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Revision 3

Liquid Waste Tank Residuals Sampling and Analysis Program Plan

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REVISION SUMMARY

REV. #	DESCRIPTION	DATE OF ISSUE
0	Initial Submittal to SCDHEC	5/10/2010
1	Incorporated Comment Submittal	2/29/2012
2	Document updated to incorporate changes, corrections, clarifications and additional information since initial submittal. Many updates reflect insight gained from LWTRSAPP and LWTRS-QAPP implementation for Tank 16 residuals sampling.	7/31/2013
3	Document updated to incorporate changes, corrections, and additional information gained from sampling cooling coil scale and wet residuals in Tank 12H.	10/26/2015

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ACRONYMS/ABBREVIATIONS

AD	Analytical Development
ADS	Analytical Development Section
ALARA	As Low As Reasonably Achievable
AOP	Analytical Operating Procedure
ARG	Analyzed Reference Glass
ASME	American Society of Mechanical Engineers
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
CFR	Code of Federal Regulation
COC	Chain-of-Custody
CQF	Cognizant Quality Function
CTS	Concentrate Transfer System
DB	Diversion Box
DOE	U.S. Department of Energy
DQA	Data Quality Assessment
DQI	Data Quality Indicator
DQO	Data Quality Objective
DWPF	Defense Waste Processing Facility
EDWS	Electronic Document Workflow System
EPA	U.S. Environmental Protection Agency
ESH&QA&CA	Environmental, Safety, Health & Quality Assurance & Contractor Assurance
FE	Fundamental Error
FFA	Federal Facility Agreement
FTF	F-Area Tank Farm
GCP	General Closure Plan
HSWA	Hazardous and Solid Waste Amendments
HRR	Highly Radioactive Radionuclide
HTF	H-Area Tank Farm
LIMS	Laboratory Information Management System
LWTRSAPP	Liquid Waste Tank Residuals Sampling and Analysis Program Plan
LWTRS-QAPP	Liquid Waste Tank Residuals Sampling-Quality Assurance Program Plan
M&O	Management and Operations
MDC	Minimum Detectable Concentration
MPC	Measurement Performance Criteria
NQA-1	Nuclear Quality Assurance
PA	Performance Assessment
PIC	Person in Charge
PP	Pump Pit
PSQ	Principal Study Question
QA	Quality Assurance
QAP	Quality Assurance Plan
QAPP	Quality Assurance Program Plan
QC	Quality Control
R&D	Research and Development

RadCon	Radiological Controls
RCRA	Resource Conservation and Recovery Act
RM&A	Regulatory Management & Administration
SA	Special Analysis
SCDHEC	South Carolina Department of Health and Environmental Control
SCO	Shielded Cells Operations
SDF	Saltstone Disposal Facility
SET	Sample Extraction Team
SLDR	Sample Location Determination Report
SOP	Standard Operating Procedure
SPF	Saltstone Production Facility
SRNL	Savannah River National Laboratory
SRNS	Savannah River Nuclear Solutions, LLC
SRR	Savannah River Remediation LLC
SRS	Savannah River Site
TFO	Tank Farm Operations
TSAP	Tank-Specific Sampling and Analysis Plan
TTQAP	Task Technical and Quality Assurance Plan
TTR	Technical Task Request
VP	Volume-Proportional

FOREWORD

This Liquid Waste Tank Residuals Sampling and Analysis Program Plan (LWTRSAPP) is designed to provide programmatic guidance for the sampling and analysis of residuals material in the liquid radioactive waste tanks being removed from service at the Savannah River Site (SRS), Aiken, South Carolina.

Waste tank residuals sampling data and the subsequent residuals characterization that the data support are used as input to complex engineering computational models to assess the long-term fate and transport of the chemical and radiological constituents in the environment. That assessment enables prediction of future performance of the engineered final closure system and prediction of future sustained conformity with relevant and appropriate environmental guidelines. Waste tank residuals sampling is not performed for the purpose of measuring concentrations against prescribed environmental limits and, as such, the samples collected and analyzed under this plan are not environmental compliance samples and are not used for environmental monitoring.

SRS operations are governed by the 1Q Manual, *Quality Assurance Manual,* as it applies to the waste tank residuals sampling and analysis. 1Q Manual Procedure 21-1, *Quality Assurance Requirements for the Collection & Evaluation of Environmental Data*, does not apply to the sampling and analysis of waste tank residuals. SRS has prepared a Liquid Waste Tank Residuals Sampling-Quality Assurance Program Plan (LWTRS-QAPP) to support field and analytical services for waste tank residuals characterization. [SRR-CWDA-2011-00117]

This LWTRSAPP has been developed consistent with U.S. Department of Energy (DOE) Manual 435.1-1 and serves as an upper-tier description document that integrates the technical, regulatory, and quality attributes[1]. This integrated information is utilized to assure that the waste tank residuals material has been accurately characterized.

This LWTRSAPP was not applicable to Tanks 5, 6, 18, or 19 which have undergone removal from service activities. The LWTRSAPP and companion LWTRS-QAPP, were approved and implemented to provide the Quality Assurance (QA)/Quality Control (QC) protocols for future residuals sampling and analysis for waste tank removal from service activities.

The objectives of this LWTRSAPP are to describe the following:

- The general methodology used to sample residuals that will remain in waste tanks following concurrence by South Carolina Department of Health and Environmental Control (SCDHEC) and U.S. Environmental Protection Agency (EPA) to proceed with sampling and analysis,
- The general Savannah River National Laboratory (SRNL) procedures used to analyze residual material samples, and
- The QA/QC activities necessary to demonstrate conformity with applicable performance objectives.

[1] DOE's submittal of this plan does not waive any DOE claim of jurisdiction over matters reserved to it under the Atomic Energy Act of 1954 as amended.

Tank-Specific Sampling and Analysis Plans (TSAPs) will be subsequently developed on a tank-by-tank basis to manage the residuals material variations and distributions expected between waste tanks. The TSAPs will be consistent with this program plan.

1.0 INTRODUCTION

Since the early 1950s, the primary mission of SRS had been to produce nuclear materials for use in national defense and deep space missions. The processes used to recover these nuclear materials from production reactor fuel and target assemblies in the chemical separations areas at SRS generated significant volumes of liquid radioactive waste. This waste is currently stored in underground waste tanks in F- and H-Areas near the center of the site. Today, the primary focus at SRS is environmental restoration with the highest priority being removal, treatment, and disposal of the liquid radioactive waste in the F-Area Tank Farm and H-Area Tank Farm (FTF/HTF).

In support of environmental remediation activities at SRS, the DOE, EPA, and SCDHEC signed a Federal Facility Agreement (FFA) pursuant to Section 120 of the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) and Sections 3008(h) and 6001 of the Resource Conservation and Recovery Act (RCRA), as amended by the Hazardous and Solid Waste Amendments of 1984 (HSWA) (usually jointly referred to as RCRA) and the Atomic Energy Act of 1954 as amended. The agreement became effective in August 1993. As part of this comprehensive agreement, DOE has committed to remove from service those waste tank systems that do not meet the standards set forth in Appendix B of the FFA. Appendix B of the FFA also defines the specific waste tank systems that are subject to the agreement. [WSRC-OS-94-42]

After completion of waste removal activities from individual waste tank systems, the waste tank systems are operationally closed under the industrial wastewater permit that regulates their operation. SCDHEC regulates the process of waste tank system removal from service via applicable South Carolina law and regulation, and the SRS FFA. The use of the terms "operational closure" and "removal from service" are considered synonymous.

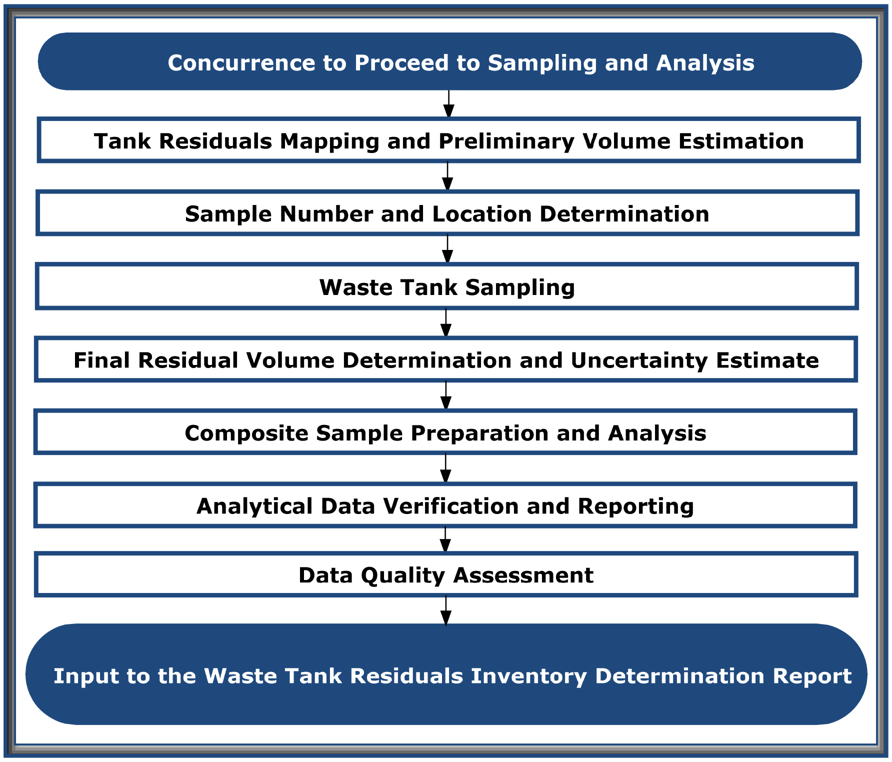
1.1 Purpose of the Sampling and Analysis Program Plan

The purpose of the LWTRSAPP is to establish the framework to characterize waste tank residuals material so that quality data is generated to support the residuals material inventory determination.

This LWTRSAPP presents the activities and organizations necessary to satisfy the Data Quality Objectives (DQOs) of the sampling and analysis effort and is based on requirements in the DOE Manual 435.1-1, guidance in EPA/240/B-06/001, and requirements of the FFA, Section IX.E (WSRC-OS-94-42). Elements of this LWTRSAPP are designed to ensure conformance with the QA/QC requirements of DOE over the life of the waste tank removal from service efforts. Ancillary structures, such as evaporators, catch tanks, pump pits, etc., will be addressed under specific sampling and analysis plans that will be consistent with the LWTRSAPP and LWTRS-QAPP. In March 2012, following comment resolution, SCDHEC approved the LWTRSAPP Revision 1 and LWTRS-QAPP Revision 0. (DHEC_03-07-2012-01) Similarly, EPA was satisfied that all of their comments had been resolved and had no further comments on the documents. (EPA_03-01-2012)

The general waste tank residuals characterization process and information flow is summarized in Figure 1.1-1.

Figure 1.1-1: General Waste Tank Residuals Characterization Process Flowchart



Note: Colored ovals are predecessor and successor activities, clear rectangles are activities associated with the LWTRSAPP and LWTRS-QAPP.

1.1.1 Document Organization for LWTRSAPP

This LWTRSAPP is organized in the following manner (Table 1.1-1).

Table 1.1-1: LWTRSAPP Organization

Section	Contents
1	Introduction
2	Savannah River Remediation LLC (SRR) Quality Assurance and Project Team Roles & Responsibilities
3	Data Quality Objectives
4	Sampling
5	Analytical Methods
6	Data and Records Management

1.2 Background and History of SRS Waste Tank Operations

The SRS mission generated liquid waste from chemical separations processes in both F and H Areas. Since the beginning of SRS operations, an integrated waste management system consisting of several facilities designed for the overall processing of liquid radioactive waste has evolved. Two of the major components of this system are the FTF and HTF located in F and H Areas, respectively, which are near the center of the site. The F- and H-Chemical Separations Facilities are where plutonium, uranium, and other radionuclides were extracted from irradiated fuel and target assemblies using chemical separation processes. The resultant liquid radioactive wastes were transferred to the FTF and HTF for storage, treatment, and disposition. The tank farms include waste tanks, evaporators, transfer line systems, and other ancillary structures and equipment.

The tank farm locations were chosen because of their favorable terrain and proximity to the F- and H-Chemical Separations Facilities, the major waste generation sources. There are four principal types of waste tanks designated as Type I, II, III/IIIA, and IV tanks in the FTF and HTF. The waste tank numbering and design type are shown in Table 1.2-1.

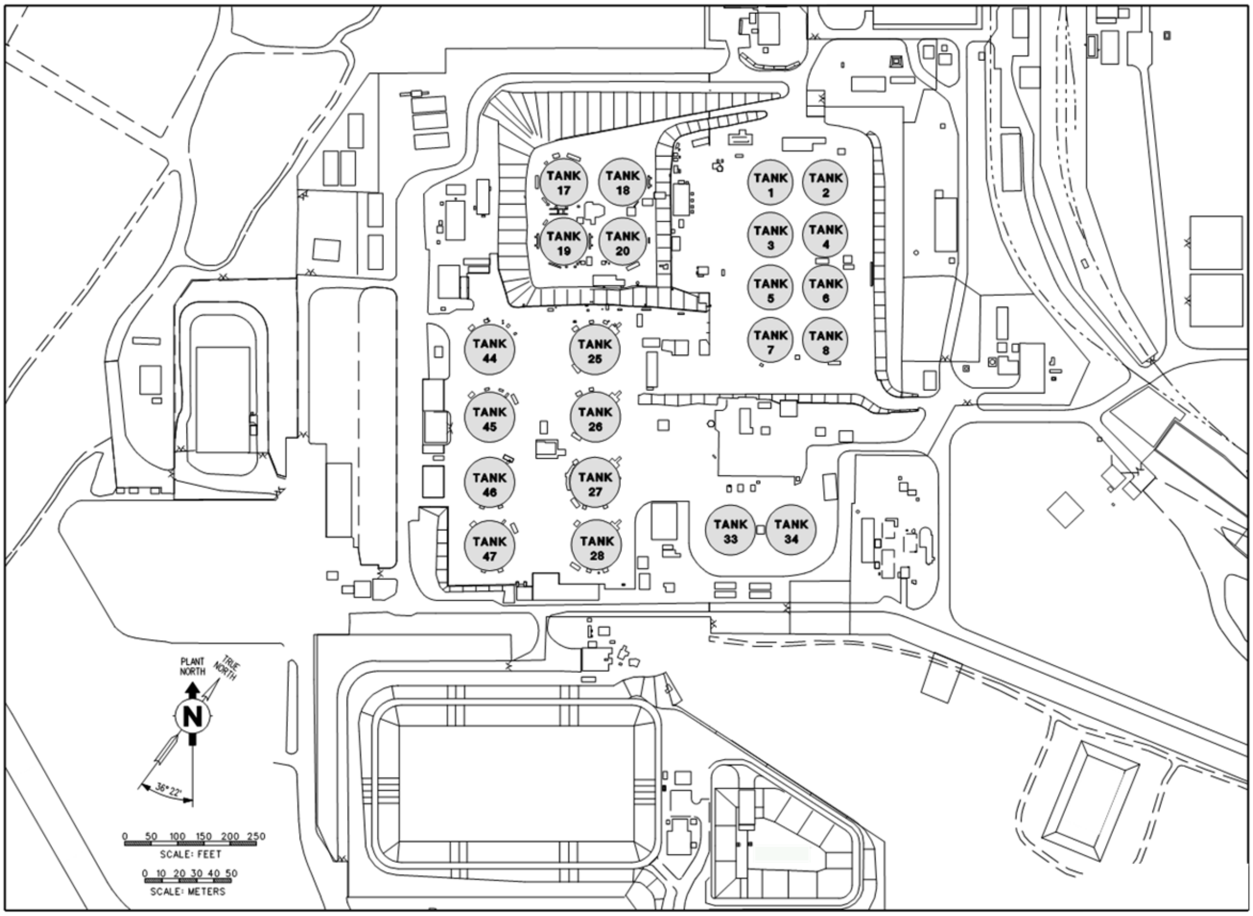
The FTF is a 22-acre site consisting of 22 liquid waste storage tanks, two evaporator systems, transfer pipelines, six diversion boxes (DBs), one catch tank, a concentrate transfer system (CTS) tank, and three pump pits (PPs). Figure 1.2-1 shows the general layout of FTF. There are three major waste tank types in FTF that range in size from 750,000 gallons (Type I tanks) to 1,300,000 gallons (Type III/IIIA and Type IV tanks) and have varying degrees of secondary containment and internal tank interferences to sampling, such as cooling coils and roof support columns. The four Type IV tanks (Tanks 17-20) and two Type I tanks (Tanks 5, 6) in the FTF have been operationally closed.

