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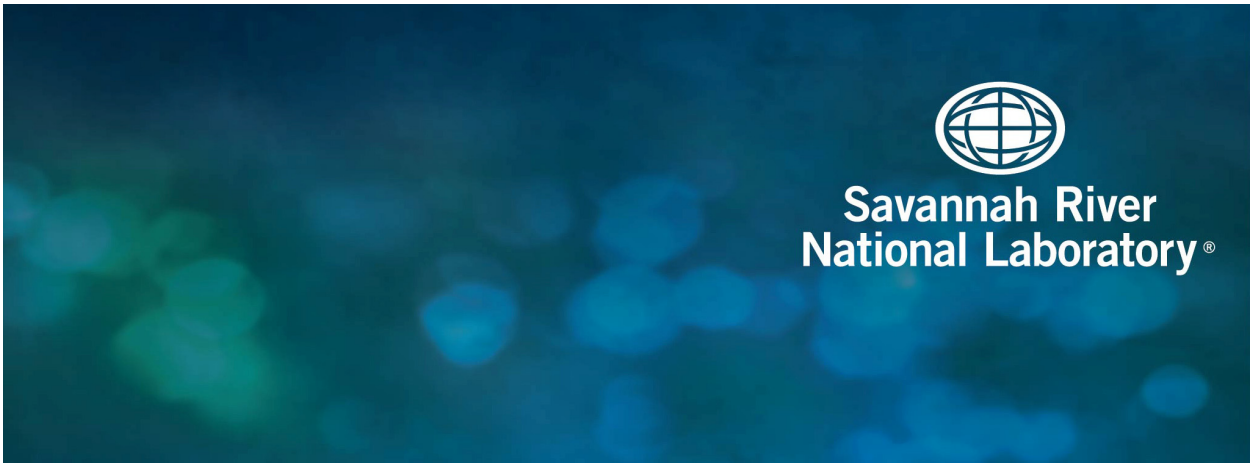

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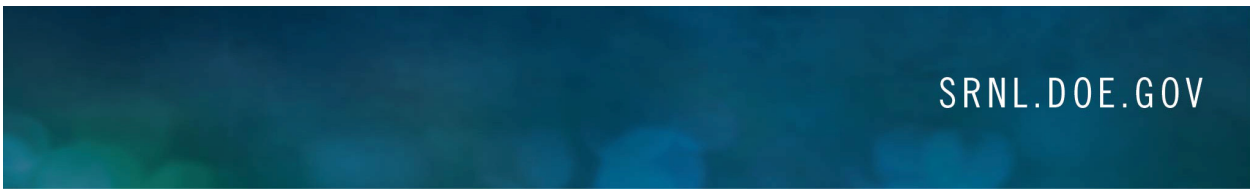
Saltstone Third Quarter Calendar Year 2021 (3QCY21) Toxicity Characteristic Leaching Procedure (TCLP) Results

K. A. Hill

February 2022

SRNL-STI-2021-00681, Revision 0



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Prepared for
U.S. Department of Energy

SRNL-STI-2021-00681
Revision 0

Keywords: *Tank 50, Regulatory, Saltstone, TCLP*

Retention: *Permanent*

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Savannah River National Laboratory is operated by Battelle Savannah River Alliance for the U.S. Department of Energy under Contract No. 89303321CEM000080.

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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). Per request of customer, X-TTR-Z-00023, Revision 0, two SDF waste form (saltstone) samples were prepared in the Savannah River National Laboratory (SRNL) from the Tank 50 Waste Acceptance Criteria (WAC) sample and Z-Area premix material for the third quarter of calendar year 2021(3QCY21). One sample contained a Full Premix which included 10:45:45 (by weight) of cement, slag and fly ash. The second sample contained 60:40 (by weight) of slag and fly ash only referred to as the "Cement-Free grout sample". Results from this technical report support Task 2: 'Grout Leaching Analyses' of the Task Technical Request (TTR) prepared by Savannah River Remediation (SRR). After at least 28 days cured, a sample of each of the SDF waste forms was collected and shipped to a certified laboratory for analysis using the Toxicity Characteristic Leaching Procedure (TCLP). The 3QCY21 saltstone (Full Premix) and the Cement-Free grout samples 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 that was in effect at the time of the tank sampling.

