Quality Assurance 9

Quality assurance is an integral part of every activity at Brookhaven National Laboratory. 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. The 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 2016 Site Environmental Report are reliable and of high quality.

9.1 QUALITY PROGRAM ELEMENTS

As required by DOE Order 458.1, Radiation Protection of the Public and Environment, and DOE Order 436.1, Departmental Sustainability, 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 414.1D, 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 operations in a reliable and effective manner to minimize any impact on the environment, safety, security, and health of the staff and public
- Standardize processes and support continual improvement
- Enable the delivery of products and services that meet customers' requirements and expectations
- Support an environment that facilitates scientific and operational excellence

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 2006) 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

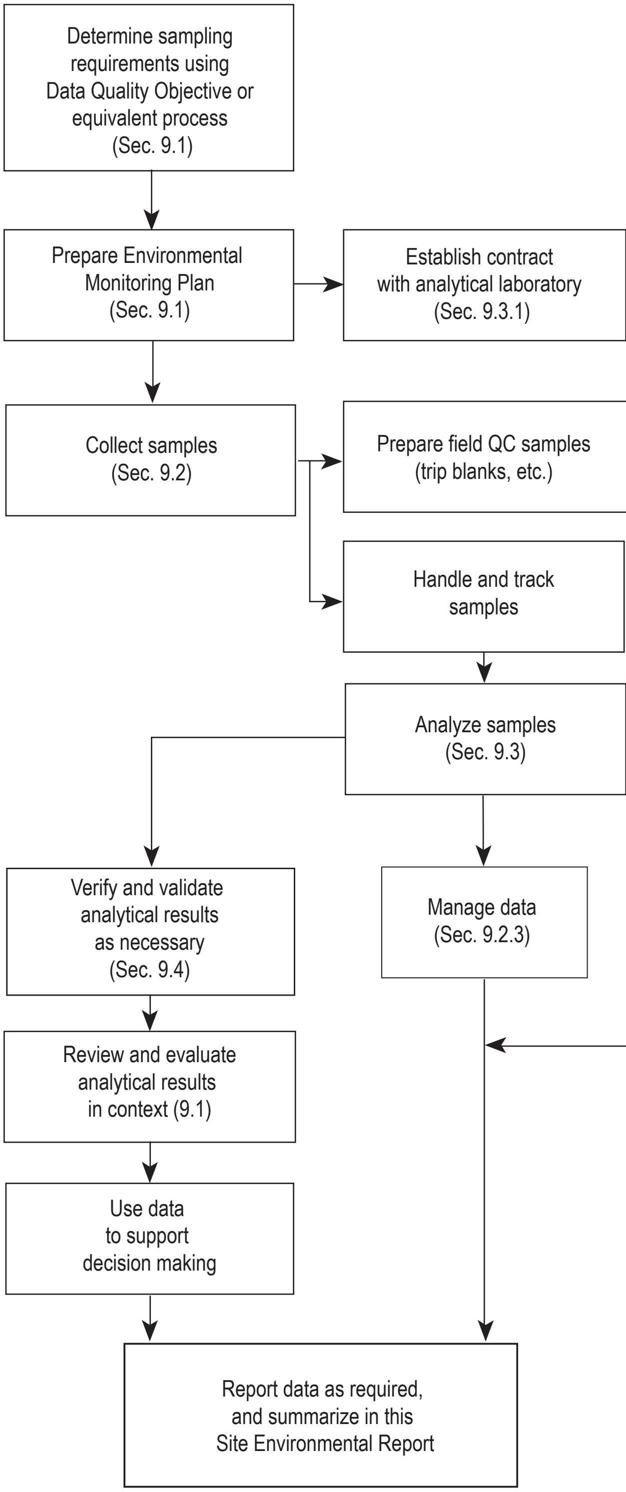


Figure 9-1. Flow of Environmental Monitoring QA/QC Program Elements.

sampling plan is then prepared, specifying the location, frequency, type of sample, analytical 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 and analyzed in the field or at a certified contract analytical laboratory. 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.). The data are used to support decision making, reported as required by regulatory permits or agreements, and summarized in this annual report.

9.2 SAMPLE COLLECTION AND HANDLING

In 2016, environmental monitoring samples were collected, as specified, by EM-SOPs, the BNL Environmental Monitoring Plan Update (BNL 2016), and project-specific work plans. 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; equipment calibration; how to properly collect, handle, and preserve samples; and how to manage any wastes generated during sampling. These procedures also ensure consistency between samples collected by Laboratory sampling personnel and contractors used to support 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.

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 2016. A sample is considered to be in the custody of a person if any or all 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 2015). All environmental monitoring samples collected in 2016 maintained a valid COC from the time of sample collection through sample disposal by the contract analytical laboratories used by BNL.

