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Saltstone 1QCY13 TCLP Results

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EXECUTIVE SUMMARY

A Saltstone waste form was prepared in the Savannah River National Laboratory (SRNL) from a Tank 50H sample and Z-Area premix material for the first quarter of calendar year 2013 (1QCY13). After a 49 day cure, samples of the saltstone were collected, and the waste form was shown to meet the South Carolina Hazardous Waste Management Regulations (SCHWMR) R.61-79.261.24 and R.61-79.268.48(a) requirements for a nonhazardous waste form with respect to RCRA metals and underlying hazardous constituents. These analyses met all quality assurance specifications of USEPA SW-846.

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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-MS	Inductively Coupled Plasma – Mass Spectrometer
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
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

1.0 Introduction

The Saltstone Production Facility (SPF) receives waste from Tank 50H for treatment. In the first quarter of the 2013 calendar year (1QCY13), Tank 50H accepted transfers of approximately 4.2 kgal from the Effluent Treatment Project (ETP), approximately 7 kgal from 211H, approximately 455.7 kgal from the Actinide Removal Process / Modular Caustic Side Solvent Extraction Unit (ARP/MCU) Decontaminated Salt Solution Hold Tank (DSS-HT), and approximately 22.5 kgal from other sources.

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).[1] During operation, samples were collected from Tank 50H and grout samples prepared to determine the non-hazardous nature of the grout to meet the requirements of the South Carolina Hazardous Waste Management Regulations (SCHWMR) R.61-79.261.24(b) and R.61-79.268.48(a).

Savannah River National Laboratory (SRNL) was asked to prepare saltstone from samples of Tank 50H obtained January 16, 2013 during 1QCY13 to determine the non-hazardous nature of the grout. The samples were cured and shipped to Southwest Research Institute (SWRI) to perform the Toxic Characteristic Leaching Procedure (TCLP) and subsequent extract analysis on saltstone samples for the analytes required for the quarterly analysis saltstone sample. In addition to the eight toxic metals—arsenic, barium, cadmium, chromium, mercury, lead, selenium and silver—analytes include underlying hazardous constituents (UHC) antimony, beryllium, nickel, thallium, benzene, phenols, and total and amenable cyanide which could not be eliminated from analysis by process knowledge.[3]

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.

