Contract No:

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July 2022 Semiannual Saltstone Toxicity Characteristic Leaching Procedure (TCLP) Results

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EXECUTIVE SUMMARY

The aqueous waste from Tank 50 (salt solution) is sampled semiannually 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-00025, 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 July 2022 semiannual Toxicity Characteristic Leaching Procedure (TCLP) samples. 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 Mission Completion (SRMC). 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 July 2022 semiannual 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.

TABLE OF CONTENTS

LIST OF TABLES vii
LIST OF ABBREVIATIONS viii
1.0 Introduction 1
2.0 Experimental 1-2
3.0 Results 2-5
4.0 Conclusions 6
5.0 References 7
Appendix A . Quality Assurance A-1

LIST OF TABLES

Table 2-1. Premix Components for January and July 2022 Semiannual TCLP Sample 2

Table 3-1. July 2022 Semiannual Saltstone Sample TCLP and Solids Analysis Results 4

Table 3-2. Mercury Speciation Data from Current and Past Tank 50 Salt Solutions 4

Table 3-3. RCRA Metal TCLP Result Concentrations, Limit of Detection, and Limit of Quantitation[12] ... 5

LIST OF ABBREVIATIONS

D&S-FE	DWPF & Saltstone Facility Engineering
DSS	Decontaminated Salt Solution
DSSHT	Decontaminated Salt Solution Hold Tank
EC&ACP	Environmental Compliance & Area Completion Projects
EPA	Environmental Protection Agency
ETF	Effluent Treatment Facility
LOD	Limit of Detection
LOQ	Limit of Quantitation
MCU	Modular Caustic Side Solvent Extraction Unit
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
SRMC	Savannah River Mission Completion
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 semiannual sampling date when regulatory saltstone samples were prepared January of calendar year 2022 to the now July semiannual sampling date. Tank 50 accepted the following transfers from January 12, 2022 to July 19, 2022 totaling ~ 1212 kgal[1]:

- ~20.7 kgal from Effluent Treatment Facility (ETF)
- ~1159 kgal from the Salt Waste Processing Facility (SWPF)
- ~0.39 kgal from bearing and flush water
- ~32.4 kgal from Other

During this same time period there was a total of 1186 kgal of Tank 50 material transferred out to SPF.[1] On July 19, 2022, 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 historical formulation for saltstone and the Cement-Free sample represents the current recipe for saltstone processing. The Cement-Free sample is the sample used to determine that saltstone produced by the SPF is a non-hazardous waste form. The July 2022 semiannual Full Premix and Cement-Free saltstone samples were prepared on August 10, 2022.[4] The samples cured for 42 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.[3, 6] The Cement-free saltstone sample determines whether the non-hazardous nature of the grout meets the requirements of the SC Code of Regulations 61-79.261.24[7] for RCRA metals and 61-79.268.48[8] for inorganic/organic UHCs (for informational purposes only[3]).

2.0 Experimental

2.1 Technical

Saltstone preparation was performed at SRNL. D&S-FE provided SRNL with the saltstone grout recipe as well as the premix components.[3] Table 2-1 shows the premix components obtained for the last semiannual TCLP sample including specific LOT numbers.[9] Premix components used for January semiannual TCLP sample were used for the July 2022 semiannual TCLP, per SRMC.[4] SRMC provided SRNL personnel with the premix formulation for the July 2022 semiannual saltstone samples.[4]

Table 2-1. Premix Components for January and July 2022 Semiannual TCLP Sample.

Premix Component	Date SRNL Received	LOT #
Holcim Cement	8/6/2021	2021-IR-05-0683
Lehigh Slag	8/6/2021	2021-IR-05-1480
SE Fly Ash	8/6/2021	2021-IR-05-0695

The July 2022 semiannual 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 work instructions[10] and the July 2022 semiannual 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 42 days. After curing, the samples were crushed and sieved using the method outlined in SRNL 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).

