Chapter 8

Quality Assurance

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[*Editor's note: During 2006, responsibility for the environmental Quality Assurance (QA) program was divided among three groups—the Environmental Monitoring Laboratory (EML), the Environmental Permitting and Monitoring group (EPM), and the Geochemical Monitoring and Data Management and Waste Engineering group (GM&DMWE)*].

SRS's environmental QA program is conducted to verify the integrity of data generated by onsite and subcontracted offsite environmental laboratories.

The program's objectives are to ensure that samples are representative of the surrounding environment, and that analytical results are accurate.

QA for EPM Program Samples

Internal Quality Assurance Program

EPM has a documented QA program that meets SRS and U.S. Department of Energy (DOE) requirements. Based on periodic inspections, no significant QA issues or corrective actions were identified during 2006.

Laboratory Certification

EPM is certified by the South Carolina Department of Health and Environmental Control (SCDHEC) Office of Laboratory Certification for field pH and total residual chlorine measurements.

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Blind pH Samples

EPM personnel routinely conduct a blind sample program for field measurements of pH to assess the quality and reliability of field data measurements.

Quality Control Sample Definitions

Blank - A sample that has not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, storage, or analysis. The blank is subjected to the usual analytical and measurement process to establish a zero-baseline or -background value, and sometimes is used to adjust or correct routine analytical results.

Instrument Blank - A clean sample (e.g., distilled water) processed through the instrumental steps of the measurement process; used to determine instrument contamination.

Method Blank - A matrix sample—similar to the batch of associated samples (when available)—that is free from the analytes of interest and is processed simultaneously with—and under the same conditions as—samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses.

Reagent Blank - A sample containing reagent(s), without the target analyte or sample matrix, that is introduced into the analytical procedure at the appropriate point—and that is carried through all subsequent steps to determine the contribution of the reagents and of the involved analytical steps.

Blind Sample - A subsample for analysis with a composition known to the submitter. The analyst/laboratory may know the identity of the sample, but not its composition. It is used to test the analyst's or laboratory's proficiency in the execution of the measurement process.

Carrier - A stable isotope of a radionuclide (usually the analyte) added to increase the total amount of that element so that a measurable mass of the element is present.

Laboratory Control Sample (LCS) - A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes or a material containing known and verified amounts of analytes. It generally is used to establish intralaboratory or analyst-specific precision and bias, or to assess the performance of all or a portion of the measurement system.

Laboratory Duplicate - Aliquot of a sample taken from the same container under laboratory conditions and processed and analyzed independently.

Spike - A known mass of target analyte added to a blank sample or subsample; used to determine recovery efficiency, or for other QC purposes.

Tracer - A radioactive isotope that chemically mimics and does not interfere with the target analyte through radiochemical separations. Isotopic tracers typically are radioactive materials (e.g., U-232, Pu-242). Tracers are added to samples to determine the overall chemical yield for the analytical preparation steps.

During 2006, blind pH field measurements were taken for 24 samples. All field pH measurements were within the U.S. Environmental Protection Agency's (EPA's) suggested acceptable control limit of ± 0.4 pH units of the true (known) value.

QA for EML Sample Analyses

Internal QA Program

EML has a documented QA program that meets SRS and DOE requirements. Instruments are calibrated with known reference standards. Instrument performance is monitored through the use of checks and control charts. Analytical batch performance is measured through the use of quality control (QC) samples (blanks, spikes, carriers, tracers, laboratory control samples, and duplicates). QC results that fall outside of specified limits may result in analytical batch or sample reruns. If a batch or sample is not rerun, the reason is documented in the data package, which includes the QA cover sheet, instrument data printouts, and associated QC data.

Based on periodic inspections of instrument records and analytical data packages, no significant quality assurance issues or corrective actions were identified during 2006.

Laboratory Certification

EML is certified by the SCDHEC Office of Laboratory Certification for the measurement of following analytes:

- total suspended solids and 27 metals (under the Clean Water Act)
- 42 volatile organic compounds (VOCs) and 28 metals (under the Resource Conservation and Recovery Act)

Blind Tritium Samples

Blind tritium samples provide an assessment of laboratory sample preparation and counting. During 2006, eight blind samples were analyzed for tritium; all of the results were within control limits. Complete results (including control limits) can be found in the "Blind Sample Results for Tritium" table on the CD accompanying this report.