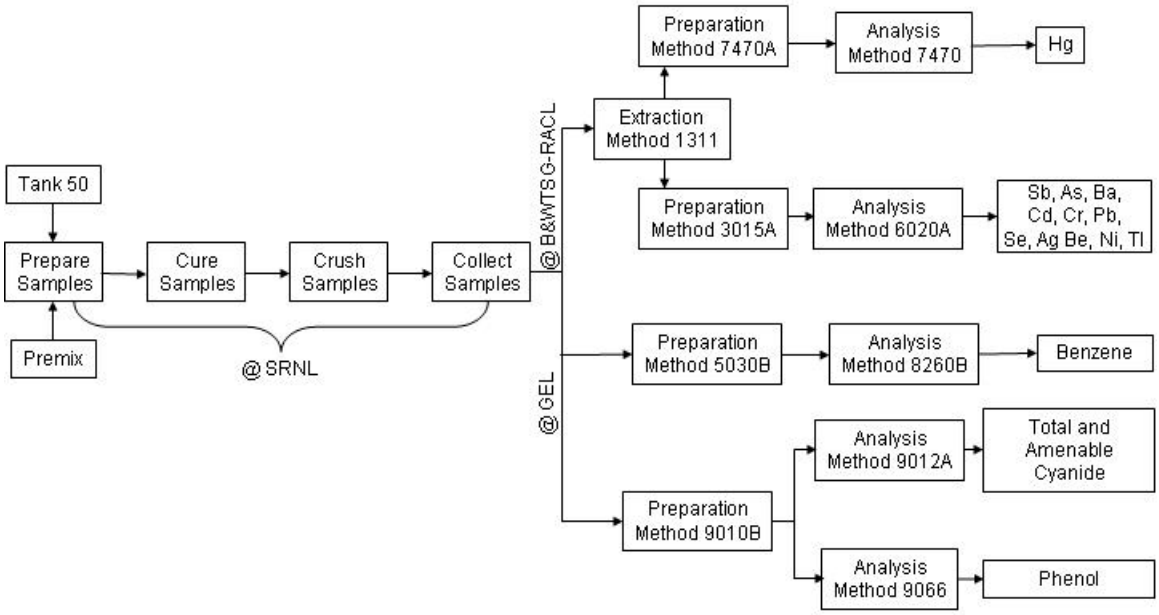

Figure 2-1. Flowchart of saltstone preparation and analysis

2.1 Saltstone Preparation

Saltstone preparation was performed at SRNL. The weight percent solids data used for the TCLP sample was taken from the quarterly Waste Acceptance Criteria (WAC) analyses performed on Tank 50H[4]. Table 2-1 lists the concentration of TCLP metals of interest in the salt solution from the WAC analysis for the sample.[4] As shown in Table 2-1 the contents of Tank 50H exceed the regulatory limits for antimony, cadmium, chromium, mercury, selenium, thallium and phenol, and therefore must be treated and disposed of in a non-hazardous waste form. Table 2-2 contains the parameters used to prepare the TCLP sample.[5]

Saltstone samples for TCLP were prepared with the Tank 50H blended salt solution and a premix of cement, slag, and fly ash. Figure 2-2 shows the formulation used to prepare these samples. The admixtures were added to the salt solution first and then the dry feeds were added to the liquid. The salt solution, admixtures and premix materials were 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 method.

After curing for not less than 28 days[1]— 49 days for the 1QCY13 sample, the saltstone was removed from the container and a portion of the saltstone was crushed to particles less than 0.9 centimeters (3/8 inch) as prescribed by Section 7.13 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."[1] ESS-WP retrieved the sample from SRNL and transported them to SWRI for extraction and analysis.

Table 2-1. Sample Results of TCLP Metals and UHCs from Tank 50 WAC Analyses

Analyte	Sample Results (mg/L) [4]	Regulatory Limits (mg/L)
	1Q13	Toxicity[a]
As	<4.12	5
Ba	<0.461	100
Cd	<0.731	1
Cr	35.5	5
Pb	0.608	5
Hg	50.3	0.2
Se	<7.90	1
Ag	<0.974	5
--		UHC[b]
Sb	<30.0	1.15
Be	<0.104	1.22
Ni	<1.80	11
Tl	4.96	0.20
-	(mg/kg)	(mg/kg)
benzene	<0.150	10
phenol	<10.0	6.2
cyanide (total)	NM	590
cyanide (amenable)	NM	30

NM – Not Measured
[a] SCHWMR R.61-79.261.24(b) "Characteristic of Toxicity."
[b] SCHWMR R.61-79.268.48 "Universal Treatment Standards."

Table 2-2. Customer Recommended Values for Preparation of TCLP Sample

Parameter	1QCY13
Water-to-Premix ratio	0.59
(Daratard 17) gal/Ton premix	0
(Dow Corning Q2-3183A) gal/Ton premix	0.20

Saltstone Mix Data Sheet

MIX # 0144		Date: 1/30/2013	
Material	**%**	**WT%**	**Grams**
Waste Solution: Tank 50 01/16/13 1Q13 **Wt% Solids #** 28.528 **Grams Water** 178.77		45.04	250.12
Admixture: Q2 Antifoam*		0.08	0.24
Admixture:			0.00
Admixture:			
Premix		54.92	305.00
Cement (% of Premix)	10	5.49	30.50
Slag (% of Premix)	45	24.71	137.25
Fly Ash (% of Premix)	45	24.71	137.25
Total	100	100.04	555.36
Water to Premix Ratio	0.59		

Calculations:
Use CBO fly ash

From customer:
0.59 W/P
0.10 gpm Antifoam
33 TPH Dry Feeds
NO Daratard

Q2 is diluted Q2 amount . In plant, diluted 1:4 in water.
* Actual amount of Q2 added to sample is 0.24 g. Q2 was diluted to a 1:4 in water and 100 uL pipette was used to add the diluted Q2 to the sample.

