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Saltstone 2QCY15 TCLP Toxicity and UTS Results

D. H. Miller

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EXECUTIVE SUMMARY

A Saltstone Disposal Facility (SDF) waste form was prepared in the Savannah River National Laboratory (SRNL) from a Tank 50H sample and Z-Area premix material for the second quarter of calendar year 2015 (2QCY15). After a 28 day cure, a sample of the SDF waste form was collected, and shipped to a certified laboratory for Toxic Characteristic and Universal Treatment Standards (UTS) analysis. The metals analysis is performed using the Toxic Characteristic Leaching Procedure (TCLP)¹. The 2QCY15 saltstone sample results meet South Carolina Hazardous Waste Management Regulations (SCHWMR) R.61-79.261.24 and R.61-79.268.48(a) requirements for a nonhazardous waste form with respect to RCRA metals and underlying hazardous constituents (UHC).

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LIST OF ABBREVIATIONS

ARP	Actinide Removal Process
CVAA	Cold Vapor Atomic Absorption
DSS-HT	Decontaminated Salt Solution Hold Tank
E&CPT	Environmental and Chemical Process Technology
ESS-WP	Environmental Services Section – Waste Programs
ETF	Effluent Treatment Facility
ICP-AES	Inductively Coupled Plasma – Atomic Emission Spectroscopy
ISWLF	Industrial Solid Waste Landfill
LCS	Laboratory Control Sample
MCL	Maximum Contaminant Level
MCU	Modular Caustic Side Solvent Extraction Unit
MS	Matrix Spike
MSD	Matrix Spike Duplicate
RCRA	Resource Conservation and Recovery Act
RL	Reporting Limit
RPD	Relative Percent Differences
SCDHEC	South Carolina Department of Health and Environmental Control
SCHWMR	South Carolina Hazardous Waste Management Regulations
SDF	Saltstone Disposal Facility
SDG	Sample Delivery Group
SPF	Saltstone Production Facility
SWLF	Solid Waste Landfill
SWRI	Southwest Research Institute
SRNL	Savannah River National Laboratory
TCLP	Toxic Characteristic Leaching Procedure
UHC	Underlying Hazardous Constituent
UTS	Universal Treatment Standards
USEPA	United State Environmental Protection Agency
WAC	Waste Acceptance Criteria

1.0 Introduction

The Saltstone Production Facility (SPF) receives waste from Tank 50H for treatment. In the second quarter of the 2015 calendar year (2QCY15), Tank 50H accepted transfers of approximately 1.4 kgal from 211H, approximately 63.1 kgal from the Actinide Removal Process / Modular Caustic Side Solvent Extraction Unit (ARP/MCU) Decontaminated Salt Solution Hold Tank (DSS-HT), and approximately 22.8 kgal from ETF².

The Saltstone Grout Sampling plan provides the South Carolina Department of Health and Environmental Control (SCDHEC) with the chemical and physical characterization strategy for the salt solution, which is to be disposed of in the Z-Area Solid Waste Landfill (SWLF)³. During operation, a salt solution sample was collected from Tank 50H and used to prepare a SDF waste form sample, referred to as a saltstone sample. This saltstone sample determines the nonhazardous nature of the grout to meet the requirements of the SCDHEC South Carolina Hazardous Waste Management Regulations (SCHWMR) R.61-79.261.24 and R.61-79.268.48⁴.

Savannah River National Laboratory (SRNL) was requested to prepare saltstone from a Tank 50H salt solution sample obtained April 7, 2015 during 2QCY15 to determine the nonhazardous nature of the grout^{5,6}. The sample was cured and shipped to Southwest Research Institute (SWRI) to analyze for Toxicity and Universal Treatment Standards (UTS). The primary eight metals for analysis include arsenic, barium, cadmium, chromium, mercury, lead, selenium and silver. In addition, analytes required include underlying hazardous constituents (UHCs) antimony, beryllium, nickel, thallium, benzene, phenols, and total and amenable cyanide, which could not be eliminated from analysis by process knowledge⁷. The primary metals and the first four UHCs are extracted for analysis by the Toxic Characteristic Leaching Procedure (TCLP)¹.

