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Saltstone Fourth Quarter Calendar Year 2018 (4QCY18) Toxicity Characteristic Leaching Procedure (TCLP) Results



K. A. Hill

July 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) sample¹ and Z-Area premix material for the fourth quarter of calendar year 2018 (4QCY18).^{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 4QCY18 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

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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 September 21 (when it was 49% full) to November 6, 2018 (when it was 39% full):⁸ During this same time period there was a total of 125.9 kgal of Tank 50 material transferred out to Z Area.

- ~2.0 kgal from 211-H
- ~17.2 kgal from the Actinide Removal Process / Modular Caustic Side Solvent Extraction Unit (ARP/MCU) Decontaminated Salt Solution Hold Tank (DSSHT)

On November 6, 2018, a salt solution sample was taken from Tank 50¹ and used to prepare a SDF waste form sample, referred to as a saltstone sample.⁹ Due to delays in TTQAP approval before work could begin, the Tank 50 salt solution sample remained in the steel sampler for 64 days before being transferred to polybottles for processing. The 4QCY18 saltstone sample was prepared on February 12, 2019.⁹ Once the 4QCY18 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 61-79.261.24⁶ for RCRA metals and 61-79.268.48⁵ for inorganic/organic UHCs (for informational purposes only³).

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 all premix components obtained to date for CY2018 samples with specific LOT numbers.⁹

Table 2-1. Premix Components for CY2018

Premix Component	Date SRNL Received	LOT #
Holcim Cement 4QCY18	2/5/2019	2018-IR-05-1717
Holcim Cement 3QCY18	10/22/2018	2018-IR-05-1644
Holcim Cement 2QCY18	8/15/2018	2018-IR-05-1545
Holcim Cement 1QCY18	3/27/2018	2018-IR-05-0371
LeHigh Slag 4QCY18	2/5/2019	2019-IR-05-0120
LeHigh Slag 3QCY18	10/22/2018	2018-IR-05-1301
LeHigh Slag 2QCY18	8/15/2018	2018-IR-05-1299
LeHigh Slag 1QCY18	3/27/2018	2018-IR-05-0366
SE Fly Ash 4QCY18	2/5/2019	2018-IR-05-0802
SE Fly Ash 3QCY18	10/22/2018	2018-IR-05-1297
SE Fly Ash 2QCY18	8/15/2018	2018-IR-05-0807
SE Fly Ash 1QCY18	3/27/2018	2018-IR-05-0546

The saltstone sample was prepared using the mixing method outlined in SRNL Environmental Stewardship (ES) work instructions and the 4Q premix components in Table 2-1.¹¹ The sample was left to cure in a Ziploc[®] sealed plastic bag for at least 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 Toxicity⁶ values and the WAC Limits are from Table 6 of the WAC⁷ 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.¹³ The TCLP extraction was performed using extraction fluid #2 which is an aqueous acetic acid solution with pH = 2.88 ± 0.05 .⁴ The reported detection limit of <0.025 mg/L for As is 2X lower than the previous quarter that reported an As detection limit of <0.05 mg/L from a diluted sample. The reported value for Se of 0.0296 mg/L was detected above the LOD of 0.025 mg/L but below the LOQ of 0.050 mg/L. Selenium was reported previously at <0.1 mg/L from a diluted sample.

The reported value from 3Q18 for Pb was 0.0102 mg/L which was above the minimum detection limit, but lower than the quantification limit. This quarter, lead was measured as less than detectable at <0.0075 mg/L. The analyzed value for Be of <0.005 mg/L is about 3X lower than the previous quarter measuring at 0.017 mg/L in 3Q18. The reported value for Cr for this quarter was measured as less than detectable at <0.005 mg/L. This value is significantly lower compared to previous quarter, 3Q18, where the value was measured from a diluted sample at 0.26 mg/L. There was no significant change in the Tank 50 supernate Cr concentration as the 4QCY18 Tank 50 average supernate Cr concentration is $53.4 \text{ mg/L} \pm 0.7 \text{ mg/L}$ ¹ compared to the prior 3QCY18 Tank 50¹⁴ average supernate Cr value of $54.6 \text{ mg/L} \pm 2.6 \text{ mg/L}$. The \pm 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 lower this quarter at 0.656 mg/L than the previous 3Q18 Ba value of 1.84 mg/L and the previous four quarter average of 1.47 mg/L. The Tank 50 supernate Ba concentration did decrease in the current 4QCY18 supernate at $<0.65 \text{ mg/L}$ ¹ vs. the previous 3QCY18 Tank 50 supernate value¹⁴ of $1.97 \text{ mg/L} \pm 0.25 \text{ mg/L}$.

