Sampling and Analysis Plan for Old Solvent Tanks S1-S22 to Address Waste Acceptance Criteria

by

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P. E. Filpus-Luyckx

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OCTOBER 2, 1997

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LIST OF ACRONYMS

AAS	Atomic Absorption Spectrophotometry
ADS	Analytical Development Section
CIF	Consolidated Incineration Facility
COC	Chain-of-Custody
CVAA	Cold Vapor Atomic Absorption
DQO	Data Quality Objective
ERD	Environmental Restoration Department
EPA	United States Environmental Protection Agency
GC	Gas Chromatography
GC/MS	Gas Chromatography/Mass Spectrometry
HLWD	High Level Waste Division
IC	Ion Chromatography
ICP-ES	Inductively-Coupled Plasma-Emission Spectroscopy
ICP-MS	Inductively-Coupled Plasma-Mass Spectrometry
IDW	Investigation-Derived Waste
LEPS	Low Energy Photon Spectroscopy
LSC	Liquid Scintillation Counting
MWSF	Mixed Waste Storage Facilities
ORWBG	Old Radioactive Waste Burial Ground
PHA	Pulse Height Analysis
QA	Quality Assurance
QAPjP	Quality Assurance Project Plan
QC	Quality Control
SAP	Sampling and Analysis Plan
SRS	Savannah River Site
SRTC	Savannah River Technology Center
SST	Solvent Storage Tank Facility
SWD	Solid Waste Division
TCLP	Toxic Characteristic Leaching Procedure
TOC	Total Organic Carbon
TRU	Transuranic
WAC	Waste Acceptance Criteria
WSRC	Westinghouse Savannah River Company
XRD	X-ray Diffraction
XRS	X-ray Spectroscopy

1. INTRODUCTION

The Environmental Restoration Department (ERD) assumed custody of the Old Solvent Tanks (Tanks S1-S22) in the Old Radioactive Waste Burial Ground (ORWBG, 643-E) from Waste Management in January 1991. The purpose of this Sampling and Analysis Plan (SAP) is to collect and analyze samples of the sludge solids, organic and aqueous phases to determine the level of radioactivity, the isotopic constituents, the specific gravity, and other physical parameters. These data must be obtained to evaluate the process safety of remediating the tanks, to determine the disposal path for the material in the tanks, and to determine the most viable closure technology for the tanks.

1.1 Objectives

Project objectives are:

- To update and supplement existing compositional information regarding the vapor phase, liquid phase(s), and solid phase(s) in each tank.
- To provide sufficient compositional information to develop the safety envelope for waste removal and tank closure in a way that will not unnecessarily constrain operations.
- To quantify the necessary constituents and physical parameters in the solvent and aqueous phases to meet the Waste Acceptance Criteria (WAC) at the Solvent Storage Facility¹ (SST) operated by Solid Waste Division (SWD) and to meet the WAC for incineration at the Consolidated Incineration Facility² (CIF).
- To quantify the necessary constituents and physical parameters in the sludge phase for acceptance at the 241-F/H Tank Farms³ operated by High Level Waste Division (HLWD).
- To quantify the necessary constituents and physical parameters in the solvent, aqueous and sludge phases to meet the Waste Acceptance Criteria (WAC) at the Mixed Waste Storage Facilities⁴ (MWSF) operated by SWD.

1.2 Scope

The scope of this SAP includes: collecting samples of the liquid phase(s), and Solid phase(s) (sludge) from specific tanks; analyzing the samples to determine compliance with the Waste Acceptance Criteria of potential accepting SRS Waste Treatment Facilities; collecting samples of the vapor phase from specific tanks; and analyzing the samples to provide the information required for environmental, safety and health considerations. ERD will develop criteria specifying which tanks will be sampled. This criteria will be based on actual tank conditions observed during previous video surveys.

2. BACKGROUND INFORMATION

2.1 Location

The ORWBG (Building Number 643-E) comprises approximately 76 acres located in the north-central portion of SRS, about five miles southeast of A-Area on the north side of Road E between F- and H-Areas (Separations). The ORWBG was a trench disposal area which received radioactive waste from 1952 until 1972. A map showing the ORWBG in relation to the locations of the major E-Area facilities is included in Figure 1.

2.2 Site Description

The 22 Old Solvent Tanks are located in a "T"-shaped block near the center of the ORWBG about 400 feet northeast of the Burial Ground Administration Building 724-7E and 500 feet north of Road E. A map showing the location of the Old Solvent Tanks S1-S22 in relation to the ORWBG is included in Figure 2. The relative location of each tank is shown in Figure 3.

2.3 Operational History and Waste Characteristics

The Old Solvent Tanks S1-S22 were single-wall steel tanks installed between 1955 and 1968 to receive spent PUREX solvent from Separations in F- and H-Areas. The tanks are all cylindrical, single-wall carbon steel tanks buried with the long axis horizontal. Each tank was installed with an approximate inclination of six inches.

In the PUREX process, targets and spent fuel rods were processed in Separations to recover activation products and to recover residual plutonium and uranium for recycling. The targets and rods were clipped into small pieces and placed in the nitric acid dissolver solution to dissolve the plutonium and uranium. The PUREX solvent, which consisted of tri-n-butyl phosphate dissolved in kerosene, was used to extract plutonium and uranium from the dissolver solution. Plutonium and uranium were recovered from the solvent and the solvent was recycled. After a number of cycles, the solvent became degraded and was neutralized with caustic compounds and stored in Tanks S1-S22 for aging. During the aging period, generally about six months, most of the short-lived, high-gamma fission products decayed and the heavy alpha emitters settled out of the solvent. The solvent separated into three phases: organic, aqueous, and sludge solids, consisting of alpha emitters in hydroxides and dibutyl phosphate complexes.

After aging, the sludge solids accumulated in the bottoms of Tanks S1-S22. The organic phase was drawn out of the tanks and burned in shallow pans placed in trenches in the Old Radioactive Waste Burial Ground. Approximately 382,750 gallons of spent solvent were burned between 1956 and 1972.