External QA Program

In 2006, EML participated in the DOE Mixed Analyte Performance Evaluation Program (MAPEP), an interlaboratory comparison program that tracks performance accuracy and tests the quality of environmental data reported to DOE. The Radiological and Environmental Sciences Laboratory (RESL), under the direction of DOE–Headquarters Environmental Safety and Health (ES&H), administers the MAPEP.

MAPEP samples include water, soil, air filter, and vegetation matrices with environmentally important stable inorganic, organic, and radioactive constituents.

In 2006, EML completed the analysis of 56 radioisotopes and 15 metals for MAPEP-15 (designation of a specific study set), and of 52 radioisotopes and 15 metals for MAPEP-16. The results show that the laboratory exceeded the expected 80-percent-acceptable-results level for each study set (table 8–1). The rating was calculated by dividing the acceptable and the acceptable-with-warning results by the total number of results.

MAPEP intercomparison study results for EML can be found in the data tables section of the CD accompanying this report.

QA for EPM Sample Analyses

Onsite and subcontract environmental laboratories providing analytical services must have documented QA programs and meet the quality requirements defined in the *WSRC Quality Assurance Manual* (WSRC 1Q).

An annual DOE Consolidated Audit Program (DOECAP) evaluation of each subcontract laboratory is performed to ensure that all the laboratories maintain technical competence and follow the required QA programs. Each evaluation includes an examination of

Table 8–1
EML Performance on Mixed Analyte Performance Evaluation Program (MAPEP)

Study Set	Matrix	EML[a]
MAPEP-06-GrF15	Air Filter	100%
MAPEP-06-GrW15	Water	100%
MAPEP-06-MaS15	Solid	100%
MAPEP-06-MaW15	Water	100%
MAPEP-06-RdF15	Air Filter	100%
MAPEP-06-MaV15	Vegetation	100%
MAPEP-06-GrF16	Air Filter	100%
MAPEP-06-GrW16	Water	100%
MAPEP-06-MaS16	Solid	92%[b]
MAPEP-06-MaW16	Water	100%
MAPEP-06-RdF16	Air Filter	100%
MAPEP-06-MaV16	Vegetation	100%

[a] Column presents the percentage of tests that exceeded the 80%-acceptable-results level.
[b] Results for Cs-134 were acceptable with warning (bias between 20% and 30%).

laboratory performance with regard to sample receipt, instrument calibration, analytical procedures, data verification, data reports, records management, nonconformance and corrective actions, and preventive maintenance. Reports of the findings and recommendations are provided to each laboratory, and follow-up evaluations are conducted as necessary. No DOECAP evaluation was conducted for onsite laboratories.

Nonradiological Liquid Effluents

National Pollutant Discharge Elimination System (NPDES) samples are analyzed by four onsite laboratories groups—EML, EPM, the Site Infrastructure & Services Department (I&SD), and Westinghouse Safety Management Solutions (WSMS)—and one offsite subcontract laboratory, Shealy Environmental Services (SES). All these laboratories must be certified by SCDHEC for NPDES analyses.

Interlaboratory Program

During 2006, all laboratories performing NPDES analyses for WSRC participated in the Environmental Resource Associates (ERA) Water Pollution (WP) performance evaluation studies for compliance with the EPA-required Discharge Monitoring Report–QA Study 26. ERA, as required by EPA, is accredited by the American Association of Laboratory Accreditation and the National Institute of Standards and Technology.

EPA and SCDHEC use the study results to certify laboratories for specific analyses. As part of the recertification process, these agencies require that laboratories investigate the unacceptable results and implement corrective actions as appropriate.

WSMS participated in the 2006 DMR–QA Study 26, while SES, EPM, EML, and I&SD participated in ERA's WP-137 and WP-138 studies.

With the exception of one parameter, all results were acceptable. The offsite laboratory received a "not acceptable" result for oil and grease in the WP-137 study. The cause of the failure was determined, and an acceptable result was obtained in the WP-138 study.

Intralaboratory Program

The environmental monitoring intralaboratory program reviews laboratory performance by analyzing duplicate and blind samples throughout the year.