Mercury TCLP value for the 4Q18 sample of 0.013 mg/L is 4.5X lower than the past three quarters (3Q18, 2Q18 and 1Q18) in the range of 0.058 to 0.059 mg/L. Prior to the 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 samples 2Q17 through 4Q18 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 3-2.¹⁵ 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 3-2¹⁵ that are in the range of 0.305 to 0.510. A noticeable difference in this 4QCY18 TCLP sample relative to previous 2018 TCLP samples is use of the Tank 50 supernate sample that had aged in the original steel sampler for 64 days prior to sampling and processing to grout. As pointed out in Reference¹⁵, the sample used for saltstone production had a measured total mercury value of only 45.9 mg/L vs. the earlier 10-day sampled value of 61.7 mg/L.

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, cadmium, selenium, 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. 4QCY18 Saltstone Sample TCLP and Solids Analysis Results

Analyte	Result ¹³ (mg/L)	Regulatory Toxicity ⁶ (mg/L)	WAC Limit ⁷ (mg/L)	Results	
				Previous Quarter ¹⁶ (mg/L)	Previous Four Quarter Average ¹⁶⁻¹⁹ (mg/L)
RCRA Metals					
Arsenic (As)	<0.025 ^U	5.0	2.5	<0.050 ^{UD}	0.0275 ⁺
Barium (Ba)	0.656 ^D	100.0	50	1.84 ^D	1.47
Cadmium (Cd)	<0.005 ^U	1.0	0.5	<0.005 ^U	0.005 [^]
Chromium (Cr)	<0.005 ^U	5.0	2.5	0.26 ^D	0.0964*
Lead (Pb)	<0.0075 ^U	5.0	2.5	0.0102 ^B	0.00755*
Mercury (Hg)	0.013	0.2	0.1	0.0583	0.0454
Selenium (Se)	0.0296 ^B	1.0	0.5	<0.1 ^{UD}	0.0547*
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	0.0238 ⁺
Beryllium (Be)	<0.005 ^U	-	-	0.017	0.008*
Nickel (Ni)	0.0185	-	-	0.0717 ^D	0.0496*
Thallium (Tl)	<0.005 ^{UD}	-	-	<0.005 ^{UD}	0.005 [^]
Select Solids Analyses of Regulatory Interest					
	(mg/kg)			(mg/kg)	(mg/kg)
Benzene	<0.00095 ^U	-	-	<0.00089 ^U	0.00094 ⁺
Amenable Cyanide	4.04	-	-	<0.232 ^{UJ}	0.311*
Total Cyanide	12.0	-	-	11.1	11.3
Phenol	<0.831 ^{UJ}	-	-	<0.771 ^{UJ}	1.22*

-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 Solutions¹⁵

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

Table 3-3. RCRA Metal TCLP Result Concentrations, Limit of Detection, and Limit of Quantitation¹³

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	50.0	100	656	D
Beryllium (Be)	6010D	5.00	10.0	<5.0	U
Cadmium (Cd)	6010D	5.00	10.0	<5.00	U
Chromium (Cr)	6010D	5.00	10.0	<5.00	U
Lead (Pb)	6010D	7.50	15.0	<7.50	U
Mercury (Hg)	7470A	1.00	2.00	13.0	-
Nickel (Ni)	6010D	5.00	10.0	18.5	-
Selenium (Se)	6010D	25.0	50.0	29.6	B
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	-	-	<0.00095	U
Amenable Cyanide	Amenable cyanide 9012B	-	0.250	4.04	-
Total Cyanide	Cyanide 9012B	-	0.181	12.0	-
Phenol	Phenol 9065	-	0.831	<0.831	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.

^B Analyte was detected at the instrument at or above Limits of Detection (LOD), but less than Limit of Quantitation (LOQ).