Tanks S1-S18 were pumped out to "unpumpable heel" in the solvent relocation program between March 1977 and May 1978. The solvent was placed in Tanks S19-S22. Tanks S19-S22 were pumped out and the solvent was transferred to Tanks S23-S30 between November 1980 and January 1981. No additional waste was placed in Tanks S1-S22 after January 1981. The total volumes of the material remaining in Tanks S1-S22 is ~8000 gallons of liquid and up to 1250 Kg solids. These values are being confirmed by remote camera surveillance.

Thus, one of the objectives of this SAP is to yield analytical results which can update and supplement existing compositional information regarding the vapor phase, liquid phase(s), and solid phase in each tank.

2.4 Disposition Strategy

The material disposition strategy that this Sampling and Analysis Plan supports is shown in Figure 4

3. DATA QUALITY OBJECTIVES (DQO)

According to the United States Environmental Protection Agency (EPA), "The DQO Process was developed to help EPA personnel collect data that are important to decision making". The process allows decision makers to define their data requirements and acceptable levels of decision errors while in the planning stage (before data are collected). Application of the DQO Process will result in the collection of data that yield results of appropriate quality for "defensible decision making". The DQO process is designed to ensure that the appropriate type, quality and quantity of environmental data match the intended applications.

The Data Quality Objectives (DQOs) for this project represent the level of analysis needed to quantify the contents adequately to allow evaluation of the process safety envelope and to evaluate transfer to SWD and/or HLW facilities.

3.1 Old Solvent Tanks S1-S22 in the Old Radioactive Waste Burial Ground 643-E DQO Process

Old Solvent Tanks S1-S22 in the Old Radioactive Waste Burial Ground 643-E are radioactively contaminated sites with the potential for commingled radiological and chemical contamination. The intended application of the Sampling and Analysis Plan is to collect data to support transfer to SWD and HLW Waste Treatment facilities. Meeting the WACs of these Waste Treatment facilities requires both radiological and chemical characterization.

3.1.1 Problem

Determine the appropriate sampling scheme necessary to characterize solvent and sludge phases of selected tanks to identify disposal options for the solvent and sludge.

3.1.2 Key Decisions

- Determine the composition of the vapor phase to provide design data and support environmental, safety and health considerations for the disposition of material in the tanks.
- Determine the chemical and radiological constituents in each liquid phase to determine compliance with the SST WAC, the CIF WAC, the TRU Pad WAC⁵ and/or the MWSF WAC.
- Determine the chemical and nuclide constituents in the sludge to evaluate the criticality safety envelope for waste removal and tank closure activities.
- Determine the chemical and radiological constituents in the sludge to determine compliance with the F/H Tank Farms WAC, the TRU Pad WAC and/or the MWSF WAC.

3.1.3 Identify the Inputs to the Decisions

- Definitive analytical data will be used to determine chemical and radiological constituents in the vapor phase.
- Definitive analytical data will be used to determine the chemical and radiological constituents in the liquid phase(s).
- Definitive analytical data will be used to determine the chemical and radiological constituents in the sludge phase.

3.1.4 Define the Boundaries of the Study

The primary goals of this sampling plan are to quantify the nature of the material within Tanks S1 through S22 to support evaluation of the safety envelope and to compare to the SST, CIF, MWSF, F/H Tank Farms, TRU Pad and the MWSF WACs.

3.1.5 Decision Rules

- If the tank has <50 gallons of solid phase, THEN no solid samples will be taken.
- IF the as-received sample has both a solid AND liquid phase(s), THEN the sample will be filtered by the Analytical Development Section (ADS) of the Savannah River Technology Center (SRTC) to segregate the solid sludge from the liquid.

- IF the liquid (or filtrate) shows both an organic AND an aqueous phase, THEN the two phases will be separated by ADS and analyzed separately.
- IF the solid material shows more than one phase, THEN the phases will be separated by ADS to the extent practicable and analyzed separately.
- IF the liquid phase(s) meet the SST and CIF WAC, THEN the liquids can be transferred to the SST facility. ELSE, the composition of the liquid phase(s) will be compared to the TRU limit.
- IF the liquid phase(s) exceed the 100 nCi TRU/g AND the TRU Pad WAC is met, THEN the liquids can be transferred to the TRU Pad. ELSE, the composition of the liquid phase(s) will be compared to the MWSF WAC.
- IF the liquid phase(s) meet the MWSF WAC, THEN the liquids can be transferred to the MWSF. ELSE, stabilization in place will be evaluated.
- IF the solid phase meets the F/H Tank Farm WAC, THEN the solids can be transferred to the F/H Tank Farm. ELSE, the composition of the solid phase will be compared to the TRU limit.
- IF the solid phase exceed the 100 nCi TRU/g AND the TRU Pad WAC is met, THEN the solids can be transferred to the TRU Pad. ELSE, the composition of solid phase will be compared to the MWSF WAC.
- IF the solid phase meets the MWSF WAC, THEN the solids can be transferred to the MWSF. ELSE, stabilization in place will be evaluated.
- IF any WAC is not met, THEN the compositional data derived from this SAP will be used to support a deviation request or evaluation for in place stabilization of the materials.

3.1.6 Tolerance for Uncertainty

The establishment of sampling protocol and use of definitive analyses will minimize potential errors introduced during field operations.

Given the nature, history, and inaccessibility of the material in the tanks, there exists the potential for the samples to be non-representative due to non-homogeneity of the tank contents. Any such additional uncertainties will likely cause the criticality safety controls to be more restrictive than otherwise, due to the conservative nature of such calculations.

3.2 Summary of DQO Evaluation

The solvent and sludge phases for each tank will need to be analyzed for all chemical and radiological constituents required by the respective WACs and to support evaluation of the safety envelope.

4. FIELD SAMPLING AND LABORATORY ANALYSIS PLAN

4.1 Overall Sampling Strategy

The overall field sampling strategy will, to the greatest extent possible, obtain samples from the tanks that will be representative of all phases present in the tank.

4.2 Liquid Phase Sampling

Organic phase and aqueous phase samples will be collected using a pumping system designed and fabricated by the Engineered Equipment & Systems Section, SRTC. The sample will be obtained at the lowest end of the tank, directly below the tank riser pipe. Certain tanks with large quantities of liquids will be sampled at both ends of the tank. The sampling devices must limit sample volume to avoid risks of criticality when working with materials of unknown composition. Specific volume limits will be documented in a Nuclear Criticality Safety Evaluation to be provided before sampling begins. The entire sample will be sent to ADS. If the as-received sample has both a solid and liquid phase(s), then the sample will be filtered by ADS to segregate the solid sludge from the liquid. If the liquid (or filtrate) shows both an organic and an aqueous phase, the two phases will be separated by ADS and analyzed separately for the constituents and physical parameters shown in Table 1. The selected parameters are those specified in the SST, CIF and MWSF WACs.