Table 1.2-1: Summary of FTF/HTF Waste Tank Design Types and Construction Details

Tank Number	Tank Farm	Type	Year Constructed	Volume
1	F	I	1952-1953	750,000
2	F	I	1952-1953	750,000
3	F	I	1952-1953	750,000
4	F	I	1952-1953	750,000
5[a]	F	I	1952-1953	750,000
6[a]	F	I	1952	750,000
7	F	I	1952-1953	750,000
8	F	I	1952-1953	750,000
9	H	I	1950's	750,000
10	H	I	1950's	750,000
11	H	I	1950's	750,000
12[b]	H	I	1950's	750,000
13	H	II	1955-1956	1,070,000
14	H	II	1955-1956	1,070,000
15	H	II	1955-1956	1,070,000
16[b]	H	II	1955-1956	1,070,000
17[a]	F	IV	1958	1,300,000
18[a]	F	IV	1958	1,300,000
19[a]	F	IV	1958	1,300,000
20[a]	F	IV	1958	1,300,000
21	H	IV	1958-1962	1,300,000
22	H	IV	1958-1962	1,300,000
23	H	IV	1958-1962	1,300,000
24	H	IV	1958-1962	1,300,000
25	F	IIIA	1978	1,300,000
26	F	IIIA	1978	1,300,000
27	F	IIIA	1978	1,300,000
28	F	IIIA	1978	1,300,000
29	H	III	1967-1970	1,300,000
30	H	III	1967-1970	1,300,000
31	H	III	1967-1970	1,300,000
32	H	III	1967-1970	1,300,000
33	F	III	1969	1,300,000
34	F	III	1972	1,300,000
35	H	IIIA	1974-1981	1,300,000
36	H	IIIA	1974-1981	1,300,000
37	H	IIIA	1974-1981	1,300,000
38	H	IIIA	1974-1981	1,300,000
39	H	IIIA	1974-1981	1,300,000
40	H	IIIA	1974-1981	1,300,000
41	H	IIIA	1974-1981	1,300,000
42	H	IIIA	1974-1981	1,300,000
43	H	IIIA	1974-1981	1,300,000
44	F	IIIA	1980	1,300,000
45	F	IIIA	1980	1,300,000
46	F	IIIA	1980	1,300,000
47	F	IIIA	1980	1,300,000
48	H	IIIA	1974-1981	1,300,000
49	H	IIIA	1974-1981	1,300,000
50	H	IIIA	1974-1981	1,300,000
51	H	IIIA	1974-1981	1,300,000

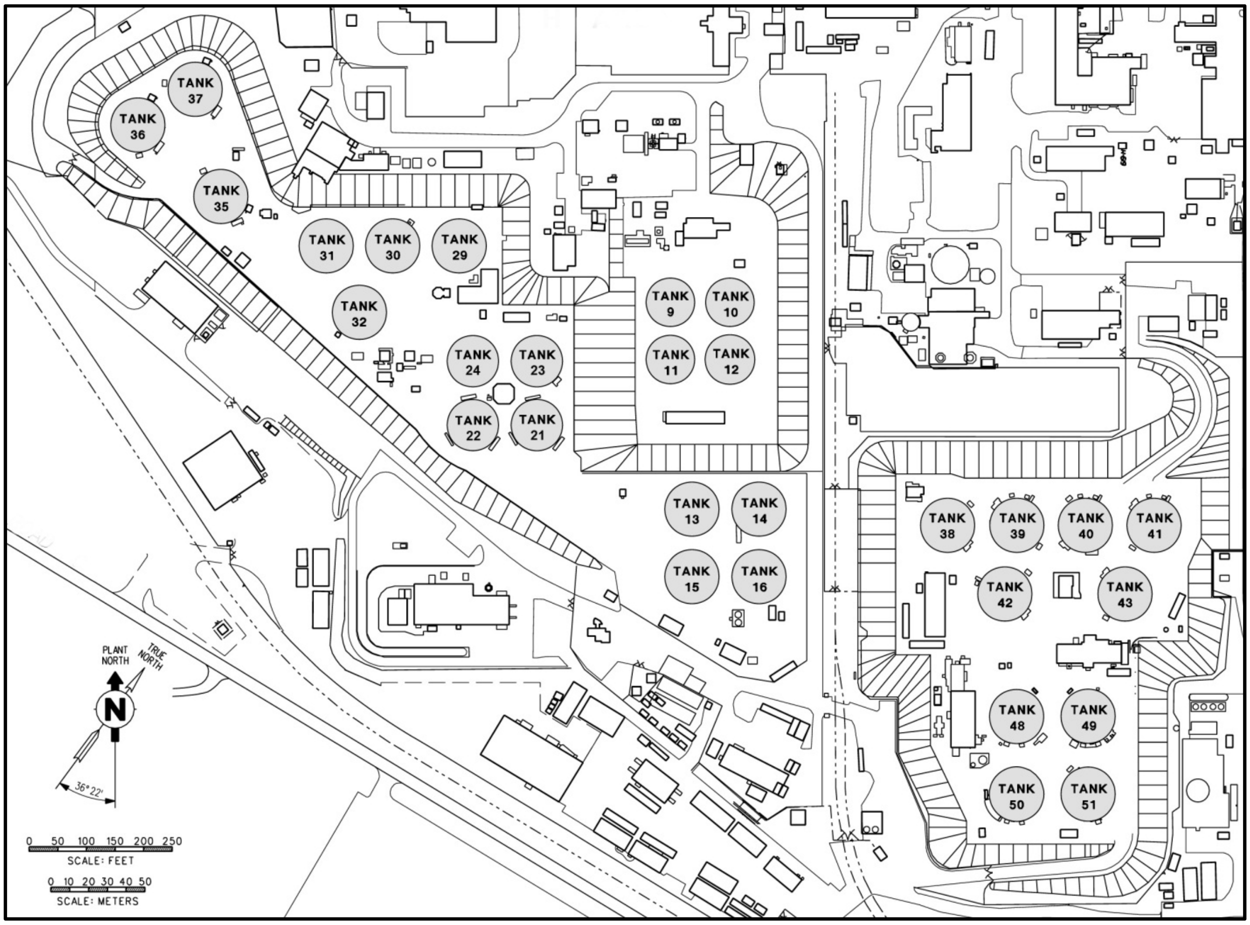
[a] Operationally closed
[b] Undergoing operational closure

Figure 1.2-1: General Layout of F-Area Tank Farm



The HTF is an approximately 45-acre site consisting of 29 liquid waste storage tanks, three evaporator systems, transfer pipelines, eight DBs, one catch tank, two CTS tanks, and 10 PPs. Figure 1.2-2 shows the general layout of HTF. There are four major waste tank types in HTF that range in size from 750,000 gallons (Type I tanks) to 1,300,000 gallons (Type III/IIIA and Type IV tanks) and have varying degrees of secondary containment and internal tank interferences to sampling. Tank 16 (Type II) and Tank 12 (Type I) are currently undergoing operational closure.

Figure 1.2-2: General Layout of H-Area Tank Farm

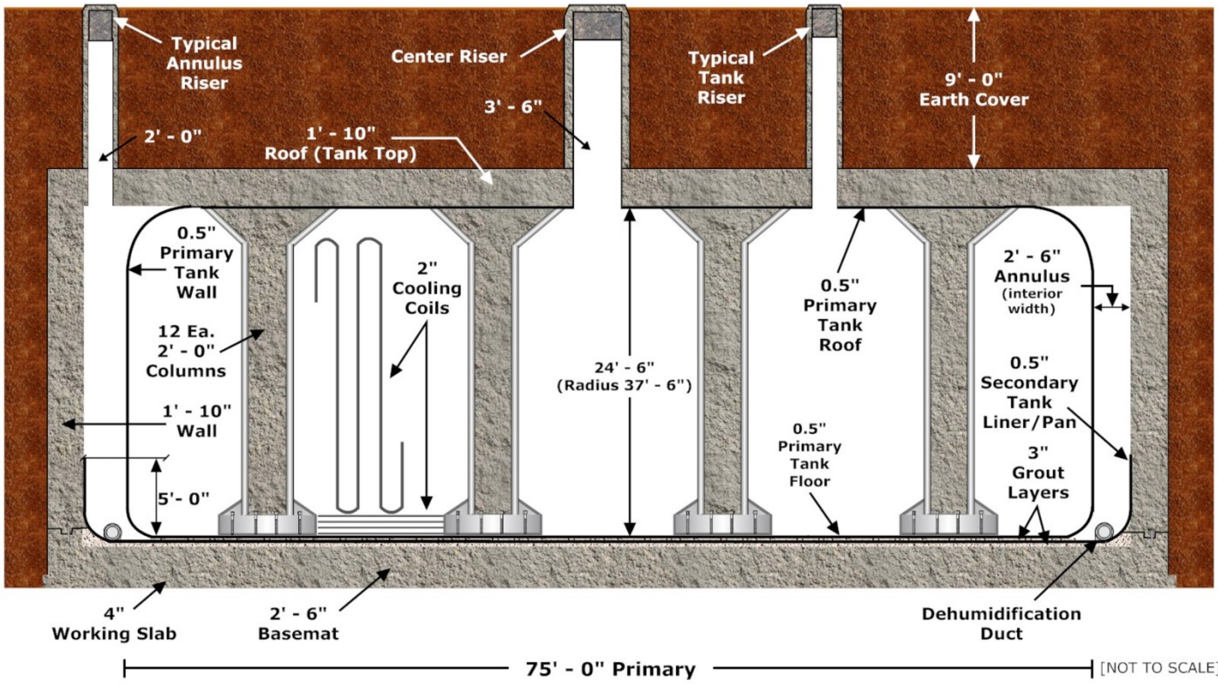


Within FTF and HTF, facilities are in place to pretreat the accumulated solids and salt solutions (supernate) to enable further waste treatment within other SRS facilities (i.e., Defense Waste Processing Facility [DWPF] and Saltstone Production Facility [SPF]). These treatment facilities convert the solids and supernate to more stable forms suitable for permanent disposal in a Federal repository or the Saltstone Disposal Facility (SDF), as appropriate. Extensive descriptions of the FTF and waste processing facilities are provided in the FTF Performance Assessment (PA). [SRS-REG-2007-00002] A description of the HTF and waste processing facilities are provided in the HTF PA. [SRR-CWDA-2010-00128]

1.2.1 Type I Waste Tanks

Eight Type I tanks are located in the FTF and four in the HTF. The Type I primary liners are 75 feet in diameter and 24.5 feet high, with a nominal operating capacity of 750,000 gallons. A typical Type I tank is shown in Figure 1.2-3. Additional details of the Type I tanks are provided in Section 3 of the FTF and HTF PAs. [SRS-REG-2007-00002, SRR-CWDA-2010-00128]

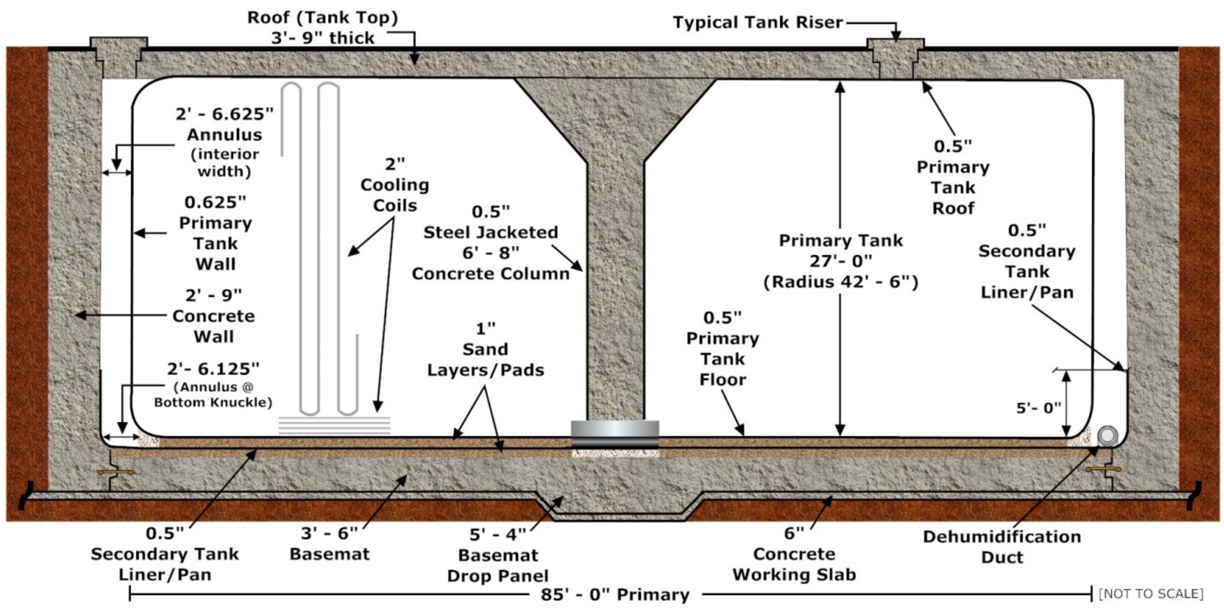
Figure 1.2-3: Typical Type I Tank



1.2.2 Type II Tanks

Four Type II tanks are located in the HTF. The Type II primary liners are 85 feet in diameter and 27 feet high, with a nominal operating capacity of 1,030,000 gallons. A typical Type II tank is shown in Figure 1.2-4. Additional details of the Type II tanks are provided in Section 3 of the HTF PA. [SRR-CWDA-2010-00128]

Figure 1.2-4: Typical Type II Tank



1.2.3 Type III/IIIA Tanks

Four Type III tanks (Tanks 29 through 32) and 13 Type IIIA tanks (Tanks 35 through 43, and 48 through 51) are located in the HTF. In FTF, there are two Type III tanks (Tanks 33 and 34) and eight Type IIIA tanks (Tanks 25 through 28 and 44 through 47). The primary liners are 85 feet in diameter and 33 feet high with a nominal operating capacity of 1,300,000 gallons. Typical HTF Type III and Type IIIA tanks are shown in Figures 1.2-5 and 1.2-6, respectively. Note that Tanks 35, 36, and 37 have been designated as Type IIIA tanks but they differ from Figure 1.2-6 in that these waste tanks have a flat roof with a uniform 4-foot concrete thickness, similar to the Type III tanks. Additionally, Tank 35 has deployable cooling coils rather than the network of cooling coils typically installed in Type IIIA tanks. Additional details of the Type III and IIIA tanks are provided in Section 3 of the FTF and HTF PAs. [SRS-REG-2007-00002, SRR-CWDA-2010-00128]

Figure 1.2-5: Typical Type III Tank

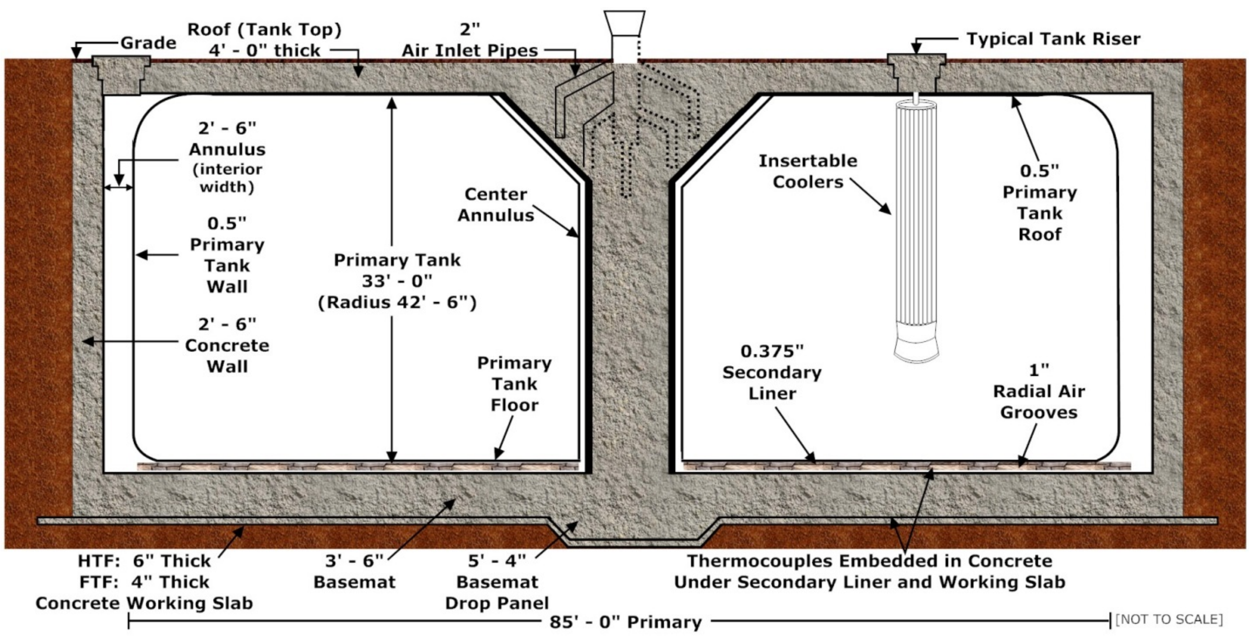
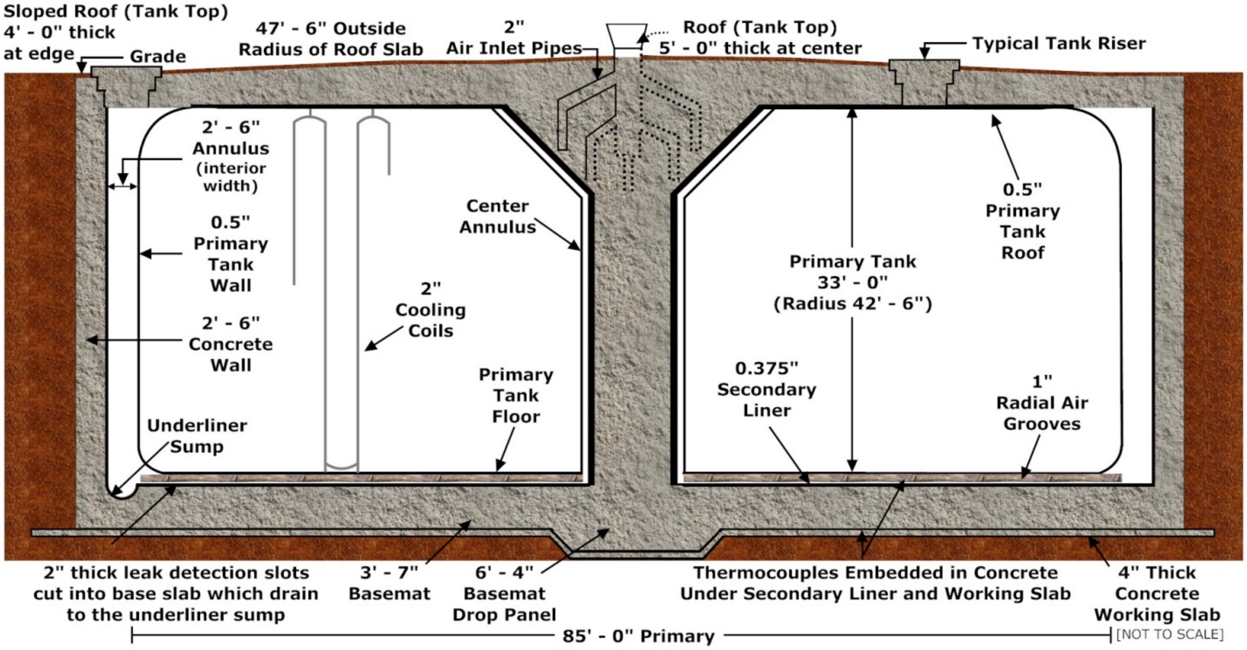


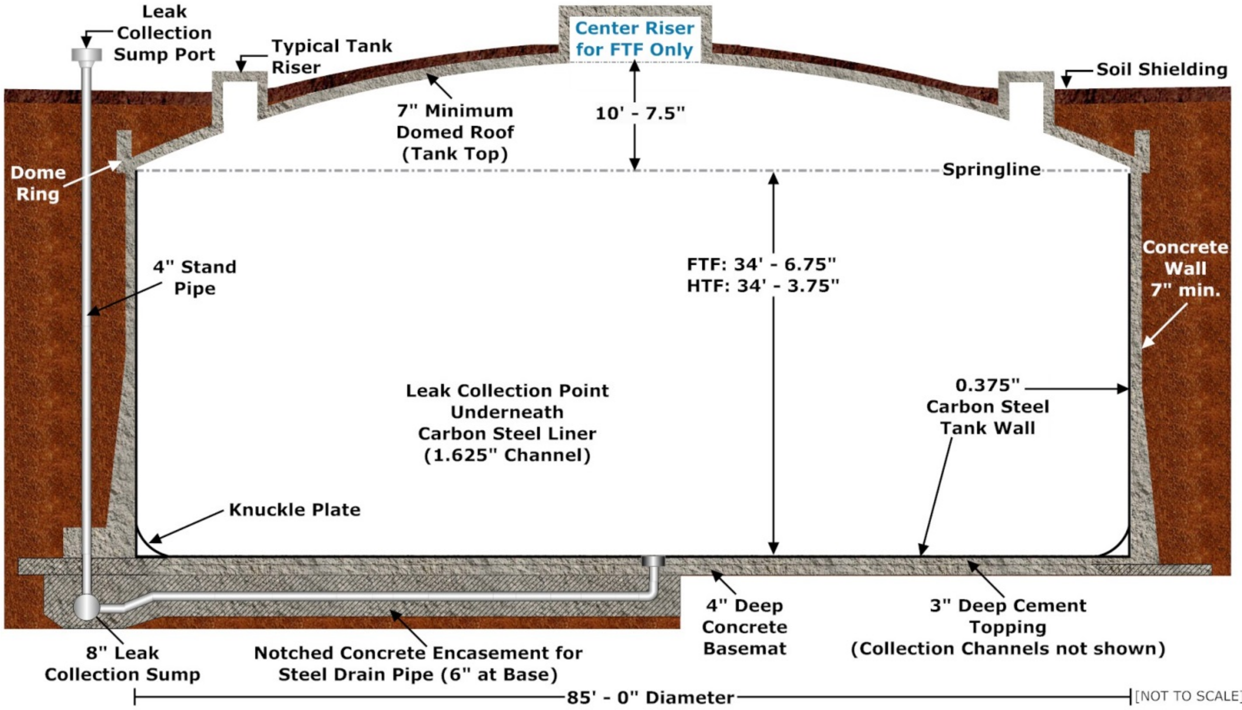
Figure 1.2-6: Typical Type IIIA Tank



1.2.4 Type IV Tanks

Four Type IV tanks are located in HTF (Tanks 21 through 24) and four are located in FTF (Tanks 17 through 20; which have been operationally closed). Type IV tanks are 85 feet in diameter and approximately 34 feet high at the side wall, with a nominal operating capacity of 1,300,000 gallons. Type IV tanks have a single liner with a self-supporting, reinforced concrete, hemispherical, domed roof. Type IV tanks in the FTF differ slightly from the HTF Type IV tanks. The FTF Type IV tanks have a central riser on the roof and the springline is 34 feet 6.75 inches above the floor. The HTF Type IV tanks do not have a central roof riser and the springline is 34 feet 3.75 inches above the floor. A typical Type IV tank configuration is shown in Figure 1.2-7. Additional details of the Type IV tanks are provided in Section 3 of the FTF and HTF PAs. [SRS-REG-2007-00002, SRR-CWDA-2010-00128]

Figure 1.2-7: Typical Type IV Tank



1.3 Analytical Laboratory Selection

The current laboratory used for FTF/HTF analytical activities associated with Liquid Waste Organization samples is the DOE's SRNL. SRNL's over 50 years of experience in developing the necessary analytical methods and analyzing waste tank samples for the anticipated target analyte suite brings continuity to the project. SRNL also has the special handling facilities necessary to receive and process these high-activity material samples.

