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# Quality Assurance

CHAPTER

# 8

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[During 2008, responsibility for the environmental Quality Assurance (QA) program continued to be divided among three groups—Environmental Monitoring Laboratory (EML), Environmental Monitoring (EM), and Data Management and Waste Engineering (DMWE).]

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*WSRC's environmental QA program is conducted to verify the integrity of analyses determined by onsite and subcontracted offsite environmental laboratories, and to ensure that quality control program requirements are met. The program's objectives are to ensure that samples are representative of the surrounding environment, and that analytical results are accurate.*

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## SRS and Environmental QA Programs Integration

The SRS comprehensive environmental QA program follows the QA requirements defined in the WSRC Quality Assurance Manual (WSRC 1Q). Each environmental organization has developed and implemented QA procedures that address these requirements. In addition, a Cognizant Quality Function (CQF) from the site's independent QA organization is assigned responsibility for environmental program oversight for each organization. The CQF periodically performs QA reviews and assessments on environmental programs to ensure compliance with site requirements. In addition, each organization assigns QA responsibilities to individuals to oversee daily QA activities for the organization. Results, improvement opportunities, and corrective actions that come from assessments and reviews are documented in the Site Tracking, Analysis and Reporting (STAR) system. Site environmental professionals periodically conduct QA self-assessments on specific environmental program activities. The results of these assessments are documented in STAR. Site management participates in the Management Field Observation process, and the results from these reviews also are documented in STAR.

## QA for EM Program Samples

### Internal Quality Assurance Program

EM has a documented QA program that meets SRS and U.S. Department of Energy (DOE) requirements (3Q1-2 Volume III, "Quality Assurance Plan"). Based on data reviews, no QA issues or corrective actions were identified during 2008.

### Laboratory Certification

EM 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. Certification is renewed every three years; the current certification expires in June of this year.

### Blind pH Samples

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

During 2008, two blind pH field measurements were taken monthly, for a total of 24 samples. All field pH

### 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.

**Cross-talk** - The fraction of all recorded pulses from alpha particles that are recorded in the beta channel due to degradation in their pulse height or the fraction of all recorded pulses from beta particles that are recorded in the alpha channel due to pulse pileup or other phenomenon.

**Field Duplicates** - Independent samples collected as closely as possible to the same point in space and time. They are two separate samples taken from the same source, stored in separate containers, and analyzed independently.

**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.

measurements were within the U.S. Environmental Protection Agency's (EPA's) suggested acceptable control limit of  $\pm 0.4$  pH units of the true (known) value. Blind pH sample results can be found in the data tables section of the CD accompanying this report ["Blind Sample Results for pH Field Measurements"].

## 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. Analytical instrumentation includes liquid scintillation and gas flow proportional counters, alpha and gamma spectrometry, inductively coupled plasma atomic emission spectrometry

(ICP-AES), inductively coupled plasma mass spectrometry (ICP-MS), flow injection mercury system (FIMS) and gas chromatography mass spectrometry (GC-MS). Analyses include tritium, carbon-14, nickel-63, gamma isotopes (cesium-137, cobalt-60, potassium-40, etc.), iodine-129, strontium 89,90, strontium-90, americium-241, curium-244, neptunium-237, plutonium-238, plutonium-239, thorium-229, thorium-230, thorium-232, uranium-234, uranium-235, uranium-238, inorganic metals, mercury, and volatile organic compounds. Total suspended solids are determined gravimetrically. Instruments are calibrated with known reference standards. Instrument performance is monitored through the use of check standards and control charts. Analytical batch performance is measured through the use of quality control (QC) samples (blanks, spikes, carriers, tracers, laboratory control samples, and laboratory duplicates). QC results that fall outside of speci-

fied limits may result in analytical batch or sample reruns. For those batches or samples that fall outside of limits but for which the results are determined to be satisfactory, the 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 corrective actions were identified during 2008.

### Laboratory Certification

EML is certified by the SCDHEC Office of Laboratory Certification for analytical measurements using the following methods:

- total suspended solids (Standard Methods, 2540D), 27 metals by ICP–AES (EPA, 200.7), mercury by FIMS (EPA, 245.2), and 18 metals by ICP–MS (EPA, 200.8)
- 42 volatile organic compounds by GC–MS (EPA, 8260B), 28 metals by ICP–AES (EPA, 6010C), mercury by FIMS (EPA, 7470A and 7471B), and 18 metals by ICP–MS (EPA, 6020A)

Certification is renewed every three years; the current certification expires in June 2009.

