

5. LABORATORY PROCEDURES

5.1 INTRODUCTION

Procedures for laboratory analysis comprise, by definition, an integral element of the environmental monitoring program; that is, both effluent monitoring and environmental surveillance are defined, in part, in DOE Order 5400.1 as “the collection and analysis of samples.” Those objectives of the EMP concerning compliance with applicable regulations and commitments, identification of facility contributions to ambient contaminant levels, and determination of the effectiveness of effluent treatment and controls would be impossible to measure without a valid and reliable analytical system.

In compliance with the *Regulatory Guide*, laboratory procedures and practices are documented in this section to show:

- sample identification systems,
- procedures preventing cross-contamination,
- analytical methods and/or modifications thereof,
- analytical capabilities,
- equipment calibration and reference source practices, and
- other QC procedures.

5.2 ORGANIZATION

Depending on the program, sampling and analytical services are provided by BWXT Y-12, L.L.C., U.T.-Battelle, or various subcontractors. Responsibilities of these organizations include (1) developing statements of work for analytical services, (2) determining appropriate analytical protocols to be applied to meet the DQOs, (3) developing technical and quality standards for requested analytical services, (4) deciding on appropriate laboratories with which to place the work, (5) negotiating schedules for provision of services and oversight to ensure schedules are met, (6) assessing laboratories to ensure they comply with quality and technical standards, and (7) ensuring appropriate validation and transmittal of data to the program customer.

5.3 GENERAL QUALITY ASSURANCE

A Laboratory Quality Assurance Plan (LQAP) shall be developed by each analytical laboratory (in-house and subcontractor) performing analyses of environmental and waste samples. The purpose of an LQAP is to ensure that quality data are generated. LQAPs will address the requirements of DOE Order 5400.1 with respect to laboratory QA and data verification and will address the laboratory procedures requirements in the *Regulatory Guide*. In the context of laboratory qualification, an LQAP provides a basis for evaluating a laboratory’s QA procedures.

This evaluation includes a critical review of the LQAP and verification of the laboratory's adherence to the LQAP through on-site audits.

The Y-12 Analytical Chemistry organization provides a wide range of analytical services supporting environmental, radiological, mixed-waste, defense, and industrial hygiene programs. The organization's qualifications include American Industrial Hygiene Association (AIHA) accreditation; state of Tennessee Drinking Water certification; state of Utah RCRA, SDWA, and CWA certification; and the state of Tennessee Approved Listing for the UST program.

5.4 ANALYTICAL METHODS

Using guidance from EPA, the laboratories document the steps in sample handling, analysis, data collection, data reduction, and approval of results. The performance of all analytical procedures is conducted using EPA-approved methods when they are available. Analytical activities are documented in SOPs and various QA records (such as logbooks and data files). Laboratories determine laboratory-specific method detection limits (MDLs), which must meet the requirements for MDLs specified by the EPA. Laboratories also establish the lowest concentration reported (LCR), which must meet the needs of the programs they support. The LCR may vary among specific samples, depending on interferences in the sample matrix. However, these LCRs have been assigned to accommodate most minor interferences. Consistent with guidance from EPA, some of the LCRs may be higher than the MDLs specified by EPA. However, any data reported below the EPA MDLs must be consistent with the laboratory-determined MDL and be supported by sound documentation.

A compilation of routine analytical services provided by the Y-12 laboratory is recorded in *Analytical Activities Summary for the Analytical Services Organization, Y/DK-1034 (BWXT Y-12 1998)*. This document lists commonly requested parameters, the laboratory test codes used to request analyses, the analytical method used, the holding time, preservation, required sample size, and routine lower reporting limits.

5.5 REGULATORY GUIDE PERFORMANCE CRITERIA

a. Laboratory procedures and practices *should be documented in the site EMP.**

Those procedures and practices that are fundamental to the analysis of environmental monitoring samples are documented in LQAPs and/or sampling and analysis plans. Standard analytical methods are specified by EPA for use with their regulations. When EPA methods are not available, other standard methods are used, such as American Society of Testing and Materials (ASTM) or Standard Methods for the Examination of Water and Wastewater. Specific laboratory procedures are developed and documented by each of the analytical laboratories in their SOPs.

b. Each monitoring and surveillance organization *should have a sample identification system that provides positive identification of samples and aliquots of samples**

throughout the analytical process. The system *should** incorporate a method for tracking all pertinent information obtained in the sampling process.

When samples are logged into the laboratory, a unique laboratory identification number is assigned. Labels bearing this number are affixed to the sample bottle.

Samples and analytical results are tracked and maintained in databases such as PEMS. Additional records are worksheets, notebooks, and laboratory computer printouts.