2.0 Experimental Procedure

This section is a summary of the approach taken to prepare and characterize the saltstone samples. The saltstone sample preparation was performed at SRNL. Saltstone sample characterization was performed at SWRI in San Antonio, Texas. Figure 2-1 is a flowchart of the steps taken to prepare and characterize the saltstone samples.

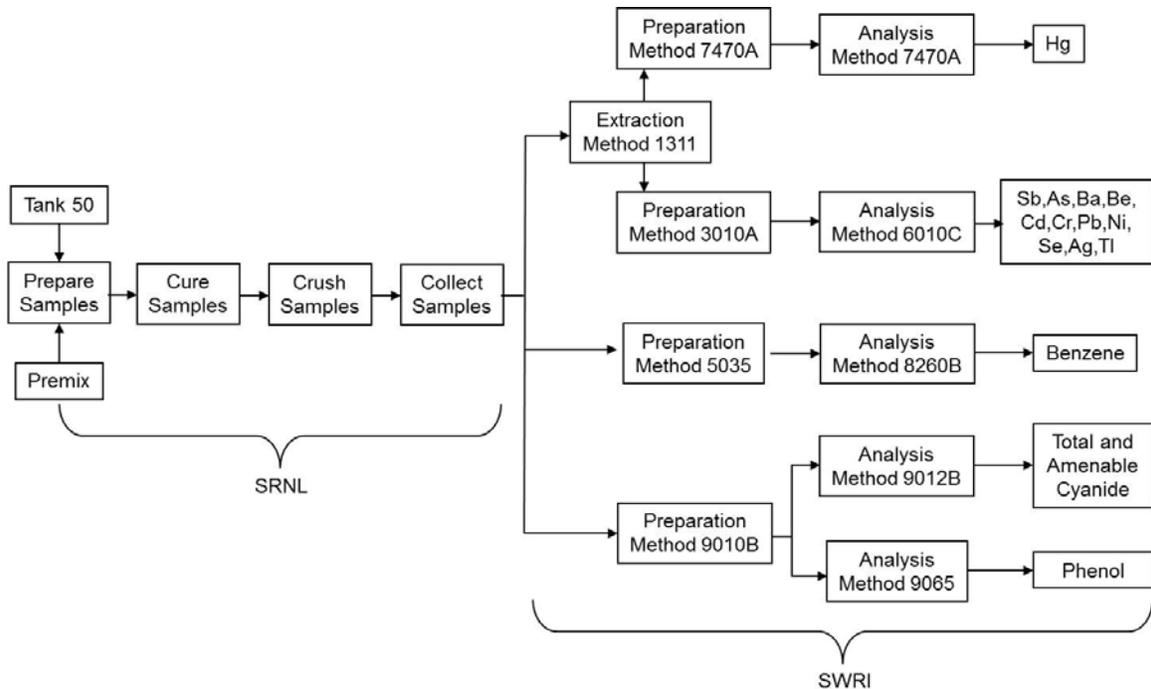


Figure 2-1: Saltstone Preparation and Analysis Flowsheet

2.1 Saltstone Sample Preparation

Saltstone sample preparation was performed at SRNL. The weight percent solids data used for the sample was taken from the 2QCY15 Waste Acceptance Criteria (WAC) analyses performed on Tank 50H⁸. Table 2-1 lists the concentration of Toxicity Characteristics and UTS of interest in the Tank 50H WAC sample⁹. Tank 50H 2QCY15 exceeds the regulatory limits for cadmium, chromium, mercury, silver, and phenol. Therefore, Tank 50H 2QCY15 salt solution must be processed and disposed of in a nonhazardous waste form. Table 2-2 contains the parameters used to prepare the saltstone sample².

The saltstone sample was prepared with the Tank 50H salt solution and a premix of cement, slag, and fly ash (obtained from the facility in February 2015). Figure 2-2 shows the formulation used to prepare the sample. The premix material was slowly added to the salt solution and mixed for approximately three minutes using a paddle blade mixer. The mixing was paused for approximately five seconds after 30 seconds of mixing to allow entrained air to escape from the grout. After the saltstone slurry was mixed, it was cast into a polyethylene zip top bag. The bag was laid flat and the air was expelled prior to sealing. The sample was cured flat in the bag to facilitate the size reduction step needed to conform to the particle size requirements of the TCLP method¹.

