

Keywords: *Saltstone*

☐ *Regulatory*

☐ *TCLP*

Retention: *Permanent*

Saltstone 4QCY10 TCLP Results

M.M. Reigel

March 2011

Savannah River National Laboratory
Savannah River Nuclear Solutions
Aiken, SC 29808

Prepared for the U.S. Department of Energy under
contract number DE-AC09-08SR22470.



DISCLAIMER

This work was prepared under an agreement with and funded by the U.S. Government. Neither the U.S. Government or its employees, nor any of its contractors, subcontractors or their employees, makes any express or implied:

1. warranty or assumes any legal liability for the accuracy, completeness, or for the use or results of such use of any information, product, or process disclosed; or
2. representation that such use or results of such use would not infringe privately owned rights; or
3. endorsement or recommendation of any specifically identified commercial product, process, or service.

Any views and opinions of authors expressed in this work do not necessarily state or reflect those of the United States Government, or its contractors, or subcontractors.

Printed in the United States of America

**Prepared for
U.S. Department of Energy**

REVIEWS AND APPROVALS

AUTHORS:

M.M. Reigel, Engineering Process Development

Date

TECHNICAL REVIEW:

R.E. Eibling, Engineering Process Development

Date

APPROVAL:

A.B. Barnes, Manager
Engineering Process Development

Date

S.L. Marra, Manager
Environmental & Chemical Process Technology Research Programs

Date

J.E. Occhipinti, Manager
Waste Solidification Engineering

Date

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 fourth quarter of calendar year 2010 (4QCY10). After the prescribed 28 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.

TABLE OF CONTENTS

LIST OF TABLES	vi
LIST OF FIGURES	vi
LIST OF ABBREVIATIONS	vii
1.0 Introduction	1
2.0 Experimental Procedure	1
2.1 Saltstone Preparation	2
2.2 Saltstone Testing.....	5
2.2.1 B&W TSG-RACL.....	5
2.2.2 GEL Laboratories, LLC	5
3.0 Results and Discussion	6
3.1 B&W TSG-RACL	6
3.1.1 Comparison of Results to Regulatory Limits	7
3.1.2 Quality Assurance	8
3.1.3 Blanks.....	8
3.1.4 Laboratory Control Samples	9
3.1.5 Matrix Spikes	10
3.1.6 Calibration Information.....	10
3.2 GEL Laboratories, LLC	11
3.2.1 Comparison of Results to Regulatory Limits	11
3.2.2 Quality Assurance	12
3.2.3 Blanks.....	12
3.2.4 Laboratory Control Samples	13
3.2.5 Matrix Spikes	13
3.2.6 Calibration Information.....	13
4.0 Conclusions	14
5.0 References	15

LIST OF TABLES

Table 2-1. Sample Results of TCLP Metals from Tank 50H WAC Analysis.	3
Table 2-2. Customer Recommended Values for Preparation of TCLP Sample.	3
Table 3-1. TCLP Leachates RCRA Metal Concentrations, DLs, and QLs.	6
Table 3-2. Saltstone TCLP Results and Corresponding Regulatory Limits.	8
Table 3-3. TCLP Blank.	9
Table 3-4. RCRA Metal Laboratory Control Sample.....	9
Table 3-5. TCLP Leachates RCRA Metal Matrix Spike and Duplicate Results.....	10
Table 3-6. Total Concentrations, DLs, and RLs.....	11
Table 3-7. Saltstone Total Results and Corresponding Regulatory Limits.	12
Table 3-8. Method Blank.....	12
Table 3-9. Laboratory Control Sample.....	13
Table 3-10. Matrix Spike Results.....	13

LIST OF FIGURES

Figure 2-1. Flowchart of Saltstone preparation and analysis.	2
Figure 2-2. Data sheet for the Saltstone mix used to prepare the 4Q10 TCLP sample.	4

LIST OF ABBREVIATIONS

B&W TSG-	B & W Technical Services Group-Radioisotope and Analytical
RACL	Chemistry Laboratory
CVAA	Cold Vapor Atomic Absorption
DL	Detection Limit
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
QL	Quantitation Limit
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
SRNL	Savannah River National Laboratory
TCLP	Toxic Characteristic Leaching Procedure
UHC	Underlying Hazardous Constituent
UTS	Universal Treatment Standards

