.	Correct problem. If required, reprep and reanalyze MB and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

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Table 5. Quality Control Requirements – Organic Analysis by Gas Chromatography

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Laboratory Control Sample (LCS)	One per preparatory batch.	A laboratory must use Table 3 and 4 limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits (see the LIMS) if project limits are not specified.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per preparatory batch.	A laboratory must use Table 3 and 4 limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits (see the LIMS) if project limits are not specified.	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	For matrix evaluation only. If MS results are outside the limits, the data shall be evaluated to determine the source(s) of difference (i.e., matrix effect or analytical error).

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Table 5. Quality Control Requirements – Organic Analysis by Gas Chromatography

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch.	A laboratory must use Table 3 and 4 limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits (see the LIMS) if project limits are not specified. $RPD \leq 30\%$ (between MS and MSD or sample and MD).	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	The data shall be evaluated to determine the source of difference. For Sample/MD: RPD criteria only apply to analytes whose concentration in the sample is greater than or equal to the LOQ.
Surrogate Spike	All field and QC samples.	QC acceptance criteria specified by the project, if available; otherwise use Table 3 and 4 limits or in-house LCS limits (see the LIMS) if analyte(s) are not listed.	Correct problem, then reprep and reanalyze all failed samples for all surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference is present, reanalysis may not be necessary, but the client must be notified prior to reporting data, and failures must be discussed in the case narrative.	Apply Q-flag to all associated analytes if acceptance criteria are not met and explain in the case narrative.	Alternative surrogates are recommended when there is obvious chromatographic interference.

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Table 5. Quality Control Requirements – Organic Analysis by Gas Chromatography

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Confirmation of Positive Results (second column)	All results greater than the DL must be confirmed (except for single column methods such as TPH by Method 8015 where confirmation is not an option or a requirement).	Calibration and QC criteria for second column are the same as for initial or primary column analysis. Results between primary and secondary column RPD \leq 40%.	NA	Apply J-flag if RPD > 40%. Discuss in the Case Narrative.	Use project-specific reporting requirements if available; otherwise, use method requirements if available; otherwise report the result from the primary column.

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