4.3 Sludge

Sludge phase samples will be collected using the same pumping system as that used to collect the liquid samples. The sludge sample will be obtained at the lowest end of the tank, directly below the tank riser pipe. Certain tanks with large quantities of sludge will be sampled at both ends of the tank. Separate sludge samples will be taken from tanks with large quantities of both liquid and sludge. The sampling devices must limit sample volume to avoid risks of criticality when working with materials of unknown composition. Specific volume limits will be documented in a Nuclear Criticality Safety Evaluation to be provided before sampling begins. The entire sample will be sent to ADS. If the as-received sample has both a solid and liquid phase(s), then the sample will be filtered by ADS to segregate the solid sludge from the liquid. The filter residue will be analyzed for the constituents and physical parameters shown in Table 1. The selected parameters are those specified in the F/H Tank Farm and the MWSF WACs.

4.4 Vapor Phase

Hydrogen samples will be collected by pumping ½ liter of tank air into a Tedlar®*. Three hydrogen samples at three different elevations will be obtained from each tank sampled

*Registered trademark for polyvinyl fluoride, owned by E. I. DuPont de Nemours & Company, Inc.

The hydrogen samples will be taken at tank bottom (approximately 6" above liquid/sludge), at mid-tank and at the top of the tank. One volatile/semi-volatile organic compound sample will be collected by pumping 100 liters of the tank air through an activated charcoal cartridge, then a silica gel cartridge using a Constant Flow Air Sampler. This sample will be taken at mid-tank elevation. The Tedlar[®] bags, activated charcoal cartridges, and silica gel cartridges will be sent to ADS and analyzed for the constituents shown in Table 1.

4.5 Analytical Methodologies

The analytical procedures that will be used by ADS are listed in Table 2, corresponding to the list of the constituents and physical parameters shown in Table 1.

Appendix A lists the applicable ADS procedure titles from the Savannah River Technology Center ADS Analytical Operating Procedures, Manual L16.1.

5. FIELD DOCUMENTATION RECORDS

5.1 Field Sample Chain-of-Custody

Samples will be assigned unique numbers in the field that identify the specific tank and sample type for each sample.

A formal quality control process (COC) will be used to ensure the integrity of the samples from the point of origin through the sample analysis is implemented. This process will demonstrate that the samples obtained in the field have been securely transported, and have reached the analytical laboratory without alteration. The COC process has been established by SRS Environmental Compliance procedures⁶. An example COC form is shown in Figure 5. The COC within the ADS laboratories will be maintained in accordance with SRTC-ADS COC procedure⁷.

5.2 Holding Times, Sample Packaging, and Shipment

The time required for shipping samples to the laboratory will be minimized to the extent possible. Sample holding times shall be commensurate with EPA Guidelines⁸. The shortest holding time for the samples that will be collected is 14 days for volatiles in aqueous samples. ADS will be responsible for ensuring that samples are analyzed within EPA specified holding times, as shown in Table 2. Samples will be packaged for shipment in accordance with WSRC Transportation Procedures (Radioactive Material Packaging Certification and Authorization Requirements). Samples will be transported in accordance with WSRC Transportation Procedures (Radioactive Materials Transported In Excepted Packages). The WSRC Transportation Procedure demonstrates compliance with Department of Energy transportation safety standards.

5.3 Investigation Derived Waste (IDW)

Pending the results of laboratory analysis, the aqueous and non-aqueous IDW will be handled in accordance with SRS Management Plan (WSRC-RP-91227) Rev. 2⁹. The laboratory results will be compared to the limits set in the Management Plan to determine the final disposition of the wastes.

6. QUALITY ASSURANCE PROJECT PLAN (QAPJP)

WSRC Procedure Quality Assurance Plan (QAP) 21-1¹⁰ establishes quality assurance requirements and responsibilities for environmental data collection and evaluation activities. This Sampling and Analysis Plan (SAP) is designed to comply with QAP 21-1 guidelines. The elements for a Quality Assurance Project Plan (QAPJP) called for under this SRS site guidance are addressed below. If the element has been previously addressed elsewhere in this document the appropriate section is referenced.

6.1 Project Description

A brief description of the project and its objectives is provided in Section 1.

6.2 Project Organization and Responsibility

The Analytical Development Section (ADS) of the Savannah River Technology Center (SRTC) will be the point of contact and will have overall responsibility for implementation of this SAP.

Environmental Restoration is responsible for the field collection of the samples.

ADS will provide technical assistance as required, and will ensure that adequately qualified personnel and necessary equipment are available to complete the laboratory work in a timely manner.

ADS will be responsible for the technical review of the data, results, and Quality Control (QC) information obtained throughout the project.

6.3 Data Quality Objectives

Data Quality Objectives are described in Section 3.

6.4 Sample Collection, Control, and Transport

Sampling methodologies are provided in Section 4. Sampling details and radiological controls will be specified in the Solvent Tank Specific Sampling Procedure and Radiation Work Permit.

6.5 Analytical Procedures

The applicable standard operating procedure for each measurement parameter is provided in Appendix A. ADS results would be Level 5 results, per WSRC Manual 1Q, Procedure 21-1.

6.6 Calibration Procedures

Calibration procedural steps are described within the applicable standard operating procedure for each measurement parameter as provided in Table 2.

6.7 Waste Disposal

The handling and disposal of waste materials resulting from field investigations is presented in Section 5.3. Waste resulting from laboratory operations will be in accordance with ADS procedures.

6.8 Internal Quality Control Checks

ADS has in place a Quality Assurance Program Plan that provides internal quality control checks on analytical work.

6.9 Performance Assessments

SRTC has in place a Quality Assurance internal assessment program that provides independent review. Non-compliance issues will be logged, documented, and controlled through assessment findings, which are attached to and become a part of the internal assessment report. These assessment findings will be directed to the appropriate supervisor for corrective action in a specified and timely manner.