After curing for 28 days, the 2QCY15 saltstone sample was removed from the container and a portion of the saltstone was crushed to pass through a 9.5 mm (3/8 inch) standard sieve as prescribed by Section 7.1.3 of the TCLP method. The crushed saltstone was packaged into containers provided by Environmental Services Section – Waste Programs (ESS-WP). After the saltstone has been crushed, sieved and packaged, the sample is deemed “collected”¹⁰. ESS-WP retrieved the sample from SRNL and transported them to SWRI for extraction and analysis.

Table 2-1: Tank 50H 2QCY15 Salt Solution Results and Toxicity and UTS Limits

Analyte	Sample Result ⁸	Regulatory Limit		Unit
	2Q15	Toxicity ^a	UTS ^b	
Arsenic (As)	<0.110	5	5.0	mg/L
Barium (Ba)	<1.06	100	21	mg/L
Cadmium (Cd)	<2.04	1	0.11	mg/L
Chromium (Cr)	35.1	5	0.60	mg/L
Lead (Pb)	0.163	5	0.75	mg/L
Mercury (Hg)	118	0.2	0.025	mg/L
Selenium (Se)	0.0487	1.0	5.7	mg/L
Silver (Ag)	<2.36	5	0.14	mg/L
Antimony (Sb)	<0.035	-	1.15	mg/L
Beryllium (Be)	<0.225	-	1.22	mg/L
Nickel (Ni)	<7.92	-	11	mg/L
Thallium (Tl)	<0.0284	-	0.20	mg/L
Benzene	<0.150	0.5 mg/L	10	mg/kg
Amendable Cyanide	NM	-	30	mg/kg
Total Cyanide	NM	-	590	mg/kg
Total Phenol	<10.0	-	6.2	mg/kg

“-” Indicates a location in the table for which an entry would not be appropriate

NM – Not Measured

^aSCDHEC R.61-79.261.24 “Toxicity Characteristic”⁴

^bSCDHEC R.61-79.268.48 “Universal Treatment Standards”, Nonwastewater Standard⁴

Table 2-2: Customer Recommended Values for Saltstone Sample Preparation

Parameter	2QCY15
Water-to-Premix Ratio	0.59
(Daratard 17) gal/Ton Premix	0
(Dow Corning Q2-3183A) gal/Ton Premix	0.00

Saltstone Mix Data Sheet

MIX # 0160		Date: 4/16/2015	
Material	%	WT%	Grams
Waste Solution: Tank 50 4/7/15 2Q15 WT% Solids # <u>27.870</u> Grams Water <u>218.09</u>		44.97	302.36
Admixture: <u>Q2 Antifoam*</u>			0.00
Admixture: _____			0.00
Admixture: _____			
Premix		55.03	370.00
Cement (% of Premix)	10	5.50	37.00
Slag (% of Premix)	45	24.76	166.50
Fly Ash (% of Premix)	45	24.76	166.50
Total	100	100.00	672.36
Water to Premix Ratio	0.59		
Calculations: Use CBO fly ash From customer: 0.59 W/P No Antifoam 33 TPH Dry Feeds NO Daratard			

Figure 2-2: Saltstone Mix Data Sheet for the 2QCY15 Saltstone Sample

2.2 Saltstone Sample Testing

Saltstone sample testing was performed by SWRI. Activities associated with the 2QCY15 saltstone sample were TCLP extraction, TCLP leachate digestion and analysis, extraction of solid subsamples and extract analysis. The sample arrived at SWRI in San Antonio, Texas on May 19, 2015 for analysis. The samples were delivered with proper chain of custody documentation and signatures. All sample containers arrived without any visible signs of tampering or breakage, as noted in the SWRI report¹¹.