Figure 2-2. Data Sheet for saltstone mix used to prepare the 1QCY13 TCLP sample

2.2 Saltstone Testing

Saltstone testing was performed by SWRI. Activities associated with the 1QCY13 saltstone sample were TCLP extraction, TCLP leachate digestion and analysis, extraction of sold

subsamples and extract analysis. The sample arrived at SWRI in San Antonio, Texas on March 21, 2013 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.

The volatile compound, benzene, was 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 TCLP metals analysis, the sample was extracted by SW-846 method 1311. It is reported from method 1311 "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 SW-846 Method 6010C.

3.0 Results and Discussion

Results summarized in the following tables are from the data package for these analyses.[6] Data is presented in these results as reported by the vendor, SWRI. 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 a "J" qualifier indicate an issue with the matrix spike recoveries. 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 three groups:

- Total phenol is flagged as having matrix spiked issues.
- Cadmium, chromium, lead, silver, antimony, thallium, beryllium, benzene, amenable cyanide, and total phenol were detected at or below the RLs.
- Arsenic, barium, mercury, selenium, nickel, and total cyanide were detected in the leachates at concentrations above the RLs.

Table 3-1. Saltstone TCLP metals and UHC results

Analyte	Results	Q	Reporting Limit	Units	Method
Arsenic	39.2	-	10.0	µg/L (TCLP)	1311/6010C
Barium	673	-	5.0	µg/L (TCLP)	1311/6010C
Cadmium	5.0	U	5.0	µg/L (TCLP)	1311/6010C
Chromium	5.0	U	5.0	µg/L (TCLP)	1311/6010C
Lead	5.0	U	5.0	µg/L (TCLP)	1311/6010C
Mercury	2.2	-	0.2	µg/L (TCLP)	1311/6010C
Selenium	25.8	-	10.0	µg/L (TCLP)	1311/6010C
Silver	5.0	U	5.0	µg/L (TCLP)	1311/6010C
Antimony	20.0	U	20.0	µg/L (TCLP)	1311/6010C
Nickel	9.2	-	5.0	µg/L (TCLP)	1311/6010C
Thallium	20.0	U	20.0	µg/L (TCLP)	1311/6010C
Beryllium	5.0	U	5.0	µg/L (TCLP)	1311/6010C
Benzene	1.8	U	1.8	µg/kg	EPA 8260
Amenable Cyanide	0.206	U	0.206	mg/kg	EPA 9012B
Total Cyanide	12.3	-	0.206	mg/kg	EPA 9012B
Total Phenol	0.659	UJ	0.659	mg/kg	EPA 9012B

- Indicates a location in the table for which an entry would not be appropriate.
[Q] Qualifier Column
[U] Indicates compound was analyzed for, but not detected.
[J] Indicates MS/MSD recoveries were not within specification

3.1 Comparison of Results to Regulatory Limits

Results from the TCLP leachate analyses from Table 3-1 are replicated in Table 3-2; however the TCLP metals units are changed from µg/L to mg/L and compared to the regulatory limits that may be applied to the Saltstone waste form. Table 3-2 includes the SCHWMR R.61-79.261.24(b) limits above which a waste is to be considered characteristically hazardous for toxicity and the SCHWMR R.61-79.268.48 Universal Treatment Standards (UTS) for hazardous constituents. In addition, Maximum Contaminant Levels (MCL's) from the State Primary Drinking Water Regulations[1] also have been included in Table 3-2. By comparing the sample results and the regulatory limits the following conclusions can be made:

- The saltstone waste form was not characteristically hazardous for toxicity.
- The leachate metals concentrations were below the Nonwastewater Standard for all of the metals and UHCs.
- Barium, chromium, lead, selenium, and silver were below the MCL's.
- Arsenic, cadmium, mercury, antimony, thallium, and beryllium exceeded the MCL.
- Nickel does not have a MCL.

The MCL is the limit for a constituent in drinking water. The MCL is used to determine the class of landfill required. At 10x MCL, a Class 3 landfill is required. The SDF vaults are permitted as a Class 3 landfill. None of the analyses were greater than 10x the MCL.

