Saltstone 3QCY14 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 third quarter of calendar year 2014 (3QCY14). After a 78 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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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-AES	Inductively Coupled Plasma – Atomic Emission Spectroscopy
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
SWLF	Solid Waste Landfill
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
WAC	Waste Acceptance Criteria

1.0 Introduction

The Saltstone Production Facility (SPF) receives waste from Tank 50H for treatment. In the third quarter of the 2014 calendar year (3QCY14), Tank 50H accepted transfers of approximately 12.2 kgal from the Effluent Treatment Project (ETP), approximately 2.2 kgal from 211H, approximately 474.2 kgal from the Actinide Removal Process / Modular Caustic Side Solvent Extraction Unit (ARP/MCU) Decontaminated Salt Solution Hold Tank (DSS-HT), approximately 13.6 kgal from 512-S, and approximately 3.1 kgal from other sources.[1]

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).[2] During operation, samples were collected from Tank 50H and grout samples prepared to determine the nonhazardous 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).[3]

Savannah River National Laboratory (SRNL) was requested [4,5] to prepare saltstone from samples of Tank 50H obtained July 8, 2014 during 3QCY14 to determine the nonhazardous nature of the grout. The samples were cured and shipped to Southwest Research Institute (SWRI) to perform the Toxic Characteristic Leaching Procedure (TCLP) [6] 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.[7]

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.

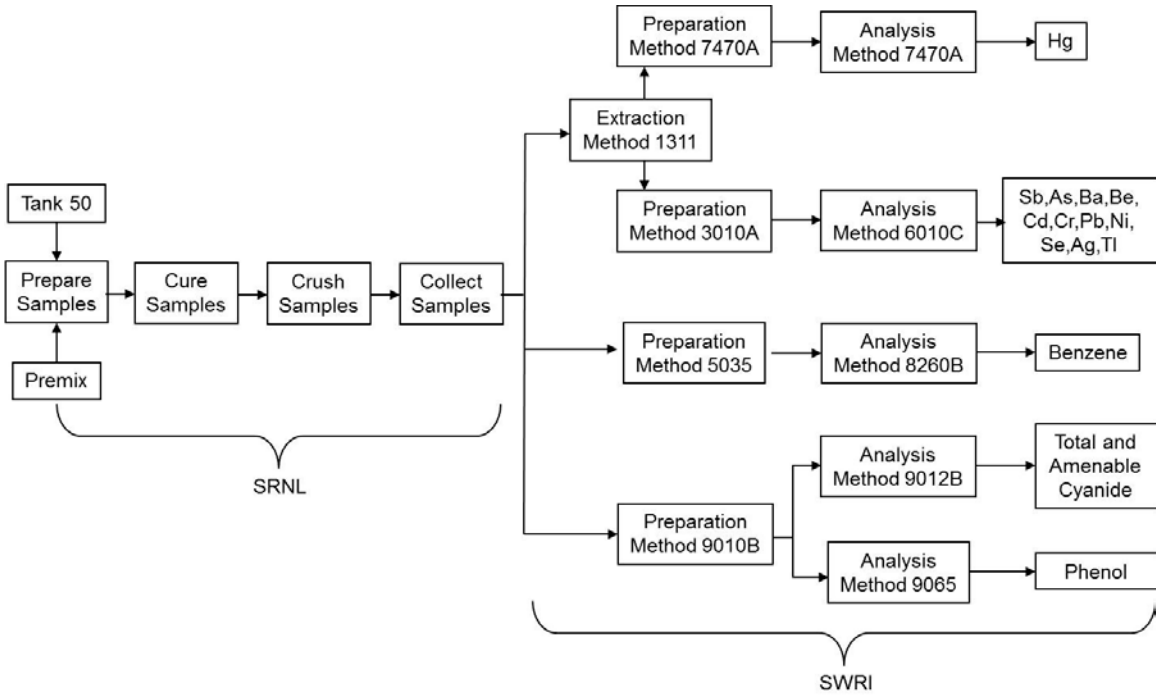

Figure 2-1: Saltstone Preparation and Analysis Flowsheet

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[8]. Table 2-1 lists the concentration of TCLP metals of interest in the salt solution from the WAC analysis for the sample.[8] As shown in Table 2-1 the contents of Tank 50H exceed the regulatory limits for antimony, cadmium, chromium, mercury, and phenol, and therefore must be treated and disposed of in a nonhazardous waste form. Table 2-2 contains the parameters used to prepare the TCLP sample.[1]

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 premix material was slowly added to the salt solution and 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 78 days, the 3QCY14 saltstone sample was removed from the container and a portion of the saltstone was crushed to pass through a 9.5 mm (3/8 inch) standard sieve as prescribed by Section 7.1.3 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."[2] ESS-WP retrieved the sample from SRNL and transported them to SWRI for extraction and analysis.