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LIST OF ABBREVIATIONS

ARP/MCU	Actinide Removal Process / Modular Caustic Side Solvent Extraction Unit
D&S-FE	DWPF & Saltstone Facility Engineering
DSSHT	Decontaminated Salt Solution Hold Tank
EC&ACP	Environmental Compliance & Area Completion Projects
EM&ES	Environmental, Materials & Energy Sciences
EPA	Environmental Protection Agency
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
RPD	Relative Percent Difference
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
SWPF	Salt Waste Processing Facility
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 Saltstone Production Facility (SPF) receives waste from Tank 50 for treatment. The following transfers were selected starting from the last quarterly sampling date when regulatory saltstone samples were prepared first quarter of calendar year 2021 (1QCY21) to the current third quarter of calendar year 2021 (3QCY21) quarterly sampling date. Tank 50 accepted the following transfers from January 19, 2021 to July 20, 2021 totaling ~ 1,597 kgal[1]:

- ~42.2 kgal from Effluent Treatment Facility (ETF)
- ~1,548.8 kgal from the Salt Waste Processing Facility (SWPF)
- ~0.15 kgal from LWHT and flush water
- ~6.28 kgal from Other

During this same time period there was a total of 1,433 kgal of Tank 50 material transferred out to Z-area.[1] On July 20, 2021, a salt solution sample was taken from Tank 50[2]. DWPF & Saltstone Facility Engineering (D&S-FE) requested the Savannah River National Laboratory (SRNL) prepare two SDF waste form samples, referred to as a Full Premix saltstone sample and Cement-Free grout sample.[3] The Full Premix sample is the baseline, historical formulation for saltstone and is the sample used to determine that saltstone produced by the SPF is a non-hazardous waste form. The Cement-Free sample is included as a preliminary examination of the potential use of a Cement-Free formulation in future saltstone processing. The 3QCY21 Full Premix and Cement-Free saltstone samples were prepared on August 9, 2021.[4] The 3QCY21 saltstone samples cured for 44 days, they were crushed, sieved, packaged, and deemed "collected".[4, 5] The samples were then shipped to Southwest Research Institute (SwRI®) to analyze for toxicity per the TCLP method.[6, 7] The full premix saltstone sample determines whether the non-hazardous nature of the grout meets the requirements of the SC Code of Regulations 61-79.261.24[8] for RCRA metals and 61-79.268.48[9] for inorganic/organic UHCs (for informational purposes only[6]).

2.0 Experimental

Saltstone preparation was performed at SRNL. D&S-FE provided SRNL with the saltstone grout recipe as well as the premix components.[6] Table 2-1 shows the premix components obtained for the last quarterly TCLP sample (1QCY21) and the premix components that have been obtained to date, including specific LOT numbers.[4] SRR provided SRNL personnel with the premix formulation for the 3QCY21 saltstone samples.[4]

Table 2-1. Premix Components for 1QCY21 and Latest 3QCY2021.

Premix Component	Date SRNL Received	LOT #
Holcim Cement 1QCY21	6/27/2019	2019-IR-05-0487
Lehigh Slag 1QCY21	11/11/2020	2020-IR-05-976
SE Fly Ash 1QCY21	11/11/2020	2020-IR-05-0910
Holcim Cement 3QCY21	8/6/2021	2021-IR-05-0683
Lehigh Slag 3QCY21	8/6/2021	2021-IR-05-1480
SE Fly Ash 3QCY21	8/6/2021	2021-IR-05-0695

The 3QCY21 salt solution had been stored since collection in a zero-headspace Teflon® bottle refrigerated at <10 ºC to preserve the mercury species present in the sample. The saltstone samples were prepared using the mixing method outlined in SRNL Environmental, Materials & Energy Sciences (EM&ES) work instructions[10] and the 3QCY21 premix components in Table 2-1.[4] One sample contained a Full Premix which included 10:45:45 (by weight) of cement, slag and fly ash. The second sample contained 60:40 (by weight) of slag and fly ash only referred to as the "Cement-Free grout sample". The samples cured in a Ziploc® sealed plastic bag for 65 days. After curing, the samples were crushed and sieved using the method outlined in EM&ES work instructions.[11] Material that passed through the 3/8-inch (0.375") sieve (9.252 mm) 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).