The volatile compound, benzene, was prepared by SW-846 Method 5035 and analyzed according to SW-846 Method 8260B. For total and amenable cyanide, the sample was prepared using SW-846 9010B and analyzed using Method 9012B. For total phenol, since the sample is a solid, Method SW-846 9065 (phenolics), a manual distillation and colorimetric procedure, was performed.

For the Toxicity Characteristic metals, the samples were extracted by a modified SW-846 Method 1311, using Extraction Fluid #2. The method was modified since a reduced sample mass (19.6 grams) was extracted due to its elevated sample activity. The extracts were prepared and analyzed for mercury by SW-846 Method 7470A. The extracts were digested according to SW-846 Method 3010A for the remaining metals. Those digestates were analyzed by ICP-AES, SW-846 Method 6010C.

2.3 Quality Assurance

Work performed on this task by Environmental and Chemical Process Technology (E&CPT) Research Programs personnel follows QA Manual (1Q) and associated implementing procedures for SRNL, as listed in Attachment 1 of the Task Technical and Quality Assurance Plan (TTQAP)⁶. Requirements for performing reviews of technical reports and the extent of review are established in Manual E.7 2.60. SRNL documents the extent and type of review using the SRNL Technical Report Design Checklist contained in WSRC-IM-2002-00011, Rev. 2.

3.0 Results and Discussion

The following tables summarize the analytical results provided by the vendor, SWRI. The entire vendor report is documented and included as a reference¹¹. Analytes that were analyzed for, but not detected, have been flagged with the “U” qualifier. This is based on required concentration dilution action and not necessarily the instrument detection limit. Analytes flagged with the “J” qualifier indicate an issue with the matrix spike recoveries or relative percent differences. In addition to the results, the reporting limit for each analyte has been given. The reporting limit (RL) is the lowest level at which an analyte may be accurately and reproducibly measured. For comparison, the previous quarter and four quarter average results are also shown. It should be noted that the qualifiers and reporting limits may vary for the previous values. Generally the previous values do not exhibit trends approaching regulatory limits. Mercury had been trending upward over the last several quarters. This quarter’s result was down, but still somewhat elevated compared to previous years.

Results in Table 3-1, when compared with the RL, can be organized into two groups:

- Detected at or below the RLs – arsenic, cadmium, chromium, lead, silver, beryllium, thallium, benzene, amenable cyanide, and total phenol
- Detected above the RLs – barium, mercury, selenium, antimony, nickel, and total cyanide

Table 3-1: 2QCY15 Saltstone Sample Toxicity Characteristic and UTS Results

Analyte	Result	Reporting Limit	Unit	Method	Results	
					Previous Quarter ^{R1}	Previous Four Quarter Average ^{R2}
Arsenic (As)	20.0 ^U	20.0	µg/L	1311/6010C	68.5	37.2* ⁺
Barium (Ba)	1510	5.0	µg/L	1311/6010C	713	902
Cadmium (Cd)	5.0 ^U	5.0	µg/L	1311/6010C	5.0 ^U	5.0 [^]
Chromium (Cr)	5.0 ^U	5.0	µg/L	1311/6010C	5.0 ^U	5.9* ⁺
Lead (Pb)	20.0 ^U	20.0	µg/L	1311/6010C	20.0 ^U	16.8* ⁺
Mercury (Hg)	12.8 ^D	0.8	µg/L	1311/7470A	15.6 ^{JD}	15.6* ⁺
Selenium (Se)	52.8	20.0	µg/L	1311/6010C	57.9	51.8 ⁺
Silver (Ag)	5.0 ^U	5.0	µg/L	1311/6010C	5.0 ^U	5.0 [^]
Antimony (Sb)	25.3	20.0	µg/L	1311/6010C	20.0 ^U	29.7* ⁺
Beryllium (Be)	5.0 ^U	5.0	µg/L	1311/6010C	5.0 ^U	5.0 [^]
Nickel (Ni)	59.7	5.0	µg/L	1311/6010C	14.4	19.5* ⁺
Thallium (Tl)	25.0 ^U	25.0	µg/L	1311/6010C	20.0 ^U	25.0* ⁺
Benzene	0.98 ^U	0.98	µg/kg	EPA 8260B	1.0 ^U	1.0* ⁺
Amenable Cyanide	0.341 ^U	0.341	mg/kg	EPA 9012B	0.427 ^U	0.388* ⁺
Total Cyanide	16.4 ^J	0.341	mg/kg	EPA 9012B	5.05 ^J	10.8* ⁺
Total Phenol	0.958 ^{UJ}	0.958	mg/kg	EPA 9065	0.680 ^{UJ}	0.842* ⁺