1.0 Introduction

The Saltstone Production Facility (SPF) receives waste from Tank 50H for treatment. In the fourth quarter of the 2010 calendar year (4QCY10), Tank 50H accepted transfers of approximately 8 kgal from the Effluent Treatment Project (ETP), approximately 1 kgal from Tank 710—the H-Canyon General Purpose Evaporator, approximately 34 kgal from the H-Canyon Super Kukla campaign, approximately 338 kgal from the Modular Caustic Side Solvent Extraction Unit (MCU) Decontaminated Salt Solution Hold Tank (DSS-HT), and approximately 6 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 (ISWLF)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 October 4, 2010 during 4QCY10 to determine the non-hazardous nature of the grout. The samples were cured and shipped to Babcock & Wilcox Technical Services Group-Radioisotope and Analytical Chemistry Laboratory (B&W TSG-RACL) to perform the Toxic Characteristic Leaching Procedure (TCLP)² 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 included the underlying hazardous constituents (UHC) antimony, beryllium, nickel, and thallium which could not be eliminated from analysis by process knowledge.³ B&W TSG-RACL provided subsamples to GEL Laboratories, LLC for analysis for the UHCs benzene, phenols and total and amenable cyanide.

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 both B&W TSG-RACL facility in Lynchburg, Virginia and the GEL laboratory facility in Charleston, South Carolina. Figure 2-1 is a flowchart of the steps taken to prepare and characterize the saltstone samples.

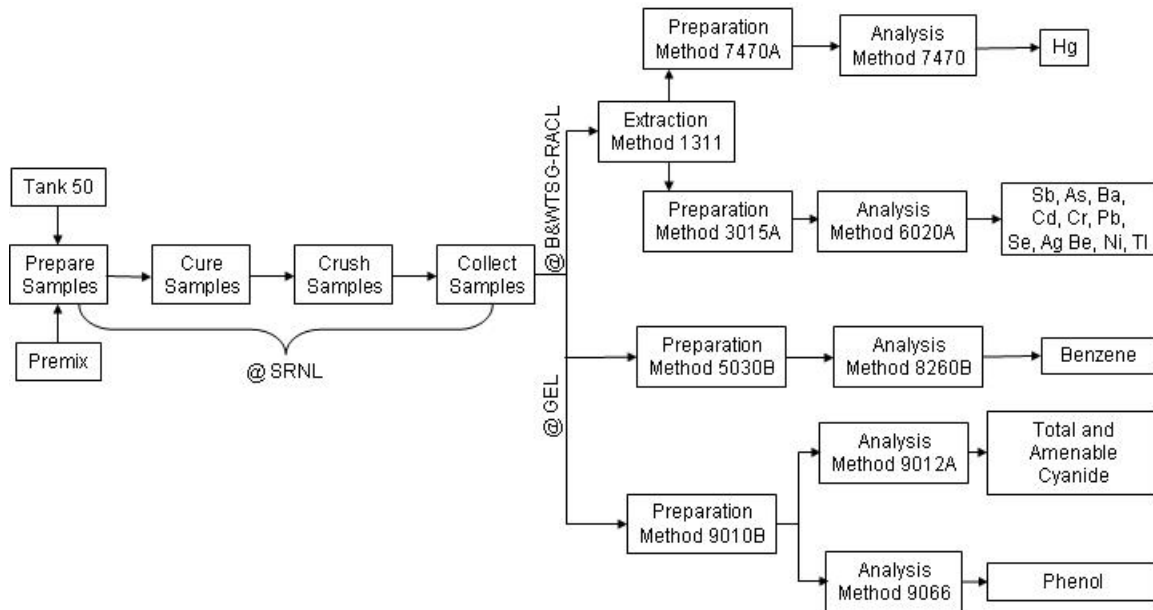


Figure 2-1. Flowchart of Saltstone preparation and analysis.

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 analysis⁴ for the sample. As shown in Table 2-1, the contents of Tank 50H exceed the regulatory limits for antimony, chromium, mercury, 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 salt solution, admixtures and premix materials were combined in a blender and mixed at low speed for one minute, inspected for incorporation of the premix, and then mixed at high speed for an additional two minutes. 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^a— 46 days for the 4Q10 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.² 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.”⁶ ESS-WP retrieved the samples from SRNL and transported them to B&W TSG-RACL for extraction and analysis. B&W TSG-RACL repackaged a portion of the sample and shipped the sample to GEL Laboratories to perform totals analysis for the UHCs benzene, phenol and total and amenable cyanide.

^a Samples are considered ready for analysis after 28 days. Samples are not crushed until shipment has been scheduled.

Table 2-1. Sample Results of TCLP Metals from Tank 50H WAC Analysis.

