

Exerpts from:

CoPhysics Corporation

STANDARD OPERATING

and

QUALITY ASSURANCE PROCEDURES

for

Radiological Laboratory Analysis

Survey Instrument Calibration

Radiological Field Services

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2 LABORATORY SERVICES

2.1 General Laboratory Procedures

The analysis procedures described in this manual are generic and may be modified for special types of analyses. Other referenced procedures such as the EPA 900 series may be used for specific analyses as requested by customers.

2.1.1 Laboratory Rules

1. All personnel must follow the "Model Rules for the Safe Use of Radioactive Material" as specified in the NYS radioactive materials license and posted in the laboratory area.
2. Use of the equipment is strictly limited to personnel designated by the laboratory manager.
3. Radioactive standards must be marked by labels on drawers or cabinets and on protective cases. Damaged standards must not be used for calibration or QA and will be disposed of properly.
4. Wipe testing of the laboratory floors and equipment surfaces must be conducted once per month. The results must be recorded and maintained in a binder or computer database.
5. All samples are to be treated carefully to avoid spillage which may result in the loss of sample and the possible contamination of the lab. No uncovered or unlabeled liquids or volatile samples are to be brought into the laboratory area.
6. Hands should be washed before and after handling samples; plastic gloves must be worn when handling radioactive solutions or chemicals.
7. Incoming and outgoing samples shall be stored in an orderly fashion using dedicated cabinets and shelves.
8. No samples with activity greater than 100 uCi are to be accepted by CoPhysics without notification of the NYS Dept. of Health. Samples that have greater than 10 uCi of radioactivity per sample, must be included in the radioactive materials inventory as would reference radioactive materials. The handling of such "hot" samples (> 10 uCi/sample) must be performed with designated "high activity" glassware and proper containment so that standard glassware, facility surfaces, and instrumentation will not experience an increase in background radioactivity. Warning of receipt of such "hot" samples should be obtained from the client, from CoPhysics personnel with knowledge of the facility of origin, from notations on the chain of custody or packing slip, or from the results of the receipt survey. The RSO may specify pre-testing of samples that are suspected of having > 10 uCi of alpha or low-energy beta emitters that would not be detectable by external measurements conducted during package opening. Samples received for analysis that contain less than 10 uCi of radioactivity per sample are not subject to radioactive material inventory tracking.
9. Samples containing tritium, radioiodine or other volatile forms of radioactivity must not be heated or processed until the RSO determines the potential for release of airborne radioactivity and specifies precautions and regulatory notifications necessary.

2.1.2 Receipt of Samples

All samples entering the laboratory must be given a sample ID number and entered into the laboratory computer database or log book. Other sample data such as customer name, analysis method to be used, customer ID number, date received, customer Chain of Custody number, etc. must also be entered upon sample receipt. The packing list or Chain of Custody record must be signed and filed by the person logging-in the samples. Such initial logging will prevent lost samples and will allow detection of broken containers, insufficient volumes, and other problems. Any such physical sample problems or discrepancies in the sample list must be resolved immediately with the customer.

Packages containing radioactive materials shall be surveyed per instructions in the Radiation Safety Manual. Packages containing samples not labeled as radioactive, but that may have the potential for $> \text{nCi}$ (or nCi/g) quantities of radioactivity, shall be checked with a GM survey probe (for beta/gamma emitters) and/or wipe or swab tested (for pure alpha emitters or tritium). The receipt of sample containers with contaminated external surfaces or packing material shall not be taken into the counting room and shall be reported to the RSO immediately.

2.1.3 Quality Assurance

1. Responsibility: It is the responsibility of the laboratory manager to ensure that all the analysis and quality assurance procedures are followed and that all customer requests and regulatory concerns regarding laboratory analysis are addressed.

2. Quality Assurance Tests: Quality assurance checks of the various analyses performed consist of:

a. Spiked samples: Calibrated (blind) samples are obtained from IAEA performance evaluation studies or similar program. If certain types of samples are not routinely available from such an independent agency, the laboratory manager will create or obtain suitable spiked samples along with supporting calculations or documentation to determine the actual activity for comparison with analysis results. Calibration standards may be used as for this purpose if analyzed as a sample. Analysis of spiked samples provides an extremely useful assessment of the **accuracy** of the laboratory.

b. Duplicate sample analysis: Duplicate, or "split", samples of water, soil, or bioassay media provide an estimate of the **precision** of the laboratory's analyses. In cases where splitting of inhomogeneous samples, such as soil, may not be possible, duplicate counts of the same sample may be substituted for analysis of duplicates.

c. Blanks: Blanks are essentially background samples that consist of actual sample media (e.g., urine, water, etc.) from an unexposed person or source. Such analyses provide evidence of proper sample handling and provide means to calculate minimum detectable activities (MDA's) for each type of analysis.

3. Control Charts: Control charts shall be maintained to track the results of calibration and background analyses. The charts shall show the means and 2-sigma and 3-sigma values. These parameters shall be updated routinely. Measurements of either variable exceeding ± 3 -sigma (control limit) shall result in an investigation of the operability of the equipment. Non-operability

(values beyond ± 3 -sigma) shall be reported to the company president immediately. Control charts shall be kept in a labeled binder or on computer.

4. QA Testing Schedule - The frequency of the quality assurance tests for each routine type of analysis performed are as follows:

- a. blind calibrated samples shall be analyzed at least once per year;
- b. duplicate samples: every 20 samples shall be accompanied by a duplicate; however, this is not applicable for wipe tests or samples containing insufficient volumes;
- c. blank or background samples: at least once per month in which the system is used;
- d. calibrations: at least once per year in which the system is used.