^U Compound not detected above the RL

^J Matrix or matrix spike duplicates criteria not met

^D Results is reported from a dilution

^{R1} See Reference 12 for data

* Contains qualifier of Q or J in at least one quarter

⁺ Contains multiple RLs

[^] Contains qualifier of Q in all quarters with same RL

^{R2} See References 12,13,14,and 15 for data

3.1 Comparison of Results to Regulatory Limits

Saltstone results from Table 3-1 are replicated in Table 3-2; however, units are converted from µg/L to mg/L and compared to the regulatory limits that may be applied to the SDF waste form. Table 3-2 includes the SCHWMR R.61-79.261.24(b) limits a waste is characteristically considered hazardous for toxicity and the SCHWMR R.61-79.268.48 UTS limits for underlying hazardous constituents. In addition, Maximum Contaminant Levels (MCL) from the State Primary Drinking Water Regulations⁴ are included in Table 3-2. By comparing the saltstone sample results and the regulatory limits the following conclusions can be made:

- The SDF waste form was not characteristically hazardous for toxicity.
- The analyte concentrations were below the UTS Non-wastewater Standard.
- Barium, chromium, and silver were below the MCL.
- Arsenic, cadmium, lead, mercury, selenium, antimony, beryllium and thallium exceed the MCL.
- Nickel, benzene, amenable cyanide, total cyanide, and total phenol do not have a MCL.

The MCL is the limit for a constituent in drinking water and used to determine the class of landfill required. At 10x MCL, a Class 3 landfill is required. The Salt Disposal Facility (SDF) vaults are permitted as a Class 3 landfill. Thallium equals 10x the MCL, because the reporting limit is used as the analytical result.

Table 3-2: 2QCY15 Saltstone Sample Results and Regulatory Limits

Analyte	Result	Unit	Regulatory Limit			Results	
			Toxicity ^a	UTS ^b	MCL ^c	Previous Quarter ^{R1}	Previous Four Quarter Average ^{R2}
Arsenic (As)	0.020 ^U	mg/L	5	5	0.010	0.0685	0.0372 ^{*+}
Barium (Ba)	1.51	mg/L	100	21	2	0.713	0.902
Cadmium (Cd)	0.005 ^U	mg/L	1	0.11	0.005	0.005 ^U	0.0050 [^]
Chromium (Cr)	0.005 ^U	mg/L	5	0.6	0.1	0.005 ^U	0.0059 [*]
Lead (Pb)	0.020 ^U	mg/L	5	0.75	0.015 ^d	0.020 ^U	0.0168 ^{*+}
Mercury (Hg)	0.0128 ^D	mg/L	0.2	0.025	0.002	0.0156 ^{JD}	0.0156 ^{*+}
Selenium (Se)	0.0528	mg/L	1	5.7	0.05	0.0579	0.0518 ⁺
Silver (Ag)	0.005 ^U	mg/L	5	0.14	0.1 ^e	0.005 ^U	0.0050 [^]
Antimony (Sb)	0.0253	mg/L	-	1.15	0.006	0.020 ^U	0.0297 ^{*+}
Beryllium (Be)	0.005 ^U	mg/L	-	1.22	0.004	0.005 ^U	0.0050 [^]
Nickel (Ni)	0.0597	mg/L	-	11	-	0.0144	0.0195 ^{*+}
Thallium (Tl)	0.025 ^U	mg/L	-	0.20	0.002	0.020 ^U	0.0250 ^{*+}
Benzene	0.001 ^U	mg/kg	-	10	-	0.001 ^U	0.001 ^{*+}
Amenable Cyanide	0.341 ^U	mg/kg	-	30	-	0.427 ^U	0.388 ^{*+}
Total Cyanide	16.4 ^J	mg/kg	-	590	-	5.05 ^J	10.8 ^{*+}
Total Phenol	0.958 ^{UJ}	mg/kg	-	6.2	-	0.680 ^{UJ}	0.842 ^{*+}

“-” Indicates a location in the table for which an entry would not be appropriate.