6.10 Preventative Maintenance

Preventative maintenance procedural steps are described within the applicable standard operating procedure for each measurement parameter as provided in Table 2. Service to all equipment, instruments, tools, gauges, etc., will be performed only by qualified personnel. Non-conforming items will be managed in accordance with the Program Plan.

6.11 Corrective Action

When a significant condition adverse to quality is noted, the cause of the condition will be determined and corrective action will be taken to preclude repetition of the same condition. Condition identification and cause, documents affected, and corrective action will be documented and reported to ADS. Implementation of corrective action will be verified by documented follow-up action. All task personnel have the responsibility, as part of the normal work duties, to promptly identify and report conditions adverse to quality. Non-conforming items or services will be documented on a Nonconformance Report Form.

6.12 Reports to Management

Field logbooks will be maintained for recording and reporting field activity. Analytical results will be compiled within ADS, including the QA status of measurement systems and data.

7. REFERENCES

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2. Procedure WAC 3.17, Low Level Radioactive Waste Acceptance Criteria, Rev. 1, WSRC Manual 1S, Savannah River Site, Aiken, SC 29808 (1997).
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10. Procedure 21-1, Quality Assurance Requirements for the Collection and Evaluation of Environmental Data, Rev. 0, WSRC Manual 1Q, Savannah River Site, Aiken, SC 29808 (1994).
11. Franson, M. A., Ed., Standard Methods for the Examination of Waste and Wastewater, 17th ed., pg. 2-71, American Public Health Association, Washington, DC (1989)

Figure 1. Location of the ORWBG in relation to other E Areas.

Figure 2. Location of Tanks S1-22 in Relation to the ORWBG

OLD SOLVENT TANK 643-E LAYOUT

Figure 3. Relative locations of Tanks S1-22.

Figure 4. Material Disposition Strategy

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Figure 5 Field Chain-of-Custody Form

Table 1. Analytical Constituents, Parameters, and Detection Limits

Constituent/Parameter	WAC(s)	Vapor	Organic Liquid	Aqueous Liquid	Solid(s)	Required Analytical Detection Limit (Note 1)	Typical Analytical Detection Limit (Note 2)
Aluminate	AlO ₂ -	F/H Tank Farms			X		
Aluminum	Al	SST, CIF	X				42 mg/L
		SST, CIF		X			0.083 mg/L
		F/H Tank Farms			X		830 mg/Kg
Ammonium	NH ₄ ⁺	F/H Tank Farms			X	≤ 0.56 wt% in solution	
Antimony	Sb	SST, CIF	X			730 mg/Kg	100 mg/L
		SST, CIF		X		730 mg/Kg	0.2 mg/L
		F/H Tank Farms			X		2000 mg/Kg
Arsenic	As	SST, CIF	X			50 mg/Kg	3 mg/L
		SST, CIF		X		50 mg/Kg	0.005 mg/L
		F/H Tank Farms			X		50 mg/Kg
Barium	Ba	SST, CIF	X			100,000 mg/Kg (CIF), ≤ 1000 mg/Kg (SST)	2 mg/L
		SST, CIF		X		100,000 mg/Kg (CIF), ≤ 1000 mg/Kg (SST)	0.004 mg/L
		F/H Tank Farms			X		40 mg/Kg
Beryllium	Be	SST, CIF	X			Detectable	2 mg/L
		SST, CIF		X		Detectable	0.004 mg/L
		F/H Tank Farms			X		40 mg/Kg
Boron	B	SST, CIF	X				10 mg/L
		SST, CIF		X			0.02 mg/L
		F/H Tank Farms			X		200 mg/Kg
Cadmium	Cd	SST, CIF	X			20 mg/Kg	1 mg/L
		SST, CIF		X		20 mg/Kg	0.002 mg/L
		F/H Tank Farms			X	20 mg/Kg (Note 3)	19 mg/Kg

Table 1. (cont.)

Constituent/Parameter		WAC(s)	Vapor	Organic Liquid	Aqueous Liquid	Solid(s)	Required Analytical Detection Limit (Note 1)	Typical Analytical Detection Limit (Note 2)
Carbonate	CO3-	F/H Tank Farms				X		
Chloride	Cl-	SST, CIF		Note 4				
		SST, CIF			X			0.2 mg/L
		F/H Tank Farms				X	$\leq 0.11 \text{ M} \approx 3.8 \text{ g/L}$	
Chromium	Cr	SST, CIF		X			2500 mg/Kg	3 mg/L
		SST, CIF			X		2500 mg/Kg	0.006 mg/L
		F/H Tank Farms				X		63 mg/Kg
Copper	Cu	SST, CIF		X				4 mg/L
		SST, CIF			X			0.009 mg/L
		F/H Tank Farms				X		87 mg/Kg
Cyanide	CN-	SST, CIF, MWSF			X			
		Tank Farms, MWSF				X		
Fluoride	F-	F/H Tank Farms				X	$\leq 0.086 \text{ M} \approx 1600 \text{ mg/L}$	0.2 mg/L
Hydroxide, Free	OH-	F/H Tank Farms				X		
Iron	Fe	SST, CIF		X				3 mg/L
		SST, CIF			X			0.006 mg/L
		F/H Tank Farms				X		64 mg/Kg
Lead	Pb	SST, CIF		X			10,000 mg/Kg	26 mg/L
		SST, CIF			X		10,000 mg/Kg	0.052 mg/L
		F/H Tank Farms				X		520 mg/Kg
Manganese	Mn	SST, CIF		X				1 mg/L
		SST, CIF			X			0.001 mg/L
		Criticality				X		13 mg/Kg

Table 1. (cont.)

