9 Quality Assurance

Quality assurance is an integral part of every activity at Brookhaven National Laboratory (BNL). A comprehensive Quality Assurance/Quality Control (QA/QC) Program is in place to ensure that all environmental monitoring samples are representative and that data are reliable and defensible. QC in the contract analytical laboratories is maintained through daily instrument calibration, efficiency and background checks, and testing for precision and accuracy. Data are verified and validated as required by project-specific quality objectives before being used to support decision making. The multilayered components of QA monitored at BNL ensure that all analytical data reported for the 2008 Site Environmental Report are reliable and of high quality.

9.1 QUALITY PROGRAM ELEMENTS

As required by DOE Order 450.1A, Environmental Protection Program, BNL has established a QA/QC Program to ensure that the accuracy, precision, and reliability of environmental monitoring data are consistent with the requirements of Title 10 of the Code of Federal Regulations, Part 830 (10 CFR 830), Subpart A, Quality Assurance Requirements (2000) and DOE Order 414.1A, Quality Assurance. The responsibility for quality at BNL starts with the Laboratory director, who approves the policies and standards of performance governing work, and extends throughout the entire organization. The purpose of the BNL Quality Management (QM) System is to implement QM methodology throughout the various Laboratory management systems and associated processes, in order to:

- Plan and perform BNL operations in a reliable and effective manner to minimize any impact on the health and safety of the public, employees, and the environment
- Standardize processes and support continual improvement in all aspects of Laboratory operations
- Enable the delivery of products and services that meet customers' requirements and expectations

For environmental monitoring, QA is deployed as an integrated system of management activities. These activities involve planning, implementation, control, reporting, assessment, and continual improvement. QC activities measure each process or service against the QA standards. QA/QC practices and procedures are documented in manuals, plans, and a comprehensive set of standard operating procedures (SOPs) for environmental monitoring (EM-SOPs). Staff members who must follow these procedures are required to document that they have reviewed and understand them.

The ultimate goal of the environmental monitoring and analysis QA/QC program is to ensure that results are representative and defensible, and that data are of the type and quality needed to verify protection of the public, employees, and the environment. Figure 9-1 depicts the flow of the QA/QC elements of BNL's Environmental Monitoring Program and indicates the sections of this chapter that discuss each element in more detail.

Laboratory environmental personnel determine sampling requirements using the EPA Data Quality Objective (DQO) process (EPA 2000) or its equivalent. During this process, the project manager for each environmental program determines the type, amount, and quality of data needed to support decision making, the legal requirements, and stakeholder concerns. An environmental monitoring plan or project-specific sampling plan is then prepared, specifying the location, frequency, type of sample, analytical