### External QA Program

In 2008, 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 2008, EML completed the analysis of 54 radioisotopes and 15 metals for MAPEP–18 (designation of a specific study set) and the analysis of 56 radioisotopes and 15 metals for MAPEP–19. Results show that the laboratory passed the 80-percent-acceptable-results level for the study set (table 8–1). The

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

Study Set	Matrix	EML <sup>1</sup>
MAPEP–07–GrF18	Air Filter	100%
MAPEP–07–GrW18	Water	100%
MAPEP–07–MaS18	Solid	100%
MAPEP–07–MaW18	Water	100%
MAPEP–07–RdF18	Air Filter	100%
MAPEP–07–MaV18	Vegetation	100%
MAPEP–08–GrF19	Air Filter	100%
MAPEP–08–GrW19	Water	100%
MAPEP–08–MaS19	Solid	100%
MAPEP–08–MaW19	Water	97% <sup>2</sup>
MAPEP–08–RdF19	Air Filter	100%
MAPEP–08–MaV19	Vegetation	100%

<sup>1</sup> Column presents percentage of tests that exceeded 80%-acceptable-results level  
<sup>2</sup> Result for Tc-99 not acceptable (bias greater than 30%)

percentage 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 [“MAPEP Performance Study Series 18” and “MAPEP Performance Study Series 19”]. 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 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 Uncertainty (Unc) Flag column is used to note uncertainty values that may be High (H) or (L), etc.

## QA for EM 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. 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. Evaluations were conducted at four laboratories in 2008, resulting in a total of 22 Priority II findings. A Priority II finding documents a deficiency which in and of itself does not represent a concern of sufficient magnitude to render the audited facility unacceptable to provide services to DOE. Each laboratory submits a corrective action response that addresses each finding. The findings are reviewed and typically closed during the next laboratory audit.

## Nonradiological Liquid Effluents

National Pollutant Discharge Elimination System (NPDES) samples are analyzed by four onsite laboratory groups—EML, EM, the Site Infrastructure

& Services Department (I&SD), and Washington 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 2008, all laboratories performing NPDES analyses for SRS participated in the EPA-required Discharge Monitoring Report (DMR) and Proficiency Testing Studies. 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 laboratories investigate the unacceptable results and implement corrective actions as appropriate.

WSMS participated in the 2008 DMR–QA Study 28, while SES, EM, EML, and I&SD participated in ERA’s water proficiency (WP)–160, WP–161, and WP–162 studies. All the studies’ results were determined to be acceptable. Interlaboratory program results can be found in the data tables section of the CD accompanying this report [“Discharge Monitoring Report - Proficiency Test Results”].

## Intralaboratory Program

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

The onsite and offsite laboratories processed 67 field duplicate analyses during 2008. Zero-difference results were reported for 49 of these analyses. Thirteen field duplicate analyses were between the zero and  $\leq 20$ -percent difference. Only five of the 67 field duplicate analyses exceeded the relative-percent ( $\leq 20$ -percent) difference.

The onsite and offsite laboratories processed 77 blind analyses during 2008. Zero-difference results were reported for 53 of these analyses. Nineteen field duplicate analyses were between the zero and  $\leq 20$ -percent difference. Only five of the 77 blind analyses exceeded the relative percent ( $\leq 20$ -percent) difference.

Results for the field duplicate and blind sam-

**Table 8–2**  
**Subcontract-Laboratory Percent Acceptable Performance for Environmental Resource Associates (ERA) Water Pollution Studies**

*Note: Laboratories met or exceeded the 80-percent-acceptable-results level.*

Study	General Engineering	TestAmerica
WS–141	93% <sup>10,14, 18, 19</sup>	97% <sup>1,17</sup>
WS–146	99% <sup>12</sup>	
WS–147		100%
WP–159	100%	
WP–161		98% <sup>2,3,4,5,8,9,13</sup>
WP–162		89% <sup>6,7,11,15,16</sup>
WP–164	100%	

  

<b>Results Not Acceptable</b>		
<sup>1</sup> 1,2,3 trichloropropane (TCP)	<sup>8</sup> Cobalt	<sup>15</sup> Toluene
<sup>2</sup> Aroclor 1242	<sup>9</sup> Dalapon	<sup>16</sup> Total organic halides (TOX)
<sup>3</sup> Aroclor 1016	<sup>10</sup> DOC	<sup>17</sup> Tetrachloroethylene
<sup>4</sup> Alkalinity as CaCO <sub>3</sub>	<sup>11</sup> Ethylbenzene	<sup>18</sup> Turbidity
<sup>5</sup> Ammonia as N	<sup>12</sup> Heterotrophic plate count	<sup>19</sup> Vanadium
<sup>6</sup> Benzene in (GRO)	<sup>13</sup> Orthophosphate as P	
<sup>7</sup> Benzene	<sup>14</sup> TOC	

pling programs indicated no consistent problems with the laboratories. Field duplicate and blind sample program results can be found in the data tables section of the CD accompanying this report [“NPDES Duplicate Sample Results” and “NPDES Blind Sample Results”].

