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Saltstone 4QCY14 TCLP Toxicity and UTS Results

D. H. Miller

April 2015

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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 fourth quarter of calendar year 2014 (4QCY14). After a 47 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 4QCY14 saltstone sample results show that the saltstone is Resource Conservation Recovery Act (RCRA) nonhazardous, but is greater than the universal treatment standard for land disposal. The Saltstone Production Facility (SPF) and SDF were in a maintenance outage during the 4QCY14. Thus no processing or disposal of saltstone, as characterized by this 4QCY14 sample, occurred.

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LIST OF ABBREVIATIONS

ARP	Actinide Removal Process
CVAA	Cold Vapor Atomic Absorption
DSS-HT	Decontaminated Salt Solution Hold Tank
ESS-WP	Environmental Services Section – Waste Programs
ETP	Effluent Treatment Project
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 fourth quarter of the 2014 calendar year (4QCY14), Tank 50H accepted transfers of approximately 1.5 kgal from 211H, approximately 188.1 kgal from the Actinide Removal Process / Modular Caustic Side Solvent Extraction Unit (ARP/MCU) Decontaminated Salt Solution Hold Tank (DSS-HT), and approximately 9.2 kgal from 512-S².

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 saltstone 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 October 2, 2014 during 4QCY14 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 Procedure¹.

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.

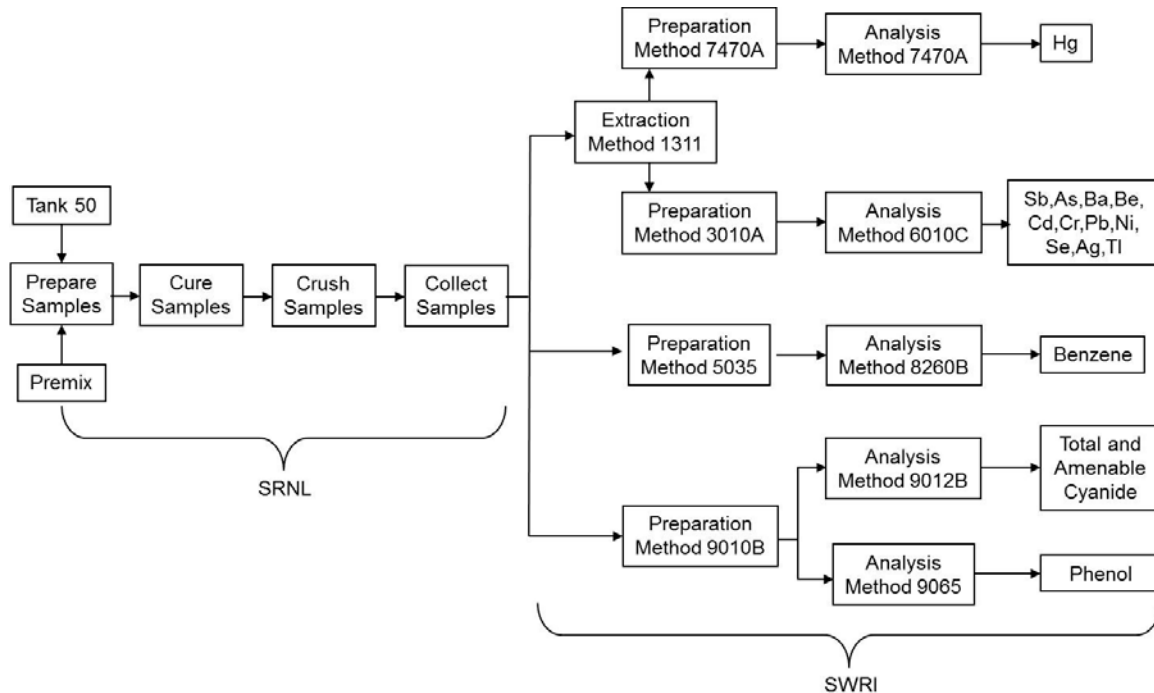


Figure 2-1: Saltstone Preparation and Analysis Flowsheet

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 4QCY14 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 sample⁸. Tank 50H 4QCY14 exceeds the regulatory limits for cadmium, chromium, mercury, and phenol. Therefore Tank 50H 4QCY14 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. 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 a polypropylene bag to facilitate the size reduction step needed to conform to the particle size requirements of the TCLP method¹.