2.2 Quality Assurance

Quality Assurance requirements for performing reviews of technical reports and the extent of review are established in manual E7 2.60.[12] SRNL documents the extent and type of review using the SRNL Technical Report Design Checklist contained in WSRC-IM-2002-00011, Rev. 2.[13] The customer requested that a Functional Classification of Safety Significant/Production Support apply to this work. Thus, a Design Verification technical review was performed via a document review according to the applicable elements detailed in Section 5.3.1 'Design Verification by Document Review' of E7 2.60.[12] Data collection and analysis methods used in this work comply with this requirement as detailed in the TTQAP.[14]

3.0 Results

Table 3-1 summarizes the analytical results provided by the vendor, SwRI.[15] 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.[15] Some of the data values are flagged with qualifier letters (U, D, J, B) that are shown as footnotes to the table. Further explanation for these qualifiers can be found in the vendor report.[15] For comparison to the current Cement-Free results, the previous quarter and four previous quarter average results for the Cement-Free 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[7] values and the WAC Limits are from Table 6 of the WAC[16] 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.[15] 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. For the July 2022 semiannual saltstone samples, the TCLP extraction was performed on the Full

Premix and the Cement-Free samples using extraction fluid #1.[15] 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.[6] 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. Determination of the extract fluid used in the TCLP uses Section 7.1.4 'Determination of Appropriate Extraction Fluid' of the TCLP method.[6] A size reduced portion of the solid is mixed with reagent 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 and heating to 50 °C is performed. Both samples resulted in a pH slightly at and above 12 of the contacted water. Therefore, the HCl step was applied. The resulting pH from the HCl step showed a pH of 4.37 for the Full Premix sample and a pH of 2.35 for the Cement-Free sample. Thus, since both of these pHs were below the specified pH of 5.0 for this step, the extraction fluid #1 was applied for the Full Premix and for the Cement-Free.[15] The final extract pH values resulting from the TCLP extraction using fluid #1 showed a pH of 11.25 for the Full Premix and a pH of 10.95 for the Cement-Free Sample.

Full Premix Saltstone

Table 3-1 shows the reported value for Se of 0.0615 mg/L is above the Limit of Detection (LOD) of 0.025 mg/L and above the Limit of Quantitation (LOQ) of 0.05 mg/L, similar to the previous semiannual (January2022) sample in which a Full premix saltstone sample was analyzed. Lead has remained as less than detectable at <0.0075 mg/L in comparison to the previous semiannual TCLP sample. The analyzed value for Be and Cd of <0.005 mg/L is below the quantitation limit, which is the same as the previous semiannual TCLP sample. The reported value for Cr was measured at 0.01 mg/L which is higher than the previous semiannual TCLP sample. The reported TCLP value for Ba of 0.093 mg/L is lower than the previous semiannual TCLP sample (0.491 mg/L)[9].

The mercury TCLP value for the July 2022 semiannual Full premix sample of 0.0054 mg/L is higher than the previous semiannual TCLP sample which measured 0.0048 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 except for amenable cyanide. 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 and amenable cyanide and phenol are higher, comparable and lower than the average for the previous four Cement-Free samples, respectively.

Table 3-2 shows the total mercury and methyl Hg expressed as mg Hg/L for the current July 2022 Tank 50 salt solution sample and the previous four quarters that generated saltstone samples. The associated Cement-Free TCLP values are also shown in the final column. For the current July 2022 and previous four samples, saltstone samples have been made with total mercury in the range of 46 to 68 mg/L and methyl mercury to total mercury ratios in the range of 0.22 to 0.46. The Cement-Free mercury TCLP values are in the range of 0.0015 to 0.0096 mg/L. This indicates that the TCLP value for mercury has remained below 0.010 mg/L, which is 5% of the regulatory toxicity limit of 0.200 mg/L.