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 are 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 2012a), field sampling technicians are also required to maintain bound, weatherproof field logbooks, which are used to record sample ID numbers, collection times, descriptions, collection methods, and COC numbers. Daily weather conditions, field measurements, and other appropriate site-specific observations also are recorded in the logbooks.

9.2.1.2 Preservation and Shipment

Before sample collection, 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, and samples are refrigerated as necessary. For example, samples collected for methyl mercury are cooled immediately and shipped to a contract analytical laboratory on the day of collection. After samples arrive at the contract analytical 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 and their intact status upon receipt indicates that custody was maintained during shipment. These procedures are outlined in EM-SOP 109, "Chain-of-Custody, Storage, Packaging, and Shipment of Samples" (BNL 2015).

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, "Quality Assurance" procedures. Equipment blanks and trip blanks were collected for all appropriate media in 2016.

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 the effectiveness of the decontamination procedures on non-dedicated 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 container occurred during shipment from the manufacturer, while the container was in storage, during shipment to a contract analytical laboratory, or during analysis of a sample at a contract analytical laboratory. 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 are not provided by the contract analytical laboratory, 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 2016.

Field blanks are collected to check for cross-contamination that may occur during sample collection. A field blank consists of an aliquot of laboratory-grade water that is poured into a sample container in the field. 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 2016 (as in other years), the most common contaminants detected in the trip, field, and equipment blanks included trace to low levels of chloroform, and methylene chloride. 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 methylene chloride and acetone, 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 2016 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 2012, 2013). 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 2016, a total of 58 duplicate samples were collected for non-radiological analyses and 62 duplicates were collected for radiologic analyses. Not all parameters were analyzed in every duplicate. The parameters in each duplicate were consistent with those required for the specific program the duplicate was monitoring. Of the 3,218 parameters analyzed, only 21 (0.7 percent) of the non-radiologic analyses failed to meet QA criteria. For the radiologic parameters, only 9 of the 349 parameters (2.6 percent) failed to meet QA criteria. The results are indicative of consistency with the contract analytical laboratories and sampling methods, resulting in valid, reproducible data.

Matrix spike and matrix spike duplicates are used 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 2016 for routine matrices such as water and soil. Non-routine 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 BNL's Environmental Information Management System (EIMS), a database system used to store, manage, verify, protect, retrieve, and archive BNL's environmental data. A small number of environmental samples that were not tracked in the EIMS were analyzed at a contract analytical laboratory; Chemtex Lab cannot produce the electronic data deliverables needed to enter the data into the EIMS. Tracking is initiated when a sample is recorded on a COC form. Copies of the COC forms and supplemental forms are 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 maintains 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. 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 2016, environmental samples were analyzed by five contract analytical laboratories, whose selection is discussed in Section 9.3.1. All samples were analyzed according to EPA-approved methods or by standard industry methods, where no EPA methods are available. 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 2016:

- American Radiation Services (ARS) in Port Allen, Louisiana, for radiological analytes
- Chemtex Lab in Port Arthur, Texas, for select nonradiological analytes
- General Engineering Lab (GEL) in Charleston, South Carolina, for radiological and nonradiological analytes
- PACE Lab in Melville, New York, for nonradiological analytes
- Test America (TA), based in St. Louis, Missouri, for radiological and nonradiological analytes

The process of selecting contract analytical laboratories involves the following 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 2016 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 undergo data verification, whereas analytical results for specific waste streams undergo a 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 – 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 2016 were verified and/or validated when project DQOs required using BNL EM-SOPs and EPA contract laboratory program guidelines (EPA 2012, EPA 2013). Radiological packages were verified and validated using BNL and DOE guidance documents (BNL 2012b). During 2016, the verifications were conducted using a combination of manually checking hard copy data packages and the use of a computer program developed at the Laboratory to verify that the information reported electronically is stored in the EIMS.

9.5 CONTRACT ANALYTICAL LABORATORY QA/QC

In 2016, 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 occur, nonconformance reports are generated. Following investigation into the root causes, corrective actions are taken and tracked to closure.

9.6 PERFORMANCE OR PROFICIENCY EVALUATIONS

Four of the contract analytical laboratories (ARS, GEL, PACE, and TA) participated in several national and state PE testing programs in 2016. Chemtex Lab did not participate in PE testing because there is no testing program for the specific analytes Chemtex analyzed for BNL (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 DOE required Mixed Analyte Performance Evaluation Program (MAPEP), Resource Technology Corporation (RTC), Phenova, and the NYSDOH Environmental Laboratory Accreditation Program (ELAP). The results from these tests are summarized in Section 9.6.1.

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, MAPEP, and ELAP programs. GEL, TA, and ARS had average overall satisfactory scores of 98, 95, and 83 percent, respectively. Additional details about the radiological assessments are discussed in Section 9.6.1.1.