-	Sample Results (mg/L) ⁴	Regulatory Limits (mg/L)
-	4Q10	Toxicity^a
As	< 0.107	5
Ba	< 0.818	100
Cd	< 0.696	1
Cr	45.8	5
Pb	< 0.216	5
Hg	10.8	0.2
Se	< 0.214	1
Ag	< 1.81	5
--	--	UHC^b
Sb	< 6.70	1.15
Be	< 0.107	1.22
Ni	9.54	11
Tl	< 0.0826	0.20
-	-	(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	4Q10
Water-to-Premix ratio	0.60
(Daratard 17) gal/Ton premix	0.00
(Dow Corning Q2-1383A) gal/Ton premix	0.15

Saltstone Mix Data Sheet

MIX # 0120		Date: 11/12/2010	
Material	%	WT%	Grams
Waste Solution: Tank 50 10/4/10 4Q10 Wt% Solids # 23.07 Grams Water 167.56		43.75	217.81
Admixture: Q2 Antifoam*		0.03	0.08
Admixture: _____			
Admixture: _____			
Premix		56.24	280.00
Cement (% of Premix)	10	5.62	28.00
Slag (% of Premix)	45	25.31	126.00
Fly Ash (% of Premix)	45	25.31	126.00
Total	100	100.01	497.89
Water to Premix Ratio	0.60		
Calculations: Use CBO fly ash From customer: 0.60 w/p, 0.10 gpm, 33T/hr dry feed Q2 is diluted Q2 amount . In plant, diluted 1:4 in water. * Actual amount of Q2 added to sample is 0.078 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 the Saltstone mix used to prepare the 4Q10 TCLP sample.⁵

2.2 Saltstone Testing

Saltstone testing was performed by B&W TSG-RACL and GEL Laboratories, LLC. Activities associated with the 4QCY10 saltstone samples were:

At B&W TSG-RACL,

- performing the TCLP extraction,
- digesting the TCLP leachate, and
- analyzing the digested leachate.

At GEL

- performing extractions on solid subsamples shipped from B&W TSG-RACL and
- analyzing extracts.

2.2.1 *B&W TSG-RACL*

The samples arrived at B&W TSG-RACL, Lynchburg, Virginia on January 20, 2011 for analysis. Shipping container temperatures were documented to be within specifications. 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 Metals method 6020A analysis was performed on an X-7 Series Inductively Coupled Plasma – Mass Spectrometer (ICP-MS). The instrument measures ions produced by a radio-frequency inductively coupled plasma. Analyte species originating in a liquid are nebulized and the resulting aerosol transported by argon gas into the plasma torch. The ions produced by high temperatures are entrained in the plasma gas and introduced, by means of an interface, into a mass spectrometer. The ions produced in the plasma are sorted according to their mass-to-charge ratios and quantified with a channel electron multiplier. Mass interferences must be assessed and valid corrections applied or the data flagged to indicate problems.

The Metals method 7470A analysis was performed on a Leman PC 200 II instrument which consists of a cold vapor atomic absorption spectrometer (CVAA) set to detect mercury at a wavelength of 253.7 nm. The mercury is reduced to the elemental state and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of an atomic absorption spectrophotometer. Absorbance (peak height) is measured as a function of mercury concentration.

A portion of the leachate from the fourth quarter sample was used as the quality control sample (matrix spike) for the ICP-MS and CVAA.

2.2.2 *GEL Laboratories, LLC*

The subsamples arrived at GEL Laboratories, LLC, Charleston, South Carolina on January 25, 2011 for analysis. Shipping container temperatures were documented to be within specifications. All sample containers arrived without any visible signs of tampering or breakage. The sample arrived with the proper chain of custody documentation and signatures. However, due to shipping delays from the Savannah River Site, the hold time for organics analyses was missed. The organics were analyzed despite the missed hold times.

The method 8260B analysis was performed with an HP6890/HP5973 gas chromatograph/mass spectrometer using a J&W DB-624 column. Methods 9012A and 9066 were performed using a Lachat QuickChem FIA+ 8000 Series.

3.0 Results and Discussion

Results summarized in the following tables are from the data package for these analyses.⁷ Data is presented in these results as reported by the vendors.

3.1 B&W TSG-RACL

Analytes detected but at concentrations too low to determine quantitatively have been flagged with the “B” qualifier. Analytes that were not detected have been flagged with the “U” qualifier. In addition to the results, Detection Limits (DLs) have been given. The DL is the minimum concentration of an analyte that can be identified, measured, and reported with 99% confidence that the concentration is above zero. The DL values given in the table are the results from this study adjusted for sample dilution. The Quantitation Limit (QL) is the lowest level at which an analyte may be accurately and reproducibly achieved.