Programmatic QA document(s), including the LWTRS-QAPP, have been developed to manage the specific requirements associated with residuals material sample collection and analysis. Any laboratory chosen to conduct analyses will be required to have the necessary facilities and equipment to handle the highly radioactive samples, the appropriate level of qualified personnel, analytical experience and methods, internal Standard Operating Procedures (SOPs), and an approved Quality Assurance Plan (QAP) meeting the same quality requirements.

1.4 Data Analysis

The FTF/HTF waste tank characterizations will provide the data that satisfy the DQOs for waste tank removal from service decisions. Data analysis is addressed in the LWTRS-QAPP. [SRR-CWDA-2011-00117]

2.0 SAVANNAH RIVER REMEDIATION QUALITY ASSURANCE AND PROJECT TEAM ROLES & RESPONSIBILITIES

A number of different contractors working for the DOE operate the SRS. Savannah River Remediation LLC (SRR) is the Liquid Waste Management contractor at SRS and has responsibility for managing the SRS liquid waste facilities and systems. Savannah River Nuclear Solutions, LLC (SRNS) is the Management and Operations (M&O) contractor for SRS. SRNL is one of the operating entities under SRNS. A common QA program is currently applied across both contractors on a site-wide basis. That common program is documented in the 1Q Manual based on Nuclear Quality Assurance (NQA-1) standards.

The SRR QA approach is consistent with the program requirements in the 1Q Manual and is described in Sections 2.1 and 2.2.

2.1 Quality Assurance Requirements Integration Across SRS

SRS operations are controlled by a common QA program documented in the 1Q Manual based on national consensus standard American Society of Mechanical Engineers (ASME) NQA-1 and DOE requirements. The 1Q Manual is the implementation document at SRS.

Figure 2.1-1 presents some of the upper tier drivers for SRS QA. As shown in Figure 2.1-2, from these high-level documents, site-specific QA program documents are developed for the residuals sampling and analysis program that address increasingly more detailed aspects as the process proceeds to field and laboratory implementation.

Figure 2.1-1: SRS Quality Assurance Requirements and Documents Hierarchical Flow-Down

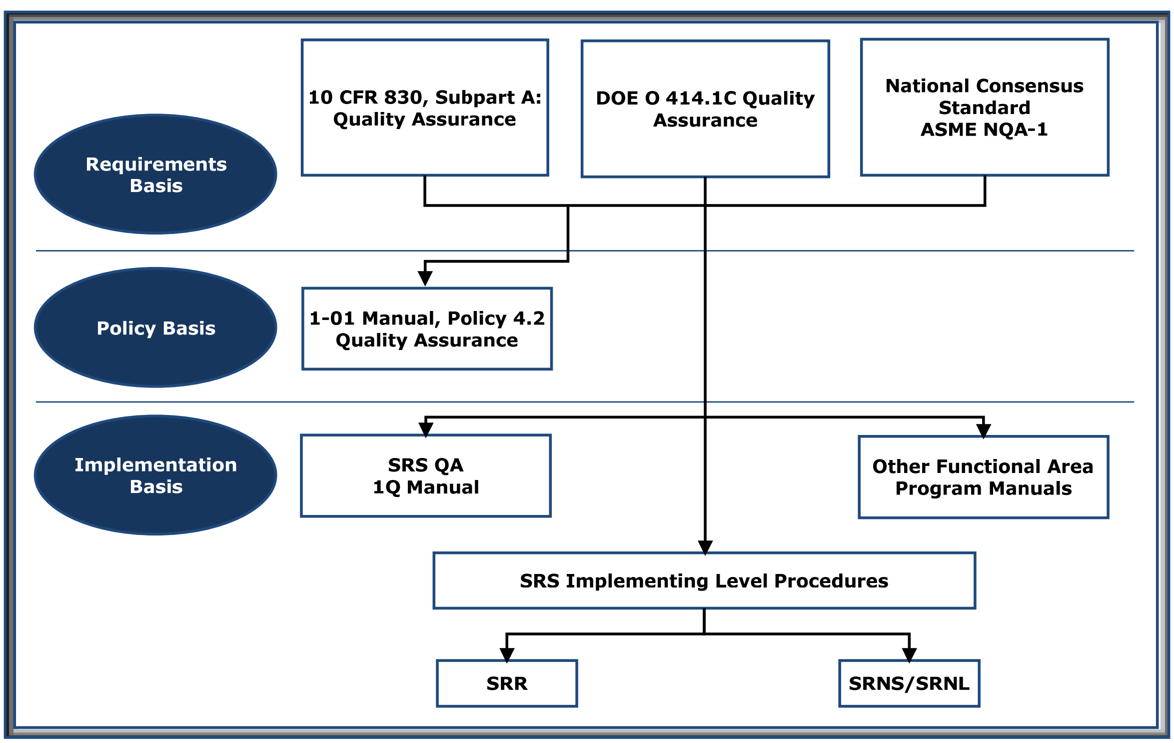
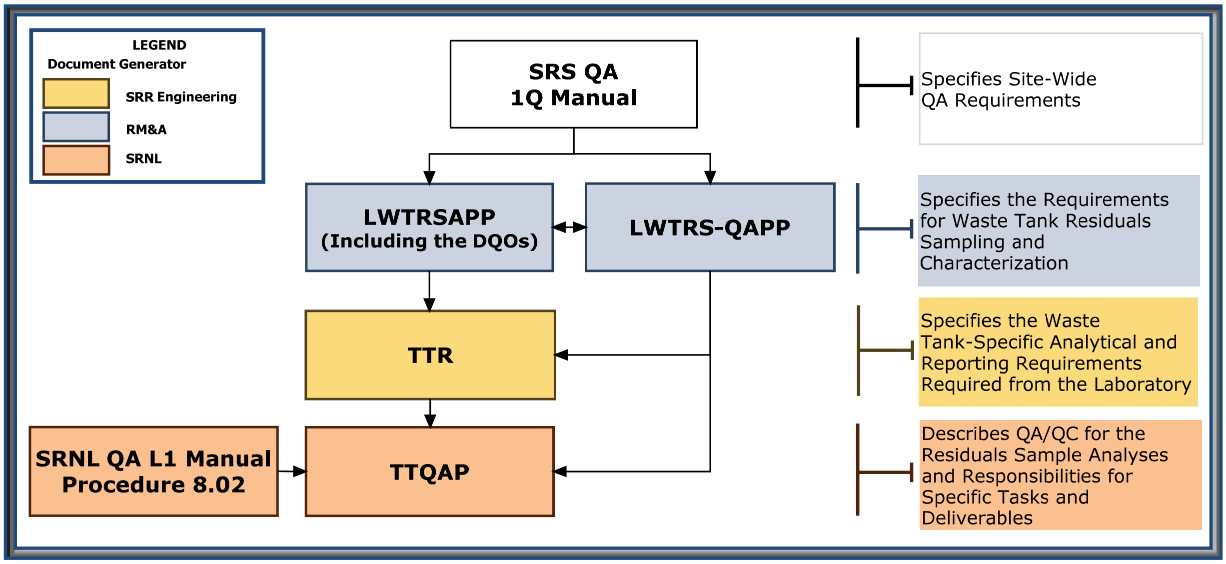


Figure 2.1-2: Relationship of the LWTRSAPP to Program Documents Associated with Waste Tank Residuals Sampling and Analysis



Note: TTQAP Task Technical and Quality Assurance Plan
TTR Technical Task Request

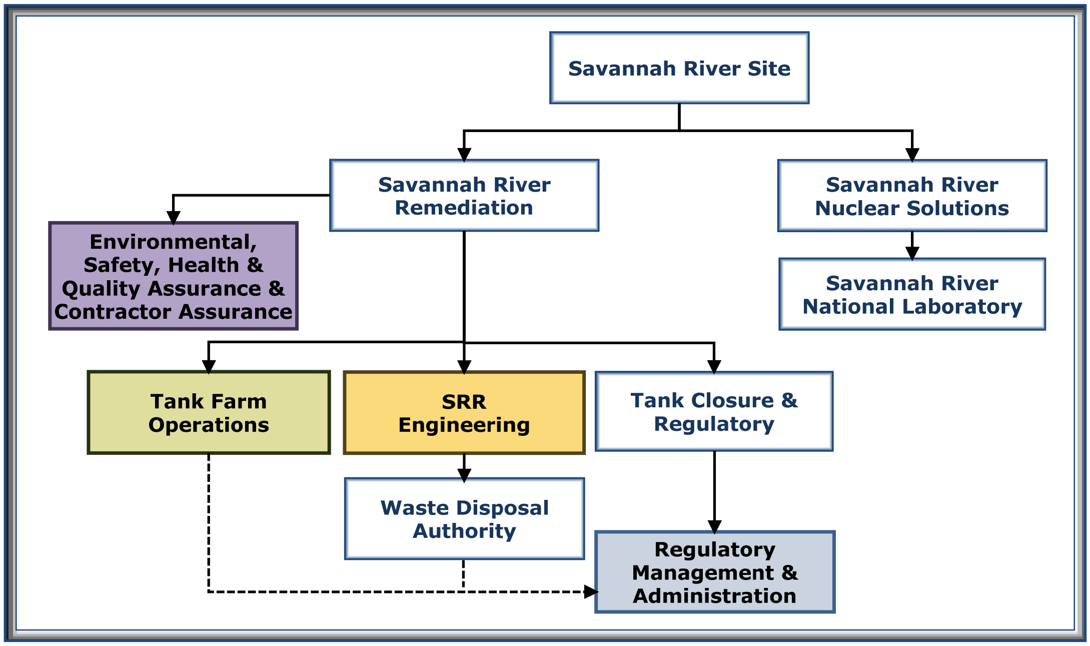
Special challenges are associated with the collection and analysis of the waste tank residuals samples, such as the high sample radioactivity, the remote handling requirements for worker protection, and the analytical complexities. The LWTRS-QAPP covers the unique aspects of the waste tank residuals sampling and analysis, and has been developed using the SCDHEC QAPP Guide Rev. 1.1, *Guidance Document for Preparing Quality Assurance Project Plans (QAPPs) for Environmental Monitoring Projects/Studies* (DHEC_QAPP_Guide_09-2008), to explain the SRS QA Program as defined in the 1Q Manual. [SRR-CWDA-2011-00117] The SCDHEC QAPP Guide is based on the EPA requirements as found in the EPA QA/G-5, *Guidance for Quality Assurance Project Plans*, and EPA QA/R-5, *EPA Requirements for Quality Assurance Project Plans*. Even though the samples are not environmental compliance samples, these reference documents were used to establish a recognized process for characterizing the residuals material remaining in the waste tanks at the time of removal of service.

The LWTRS-QAPP establishes the specific QA/QC requirements necessary for program application across the SRS organization and to produce defensible data to meet the LWTRSAPP DQOs. Figure 2.1-2 illustrates the relationship of the higher-level program documents associated with the LWTRSAPP and LWTRS-QAPP. Additional detail on the documents is provided in Section 2.3.

2.2 Liquid Waste Tank Residuals Sampling Project Organization

SRR is a project organization with subject matter experts organized by functional areas to provide matrix support. Figure 2.2-1 shows the SRR and SRNS/SRNL groups that support the residuals sampling and analysis under the umbrella requirements of the LWTRSAPP and LWTRS-QAPP.

Figure 2.2-1: Organizational Structure for LWTRSAPP and LWTRS-QAPP Implementation



2.2.1 Organization Areas of Responsibility

Project direction and management of waste tank removals from service are the responsibilities of the Waste Removal and Tank Closure Manager. These responsibilities would include implementation of this LWTRSAPP, LWTRS-QAPP, and related quality management expectations for waste tank removals from service. Waste Removal and Tank Closure is supported by Regulatory Management & Administration (RM&A); SRR Engineering; Tank Farm Operations (TFO); Environment, Safety, Health & Quality Assurance & Contractor Assurance (ESH&QA&CA); and SRNL.

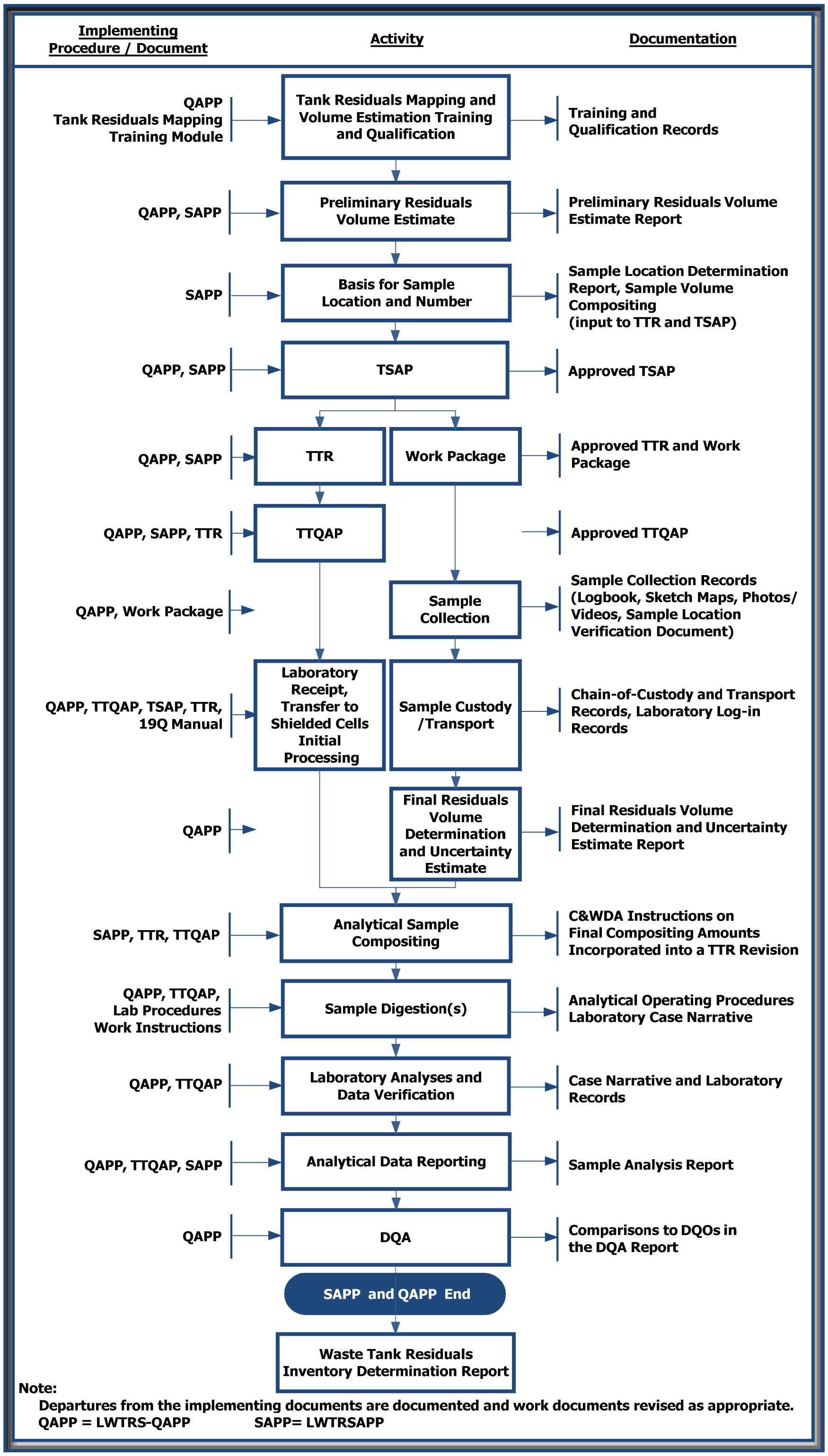
RM&A has the responsibility for maintaining, updating, and distributing the LWTRSAPP and LWTRS-QAPP, as directed, for SRR. The implementing organization involved will coordinate and be responsible for activities under their area of operation. The organizations designate individuals to oversee sampling and analysis activities or interface with the other organizations as part of the Sampling and Analysis Project Team as discussed in Section 2.4.

Additional details on specific roles and responsibilities are presented in Section A4.2 of the LWTRS-QAPP. [SRR-CWDA-2011-00117]

2.3 SRR Implementation for Waste Tank Residuals Sampling and Analysis Program

Figure 2.3-1 shows the activities and tasks related to the waste tank residuals sampling and analysis, the implementing procedures, and documentation generated.

Figure 2.3-1: Waste Tank Residuals Sampling and Analysis Implementation Procedures, Activities, and Documentation



2.4 Sampling and Analysis Project Team Roles and Responsibilities

SRR uses an informal Sampling and Analysis Project Team composed of personnel from various SRS organizations to interact with other organizations to implement the waste tank sampling and analysis and oversee activities in their area of responsibility. To that end, implementing documents are generated, reviewed and approved by the parties shown in Figure 2.4-1.

The numbered documents shown on Figure 2.4-1 and their implementation are summarized below.