### 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 2008. SES and EML reported approximately 3,000 analyses for this program. Greater than 95 percent of the approximately 1,100 field duplicate results were within acceptable limits ( $\leq$  20-percent difference). Results for the field duplicate sampling program indicated no consistent problems with the laboratories. Detailed stream and Savannah River field duplicate sample results can be found in the data tables section

of the CD accompanying this report [“Duplicate Sample Results”].

### QA for DMWE Sample Analyses

Groundwater analyses at SRS are performed by offsite (subcontract) and onsite laboratories. During 2008, General Engineering Laboratories and TestAmerica, Inc., were the primary full-service subcontractors. The EML performed groundwater analyses for DMWE during 2008. Eberline Services Oak Ridge Lab (radiological only) and Lionville Laboratory (nonradiological only) were subcontracted laboratories; however, each was minimally used during 2008.

During 2008, General Engineering and TestAmerica participated in various WP and water supply (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

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

Study	Matrix	General Engineering	TestAmerica
MAPEP–07–MaS18	Soil	97% <sup>1,7,8</sup>	97% <sup>2,9,13</sup>
MAPEP–07–MaW18	Water	97% <sup>9</sup>	97% <sup>6</sup>
MAPEP–07–OrW18	Water	96% <sup>10,(11),12</sup>	97% <sup>12,(14)</sup>
MAPEP–07–GrW18	Water	100%	100%
MAPEP–08–MaS19	Soil	98% <sup>3,(5)</sup>	98% <sup>3,9</sup>
MAPEP–08–MaW19	Water	100%	97% <sup>(9)</sup>
MAPEP–08–OrW19	Water	99% <sup>7</sup>	99% <sup>4</sup>
MAPEP–08–GrW19	Water	100%	100%

<sup>1</sup> Results for selenium were not acceptable.  
<sup>2</sup> Results for antimony were not acceptable.  
<sup>3</sup> Results for mercury were not acceptable.  
<sup>4</sup> Results for gamma-BHC (lindane) were not acceptable.  
<sup>5</sup> Results for endrin ketone were not acceptable.  
<sup>6</sup> Results for hydrogen-3 were not acceptable.  
<sup>7</sup> Results for heptachlor were not acceptable.  
<sup>8</sup> Results for iron-55 were not acceptable.  
<sup>9</sup> Results for nickel-63 were not acceptable.  
<sup>10</sup> Results for benzo(a)anthracene were not acceptable.  
<sup>11</sup> Results for chrysene were not acceptable.  
<sup>12</sup> Results for hexachlorobenzene were not acceptable.  
<sup>13</sup> Results for bis(2-ethylhexyl)phthalate were not acceptable.  
<sup>14</sup> Results for methoxychlor were not acceptable.  
<sup>( )</sup> False positive  
<sup>[ ]</sup> False negative

studies associated with contracted analyses performed for SRS.

Results from the subcontract-laboratory performance on MAPEP are summarized in table 8–3. The results show that all laboratories exceeded the 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 contract scope.

To help participants identify, investigate, and resolve potential quality concerns, the MAPEP issues a letter of concern to a participating laboratory upon identification of a potential analytical data quality problem in the MAPEP results. Letters of concern have been issued since 1996, shortly after the beginning of the MAPEP program. A copy of the letter is sent to DOE/contractor oversight points of contact (POCs), including DOE Field Office and Headquarters POCs and contractor sample management POCs. Intended to be informative and not punitive, each letter states,

“This letter is solely intended to alert your laboratory to a potential quality concern that you may wish to investigate for corrective action.” Table 8–4 summarizes MAPEP concerns from 2008 for the primary full-service subcontracted laboratories.

#### 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 DMWE according to EPA standards for analytical data quality, or as specified by SRS onsite 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 and document acceptance. Hence, the SRS 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 2008
- “*DOE Quality Systems for Analytical Services*,” Revision 2.4, October 2008

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 DMWE personnel.

**Data Review**

The QA program’s detailed data review for ground-water and soil/sediment analyses is described in WSRC-3Q1-2, Section 1100.

One item that still required resolution in 2008 was closed as follows:

- Gas-flow proportional counting without daily cross-talk checks was resolved at two laboratories.

Items that are ongoing each year and are resolved on a case-by-case basis with each laboratory include

- incomplete record packages for validation
- omissions and logic failures in electronically reported data

The identification and resolution of quality and technical issues illustrates that, although laboratory procedures are well defined, analytical data quality does benefit from technical scrutiny.

**Table 8-4  
Subcontract-Laboratory Performance MAPEP Letters of Concern**

General Engineering	TestAmerica
Selenium (series 18)	Antimony (series 18)
	Hydrogen-3 (series 18)
	Strontium-90 (series 18)
	Nickel-63 (series 19)