The onsite and offsite laboratories processed 89 duplicate analyses during 2006. Zero-difference results were reported for 45 of these analyses. Eight of the 89 duplicate analyses exceeded the relative-percent difference (± 20-percent difference).

The onsite and offsite laboratories processed 74 blind analyses during 2006. Zero-difference results were reported for 45 of these analyses. Seven of the 74 blind analyses exceeded the relative percent difference (± 20-percent difference). Results for the duplicate and blind sampling programs showed no indications of consistent problems in the laboratories.

Stream and River Water Quality

SRS's water quality program requires checks of 10 percent of the samples to verify analytical results. Duplicate grab samples from SRS streams and the Savannah River were analyzed by SES and EML in 2006. SES and EML reported 564 analyses for this program. Most results were within acceptance limits (+ 20-percent difference). Approximately 10 percent of the 277 duplicate results evaluated fell outside the acceptance limits. Results for the duplicate sampling program showed no indications of consistent problems with the laboratories. Detailed stream and Savannah River duplicate sample results can be found in the data tables section of the CD accompanying this report.

QA for SGCP Sample Analyses

Groundwater analyses at SRS are performed by subcontract and onsite laboratories. During 2006, General Engineering and Severn Trent were the primary full-service subcontractors; Eberline Services Oak Ridge Lab (radiological only) and Lionville (nonradiological only) were used to a lesser extent; and MicroSeeps, Inc., performed special analyses. In addition to the subcontract laboratories, EML performed groundwater analyses on site.

During 2006, General Engineering Laboratories, Severn Trent Laboratories, Inc., and Lionville Laboratory, Inc., participated in various WP and WS studies. These laboratories are required by contract to participate in the WP and WS studies. The results for WP–130 through WP–141 and WS–114 through WS–123 (table 8–2) show that the laboratories met or exceeded the 80-percent-acceptable-results level. The table reflects only the studies in which the laboratories actually participated.

Table 8–2

Subcontract-Laboratory Performance Environmental Resource Associates (ERA) Water Pollution Studies

Study	General Engineering		Severn Trent		Lionville	
	Acceptable	*Fail*	*Acceptable*	*Fail*	*Acceptable*	*Fail*
WP-130	100%					
WP-132			95%	5%	96%	4%
WP-135	99%	1%				
WP-138	98%	2%	99%	1%	98%	2%
WP-139	100%					
WP-140	100%					
WP-141	100%					
WS-114	94%	6%				
WS-117	86%	14%	100%			
WS-120	92%	8%				
WS-121	100%					
WS-122	80%	20%				
WS-123	100%		92%	8%		

Acceptable = Reported value falls within acceptance limits
Fail = Reported value falls outside acceptance limits

Results from the laboratories are summarized in table 8–3. The results show that all but one laboratory exceeded the expected 80-percent-acceptable-results level for all studies for both the soil and groundwater matrices. Following receipt of the results, samples have not been sent to the laboratory that did not meet the expected 80-percent-acceptable-results level. Samples will not be sent to this laboratory until it provides a corrective action response. The air filter and vegetation matrices are not included in the subcontract laboratory performance summary because these matrices are not part of the SGCP program.

Soil/Sediment

Environmental investigations of soils and sediments, primarily for RCRA/CERCLA units, are performed by subcontract laboratories. Data are validated by SGCP according to EPA standards for analytical data quality, or as specified by SRS customers.

The environmental validation program is based in part on two EPA guidance documents, "*Guidance for the Data Quality Objectives Process for Superfund*" (EPA–540–R–93–071) and "*Data Quality Objectives Process for Waste Site Investigations*" (QA/G–4HW) (EPA–600/R–00–007). These documents identify QA issues to be addressed, but they do not formulate a procedure for data evaluation or provide pass/fail criteria to apply to data and document acceptance. Hence, the validation program contains elements from—and is influenced by—several other references, including

Table 8–3
Subcontract-Laboratory Performance on Mixed-Analyte Performance Evaluation Program (MAPEP)

