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Disclaimer:

Saltstone First Quarter Calendar Year 2019 (1QCY19) Toxicity Characterstic Leaching Procedure (TCLP) Results

K. A. Hill

August 2019

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EXECUTIVE SUMMARY

The aqueous waste from Tank 50 (salt solution) is sampled quarterly for transfers to the Saltstone Production Facility (SPF). Salt solution is treated at SPF and disposed of in the Saltstone Disposal Facility (SDF). A SDF waste form (saltstone) was prepared in the Savannah River National Laboratory (SRNL) from the Tank 50 Waste Acceptance Criteria (WAC) sample[1] and Z-Area premix material for the first quarter of calendar year 2019 (1QCY19).[2,3] Results from this memorandum support Task 2: 'Grout Leaching Analyses' of the Task Technical Request (TTR)[3] prepared by Savannah River Remediation (SRR). After a 28 day cure, a sample of the SDF waste form was collected and shipped to a certified laboratory for analysis using the Toxicity Characteristic Leaching Procedure (TCLP).[4] The 1QCY19 saltstone sample met the South Carolina (SC) Code of Regulations for Hazardous Waste Management Regulations (HWMR) 61-79.261.24 and 61-79-268.48 requirements for a non-hazardous waste form with respect to Resource Conservation and Recovery Act (RCRA) metals and Underlying Hazardous Constituents (UHCs), and also met the SPF WAC.[5-7]

TABLE OF CONTENTS

LIST OF TABLES

LIST OF ABBREVIATIONS

D&S-FE	DWPF & Saltstone Facility Engineering
EC&ACP	Environmental Compliance & Area Completion Projects
EPA	Environmental Protection Agency
ES	Environmental Stewardship
ETF	Effluent Treatment Facility
LOD	Limit of Detection
LOQ	Limit of Quantitation
MRL	Minimum Reporting Limit
MS	Matrix Spike
MSD	Matrix Spike Duplicate
NRC	Nuclear Regulatory Commission
RCRA	Resource Conservation and Recovery Act
RL	Reporting Limit
SDF	Saltstone Disposal Facility
SPF	Saltstone Production Facility
SRNL	Savannah River National Laboratory
SRNS	Savannah River Nuclear Solutions
SRR	Savannah River Remediation
SwRI®	Southwest Research Institute
TCLP	Toxicity Characteristic Leaching Procedure
TTQAP	Task Technical and Quality Assurance Plan
TTR	Technical Task Request
UHC	Underlying Hazardous Constituents
WAC	Waste Acceptance Criteria

1.0 Introduction

The SPF receives waste from Tank 50 for treatment. The following dates were selected starting from the last quarterly sampling date to the current quarterly sampling date. Tank 50 accepted the following transfers from November 1, 2018 (when it was 39% full) to February 5, 2019 (when it was 40% full):[8] During this same time period there was a total of 39.8 kgal of Tank 50 material transferred out to Z Area.

- ~2.3 kgal from 211-H
- ~24.3 kgal from Effluent Treatment Facility (ETF)
- ~30.1 kgal from the Actinide Removal Process / Modular Caustic Side Solvent Extraction Unit (ARP/MCU) Decontaminated Salt Solution Hold Tank (DSSHT)

On February 5, 2019, a salt solution sample was taken from Tank 50[1] and used to prepare a SDF waste form sample, referred to as a saltstone sample.[9] The 1QCY19 saltstone sample was prepared on March 5, 2019.[9] Once the 1QCY19 saltstone sample cured for 28 days, it was crushed, sieved, packaged, and deemed "collected".[10] The sample was then shipped to Southwest Research Institute (SwRI®) to analyze for toxicity per the TCLP method.[2,4] This saltstone sample determines whether the non-hazardous nature of the grout meets the requirements of the SC Code of Regulations 61-79.261.24[6] for RCRA metals and 61-79.268.48[5] for inorganic/organic UHCs (for informational purposes only[3]).