Constituent/Parameter		WAC(s)	Vapor	Organic Liquid	Aqueous Liquid	Solid(s)	Required Analytical Detection Limit (Note 1)	Typical Analytical Detection Limit (Note 2)
Mercury	Hg	SST, CIF		X			260 mg/Kg	100 mg/L
		SST, CIF			X		260 mg/Kg	0.2 mg/L
		F/H Tank Farms				X		2000 mg/Kg
Nickel	Ni	SST, CIF		X			600 mg/L	5 mg/L
		SST, CIF			X		600 mg/L	0.01 mg/L
		Criticality				X		96 mg/Kg
Nitrate	NO3-	SST, CIF			X			1 mg/L
		F/H Tank Farms				X	≤ 8.5 M ≈ 535,000 mg/L, Dependent on OH- and NO2- concentration.	
Nitrite	NO2-	F/H Tank Farms				X	Dependent on OH- and NO3-concentration.	1 mg/L
Oxalate	C2O4-	F/H Tank Farms				X		1 mg/L
Phosphate	(PO4) -3	SST, CIF			X			1 mg/L
		F/H Tank Farms				X		
Potassium	K	F/H Tank Farms				X		400 mg/Kg
Selenium	Se	SST, CIF		X			0.1 mg/L	3 mg/L
		SST, CIF			X		0.1 mg/L	0.005 mg/L
		F/H Tank Farms				X		50 mg/Kg
Silver	Ag	SST, CIF		X			7,300 mg/Kg	28 mg/L
		SST, CIF			X		7,300 mg/Kg	0.055 mg/L
		F/H Tank Farms				X	None detectable	550 mg/Kg
Sodium	Na	F/H Tank Farms				X		700 mg/Kg
Sulfate	(SO4) -2	F/H Tank Farms				X	≤ 0.18 M ≈ 17,000 mg /L	0.5 ppm
Thallium	Tl	SST, CIF		X			730 mg/Kg	30 mg/L
		SST, CIF			X		730 mg/Kg	0.06 mg/L

Table 1. (cont.)

Constituent/Parameter		WAC(s)	Vapor	Organic Liquid	Aqueous Liquid	Solid(s)	Required Analytical Detection Limit (Note 1)	Typical Analytical Detection Limit (Note 2)
Titanium	Ti	F/H Tank Farms				X		50 mg/Kg
Uranium	U	SST, CIF		X				18 mg/L
		SST, CIF			X			0.036 mg/L
Zinc	Zn	SST, CIF		X				1 mg/L
		SST, CIF			X			0.002 mg/L
		Criticality				X		16 mg/Kg
Crystallography		None, to support grout formulation				X		
TCLP		None, to support grout formulation				X		
Gas Constituents		None, for environmental, safety and health.	X					
Any known volatiles > 20 mg/L		F/H Tank Farms				Note 5		
n-Butanol		F/H Tank Farms						
Phenol/phenoxide/nitrophenols		SST, CIF, F/H Tank Farms		Note 6	Note 6	Note 6		
Semi-Volatile Organics		SST, CIF	X	Note 7	Note 7			
Total Organic Carbon		SST, CIF, F/H Tank Farms		X	X	X		
Volatile % Organics		F/H Tank Farms				X	< 0.5 vol%	
Volatile Organics		SST, CIF	Note 5	Note 5	Note 5			
PCBs		SST, CIF, MWSF		X	X	X	< 50 ppm	
Kepone, p-Nitrophenol		SST, CIF		Note 7	Note 7		Detectable (SST)	

Table 1. (cont.)

Constituent/Parameter	WAC(s)	Vapor	Organic Liquid	Aqueous Liquid	Solid(s)	Required Analytical Detection Limit (Note 1)	Typical Analytical Detection Limit (Note 2)
Benzene	SST, CIF		Note 5	Note 5			
MEK, PCE	SST, CIF		Note 5	Note 5			
Acidity	SST, CIF			X			
Boiling Point	SST, CIF		X	X		≥ 135°F	
BTU/lb	SST, CIF		X	X		≥ 5000	
Flash Point	SST, CIF		X	X		≥ 140°F	
Freezing Point	SST, CIF		X	X		≤ 40°F	
Paint Filter Liquids Test	SST, CIF		X	X			
pH	SST, CIF, MWSF			X		4 ≤ pH ≤ 12.5	
	F/H Tank Farms, MWSF				X	pH > 9.5	
Specific Gravity	SST, CIF, F/H Tank Farms		X	X	X	≤ 1.15	
Total Dissolved Solids	SST, CIF, F/H Tank Farms, MWSF		X	X	X		
Total Insoluble/Suspended Solids	SST, CIF, F/H Tank Farms, MWSF		X	X	X	≤ 10 wt% Total Suspended Solids	
Viscosity	SST, CIF		X	X		0.002-0.045 Pas @ 25°C	
Water, wt%	SST, CIF		X	X			0.002 wt%
Am-241	ES&H	Note 8				None Specified	
	SST, CIF, MWSF		X	X		1.00E-05 µCi /cc (Note 9)	9E-6 µCi /mL
	MWSF				X	1.00E-05 µCi /g (Note 9)	
Am Series, Total	F/H Tank Farms				Note 10		

Table 1. (cont.)

Constituent/Parameter	WAC(s)	Vapor	Organic Liquid	Aqueous Liquid	Solid(s)	Required Analytical Detection Limit (Note 1)	Typical Analytical Detection Limit (Note 2)
C-14	SST, CIF, MWSF		X	X		1.00E-05 $\mu\text{Ci/cc}$ (Note 9)	1E-5 $\mu\text{Ci/mL}$
	F/H Tank Farms, MWSF				X	1.00E-05 $\mu\text{Ci/g}$ (Note 9)	
Ce-144	ES&H	Note 8				None Specified	
	SST, CIF, MWSF		X	X		< 1% Total Activity (Note 11)	1E-5 $\mu\text{Ci/mL}$
	F/H Tank Farms, MWSF				X	< 1% Total Activity (Note 11)	
Cm Series	F/H Tank Farms				Note 12		1E-5 $\mu\text{Ci/mL}$
Co-60	ES&H	Note 8				None Specified	
	SST, CIF, MWSF		X	X		1.00E-04 $\mu\text{Ci/cc}$ (Note 9)	3E-6 $\mu\text{Ci/mL}$
	F/H Tank Farms, MWSF				X	1.00E-04 $\mu\text{Ci/g}$ (Note 9)	
Cs-134	F/H Tank Farms				X		2E-6 $\mu\text{Ci/mL}$
Cs-137	ES&H	Note 8				None Specified	
	SST, CIF, MWSF		X	X		1.00E-05 $\mu\text{Ci/cc}$ (Note 9)	4E-6 $\mu\text{Ci/mL}$
	F/H Tank Farms, MWSF				X	< 3.48E+03 dpm/ml	
Eu-154	F/H Tank Farms				X		3E-6 $\mu\text{Ci/mL}$
Gross Alpha	ES&H	X				None Specified	
	SST, CIF, MWSF		X	X		5 nCi/g = 5E-3 $\mu\text{Ci/g}$	9E-6 $\mu\text{Ci/mL}$
	F/H Tank Farms, MWSF				X		
H-3	ES&H	X				None Specified	
	SST, CIF, MWSF		X	X		1.00E-04 $\mu\text{Ci/cc}$ (Note 9)	1E-4 $\mu\text{Ci/mL}$
	F/H Tank Farms, MWSF				X	1.00E-04 $\mu\text{Ci/g}$ (Note 9)	

Table 1. (cont.)