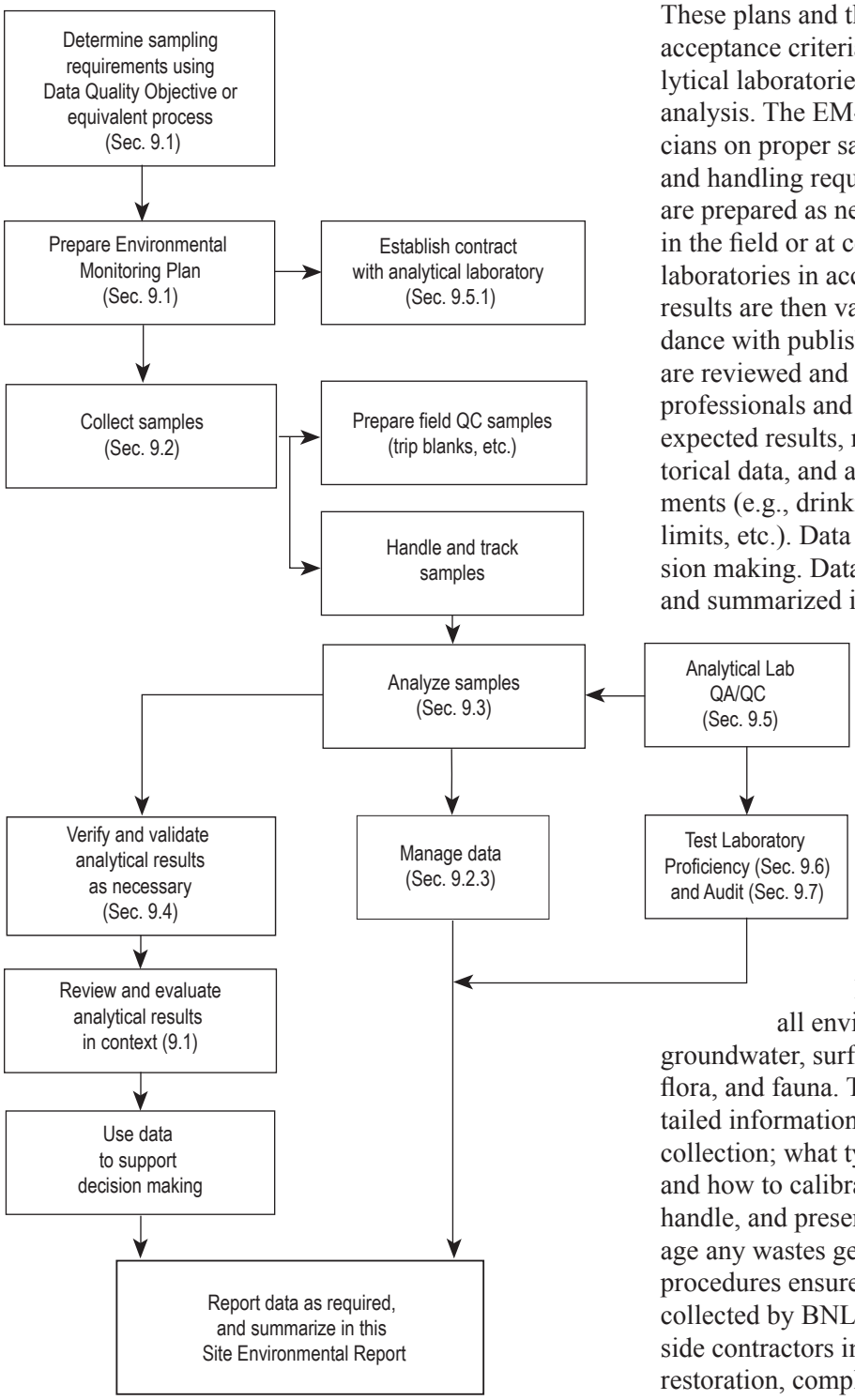


Figure 9-1. Flow of Environmental Monitoring QA/QC Program Elements.

methods to be used, and a sampling schedule. These plans and the EM-SOPs also specify data acceptance criteria. Contracts with off-site analytical laboratories are established for sampling analysis. The EM-SOPs direct sampling technicians on proper sample collection, preservation, and handling requirements. Field QC samples are prepared as necessary. Samples are analyzed in the field or at certified contract analytical laboratories in accordance with EM-SOPs. The results are then validated or verified in accordance with published procedures. Finally, data are reviewed and evaluated by environmental professionals and management in the context of expected results, related monitoring results, historical data, and applicable regulatory requirements (e.g., drinking water standards, permit limits, etc.). Data are then used to support decision making. Data are also reported as required and summarized in this annual report.

9.2 SAMPLE COLLECTION AND HANDLING

In 2008, environmental monitoring samples were collected as specified by EM-SOPs, the BNL Environmental Monitoring Plan Update (BNL 2008), and project-specific work plans, as applicable. BNL has sampling SOPs for all environmental media, including groundwater, surface water, soil, sediment, air, flora, and fauna. These procedures contain detailed information on how to prepare for sample collection; what type of field equipment to use and how to calibrate it; how to properly collect, handle, and preserve samples; and how to manage any wastes generated during sampling. The procedures ensure consistency between samples collected by BNL sampling personnel and outside contractors in support of the environmental restoration, compliance, and surveillance programs.

QC checks of sampling processes include the collection of field duplicates, matrix spike samples, field blanks, trip blanks, and equipment blanks. For example, field readings of water

quality parameters are taken until all parameters are within acceptable limits. Also, specific sampling methodologies include QC checks. An example of this is the low-flow groundwater sampling technique, which includes checks to ensure that monitoring wells are properly purged before readings are taken.