1. The LWTRSAPP provides the basis, procedures, and general methodology for sampling waste tank residuals.
2. The LWTRS-QAPP provides the program-specific QA/QC requirements for characterization task performance.
3. A preliminary residuals volume estimate is determined by trained personnel from SRR Engineering and RM&A using the tank mapping methodology. [SRR-LWE-2010-00240] SRR Engineering produces a Preliminary Residuals Volume Estimate Report showing the distribution and volume(s) for distinguishable areas of the residual material on the waste tank floor.
4. RM&A personnel use the preliminary mapping and volume(s) to determine initial sample locations and to calculate preliminary compositing instructions as described in Section 4. The preliminary sample location maps and compositing instructions are discussed and reviewed by SRR Engineering and TFO. If necessary, the locations are adjusted to account for accessibility, sample collection method limitations, radiological conditions, (e.g., As Low As Reasonably Achievable [ALARA]), and any other Project Team concerns. RM&A writes a Sample Location Determination Report (SLDR) documenting the basis for the locations selected.
5. The SLDR is used by SRR Engineering staff as the basis for generating the TSAP. The TSAP is reviewed for completeness and accuracy by the organizations involved in the sampling. The TSAP is revised during the sampling process, as needed, to document "as found" conditions, sampling method modifications, or sample location changes.
6. The TSAP is also used by SRR Engineering staff as the basis for generating the Technical Task Request (TTR). SRR Engineering uses the TTR to provide SRNL direction for sample preparation, applicable analytes, desired analytical sensitivities, and reporting. The TTR will reference the LWTRS-QAPP as appropriate and will be revised to document changes occurring during the sampling. The TTR will be revised at the end of sampling activities to provide the final compositing instructions to the laboratory.
7. The TSAP is used to generate the waste tank-specific work package for the TFO Sample Extraction Team (SET) to perform the sampling. Prior to sampling, the work package is reviewed and approved by the TFO SET and other involved organizations such as Radiological Control. Any special tools or equipment needed are developed, fabricated and tested.

Figure 2.4-1: Roles and Responsibilities for Waste Tank Characterization Implementing Procedure/Activity/Document

Note: Any changes/deviations must include same level of review and approval as original document or activity.

Description in Section 2.4	Implementing Procedure/ Activity/Document	SRR					SRNS	SRNL [2]	DOE	SCDHEC	EPA	Documentation
		SRR Engineering	Tank Farm Operations [1]	RM&A	Waste Determination Director	Quality Assurance	Quality Assurance					
1	LWTRSAPP	R A I	I	G R A I	R A	R	R	R A I	R A	R A	R	LWTRSAPP
2	LWTRS-QAPP	R I	R I	G R A I	R A	R A I	R I	R A I	R A	R A	R	LWTRS-QAPP
3	Preliminary Residuals Volume Estimate	G A	—	R	—	—	—	—	—	—	—	Preliminary Residuals Volume Estimate Report
4	SLDR	R	—	G A	—	—	—	—	—	—	—	SLDR
5	TSAP	G A	R I	R A	—	—	—	—	—	—	—	TSAP
6	TTR	G A	—	R A	—	—	—	R A I	—	—	—	TTR
7	Sampling Work Package	R A	G A I	—	—	—	—	—	—	—	—	Sampling Work Package
8	TTQAP	R A	—	R A	—	R	R A	G A I	—	—	—	TTQAP
9	Sample Collection and Location Documentation	G A I	R	R	—	—	—	—	—	—	—	Logbook, Sketch Maps, Photos/ Videos, Sample Location Verification Document
10	Sample Transport and Laboratory Receipt	—	G I	—	—	—	—	G I	—	—	—	Chain-of-Custody and Transport Records, Laboratory Log-In Records
11	Final Residuals Volume Determination and Uncertainty Estimate	G A	—	R	—	—	—	—	—	—	—	Final Residuals Volume Determination and Uncertainty Estimate Report
12	Analytical Sample Compositing Instructions	R	—	G R A	—	—	—	R I	—	—	—	Revision to TTR
13	Sample Compositing, Digestion and Analysis Records	—	—	—	—	—	—	G A I	—	—	—	Analytical Operating Procedures
14	Sample Analysis Report	R A	—	R A	—	R	—	G A I	—	—	—	Sample Analysis Report
15	DQA [3,4]	—	—	R A	—	R	—	R	—	—	—	DQA Report
LWTRSAPP and LWTRS-QAPP END												
16	Waste Tank Residuals Inventory Determination [5]	R	—	R	—	—	—	—	—	—	—	Waste Tank Residuals Inventory Determination Report
17	Waste Tank-Specific Closure Module	R	—	G A	R A	—	—	—	R A	R A	R	Waste Tank-Specific Closure Module

1 Includes Radiological Control as Necessary
2 Laboratory Responsible for All Internal Activities (e.g., Laboratory QA)
3 Generated by Third-Party Assessor
4 Reviewed and Approved by WDA
5 Generated by WDA

A = Approval Authority
G = Document Generator
I = Implementing Organization
R = Reviewer
— Not Applicable

8. SRR Engineering sends the TTR, which references the LWTRS-QAPP, and TSAP to SRNL as input for their Task Technical and Quality Assurance Plan (TTQAP) preparation. The TTQAP governs the SRNL QA/QC activities and measures required for the analyses and reporting requested in the TTR. The TTQAP references the LWTRS-QAPP and LWTRSAPP as applicable to assure project consistency.
9. Sample collection locations are documented by the SET team using photographs and/or video footage during collection. Simple location sketch maps are also be prepared by the SET. SRR Engineering generates a Sample Location Verification Document to authenticate the actual locations sampled.
10. Samples are transported to SRNL using the *CST Sample Manual* requirements which incorporate a chain-of-custody (COC) developed specifically for these characterization samples. [SW11.1-SAMPLE Section 7.18] As described in the LWTRS-QAPP, a COC is prepared for each group of samples being transmitted by the SET. Samples are typically received at the Shielded Cells Operations (SCO) facility for initial log-in, material inspection and processing that includes: material transfer into standard containers, photography, density measurements, drying, grinding, and sieving in preparation for compositing.
11. SRR Engineering prepares the Final Residuals Volume Determination and Uncertainty Estimate Report using the tank mapping methodology. [SRR-LWE-2010-00240]
12. RM&A uses the final volume determination and uncertainty estimate to calculate the final sample compositing instructions for the analytical sample creation. The TTR is revised and the compositing instructions are transmitted to SRNL. If necessary, the TTQAP is updated.
13. SRNL prepares, composites, and analyzes the characterization samples using the instructions in the TTR, TTQAP, and specific Analytical Operating Procedures (AOPs).
14. The analytical data are reviewed and verified by the appropriate SRNL staff and after acceptance by SRR Engineering and RM&A, are formally submitted as a Sample Analysis Report.
15. The Sample Analysis Report and the verified data are evaluated in a Data Quality Assessment (DQA) to ensure the DQOs are satisfied. The DQA is performed by an independent evaluator not associated with the data collection or use. The data are also reviewed by RM&A and subsequently used to prepare the Waste Tank Residuals Inventory Determination Report.

Any required changes to the implementing documents will have the same level of review and approval as the original document or activity. Changes affecting sampling will be documented as revisions to the TSAP or TTR, as appropriate, and transmitted to SRNL. When needed, the TTQAP will be revised to incorporate those changes.

A copy of the TSAP, reflecting any changes required by field conditions, will be provided to SCDHEC. Periodic briefings will be held with SCDHEC during the sampling and analysis process to provide updates on the process.

2.5 LWTRSAPP Implementing Document Descriptions

The implementing documents for the LWTRSAPP are summarized on Table 2.5-1.

Table 2.5-1: LWTRSAPP Implementing Document Descriptions

Document	Description
LWTRSAPP	Programmatic document requiring regulatory approval that describes the sampling and analysis process/methodology to guide the SET in data collection and the SRNL sample analysis efforts to support characterization of the waste tank residuals material. References the LWTRS-QAPP for quality direction and analytical procedures.
LWTRS-QAPP	Programmatic document requiring regulatory approval that establishes the waste tank sampling and analysis quality program protocols and requirements necessary to characterize the waste tank residuals material.
Preliminary Residuals Volume Estimate Report	Prepared by SRR Engineering to provide initial information, including diagrams and volume maps on the estimated distribution of residuals material within the specific waste tank.
SLDR	Prepared by RM&A to determine the sample locations using the preliminary residuals material volume and distribution, and applying the sample plan design approach described in the LWTRSAPP. The SLDR is used to prepare the TSAP.
TSAP	Provides technical direction to the sampling team on specific sample numbers and locations to be collected within the waste tank. References the LWTRS-QAPP for QA requirements and the LWTRSAPP for general sampling instructions. The TSAP also includes applicable analyses for characterization.
TTR	Provides technical direction to the analytical laboratory (SRNL) to perform and report sample analyses as defined in the LWTRS-QAPP.
Sampling Work Package	Provides specific directions to the SET to implement the requirements of the TSAP and applicable QA requirements.
TTQAP	Prepared by SRNL to define the task(s) specified in the TTR along with responsibilities, deliverables, and specific instructions. The TTQAP also identifies the applicable sections of the SRS QA Manual (1Q Manual) and associated implementing procedures.
Sample Location Verification Document	Prepared by SRR Engineering to photographically document the actual sample locations within the waste tank.
Final Residuals Volume Determination and Uncertainty Estimate Report	Prepared by SRR Engineering as input to RM&A in preparing compositing instructions for the laboratory and the Waste Tank Residuals Inventory Determination Report for the specific waste tank.
Analytical Sample Compositing Instructions	Prepared by RM&A to provide SRNL the final compositing instructions when composite samples are used for residuals characterization.
AOPs	Prepared by SRNL to provide general analytical instructions. They are used with Research and Development (R&D) directions to provide direction on sample digestion, separations, and analysis.
Sample Analysis Report	Prepared by SRNL to document the results of the sample analyses and associated data verification. Provided to RM&A to perform the waste tank DQA and prepare the Waste Tank Residuals Inventory Determination Report and associated closure documents for the specific waste tank.
DQA Report	Conducted by an independent third-party to assess the usability of the data for developing the Waste Tank Residuals Inventory Determination Report.

Note: See Figure 2.4-1 for specific roles and responsibilities associated with these documents.

3.0 DATA QUALITY OBJECTIVES

Consistent with the DOE Guide 435.1-1 and EPA/240/B-06/001, the seven-step DQO development process was followed to identify the type, quantity, and quality of characterization data needed to support the FTF/HTF waste tank removal from service activities. The DQOs are summarized below.

The scope of the LWTRSAPP DQOs is outlined in the following statements:

- The DQO process will only address the sampling plan design and sample analyses for waste tank residuals material.
- The waste tank system within the footprint of the waste tank is the sampling and analysis action boundary. These DQOs will not address any closure actions associated with contaminated soil or ancillary tank farm structures or equipment.

3.1 State the Problem

Based on the intent and commitments of DOE and conforming with relevant and appropriate regulatory guidelines as stated in the FTF and HTF PAs (SRS-REG-2007-00002, SRR-CWDA-2010-00128), the problem to be addressed can be written as:

Provide waste tank residuals material characterization (concentrations) that enable residuals material inventory determinations to support tank closure decisions.

The question the waste tank residuals characterization will attempt to resolve and the actions that may result are defined in the principal study questions (PSQs). The PSQ that addresses the LWTRSAPP problem statement is:

- Do the sample analyses provide the necessary concentration data to support the residuals material inventory determination?

The waste tank residuals material sampling and analyses generate the concentration data used to support the inventory determination. The characterization data is used in analyses to demonstrate conformance with both qualitative and quantitative Federal and State performance objectives and allow DOE to make closure decisions related to waste tank removal from service. For FTF, the protocol DOE uses to remove waste tanks from service is given in the FTF Industrial Waste Water General Closure Plan (GCP), which has been approved by SCDHEC. [LWO-RIP-2009-00009] A similar HTF GCP provides the protocol for HTF waste tank and ancillary structure removals from service. [SRR-CWDA-2011-00022] The characterization data for the radiological and hazardous constituents together with the final residuals volume determination and uncertainty estimate are used to develop the waste tank-specific residuals inventory determination report.

3.2 Identify the Decision

Waste tank residuals sampling data and the subsequent residuals characterizations that the data support are used as input to complex engineering computational models such as the Special Analysis (SA) to assess the long-term fate and transport of the chemical and radiological constituents in the environment. The SA enables prediction of future sustained conformity with these performance objectives. Waste tank residuals sampling is not performed for comparing

residual concentrations against prescribed environmental limits and, as such, the samples to be taken and analyzed under this plan are not environmental compliance samples and are not used for environmental monitoring.

The characterization data is used to determine the tank-specific residuals inventory, which is then compared in the SA to the residuals inventory used in the PA and to support making decisions as to whether other operational closure criteria are met such as:

- Greater than Class C Waste Determination
- Removal of Highly Radioactive Radionuclides (HRRs) to the Maximum Extent Practical
- Decontamination of the waste tanks to the extent technically practicable from an engineering perspective

These are not pass-fail comparisons. If the necessary concentration data are not obtained, the operational closure decisions may be impacted and alternative actions to finalize the residuals inventory may be necessary. If necessary, any required action will be decided on a case-by-case basis in consultation with the appropriate regulatory authorities.

This LWTRSAPP applies to the waste tank residuals sampling and analysis from the start of sampling through the DQA. This LWTRSAPP is not applicable to Tanks 5, 6, 18, or 19 which have undergone removal from service activities.

3.3 Identify Inputs to the Decision

Four types of data are required to address the problem statement and the decision statement for the residuals material characterization to enable waste tank closure decisions:

- Radionuclide concentrations
- Chemical constituent concentrations
- Density of the samples
- Residuals volume data

Residuals volume data, along with sample densities, and the concentrations will be used to determine the constituent inventory in the waste tank. The constituent concentrations and inventories are required to address the program end-state requirements.

3.4 Define the Study Boundaries

The spatial boundary defined in this LWTRSAPP is the waste tank system within the footprint of the waste tank. This would include the waste tank annular space for applicable waste tanks. Samples of the waste tank residuals will be collected after SCDHEC, EPA, and DOE reach mutual agreement that waste removal activities can preliminarily cease and DOE can proceed to the sampling and analysis phase. Other than radioactive decay and additional drying resulting from waste tank ventilation operation, minimal changes are expected to occur in the residuals material after the temporary cessation of waste removal and before sample analysis.

3.5 Develop an Analytical Approach and a Decision Rule

For the LWTRSAPP PSQ, if the necessary concentration data are not obtained, the operational closure decisions may be impacted and alternative actions to finalize the residuals inventory may

be necessary. If necessary, any required action will be decided on a case-by-case basis in consultation with the appropriate regulatory authorities.

3.6 Specify Limits on Decision Error

The probability of determining an incorrect waste tank-specific inventory is related to inaccuracies in the volume determination and in the representative sampling and analysis of the residual materials.

The residuals mapping and volume estimation process is intended to not only determine the residuals volume and uncertainty in the volume estimate; it also looks for areas of possible heterogeneity. The basic sampling plan design requires a sufficient number of samples of adequate size from the segments present in the residual material for the sample compositing. Incomplete sampling and/or insufficient material recovery could impact the data usability for the waste tank inventory determination. Those impacts would need evaluation before the tank-specific sampling finishes so that steps could be taken to ensure that the sampling representativeness and completeness goals are met.

Uncertainty in the residuals final volume determination is incorporated into the sample compositing as described in Appendix A. However, the usability of a large uncertainty in the volume determination would need to be evaluated for its impact on the inventory determination.

The error associated with a sample is highly dependent upon two components: (1) the size of the largest particles; and, (2) the mass of the sample, which is called the sample support. Sampling errors are increased substantially as the particle size increases and as the sample support decreases. These parameters contribute heavily to the fundamental error (FE), an incompressible minimum error that originates from the sample or subsample support, composition, shape, particle size distribution, and chemical properties of the material. Pitard recommends that the FE be kept below 15%. [ISBN 0849389178] The FE and sampling errors in a volume-proportional composite sample will be minimized by (1) creating a composite sample of sufficient support (70 grams), and (2) by reducing particle sizes to the extent practicable. The combined FE for the composite samples for analysis is estimated at approximately 5%. The minimization of sampling errors and FE is described in Appendix A.

Analytical errors that could affect the inventory determination are related to interferences for some determinations and the overwhelming of minor constituents by large amounts of other constituents. The decision error resulting from the incomplete or incorrect measurement of a constituent concentration can be minimized to the extent practicable by careful and sophisticated separation and analysis techniques. Analytical uncertainty is discussed further in Section A7.2 of the LWTRS-QAPP. [SRR-CWDA-2011-00117] Precision and accuracy criteria for analytical data acceptance are presented in Table A7-1 of the LWTRS-QAPP. Recoveries for some of the minor radionuclides may introduce a large uncertainty in the measured concentration that will need evaluation in the DQA. The DQA will also review the Data Quality Indicators (DQIs) and evaluate any nonconformities for impacts on the data.

The decision error (i.e., errors in the inventory) resulting from the uncertainties described above is difficult to quantify. Since the inventory determination is an estimation problem and the data generated is limited by what material can be collected and by analytical success, data

uncertainties do not necessary result in data rejection. When data uncertainties are recognized, or cannot be quantified, the impact is evaluated and typically biased towards conservatism.

3.7 Optimize the Design for Obtaining Data

The basis for the residuals characterization sampling approach is presented in Appendix A. In general, stratified random sampling with volume-proportional compositing is expected to be the primary approach used to characterize the residuals material. The material distribution and volume will be used to design the three sampling arrays, and to calculate a volume-proportional mass needed from each of the five sample locations per array for compositing as described in Section 4. The approach was statistically evaluated and determined that the built-in variations in the volumetric proportions produced valid statistical sampling results for the concentrations of residuals material. [SRNL-STI-2011-00323]

The waste tank-specific sampling plan design and sampling locations will be developed based on the preliminary residuals material volume and distribution. The sampling design will be documented in the SLDR and TSAP.

3.7.1 Practical Constraints

Sampling in the FTF/HTF waste tanks is largely constrained by the access to the residuals material (riser locations, in-tank obstructions), the sampling devices available, the physical condition of the residuals material (dry versus wet, accumulations versus thin layer), and worker risk issues such as exposure to high radiation fields or physical hazards associated with sampling in the waste tank farms. Other considerations include difficulties analyzing the complex sample matrices to the desired sensitivities, and possible modifications to the characterization process due to low residual volume conditions.

3.8 Performance and Acceptance Criteria

The analytical results will be evaluated during the DQA process against the LWTRSAPP DQOs and Measurement Performance Criteria (MPC) for the analytical data. The MPC for analytical data are presented in the LWTRS-QAPP, Section A7. The evaluation may also include waste tank usage history, any historic or in-process waste samples, or a simple statistical analysis of the constituent results. The acceptance criteria provide reasonable assurance that the reported concentrations are representative of the residuals material.

The DQA will assess any impacts to the data introduced by sampling or analytical constraints, and those impacts will be evaluated in the waste tank residuals inventory determination.

3.9 Plan to Obtain Data

This LWTRSAPP presents the general approach that will be used to characterize residuals materials in the FTF/HTF waste tanks. Because the final material distribution and access conditions will differ for each waste tank, TSAPs will be developed to address the specific waste tank sampling conditions. The general process for waste tank characterization is described in Section 1 and shown on Figure 1.1-1.

3.9.1 Representative Sampling

Representative sampling is by definition the collection of a sample that is representative of the entire population in question. Implied in the definition is that the population varies and

that capturing the variations must be taken into account when designing the sampling plan. For residuals sampling, variations could be areas (segments) of different concentration, color, thickness, or other distinguishable attributes.