^U Compound was analyzed for, but not detected above the RL

^J MS/MSD recoveries were not within specification

^D Results reported from a dilution

^a SCDHEC R.61-79.261.24(b) “Characteristic of Toxicity”

^b SCDHEC R.61-79.268.48 “Universal Treatment Standards”, Non- waste water standard

^c SCDHEC R.61-58.5(B) “Maximum Contaminant Levels for Inorganic Chemicals”

^d Lead action level from SCDHEC R.61-58.11.B

^e Secondary drinking water parameter

^{*} Contains qualifier of Q or J in at least one quarter

⁺ Contains multiple RLs

[^] Contains qualifier of Q in all quarters with same RL

^{R1} See Reference 12 for data

^{R2} See References 12, 13, 14, and 15 for data

3.2 Analytical Quality Control

The following subsections include summaries of results from blanks, laboratory control samples, matrix spikes, and matrix spike duplicates. The data package also includes data for calibration verifications, interference checks, and serial dilutions¹¹.

3.2.1 Blanks

Blank concentrations are given in Table 3-3. In the Method Blanks, all the analyte concentrations were below the RLs.

Table 3-3: Method Blanks

Analyte	Blank	Unit
Arsenic (As)	20.0 ^U	µg/L
Barium (Ba)	5.0 ^U	µg/L
Cadmium (Cd)	5.0 ^U	µg/L
Chromium (Cr)	5.0 ^U	µg/L
Lead (Pb)	20.0 ^U	µg/L
Mercury (Hg)	0.20 ^U	µg/L
Selenium (Se)	20.0 ^U	µg/L
Silver (Ag)	5.0 ^U	µg/L
Antimony (Sb)	20.0 ^U	µg/L
Beryllium (Be)	5.0 ^U	µg/L
Nickel (Ni)	5.0 ^U	µg/L
Thallium (Tl)	25.0 ^U	µg/L
Benzene	0.5 ^U	µg/kg
Amenable	0.458 ^U	mg/kg
Total Cyanide	0.458 ^U	mg/kg
Total Phenol	0.987 ^U	mg/kg

^U Compound was analyzed for, but not detected above the RL

3.2.2 Laboratory Control Samples

Results from the Laboratory Control Sample (LCS) are given in Table 3-4. The LCS post spike recoveries met USEPA SW-846 acceptance limits for all elements. Laboratory Control Samples are clean aqueous solutions analyzed to assure integrity of the analytical technique exclusive of matrix effects.

Table 3-4: Laboratory Control Samples

Analyte	LCS		Unit	Recovery (%)
	True	Recovery		
Arsenic (As)	4000	3860	µg/L	96.5
Barium (Ba)	4000	3890	µg/L	97.2
Cadmium (Cd)	100	98.3	µg/L	98.3
Chromium (Cr)	400	383	µg/L	95.8
Lead (Pb)	1000	934	µg/L	93.4
Mercury (Hg)	1.00	0.98	µg/L	98.1
Selenium (Se)	4000	3680	µg/L	92.0
Silver (Ag)	100	94.6	µg/L	94.6
Antimony (Sb)	1000	979	µg/L	97.9
Beryllium (Be)	100	97.1	µg/L	97.1
Nickel (Ni)	1000	955	µg/L	95.5
Thallium (Tl)	4000	3880	µg/L	97.0
Benzene	10	7.5	µg/kg	75
Amenable Cyanide	NA	NA	mg/kg	NA
Total Cyanide	53.9	57.1	mg/kg	106
Total Phenol	NA	NA	mg/kg	NA

NA – Not Applicable

3.2.3 Matrix Spikes

Results from analysis of the matrix spike (MS) and matrix spike duplicates (MSD) are given in Table 3-5. These results show that:

