Chapter 7

Quality Assurance

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[Editor's note: The Environmental Monitoring Section (EMS) of the Savannah River Site (SRS) Environmental Protection Department (EPD) maintained the environmental quality assurance (QA) program in 2002. As part of the site's reorganization, effective the beginning of 2003, this responsibility has been divided among three groups—the Environmental Monitoring Laboratory (EML), the Environmental Monitoring and Analysis group (EMA), and the Geochemical Monitoring group (GM). When referencing results specific to 2002, this chapter will continue to cite EMS.]

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

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

This chapter summarizes the 2002 QA program. Guidelines and applicable standards for the program are referenced in appendix A, "Applicable Guidelines, Standards, and Regulations."

Tables containing the 2002 QA data and the nonradiological detection limits can be found on the CD accompanying this report.

A more complete description of the QA program can be found in *Savannah River Site Environmental Monitoring Program* (WSRC–3Q1–2, Section 1100) and in the *Savannah River Site Environmental Monitoring Section Quality Assurance Plan* (WSRC–3Q1–2, Section 8000).

The 2002 QA data and program reviews demonstrate that the data in this annual report are reliable and meet applicable standards.

QA for EMA Laboratories

Internal Quality Assurance Program

Field Sampling Group

EMA and EML personnel routinely conduct a blind sample program for field measurements of pH to assess the quality and reliability of field data measurements. EMA personnel also measure total residual chlorine, dissolved oxygen, and temperature in water samples; but because of the difficulties in providing field standards, these measurements are not suitable for a blind sample program.

During 2002, 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.

Chemistry and Counting Laboratories

Blind Tritium Samples Blind tritium samples provide a continuous assessment of laboratory sample preparation and counting. During 2002, six blind samples were analyzed for tritium. All tritium results were within the control limits.

Laboratory Certification EML is certified by the South Carolina Department of Health and Environmental Control (SCDHEC) Office of Laboratory Certification for the following analytes:

- under the Clean Water Act (CWA)—chemical oxygen demand, total suspended solids, field pH, total residual chlorine, temperature, and 26 metals
- under the Resource Conservation and Recovery Act (RCRA)—50 volatile organic compounds (VOCs) and 27 metals

External Quality Assurance Program

In 2002, the EMS laboratory participated in the U.S. Department of Energy (DOE) Quality Assurance

Table 7–1 Subcontract Laboratory Performance in ERA Water Pollution and Water Supply Studies

Laboratory	Water Pollution Studies (Percent Acceptable)[a]	Water Supply Studies (Percent Acceptable)
Lionville	WP 90 (98%)[b]	WS 72 (98%)[c]
General Engineering	WP 90 (100%)	WS 69 (94%)[d]
General Engineering Mobile Lab	WP 87 (99%)[e]	
Shealy Environmental Services	WP 84 (97%)[f]	WS 71 (98%)[g]

a Laboratories are expected to exceed 80 percent acceptable results.

b The result for methylene chloride was not acceptable.

c Results for chloride and orthophosphate were not acceptable.

d Results for total xylenes, chloromethane, 1,3–dichloropropane, conductivity, orthophosphate, and bromide were not acceptable.

e Results for 1,1–dichloroethylene and cis–1,2dichloroethylene were not acceptable.

f Results for aluminum, copper, chloride, conductivity, total hardness, turbidity, and benzo(k)fluoranthene were not acceptable.

g The result for aluminum was not acceptable.

Program (QAP), an interlaboratory comparison program that tracks performance accuracy and tests the quality of environmental data reported to DOE by its contractors.

For a radiological laboratory intercomparison in 2002, the analysis of 43 isotopes was completed in March on the 56th set of QAP samples and the analysis of 44 isotopes was completed in September on the 57th set. A performance rating of 84 percent acceptable was achieved on the 56th set; the rating for the 57th set was 91 percent acceptable. This rating was calculated by dividing the “acceptables” and the “acceptable with warnings” by the total number of results. Environmental QA personnel consider 80 percent to be the minimum acceptance rate in this program.

The March results, which were considerably lower than normal, are attributed to the disruption of operations during the move of the laboratory to a new building.

Detailed QAP intercomparison study results can be found in the data tables section of the CD accompanying this report.

QA for Subcontracted Laboratories/EMA Laboratories

Subcontracted environmental laboratories providing analytical services must have a documented QA program and meet the quality requirements defined in *WSRC Quality Assurance Manual* (WSRC–1Q).