2.0 Experimental

Saltstone preparation was performed at SRNL. DWPF & Saltstone Facility Engineering (D&S-FE) provided SRNL with the saltstone grout recipe as well as the premix components.[3,9] Table 2-1 shows the premix components obtained to date for CY2019 sample with specific LOT numbers.[9]

Table 2-1. Premix Components for CY2019

Premix Component	Date SRNL Received	LOT #
Holcim Cement 1QCY19	3/04/2019	2019-IR-05-1666
LeHigh Slag 1QCY19	3/04/2019	2019-IR-05-0120
SE Fly Ash 1QCY19	3/04/2019	2019-IR-05-0195

The saltstone sample was prepared using the mixing method outlined in SRNL Environmental Stewardship (ES) work instructions and the 1QCY19 premix components in Table 2-1.[11] The sample cured in a Ziploc® sealed plastic bag for 28 days. After curing, the sample was crushed and sieved using the method outlined in ES work instructions.[12] Material that passed through the 3/8-inch sieve was subsequently screened through a No. 4 sieve (4.76 mm). The material retained on the No. 4 sieve was packaged in a primary container (250 mL High Density Polyethylene (HDPE) bottle) and shipped on the same day that it was prepared to SwRI® by Environmental Compliance & Area Completion Projects (EC&ACP).[3]

3.0 Results

Table 3-1 summarizes the analytical results provided by the vendor, SwRI.[13] The first eight rows show data for the RCRA metals and the next four rows show data for the UHC metals from the TCLP leachates. The last four rows show results from solids analyses of the saltstone for benzene, phenol, total and amenable cyanide. The entire vendor report is documented and included as a reference.[13] For comparison, the previous quarter and four quarter average results are shown. The Regulatory Toxicity[6] values and the WAC Limits are from Table 6 of the WAC[7] and reflect the requirements in the applicable version of the document. Note that the vendor used a "modified" Method 1311 where sample mass was restricted due to the elevated activity of the sample.[13] This methodology is consistent with the joint guidance from the Nuclear Regulatory Commission (NRC) and Environmental Protection Agency (EPA) for mixed radioactive and hazardous waste.[14] For 4QCY18 TCLP sample, extraction fluid #2 was used, which is an aqueous acetic acid solution with pH = 2.88 ± 0.05 prepared by diluting 5.7 mL of glacial acetic acid into reagent water for a total volume of 1 Liter.[15] However, for 1QCY19 the TCLP extraction was performed using extraction fluid #1 which is an aqueous acetic acid solution with pH = 4.93 ± 0.05 prepared by diluting 5.7 mL of glacial acetic acid along with 64.3 mL of 1N NaOH to a total volume of 1 Liter using reagent water.[13]

The reported detection limit for As has remained the same relative to the previous quarter. The reported detection limit was <0.025 mg/L for As. The reported value for Se of 0.0549 mg/L was detected above the LOD of 0.025 mg/L and above the LOQ of 0.050 mg/L.

Lead has remained as less than detectable at <0.0075 mg/L in comparison to the previous quarter. The analyzed value for Be of <0.005 mg/L is at least 3X lower for this quarter and the previous quarter (<0.005 mg/L) than measured at 0.017 mg/L in 3Q18. The reported value for Cr for this quarter was measured at 0.0137 mg/L in comparison to less than detectable at <0.005 mg/L in 4Q18. There was no significant change in the Tank 50 supernate Cr concentration as the 1QCY19 Tank 50 average supernate Cr concentration is 49.0 mg/L ± 2.36 mg/L[1] compared to the prior 4QCY18 Tank 50[16] average supernate Cr value of 53.4 mg/L ± 0.7 mg/L. The ± values indicated are the one-sigma uncertainties calculated from triplicate analyses of the Tank 50 supernate. These uncertainties do not factor in any method/instrument uncertainties.

The reported TCLP value for Ba is 7X lower this quarter at 0.0914 mg/L than the previous 4Q18 Ba value of 0.656 mg/L. The Tank 50 supernate Ba concentration did decrease in the current 1QCY19 supernate at 7.00 mg/L ± 0.76 mg/L [1] vs. the previous 4QCY18 Tank 50 supernate value[16] of <0.65 mg/L.

Mercury TCLP value for the 1Q19 sample of 0.005 mg/L is 2.5X lower than the previous quarter measuring 0.013 mg/L and 11X lower than the past three quarters (3Q18, 2Q18 and 1Q18) in the range of 0.058 to 0.059 mg/L.

Prior to the 1Q19 and 2018 samples, the 2Q17 and 3Q17 Hg TCLP values were in the lower range of 0.0134 mg/L and 0.0051 mg/L, respectively. There was no data collected for the 4Q17 sample. Mercury speciation analyses for recent past 2Q17 through 1Q19 show that the total mercury levels in the Tank 50 supernate have ranged from a low of 61.7 mg/L for 4Q18 to a high of 81.4 mg/L for the 3Q17 sample as shown in Table 3-2.[17] The corresponding methyl Hg values expressed as mg Hg/L ranged from 18.8 mg/L to 36.6 mg/L. The methyl Hg species is the dominant Hg species in the Tank 50 supernate (relative to other Hg species measured like elemental Hg(0) or ionic Hg(I) and/or Hg(II)) with methyl Hg to total Hg ratios shown in Table 3-2[17] that are in the range of 0.3 to 0.5.