- The percent recoveries (%R) obtained from the MS analyses met the recommended quality control acceptance criteria for percent recoveries, 75 – 125% (70 – 130% for benzene), for all applicable analytes except for phenol. Post digestion spikes were performed for total phenol and found to be acceptable.
- The percent recoveries (%R) obtained from the MSD analyses met the recommended quality control acceptance criteria for percent recoveries, 75 – 125% (70 – 130% for benzene), for all applicable analytes except benzene, phenol and total cyanide. Post digestion spikes were performed for total cyanide and phenol and found to be acceptable.
- The Relative Percent Difference(s) (RPD) between the MS and MSD met the acceptance limits (0 – 20%) for all metals. Phenol and total cyanide were outside the limits but the duplicates were acceptable.

Table 3-5: Matrix Spike and Matrix Spike Duplicate Results

Analyte	Unit	Initial Concentration		Spiked Sample		Recovery (%)		
		Sample	Spike Added	Spike	Spike Duplicate	Spike	Spike Duplicate	
Arsenic (As)	µg/L	20.0*	2500	2560	2520	102.4	100.8	
Barium (Ba)	µg/L	1510	5000	5840	5760	86.6	85.0	
Cadmium (Cd)	µg/L	5.0*	500	441	434	88.2	86.8	
Chromium (Cr)	µg/L	5.0*	1000	850	836	85.0	83.6	
Lead (Pb)	µg/L	20.0*	2500	2100	2060	84.0	82.4	
Mercury (Hg)	µg/L	12.8 ^D	4.0	16.8	16.7	100.0	97.5	
Selenium (Se)	µg/L	52.8	2500	2520	2480	98.7	97.1	
Silver (Ag)	µg/L	5.0*	500	478	472	95.6	94.4	
Antimony (Sb)	µg/L	25.3	5000	4970	4910	98.9	97.7	
Beryllium (Be)	µg/L	5.0*	500	460	453	92.0	90.6	
Nickel (Ni)	µg/L	59.7	2500	2150	2110	83.6	82.0	
Thallium (Tl)	µg/L	25.0*	2500	2230	2180	89.2	87.2	
Benzene ^J	µg/kg	0.98*	20	19	26	25	130	132
Amenable Cyanide	mg/kg	0.341*	NA		NA	NA	-	-
Total Cyanide ^J	mg/kg	16.4	23.5	14.3	37.9	24.7	91.5	58.0
Phenol ^J	mg/kg	0.958*	20.4	21.9	0.816	1.03	0.0	4.70

NA - Not Applicable

“-“ Indicates a location in the table for which an entry would not be appropriate.

* Compound was analyzed for, but was not detected above the RL on original sample

^J Matrix spike/matrix spike duplicate recovery is outside specified limits.

^D Result reported from a dilution

3.2.4 Duplicates

Results from the duplicates testing are shown in Table 3-6. The RPD(s) between the sample and sample duplicate met the acceptance limit. Phenol and amenable cyanide are reported as zero since the initial values were below the detection limit.

Table 3-6: Duplicates and Relative Percent Difference

Analyte	Unit	Initial Sample	Duplicate Sample	RPD
Arsenic (As)	µg/L	20.0 ^U	20.0 ^U	-
Barium (Ba)	µg/L	1510	1490	1.3
Cadmium (Cd)	µg/L	5.0 ^U	5.0 ^U	-
Chromium (Cr)	µg/L	5.0 ^U	5.0 ^U	-
Lead (Pb)	µg/L	20.0 ^U	20.0 ^U	-
Mercury (Hg)	µg/L	12.8	13.0	1.6
Selenium (Se)	µg/L	52.8	49.8	5.8
Silver (Ag)	µg/L	5.0 ^U	5.0 ^U	-
Antimony (Sb)	µg/L	25.3	30.1	17.3
Beryllium (Be)	µg/L	5.0 ^U	5.0 ^U	-
Nickel (Ni)	µg/L	59.7	58.7	1.7
Thallium (Tl)	µg/L	25.0 ^U	25.0 ^U	-
Benzene	µg/kg	0.98 ^U	-	-
Amenable Cyanide	mg/kg	0.341 ^U	0.594 ^U	0.0
Total Cyanide	mg/kg	16.4 ^J	16.4	0.0
Total Phenol	mg/kg	0.958 ^{UJ}	0.970 ^U	0.0

“-“ Indicates a location in the table for which an entry would not be appropriate.