An annual evaluation of each subcontracted laboratory is performed to ensure that all the laboratories maintain technical competence and follow the required QA programs. Each 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

Effluent samples are analyzed by five laboratories—three onsite laboratories and two subcontracted laboratories. Laboratories must be certified by SCDHEC for all analyses.

Interlaboratory Comparison Program

During 2002, EMS and a number of its subcontracted laboratories participated in the Environmental Resource Associates (ERA) WatR™ Pollution Proficiency Testing (PT) Studies, which include various InterlaB WatR™ Supply Water Pollution (WP) and Water Supply (WS) Performance Evaluation Programs. Performance results by the subcontracted laboratories can be found in table 7–1.

The proficiency rating is calculated as follows: acceptable parameters divided by total parameters analyzed, multiplied by 100.

EPA uses PT results to certify laboratories for specific analyses. As part of the recertification process, EPA requires that subcontracted laboratories investigate the outside-acceptance-limit results and implement corrective actions as appropriate.

Laboratories (commercial and government) that analyze National Pollutant Discharge Elimination System (NPDES) samples participate in the Discharge Monitoring Report–Quality Assurance (DMR–QA) study or the WP study. Under this program, the laboratories obtain test samples from ERA. This provider, as required by EPA, is accredited by the National Institute of Standards and Technology. For the 2002 DMR–QA study, Shealy Environmental Services, Inc. (SES) used the WP 89 study.

SES reported acceptable results for 16 of 16 NPDES parameters and 10 of 10 voluntary analytes. EMS reported acceptable results for 14 of 14 NPDES parameters and eight of 11 voluntary analytes. The Site Utilities Division (SUD) Wastewater Laboratory reported acceptable results for three of three NPDES parameters. The TNX Effluent Treatment Facility did not participate in the PT studies. EML has a corrective action plan in place to investigate and correct PT failures. Subsequent samples for the failed voluntary parameters will be analyzed in 2003. Until acceptable results are obtained with the voluntary analytes, EML will not analyze samples for cobalt, potassium, and sodium.

Intralaboratory Comparison Program

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

SES and the EMS laboratory analyzed a total of 95 duplicate samples during 2002. Nondetectable results were reported for 70 of these duplicate samples.

Percent difference calculations showed that 11 of the 95 duplicate samples analyzed were outside the EMS internal QA requirement (± 20 percent of the true value). These exceptions appeared to be related to an analytical error, sample contamination, or improper sampling techniques. Generally, exceptions in this range are not considered a problem.

SES and EMS analyzed a total of 91 blind samples during 2002. Nondetectable results were reported for 75 of these samples.

Percent difference calculations showed that seven of the 91 blind samples analyzed were outside the EMS internal QA requirement (± 20 percent of the true value). These exceptions appeared to be related to an analytical error, sample contamination, or improper sampling techniques. Generally, exceptions in this range are not considered a problem.

Results for the duplicate and blind sampling programs met expectations, with 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 the EMS laboratory in 2002. SES analyzed samples for hardness, herbicides, nitrate + nitrite, phosphorus, pesticides, and total organic carbon. EMS analyzed duplicate samples for chemical oxygen demand, metals, and total suspended solids. Only one analysis result was outside the ± 20 percent acceptance limit. Detailed stream and Savannah River water quality duplicate sample results can be found in the data tables section of the CD accompanying this report.

Groundwater

Groundwater analyses at SRS are performed by subcontracted laboratories. SRS requires that the laboratories investigate the outside-acceptance-limit results and implement corrective actions as appropriate.

Internal QA

During 2002, approximately 5 percent of the samples collected (radiological and nonradiological) for the RCRA and the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) programs were submitted to the primary laboratory for analysis as blind duplicates and to a different laboratory as a QA check. The laboratories' results were evaluated on the basis of the percentage within an acceptable concentration range.

Generally, results for all QA evaluations were found to be within control limits in 2002. Full results for all QA evaluations can be obtained by contacting the EMA manager at 803–952–6931.

External QA (Environmental Resource Associates Standards)

Water Pollution and Water Supply Studies During 2002, General Engineering, General Engineering Mobile, and Lionville participated in various WP and WS studies (WP and WS studies are described on page 62). The results show that all laboratories exceeded the 80-percent acceptable results level that is expected. Performance result summaries can be found in table 7–1.