Amenable cyanide value for 1Q19 of 1.50 mg/kg[13] is 2.5X lower than the previous quarter (4Q18) measuring 4.04 mg/kg.[18]

Table 3-3 provides comparison between analytical results for each analyte to SwRI®'s Limit of Detection (LOD) and Limit of Quantitation (LOQ) for the TCLP leachates and to the Reporting Limits (RL) for the solids analyses. Antimony, arsenic, beryllium, cadmium, lead, nickel, silver, thallium, benzene, and phenol were all less than the detection limit or reporting limit. Appendix A includes summaries of results from blanks, laboratory control samples, matrix spikes, and matrix spike duplicates.

Table 3-1. 1QCY19 Saltstone Sample TCLP and Solids Analysis Results

Analyte	Result[15] (mg/L)	Regulatory Toxicity[6] (mg/L)	WAC Limit[7] (mg/L)	Results	
				Previous Quarter[18] (mg/L)	Previous Four Quarter Average[18-21] (mg/L)
RCRA Metals					
Arsenic (As)	<0.025[U]	5.0	2.5	<0.025[U]	0.0288[+]
Barium (Ba)	0.0914	100.0	50	0.656[D]	1.51
Cadmium (Cd)	<0.005[U]	1.0	0.5	<0.005[U]	0.005^
Chromium (Cr)	0.0137	5.0	2.5	<0.005[U]	0.0964*
Lead (Pb)	<0.0075[U]	5.0	2.5	<0.0075[U]	0.00818*
Mercury (Hg)	0.005[J]	0.2	0.1	0.013	0.0474
Selenium (Se)	0.0549	1.0	0.5	0.0296[B]	0.0492*
Silver (Ag)	<0.010[U]	5.0	2.5	<0.010[U]	0.01^
Underlying Hazardous Constituents (UHCs)					
Antimony (Sb)	<0.025[U]	-	-	<0.025[U]	0.0250^
Beryllium (Be)	<0.005[U]	-	-	<0.005[U]	0.008*
Nickel (Ni)	<0.005[U]	-	-	0.0185	0.0530
Thallium (Tl)	<0.005[UD]	-	-	<0.005[UD]	0.005^
Select Solids Analyses of Regulatory Interest					
	(mg/kg)			(mg/kg)	(mg/kg)
Benzene	<0.00094[U]	-	-	<0.00095[U]	0.00094[+]
Amenable Cyanide	1.50	-	-	4.04	1.20*
Total Cyanide	13.6	-	-	12.0	11.3
Phenol	<0.883[UJ]	-	-	<0.831[UJ]	0.962*

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

[U] Non-detected analyte
[D] Results reported from a dilution.
[J] Matrix spike and/or matrix spike duplicate criteria was not met.
[B] Analyte was detected at the instrument at or above Limits of Detection (LOD), but less than Limit of Quantitation (LOQ).
* Contains qualifier of "U" in at least one quarter.
[+] Contains qualifier of "U" in all quarters with multiple Reporting Limits (RL) or Limits of Detection (LOD).
^ Contains qualifier of "U" in all quarters with same RL or LOD.

Table 3-2. Mercury Speciation Data from Past Tank 50 Salt Solutions[17]

Tank 50 Sample	Total Hg (mg/L)	Methyl Hg (mg/L)	Ratio Methyl Hg/Total Hg
2QCY17	72.2	32.2	0.446
3QCY17	81.4	28.2	0.346
1QCY18	71.8	36.6	0.510
2QCY18	69.8	28.5	0.408
3QCY18	70.4	30.7	0.436
4QCY18	61.7	18.8	0.305
1QCY19	67.4	24.0	0.356

Table 3-3. RCRA Metal TCLP Result Concentrations, Limit of Detection, and Limit of Quantitation[13]