All wastes generated during sampling (contaminated equipment, purge water from wells, etc.) are managed in accordance with applicable requirements. A factor considered during sample collection is minimizing the amount of waste generated, consistent with the Pollution Prevention Program described in Chapter 2.

9.2.1 Field Sample Handling

To ensure the integrity of samples, chain-of-custody (COC) was maintained and documented for all samples collected in 2008. A sample is considered to be in the custody of a person if any of the following rules of custody are met: 1) the person has physical possession of the sample, 2) the sample remains in view of the person after being in possession, 3) the sample is placed in a secure location by the custody holder, or 4) the sample is in a designated secure area. These procedures are outlined in EM-SOP 109, "Chain-of-Custody, Storage, Packaging, and Shipment of Samples" (BNL 2008). All environmental monitoring samples in 2008 maintained a valid COC from the time of sample collection through sample disposal by the contract analytical laboratories.

9.2.1.1 Custody and Documentation

Field sampling technicians are responsible for the care and custody of samples until they are transferred to a receiving group or contract analytical laboratory. Samples requiring refrigeration are placed immediately into a refrigerator or a cooler with cooling media, and kept under custody rules. The technician signs the COC form when relinquishing custody, and contract analytical laboratory personnel sign the COC form when accepting custody.

As required by EM-SOP-201, "Documentation of Field Activities" (BNL 2007a), the field sampling technician is also required to maintain a bound, weatherproof field logbook, which is used to record sample ID number, collection time, description, collection method, and COC number. Daily weather conditions, field measurements, and other appropriate site-specific observations also are recorded in the logbook.

9.2.1.2 Preservation and Shipment

Before sample collection, the field sampling technicians prepare all bottle labels and affix them to the appropriate containers, as defined in the QA program plan or applicable EM-SOPs. Appropriate preservatives are added to the containers before or immediately after collection; in appropriate cases, samples are refrigerated. For example, samples collected for methyl mercury are cooled immediately and shipped to the contract analytical laboratory on the day of collection. After samples arrive at the laboratory, they are preserved with hydrochloric acid.

Sample preservation is maintained as required throughout shipping. If samples are sent via commercial carrier, a bill-of-lading is used. COC seals are placed on the shipping containers; their intact status upon receipt indicates that custody was maintained during shipment. These procedures are outlined in EM-SOP 109.

9.2.2 Field Quality Control Samples

Field QC samples collected for the environmental monitoring program include equipment blanks, trip blanks, field blanks, field duplicate samples, and matrix spike/matrix spike duplicate samples. The rationale for selecting specific field QC samples, and minimum requirements for their use in the environmental monitoring program, are provided in the BNL EM-SOP 200 series. Equipment blanks and trip blanks (see below) were collected for all appropriate media in 2008.

An ***equipment blank*** is a volume of solution (in this case, laboratory-grade water) that is used to rinse a sampling tool after decontamination. The rinse water is collected and tested to verify that the sampling tool is not contaminated. Equipment blank samples are collected, as needed, to verify the effectiveness of the decontamination procedures on nondedicated or reusable sampling equipment.

A ***trip blank*** is provided with each shipping container of samples to be analyzed for volatile organic compounds (VOCs). The use of trip blanks provides a way to determine whether contamination of a sample occurred during shipment from the manufacturer, while in bottle storage, in shipment to a contract analytical laboratory, or during analysis at a lab. Trip blanks consist of an aliquot of laboratory-grade water sealed in a sample bottle, usually prepared by the contract analytical laboratory prior to shipping the sample bottles to BNL. If trip blanks were not provided by the lab, then field sampling technicians prepare trip blanks before they collect the samples. Trip blanks were included with all shipments of aqueous samples for VOC analysis in 2008.

Field blanks are collected to check for cross-contamination that may occur during sample collection. For the Groundwater Monitoring Program, one field blank is collected for every 20 samples, or one per sampling round, whichever is more frequent. Field blanks are analyzed for the same parameters as groundwater samples. For other programs, the frequency of field blank collection is based on their specific DQOs.