After curing for 47 days, the 4QCY14 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”⁹. ESS-WP retrieved the sample from SRNL and transported them to SWRI for extraction and analysis.

Table 2-1: Tank 50H 4QCY14 Salt Solution Results and Toxicity and UTS Limits

Analyte	Sample Result ⁸	Regulatory Limit		Unit
	4Q14	Toxicity ^a	UTS ^b	
Arsenic (As)	<0.0937	5.0	5.0	mg/L
Barium (Ba)	<0.900	100.0	21	mg/L
Cadmium (Cd)	<1.73	1.0	0.11	mg/L
Chromium (Cr)	33.8	5.0	0.60	mg/L
Lead (Pb)	0.222	5.0	0.75	mg/L
Mercury (Hg)	79.3	0.2	0.025	mg/L
Selenium (Se)	<0.187	1.0	5.7	mg/L
Silver (Ag)	<2.01	5.0	0.14	mg/L
Antimony (Sb)	<0.0298	-	1.15	mg/L
Beryllium (Be)	<0.191	-	1.22	mg/L
Nickel (Ni)	<6.73	-	11	mg/L
Thallium (Tl)	<0.0242	-	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	4QCY14
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 # 0153		Date: 10/21/2014	
Material	%	WT%	Grams
Waste Solution: Tank 50 10/02/14 4Q14 Wt% Solids # 28.090 Grams Water 208.90		45.00	290.51
Admixture: Q2 Antifoam*			0.00
Admixture:			0.00
Admixture:			
Premix		55.00	355.00
Cement (% of Premix)	10	5.50	35.50
Slag (% of Premix)	45	24.75	159.75
Fly Ash (% of Premix)	45	24.75	159.75
Total	100	100.00	645.51
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 4QCY14 Saltstone Sample

2.2 Saltstone Sample Testing

Saltstone sample testing was performed by SWRI. Activities associated with the 4QCY14 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 December 10, 2014 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 report¹⁰.

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. The method was modified since a reduced sample mass 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

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 reference¹⁰. Analytes that were analyzed for, but not detected, have been flagged with the “U” qualifier. This is based on necessary 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. Post digestion spike recoveries were performed for these analytes as confirmation of results. 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.

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

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

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

Analyte	Result	Qualifier	Reporting Limit	Unit	Method
Arsenic (As)	16.2		10.0	µg/L	1311/6010C
Barium (Ba)	112		5.0	µg/L	1311/6010C
Cadmium (Cd)	5.0	U	5.0	µg/L	1311/6010C
Chromium (Cr)	8.79		5.0	µg/L	1311/6010C
Lead (Pb)	25.0	U	25.0	µg/L	1311/6010C
Mercury (Hg)	29.4		2.0	µg/L	1311/7470A
Selenium (Se)	72.6		20.0	µg/L	1311/6010C
Silver (Ag)	5.0	U	5.0	µg/L	1311/6010C
Antimony (Sb)	40.0	U	40.0	µg/L	1311/6010C
Beryllium (Be)	5.0	U	5.0	µg/L	1311/6010C
Nickel (Ni)	5.0	U	5.0	µg/L	1311/6010C
Thallium (Tl)	40.0	U	40.0	µg/L	1311/6010C
Benzene	1.1	U	1.1	µg/kg	EPA 8260B
Amenable Cyanide	0.456	U	0.456	mg/kg	EPA 9012B
Total Cyanide	13.5		0.456	mg/kg	EPA 9012B
Total Phenol	0.900	U	0.900	mg/kg	EPA 9065

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

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 SCDHEC SCHWMR R.61-79.261.24 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 with the exception of mercury.
- 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. Mercury slightly exceeds 10x of the MCL. Thallium exceeds 10x the MCL because the reporting limit is used as the analytical result.