Table 3-1. July 2022 Semiannual Saltstone Sample TCLP and Solids Analysis Results

Analyte	Full Premix Result (mg/L)[15]	Cement-Free Result (mg/L)[15]	Regulatory Toxicity[7] (mg/L)	WAC Limit[16] (mg/L)	Results Cement-Free	
					Previous Cement-Free Sample (mg/L)[9]	Previous Four Cement-Free Average[9, 17-19] (mg/L)
RCRA Metals						
Arsenic (As)	<0.025[U]	<0.025[U]	5.0	2.5	<0.025[U]	0.024*
Barium (Ba)	0.093	0.089	100.0	50	0.656	0.458
Cadmium (Cd)	<0.005[U]	<0.005[U]	1.0	0.5	<0.005[U]	0.005^
Chromium (Cr)	0.01	<0.005[U]	5.0	2.5	<0.005[U]	0.005^
Lead (Pb)	<0.0075[U]	<0.0075[U]	5.0	2.5	<0.0075[U]	0.008^
Mercury (Hg)	0.0054	0.006	0.2	0.1	0.0061	0.006
Selenium (Se)	0.0615	0.053	1.0	0.5	0.037[B]	0.035*
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[U]	0.005^
Nickel (Ni)	<0.005[U]	<0.005[U]	-	-	<0.006[U]	0.0010*
Thallium (Tl)	<0.075[U]	<0.075[U]	-	-	<0.075[U]	0.040+
Select Solids Analyses of Regulatory Interest						
	(mg/kg)	(mg/kg)			(mg/kg)	(mg/kg)
Benzene	<0.001[U]	<0.001[U]	-	-	<0.001[U]	0.00098+
Amenable Cyanide	<0.995[U]	3.7	-	-	<0.400[U]	2.2*
Total Cyanide	16.0[DJ]	20.7[DJ]	-	-	19.8[D]	20.9
Phenol	<0.762[UJ]	<0.904[UJ]	-	-	<0.836[UJ]	1.33+

-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	Cement-Free TCLP (mg/L) [a]
3QCY19 [a]	67.6	19.9	0.294	0.0015
1QCY21 [a]	59.0	13.1	0.222	0.0079
3QCY21 [a]	62.2	24.6	0.395	0.0096
Jan. 2022 [a]	45.9	20.9	0.455	0.0061
July 2022	59.2	17.3	0.292	0.0061

"a" Past four quarter average Cement-Free TCLP = 0.0063 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, 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 sample. 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[15]

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	25.0	50.0	<25.0[U]	<25.0[U]
Barium (Ba)	6010D	5.00	10.0	93.4	89.5
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	10.0	<5.00[U]
Lead (Pb)	6010D	7.50	15.0	<7.50[U]	<7.50[U]
Mercury (Hg)	7470A	0.500	1.00	5.44	6.07
Nickel (Ni)	6010D	5.00	10.0	<5.00[U]	<5.00[U]
Selenium (Se)	6010D	25.0	50.0	61.5	53.0
Silver (Ag)	6010D	10.0	20.0	<10.0[U]	<10.0[U]
Thallium (Tl)	6010D	75.0	150	<75.0[U]	<75.0[U]
-	-	-	RL (mg/kg)	(mg/kg)	(mg/kg)
Benzene	8260D	-	0.00099	<0.001[U]	<0.001[U]
Amenable Cyanide	9012C	-	0.995 & 0.973	<0.995[U]	3.70
Total Cyanide	9012C	-	0.995 & 0.973	16.0[DJ]	20.7[DJ]
Phenol	9065	-	0.762 & 0.904	<0.762[UJ]	<0.904[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 Full Premix and Cement-Free waste forms prepared from the July 2022 semiannual 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.[7]
- 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.[8]
- The measured concentrations of the TCLP RCRA metals met the SPF WAC.[16]

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.[15]

Table A- 1 shows all TCLP extraction fluid blank concentrations and the solid matrix blank concentrations. In the extraction fluid blank, antimony, arsenic, beryllium, cadmium, chromium, lead, mercury, nickel, selenium, silver, and thallium were all less than detection limit or reporting limit. Barium was detected at the instrument above the LOD and 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[15]