In 2008 (as in other years), the most common contaminants detected in the trip, field, and equipment blanks included methylene chloride, methyl chloride, toluene, and chloroform. These compounds are commonly detected in blanks and do not pose significant problems with the reliability of the analytical results. Several other compounds were also detected, such as acetone and strontium-90 (Sr-90), at low levels. When these contaminants are detected, validation or verification procedures are used, where applicable, to qualify the associated data as "nondetects," (see Section 9.4). The results from blank samples collected during 2008 did not indicate any significant impact on the quality of the results.

Field duplicate samples are analyzed to check the reproducibility of sampling and analytical results, based on EPA Region II guidelines (EPA 2006). For example, in the Groundwater Monitoring Program, duplicates are collected for 5 percent of the total number of samples collected for a project per sampling round. During 2008, 42 duplicate samples were collected for nonradiological analyses, and 66 duplicate samples were collected for radiological analyses. All duplicate samples were acceptable for input into BNL's Environmental Information Management System (EIMS) database, which is used to manage the Laboratory's environmental data. Duplicates were analyzed only for the parameters relevant to the program they monitored. Of the 4,160 nonradiological parameters analyzed in 2008, 99 percent of the analyses met QA criteria. Of the 288 radiological parameters monitored, 99 percent met QA criteria.

Matrix spike and ***matrix spike duplicates*** are performed to determine whether the sample matrix (e.g., water, soil, air, vegetation, bone, or oil) adversely affected the sample analysis. A *spike* is a known amount of analyte added to a sample. Matrix spikes are performed at a rate specified by each environmental program's DQOs. The rate is typically one per 20 samples collected per project. No significant matrix effects were observed in 2008 for routine matrices such as water and soil. Nonroutine matrices, such as oil, exhibited the expected matrix issues.

9.2.3 Tracking and Data Management

Most environmental monitoring samples and analytical results were tracked in the EIMS. The small number of environmental samples that were not tracked in the EIMS were from Chemtex Lab, which cannot produce the electronic data deliverables needed to enter the data into BNL's EIMS. Tracking was initiated when a sample was recorded on a COC form. Copies of the COC form and supplemental forms were provided to the project manager or the sample coordinator and forwarded to the data coordinator to be entered into the EIMS. Each contract analytical laboratory also maintained its own internal sample tracking system.

Following sample analysis, the contract analytical laboratory provides the results to the project manager or designee and, when applicable, to the validation subcontractor, in accordance with their contract. Once results of the analyses are entered into the EIMS, reports can be generated

by project personnel and DOE Brookhaven Site Office staff using a web-based data query tool.

9.3 SAMPLE ANALYSIS

In 2008, environmental samples were analyzed by one of five contract laboratories, whose selection is discussed in Section 9.3.1. All samples were analyzed according to EPA-approved methods, where such methods exist, and by standard industry methods where there are no EPA methods. In addition, field sampling technicians performed field monitoring for parameters such as conductivity, dissolved oxygen, pH, temperature, and turbidity.

9.3.1 Qualifications

BNL used the following contract analytical laboratories for analysis of environmental samples in 2008:

- General Engineering Lab (GEL) in Charleston, South Carolina, for radiological and nonradiological analytes
- H2M Lab in Melville, New York, for nonradiological analytes
- Test America (TA), formerly Severn-Trent Lab, based in St. Louis, Missouri, for radiological and nonradiological analytes
- Chemtex Lab in Port Arthur, Texas, for select nonradiological analytes
- Brooks Rand in Seattle, Washington, for mercury and methylmercury analyses

The process of selecting off-site contract analytical laboratories involves a number of factors: 1) their record on performance evaluation (PE) tests, 2) their contract with the DOE Integrated Contract Procurement Team, 3) pre-selection bidding, and 4) their adherence to their own QA/QC programs, which must be documented and provided to BNL. Routine QC procedures that laboratories must follow, as discussed in Section 9.5, include daily instrument calibrations, efficiency and background checks, and standard tests for precision and accuracy. All the laboratories contracted by BNL in 2008 were certified by the New York State Department of Health (NYSDOH) for the relevant analytes, where such certification existed. The laboratories also were subject to PE testing and DOE-sponsored audits (see Section 9.7).

