Quality Assurance

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[*During 2007, responsibility for the environmental Quality Assurance (QA) program continued to be 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 analyses determined by onsite and subcontracted offsite environmental laboratories, and that quality control program requirements were met. 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 (Procedure Manual L3.25, "Environmental Monitoring Quality Assurance Procedures"). Based on data reviews, no QA issues or corrective actions were identified during 2007.

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.

Blind pH Samples

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

During 2007, at least two blind pH field measurements were taken monthly, for a total of 26 samples. All but one field pH measurement was within the U.S. Environmental Protection Agency's (EPA's) suggested acceptable control limit of ± 0.4 pH units of the true (known) value. That one value was just outside of the acceptable control limit range by 0.04 pH units. Blind pH sample results can be found in the data tables section of the CD accompanying this report.

QA for EML Sample Analyses

Internal QA Program

EML has a documented QA program (Procedure Manual L3.25, "Environmental Monitoring Quality Assurance Procedures") 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

Quality Control Sample Definitions

Blank - A sample that has not been exposed to the 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.

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 (see LCS) or subsample (a matrix spike); 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.

reason is documented in the data package, which includes the QA cover sheet, instrument data printouts, and associated QC data.

Based on inspections of instrument records and analytical data packages, no QA issues or corrective actions were identified during 2007.

Laboratory Certification

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

- total suspended solids and 27 metals by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP–AES) (under the Clean Water Act (CWA))
- 42 volatile organic compounds (VOCs) by gas chromatography/mass spectrometry and 28 metals by ICP–AES (under the Resource Conservation and Recovery Act (RCRA))

An ICP mass spectrometer (ICP–MS) was purchased in 2007, and certification was obtained from SCDHEC for 18 metals for both the CWA and RCRA ICP–MS methods.

Blind Tritium Samples

The blind tritium program was discontinued in 2007 because of the limited number of analyses used to support the program. Tritium data acceptability is demonstrated through batch quality control acceptance and Mixed Analyte Performance Evaluation Program (MAPEP) study results.

External QA Program

In 2007, EML participated in the DOE 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 2007, EML completed the analysis of 53 radioisotopes and 15 metals for MAPEP–17 (designation of a specific study set). Results show that

the laboratory passed the 80-percent-acceptable-results level for the study set (table 8–1). The percentage was calculated by dividing the acceptable and the acceptable-with-warning results by the total number of results, and multiplying the result (of that division) by 100.

MAPEP intercomparison study results for EML can be found in the data tables section of the CD accompanying this report. The MAPEP information has been copied from the actual MAPEP final report; "NR" in the report stands for "not reported," which indicates that the laboratory did not submit any data for that particular analysis. The Flag column is used to denote if a result is Acceptable (A), Not Acceptable (N), Warning (W), etc., and the Unc Flag column is used to note uncertainty values that may be High (H) or (L), etc.

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. One subcontract laboratory evaluation was conducted in 2007. The evaluation includes an examination of 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.

Nonradiological Liquid Effluents

National Pollutant Discharge Elimination System (NPDES) samples are analyzed by four onsite laboratory 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 are certified by SCDHEC for NPDES analyses.

Interlaboratory Program

During 2007, all laboratories performing NPDES analyses for WSRC participated in the EPA-required Discharge Monitoring Report–QA Study 27. All laboratories utilized Environmental Resource Associates (ERA) as the accredited Proficiency Testing provider. ERA, as required by EPA, is accredited by the American Association of Laboratory Accreditation.

EPA and SCDHEC use the study results to certify laboratories for specific analyses. As part of the recertification process, these agencies require that

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

Study Set	Matrix	EML[a]
MAPEP–07–GrF17	Air Filter	100%
MAPEP–07–GrW17	Water	100%
MAPEP–07–MaS17	Solid	100%
MAPEP–07–MaW17	Water	100%
MAPEP–07–RdF17	Air Filter	100%
MAPEP–07–MaV17	Vegetation	90%[b]

[a] Column presents percentage of tests that exceeded 80%-acceptable-results level
[b] Result for Zn-65 not acceptable (bias greater than 30%)

laboratories investigate the unacceptable results and implement corrective actions as appropriate.