- c. **Each laboratory *should** establish and adhere to written procedures to minimize the possibility of cross-contamination between samples. High-activity samples *should** be kept separate from low-activity samples.**

Written procedures for cleaning glassware are followed at each laboratory cleaning facility for this purpose. Single-use disposable lab equipment is used where practical to minimize the need for washing. Some equipment (e.g., gas chromatographs and gas chromatographs/mass spectrometers) is specified for low-level concentrations only. The analysis of field and preparation blanks gives an indication of contamination levels.

Volatile samples should be stored separately from other sample types. Additionally, refrigerator blanks should be kept in refrigerators where volatile samples are stored and should be analyzed for contamination.

Historical data and in-lab screening procedures are used to segregate samples. High-level radioactive samples and low-level samples are sent to different laboratory work areas specifically designated for each radioactivity level.

- d. **The integrity of samples *should** be maintained (i.e., minimize degradation of samples by using proper preservation and handling practices that are compatible with analytical methods).**

Samples requiring preservative arrive at the analytical laboratory with the preservative added. This fact is noted on the chain-of-custody form. Samples that need to be kept at 4°C are placed in monitored refrigerators. All samples are kept in secure storage.

- e. **Specific analytical methods *should** be identified, documented, and used to identify and quantify all radionuclides in the facility inventory or effluent that contributes 10% or more to the public dose or environmental contamination associated with the site.**

Laboratories will follow the sampling and analysis plans prepared by EM staff as to the required methods for analysis. These will be documented in the request for analytical services and will be contained in the laboratory information management system.

- f. Standard analytical methods *should** be used for radionuclide analyses (when available). Any modification of standard methods *should** be documented.**

EPA methods are the methods of choice. Only when such a method is not available are other standard methods used. Laboratory staff work with program coordinators to identify the methods required for the program, and the program coordinator must approve the use of alternate methods. Laboratories must keep documentation of the procedures used and any method modifications. Modifications may be made following procedures outlined in the sampling and analysis plans.

- g. Methods, requirements, and necessary documentation *should** be specified in analytical contracts.**

Analytical contracts with commercial laboratories are negotiated by the sample management organization for each program. These laboratories are all prequalified to perform work for DOE contractors. Any requests sent to these laboratories will include a listing of the methods to be used.

- h. All sites that release or could release gamma-emitting radionuclides *should** have the capability (either in-house or outside) of having samples (routine, special, or emergency) analyzed by gamma-ray spectroscopy systems.**

Multiple analyzer capability or backup for all the tests required for inorganic, organic, and radiochemical measurements are available at DOE-owned and commercial laboratories. This duplication provides assurance that prompt attention is available for special or emergency samples.

- i. Counting equipment *should** be calibrated using, at a minimum, the calibration frequency recommendations of the manufacturers to obtain accurate results.**

Equipment used for NPDES, RCRA, or compliance work is calibrated according to EPA procedures. In the absence of EPA guidelines, manufacturers' recommendations are used. QC samples and calibration standards from separate sources provide additional assurance.

- j. Check sources *should** be counted periodically on all counters to verify that the counters are giving correct results.**

Check sources or common standards are used periodically with multiple instrumentation to ensure a common basis. Internal and external control and standards from another vendor are used to verify the correctness of the check sources.

- k. **Samples that are sent off-site for analysis or for laboratory intercomparison *should** be monitored for contamination and radiation levels and *should** be packaged in a manner that meets applicable transportation regulations and requirements.**

All samples sent off site are controlled by Radiological Control and Department of Transportation regulations. Samples are surveyed and tagged by Health Physics personnel for surface contamination. In addition, samples are prescreened for radiological content based on process knowledge, historical data, or analytical data. If measurable radioactivity is present, the off-site laboratory is contacted to confirm that they can receive the samples and that their Nuclear Regulatory Commission (NRC) license, state license, or Letter of Exclusion will not be exceeded.

- l. **As they apply to laboratory procedures, the general QA program provisions of Chap. 10 of this guide (the *Regulatory Guide*) *should** be followed.**

Audits and surveillances are planned, conducted, and documented to verify compliance with the QA program. Laboratory activities are conducted with full cognizance of ASME NQA-1 requirements. Analytical laboratory procedures also meet the quality requirements of DOE contractors and federal regulators.

Analytical laboratories participate in DOE's QA program for those nuclides and media that they regularly measure. Spiked samples and blanks are routinely used for QC and QA.

In addition, the laboratories analyze standards prepared by EPA to support the NPDES Program. They analyze for parameters on the permit and may choose to analyze other parameters on a voluntary basis for documentation or method checking. Laboratories performing work for NPDES compliance participate in an external measurement control program using samples purchased from commercial suppliers. Evaluation reports are reviewed internally to determine if there are any quality problems.