3.0 Results

Table 3-1 summarizes the analytical results provided by the vendor, SwRI.[12] 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.[12] Some of the data values are flagged with qualifier letters (U, L, D, J, B) that are shown as footnotes to the table. Further explanation for these qualifiers can be found in the vendor report.[12] For comparison to the current Full Premix results, the previous quarter and four previous quarter average results for the Full Premix sample are shown in the final two columns. The four-quarter average values contain a qualifier (*, +, ^) if past values have been reported as a non-detectable analyte ('U'). The Regulatory Toxicity[8] values and the WAC Limits are from Table 6 of the WAC[12] 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.[12] 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.[12] For 3QCY21 the TCLP extraction was performed on the Full Premix and the Cement-Free samples using extraction fluid #2.[3] Extraction fluid #2 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. Extraction fluid #1 is an aqueous acetic acid solution with pH= 4.93 ± 0.05 prepared by diluting 5.7 mL of glacial acetic acid into 500 mL of reagent water, then adding 64.3 mL of sodium hydroxide and diluting with reagent water for a total volume of 1 Liter.[7] Determination of the extract fluid used in the TCLP uses Section 7.1.4 'Determination of Appropriate Extraction Fluid' of the TCLP method.[7] A size reduced portion of the solid is mixed with water and if the resulting pH of the contacted water is below pH 5.0, then extraction fluid #1 is used. However, if the resulting pH is above 5.0, then an additional step using HCl is performed. Both samples resulted in high pH slightly above 12 of the contacted water so the HCl step was applied. The resulting pH from the HCl step showed a pH of 11.78 for the Full Premix sample and a pH of 11.84 for the Cement-Free sample. Thus the extraction fluid #2 was applied for the Full Premix and for the Cement-Free.[12] The final extract pH values resulting from the TCLP extraction using fluid #2 showed a pH of 6.8 for the Full Premix and a pH of 7.3 for the Cement-Free Sample.

Full-Premix Saltstone

Table 3-1 shows the reported value for Se of 0.042 mg/L is also above the Limit of Detection (LOD) of 0.025 mg/L and below the Limit of Quantitation (LOQ), similar to the previous quarter (1QCY21) in which a quarterly full-premix saltstone sample was analyzed. 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 below the quantitation limit, which is the same as the previous quarter. The reported value for Cr for this quarter was measured at <0.005 mg/L and is similar to the previous quarter. The reported TCLP value for Ba of 0.367 mg/L is comparable to the previous quarter and to the previous four quarter average.

The mercury TCLP value for the 3QCY21 sample of 0.0092 mg/L is higher than the previous TCLP regulatory quarterly (1QCY21) sample which measured 0.0045 mg/L and the past four quarters that generated saltstone samples with an average of 0.004 mg/L. Table 3-2 shows the total mercury and methyl Hg expressed as mg Hg/L for the current 3QCY21 and the previous four quarters that generated saltstone samples. The associated TCLP values are also shown in the final column. These data indicate for the past five quarters that saltstone samples have been made, with total mercury in the range of 59 to 68 mg/L and methyl mercury to total mercury ratios in the range of 0.22 to 0.40, that the TCLP value for mercury has remained below 5% of the regulatory toxicity limit of 0.200 mg/L.

Cement-Free Saltstone

All of the RCRA metals and UHCs shown in Table 3-1 for the Full Premix and Cement-Free samples are comparable, i.e., within 50% of the value. The amenable and total cyanide and phenol for the Cement-Free sample are higher than for the Full Premix sample. The Cement-Free values for total cyanide and phenol are also higher than the four-quarter average of the Full Premix.