Table 2-1: TCLP Metals and UHCs from Tank 50H WAC Analyses Results and Limits

Analyte	Sample Result [8] 3Q14 (mg/L)	Regulatory Limit Toxicity[a] (mg/L)
As	<0.0945	5
Ba	0.584	100
Cd	<1.09	1
Cr	31.5	5
Pb	0.212	5
Hg	59.2	0.2
Se	<0.189	1
Ag	<1.08	5
Analyte	**3Q14 (mg/L)**	**UHC[b] (mg/L)**
Sb	<35.2	1.15
Be	<0.120	1.22
Ni	<4.24	11
Tl	<0.0244	0.20
Analyte	**3Q14 (mg/kg)**	**UHC[b] (mg/kg)**
Benzene	<0.150	10
Amendable Cyanide	NM	30
Total Cyanide	NM	590
Total Phenol	<10.0	6.2

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 TCLP Sample Preparation

Parameter	3QCY14
Water-to-Premix Ratio	0.59
(Daratard 17) gal/Ton Premix	0
(Dow Corning Q2-3183A) gal/Ton Premix	0.00

Saltstone Mix Data Sheet

MIX # 0152		Date: 7/28/2014	
Material	**%**	**WT%**	**Grams**
Waste Solution: Tank 50 07/08/14 3Q14 **Wt% Solids #** 27.858 **Grams Water** 231.32		44.99	320.65
Admixture: Q2 Antifoam*			0.00
Admixture:			0.00
Admixture:			
Premix		55.01	392.00
Cement (% of Premix)	10	5.50	39.20
Slag (% of Premix)	45	24.75	176.40
Fly Ash (% of Premix)	45	24.75	176.40
Total	100	100.00	712.65
Water to Premix Ratio	0.59		

Calculations:
Use CBO fly ash

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

Figure 2-2: Saltstone Mix Data Sheet for the 3QCY14 TCLP Sample

2.2 Saltstone Testing

Saltstone testing was performed by SWRI. Activities associated with the 3QCY14 saltstone sample were TCLP extraction, TCLP leachate digestion and analysis, extraction of solid subsamples and extract analysis. The sample arrived at SWRI in San Antonio, Texas on October 16, 2014 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, as noted in the SWRI report[9].

The volatile compound, benzene, was prepared by SW-846 Method 5035 and 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 samples were extracted by a modified SW-846 Method 1311. The method was 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-AES, SW-846 Method 6010C.

2.3 Quality Assurance

Requirements for performing reviews of technical reports and the extent of review are established in Manual E.7 2.60. SRNL documents the extent and type of review using the SRNL Technical Report Design Checklist contained in WSRC-IM-2002-00011, Rev. 2.

3.0 Results and Discussion

The following tables summarize the analytical results provided by the vendor, SWRI. The entire vendor report is documented and included as a reference.[9] 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 or relative percent differences. 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 two groups:

- Detected at or below the RLs - arsenic, cadmium, chromium, silver, thallium, beryllium, benzene, amenable cyanide, and total phenol.
- Detected above the RLs - barium, lead, mercury, selenium, antimony, nickel, and total cyanide