Results in Table 3-1, when compared with the DLs and QLs, can be organized into three groups:

- Beryllium, silver and thallium were not detected in the leachate.
- Antimony, cadmium, chromium, lead, and nickel were detected below the QLs.
- Arsenic, barium, mercury, and selenium were detected in the leachates at concentrations above the QLs.

Table 3-1. TCLP Leachates RCRA Metal Concentrations, DLs, and QLs.

-	Methods	Sample Limits (µg/L)	Sample Limits (µg/L)	Sample Results (µg/L)
SRS ID	-	-	-	4Q10
B&W ID	-	DL	QL	1101017-01A
Sb	3015, 6020A	0.13	11.11	^B 1.9
As	3015, 6020A	0.10	5.56	11.6
Ba	3015, 6020A	0.44	55.6	258
Cd	3015, 6020A	0.11	5.56	^B 0.28
Cr	3015, 6020A	0.31	11.1	^B 7.2
Pb	3015, 6020A	0.48	5.56	^B 5.3
Hg	7470A	0.07	0.200	^N 3.04
Se	3015, 6020A	0.24	27.8	^E 125
Ag	3015, 6020A	0.06	5.56	^{U,N,*} 0.06
Be	3015, 6020A	0.16	5.56	^U 0.16
Ni	3015, 6020A	1.6	5.56	^B 5.1
Tl	3015, 6020A	0.21	5.56	^U 0.21

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

^U Final concentration of the analyte was found to be below the DL.

^B Analyte is present at a concentration above the DL but less than the QL

^N Associated Matrix Spike is outside percent recovery quality control criteria.

* Associated Duplicate is outside relative percent difference quality control criteria

^E Associated Serial Dilution is outside percent difference quality control criteria.

3.1.1 *Comparison of Results to Regulatory Limits*

Results from the TCLP leachate analyses from Table 3-1 are replicated in Table 3-2 — with units changed from µg/L to mg/L—along with 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^a also have been included in Table 3-2. By comparing the sample results and the regulatory limits in, 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.
- Antimony, barium, beryllium, cadmium, chromium, lead, silver and thallium were below the MCL's.
- Arsenic, mercury, and selenium 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.

^a Regulations 61-58 through 61-58.15 are promulgated pursuant to S.C. Code Sections 44-55-10 et seq. and are collectively known as the State Primary Drinking Water Regulations.

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

-	Sample Results (mg/L)	Regulatory Limits		
SRS ID	4Q10	Toxicity ^a	UTS ^b	MCL ^c
B&W ID	1101017-01A	(mg/L)	Nonwastewater Standard (mg/L TCLP)	(mg/L)
Sb	^B 1.90E-03	-	1.15	0.006
As	1.16E-02	5	5	0.010
Ba	2.58E-01	100	21	2
Cd	^B 2.80E-04	1	0.11	0.005
Cr	^B 7.20E-03	5	0.6	0.1
Pb	^B 5.30E-03	5	0.75	0.015 ^d
Hg	^N 3.04E-03	0.2	0.025	2E-03
Se	^E 1.25E-01	1	5.7	0.05
Ag	^{U,N,*} 6.00E-05	5	0.14	0.1 ^e
Be	^U 1.60E-04	-	1.22	4E-03
Ni	^B 5.10E-03	-	11	-
Tl	^U 2.10E-04	-	0.20	2E-03

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

^U Final concentration of the analyte was found to be below the DL.

^B Analyte is present at a concentration above the DL but less than the QL.

^N Associated Matrix Spike is outside percent recovery quality control criteria.

* Associated Duplicate is outside relative percent difference quality control criteria

^E Associated Serial Dilution is outside percent difference quality control criteria.

^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.

3.1.2 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.⁷

3.1.3 Blanks

Blank concentrations are given in Table 3-3. In the TCLP Blank, none of the elements were present at levels above the quantitation limit. Antimony, arsenic, chromium, lead, and selenium were present at levels above their respective DLs, but below their respective QLs. Barium, beryllium, cadmium, mercury, nickel, silver and thallium were found to be below the DLs.

Table 3-3. TCLP Blank.

Analyte	TCLP Blank (µg/L)
Sb	^B 0.20
As	^B 4.65
Ba	^U 0.44
Cd	^U 0.11
Cr	^B 0.56
Pb	^B 1.79
Hg	^U 0.068
Se	^B 22.9
Ag	^U 0.06
Be	^U 0.16
Ni	^U 1.58
Tl	^U 0.21

^B Analyte is present at a concentration above the DL but less than the QL.

^U Final concentration of the analyte was found to be below the DL.