Table 3-2: 4QCY14 Saltstone Sample Results and Regulatory Limits

Analyte	Result	Qualifier	Unit	Regulatory Limit		
				Toxicity ^a	UTS ^b	MCL ^c
Arsenic (As)	0.016		mg/L	5.0	5.0	0.010
Barium (Ba)	0.112		mg/L	100.0	21	2.0
Cadmium (Cd)	0.005	U	mg/L	1.0	0.11	0.005
Chromium (Cr)	0.00879		mg/L	5.0	0.60	0.1
Lead (Pb)	0.025	U	mg/L	5.0	0.75	0.015 ^d
Mercury (Hg)	0.029		mg/L	0.2	0.025	0.002
Selenium (Se)	0.0726		mg/L	1.0	5.7	0.05
Silver (Ag)	0.005	U	mg/L	5.0	0.14	0.1 ^e
Antimony (Sb)	0.040	U	mg/L	-	1.15	0.006
Beryllium (Be)	0.005	U	mg/L	-	1.22	0.004
Nickel (Ni)	0.005	U	mg/L	-	11	-
Thallium (Tl)	0.040	U	mg/L	-	0.20	0.002
Benzene	0.0011	U	mg/kg	0.5 mg/L	10	-
Amenable Cyanide	0.456	U	mg/kg	-	30	-
Total Cyanide	13.5		mg/kg	-	590	-
Total Phenol	0.90	U	mg/kg	-	6.2	-

“-“ 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

^a SCDHEC R.61-79.261.24 “Toxicity Characteristics”⁴

^b SCDHEC R 61-79.268.48 “Universal Treatment Standards”, Nonwastewater Standard⁴

^c SCDHEC R.61-58.5(B) “Maximum Contaminant Levels for Inorganic Chemicals”⁴

^d SCDHEC R.61-58.11.B “Control of Lead and Copper”, General Requirements⁴

^e SCDHEC R.61-58.5.R “Maximum Contaminant Levels in Drinking Water”, Secondary Maximum Contaminant Levels⁴

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 dilutions.¹⁰

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	Qualifier
Arsenic (As)	10.0	µg/L	U
Barium (Ba)	5.0	µg/L	U
Cadmium (Cd)	5.0	µg/L	U
Chromium (Cr)	5.0	µg/L	U
Lead (Pb)	25.0	µg/L	U
Mercury (Hg)	0.20	µg/L	U
Selenium (Se)	20.0	µg/L	U
Silver (Ag)	5.0	µg/L	U
Antimony (Sb)	40.0	µg/L	U
Beryllium (Be)	5.0	µg/L	U
Nickel (Ni)	5.0	µg/L	U
Thallium (Tl)	40.0	µg/L	U
Benzene	0.6	mg/kg	U
Amenable Cyanide	0.476	mg/kg	U
Total Cyanide	0.476	mg/kg	U
Total Phenol	0.973	mg/kg	U

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	3870	µg/L	96.8
Barium (Ba)	4000	3600	µg/L	90.0
Cadmium (Cd)	100	98.3	µg/L	98.3
Chromium (Cr)	400	377	µg/L	94.2
Lead (Pb)	1000	933	µg/L	93.3
Mercury (Hg)	1.0	0.965	µg/L	96.5
Selenium (Se)	4000	3760	µg/L	94.0
Silver (Ag)	100	89.6	µg/L	89.6
Antimony (Sb)	1000	992	µg/L	99.2
Beryllium (Be)	100	98.0	µg/L	98.0
Nickel (Ni)	1000	904	µg/L	90.4
Thallium (Tl)	4000	3940	µg/L	98.5
Benzene	10	10	µg/kg	100
Amenable Cyanide	NA	NA	mg/kg	NA
Total Cyanide	76.5	67.1	mg/kg	87.7
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 and Table 3-6, respectfully. 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 total cyanide and 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 total cyanide and phenol. Post digestion spikes were performed for total phenol and found to be acceptable.
- The RPD(s) between the MS and MSD met the acceptance limits (0 – 30%).
- The RPD(s) between the Sample and Sample Duplicate met the acceptance limits. Phenol and amenable cyanide are reported as zero since the initial values were below the detection limit.