To achieve representation of the possible variations in the residuals material, the residual material volume and distribution will be assessed. The TSAP will be designed to sample areas that could represent the various segments in the waste tank. Sample locations would be shifted towards areas with more material, and more material from those areas will be used in the final composite samples. The final compositing will also be adjusted for uncertainty in the volume estimate.

Situations could arise where waste removal is so successful that volume estimation and sample collection will be difficult. Section 4 discusses possible alternative sampling approaches and impacts for low-volume situations.

3.9.2 Sampling Approach

The residuals characterization uses a stratified random sampling approach for sample collection. Waste removal activities mix and move the material and typically produce segments (areas) of relatively uniform thickness and accumulations of variable thickness (mounds). The sampling plan design delineates these areas into segments and determines the number of samples collected in each segment based on the relative volume of that segment. The material within each segment is then sampled to collect material for the final composite sample used for analysis. The samples within each segment are considered random samples due to the randomization of the material during mixing and by spreading the sample locations while considering accessibility restrictions. The amount of material used for compositing from each sample location is calculated based on the volumetric proportion of the segment and is varied to incorporate the segment's uncertainty estimated during the volume determination. Based on this approach, all the sample analyses and statistics are considered applicable to the waste tank residual material. Details are presented in Appendix A.

The segments and boundaries are determined by evaluating the preliminary residuals material distribution map for the waste tank. The boundary determinations are aided by tracking material movement or stability during waste removal efforts, color contrasts, or surface texture variations observed during the mapping or video surveys.

As detailed in Appendix A, considering each sample weight for compositing (between 10 and 30 grams) and the maximum particle size expected in the residuals material (0.05 to 0.1 cm), between 5 and 15 sample locations per composite will be sufficient to control FE in the sampling. Five sample locations were determined to be an optimum starting point for each array based on the amount of mixing that occurs during the bulk waste removal process. Three sampling arrays will be necessary to produce three composite samples, the minimum number necessary to manage sampling uncertainty.

The material distribution and volume is used to design three sampling arrays, and to calculate a volume-proportional mass needed from each sample location for compositing. The sampling design is documented in the SLDR and used to develop the TSAP.

The background for using this sampling approach is presented in Appendix A. The stratified random sampling approach is described in Section 4.

3.10 Managing Uncertainty

Uncertainty arises in several areas during the waste tank residuals characterization and must be effectively managed to produce representative results. Uncertainty is present in sampling, sample preparation, measurement and analysis, and volume determination steps. An approach to managing uncertainties is incorporated into the LWTRSAPP for residuals sampling and analysis as discussed below to ensure that quality results are obtained. Additional discussion on addressing uncertainty in the sampling and material processing is presented in Appendix A.

3.10.1 Sampling Uncertainty

Sampling is the major source of uncertainty. As recent research has shown, one of the largest uncertainties in site characterization data sets is due to sampling variability as a direct consequence of heterogeneity of the sample matrices. [DOI: 10.1021/es012490g] Sampling uncertainty arises because of heterogeneity in the physical (distribution), chemical, and radiological composition of the material. Sampling in SRS waste tanks is even more challenging given limited access, internal waste tank interferences, worker exposure concerns, and availability of suitable sampling technology.

To manage the sampling uncertainty, the sampling plan design must address the possible segments, sample size and sample support. As described further in Appendix A, the sampling plan design places sample locations in the segments present in the material. Any horizontal and vertical heterogeneity is represented by collecting material from different areas and depths if the material thickness is sufficient to do so. The segments will be represented in the final sample using volume-proportional compositing.

3.10.2 Sample Preparation Uncertainty

The radiological nature of the residuals material necessitates sample handling and preparation in shielded cells with manipulators. SOPs have been developed to prevent cross contamination and sample preparation in accordance with those needs. For example, possible cross-contamination is minimized by decontaminating the shielded cell and covering the cell floor before use, and changing manipulator fingers before samples from different waste tanks are processed inside that cell.

"Sample support" is the term used to describe the physical sample attributes that help determine the analytical result and is a critical variable for representative data. [NEMC 2004-20] As described in Appendix A, for the expected particle size range in the residuals material after grinding and homogenizing and the subsample weights used to create the composite samples, the combined FE is much less than Pitard's recommended 15% maximum. [ISBN: 0849389178]

Studies on residual material have demonstrated good sample homogeneity for the entire analytical process of sub-sampling the dried, ground and sieved sample material, dissolution in the shielded cells, and elemental analyses. [SRNL-STI-2010-00386]

3.10.3 Analytical Measurement Uncertainty

Unique extraction and separation techniques are often required to cope with interferences affecting these sample analyses. Negative effects on data quality are minimized by adherence to laboratory AOPs for analytical instrument operation, calibration, and frequency of calibration. Standards and blanks are used as process and equipment check samples.

Analyzed reference glass (ARG) standards are weighed and digested in the cells in a manner similar to the process used for residuals samples. ARG aliquots serve as laboratory control standards for some of the elemental analyses and as blanks for radionuclide analyses.

The radiological nature of the residual materials prohibits some of the routine sample preservation techniques, such as temperature control or adding preservative. As explained in Section B3 of the LWTRS-QAPP, sample preservation and holding times are not applicable for these samples. [SRS-CWDA-2011-00117]

3.10.4 Volume Uncertainty

Volume uncertainty will be managed by standardizing volume determination methodologies. Human interpretation of collected photographic and video data is the major source for variability. A residuals mapper training program has been developed to standardize interpretation of photographic and video data.

A methodology has been developed to estimate the uncertainty associated with volume determination. [SRR-LWE-2010-00240] The uncertainty for the residuals volume determination is performed by reviewing selected areas across the waste tank floor or the entire waste tank floor by trained volume estimators. Video inspections and photographs for these areas are given to the mapping team. The mapping team then determines a low-end and a high-end volume estimate for each area. If landmarks are not visible to aid in the thickness estimation, then there will typically be a larger difference between the low-end and high-end estimates because of greater uncertainty in determining the solids heights. The volume uncertainty incorporation into the composite sample creation is described in Appendix A.

4.0 SAMPLING

Waste tank residuals will be collected and analyzed for a specific list of analytes to satisfy South Carolina and DOE requirements for removing FTF/HTF waste tanks from service. Sample collection and analysis will be designed to satisfy the LWTRSAPP DQOs by implementing the stratified random and volume-proportional compositing approach presented in Appendix A. The sampling approach, sample collection equipment and field procedures are described below. The basis for this sampling approach is presented in Appendix A. Additional details on waste tank residuals sampling is presented in Section B of the LWTRS-QAPP. [SRR-CWDA-2011-00117]

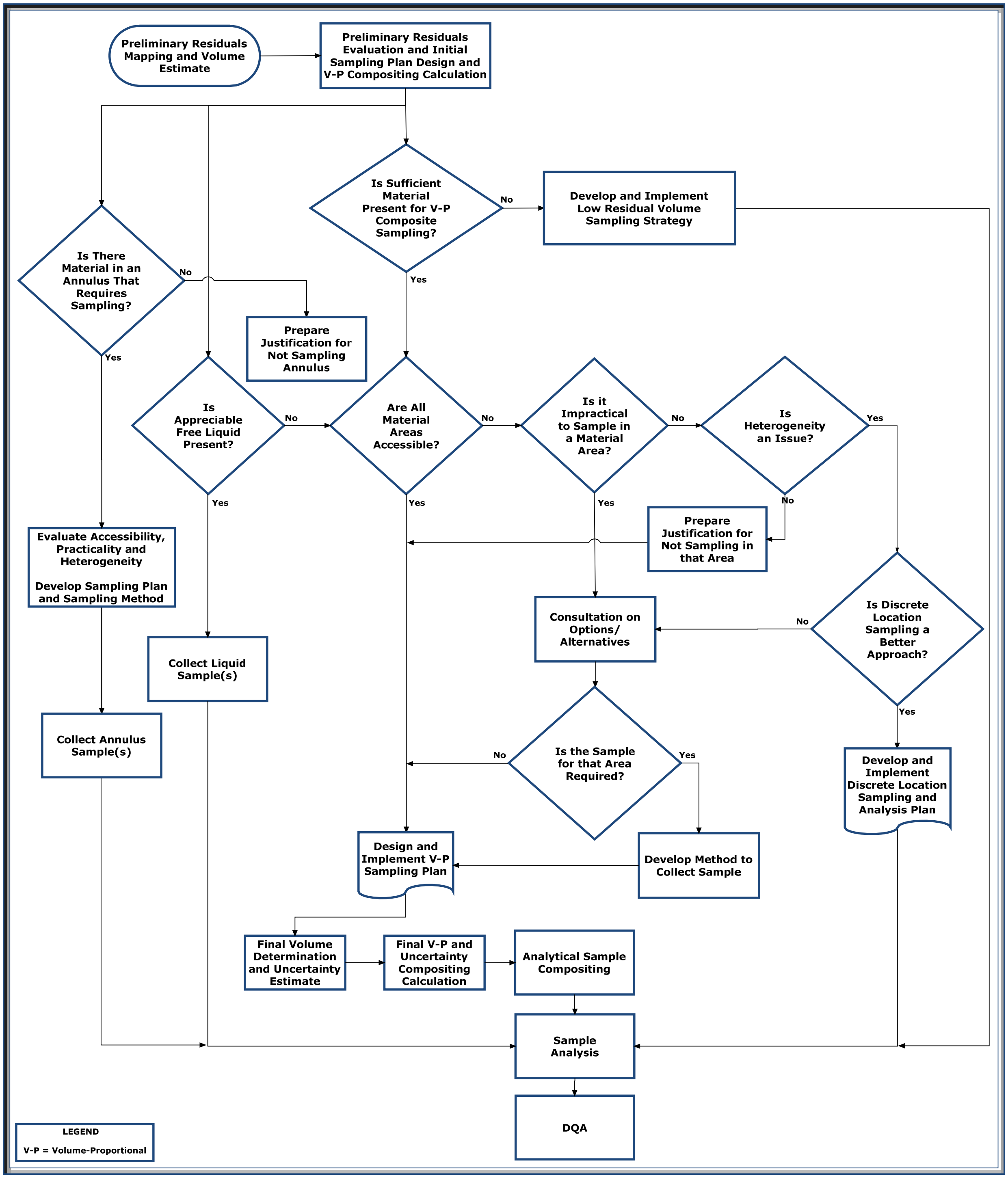
4.1 Sample Collection

Sample collection in the waste tanks is complicated by limited access points to the interior, internal obstructions, and high radiological conditions. Material accessibility, ALARA issues, and sampling equipment capabilities will be evaluated during the sampling plan design phase and used to develop the TSAPs.

4.1.1 Sampling and Analysis Planning Process

The sampling and analysis planning process is shown in Figure 4.1-1. Beginning with the preliminary residuals material mapping and volume estimate, Project Closure personnel, primarily SRR Engineering and RM&A, will evaluate the distribution, possible segments present, presence of solids accumulations, and accessibility issues. This information will be used to develop initial TSAPs. The evaluations will be performed and reviewed by SRR Engineering and RM&A personnel to ensure that the residuals material in a waste tank will be representatively sampled to satisfy the LWTRSAPP DQOs. The basis for the final sampling design will be described in the SLDR. Sample locations may change to reflect updated information on material volume and distribution developed during the sampling or as sample collection problems dictate. SRR Engineering will document the final sample locations in a Sample Location Verification Document.

Figure 4.1-1: Sampling and Analysis Planning Process



As shown on Figure 4.1-1, the waste tank-specific residuals amount, distribution, and accessibility will determine the final sampling approach and design. In some instances, discrete location sampling and analysis may be a better approach than compositing for residuals characterization. If present in appreciable quantities after all cleaning and drying efforts end, free liquids or material in a waste tank annulus may require sampling in order to characterize the waste tank. If it is impractical to sample an area due to accessibility or other issues, a consultation will be conducted to evaluate other options or alternatives for characterizing or estimating those material concentrations. The special sampling situation where only minimal material remains is discussed in Section 4.1.2.5.

4.1.2 Sampling Plan Design

The stratified random sampling approach description and appropriateness to characterize the residuals material is presented in Appendix A.

As described in Section 3.9.2, a basic sampling plan using three sample arrays (patterns), each with a minimum of five sample locations, will be developed for each waste tank to be sampled. The five samples in an array will be used to construct one composite sample for analysis, resulting in three composite samples per waste tank. In general, each array would be designed to collect material from a different segment or depth in the residuals material, and the three composite samples collectively would represent the entire residual waste tank mass. If dictated by the material distribution, more arrays could be designed or more samples per array could be added to adequately represent the residuals material variability. The need for a different sampling plan design to capture the material variability would be made by RM&A and SRR Engineering after evaluating the material distribution.

The initial array designs and sample locations will be determined by RM&A and SRR Engineering, with TFO input on sample location accessibility, using the preliminary residuals mapping and volume estimate. The final sampling plan will account for accessibility or other issues to best satisfy the LWTRSAPP DQOs before incorporation into the TSAP.

4.1.2.1 Determination of Samples per Area or Feature

The preliminary residuals volume estimate would first be used to determine the relative volume (percentage) of material contained in a segment (waste tank floor area or feature such as a mound). Next, the number of samples in the array to be collected from that segment or feature would be calculated by:

$$\frac{V_f}{V_T} \times 5 = n_{SL}$$

Where:

V_f	=	Feature volume
V_T	=	Total residuals material volume
n_{SL}	=	Number of sample locations per feature

If more than one sample is indicated in a segment or feature, the volume proportion used for compositing would be adjusted by dividing the volume of that area or feature by the number

of samples collected. If multiple samples are required from a mound, special sampling techniques or tools may be used to obtain material from different depths.

4.1.2.2 Determination of Volume-Proportioning Amounts

Each composite sample for analysis will be constructed using a volume-proportioning methodology. The amount used from each sample location will reflect the proportion of the total waste tank residuals material present at that location as its portion of the final desired 70 gram sample weight. For example, if one sample were collected in a segment that represented 25% of the total residuals material, 25% of a hypothetical total mass of 70 grams (17.5 grams) would be taken from that location's sample. Additional details of the proportioning and adjustment for volume uncertainty are presented in Appendix A.

4.1.2.3 Sample Compositing

A minimum of 20 to 30 grams of material is desired at each sample location. At the laboratory, each sample has its density measured, and then it is separately air dried, ground and sieved to a particle size less than 1,000 microns, homogenized, and weighed. The composite sample for analysis is created by removing (subsampling) the calculated proportional amount required from each location sample. Each composite sample would weigh approximately 70 grams. Based on SRNL calculations, a 70 gram sample mass is desired to ensure enough material is available for re-analyses or complex separations necessary to reach the Minimum Detectable Concentration (MDCs) typically requested. [SRNL-RP-2010-00084]

In general, a minimum of three composite samples will be produced from the minimum 15 location samples collected in a waste tank. As mentioned earlier, depending on the final material volume and distribution, fewer or more samples per array, fewer or more arrays, or more than three composite samples may be required to representatively characterize the residuals. Composite samples greater than 70 grams could be created if more than five samples per array are collected. More composite samples could be created if more arrays are sampled.

The number of samples, locations, and the proportional subsample mass needed for each location will be developed based on the residual material distribution and volume, and documented in TSAP and TTR revisions.

Any excess sample material will be archived for a length of time to be decided on a waste tank-by-tank basis. If necessary, the excess material could be used to create additional samples for analysis, but the MDCs, analyte list, and representativeness for waste tank characterization will need to be evaluated.

4.1.2.4 Example Sample Plan Designs and Calculations

Figure 4.1-2 shows a hypothetical sampling plan design that could be used for a waste tank with a uniform material layer on the floor. The three sample arrays with 15 total sample locations would collect material in all waste tank floor areas.

Figure 4.1-2: Hypothetical Sampling Arrays and Sample Locations in a Waste Tank with a Uniform Material Layer on the Floor

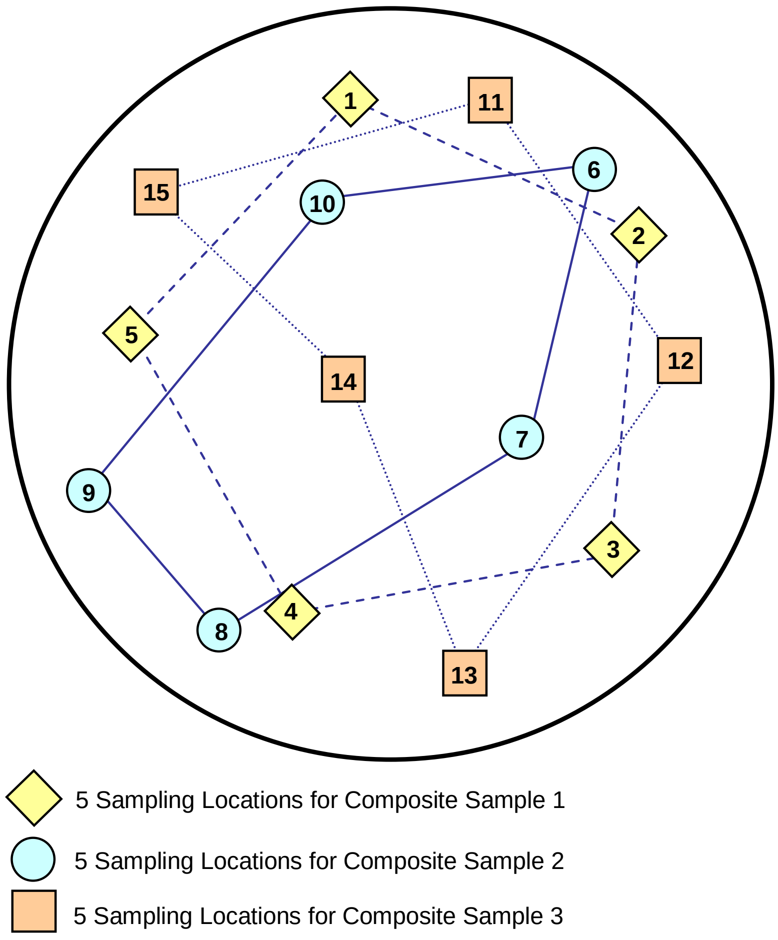
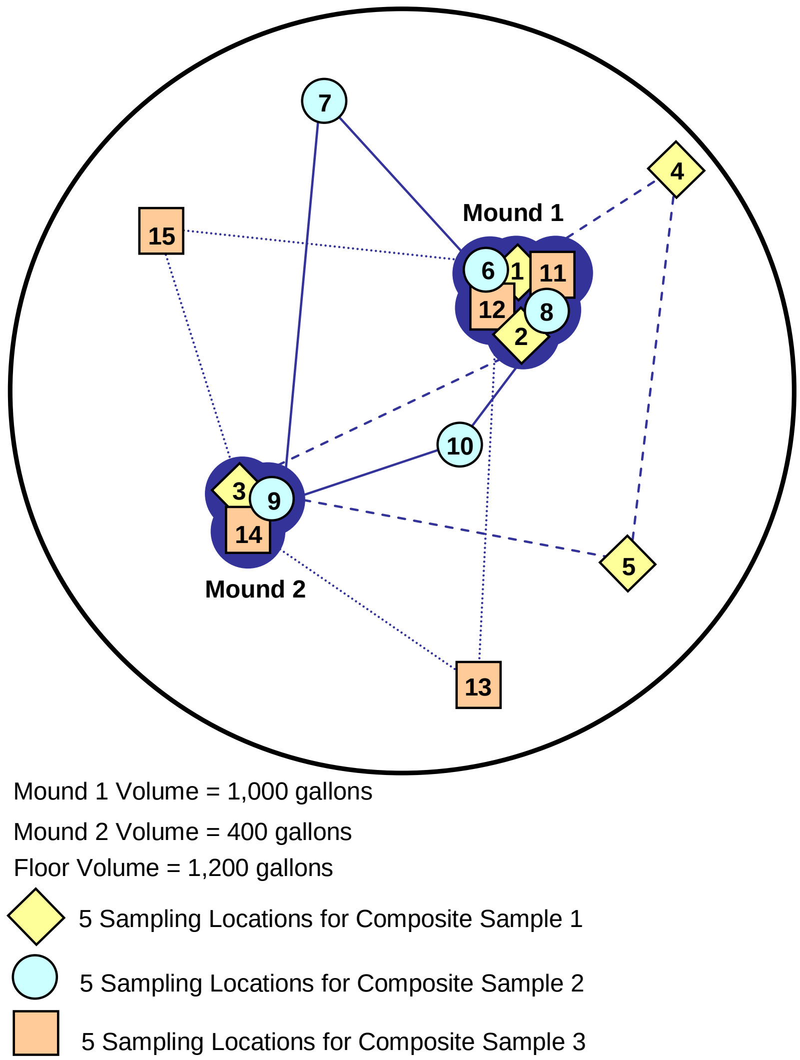


Figure 4.1-3 shows the hypothetical sampling arrays and locations that could be used in a waste tank containing two mounds and a uniform material layer on the floor. As explained above, sample locations will be placed to collect more samples in segments or features (mounds) containing more material. Volume-proportional compositing would be used to relatively represent those materials in the final composite sample.