Table 3-2. Saltstone TCLP and UHC Results and Corresponding Regulatory Limits

Analyte	Results (mg/L)	Q	Regulatory Limits		
			Toxicity[a]	UTS[b]	MCL[c]
			(mg/L)	Nonwastewater Standard (mg/L TCLP)	(mg/L)
Arsenic	3.92E-02	-	5	5	0.010
Barium	6.73E-01	-	100	21	2
Cadmium	5.00E-03	U	1	0.11	0.005
Chromium	5.00E-03	U	5	0.6	0.1
Lead	5.00E-03	U	5	0.75	0.015[d]
Mercury	2.2E-03	-	0.2	0.025	2E-03
Selenium	2.58E-02	-	1	5.7	0.05
Silver	5.00E-03	U	5	0.14	0.1[e]
Antimony	2.00E-02	U	-	1.15	0.006
Nickel	9.2E-03	-	-	11	-
Thallium	2.00E-02	U	-	0.20	2E-03
Beryllium	5.00E-03	U	-	1.22	4E-03
-	**Results (mg/kg)**	-	-	**UTS[b] (mg/kg)**	-
Benzene	1.8E-03	U	-	10	-
Amenable Cyanide	2.06E-01	U	-	30	-
Total Cyanide	1.23E+01	-	-	590	-
Total Phenol	6.59E-01	UJ	-	6.2	-

- Indicates a location in the table for which an entry would not be appropriate.
[Q] Qualifier Column
[U] indicates compound was analyzed for, but not detected.
[J] Indicates MS/MSD recoveries were not within specification
[a] R.61-79.261.24(b) "Characteristic of Toxicity."
[b] R.61-79.268.48 "Universal Treatment Standards."
[c] SCDHEC State Primary Drinking Water Regulation Maximum Contaminant Levels.
[d] Lead action level from SCDHEC 61-58.11.B.
[e] Secondary drinking water parameter.

3.2 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.[6]

3.2.1 *Blanks*

Blank concentrations are given in Table 3-3. In the TCLP Blank, all the analyte concentrations were below the RLs.

Table 3-3. Method Blanks

Analyte	Blank	Units	Q
Arsenic	10.0	µg/L	U
Barium	5.0	µg/L	U,J
Cadmium	5.0	µg/L	U
Chromium	5.0	µg/L	U
Lead	5.0	µg/L	U
Mercury	0.20	µg/L	U
Selenium	10.0	µg/L	U
Silver	5.0	µg/L	U
Antimony	20.0	µg/L	U
Nickel	5.0	µg/L	U
Thallium	20.0	µg/L	U
Beryllium	5.0	µg/L	U
Benzene	1.0	µg/kg	U
Amenable Cyanide	0.247	mg/kg	U
Total Cyanide	0.247	mg/kg	U
Total Phenol	0.934	mg/kg	U

[Q] Qualifier Column
[U] Indicates compound was analyzed for, but not detected above the RL
[J] low MS/MSD recoveries

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		Units	Recovery (%)
	True	Recovery		
Arsenic	4000.0	3664.88	µg/L	91.6
Barium	4000.0	3683.42	µg/L	92.1
Cadmium	100.0	93.15	µg/L	93.2
Chromium	400.0	366.2	µg/L	91.6
Lead	1000.0	896.16	µg/L	89.6
Mercury	1.0	1.01	µg/L	101.3
Selenium	4000.0	3419.47	µg/L	85.5
Silver	100.0	91.09	µg/L	91.1
Antimony	1000.0	908.98	µg/L	90.9
Nickel	1000.0	890.45	µg/L	89.0
Thallium	4000.0	3730.06	µg/L	93.3
Beryllium	100.0	90.63	µg/L	90.6
Benzene	10	10	µg/kg	100
Amenable Cyanide	NA	NA	mg/kg	NA
Total Cyanide	67.7	50.2	mg/kg	74.2
Total Phenol	0.500	0.497	mg/kg	99.4

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 silver.

- 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 silver.

- The RPD(s) between the MS and MSD met the acceptance limits (0 – 30%). The result for silver are "J" flagged due to the MS and MSD recoveries. A post-digestion spike was performed for silver and the silver recovery was 100.5%.