WSMS participated in the 2007 DMR–QA Study 27, while SES, EP&M, EML, and I&SD participated in ERA's water proficiency (WP)–149, WP–150, and WP–151 studies. With the exception of one parameter, all the studies' results were acceptable. The offsite laboratory (SES) received a "not acceptable" result for ammonia in the WP–149 study; the cause of the failure was determined to be a reporting error. An acceptable ammonia result was obtained from SES in the WP–151 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 66 duplicate analyses during 2007. Zero-difference results were reported for 51 of these analyses. The remaining 15 duplicate analyses were between zero and ≤ 20-percent difference. Only four of the 66 duplicate analyses exceeded the relative-percent difference (≤ 20-percent difference).

The onsite and offsite laboratories processed 67 blind analyses during 2007. Zero-difference results were reported for 50 of these analyses. Only two of the 67 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 any of 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 2007. SES and EML reported 2,298 analyses for this program. Greater than 95 percent of the 1,129 duplicate results were within acceptance limits (≤ 20-percent difference). 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 2007, General Engineering Laboratories and TestAmerica, Inc. (formerly Severn Trent), were the primary full-service subcontractors; Eberline Services Oak Ridge Lab (radiological only) and Lionville Laboratory, Inc. (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 2007, General Engineering, TestAmerica, and Lionville participated in various WP and water supply (WS) studies. These laboratories are required by contract to participate in the WP and WS studies. The WP study results (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.

Results from the subcontract-laboratory performance on MAPEP are summarized in table 8–3. The results show that all laboratories exceeded the expected 80-percent-acceptable-results level for all studies for both the soil and groundwater matrices. The air filter and vegetation matrices are not included in the subcontract-laboratory performance summary because these matrices are not part of the Soil & Groundwater Closure Projects (SGCP) program. Table 8–4 is a summary of the MAPEP issues noted during 2007.

Soil/Sediment

Environmental investigations of soils and sediments, primarily for RCRA/Comprehensive Environmental Response, Compensation, and Liability Act 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 "*Systematic Planning: A Case Study for Hazardous Waste Site Investigations*" (QA/CS–1) (EPA/240/B–06/004). 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

Table 8–2
Subcontract-Laboratory Acceptable Performance

Study	General Engineering	TestAmerica	Lionville
WP–144		**96%** [55, 21, 46, 45, 40, 32, 43, 39, 29, 41, 22, 34, 56, 62]	**98%** [46,1,2,35,33]
WP–147	**99%** [57]		
WP–150		**96%** [26, 31, 25, 38, 49, 61, 52, 9, 15, 7, 11, 13, 44, 10, 14, 18, 47, 8, 12]	**98%** [20,27,11,13,26]
WP–153	**98%** [54,19]		**100%**
WS–126	**98%** [46, 28, 42]		**99%** [64]
WS–129	**87%** [4, 28, 36]	**88%** [24, 48, 30, 16, 17, 3, 5]	
WS–132	**91%** [53, 60, 63, 58, 63, 59, 24, 51, 37]		**100%**
WS–135	**82%** [50, 58, 46]	**95%** [52, 6, 23]	

[1] 1,1 Dichloroethane
[2] 1,1 Dichloroethylene
[3] 1,1,1,2 tetrachloroethane
[4] 1,2 Dichloroethylene
[5] 1,2,3 Trichlorobenzene
[6] 1,2,4 Trichlorobenzene
[7] 1,3 Dinitrobenzene
[8] 1,3,5 Trinitrobenzene
[9] 2 Amino 4,6 dinitrotoluene
[10] 2 Nitrotoluene
[11] 2,4 Dinitrotoluene
[12] 2,4,6 Trinitrobenzene
[13] 2,6 Dinitrotoluene
[14] 3 Nitrotoluene
[15] 4 Amino 2,6 dinitrotoluene
[16] 4 Chlorotoluene
[17] 4 Isopropyltoluene
[18] 4 Nitrotoluene
[19] 4-Methylphenol
[20] Acenaphthylene
[21] Ammonia as N
[22] Benzene in GRO
[23] Bromodichloromethane
[24] Bromoform
[25] Cadmium
[26] Calcium
[27] Carbazole
[28] Carbon Tetrachloride
[29] Chlordane, technical
[30] Chloromethane
[31] COD
[32] Conductivity at 25ºC
[33] Diesel Range Organics
[34] Ethybenzene in GRO
[35] Ethyl Parathion
[36] Ethylbenzene
[37] Ethylene Dibromide
[38] Fluoride
[39] Hexachlorobutadiene
[40] Iron
[41] MCPP
[42] Methylene chloride
[43] Nitrite as N
[44] Nitrobenzene
[45] Oil & Grease (Gravimetric)
[46] Ortho-Phosphate as P
[47] RDX
[48] Sec-Butylbenzene
[49] Sulfide
[50] Surfactants-MBAS
[51] Tert-Butylbenzene
[52] Tetrachloroethylene
[53] Thallium
[54] Titanium
[55] TOC
[56] Toluene in GRO
[57] Total Organic halides (TOX)
[58] Total Residual Chlorine
[59] Uranium (Nat)
[60] Vanadium
[61] Volatile Solids
[62] Xylenes, total in GRO
[63] Zinc
[64] pH