Table 3-1. 3QCY21 Saltstone Sample TCLP and Solids Analysis Results

Analyte	Full Premix Result (mg/L)[12]	Cement-Free Result (mg/L)[12]	Regulatory Toxicity[8] (mg/L)	WAC Limit[14] (mg/L)	Results Full Premix	
					Previous Quarter (mg/L)[15]	Previous Four Quarter Average[15-18] (mg/L)
RCRA Metals						
Arsenic (As)	<0.025[U]	<0.025[U]	5.0	2.5	0.032	0.027[*]
Barium (Ba)	0.367	0.539	100.0	50	0.626[D]	0.409
Cadmium (Cd)	<0.005[U]	<0.005[U]	1.0	0.5	<0.005[U]	0.005^
Chromium (Cr)	<0.005[U]	<0.005[U]	5.0	2.5	<0.005[U]	0.007*
Lead (Pb)	<0.0075[U]	<0.0075[U]	5.0	2.5	<0.0075[U]	0.008^
Mercury (Hg)	0.0092	0.0096	0.2	0.1	0.0045	0.004
Selenium (Se)	0.042[B]	0.030[B]	1.0	0.5	<0.043[B]	0.039*
Silver (Ag)	<0.010[U]	<0.010[U]	5.0	2.5	<0.010[U]	0.010[^]
Underlying Hazardous Constituents (UHCs)						
Antimony (Sb)	<0.025[U]	<0.025[U]	-	-	<0.025[U]	0.0250[^]
Beryllium (Be)	<0.005[U]	<0.005[U]	-	-	<0.005[UD]	0.005*
Nickel (Ni)	<0.005[U]	<0.005[U]	-	-	<0.025[U]	0.010+
Thallium (Tl)	<0.075[U]	<0.075[U]	-	-	<0.005[UD]	0.005^
Select Solids Analyses of Regulatory Interest						
	(mg/kg)	(mg/kg)			(mg/kg)	(mg/kg)
Benzene	<0.001[U]	<0.001[U]	-	-	<0.00099[U]	0.00098[+]
Amenable Cyanide	<0.384[U]	0.700	-	-	1.90[J]	0.953*
Total Cyanide	15.1	20.3[D]	-	-	18.3	13.9
Phenol	<0.800[UJ]	<0.995[UJ]	-	-	<0.940[UJ]	0.875[+]

-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 Current and Past Tank 50 Salt Solutions

Tank 50 Sample	Total Hg (mg/L)	Methyl Hg (mg/L) as Hg	Ratio Methyl Hg as Hg /Total Hg	TCLP (mg/L)[a]
1QCY19 [a]	67.4	24.0	0.356	0.0056
2QCY19 [a]	63.0	19.3	0.306	0.0036
3QCY19 [a]	67.6	19.9	0.294	0.0016
1QCY21 [a]	59.0	13.1	0.222	0.0045
3QCY21	62.2	24.6	0.395	0.0092

"[a]" Past four quarter average TCLP = 0.0038 mg/L ± 0.0017 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, beryllium, cadmium, chromium, lead, nickel, silver and thallium were all less than the detection limit or reporting limit for the Full Premix sample. The same was true for the Cement-Free. Appendix A includes summaries of results from blanks, laboratory control samples, matrix spikes, and matrix spike duplicates.

Table 3-3. RCRA Metal TCLP Result Concentrations, Limit of Detection, and Limit of Quantitation[12]

Analyte	Methods	LOD	LOQ	Full premix Sample Results	Cement Free Sample Results
		(µg/L)	(µg/L)	(µg/L)	(µg/L)
Antimony (Sb)	6010D	25.0	50.0	<25.0[U]	<25.0[U]
Arsenic (As)	6010D	25.00	50.0	<25.0[U]	<25.0[U]
Barium (Ba)	6010D	5.0	10.0	367	539
Beryllium (Be)	6010D	5.00	10.0	<5.00[U]	<5.00[U]
Cadmium (Cd)	6010D	5.00	10.0	<5.00[U]	<5.00[U]
Chromium (Cr)	6010D	5.00	10.0	<5.00 [U]	<5.00[U]
Lead (Pb)	6010D	7.50	15.0	<7.50[U]	<7.50[U]
Mercury (Hg)	7470A	0.05	0.100	9.20	9.60
Nickel (Ni)	6010D	5.0	10.0	<5.00[U]	<5.00[U]
Selenium (Se)	6010D	25.0	50.0	42.0[B]	30.0[B]
Silver (Ag)	6010D	10.0	20.0	<10.0[U]	<10.0[U]
Thallium (Tl)	6010D	75.00	150.0	<75.0[U]	<75.0[U]
-	-	-	**RL (mg/kg)**	**(mg/kg)**	
Benzene	8260D	-	-	<0.001[U]	<0.001[U]
Amenable Cyanide	9012C	-		<0.384[U]	0.700
Total Cyanide	9012C	-		15.1	20.3[D]
Phenol	9065	-		<0.800[UJ]	<0.995[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 Full Premix and Cement-Free waste forms prepared from the 3QCY21 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.[8]