Constituent/Parameter	WAC(s)	Vapor	Organic Liquid	Aqueous Liquid	Solid(s)	Required Analytical Detection Limit (Note 1)	Typical Analytical Detection Limit (Note 2)
I-129	SST, CIF, MWSF		X	X		1.00E-06 $\mu\text{Ci/cc}$ (Note 9)	1E-6 $\mu\text{Ci/mL}$
	F/H Tank Farms, MWSF				X	1.00E-06 $\mu\text{Ci/g}$ (Note 9)	
Nb-94	F/H Tank Farms				X		3E-6 $\mu\text{Ci/mL}$
Ni-59	SST, CIF, MWSF		X	X		1.00E-04 $\mu\text{Ci/cc}$ (Note 9)	1E-4 $\mu\text{Ci/mL}$
	F/H Tank Farms, MWSF				X	1.00E-04 $\mu\text{Ci/g}$ (Note 9)	
Ni-63	F/H Tank Farms				X		1E-7 $\mu\text{Ci/mL}$
Gross Beta	ES&H	X				None Specified	
	SST, CIF, MWSF		X	X		2600 nCi/g = 2.6 $\mu\text{Ci/g}$	9E-6 $\mu\text{Ci/mL}$
	F/H Tank Farms, MWSF				X		
Np-237	ES&H	Note 8				None Specified	
	SST, CIF, MWSF		X	X		1.00E-06 $\mu\text{Ci/cc}$ (Note 9)	5E-6 $\mu\text{Ci/mL}$
	F/H Tank Farms, MWSF				X	1.00E-06 $\mu\text{Ci/g}$ (Note 9)	
Plutonium, Total	F/H Tank Farms				Note 13		
Plutonium-238	SST, CIF, MWSF		X	X		1.00E-06 $\mu\text{Ci/cc}$ (Note 9)	1E-6 $\mu\text{Ci/mL}$
	F/H Tank Farms, MWSF				X	1.00E-06 $\mu\text{Ci/g}$ (Note 9)	
Plutonium-239	SST, CIF, MWSF		X	X		1.00E-06 $\mu\text{Ci/cc}$ (Note 9)	1E-6 $\mu\text{Ci/mL}$
	F/H Tank Farms, MWSF				X	1.00E-06 $\mu\text{Ci/g}$ (Note 9)	
Plutonium-240	SST, CIF, MWSF		X	X		1.00E-06 $\mu\text{Ci/cc}$ (Note 9)	1E-6 $\mu\text{Ci/mL}$
	F/H Tank Farms, MWSF				X	1.00E-06 $\mu\text{Ci/g}$ (Note 9)	
Plutonium-241	SST, CIF, MWSF		X	X		1.00E-05 $\mu\text{Ci/cc}$ or 1% Total Activity (Note 14)	1E-6 $\mu\text{Ci/mL}$
	F/H Tank Farms, MWSF				X	1.00E-05 $\mu\text{Ci/cc}$ or 1% Total Activity (Note 14)	

Table 1. (cont.)

Constituent/Parameter	WAC(s)	Vapor	Organic Liquid	Aqueous Liquid	Solid(s)	Required Analytical Detection Limit (Note 1)	Typical Analytical Detection Limit (Note 2)
Plutonium-242	SST, CIF, MWSF		X	X		1.00E-06 $\mu\text{Ci/cc}$ (Note 9)	
	F/H Tank Farms, MWSF				X	1.00E-06 $\mu\text{Ci/g}$ (Note 9)	
Pm-147	ES&H	Note 8				None Specified	
	SST, CIF, MWSF		X	X		< 1% Total Activity (Note 11)	1E-5 $\mu\text{Ci/mL}$
	F/H Tank Farms, MWSF				X	< 1% Total Activity (Note 11)	
Ru-106	ES&H	Note 8				None Specified	
	SST, CIF, MWSF		X	X		< 1% Total Activity (Note 11)	3E-5 $\mu\text{Ci/mL}$
	F/H Tank Farms, MWSF				X	< 1% Total Activity (Note 11)	
Sb-125	F/H Tank Farms				X		8E-6 $\mu\text{Ci/mL}$
Se-79	SST, CIF, MWSF		X	X		< 1% Total Activity (Note 11)	1E-5 $\mu\text{Ci/mL}$
	F/H Tank Farms, MWSF				X	< 1% Total Activity (Note 11)	
Sn-126	SST, CIF, MWSF		X	X		< 1% Total Activity (Note 11)	2E-6 $\mu\text{Ci/mL}$
	F/H Tank Farms, MWSF				X	< 1% Total Activity (Note 11)	
Sr-89	F/H Tank Farms				Note 15		
Sr-90	SST, CIF, MWSF		X	X		1.00E-04 $\mu\text{Ci/cc}$ (Note 9)	1E-5 $\mu\text{Ci/mL}$
	F/H Tank Farms, MWSF				X	1.00E-04 $\mu\text{Ci/g}$ (Note 9)	

Table 1. (cont.)