9.4 VERIFICATION AND VALIDATION OF ANALYTICAL RESULTS

Environmental monitoring data are subject to data verification and, in certain cases, data validation, when the data quality objectives of the project require this step. For example, groundwater samples collected for the Long Term Remedial Action (LTRA) group undergo data verification, whereas specific data collected for specific waste streams undergo full validation.

The data ***verification*** process involves checking for common errors associated with analytical data. The following criteria can cause data to be rejected during the data verification process:

- *Holding time missed* – The analysis is not initiated or the sample is not extracted within the time frame required by EPA or by the contract.
- *Incorrect test method* – The analysis is not performed according to a method required by the contract.
- *Poor recovery* – The compounds or radioisotopes added to the sample before laboratory processing are not recovered at the recovery ratio required by the contract.
- *Insufficient QA/QC data* – Supporting data received from the contract analytical laboratory are insufficient to allow validation of results.
- *Incorrect minimum detection limit (MDL)* – The contract analytical laboratory reports extremely low levels of analytes as "less than minimum detectable," but the contractually required limit is not used.
- *Invalid chain-of-custody* – There is a failure to maintain proper custody of samples, as documented on COC forms.
- *Instrument failure* – The instrument does not perform correctly.
- *Preservation requirements not met* – The requirements identified by the specific analytical method are not met or properly documented.
- *Contamination of samples from outside sources* – These possible sources include sampling equipment, personnel, and the contract analytical laboratory.

- *Matrix interference* – Analysis is affected by dissolved inorganic/organic materials in the matrix.

Data ***validation*** involves a more extensive process than data verification. Validation includes all the verification checks, as well as checks for less common errors, including instrument calibration that was not conducted as required, internal analyte standard errors, transcription errors, and calculation errors. The amount of data checked varies, depending on the environmental media and on the DQOs for each project. Data for some projects, such as long-term groundwater monitoring, may require only verification. Data from some waste streams receive the more rigorous validation testing, performed on 20 to 100 percent of the analytical results. The results of the verification or validation process are entered into the EIMS.

9.4.1 Checking Results

Nonradiological data analyzed in 2008 were verified and/or validated, when project DQOs required, using BNL EM-SOPs in the 200 Series and EPA contract laboratory program guidelines (EPA 1992, 2006). Radiological packages were verified and validated using BNL and DOE guidance documents (BNL 2002, DOE 1994). During 2008, the verifications were conducted using a combination of manually checking the hard copy data packages and the use of a computer program developed at BNL to verify that the information reported electronically is stored in the EIMS.

9.5 CONTRACT ANALYTICAL LABORATORY QA/QC

In 2008, procedures for calibrating instruments, analyzing samples, and assessing QC were consistent with EPA methodology. QC checks performed included: analyzing blanks and instrument background; using Amersham Radiopharmaceutical Company or National Institute for Standards and Technology (NIST) traceable standards; and analyzing reference standards, spiked samples, and duplicate samples. Analytical laboratory contracts specify analytes, methods, required detection limits, and deliverables—which include standard batch QA/QC performance checks. As part of the laboratory selection process, candidate laboratories are required to provide BNL with copies of their QA/QC manuals and QA program plans.

When discrepancies were found in field sampling designs, documented procedures, COC forms, data analyses, data processing systems, and QA software, or when failures in PE testing occurred, nonconformance reports were generated. Following investigation into the root causes, corrective actions were taken and tracked to closure.