Table 3-1: TCLP Metals and UHCs Results

Analyte	Result	Q*	Reporting Limit	Unit	Method
Arsenic	20.0	U	20.0	µg/L	1311/6010C
Barium	1870		5.0	µg/L	1311/6010C
Cadmium	5.0	U	5.0	µg/L	1311/6010C
Chromium	5.0	U	5.0	µg/L	1311/6010C
Lead	13.4		5.0	µg/L	1311/6010C
Mercury	12.6		0.4	µg/L	1311/7470A
Selenium	37.8		20.0	µg/L	1311/6010C
Silver	5.0	U	5.0	µg/L	1311/6010C
Antimony	38.7		20.0	µg/L	1311/6010C
Nickel	47.9		5.0	µg/L	1311/6010C
Thallium	20.0	U	20.0	µg/L	1311/6010C
Beryllium	5.0	U	5.0	µg/L	1311/6010C
Benzene	1.1	U	1.1	µg/kg	EPA 8260B
Amenable Cyanide	0.193	U	0.193	mg/kg	EPA 9012B
Total Cyanide	8.63		0.193	mg/kg	EPA 9012B
Total Phenol	0.871	U	0.871	mg/kg	EPA 9065

"-" Indicates a location in the table for which an entry would not be appropriate.
*Q - Qualifier Column
U - compound was analyzed for, but not detected above the RL

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 converted 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) from the State Primary Drinking Water Regulations[3] are 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 Non-wastewater Standard for all of the metals and UHCs.
- Barium, chromium, mercury, selenium, silver and antimony were below the MCL.
- Arsenic, cadmium, lead, 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 Salt Disposal Facility (SDF) vaults are permitted as a Class 3 landfill. None of the analytes were greater than 10x the MCL.

Table 3-2: TCLP and UHC Results and Corresponding Regulatory Limits

Analyte	Result (mg/L)	Q*	Regulatory Limit		
			Toxicity[a]	UTS[b]	MCL[c]
			(mg/L)	(mg/L)	(mg/L)
Arsenic	2.0E-02	U	5	5	0.010
Barium	1.87E+00		100	21	2
Cadmium	5.00E-03	U	1	0.11	0.005
Chromium	5.00E-03	U	5	0.6	0.1
Lead	1.34E-02		5	0.75	0.015[d]
Mercury	1.26E-02		0.2	0.025	2E-03
Selenium	3.78E-02		1	5.7	0.05
Silver	5.00E-03	U	5	0.14	0.1[e]
Antimony	3.87E-02		-	1.15	0.006
Nickel	4.79E-02		-	11	-
Thallium	2.00E-02	U	-	0.20	2E-03
Beryllium	5.00E-03	U	-	1.22	4E-03
Analyte	**Result**	**Q***	**Toxicity[a]**	**UTS[b]**	**MCL[c]**
	(mg/kg)		**(mg/L)**	**(mg/kg)**	**(mg/L)**
Benzene	1.1E-03	U	-	10	-
Amenable Cyanide	1.93E-01	U	-	30	-
Total Cyanide	8.6E+00		-	590	-
Total Phenol	8.71E-01	UJ	-	6.2	-

"-" Indicates a location in the table for which an entry would not be appropriate.
*Q - Qualifier Column
U - compound was analyzed for, but not detected above the RL
J - MS/MSD recoveries were not within specification
[a] SCDHEC R.61-79.261.24(b) "Characteristic of Toxicity"
[b] SCDHECR 61-79.268.48 "Universal Treatment Standards", Non- waste water standard
[c] SCDHEC R.61-58.5(B) " Maximum Contaminant Levels for Inorganic Chemicals"
[d] Lead action level from SCDHEC R.61-58.11.B
[e] Secondary drinking water parameter

3.2 Analytical Quality Control

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. [9]

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	Unit	Q*
Arsenic	20.0	µg/L	U
Barium	5.0	µg/L	U
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	20.0	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	0.5	µg/kg	U
Amenable Cyanide	0.217	mg/kg	U
Total Cyanide	0.217	mg/kg	U
Total Phenol	0.892	mg/kg	U

*Q - Qualifier Column
U- compound was analyzed for, but not detected above the RL

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		Unit	Recovery (%)
	True	Recovery		
Arsenic	4000	3440	µg/L	86.0
Barium	4000	3280	µg/L	82.0
Cadmium	100	86.4	µg/L	86.4
Chromium	400	334	µg/L	83.5
Lead	1000	843	µg/L	84.3
Mercury	1.0	1.08	µg/L	108.0
Selenium	4000	3310	µg/L	82.8
Silver	100	80.7	µg/L	80.7
Antimony	1000	839	µg/L	83.9
Nickel	1000	802	µg/L	80.2
Thallium	4000	3490	µg/L	87.2
Beryllium	100	87.3	µg/L	87.3
Benzene	10	12	µg/kg	120
Amenable Cyanide	NA	NA	mg/kg	NA
Total Cyanide	76.5	47.3	mg/kg	61.8
Total Phenol	NA	NA	mg/kg	NA

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. Post digestion spikes were performed for total phenol and found to be acceptable.