Analyte	Methods	LOD	LOQ	Sample Results	Qualifiers
		(µg/L)	(µg/L)	(µg/L)	
Antimony (Sb)	6010D	25.0	50.0	<25.0	U
Arsenic (As)	6010D	25.0	50.0	<25.0	U
Barium (Ba)	6010D	5.00	10.0	91.4	-
Beryllium (Be)	6010D	5.00	10.0	<5.00	U
Cadmium (Cd)	6010D	5.00	10.0	<5.00	U
Chromium (Cr)	6010D	5.00	10.0	13.7	-
Lead (Pb)	6010D	7.50	15.0	<7.50	U
Mercury (Hg)	7470A	1.00	2.00	5.61	J
Nickel (Ni)	6010D	5.00	10.0	<5.00	U
Selenium (Se)	6010D	25.0	50.0	54.9	-
Silver (Ag)	6010D	10.0	20.0	<10.0	U
Thallium (Tl)	6020B	5.00	10.0	<5.00	UD
-	-	-	**RL (mg/kg)**	**(mg/kg)**	-
Benzene	8260C	-	-	<0.00094	U
Amenable Cyanide	Amenable cyanide 9012B	-	0.168	1.50	J
Total Cyanide	Cyanide 9012B	-	0.168	13.6	-
Phenol	Phenol 9065	-	0.883	<0.883	UJ

- Indicates a location in the table for which an entry would not be appropriate.
U Result is less than the Limit of Detection (LOD) and/or Reporting Limit (RL).
D Result is reported from a dilution.
J Matrix spike and/or matrix spike duplicate criteria was not met.

4.0 Conclusions

Analyses of the SDF waste form prepared from the 1QCY19 Tank 50 salt solution sample and premix material resulted in the following findings.

- The RCRA metal TCLP result concentrations met the SC Code of Regulations 61-79.261.24 requirements for a nonhazardous waste form.[6]

- The measured concentrations of the TCLP RCRA metals and additional inorganic/organic UHCs met the SC Code of Regulations 61-79.268.48 non-wastewater standards.[5]

- The measured concentrations of the TCLP RCRA metals met the SPF WAC.[7]

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Appendix A. Quality Assurance

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.[13]

Table A- 1 shows all TCLP extraction fluid blank concentrations and the solid matrix blank concentrations. In the extraction fluid blank, arsenic, barium, beryllium, cadmium, chromium, lead, mercury, nickel, selenium, silver and thallium were all less than detection limit or reporting limit. Antimony was detected at the instrument at or above the LOD, but less than the LOQ. Benzene, amenable cyanide, total cyanide and phenol were all less than detection limit or reporting limit.

Table A- 1. TCLP Extraction Fluid Blank and Solid Matrix Blank[13]

Analyte	TCLP Blank (µg/L)	Qualifiers
Antimony (Sb)	<25.0	U
Arsenic (As)	<25.0	U
Barium (Ba)	<5.00	U
Beryllium (Be)	<5.00	U
Cadmium (Cd)	<5.00	U
Chromium (Cr)	<5.00	U
Lead (Pb)	<7.50	U
Mercury (Hg)	<0.100	U
Nickel (Ni)	5.10	B
Selenium (Se)	<25.0	U
Silver (Ag)	<10.0	U
Thallium (Tl)	<5.00	UD
Analyte	Solid Matrix Blank (mg/Kg)	Qualifiers
Benzene	<0.00050	U
Amenable Cyanide	<0.249	U
Total Cyanide	<0.249	U
Phenol	<0.978	U

[U] Result is less than the Limit of Detection (LOD) and/or Reporting Limit (RL).
[D] Result is reported from a dilution.
[B] Analyte was detected at the instrument at or above LOD, but less than LOQ.

Table A- 2 shows all LCS recoveries meet SwRI®'s acceptance limit in the range of 80% to 120% for metals and phenol, 70% to 130% for benzene and 99.6% for total cyanide, which was within the manufacturers acceptance limit.[13] The laboratory control samples are clean aqueous solutions analyzed to assure integrity of the analytical technique exclusive of matrix effects.

Table A- 2 Laboratory Control Sample[13]

Analyte	Laboratory Control (µg/L)		Recovery (%)
	True	Found	
Antimony (Sb)	500	490	98.0%
Arsenic (As)	2000	1970	98.5%
Barium (Ba)	2000	1980	99.0%
Beryllium (Be)	50.0	55.0	110.0%
Cadmium (Cd)	50.0	48.1	96.2%
Chromium (Cr)	200	194	97.0%
Lead (Pb)	500	469	93.8%
Mercury (Hg)	1	1.04	104.0%
Nickel (Ni)	500	483	96.6%
Selenium (Se)	2000	1870	93.5%
Silver (Ag)	50.0	49.1	98.2%
Thallium (Tl)	2000	1920	96.0%
Analyte	**Laboratory Control (mg/Kg)**		**Recovery (%)**
	True	**Found**	
Benzene	10	12.0	120.0%
Amenable Cyanide	-	-	-
Total Cyanide	42.2	43.3	103.0%
Phenol	25.0	24.0	96.0%