9.6 PERFORMANCE OR PROFICIENCY EVALUATIONS

Four of the contract analytical laboratories (GEL, TA, H2M, and Brooks Rand) participated in several national and state PE testing programs in 2008. The fifth contractor, Chemtex Laboratory, did not participate in PE testing because there is no testing program for the specific analytes Chemtex analyzed: tolytriazole, polypropylene glycol monobutyl ether, and 1,1-hydroxyethylidene diphosphonic acid. Each of the participating laboratories took part in at least one testing program, and several laboratories participated in multiple programs. Results of the tests provide information on the quality of a laboratory's analytical capabilities. The testing was conducted by Environmental Resource Associates (ERA), the National Voluntary Laboratory Accreditation Program (NVLAP), the voluntary Mixed Analyte Performance Evaluation Program (MAPEP), and NYSDOH Environmental Laboratory Accreditation Program (ELAP). The results from these tests are summarized in Section 9.6.1. Because Brooks Rand only analyzed samples for mercury and methylmercury, their PE results are not summarized. Brooks Rand maintained the required certification when performing analyses for BNL in 2008.

9.6.1 Summary of Test Results

In Figures 9-2 and 9-3, results are plotted as percentage scores that were "Acceptable," "Warning (But Acceptable)," or "Not Acceptable." A Warning (But Acceptable) is considered by the testing organization to be "satisfactory." An "average overall satisfactory" score is the

sum of results rated as Acceptable and those rated as Warning (But Acceptable), divided by the total number of results reported. A Not Acceptable rating reflects a result that is greater than three standard deviations from the known value—a criterion set by the independent testing organizations.

Figure 9-2 summarizes radiological performance scores in the ERA and MAPEP programs. As in 2007, during 2008 the New York State ELAP did not provide radiological samples for PE testing, so there were no ELAP scores. GEL and TA had average overall satisfactory scores of 85 and 95 percent, respectively. More details about the radiological assessments are in Section 9.6.1.1.

Figure 9-3 summarizes the nonradiological performance results of the three participating laboratories (GEL, H2M, and TA) in the ERA, MAPEP, and ELAP tests. For nonradiological tests, the average overall satisfactory results ranged from 92 to 100 percent. Additional details on nonradiological evaluations are in Section 9.6.1.2.

9.6.1.1 Radiological Assessments

Both GEL and TA participated in the ERA radiological PE studies. Of GEL's tests on radiological samples, 72.5 percent were in the Acceptable range and 94.1 percent were Acceptable**.**

GEL and TA also participated in the MAPEP evaluations: 90.6 percent of GEL's tests on radiological samples were in the Acceptable range and 6.6 percent were in the Warning (But Acceptable) range. Of TA's MAPEP tests on radiological samples, 92.3 percent were in the Acceptable range and 3.8 percent were in the Warning (But Acceptable) range.

9.6.1.2 Nonradiological Assessments

In 2008, H2M and GEL participated in the NYSDOH ELAP evaluations of performance on tests of nonpotable water, potable water, and solid wastes. NYSDOH found 97.9 percent of H2M's nonradiological tests to be in the Acceptable range and 90.1 percent of GEL's nonradiological tests to be in the Acceptable range. TA, which is certified through the National Environmental Laboratory Accreditation Conference (NELAC), was not required to participate in ELAP evaluations.

H2M, TA, and GEL voluntarily participated in the ERA water supply and water pollution studies, although this evaluation is not required for New York State certification. ERA found that 100 percent of H2M's tests were in the Acceptable range and 98.5 percent of TA's tests were in the Acceptable range, as were 94.4 percent of GEL's tests**.**