5. QA Criteria - The laboratory manager shall review and analyze the data with comparisons to the above guides at least on a quarterly basis. If the laboratory fails to meet any one of these guides, then the laboratory manager shall notify the company president in writing. The notification shall include the test results in question and an explanation of the criteria used to initiate this action. Actions necessary to correct the problem will be taken immediately. The laboratory must comply with the following criteria for each type of quality control check:

- a. blind sample analysis: the results must fall within the agency's control limits as specified in the published results of the performance studies.
- b. duplicates: 90% of the results must fall within 2 standard deviations of each other;
- c. blanks and calibration checks: the results must be less than ± 3 -sigma of the mean (the mean and standard deviation are calculated from the previous twenty results).

2.1.4 Disposition of Samples

Normally, samples from customers will be returned to them after analysis. If the customer requests disposal of the remaining media, the samples will be transferred to a licensed waste service. If the customer request storage of samples for possible later follow-up analysis, CoPhysics can provide this service. However, in no case shall non-disposable samples be retained by the laboratory.

2.1.5 Record Keeping

All quality assurance results, raw data, calibrations and calculations shall be recorded and kept on file for 5 years.

2.3 Gamma Spectroscopic Analysis Procedures

2.3.1 Introduction

The identification and quantification of gamma-emitting radionuclides in various sample media such as liquids, soil, and air filters is necessary for environmental analyses and related health physics purposes. The solid state gamma-ray spectrometer makes this possible through the use of advanced electronics and computer software technology.

2.3.2 Equipment

- High-purity germanium detector (HPGe) at least 10% efficient relative to a 3"x3" NaI detector
- Liquid Nitrogen
- Pb shield of at least 4" thickness with graded Z inner lining
- Computer-based Multichannel analyzer
- HV Power Supply
- NIST-traceable standards

2.3.3 Sample Preparation and Analysis

1. Enter the description of each sample under the next available ID number in the sample database or log book. Label sample containers with the corresponding log ID numbers. Also enter any supporting data (e.g., date of count, customer number, initial weight, etc.).
2. Weigh the sample and record the data in the database.
3. If the sample is to be dried, place the sample into an oven or under a heat lamp in the hood for a time sufficient to dry the sample to a constant weight. Re-weigh the dried sample and enter the dry weight into the sample database.
4. Place the sample into the desired counting geometry: either a 10 mL petri dish or 200 mL plastic sample jar, Marinelli beaker, or various other custom geometries.
5. Place the sample on the germanium crystal inside its shielded housing. Be careful to place the sample gently and on center on top of the crystal. Keep a plastic liner between the sample container and the crystal. Close the shield door carefully and completely.
6. Refer to the computer software user's manual to initiate the counting sequence. The software will acquire a spectrum, store it, and analyze it. The results will be printed and/or transferred to the main sample database.
7. The laboratory manager shall predetermine the count time needed to detect known or suspected nuclides in the sample based on sample mass, geometry and detector efficiency as used in MDA calculations.

2.3.4 Background Counting

To obtain background spectra for the spectroscopic analysis software, perform the following procedure:

1. Prepare a background sample consisting of distilled water using the same counting geometry as the sample(s) to be counted.
2. Count the background sample for at least 20,000 seconds.
3. Refer to the computer software user's manual to initiate the counting sequence. The software will acquire a spectrum, store it, and analyze it. The results will be printed and/or transferred to the main sample database.
4. Enter the peak search results into the spectroscopy software's "peak background file".
5. The background procedure shall be conducted at least once per year or when the laboratory manager has reason to suspect a change in background has occurred.

2.3.5 Calibration

NIST-traceable radioactivity standards should be configured into all counting geometries used.

Perform the following procedure for each counting geometry:

1. Count the standard for at least 1000 sec.
2. Refer to the computer software user's manual to initiate the counting sequence. The software will acquire a spectrum, store it, and analyze it. The results will be printed and/or transferred to the main sample database.
3. If this is the first calibration for the geometry used, enter the new photopeak efficiencies into the spectroscopy software's efficiency file and perform the efficiency curve fit per the software manual instructions. Subsequent counts of the standards for each geometry may be used as calibration checks, i.e., keep the original calibration curve in the efficiency file and only use the recent count to check the calibration compare them to the new photopeak efficiencies.

This calibration procedure shall be performed at least annually or when the laboratory manager has reason to suspect that the detector efficiency or energy calibration may have changed.

2.3.6 Quality Assurance

All applicable quality assurance requirements discussed in Section 2.1.3 shall apply to HPGe analysis. Specific quality assurance checks of the HPGe system include:

1. Blind unknowns: Calibrated (blind) fission/activation product samples are periodically obtained from the USEPA, USDOE or IAEA performance evaluation programs.
2. Duplicate sample analysis: 1 of every 20 samples shall be analyzed twice or, if available, a duplicate should be analyzed.
3. Blanks: at least 1 per month in which the system is used
4. Calibrations: annually. New efficiency, resolution and energy calibration curves shall be generated during the calibration.
5. Calibration checks: at least 1 per month in which the system is used. Two (2) photopeak efficiencies shall be tracked in a QA chart per section 2.1.3. The photopeak resolution shall also be checked by the QA reviewer to ensure that detector degradation has not occurred.