Table 3-5. Matrix Spike and Matrix Spike Duplicate Results

Analyte	Units	Initial Concentration			Spiked Sample		Recovery	
		Sample	Spike Added		Spike	Spike Duplicate	Spike	Spike Duplicate
Arsenic	µg/L	39.19	2500.00		2475.13	2509.54	97.4	98.8
Barium	µg/L	672.86	5000.00		5076.16	5103.74	88.1	88.6
Cadmium	µg/L	5.0	500.00		426.78	429.89	85.4	86.0
Chromium	µg/L	5.0	1000.00		846.15	851.21	84.6	85.1
Lead	µg/L	5.0	2500.00		2089.67	2092.02	83.6	83.7
Mercury	µg/L	2.164	2.00		3.984	3.922	91.0	87.9
Selenium	µg/L	25.78	2500.00		2405.43	2403.04	95.2	95.1
Silver[J]	µg/L	5.0	500.00		277.25	277.94	55.4	55.6
Antimony	µg/L	20.0	5000.00		4668.39	4765.96	93.4	95.3
Nickel	µg/L	9.17	2500.00		2018.18	2034.31	80.4	81.0
Thallium	µg/L	20.0	2500.00		2300.11	2312.56	92.0	92.5
Beryllium	µg/L	5.0	500.00		443.99	447.53	88.8	89.5
Benzene	µg/kg	0	19	19	19	18	100	95
Amenable Cyanide[U]	mg/kg	0.206	NA		NA	NA	-	-
Total Cyanide	mg/kg	12.3	23.9		32.4	-	84.1	-
Total Phenol[U,J]	mg/kg	0.659	21.3	16.4	1.03	16.7	4.84	102

- Indicates a location in the table for which an entry would not be appropriate.
[Q] Qualifier Column
[U] Indicates compound was analyzed for, but not detected above the RL
[J] low MS/MSD recoveries

Table 3-6. Duplicates and Relative Percent Difference

Analyte	Units	Q	Initial Sample	Duplicate Sample	RPD
Arsenic	µg/L	-	39.19	38.34	2.2
Barium	µg/L	-	672.86	667.62	0.8
Cadmium	µg/L	U	5.0	5.0	-
Chromium	µg/L	U	5.0	5.0	-
Lead	µg/L	U	5.0	5.0	-
Mercury	µg/L	-	2.164	2.188	1.1
Selenium	µg/L	-	25.78	27.09	5.0
Silver	µg/L	U	5.0	5.0	-
Antimony	µg/L	U	20.0	20.0	-
Nickel	µg/L	-	9.17	8.53	7.2
Thallium	µg/L	U	20.0	20.0	-
Beryllium	µg/L	U	5.0	5.0	-
Benzene	µg/L	U	1.8	-	-
Amenable Cyanide	mg/kg	U	0.206	--	-
Total Cyanide	mg/kg	-	12.3	10.5	15.8
Total Phenol	mg/kg	U J	0.659	0.831	-

- Indicates a location in the table for which an entry would not be appropriate.
[Q] Qualifier Column
[U] Indicates compound was analyzed for, but not detected above the RL
[J] Indicates RPD is out of specification

3.2.4 *Calibration Information*

- All initial calibration requirements have been met for this sample delivery group (SDG).
- All Contract Required Reporting Limit requirement(s) met the referenced advisory control limits.
- 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 continuing calibration verifications bracketing this SDG met the acceptance criteria.

4.0 Conclusions

Preparation of the 1QCY13 saltstone samples and the subsequent TCLP analyses showed that:

- The saltstone waste form disposed of in the Saltstone Disposal Facility in 1QCY13 was not characteristically hazardous for toxicity.
- The concentrations of the eight RCRA metals and UHCs identified as possible in the saltstone waste form were present at levels below the UTS.
- Analyses met all quality assurance specifications of USEPA SW-846.

The saltstone waste form placed in the Saltstone Disposal Facility in 1QCY132 met the SCHWMR R.61-79.261.24(b) RCRA metals requirements for a nonhazardous waste form. The TCLP leachate concentrations were less than 10x the MCLs in SCDHEC Regulations R.61-107.19, Part I C.

The saltstone waste form placed in the Saltstone Disposal Facility in 1QCY13 met the R.61-79.268.48(a) non wastewater treatment standards.

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.

5.0 References

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