Figure 9-3 summarizes the nonradiological performance results of three of the four participating laboratories (GEL, Pace, and TA) in the ERA, MAPEP, Phenova, and ELAP tests. For nonradiological tests, the average overall satisfactory results ranged from 97 to 100 percent for the three laboratories. Additional details on nonradiological evaluations are discussed in Section 9.6.1.2.

9.6.1.1 Radiological Assessments

ARS, GEL and TA participated in the ERA and MAPEP radiological PE studies. Of ARS's radiological test results, 83 percent were in the Acceptable range; of GEL's radiological test results, 98 percent were in the Acceptable range; and of TA's radiological test results, 95 percent were in the Acceptable range. TA participated in the ELAP evaluations; 90 percent of TA's ELAP tests on radiological samples were in the Acceptable range. The ELAP testing is based on a small sample group (19 tests), while the ERA and MAPEP studies use a much larger sample size (more than 100 tests per year).

9.6.1.2 Nonradiological Assessments

During 2016, PACE participated in the NYSDOH ELAP evaluations of performance on tests of nonpotable water, potable water, and solid wastes. NYSDOH found 98 percent of PACE's nonradiological tests to be in the Acceptable range. GEL participated in the ERA water supply and water pollution studies. ERA found that 96 percent of GEL's tests were in the Acceptable range. TA and GEL participated in the MAPEP water supply and water pollution studies. MAPEP found that 98 percent of TA's and GEL's results were in the Acceptable range. TA and GEL participated in the Phenova Soil/Hazardous Waste and Water Pollution proficiency testing programs. Phenova found that 96 percent of TA's results were in the Acceptable range, and 97 percent of GEL's results were in the Acceptable range. GEL also participated in RTC nonradiological evaluations; 100 percent of GEL's results were in the Acceptable range.

9.7 AUDITS

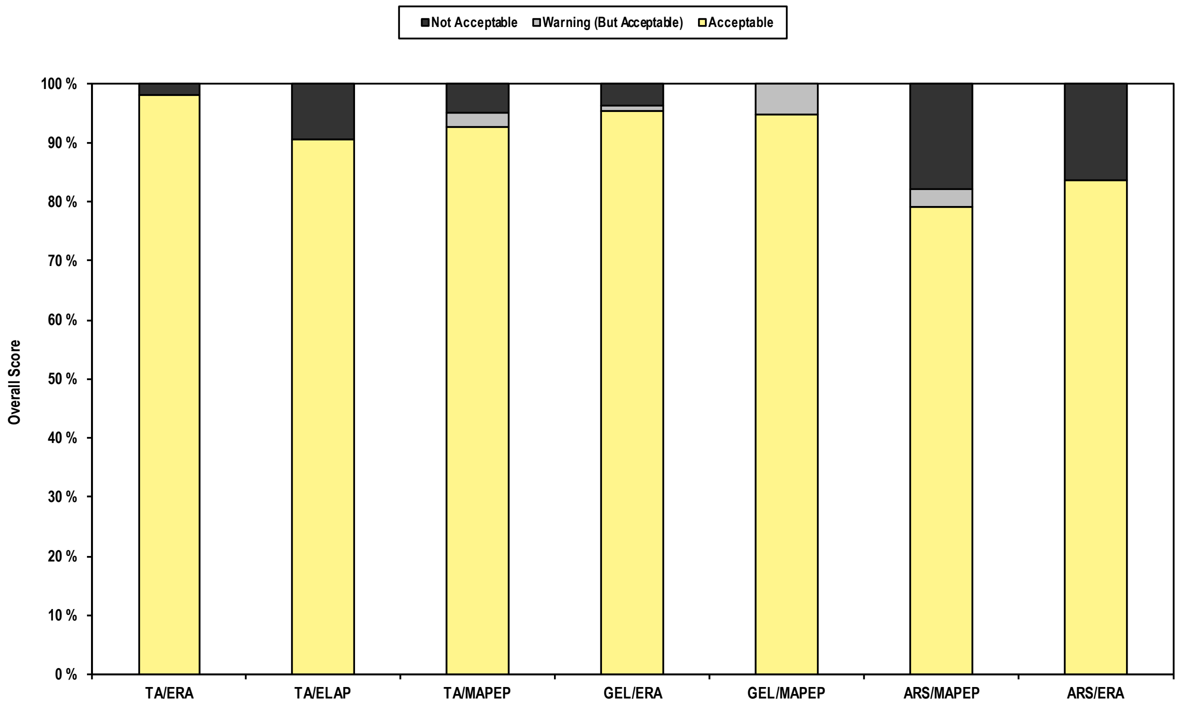
As part of DOE's Integrated Contract Procurement Team Program, TA, GEL, and ARS were audited during 2016 (DOE 2016a,b,c). During the audits, errors are categorized into Priority I and Priority II findings. Priority I status indicates a problem that can result in unusable data or a finding that the contract 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 during 2016 that affected samples analyzed for BNL.