^U Compound was analyzed for, but not detected above the RL

^J Matrix spike/matrix spike duplicate recovery is outside specified limits.

3.2.5 Calibration Information

- All initial calibration requirements have been met for this sample delivery group (SDG).
- All interference check samples associated with this SDG met the established acceptance criteria.
- All continuing calibration blanks bracketing this batch met the established acceptance criteria.
- All initial and continuing calibration verifications bracketing this SDG met the acceptance criteria.

4.0 Conclusions

Preparation of the 2QCY15 saltstone samples and the subsequent analyses showed that:

- The SDF waste form prepared from the 2QCY15 Tank 50H WAC sample and premix was not characteristically hazardous for toxicity.
- The concentrations of the eight RCRA metals and UHCs in the 2QCY15 SDF waste form were present at levels below the UTS limits.
- Analyses met all USEPA SW-846 quality assurance requirements. All other limits on holding times, laboratory control sample recoveries, matrix spike recoveries, serial dilution results when applicable, calibration verification, and interference checks were within the quality assurance requirements.

5.0 References

1. "Toxicity Characteristic Leaching Procedure", EPA SW-846, Procedure 1311.
2. Miller, D. H., "2Q15 Saltstone TCLP", Experiment T8786-00095-07, SRNL E-Notebook (Production); Savannah River National Laboratory, Aiken, SC 29808, (2014).
3. Liner, K. R., "Saltstone Grout Sampling", Savannah River Site, ESH-EPG-2004-00318, Rev. 0, December 2004.
4. South Carolina Code of Regulations, Chapter 61-Department of Health and Environmental Control.
5. Freed, E. J., "Technical Task Request: Routine Saltstone Support - FY2015", Savannah River Remediation, X-TTR-Z-00005, Rev. 1, October 2014.
6. Miller, D. H., "Task Technical and Quality Assurance Plan for SRNL Support of TCLP Preparation and Analysis for Saltstone - FY2015", Savannah River National Laboratory, SRNL-RP-2014-01020, Rev. 0, September 2014.
7. Britt, T. E., "Assessment of Regulated Organics Under 40 CFR Part 268, Section 49, Universal Treatment Standards, Relative to SRS Tank Farm Waste," Savannah River Site, LWO-LWE-2007-00052, Rev 0, March 2007.
8. Crawford, C. L., "2QCY15 Tank 50 WAC", Experiment B9108-00026-22, SRNL E-Notebook (Production); Savannah River National Laboratory, Aiken, SC 29808, (2015).
9. Crawford, C. L., "Tables Containing Results for the Second Quarter Calendar Year 2015 Tank 50 WAC Slurry Sample: Chemical and Radionuclide Contamination Results", Savannah River National Laboratory, SRNL-L3100-2015-00107, Rev. 0, July 2015.
10. Miller, D. H., "Definition of TCLP Sample Term Collected", Savannah River National Laboratory, SRNL-L3100-2015-00081, Rev. 0, April 2015.
11. Miller, D. H., "Data Package from Vendor for 2QCY15 Saltstone TCLP Analysis", Savannah River National Laboratory, SRNL-L3100-2015-00124, Rev. 0, July 2015.
12. Miller, D. H., "Saltstone 1QCY15 TCLP Toxicity and UTS Results", SRNL-STI-2015-00264 Rev. 0, Savannah River National Laboratory, July 2015.
13. Miller, D. H., "Saltstone 4QCY14 TCLP Toxicity and UTS Results", SRNL-STI-2015-00106 Rev. 0, Savannah River National Laboratory, April 2015.
14. Miller, D. H., "Saltstone 3QCY14 TCLP Results", SRNL-STI-2014-00595 Rev. 0, Savannah River National Laboratory, December 2014.
15. Miller, D. H., "Saltstone 2QCY14 TCLP Results", SRNL-STI-2014-00487 Rev. 0, Savannah River National Laboratory, October 2014.

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