and document acceptance. Hence, the validation program contains elements from—and is influenced by—several other references, including

- "*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*, Physical/Chemical Methods," EPA, November 1986, SW–846, Third Edition; Latest Update, February 2007
- "*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

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

Study	Matrix	General Engineering	TestAmerica	Eberline	SRS (EML)	Lionville
MAPEP–07–MaS17	Soil	99%(1)	98%(1),3,9,a,b,c,d	100%*	100%*	94% 2,4,5,6,7,8,†
MAPEP–07–MaW17	Water	100%	97%9	100%*	100%	100%†
MAPEP–07–OrW17	Water	100%	100%	No Data	No Data	100%†
MAPEP–07–GrW17	Water	100%	100%	No Data	100%	No Data

[1] Results for selenium were not acceptable.
[2] Results for uranium (total) were not acceptable.
[3] Results for antimony were not acceptable.
[4] Results for endosulfan II were not acceptable.
[5] Results for heptachlor epoxide were not acceptable.
[6] Results for 4,4'-DDE were not acceptable.
[7] Results for 4,4'-DDD were not acceptable.
[8] Results for endrin Aldehyde were not acceptable.
[9] Results for hydrogen-3 were not acceptable.

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

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

associated analytical records. A summary of findings is presented in each project narrative or validation report prepared by SGCP personnel.

Data Review

Major QA issues identified during 2007—as well as those identified in prior years that are still undergoing resolution—are summarized in this section. The detailed data review program for groundwater and soil/sediment analyses is described in WSRC–3Q–2, Volume 1, "Plans and Procedures," Section 1100, *Environmental Monitoring Program*, and in the following SGCP procedures: ER–AP–302, "Data Summary Report"; ER–AP–303, "Analytical Data Validation Report"; and ER–AP–306, "Laboratory Data Records Reviews."

In 2007, the major QA issues discovered and addressed in connection with these programs for soil/sediment and groundwater analyses included the following:

- Operating to EPA method deviations without proper justification, authorization, and acceptance at one laboratory

Previously identified items resolved in 2007 included the following:

- Outdated interelement corrections for ICP-AES metals at two laboratories
- Calibration spreadsheet errors for strontium-90 and gross alpha/beta 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

Table 8–4
Subcontract-Laboratory MAPEP Issues

Eberline Services	General Engineering	TestAmerica	Lionville
Cesium-134	Antimony	None	Uranium–Total
	Plutonium-238		
	Zinc-65		

- Nitrate-nitrite analysis without reduction checks at one laboratory

Previously identified items still being addressed include the following:

- Inadequate internal standardization for total uranium by ICP-MS at one laboratory
- Calibrated region deviations for alpha spectroscopy analytes at one laboratory
- Calibration stability problems for isobutanol at one laboratory
- Gas-Flow Proportional Counting without daily cross-talk checks at two laboratories (This issue is being resolved through a formal DOE resolution process; cross-talk calibrations have been corrected, only daily checks remain to be corrected)
- 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 in 2008. Corrective action plans can range from proposed changes by a laboratory (software upgrades, new software purchases/creation, and procedure revisions), to demonstration by a lab that a deficiency is an acceptable deviation, to the site's decision to no longer use a lab that refuses to correct deficiencies.