Constituent/Parameter	WAC(s)	Vapor	Organic Liquid	Aqueous Liquid	Solid(s)	Required Analytical Detection Limit (Note 1)	Typical Analytical Detection Limit (Note 2)
Tc-99	SST, CIF, MWSF		X	X		1.00E-06 $\mu\text{Ci/cc}$ (Note 9)	1E-6 $\mu\text{Ci/mL}$
	F/H Tank Farms, MWSF				X	1.00E-06 $\mu\text{Ci/g}$ (Note 9)	
Uranium-233	SST, CIF, MWSF		X	X		1.00E-06 $\mu\text{Ci/cc}$ or 1% Total Activity (Note 14)	1E-6 $\mu\text{Ci/mL}$
	F/H Tank Farms, MWSF				X	1.00E-06 $\mu\text{Ci/cc}$ or 1% Total Activity (Note 14)	
Uranium-234	SST, CIF, MWSF		X	X		1.00E-06 $\mu\text{Ci/cc}$ (Note 9)	1E-6 $\mu\text{Ci/mL}$
	F/H Tank Farms, MWSF				X	1.00E-06 $\mu\text{Ci/g}$ (Note 9)	
Uranium-235	SST, CIF, MWSF		X	X		1.00E-06 $\mu\text{Ci/cc}$ or 1% Total Activity (Note 14)	1E-6 $\mu\text{Ci/mL}$
	F/H Tank Farms, MWSF				X	1.00E-06 $\mu\text{Ci/cc}$ or 1% Total Activity (Note 14)	
Uranium-236	SST, CIF, MWSF		X	X		1.00E-06 $\mu\text{Ci/cc}$ or 1% Total Activity (Note 14)	1E-6 $\mu\text{Ci/mL}$
	F/H Tank Farms, MWSF				X	1.00E-06 $\mu\text{Ci/cc}$ or 1% Total Activity (Note 14)	
Uranium-238	SST, CIF, MWSF		X	X		1.00E-06 $\mu\text{Ci/cc}$ or 1% Total Activity (Note 14)	1E-6 $\mu\text{Ci/mL}$
	F/H Tank Farms, MWSF				X	1.00E-06 $\mu\text{Ci/cc}$ or 1% Total Activity (Note 14)	

Table 1 Notes:

1. Required detection limits are from the respective WACs.

2. Typical detection limits are listed for informational purposes, the actual detection limits obtained are sample-specific and may be higher or lower than those shown. The typical detection limits for the elemental analyses of the organic liquid assumed a 1 ml sample digested and diluted to 50 mL, with a 10-fold dilution prior to the spectroscopic measurement. The typical detection limits for the elemental analyses of the solid assumed a 0.25 g sample digested and diluted to 250 mL, with a 10-fold dilution prior to the spectroscopic measurement.
3. The required detection limit for these RCRA metals in the solid sample is assumed to be 20 times the leachate limit from the TCLP. The planned analyses will be total metal digestions, not leachings, these values account for the 20-fold excess used in the TCLP.
4. Chlorine in the organic phase will be determined by summing the chlorinated hydrocarbons identified in the VOC and SVOC analyses.
5. The Volatile Organic Compound analysis addresses these specific WAC requirements: benzene, methyl ethyl ketone, perchloroethylene, n-butanol, any known volatiles > 20 mg/L. It does not address methanol.
6. The addition of nitric acid to alkaline wastes may create nitrated organic compounds that did not originate in the sample.
7. The Semi-Volatile Organic Compound analysis addresses these specific WAC requirements: kepone and p-nitrophenol.
8. Gamma spectroscopy of the vapor phase cartridges will identify Cs-137 and other gamma emitting radionuclides in the vapor phase.
9. WAC 2.02 in the WSRC 1S Manual requires documentation of the technical justification for exceeding these Maximum Allowable Lower Limit of Detection values.
10. Total americium will be the sum of any identified Am-241 and Am-243.
11. WAC 2.02 in the WSRC 1S Manual defines a significance level of 1 Ci% for nuclides that do not have specific limits in WAC 3.17.
12. Total curium will be the sum of Cm-244 and any other Cm isotopes identified by alpha spectrometry.
13. Total plutonium will be the sum of all identified Pu isotopes.
14. The required detection limit is assumed to be the greater of 1 Ci % or the Maximum Allowable Lower Limit of Detection value.
15. Sr-89 will not be analyzed for due to the short half-life (50.5 days) and the age of the material in the tanks.

Table 2. Analytical Methods and Hold Times

Constituent/Parameter		Technique	L16.1 Procedure	Hold Time (per SW-846 ⁸)
Aluminate, Carbonate, and Free Hydroxide	AlO_2^- , CO_3^{2-} , OH^-	Titration	ADS-2312	
Aluminum, Antimony, Barium, Beryllium, Boron, Cadmium, Chromium, Copper, Iron, Lead, Manganese, Nickel, Silver, Sodium, Thallium, Titanium, Uranium, Zinc	Al, Sb, Ba, Be, B, Cd, Cr, Cu, Fe, Pb, Mn, Ni, Ag, Na, Tl, Ti, U, Zn	ICP-ES	ADS-1509	6 months
Ammonium, Chloride, Fluoride, Nitrate, Nitrite, Oxalate, Phosphate, Sulfate	NH_4^+ , Cl^- , F^- , NO_3^- , NO_2^- , $\text{C}_2\text{O}_4^{2-}$, $(\text{PO}_4)^{3-}$, $(\text{SO}_4)^{2-}$	IC	ADS-2306	Not specified in SW-846
Cyanide	CN^-		ADS-2278	
Arsenic, Selenium	As, Se	Hydride AAS	ADS-1557	6 months
Mercury	Hg	CVAA	ADS-1556	28 days
Potassium	K	Flame AAS	ADS-1549	6 months
Crystallography		XRD	ADS-1101	
TCLP		Modified TCLP	ADS-2512	
Gas Constituents		GC, GC/MS	ADS-1600	
Phenol/phenoxide/nitro phenols		HPLC	Task Technical Plan	Extract within 14 days, analyze within 40 days of extraction
Semi-Volatile Organics		GC/MS	ADS-2657	Extract within 14 days, analyze within 40 days of extraction
Total Organic Carbon		TOC	ADS-1200	Not specified in SW-846
Volatile % Organics		Oil Content Analyzer	ADS-2213	Not specified in SW-846
Volatile Organics		GC/MS	ADS-2656	14 days
PCBs		GC, GC/MS	ADS-2659	Extract within 14 days, analyze within 40 days of extraction
Acidity		Titration	ADS-2301	
Boiling Point			ADS-2280	
BTU/lb			ADS-2277	
Flash Point			ADS-2250	

Table 2 (cont.)