- 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.[9]

- The measured concentrations of the TCLP RCRA metals met the SPF WAC.[14]

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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.[12]

Table A- 1 shows all TCLP extraction fluid blank concentrations and the solid matrix blank concentrations. In the extraction fluid blank, antimony, arsenic, barium, cadmium, chromium, lead, mercury, nickel, selenium, silver and thallium were all less than detection limit or reporting limit. Thallium and beryllium were 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[12]

Analyte	TCLP Blank (µg/L)	Qualifiers
Antimony (Sb)	<25.0	U
Arsenic (As)	<25.00	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)	<5.0	U
Selenium (Se)	<25.0	U
Silver (Ag)	<10.0	U
Thallium (Tl)	<75.00	U
Analyte	**Solid Matrix Blank (mg/Kg)**	**Qualifiers**
Benzene	<0.00050	U
Amenable Cyanide	<0.400	U
Total Cyanide	<0.400	U
Phenol	<0.988	U

[U] Result is less than the Limit of Detection (LOD) and/or Reporting Limit (RL).

Table A- 2 shows all Laboratory Control Sample (LCS) recoveries meet the SwRI®'s acceptance limit in the range of 80% to 120% for metals and phenol, 70% to 130% for benzene and 122.7% for total cyanide, which was within the manufacturers acceptance limit.[17] 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[12]

Analyte	Laboratory Control (µg/L)		Recovery (%)
	True	Found	
Antimony (Sb)	500	510	102.0%
Arsenic (As)	2000	2080	104.0%
Barium (Ba)	2000	2110	105.5%
Beryllium (Be)	50.0	55.5	111.0%
Cadmium (Cd)	50.0	50.1	100.2%
Chromium (Cr)	200	212	106.0%
Lead (Pb)	500	498	99.6%
Mercury (Hg)	1.00	1.13	113.0%
Nickel (Ni)	500	526	105.2%
Selenium (Se)	2000	1920	96.0%
Silver (Ag)	50.0	49.4	98.8%
Thallium (Tl)	2000	2080	104.0%
Analyte	**Laboratory Control (mg/Kg)**		**Recovery (%)**
	True	**Found**	
Benzene	0.020	0.021	105%
Amenable Cyanide	-	-	-
Total Cyanide	58.1	71.3	122.7%
Phenol	25.0	23.0	92.0%

- 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. The 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%). 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 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[12]

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	500	522	524	104.4	104.8	0.4
Arsenic (As)	<25.0	U	500	514	527	102.8	105.4	2.5
Barium (Ba)	539	-	500	1030	1020	98.2	96.2	2.1
Beryllium (Be)	<5.00	U	500	507	511	101.4	102.2	0.8
Cadmium (Cd)	<5.00	U	500	465	470	93.0	94.0	1.1
Chromium (Cr)	<5.00	U	500	488	492	97.6	98.4	0.8
Lead (Pb)	<7.50	U	500	437	439	87.4	87.8	0.5
Mercury (Hg)	9.60	-	500	500	519	98.1	101.9	3.8
Nickel (Ni)	<25.0	U	500	481	492	96.2	98.4	2.3
Selenium (Se)	30.0	B	500	512	516	96.4	97.2	0.8
Silver (Ag)	<10.0	U	500	432	427	86.4	85.4	1.2
Thallium (Tl)	<75.00	U	500	474	455	94.8	91.0	4.1

[U] Result is less than the Limit of Detection (LOD) and/or Reporting Limit (RL).
[B] Analyte was detected at the instrument at or above Limits of Detection (LOD), but less than Limit of Quantitation (LOQ).
"-" No Qualifier
** SwRI® Sample ID = W-18424-00002 MS/MSD

Table A-4. Organic UHCs Matrix Spike and Duplicate Results[12]

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	-	0.038	0.036	0.036	0.036	95	100	20.0
Amenable Cyanide	-	-	-	-	-	-	-	-	-
Total Cyanide***	20.3**	D	1.18	1.13	20.3	20.2	0.0	8.8	200.0
Phenol***	0.995	UJ	24.0	20.0	0.783	0.960	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.
[D] Result is reported from a Dilution
* SwRI® Sample ID = W-18424-00002
**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-18424-00002 MS/MSD