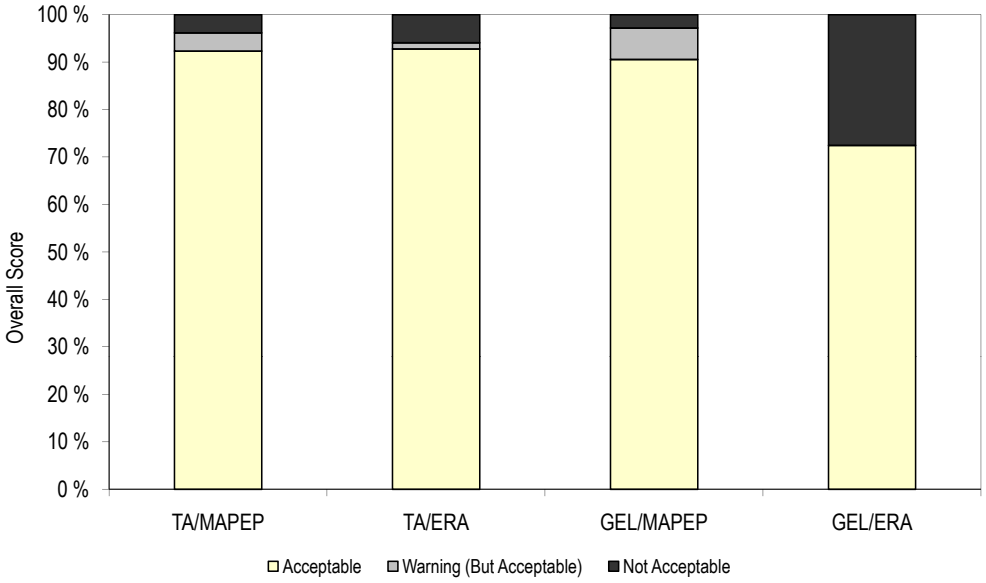
TA and GEL also voluntarily participated in MAPEP nonradiological evaluations, which showed that 96.5 percent of TA's tests were in the Acceptable range and 95.94 percent of GEL's were in the Acceptable range.

9.7 AUDITS

As part of DOE's Integrated Contract Procurement Team Program, TA and GEL were audited during 2008 (DOE 2008a, b). During the audits, errors are categorized into Priority I and Priority II findings. Priority I status indicates a problem that can result in unusable data or a finding that the contract analytical laboratory cannot adequately perform services for DOE. Priority II status indicates problems that do not result in unusable data and do not indicate that the contract analytical laboratory cannot adequately perform services for DOE (DOE 2002). There were no Priority I findings for GEL. TA had one Priority I finding and one Priority II finding.

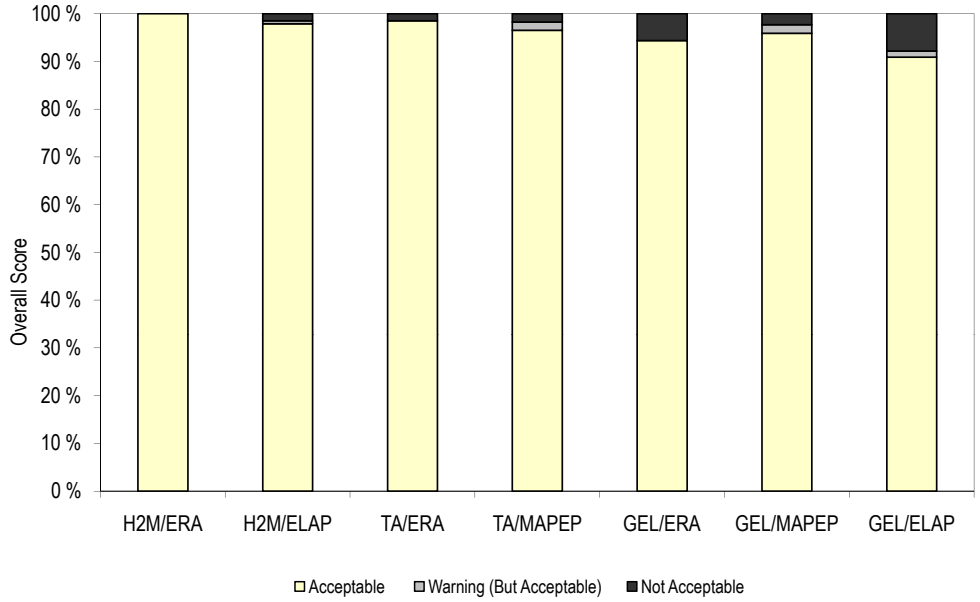
The results of the TA audit included one Priority I finding from their inorganic division and one Priority II finding, also from their inorganic division. The Priority I finding originated from a failure to obtain acceptable PE test results for antimony in a soil matrix. After the audit findings were issued, TA adjusted their soil preparation methods and received passing scores on the next two PE samples for antimony in a soil matrix. DOE accepted these results and closed this finding on August 28, 2008 (DOE 2008c). The Priority II finding dealt with inorganic lab practices that did not exactly meet internal SOPs. TA updated their SOPs, and this finding was also closed on August 28, 2008.