Study	Matrix	General Engineering	Severn Trent	Eberline	SRS (EBL)	Lionville
MAPEP–06–MaS15	Water	98%[1,15,a,d]	89%[1,2,4,6,8,9,10,14,16,17,18]	100%	100%*	99%†
MAPEP–06–MaW15	Water	97%[3]	100%	100%*	100%	87%[(10),13,14†]
MAPEP–06–OrW15	Water	100%	97%[(19),(20)]	No Data	No Data	100%
MAPEP–06–GrW15	Solid	100%	100%	50%[12]	100%	No Data
MAPEP–06–MaS16	Water	99%[(7),b,c]	97%[2,(7),8]	94%[5]*	100%*	94%[3,(7),9,(10),(11)†]
MAPEP–06–MaW16	Water	100%	100%	100%*	100%	100%
MAPEP–06–OrW16	Water	100%	100%	No Data	No Data	100%
MAPEP–06–GrW16	Solid	100%	100%	100%	100%	No Data

[1] Results for strontium-90 were not acceptable.
[2] Results for antimony were not acceptable.
[3] Results for iron-55 were not acceptable.
[4] Results for chromium were not acceptable.
[5] Results for nickel-63 were not acceptable.
[6] Results for uranium-238 were not acceptable.
[7] Results for endosulfan II were not acceptable.
[8] Results for zinc were not acceptable.
[9] Results for uranium (total) were not acceptable.
[10] Results for beta (BHC) were not acceptable.
[11] Results for methoxychlor were not acceptable.
[12] Results for gross beta were not acceptable.
[13] Results for cadmium were not acceptable.
[14] Results for copper were not acceptable.
[15] Results for cesium-134 were not acceptable.
[16] Results for beryllium were not acceptable.
[17] Results for nickel were not acceptable.
[18] Results for vanadium were not acceptable.
[19] Results for 4-chloro-3-methylphenol were not acceptable.
[20] Results for 3-methyl and 4-methylphenol were not acceptable.

[a] Results for nickel-63 were acceptable with warning.
[b] Results for americium-241 were acceptable with warning.
[c] Results for uranium (total) were acceptable with warning.
[d] Results for uranium-235 were acceptable with warning.

* Only radiological analytes reported
† Only nonradiological analytes reported
() False positive

- "*Guidance on Environmental Data Verification and Data Validation*" (QA/G–8), EPA–240/R–02/004
- "*USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review*," EPA–540/R–99/008
- "*USEPA Contract Laboratory Program National Functional Guidelines for Chlorinated Dioxin/Furan Data Review*," EPA–540/R–05/001
- "USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review," EPA–540/R–04/004

- "*Test Methods for Evaluating Solid Waste*," EPA, November 1986, SW–846, Third Edition; Latest Update, July 2005
- "*DOE Quality Systems for Analytical Services*," Revision 2.2, October 2006

Many QA parameters are evaluated by automated processing of electronically reported data. Others are selectively evaluated by manual inspection of associated analytical records. A summary of findings is presented in each project narrative or validation report prepared by SGCP personnel.

Data Review

The QA program's detailed data review for groundwater and soil/sediment analyses is described in WSRC–3Q1–2, Section 1100.

In 2006, the major QA issues discovered and addressed in connection with these programs for soil/sediment and groundwater analyses included the following:

- Inadequate internal standardization for total uranium by ICP–MS at one laboratory
- Outdated interelement corrections for ICP–AES metals at two laboratories
- Calibrated region deviations for alpha spectroscopy analytes at one laboratory
- Calibration spreadsheet errors for strontium-90 and gross alpha/beta at one laboratory
- Calibration stability problems for isobutanol at one laboratory
- Uncertain identification for total dioxins due to combined standards and co-elution
- Liquid scintillation counting without standard quench correction at one laboratory

Previously identified items resolved in 2006 included the following:

- Cyanide analysis without primary distillation checking at one laboratory
- Nitrate-nitrite analysis without reduction checks at one laboratory

Previously identified items still being addressed include the following:

- Gas-flow proportional counting without complete cross-talk calibration at two laboratories (This issue is being resolved through a formal DOE resolution process.)
- Incomplete record packages for validation (ongoing)
- Omissions and logic failures in electronically reported data (ongoing)

These findings illustrate that, although laboratory procedures are well defined, analytical data quality does benefit from technical scrutiny. A corrective action plan has been put into place to address these issues, which are expected to be resolved during 2007.