The results of the TA audit included two Priority I findings and 17 Priority II findings. The Priority I findings were for failing consecutive rounds of proficiency testing for iron-55 and zinc-65 in a filter matrix. TA did not perform the analyses mentioned in the Priority I findings for BNL during 2016. The Priority II findings included: three in Quality Assurance Management Systems, five in the Data Quality for Organic Analyses, three in the Data Quality for Inorganic and Wet Chemistry Analyses, three in Data Quality for Radionuclide Analyses, and three in Hazardous and Radioactive Materials Management.

The results of the GEL audit included seven Priority II findings: two in the Quality Assurance Department, one in Data Quality for Organic Analyses, two in Data Quality for Inorganic and Wet Chemistry Analyses, one in Laboratory Information Management System (LIMS)/Electronic Data Management, and one in Hazardous and Radioactive Materials Management.

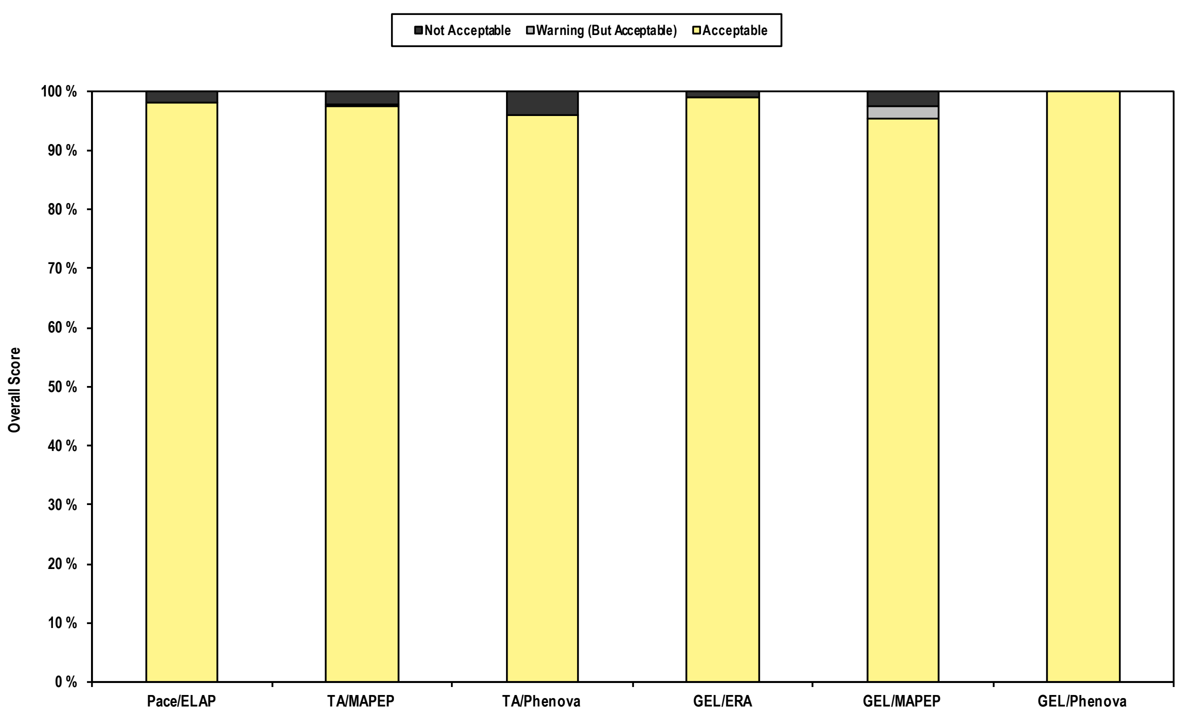
The results of the ARS audit included three Priority I findings and 17 Priority II findings. This is the first year ARS was audited for nonradiological parameters. ARS did not perform the analyses mentioned in the Priority I finding for BNL during 2016. The Priority I Findings were issued for misrepresenting the identity of a subcontracted laboratory and failures for consecutive rounds of proficiency testing for arsenic and selenium in a soil matrix. The Priority II findings included: four Priority II findings in the Quality Assurance Department, six findings in Data Quality for Organic Analyses, one finding in Data Quality for Inorganic and Wet Chemistry Analyses, three findings in Data Quality for Radiochemistry Analyses, and three in Hazardous and Radioactive Materials Management.

Based on the audits, the analytical laboratories met DOE and BNL criteria for Acceptable status.



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.

9.8 CONCLUSION

Based on the data validations, data verifications, and results of the independent PE assessments, the chemical and radiological results reported in this 2016 Site Environmental Report are of acceptable quality.

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