Constituent/Parameter	Technique	L16.1 Procedure	Hold Time (per SW-846 ⁸)
Freezing Point		ADS-2279	
Paint Filter Liquids Test		ADS-2275	Not specified in SW-846
pH		ADS-2309	Not specified in SW-846
Specific Gravity			
Total Dissolved, Insoluble/Suspended Solids		Standard Method 2540 ¹¹	
Viscosity		ADS-2276	
Water, wt%	Karl-Fischer	ADS-2210	
Am-241, Ce-144, Co-60, Cs-134, Cs-137, Eu-154, Nb-94, Np-237, Ru-106, Sb-125, Sn-126	Gamma PHA	ADS-2420	
Am Series, Total	Gamma and Alpha PHA	ADS-2420, ADS-2402	
C-14	Separation & LSC		
Cm Series	Alpha PHA	ADS-2402	
Gross Alpha	Gross Alpha	ADS-2405	Not specified in SW-846
H-3	Distillation & LSC	ADS-2444	
I-129	Separation & LEPS		
Ni-59	Separation & XRS	ADS-2452	
Ni-63	Separation & LSC	ADS-2452	
Gross Beta	LSC	ADS-2424	Not specified in SW-846
Plutonium, Total	Summation		
Pu-238, Pu-239, Pu-240, Pu-242, U-233, U-234, U-236, U-238	ICP-MS & Alpha PHA	ADS-1551, ADS-2402	
Plutonium-241	Separation & LSC		
Pm-147	Separation & LSC		
Se-79	Separation & LSC		

Table 2 (cont.)

Constituent/Parameter	Technique	L16.1 Procedure	Hold Time (per SW-846 ⁸)
Sr-90	Separation & LSC	ADS-2447	
Tc-99	Separation & LSC	ADS-2445	
Uranium-235	ICP-MS, Gamma PHA, Alpha PHA	ADS-1551, ADS- 2420, ADS-2402	

8. APPENDIX A. LIST OF ANALYTICAL PROCEDURES

L16.1 Procedure	Revision	Title	Year
ADS-1101	Rec. 3	Siemens D-500 Contained X-Ray Diffraction System (U)	1995
ADS-1509	Rev. 3	Inductively Coupled Plasma Emission Spectrometer for Aqueous Liquid Sample Analysis ARL 3580 (U)	1996
ADS-1549	Rev. 1	Procedure for Operating Varian SpectraAA-400 Atomic Absorption Spectrophotometer (U)	1997
ADS-1551	Rev. 0	Inductively Coupled Plasma-Mass Spectrometry Elemental and Isotopic Analysis for Aqueous Liquid Sample Fisons Plasmaquad I (U)	1996
ADS-1556	Rev. 0	Procedure for Cold Vapor Atomic Absorption Determination of Mercury on the Varian SpectraAA-400 Atomic Absorption Spectrophotometer (U)	1996
ADS-1557	Rev. 0	Procedure for Hydride Generation/Atomic Absorption (U)	1997
ADS-1600	Rev. 4	Procedure for Gas Chromatography (U)	1996
ADS-2210	Rev. 6	Water Determination with the Karl Fischer Reagent (U)	1997
ADS-2213	Rev. 4	Measurement of Oil and Grease in Aqueous Suspensions (U)	1996
ADS-2250	Rev. 2	Flashpoint Determination Using Petrolab Mini-Flash Flashpoint Tester (U)	1996
ADS-2275	Rev. 0	EPA SW-846 Method 9095 Paint Filter Liquids Test(U)	1997
ADS-2276	Rev. 0	Measurement of Viscosity using Glass Capillary Kinematic Opaque Viscosmeter	1997
ADS-2277	Rev. 0	Measurement of Heat of Combustion of Liquids by Bomb Calorimetry	1997
ADS-2278	Rev. 0	Measurement of Cyanide Ion in Aqueous Solutions	1997
ADS-2279	Rev. 0	Freezing Point Determination of Liquids	1997
ADS-2280	Rev. 0	Boiling Point Determination of Liquids	1997
ADS-2276	Rev. 0	Measurement of Viscosity using Glass Capillary Kinematic Opaque Viscosmeter	1997
ADS-2301	Rev. 5	Determination of Total Acid by Autotitration (U)	1996
ADS-2306	Rev. 6	Analysis of Solutions by Ion Chromatography (U)	1997
ADS-2309	Rev. 4	pH Determination of Aqueous Liquids (U)	1996
ADS-2312	Rev. 5	Automatic Determination of Hydroxide, Aluminate and Carbonate in Alkaline Solutions of Nuclear Waste (U)	1996
ADS-2401	Rev. 2	Beta Liquid Scintillation (U)	1995
ADS-2402	Rev. 2	Alpha Pulse Height Analysis (U)	1994
ADS-2405	Rev. 2	Alpha and Beta Plate Making Direct Mount and Count (U)	1995
ADS-2420	Rev. 3	Gamma Sample Preparation and Analysis (g-PHA) (U)	1997
ADS-2424	Rev. 4	Gross Alpha/Beta Determination by Liquid Scintillation Counting (U)	1996
ADS-2444	Rev. 1	Tritium in Environmental Samples - A Distillation Procedure	1996
ADS-2445	Rev. 1	Technicium-99 Extraction and Chromatography	1996
ADS-2447	Rev. 1	Strontium-90 in Environmental Samples	1996
ADS-2452	Rev. 0	Ni-59,63 in Environmental and High Activity Samples	1997

Appendix A (cont.)

L16.1 Procedure	Revision	Title	Year
ADS-2512	Rev. 2	Modified Toxic Characteristic Leaching Procedure	1996
ADS-2655	Rev. 2	High Performance Liquid Chromatography Operating Procedure (U)	1996
ADS-2656	Rev. 1	Gas Chromatography/Mass Spectrometry for Volatile Organics: Contract Laboratory Program Methods (U)	1996
ADS-2657	Rev. 1	Gas Chromatography/Mass Spectrometry for Semi-Volatile Organics: Contract Laboratory Program Methods (U)	1997
ADS-2659	Rev. 0	Gas Chromatography and Gas Chromatography/Mass Spectrometry for Polychlorinated Biphenyls Analysis: Contract Laboratory Program Methods (U)	1995