The results of the GEL audit included two Priority II findings. The first Priority II finding



Note that the Acceptable scores and the Warning (But Acceptable) scores combined constitute the "overall satisfactory" category referred to in the text of this chapter.

Figure 9-2. Summary of Scores in the Radiological Proficiency Evaluation Programs.



Note that the Acceptable scores and the Warning (But Acceptable) scores combined constitute the "overall satisfactory" category referred to in the text of this chapter.

Figure 9-3. Summary of Scores in the Nonradiological Proficiency Evaluation Programs.

stated that the GEL SOP for semivolatile analysis did not contain sufficient information on method blanks. The lab's corrective action for this finding was to update the SOP. The second Priority II finding indicated a failure to pass the PE sample for selenium in a soil matrix. The manufacturers of the PE sample were contacted, and due to the difficulty of analysis of this ana-

lyte, it was determined that this finding would be a Priority II, rather than a Priority I finding. Since this PE result, GEL has changed analysis techniques for selenium in soil and has obtained acceptable results on PE samples. DOE has accepted these corrective actions and both these findings were closed on August 6, 2008 (DOE, 2008d).

Based on the audits, the analytical data met DOE's criteria for Acceptable status.

9.8 CONCLUSION

Based on the data validations, data verifications, and results of the independent Performance Evaluation assessments, the chemical and radiological results reported in this 2008 Site Environmental Report are of acceptable quality.

REFERENCES AND BIBLIOGRAPHY

10 CFR 830 Subpart A. U.S. Department of Energy. *Quality Assurance Requirements.* U.S. Code of Federal Regulations. 2000.

BNL. 2006. EM-SOP 209. "Radiochemical Data Validation." Brookhaven National Laboratory, Upton, NY.

BNL 2007a. EM-SOP-201 "Documentation of Field Activities." Brookhaven National Laboratory, Upton, NY.

BNL. 2008. *Brookhaven National Laboratory Environmental Monitoring Plan Update January 2008.* BNL-52676-2006. Brookhaven National Laboratory, Upton, NY.

BNL. 2008. EM-SOP 109. "Chain-of-Custody, Storage, Packaging, and Shipment of Samples." Brookhaven National Laboratory. Upton, NY.

DOE. 1994. *RD-4 Guidance for Radiochemical Data Validation.* Draft. Report EM-73. U.S. Department of Energy, Washington, DC.

DOE. 2008a. *Continuing Qualification Audit of Severn Trent Laboratories, Inc.* Earth City, Mo. June 16, 2008.

DOE. 2008b. *Continuing Qualification Audit of General Engineering Laboratories, Inc.* Charleston, South Carolina. June 25, 2008.

DOE 2008c. Corrective Action Plan Acceptance for Audit ID: 080424-TAS, August 28, 2008.

DOE 2008d. Corrective Action Plan Acceptance for Audit ID: 080508-GEL, August 6, 2008.

DOE. Order 450.1A. 2008. *Environmental Protection Program.* U.S. Department of Energy. Washington, DC. 1-15-03.

DOE. Order 414.1A. 2001. *Quality Assurance.* U.S. Department of Energy. Washington, DC. November 24, 1998.

DOE. 2002. *Environmental Management Consolidated Audit Program EMCAP Procedure Number 1.0 Revision 2.* October 1, 2002.

EPA. 1992. *Evaluation of Metals Data for the Contract Laboratory Program.* EPA Region-II SOP HW-2. U.S. Environmental Protection Agency, Washington, DC.

EPA. 1998. *EPA Requirements for Quality Assurance Project Plans for Environmental Data Operations.* Report QA/R-5. U.S. Environmental Protection Agency, Washington, DC.

EPA. 2000. "Guidance for the Data Quality Objectives Process (QA/G-4)." U.S. Environmental Protection Agency, Washington, DC.

EPA. 2006. *CLP Organic Data Review and Preliminary Review.* EPA Region-II SOP HW-6. U.S. Environmental Protection Agency, Washington, DC. Revision 14, September 2006.

Intentionally Left Blank