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	16.2	2500		2670	2670	106.2	106.2
Barium (Ba)	µg/L	112	5000		4190	4210	81.6	82.0
Cadmium (Cd)	µg/L	5.0*	500		489	491	97.8	98.2
Chromium (Cr)	µg/L	8.79	1000		905	907	89.6	89.8
Lead (Pb)	µg/L	25.0*	2500		2290	2290	91.6	91.6
Mercury (Hg)	µg/L	-	-		-	-	-	-
Selenium (Se)	µg/L	72.6	2500		2640	2650	102.7	103.1
Silver (Ag)	µg/L	5.0*	500		387	382	77.4	76.4
Antimony (Sb)	µg/L	40.0*	5000		5120	5150	102.4	103.0
Beryllium (Be)	µg/L	5.0*	500		497	498	99.4	99.6
Nickel (Ni)	µg/L	5.0*	2500		2190	2190	87.6	87.6
Thallium (Tl)	µg/L	40.0*	2500		2450	2460	98.0	98.4
Benzene	µg/kg	1.1 *	21	20	20	22	95	110
Amenable Cyanide	mg/kg	0.456 *	NA		NA	NA	-	-
Total Cyanide ^J	mg/kg	13.5	24.8	21.1	22.1	25.6	34.7	57.3
Phenol ^J	mg/kg	0.900*	21.5	22.2	0.859	0.887	0.0	0.0

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.

Table 3-6: Duplicates and Relative Percent Difference

Analyte	Unit	Qualifier	Initial Sample	Duplicate Sample	RPD
Arsenic (As)	µg/L		16.2	16.8	3.6
Barium (Ba)	µg/L		112	112	0
Cadmium (Cd)	µg/L	U	5.0	5.0	-
Chromium (Cr)	µg/L		8.79	9.84	11.3
Lead (Pb)	µg/L	U	25.0	25.0	-
Mercury (Hg)	µg/L				-
Selenium (Se)	µg/L		72.6	70.9	2.4
Silver (Ag)	µg/L	U	5.0	5.0	-
Antimony (Sb)	µg/L	U	40	40	-
Beryllium (Be)	µg/L	U	5.0	5.0	-
Nickel (Ni)	µg/L	U	5.0	5.0	-
Thallium (Tl)	µg/L	U	40.0	40.0	-
Benzene	µg/kg	U	1.1	-	-
Amenable Cyanide	mg/kg	U	0.456	0.499	0.0
Total Cyanide	mg/kg		13.5	11.3	17.7
Total Phenol	mg/kg	U	0.900	0.881	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

3.2.4 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

The 4QCY14 SDF waste form, prepared from the 4QCY14 Tank 50H WAC sample and premix, analyses showed that:

- The concentrations of the RCRA metals and UHCs were present at levels below the Toxicity Characteristic limits.
- The concentrations of the RCRA metals and UHCs were present at levels below the UTS limits, with the exception of mercury.
- 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.
- No saltstone was placed into the SDF in 4QCY14.

5.0 References

1. "Toxicity Characteristic Leaching Procedure," EPA SW-846, Procedure 1311.
2. Miller, D. H., "4Q14 Saltstone TCLP", Experiment T8786-00095-05, SRNL E-Notebook (Production); Savannah River National Laboratory, Aiken, SC 29808, (2014).
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