Analyte	TCLP Blank (µg/L)	Qualifiers
Antimony (Sb)	<25.0	U
Arsenic (As)	<25.0	U
Barium (Ba)	10.3	-
Beryllium (Be)	<5.00	U
Cadmium (Cd)	<5.00	U
Chromium (Cr)	<5.00	U
Lead (Pb)	<7.50	U
Mercury (Hg)	<0.500	U
Nickel (Ni)	<5.00	U
Selenium (Se)	<25.0	U
Silver (Ag)	<10.0	U
Thallium (Tl)	<75.0	U
Analyte	**Solid Matrix Blank (mg/Kg)**	**Qualifiers**
Benzene	<0.00050	U
Amenable Cyanide	<0.200	U
Total Cyanide	<0.200	U
Phenol	<0.975	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 85% to 115% for total cyanide. 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[15]

Analyte	Laboratory Control (µg/L)		Recovery (%)
	True	Found	
Antimony (Sb)	500	512	102.4%
Arsenic (As)	2000	2070	103.5%
Barium (Ba)	2000	2090	104.5%
Beryllium (Be)	50.0	54.4	108.8%
Cadmium (Cd)	50.0	50.6	101.2%
Chromium (Cr)	200	203	101.5%
Lead (Pb)	500	503	100.6%
Mercury (Hg)	1.00	1.03	103.0%
Nickel (Ni)	500	492	98.4%
Selenium (Se)	2000	1910	95.5%
Silver (Ag)	50.0	49.6	99.2%
Thallium (Tl)	2000	2090	104.5%
Analyte	**Laboratory Control (mg/Kg)**		**Recovery (%)**
	True	**Found**	
Benzene	0.020	0.021	105%
Amenable Cyanide	-	-	-
Total Cyanide	0.700	0.764	109.1%
Phenol	22.6	22.4	88.9%

- 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 80% to 120%. The results for total cyanide did not meet the recommended quality control acceptance criteria for MS, MSD and RPDs. However, a post-digestion spike recovery sample showed a total cyanide recovery of 91.5% within the control limit of 75% to 125%.

Table A- 3 TCLP Leachates Matrix Spike and Duplicate Results[15]

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	528	531	105.6	106.2	0.6
Arsenic (As)	<25.0	U	500	543	529	108.6	105.8	2.6
Barium (Ba)	93.4	-	500	584	586	98.1	98.5	0.4
Beryllium (Be)	<5.00	U	500	482	485	96.4	97.0	0.6
Cadmium (Cd)	<5.00	U	500	466	469	93.2	93.8	0.6
Chromium (Cr)	10.0	-	500	465	471	91.0	92.2	1.3
Lead (Pb)	<7.50	U	500	442	442	88.4	88.4	0.0
Mercury (Hg)	5.44	-	500	400	407	78.9	80.3	1.8
Nickel (Ni)	<5.00	U	500	451	454	90.2	90.8	0.7
Selenium (Se)	61.5	-	500	559	567	99.5	101.1	1.6
Silver (Ag)	<10.0	U	500	508	466	101.6	93.2	8.6
Thallium (Tl)	<75.0	U	500	507	464	101.4	92.8	8.9

[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-18536-00001 MS/MSD

Table A-4. Organic UHCs Matrix Spike and Duplicate Results[15]

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	-	0.037	0.037	0.035	0.041	95	111	16.0
Amenable Cyanide	-	-	-	-	-	-	-	-	-
Total Cyanide	16.0	DJ	22.0	19.2	15.4	16.9	2.7	4.7	53.0
Phenol	0.762	UJ	19.4	20.4	0.775	0.818	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-18536-00001
- Indicates a location in the table for which an entry would not be appropriate.
*** SwRI® Sample ID = W-18536-00001 MS/MSD

Distribution:

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S. J. Harrington	A. V. Staub
E. W. Harrison	M. E. Stone
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