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

Results from analysis of the matrix spike (MS) and the matrix spike duplicate (MSD) are given in Table A-3 and Table A- **4**. These results shown in Table A- 3 indicate all analytes met the recommended quality control acceptance criteria for MS and MSD percent recoveries (75-125%) and the Relative Percent Difference (RPD) acceptance limits (0-20%) except for Hg which showed only ~ 55% MS and MSD percent recoveries. In Table A- **4**, results show benzene met the recommended quality control acceptance criteria for MS, MSD and RPDs. In Table A- **4**, results show total cyanide and phenol did not meet the recommended quality control acceptance criteria for MS, MSD and RPDs. However, a post-digestion spike recovery sample showed a phenol recovery ≥ 75% within the control limit of 60% to 120%.

Table A- 3 TCLP Leachates Matrix Spike and Duplicate Results[13]

Analyte	Initial Concentrations (µg/L)			Spiked Sample** (µg/L)		Recovery (%)		RPD (%)
	Parent Sample Result	Qualifiers	Spike Added	Spike	Spike Duplicate	Spike	Spike Duplicate	
Antimony (Sb)	<25.0	U	5000	5140	5190	102.8	103.8	1.0
Arsenic (As)	<25.0	U	2500	2560	2610	102.4	104.4	1.9
Barium (Ba)	91.4	-	5000	4790	4780	94.0	93.8	0.2
Beryllium (Be)	<5.00	U	500	514	518	102.8	103.6	0.8
Cadmium (Cd)	<5.00	U	500	467	479	93.4	95.8	2.5
Chromium (Cr)	13.7	-	1000	931	950	91.7	93.6	2.1
Lead (Pb)	<7.50	U	2500	2280	2360	91.2	94.4	3.4
Mercury (Hg)	5.61	J	10.0	11.1	11.2	**54.9**	**55.9**	1.8
Nickel (Ni)	<5.00	U	2500	2270	2360	90.8	94.4	3.9
Selenium (Se)	54.9	-	2500	2490	2560	97.4	100.2	2.8
Silver (Ag)	<10.0	U	500	510	510	102.0	102.0	0.0
Thallium (Tl)	<5.00	UD	2500	2260	2280	90.4	91.2	0.9

[U] Result is less than the Limit of Detection (LOD) and/or Reporting Limit (RL).
[D] Result is reported from a dilution.
[B] Analyte was detected at the instrument at or above Limits of Detection (LOD), but less than Limit of Quantitation (LOQ).
[J] Matrix spike and/or matrix spike duplicate criteria was not met.
- Indicates a location in the table for which an entry would not be appropriate.
** SwRI® Sample ID = W-18131-00001 MS/MSD

Table A- 4 **Organic UHCs Matrix Spike and Duplicate Results[13]**

Analyte	Initial Concentrations (mg/kg)				Spiked Sample (mg/kg)		Recovery (%)		RPD (%)
	Result	Qualifiers	MS-Spike Added	MSD-Spike Added	Spike	Spike Duplicate	Spike	Spike Duplicate	
Benzene**	0.0	U	0.020	0.020	0.022	0.020	110	100	20.0
Amenable Cyanide	-	-	-	-	-	-	-	-	-
Total Cyanide**	13.6	-	1.91	1.80	16.6	12.2	157	-77.8	593*
Phenol**	0.883	UJ	21.4	24.9	0.855	0.997	0.0	0.0	0.0

[U] Result is less than the Limit of Detection (LOD) and/or Reporting Limit (RL).
[J] Matrix spike and/or matrix spike duplicate criteria was not met.
*Parent value exceeded 4 times the spike added; therefore, MS/MSD %Recovery and %RPD are not required for evaluation
- Indicates a location in the table for which an entry would not be appropriate.
** SwRI® Sample ID = W-18131-00001 MS/MSD

Distribution:

M. R. Alexander	J. J. Mayer
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A. P. Fellinger	A. Samadi-Dezfouli
S. D. Fink	D. C. Sherburne
E. J. Freed	F. M. Smith
N. V. Halverson	A. V. Staub
E. K. Hansen	J. Stevens
S. J. Harrington	M. Stone
E. W. Harrison	P. C. Suggs
C. C. Herman	B. J. Wiedenman
K. A. Hill	T. L. White
P. J. Hill	A. W. Wiggins
J. F. Iaukea	W. R. Wilmarth
V. Jain	L. A. Wooten
V. M. Kmiec	R. H. Young
C. A. Langton	Records Administration (EDWS)
J. D. Ledbetter	
K. R. Liner	
M. J. Mahoney	
J. Manna	
K. B. Martin	