- 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. Post digestion spikes were performed for total phenol and found to be acceptable.

- The RPD(s) between the MS and MSD met the acceptance limits (0 – 30%).

- The RPD(s) between the Sample and Sample Duplicate met the acceptance limits. Phenol and amenable cyanide are reported as zero since the initial values were below the detection limit.

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

Analyte	Unit	Initial Concentration			Spiked Sample		Recovery (%)	
		Sample	Spike Added		Spike	Spike Duplicate	Spike	Spike Duplicate
Arsenic	µg/L	20.0*	2500		2470	2540	98.8	101.6
Barium	µg/L	1870	5000		5810	5940	78.8	81.4
Cadmium	µg/L	5.0*	500		425	435	85.0	87.0
Chromium	µg/L	5.0*	1000		813	832	81.3	83.2
Lead	µg/L	13.4	2500		2060	2120	81.9	84.3
Mercury	µg/L	12.6	4.00		15.7	15.8	77.5	80.0
Selenium	µg/L	37.8	2500		2460	2530	96.9	99.7
Silver	µg/L	5.0*	500		457	467	91.4	93.4
Antimony	µg/L	38.7	5000		4630	4730	91.8	93.8
Nickel	µg/L	47.9	2500		1980	2020	77.3	78.9
Thallium	µg/L	20.0*	2500		2180	2230	87.2	89.2
Beryllium	µg/L	5.0*	500		455	465	91.0	93.0
Benzene	µg/kg	1.1*	24	23	23	22	96	96
Amenable Cyanide	mg/kg	0.193*	NA		NA	NA	-	-
Total Cyanide	mg/kg	8.63	17.4	18.6	27.0	23.4	106	79.4
Total Phenol [J]	mg/kg	0.871*	23.8	17.3	0.984	0.929	4.13	5.37

NA - Not Applicable
"-" Indicates a location in the table for which an entry would not be appropriate.
* compound was analyzed for, but was not detected above the RL on original sample
[J] Matrix spike/matrix spike duplicate recovery is outside specified limits – Note: Post prepared spike was ≥75%

Table 3-6: Duplicates and Relative Percent Difference

Analyte	Unit	Q*	Initial Sample	Duplicate Sample	RPD
Arsenic	µg/L	U	20.0	20.0	-
Barium	µg/L		1870	1820	2.7
Cadmium	µg/L	U	5.0	5.0	-
Chromium	µg/L	U	5.0	5.0	-
Lead	µg/L	D	13.4	13.2	1.5
Mercury	µg/L		12.6	12.7	0.8
Selenium	µg/L		37.8	37.5	0.8
Silver	µg/L	U	5.0	5.0	-
Antimony	µg/L		38.7	28.2	31.4
Nickel	µg/L		47.9	48.1	0.4
Thallium	µg/L	U	20.0	20.0	-
Beryllium	µg/L	U	5.0	5.0	-
Benzene	µg/kg	U	1.1	-	-
Amenable Cyanide	mg/kg	U	0.193	0.201	0.0
Total Cyanide	mg/kg		8.63	7.11	19.3
Total Phenol	mg/kg	U	0.871	0.901	0.0

"-" Indicates a location in the table for which an entry would not be appropriate.
*Q - Qualifier Column
U - compound was analyzed for, but not detected above the RL
D - result is reported from a dilution

3.2.4 *Calibration Information*

- All initial calibration requirements have been met for this sample delivery group (SDG).
- All Contract Required Reporting Limit requirements meet 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 3QCY14 saltstone samples and the subsequent TCLP analyses showed that:

- The saltstone waste form disposed of in the Saltstone Disposal Facility in 3QCY14 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 3QCY14 met the SCHWMR R.61-79.261.24(b) RCRA metals requirements for a nonhazardous waste form. The sample 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 3QCY14 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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