An example calculation for the number of samples per feature, the volume-proportioning calculation and volume uncertainty adjustment is presented in Appendix A.

Figure 4.1-3: Hypothetical Sampling Arrays and Sample Locations in a Waste Tank Containing Two Mounds and a Uniform Material Layer on the Floor



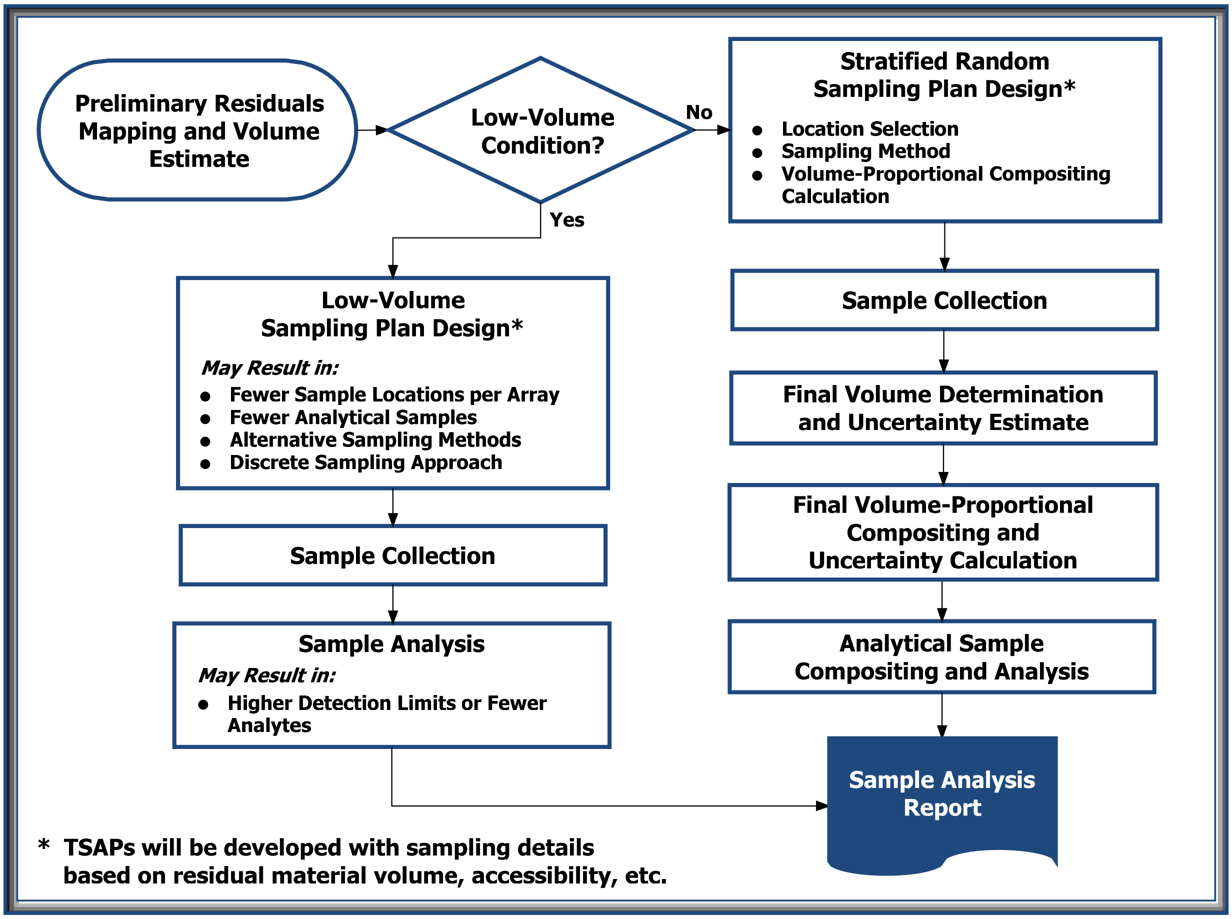
4.1.2.5 Low-Volume Condition Sampling

A low-volume condition might exist when only a thin layer of residual material remains on the floor, or material is only present in one part of the waste tank. In such cases, and if it can be used, the volume-proportional compositing approach will require modification.

With less material available, fewer sample locations per array or fewer arrays could force a reduction in the size (mass) or number of composite samples. Smaller composite sample sizes could require raising the MDCs or possibly impact the analyte list by limiting the material available for complex separation techniques or re-analyses. In some cases, discrete locations sampling and analysis might be more appropriate for residuals material characterization. In extreme low-volume cases, alternative collection methods, such as swipe sampling, might be necessary. Figure 4.1-4 is a flowchart showing the options for sampling under low-volume residuals material conditions. The actual material amount(s) recovered

during sampling would determine the number of final samples and analytical parameters. The decision to use the low-volume approach will be determined using best professional judgment of the actual waste tank residuals volume and distribution.

Figure 4.1-4: Flowchart Showing the Possible Options for Sampling Under Low-Volume Conditions



4.1.3 Sample Collection Equipment

Sampling the residuals material in a waste tank requires specialized equipment. Sampler design and use must take into account several considerations, including safety and minimizing personnel exposure. Ease of operation is an important criterion, which must integrate with quick, efficient sample handling after retrieval from the waste tank riser. The sample quantity required for laboratory analysis, the transport container size and configuration, and the need for camera support to perform remote sampling must also be factored into the sampling equipment design.

The sampling equipment must accommodate the physical limitations and access constraints as much as possible to obtain representative samples. Ongoing equipment research and design will continue throughout the waste tank removal from service effort. Sampling designs or collection methods, including any alternative methods other than those described

above, will be documented in TSAPs. Typical sampling equipment that may be used to collect waste tank residual waste material is described below.

4.1.3.1 Scrape Sampling

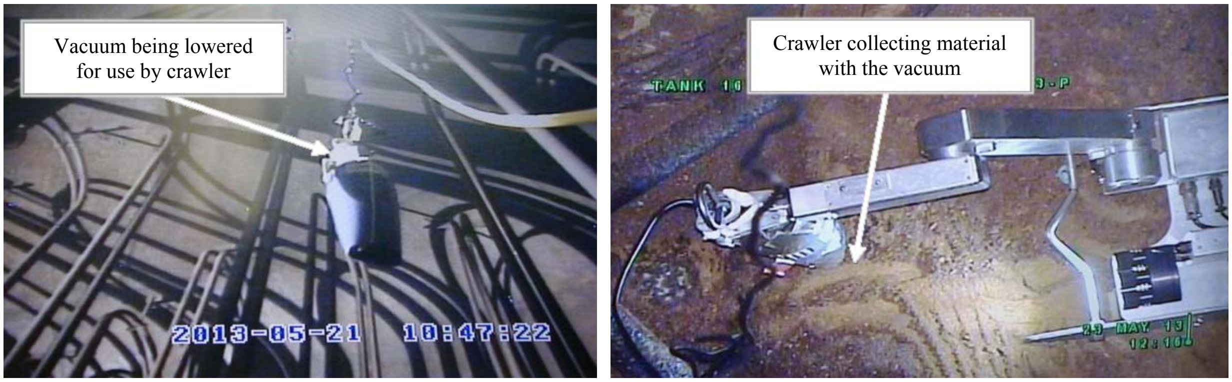
Scrape sampling is a practical method to collect material directly beneath waste tank risers. However, scrape samplers must be attached to poles 40 to 50 feet in length to reach material on the waste tank floor and the accessible area under a riser is limited. To collect samples further away from the risers, a robotic sample crawler may be used. A typical scrape sampler is shown in Figure 4.1-5.

Figure 4.1-5: Circle Scrape Sampler

4.1.3.2 Vacuum Sampling

In some instances where friable residual material is present, a narrow diameter vacuum cleaner has been used to collect samples. The vacuum can be either attached to a pole or manipulated by the mechanical arm of the robotic crawler. Figure 4.1-6 shows a vacuum being lowered into a waste tank and being used by the crawler to collect material.

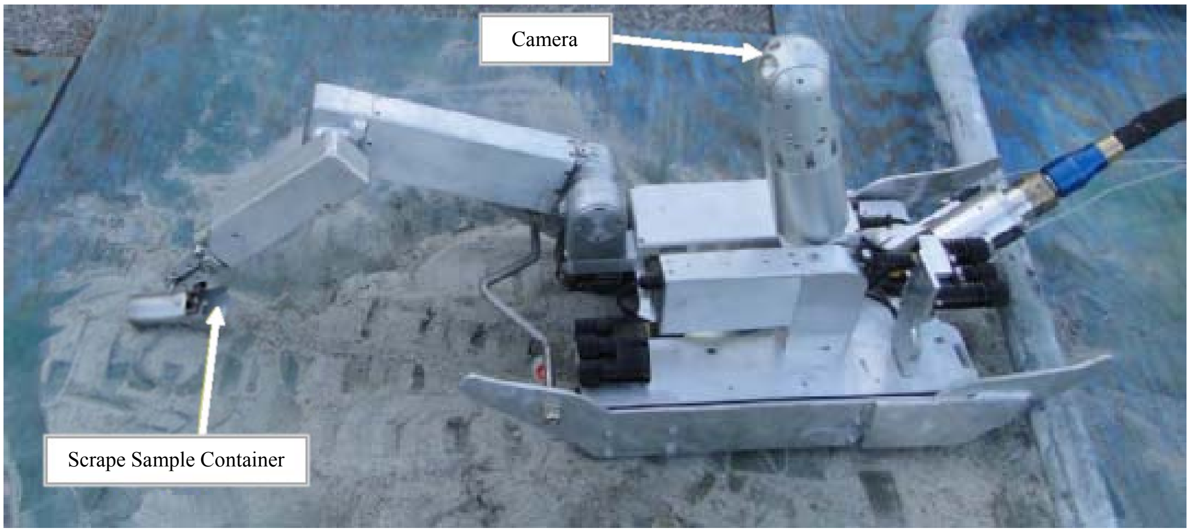
Figure 4.1-6: Sample Collection Using a Vacuum



4.1.3.3 Robotic Crawler Sampling

A robotic crawler may be used to sample some waste tank locations. An example of such a crawler is the modified Versatrak 450™ TTC crawler with attached scrape sampling device (Figure 4.1-7) which has been successfully used to collect samples in FTF Type I tanks.

Figure 4.1-7: Waste Tank Crawler Sampler



The crawler offers numerous advantages over other sampling devices in some situations. Because the sampling technician operates the device remotely, radiological exposure is avoided. The crawler uses a modular design and can be lowered through riser openings greater than 18 inches in diameter. Wet or dry material can be collected by dragging a specially designed scrape sample container across the waste tank floor. If necessary, multiple passes can be made to fill the container. Sample containers can be switched while the crawler is inside the waste tank so that multiple samples can be collected at one time. A basket-type carrying device holding up to two sample containers is used during sample collection. The mechanical arm can also be adapted to manipulate a sample collection vacuum. Additional information on crawler sampling is presented in Section B2 of the LWTRS-QAPP.

Although the crawler is able to negotiate much of the difficult terrain of the waste tank floor, it may not be able to reach all floor areas for sampling. Open riser availability for inner waste tank access, limited capability to negotiate between cooling coils rows or to crawl over horizontal cooling coil rows near the floor, and control cable management issues can restrict crawler movement.

4.1.3.4 Core Sampler

A core sampler is shown in Figure 4.1-8. Coring can be used if an area beneath a riser has a sufficient residual material thickness for penetration and capture inside the device. Since coring would be performed using 40 to 50-foot long poles, it can only be used in the limited area beneath waste tank risers. If necessary, the use of a core sampler and core sampling locations on a waste tank floor would be specified in the TSAP.

Figure 4.1-8: Core Sampler

4.1.3.5 Liquid Sampling

If free standing liquid (e.g., liquid not integral to the residual solids material) is present after cleaning and drying efforts cease, liquid samples may need to be collected. The Closure Project Team, primarily RM&A, would develop the analyte list and target detection limits by evaluating the waste tank history and expected soluble constituents. The liquid collection methodology and analysis would be developed and detailed in the TSAP.

4.1.3.6 Waste Tank Liner Metal Sampling

If characterization requires sampling the interior steel liner of a waste tank, the sampling would be performed using a methodology similar to that used for previous wall samplings in Tanks 18 and 19. The first few thousandths of an inch of the inner liner wall surface will be collected using a special drill fitted with filter pads that catch the cuttings. [SRNL-STI-2009-00802, SRNL-STI-2009-00799]

4.1.3.7 Cooling Coil Metal Sampling

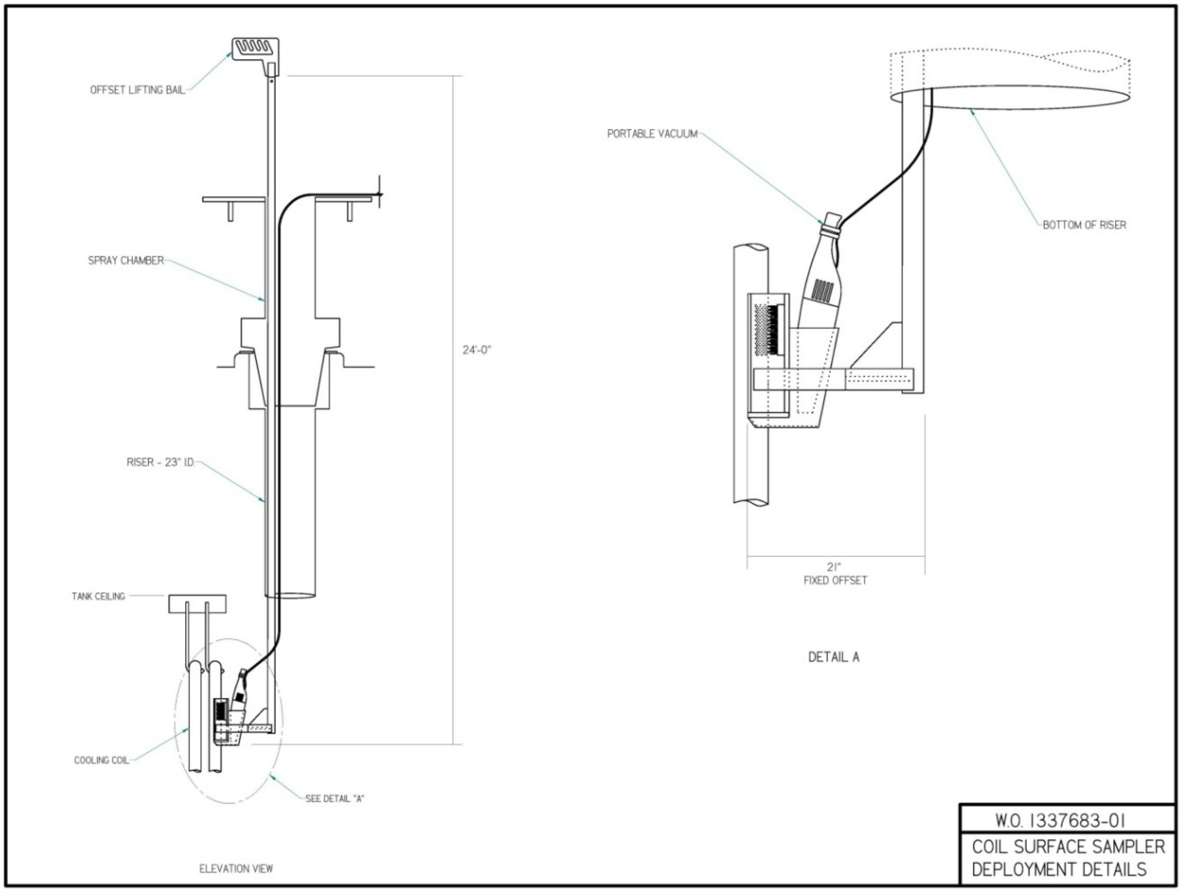
An evaluation was conducted on cooling coil sections collected from Tank 5 to assess potential residual contamination following internal flushing and external cleaning. As part of the waste removal in Tank 5, a series of oxalic acid spray washes followed by deionized water washes were performed. The inventory determined for the coil surfaces was orders of magnitude less than the floor residuals material inventory. [SRR-CWDA-2012-00027] For similarly cleaned waste tanks, the cooling coil inventory is also expected to be much less than the floor residuals material inventory. Therefore, additional cooling coil metal sampling and characterization are not anticipated.

4.1.3.8 Cooling Coil or Waste Tank Interior Scale Sampling

During the Tank 12 characterization, samples of a crystalline scale material coating the upper portions of the cooling coils were collected using a specially developed scraping and vacuuming tool (Figure 4.1-9). If similar scale is encountered in future waste tank

characterizations, the tool used in Tank 12 might be used again (Figure 4.1-9). If scale sampling is performed on different interior tank surfaces, another specialized tool might need to be developed.

Figure 4.1-9: Tank 12 Cooling Coil Scale Sample Collection Tool



[SRR-LWE-2014-00051]

4.1.3.9 Abandoned Equipment in the Waste Tank

No sampling of equipment that will be abandoned in the waste tank is planned due to the relatively small volume of residual material that might remain internally as "hold up". SRR Engineering uses process knowledge and equipment designs to estimate the appropriate residuals volume remaining for use in the inventory determination.

4.1.4 Sample Collection Procedure

Sample collection will be performed and recorded using procedures described in SOPs and associated SET work packages. Laboratory subsampling of the collected material will be performed using instructions described in the TTR.

4.1.4.1 Sample Location Documentation

Sample locations will be documented during sampling using video footage and still camera photographs. Locations will also be identified and documented on waste tank floor

diagrams. SRR Engineering will issue a Sample Location Verification Document to authenticate the actual locations sampled.