Table 7–2 Subcontract Laboratory Performance on Environmental Resource Associates Standards

Laboratory	Percent Within Limits[a] 1st Quarter 2002	2nd Quarter 2002	3rd Quarter 2002
EMS	100	90.9[b]	96.7[c]
General Engineering	98.3[d]	97.8[e]	96.8[f]
General Engineering – Mobile Lab	97.7[g]	99.2[h]	99.2[i]
Lionville	97.6[j]	98.2[k]	93.7[l]
Microseeps	88.1[m]	89.2[n]	96.0[o]

a Laboratories are expected to exceed 80 percent acceptable results.

b Results for mercury and strontium were not acceptable.

c Result for zinc was not acceptable.

d Results for 4–chlorophenol phenyl ether, 2,4–D, and total phosphates (as P) were not acceptable.

e Results for alkalinity (as $CaCO_3$), carbon tetrachloride, PCB 1016, and PCB 1242 were not acceptable.

f Results for ammonia nitrogen, butylbenzyl phthalate, nitrate as nitrogen, nitrate nitrite (as nitrogen) [inorganics], nitrate nitrite (as nitrogen) [simple nutrients], and specific conductance were not acceptable.

g Results for bromoform, endrin, and hexaclorobutadiene were not acceptable.

h Result for 2–nitrophenol was not acceptable.

i Results for chrysene, fluoride, pentachlorophenol, and pH were not acceptable.

j Results for chloride, dichloromethane, and PCB 1016 were not acceptable.

k Results for aldrin, chloride, dieldrin, dichloromethane, endrin, heptachlor, lindane, methoxychlor, PCB 1016, PCB 1254, and toxaphene were not acceptable.

l Results for benzo[*k*]fluoranthene, bis(2–chloroethoxy methane), chloride, and fluoride were not acceptable.

m Results for acetone, benzo[*b*]fluoranthene, benzo[*a*]pyrene, chromium, 2,4–D, di–n–octyl phthalate, iron, manganese, nickel, silver, 2,4,5–T, and zinc were not acceptable.

n Results for aldrin, benzo[*a*]anthracene, 1,1–dichloroethane, 1,2–dichloroethane, dieldrin, endrin, heptachlor, heptachlor epoxide, lindane, methoxychlor, and 1,1,1–trichloroethane were not acceptable.

o Results for copper, heptachlor, and 2,4,5–T were not acceptable.

Quarterly Assessments During 2002, EMS conducted quality assessments of the primary analytical laboratories to review their performance on certain analyses. Each laboratory received a set of certified environmental quality control standards from ERA, and its results were compared with the ERA-certified values and performance acceptance limits. The performance acceptance limits closely approximate the 95 percent confidence interval.

Results from the laboratories (EMS, General Engineering, General Engineering Mobile, Lionville, and Microseeps,) for the first three quarters are summarized in table 7–2. The results show that all laboratories exceeded the 80-percent acceptable results level that is expected. Fourth-quarter results were not available in time for publication in this report.

Soil/Sediment

Environmental investigations of soils and sediments, primarily for RCRA/CERCLA units, are performed by subcontracted laboratories. Data were validated by EMS in 2002 according to EPA standards for analytical data quality unless specified otherwise by site customers.

The environmental validation program is based on two EPA guidance documents, *Data Quality Objectives Process for Superfund* (EPA–540–R–93–071) and *Data Quality Objectives Process for Hazardous 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 how to evaluate these inputs, nor do they propose pass/fail criteria to apply to data and documents. Hence, the validation program necessarily contains elements from—and is influenced by—several other sources, including

- *Guidance on Environmental Data Verification and 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 Inorganic Data Review*, EPA–540/R–01/008
- T*est Methods for Evaluating Solid Waste*, EPA, November 1986, SW–846, Third Edition
- *Data Validation Procedures for Radiochemical Analysis*, WHC–SD–EN–SPP–001

Relative percent difference for the soil/sediment program is calculated for field duplicates and laboratory duplicates.Generally, results for all QA evaluations were found to be within control limits in 2002. A summary of this information is presented in each project report prepared by GM 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 2002, the major QA issues that were discovered and addressed in connection with these programs included the following at two laboratories (of the five that conduct groundwater and soil/sediment analyses):

- inadequate chromatographic separation of certain pesticides
- repeated failure of calibration verifications for organics, and unorthodox responses
- nonstandard and unapproved uncertainty calculation method for undetected gamma nuclides
- systematic calculation errors for two gamma nuclides
- inadequate radiological batch quality control association
- inability to demonstrate the absence of spectral interference for liquid scintillation counter radioisotopes

Also, inconsistent application of the blank qualification policy was discovered across all the laboratories.

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 2003.