4.0 Conclusions

Analyses of the SDF waste form prepared from the 4QCY18 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

5.0 Reference

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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.²⁰

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^{13,20}

Analyte	TCLP Blank (µg/L)	Qualifiers
Antimony (Sb)	28.4	B
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)	<5.00	U
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	<0.974	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.^{13,20} 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^{13,20}

Analyte	Laboratory Control (µg/L)		Recovery (%)
	True	Found	
Antimony (Sb)	500	478	95.6%
Arsenic (As)	2000	1900	95.0%
Barium (Ba)	2000	1900	95.0%
Beryllium (Be)	50.0	50.0	100.0%
Cadmium (Cd)	50.0	45.7	91.4%
Chromium (Cr)	200	181	90.5%
Lead (Pb)	500	449	89.8%
Mercury (Hg)	1	0.941	94.1%
Nickel (Ni)	500	460	92.0%
Selenium (Se)	2000	1800	90.0%
Silver (Ag)	50.0	48.7	97.4%
Thallium (Tl)	2000	1990	99.5%
Analyte	Laboratory Control (mg/Kg)		Recovery (%)
	True	Found	
Benzene	10	9.8	98.0%
Amenable Cyanide	-	-	-
Total Cyanide	78.3	78.3	99.6%
Phenol	25.0	25.2	100.8%

- 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 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%). 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 of 103.8% within the control limit of 60% to 120%.

Table A- 3 TCLP Leachates Matrix Spike and Duplicate Results^{13,20}

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	4880	4890	97.6	97.8	0.2
Arsenic (As)	<25.0	U	2500	2470	2500	98.8	100	1.2
Barium (Ba)	654	D	5000	5570	5550	98.3	97.9	0.4
Beryllium (Be)	<5.00	U	500	467	461	93.4	92.2	1.3
Cadmium (Cd)	<5.00	U	500	435	439	87.0	87.8	0.9
Chromium (Cr)	<5.00	U	1000	877	870	87.8	87.0	0.8
Lead (Pb)	<7.50	U	2500	2170	2180	86.8	87.2	0.5
Mercury (Hg)	13.0	-	10.0	22.7	22.9	97.0	99.0	2.0
Nickel (Ni)	18.5	-	2500	2220	2210	88.1	87.7	0.5
Selenium (Se)	29.6	B	2500	2380	2380	94.0	94.0	0.0
Silver (Ag)	<10.0	U	500	424	436	84.8	87.2	2.8
Thallium (Tl)	<5.00	UD	2500	2410	2430	96.4	97.2	0.8

^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).

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

** SwRI[®] Sample ID = W-18120-00001 MS/MSD

Table A- 4 Organic UHCs Matrix Spike and Duplicate Results^{13,20}

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	0.019	0.019	0.021	0.019	111	100	10.0
Amenable Cyanide	-	-	-	-	-	-	-	-	-
Total Cyanide**	12.0	-	1.73	1.73	11.7	11.6	-17.3	-23.1	14.3*
Phenol**	0.831	UJ	21.9	-	0.876	-	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-18120-00001 MS/MSD

Distribution:

M. R. Alexander	J. J. Mayer
J. P. Arnold	M. W. McCoy
M. J. Barnes	R. T. McNew
M. N. Borders	D. J. McCabe
J. M. Bricker	G. A. Morgan
K. M. Brotherton	P. W. Norris
L. W. Brown	J. E. Occhipinti
N. F. Chapman	F. M. Pennebaker
J. H. Christian	R. C. Player
W. A. Condon	J. Polk
A. D. Cozzi	P. A. Polk
C. L. Crawford	M. M. Potvin
J. Crenshaw	A. A. Ramsey
D. A. Crowley	W. G. Ramsey
C. C. DiPrete	J. W. Ray
K. D. Dixon	C. Ridgeway
R. E. Edwards	L. B. Romanowski
A. P. Fellingner	K. H. Rosenberger
S. D. Fink	A. Samadi-Dezfouli
E. J. Freed	D. C. Sherburne
N. V. Halverson	F. M. Smith
E. K. Hansen	A. V. Staub
S. J. Harrington	J. Stevens
E. W. Harrison	M. Stone
C. C. Herman	C. B. Sudduth
K. A. Hill	P. C. Suggs
P. J. Hill	B. J. Wiedenman
J. F. Iaukea	T. L. White
V. Jain	A. W. Wiggins
C. A. Langton	W. R. Wilmarth
J. D. Ledbetter	L. A. Wooten
K. R. Liner	R. H. Young
M. J. Mahoney	Records Administration (EDWS)
J. Manna	
K. B. Martin	