4.1.5 Field Radiological Control Screening

Sampling is one of the highest worker dose activities performed during tank closure. Whole body dose, extremity dose, and beta dose to eyes are all of concern during equipment insertions and removals through risers, sample material and tool retrieval, and sample packaging for shipment operations. Activities relating to the sampling, sample handling, and characterization of the FTF/HTF residual materials will be done in accordance with requirements and procedures of the SRS 5Q Manual, *Radiological Control Manual*, to minimize worker exposure.

Shipping containers will be surveyed by Radiological Controls (RadCon) before use to verify they are uncontaminated. The exterior of the overpack containers holding samples are surveyed before placement inside the shipping container. Sample transport is described in Section 4.1.8.

4.1.6 Sample Containers

Scrape sample collection devices will be fabricated out of stainless steel or other appropriate material, and will not be reused. After fabrication, the collection devices are cleaned by wire brushing and then washed and dried prior to use. If vacuums are used, they are checked for foreign matter, cleaned if necessary, and tested. Unique sample identification information is marked on the exterior of each sample container or vacuum. Additional details are provided in Section B3 of the LWTRS-QAPP.

4.1.7 Sample Preservation

Sample preservation in the field is not feasible because the high radioactivity of the samples prohibits direct handling of the sample containers and residual materials. Sample preservation and holding times are not applicable for these samples as discussed in Section B3 of the LWTRS-QAPP.

4.1.8 Sample Transport

Samples and sampling equipment typically are removed from the waste tanks into a glove bag assembly fitted to the top of the waste tank riser. The sample basket or other sampling device is placed into plastic bags to contain any spillage and those bags are then placed inside specially fabricated plastic pipe overpack tubes and capped. The overpack exteriors are screened and transported to the laboratory as appropriate for highly radioactive samples inside a shielded shipping container. Additional details are presented in Section B3 of the LWTRS-QAPP.

SRS Sample Transport personnel follow *CST Sample Manual* procedures and fill out the required transport records and log. [SW11.1-SAMPLE] Sample custody is also tracked using the Liquid Waste Tank Residuals Sampling COC form as described in Section B3 of the LWTRS-QAPP. At the SRNL SCO facility, the shipping container is offloaded and the shipping and sample information is recorded into a sample receiving logbook. The SRNL sample receiver signs the transport records. Sample custody is transferred using the COC, and a copy of the COC form is returned to the Person in Charge (PIC) of the sample

collection. Actual verification of the sample identification and material condition is performed when the overpack containers are placed in the hot cell, opened, and the sample is unloaded and examined. The final sample identification and any sample condition problems are added to the COC and a copy sent to the PIC. The original COC is given to the SRNL Principal Investigator for inclusion in the Sample Analysis Report. Additional details are presented in LWTRS-QAPP Section B3.

4.2 Training

Details on worker training and certifications are presented in Section A8 of the LWTRS-QAPP.

5.0 ANALYTICAL METHODS

Residual material samples require both radionuclide and chemical constituent analyses. These analyses require special procedures to reduce worker exposure and meet the complex analytical requirements for some radioisotope measurements. The following subsections describe, in general terms, the analytical guidelines for these analyses; more detail is provided in the LWTRS-QAPP. Sample analysis is designed to meet the LWTRSAPP DQOs discussed in Section 3.

5.1 Sample Preparation and Analysis

5.1.1 Sample Preparation

Volume-proportioning determinations for composite sample preparation will be performed as described in Section 4.1.2. Final subsampling weights for the compositing will be provided to the laboratory as a TTR revision following the final waste tank mapping and uncertainty determination. As discussed in Appendix A, each location sample will have the density measured and will then be air-dried, weighed, ground and sieved to a particle size less than 1,000 microns, and then be homogenized before subsampling to make the composite sample. Basic sample digestion is discussed in the LWTRS-QAPP. All initial sample preparations will be performed in the SCO facility of SRNL to reduce worker exposure.

If any liquid, scale, or other residual material type samples are required for characterization, the preparation methodology, analytes, and desired detection limits would be specified in the TTR and TTQAP.

5.1.2 Sample Analysis

Sample analyses are governed by the requirements of the LWTRS-QAPP. The waste tank-specific analytes will be specified in the TTR by SRR Engineering prior to sample receipt at the laboratory. The analytical requirements will be approved by appropriate SRNL and SRR Engineering personnel, and will serve as the primary input for the TTQAP.

5.1.2.1 Chemical Constituent Analytes

The chemical analyte list will be specified in the TTR on a waste tank-specific basis after a screening process is performed to determine the applicable analytes. Table A7-1 of the LWTRS-QAPP shows typical chemical constituent analytes and analytical techniques for residuals (solids) samples.

5.1.2.2 Radionuclide Constituent Analytes

At a minimum, the list of radionuclide analytes will be consistent with the HRRs listed in the *Basis for Section 3116 Determination for Closure of F-Tank Farm at the Savannah River Site* and the *Basis for Section 3116 Determination for Closure of H-Tank Farm at the Savannah River Site*. [DOE/SRS-WD-2012-001, DOE/SRS-WD-2014-001] Based on differences in FTF/HTF operations, specific waste tank usage history, advancements in the waste tank closure program, and needs of the respective FTF/HTF PA modeling, these analyte lists may be modified in the future. The analyte list for each waste tank will be specified in the TTR and TTQAP and justified in the subsequent Closure Module.

The necessary analytical requirements (MPC) for HRR sample analyses sufficient to meet the project DQOs are documented in the LWTRS-QAPP. In cases where waste tank characterization requires additional analyte-specific performance criteria, the requirements will be specified in the TTR and TTQAP.

Additional analyses may be conducted to determine special constituents as requested in the TTR. Those analytical procedures may be supplemented by research and development (R&D) directions. R&D directions are developed and managed in accordance with SRNL PS Manual, Procedure PL-AP-4006, *Administration of SRNL Research and Development Administrative and Work Control Documents*. Situations arising where the requested analyte resolution or detection limits may be impacted are documented, and the Project Manager and SRR Engineering are informed.

5.1.2.3 Analytical Sample Mass (Volume) Requirements

A 70 gram composite sample weight is desired to meet the MDCs for the analytes typically requested for waste tank residual samples. If insufficient material is recovered to create the composite sample, the Closure Project Team will be notified and appropriate action will be taken to produce a sample that still satisfies the LWTRSAPP DQOs.

If circumstances dictate smaller sample sizes, project personnel will consult with laboratory personnel to determine the impacts to the MDCs. The impact of higher MDCs will be evaluated against the LWTRSAPP DQOs in the subsequent DQA.

Section 4.1.2.5 discusses low-volume situations where only minimal residual material remains for sampling and the possible actions that could be taken.

5.2 Laboratory Sample Tracking

Samples will be received and tracked at the SCO facility in accordance with L1 Manual, Procedure 2.33. Sample receipt in the SCO is documented by the Sample Custodian on a sample receipt form in accordance with L7.7 Manual, Procedure 1.15, *SRNL Receipt of Radioactive Material*. The laboratory will log in the samples and use internal tracking and Laboratory Information Management System (LIMS) procedures to track material and aliquots for analysis.

For samples or subsamples sent to the Analytical Development Section (ADS), the Sample Custodian obtains a unique Analytical Development (AD) LIMS number that is also recorded in the laboratory notebooks. Samples and subsamples will be tracked by referencing the laboratory notebooks.

Because the volume-proportional compositing requires subsampling, the laboratory section performing the physical sample processing will generate a sample apportionment and compositing record and various internal aliquot-tracking records at the time of sample aliquot/split handling.

5.3 Laboratory Quality Control

SRNL will develop and adhere to QA/QC procedures to demonstrate consistency with the LWTRSAPP DQOs. The LWTRS-QAPP establishes laboratory criteria for the formulation, analysis, tolerance (acceptance levels) of the QA/QC samples for the HRRs, additional radionuclides, and chemical analytes. Sufficient numbers of QA/QC samples as specified in the

MPC will be analyzed with each batch of radionuclide and chemical analyses to ensure analytical objectives are met.

If available, standards for the preparation of laboratory control standards or matrix spike samples will be from a source independent of the laboratory standards, and a different lot number used for instrument calibration and meet the criteria defined in 1Q Manual, Procedure 2-7, *QA Program Requirements for Analytical Measurement Systems*.

5.3.1 Instrument Calibration

Analytical instrumentation will be calibrated before use and on an established frequency, and in accordance with both initial and continuing calibration requirements as described in Section B7 of the LWTRS-QAPP.

5.3.2 Background Measurement

For radiochemical analyses, regular background measurements will be made and monitored using control charts or tolerance charts to ensure that the laboratory maintains its capability to meet the required MPC.

5.4 Preventive Maintenance Procedures and Frequency

Maintenance will be performed by, or under the supervision of the Principal Investigator on laboratory instrumentation in accordance with the manufacturer's specifications as described in Section B6 of the LWTRS-QAPP.

5.5 Laboratory Assessment and Corrective Action

System and performance reviews will be performed as necessary by the appropriate Cognizant Quality Function (CQF) and corrective actions implemented as specified in the LWTRS-QAPP.

5.6 Data Reduction

Laboratory data reduction involves converting the sample and QC outputs of the analytical instruments into results usable by project personnel. This may involve separate calculations to adjust for instrument drift, sample dilutions, or isotopic ratios.

If additional data reduction is necessary for data use at the project level, it will be requested in the form suitable for task completion. Those data reduction procedures/techniques employed by the laboratory will follow the E7 Manual, *Conduct of Engineering,* and include a peer-reviewed check on the output data. The process will be documented and described in the Sample Analysis Report from the laboratory.

5.6.1 Measurement Uncertainty

Measurement uncertainty is discussed in Section A7.2 of the LWTRS-QAPP. For each radionuclide, two types of uncertainty will be reported:

1. The standard deviation of the mean result, and
2. The average of one sigma uncertainty arising from counting statistics.

For each chemical constituent, one type of uncertainty will be reported – the standard deviation of the mean result.

5.7 Data Verification

The data will be verified using the criteria specified in Section D of the LWTRS-QAPP. The analytical data verification will be performed and independently reviewed by qualified laboratory personnel. Verification checklists for the waste tank residuals sampling program are provided as attachments to the LWTRS-QAPP.

5.8 Data Validation

As described in Section D2 of the LWTRS-QAPP, a data validation for a limited suite of radionuclides and chemicals, and similar to the validation performed for the Tanks 5 and 6 Data Quality Summary Report, will be performed for every tenth waste tank characterized following Tank 6. Validation may also be performed if any anomalies are discovered during the data verification or data quality assessment steps. The validation will use data validation protocols and checklists specifically developed for the waste tank residuals sampling program. The validation protocols and checklists are provided as attachments to the LWTRS-QAPP.

5.9 Data Reporting

The TTR and LWTRS-QAPP will define the specific data deliverable requirements and if database uploading is necessary, specify the required electronic format.

6.0 DATA AND RECORDS MANAGEMENT

6.1 Records Management

Data management will consist of compiling and controlling the data generated during the waste tank residuals sampling and analysis effort. Data will be reviewed by the responsible organizational authority for completeness and accuracy before use, distribution, or storage. The data management system for records associated with the waste tank residuals characterization is described in Section B10 of the LWTRS-QAPP.

SRR Engineering and RM&A will be responsible for providing waste tank characterization records for retention. Figure 6.1-1 shows the sampling and analysis records that will be generated during the waste tank characterization. Any additional documents capturing changes to the sampling necessitated by occurrences or conditions would also be included.

SRNL will manage their records as described in the LWTRS-QAPP, Section B10.

6.2 Document Control

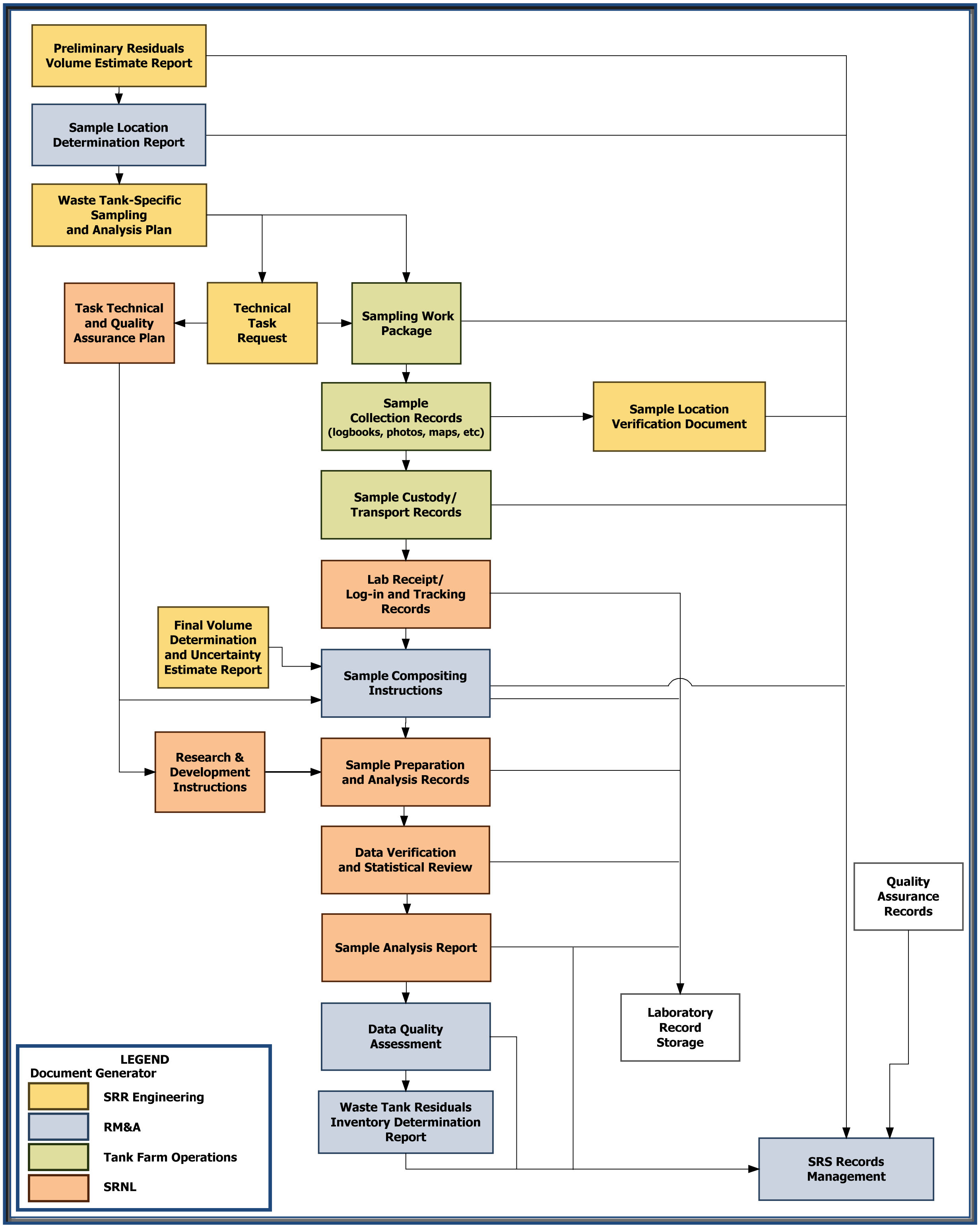
The documentation for the waste tank characterization program will be generated, reviewed, and approved by the organizations discussed in Section 2 and shown on Figure 2.5-1.

6.2.1 Waste Tank Sampling Records

Hard copy records are scanned and filed along with soft copy records in the Electronic Document Workflow System (EDWS) for long-term record retention and subsequent retrieval following SRS records management requirements.

The SRR Waste Tank Inspection and Monitoring Group is responsible for the collection and processing of video and photographic images. They also perform the indexing and storage of photographs and video footage of waste tank mapping and sampling activities.

Figure 6.1-1: Records Generated During Waste Tank Characterization Activities



6.2.2 Laboratory Records

As shown on Figure 6.1-1, the laboratory will generate TTQAPs, sample management, sample analysis, data verification checklists, and the Sample Analysis Report. Sample management records document sample receipt, sample login, and the assigned LIMS numbers used to track sample processing and analysis inside the laboratory. Sample analysis records contain information on the actual analysis of the samples and will typically include: LIMS sample tracking number, R&D directions or analytical method used, analytical instrument used, instrument calibrations, analysis date and time, dilutions, deviations from R&D directions or operating procedures, corrective actions, and detection and reporting limits, and test-specific QC criteria. A case narrative describing the analytical steps and any problems will also be generated. The data verification checklists, which provide traceability for the underlying analytical information, will be submitted as a separate deliverable to support the Sample Analysis Report.

Original analytical data and reports will be retained by the laboratory and, at the discretion of the laboratory manager or QA officer, may be transferred and stored in an electronic data management system. Records retention is discussed in LWTRS-QAPP Section A9.4.

6.2.3 QA/QC Records

SRNL QA records will be processed in accordance with 1Q Manual, Procedure 17-1, *Quality Assurance Records Management*. SRR QA records will be processed in accordance with 1Q Manual, Procedure 17-1Q, *Quality Assurance Records Management*. Preparation and processing of any controlled documents required will be in accordance with 1B Manual, Procedure 3.32, *Document Control*.

7.0 REFERENCES

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APPENDIX A

A.0 Technical Description of the Stratified Random Sampling with Volume-Proportional Compositing Approach

This appendix describes the basis for the approach used to sample residual waste tank material. The final sampling design will depend on the waste tank-specific conditions such as the final residual volume, distribution, and access. The characterization objective is to determine the average radionuclide and chemical constituent concentrations in the waste tank residuals. A stratified random sampling approach will be used for sampling to represent the residual material while volume-proportional compositing will be used to represent the material distribution in the waste tank. This approach was chosen to be the most appropriate to control sample error and meet the characterization objective while accommodating sampling equipment and access limitations.

Depending on the final residuals material volume and distribution, modifications to this approach, or an alternate approach, may be more appropriate for waste tank residuals characterization. The sampling and analysis process and low-volume sampling situation are described and discussed in Section 4 of the LWTRSAPP.

A.1 Sampling Errors

The error associated with a sample is highly dependent upon two components: (1) the size of the largest particles; and, (2) the mass of the sample, which is called the sample support. Sampling errors are increased substantially as the particle size increases and as the sample support decreases. These parameters contribute heavily to the Fundamental Error (FE), an incompressible minimum error that originates from the sample or subsample support, composition, shape, particle size distribution, and chemical properties of the material. The FE in a volume-proportional composite sample would be minimized by (1) creating a composite sample of sufficient support; and, (2) by reducing particle sizes to the extent practicable.

A.2 Sampling Error Management for FTF/HTF Waste Tank Residuals Characterization

The residuals material in the waste tanks consists of small particles of various size fractions. Despite material mixing during operations and waste removal, compositional variations will exist within the material. Even low levels of material heterogeneity can cause individual sample compositions to vary from the overall residual mass composition. The volume-proportional composite sampling approach applies Gy's sampling theory (ISBN: 0444420797) and practice to manage the previously mentioned sampling errors during waste tank characterization.

Pitard recommends the FE be less than 15% (ISBN: 0849389178) using Meyers FE formula (Equation 1), Ramsey's sampling constant of 20, and an assumed particle size of 500 microns, the required mass calculated for a sample increment of material would be less than 1 gram. [ISBN: 978-0-471-28556-4, EnviroStat_2008]

Because the material particles will clump during drying, the sample material will be ground and sieved to a size less than 0.1 centimeter (less than 1,000 microns). The set of parameters used in Equation 2 reflect those waste tank conditions and yield an FE of 1.69%. The small FE for field sampling (FE_{Field}) value resulting from Equation 2 indicates, that due to the small particle size of the residual material, the sample support (mass) would be driven by laboratory requirements.

Based on SRNL calculations to meet the detection limits requested for Tank 18 samples, a 70 gram sample mass is desired. [SRNL-RP-2010-00084]

$$m_i = \frac{20(0.05^3)}{0.15^2} = 0.11\,grams \qquad \text{Equation 1}$$

$$FE_{Field} = \sqrt{\frac{Cd^3}{m_s}} = \sqrt{\frac{20(0.1)^3}{70}} = 1.69\% \qquad \text{Equation 2}$$

Where:

FE	=	Fundamental Error
C	=	Sampling Constant
d	=	Diameter of the largest particles (cm)
m_i	=	Mass of sample increment required
m_s	=	Mass of composite sample
0.15	=	15% FE limit

An additional FE is incurred when the volume-proportions to create the composite sample are weighed in the laboratory. The density of the material collected at each sample location will be measured, the sample will then be homogenized, ground and sieved before the calculated volume proportion is weighed out (subsampled) to create the composite sample. For an assumed particle size of 0.1 centimeter, using the parameters above with an assumed subsample weight of 15 grams, the $FE_{Subsample}$ (Equation 3) is equal to:

$$FE_{Subsample} = \sqrt{\frac{Cd^3}{m_s}} = \sqrt{\frac{20(0.1)^3}{15}} = 3.65\% \qquad \text{Equation 3}$$

The combined FE, (FE_{Field} + $FE_{Subsample}$) would be approximately 5.34%.

Table A-1 shows the FE for various particle sizes and sample support weights (grams). Particle sizes for waste tank samples are anticipated to be less than 1 millimeter in the processed material that is subsampled to create the composite sample. The composite sample support is expected to be 70 or more grams and individual subsample weights are expected to range from 20 to 30 grams.

Table A-1: Fundamental Error for Various Particle Sizes and Composite Sample Supports

Maximum Particle Size (microns)	Maximum Particle Size (centimeters)	Fundamental Error for Composite Sample Supports (%)				
		5g	10g	30g	50g	70g
2,000	0.2	17.9	12.7	7.3	5.7	4.8
1,000	0.1	6.3	4.5	2.6	2.0	1.7
500	0.05	2.2	1.6	0.9	0.7	0.6
50	0.005	0.07	0.05	0.03	0.022	0.019

A.3 Number of Sample Locations

For unhomogenized materials with maximum particle sizes in the 0.2 to 0.5 centimeter range, Pitard suggests collecting material from 10 to 25 locations. [ISBN: 0849389178] For materials that have less heterogeneity and smaller particle sizes, fewer sample locations are necessary to control FE. Because the waste tank residuals material are expected to have maximum particle sizes between 0.05 and 0.1 centimeter and have been mixed during the waste removal process, from 5 to 15 sample locations in a waste tank would be sufficient to control the FE. Five sample locations were decided to be an optimum starting point for each array based on the assumption that the material is reasonably well-mixed during the bulk waste removal process. Three composite samples were determined to be the minimum number necessary to control uncertainty.

A.4 Sample Location Selection and Compositing

Sample locations will be selected to collect material from the segments (various material areas) identified in the waste tank for characterization. The logic for using stratified random sampling is to effectively represent material variability. The material variability across the waste tank would be captured and represented by volume-proportional compositing to create samples for analysis. Volume-proportioned composite samples will provide average residuals material concentrations.

To measure the representativeness and control the FE, a sampling design using a minimum of three sample arrays, each with at least five sample locations, will be developed for each waste tank to be sampled. The five samples from an array would be used to construct one composite sample for analysis, resulting in three samples per waste tank. The array designs and sample locations will be determined using the mapped residual material distribution. As explained below, sample locations will be selected to collect more samples in segments or features (mounds) that contain more material. The relative constituent concentrations in those materials would be represented in the composite samples using a volume-proportional compositing scheme. An example sample location determination and compositing calculation is presented in Section A.5.

Figure A-1 shows three hypothetical sample arrays for a waste tank containing a uniform layer of material on the floor. Figure A-2 shows hypothetical sample arrays for a waste tank with two mounds and a uniform material layer on the floor. Figure A-3 illustrates how the collected material will be processed to create the final volume-proportional composite samples.

Figure A-1: Hypothetical Sampling Arrays and Sample Locations in a Waste Tank Containing a Uniform Layer of Material on the Floor

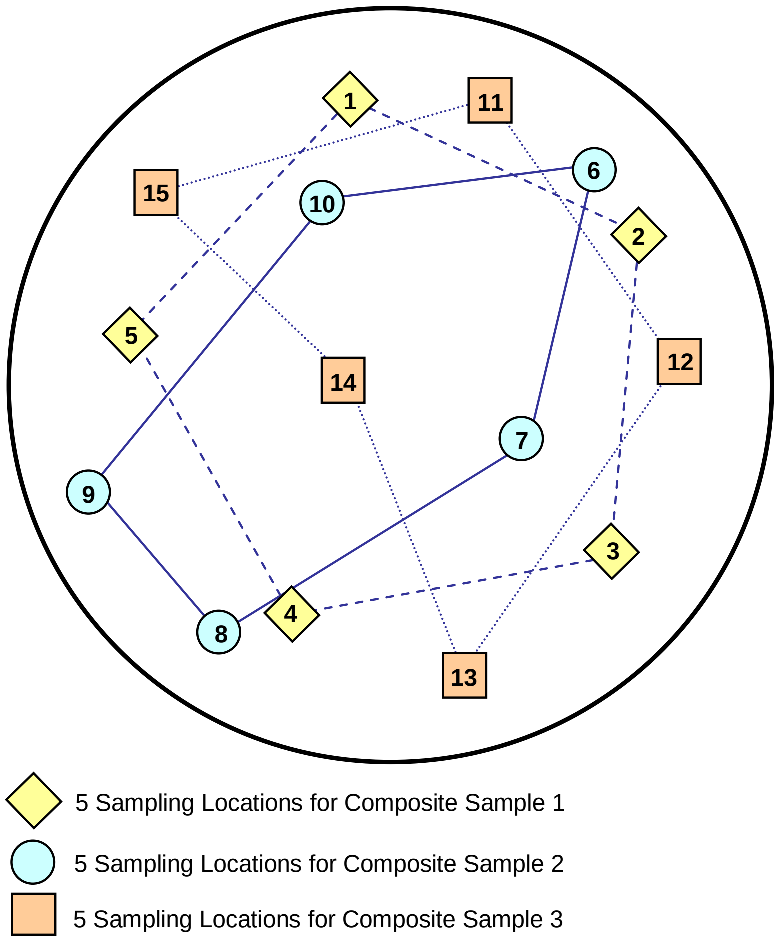


Figure A-2: Hypothetical Sampling Arrays and Sample Locations in a Waste Tank Containing Two Mounds and a Uniform Layer of Material on the Floor

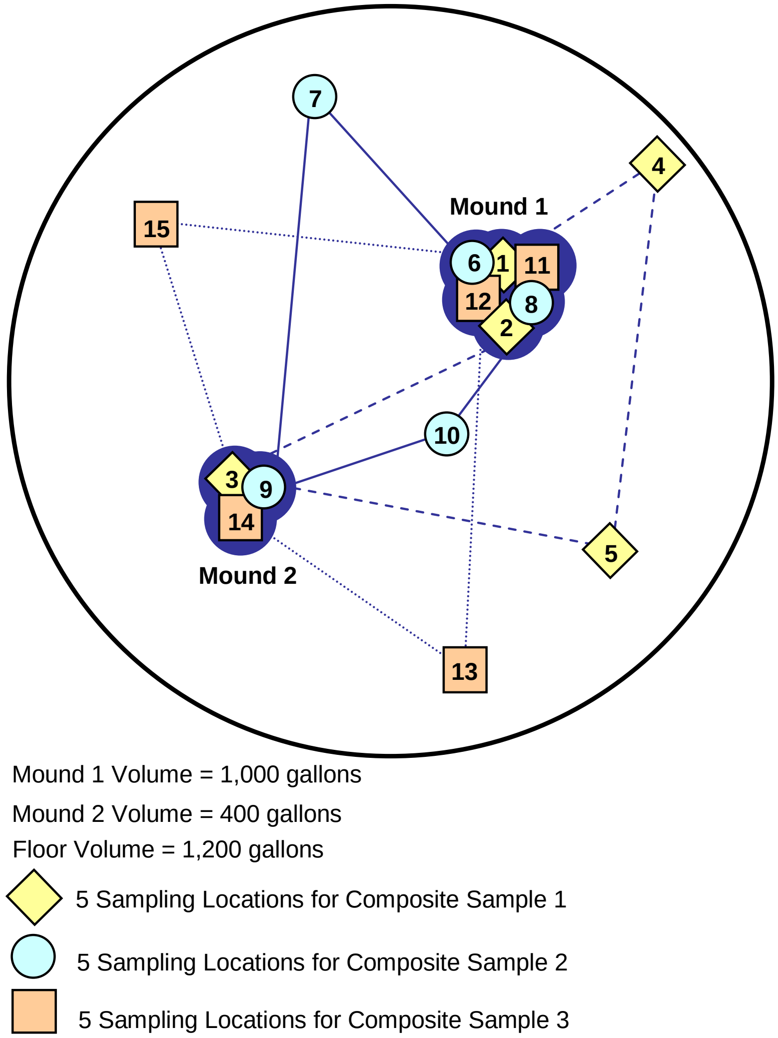
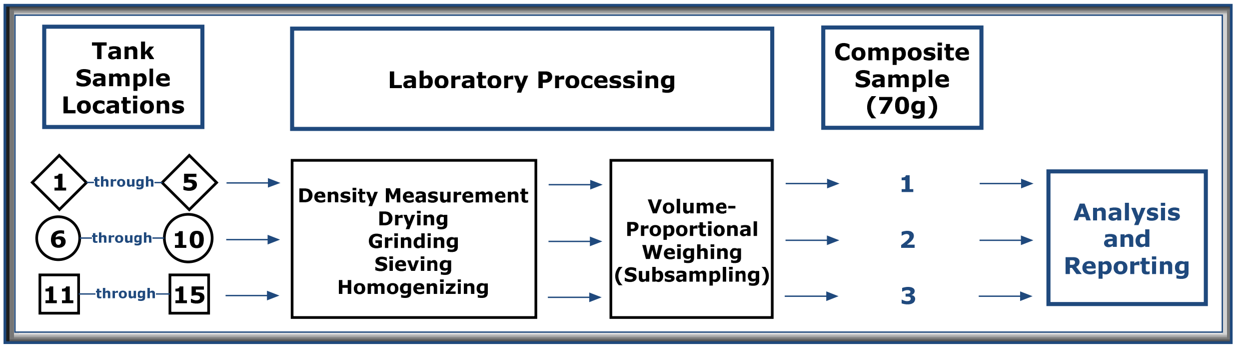


Figure A-3: Sample Material Processing Steps to Construct Composite Samples



The number of waste tank sampling locations would be specified in the TSAP. The location selection would be documented in the Sample Location Determination Report (SLDR) as described in Section 4 of the LWTRSAPP. It is anticipated that three composite samples, one per five-sample location array, would be adequate to characterize each waste tank.

The composite sample would be constructed using a volume-proportioning scheme. The composite sample is initially assumed to have a volume of 70 milliliters with an assumed density of 1.0 gram/milliliter. This 70 milliliters = 70 grams assumption will be used to estimate the approximate sample mass needed from a sample location for compositing.

To calculate the actual subsample mass from a location needed for compositing, the volume would be corrected for the actual sample density using:

$$M_s = (V_c)\,(V_p)\,\rho \qquad \text{Equation 4}$$

Where:

M_s	=	Volumetric sample mass (grams)
V_c	=	Composite sample volume (mL)
V_p	=	Proportion of (volume at sample location or area) to (total residual material volume)
ρ	=	measured sample density (grams/mL)

A.5 Example Location Determination and Compositing Calculation

The example described below shows how the general volume-proportioning approach would be used to determine the number of sample locations per area or feature and the volume-proportioning calculation to construct one composite sample. The compositing methodology would be similar for the other two analytical samples.

This example uses the hypothetical waste tank shown in Figure A-2, containing two mounds and a uniform material layer on the floor. It is assumed that Mound 1 contains 1,000 gallons; Mound 2 contains 400 gallons; and the floor layer volume is 1,200 gallons for a total residual volume of 2,600 gallons.

Determination of Samples per Area or Feature

The preliminary waste tank mapping volume estimate would be used to determine the relative volume (percentage) of material contained in a waste tank floor segment or feature such as a mound. Next, the number of samples in each array to be collected from that segment or feature would be calculated as shown in Table A-2 by:

$$\frac{V_f}{V_T} \times 5 = n_{SL} \qquad \text{Equation 5}$$

Where:

V_f = Segment or Feature volume

V_T = Total residual material volume

n_{SL} = Number of sample locations per feature

As shown in Table A-2, if more than one sample will be collected in a segment or feature, the volume proportion used for compositing would be adjusted by dividing the volume of that segment or feature by the number of samples collected. If multiple samples are collected from a mound of sufficient thickness, coring or depth sampling may be performed and material from different depths used for the compositing.

Table A-2: Sample Number and Volume-Proportioning Calculation for the Hypothetical Waste Tank shown in Figure A-2

Area or Feature (See Figure A-2)	**Area or Feature Volume (gallons)**	**Percentage of Final Residual Volume (%)**	**Calculated Samples per Five Sample Array**	**Samples to be Collected in Each Area or Feature**	**Proportion of Each Sample Location to be Used for Compositing (%)**
Mound 1	1,000	38.5	1.9	2	38.5/2 = 19.25
Mound 2	400	15.4	0.77	1	15.4/1 = 15.4
Floor	1,200	46.2	2.31	2	46.2/2 = 23.1

Determination of Volume-Proportioning Amounts

The amount taken from each location sampled would reflect the proportion of the total waste tank residuals volume present at that location as its portion of the final 70 gram sample weight. The composite sample is initially assumed to have a volume of 70 milliliters with an assumed density of 1.0 gram/milliliter. This 70 milliliters = 70 grams assumption is used to estimate the approximate sample mass needed from any location sample in an array to make the composite sample. For example, if one sample were collected in a segment that represented 23.1% of the total residuals volume, 23.1% of a hypothetical total mass of 70 grams (16.2 grams) would be used for the composite sample. The proportioning amounts for the example waste tank are shown in Table A-2.

Sample Compositing

To calculate the actual subsample mass needed from a sample location, the volume would be corrected for the actual sample density using the previously described relationship in Equation 4:

$$M_s = (V_c)\,(V_p)\,\rho$$

If enough material has been collected, either a composite sample greater than the 70 gram minimum could be constructed or the volumetric sample masses could be adjusted as shown in Table A-3.

Table A-3: Calculated Sample Masses and Density Adjustments Used for the Hypothetical Composite Sample

Sample Location (See Figure A-2)	Proportional Sample Location Volume (%)	Proportion of the 70 gram Composite Sample (%)	Measured Density[a]	Volumetric Sample Mass[a] (grams)	Required Sample Mass for a 70 gram Composite Sample[a] (grams)
Mound 1	19.25	13.5	1.36	18.3	13.3
Mound 1	19.25	13.5	1.25	16.9	12.3
Mound 2	15.4	10.8	1.54	16.6	12.0
Floor	23.1	16.2	1.32	21.4	15.3
Floor	23.1	16.2	1.43	23.2	16.8
Total Composite Sample Weight				**96.4**	**69.7**

a Hypothetical values

Volume Uncertainty Adjustment

The final waste tank mapping report will provide the final volume determination and an uncertainty estimate (minimum and maximum values) for that reported volume. As part of the mapping process, distinct segments or regions, such as the waste tank floor or mounds, could be delineated and have a volume uncertainty assigned to their volume. Because a composite sample design is used, the volume uncertainty must be incorporated to provide representative results. This volume uncertainty can be taken into account by incorporating the volumetric uncertainties for each region into the compositing for each analytical sample.

If the volume uncertainty is incorporated into the compositing, the variation between composite sample results will reflect all the uncertainties associated with the sampling process. With this variation, the total uncertainty can be factored into the statistical parameter estimate process (e.g. confidence limits). In general, the confidence limit of the mean includes the total uncertainty of the mean concentration estimate.

The volume uncertainty is incorporated into the composite samples by adjusting their compositing portions for the volumetric uncertainty in the following way.

1. For each segment or feature, a random number is generated from a triangular distribution that ranges from the low to the high volume estimated for that feature or segment. That

number will be used as the volume for that segment or feature for the compositing calculation.

2. The process is repeated for each segment or feature.
3. The compositing proportions are calculated.
4. For each composite sample created, steps 1 through 3 are repeated.

Table A-4 shows volumes and uncertainty ranges for a hypothetical waste tank with three segments. Table A-5 shows the hypothetical segment volumes generated using the procedure described above.

Table A-4: Volumes and Uncertainty Ranges for Hypothetical Waste Tank with Three Segments of Residual Material

	Min Volume (Low Estimate) (gal)	Determined (Reported) Volume (gal)	Max Volume (High Estimate) (gal)
Segment 1	100	200	300
Segment 2	300	400	500
Segment 3	700	800	900

Table A-5: Random Volumes Generated for a Hypothetical Waste Tank with Three Segments of Residual Material

	Low Volume Estimate (gal)	High Volume Estimate (gal)	Randomly Generated Segment Volume Estimate (gal)		
			Composite Sample 1	Composite Sample 2	Composite Sample 3
Segment 1	100	300	250	190	130
Segment 2	300	500	360	420	460
Segment 3	700	900	780	820	800

Table A-6 presents the volume and proportion that would be used to composite Sample 1 for the hypothetical waste tank.

Table A-6: Sample 1 Compositing Using the Randomly-Generated Segment Volumes

	Randomly Generated Segment Volume (Table A-5)	Segment Volumetric Proportion of Waste Tank Volume	Composite Sample Volumetric Proportion
Segment 1	250	18%	18%
Segment 2	360	26%	26%
Segment 3	780	56%	56%
Total Waste Tank Volume	**1,390**	**100%**	**100%**

As stated above, for each composite sample required to characterize the residual material, Steps 1 through 6 would be repeated. Table A-7 shows the compositing volumes that would be used for the hypothetical waste tank with three samples.

Table A-7: Volume Proportions for the Hypothetical Waste Tank with Three Composite Samples

	Randomly Generated Segment Volume (Table A-5)	Volumetric Proportions for Composite 1	Randomly Generated Segment Volume (Table A-5)	Volumetric Proportions for Composite 2	Randomly Generated Segment Volume (Table A-5)	Volumetric Proportions for Composite 3
Segment 1	250	18%	190	13%	130	9%
Segment 2	360	26%	420	30%	460	33%
Segment 3	780	56%	820	57%	800	58%
Total	**1,390**	**100%**	**1,430**	**100%**	**1,390**	**100%**