3.1.4 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. RCRA Metal Laboratory Control Sample

Analyte	Laboratory Control (µg /L)		Recovery (%) (80 – 120)
	True	Measured	
-			
Sb	328.0	313.39	96
As	719.0	744.44	104
Ba	463.0	469.28	101
Cd	476.0	463.22	97
Cr	162.0	165.50	102
Pb	280.0	289.44	103
Hg	2.7	3.02	111.9
Se	1000.0	987.22	99
Ag	194	193.78	100
Be	136.0	130.67	96
Ni	533.0	533.89	100
Tl	142.0	130.06	92

3.1.5 Matrix Spikes

Results from analysis of the matrix spike (MS) and matrix spike duplicates (MSD) are given in Table 3-5. The initial concentrations in the second column are reproduced from Table 3-1. 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%) for all applicable analytes with the exception of mercury and silver.
- The percent recoveries (%R) obtained from the MSD analyses met the recommended quality control acceptance criteria for percent recoveries (75 – 125%) for all applicable analytes with the exception of mercury.
- The RPD(s) between the MS and MSD met the acceptance limits (0 – 20%) with the exception of silver.

Table 3-5. TCLP Leachates RCRA Metal Matrix Spike and Duplicate Results.

Analyte	Initial Concentrations (µg /L)		Spiked Sample (µg /L)		Recovery (%)		RPD (%)
	B&W ID 1101017- 01A	Spike Added	Spike	Spike Duplicate	Spike	Spike Duplicate	
Sb	^B 1.9	1000	989	1103	99	110	11
As	11.6	500	567	624	111	123	10
Ba	258	2500	2854	3215	104	118	12
Cd	^B 0.28	250	239	266	95	106	11
Cr	^B 7.2	1000	996	1096	99	109	10
Pb	^B 5.3	500	541	611	107	121	12
Hg	^N 3.04	5.00	5.51	5.45	49.4	48.2	1.1
Se	125	250	375	423	100	119	12
Ag	^{U,N} 0.06	250	111	195	45	78	55
Be	^U 0.16	250	242	268	97	107	10
Ni	^B 5.1	1000	942	1038	94	103	10
Tl	^U 0.21	250	254	291	102	117	14

^U Final concentration of the analyte was found to be below the DL.

^B Analyte is present at a concentration above the DL but less than the QL.

^N Associated Matrix Spike is outside percent recovery quality control criteria.

3.1.6 Calibration Information

- All initial calibration requirements have been met for this sample delivery group (SDG).
- All Contract Required Reporting Limit standard(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 except for selenium and thallium.
- All continuing calibration verifications bracketing this SDG met the acceptance criteria.

3.2 GEL Laboratories, LLC

GEL reports general chemistry analyses on the organics in the sample. If the concentrations of benzene, phenol and cyanide are not detected or below the detection limit (<MDL) the result is reported as “ND”. Analytes detected but at concentrations too low to determine quantitatively have been flagged with the “J” qualifier. Analytes that were not detected have been flagged with the “U” qualifier. In addition to the results, Detection Limits (DLs) and Reporting Limits (RLs) have been given. The DL is the minimum concentration of an analyte that can be identified, measured, and reported with 99% confidence that the concentration is above zero. The DL values given in Table 3-6 are the results from this study adjusted for sample dilution. The RL is the lowest level at which an analyte may be accurately and reproducibly quantitated.

Table 3-6. Total Concentrations, DLs, and RLs.

-	Methods	Sample Limits (µg/kg)	Sample Limits (µg/kg)	Sample Results*
SRS ID	-	-	-	4Q10
GEL ID	-	DL	RL	271001001
benzene	5030, 8260B	40.3	134	^U ND
phenol	9010B, 9066	113	352	1340
cyanide (total)	9010B, 9012A	104	326	5740
cyanide (amenable)	9012A	104	326	^U ND

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

^U Final concentration of the analyte was found to be below the DL.

* The sample hold time was exceeded

3.2.1 Comparison of Results to Regulatory Limits

Results from the analyses from Table 3-6 are replicated in Table 3-7 — with units changed from µg/kg to mg/kg — along with the regulatory limits that may be applied to the Saltstone waste form. Table 3-7 includes the SCHWMR R.61-79.268.48 Universal Treatment Standards (UTS) for hazardous constituents. By comparing the sample results and the regulatory limits in Table 3-7, it can be concluded that for all of the analytes, the concentrations were below the Nonwastewater Standard.

Table 3-7. Saltstone Total Results and Corresponding Regulatory Limits.

-	Sample Results (mg/kg)	Regulatory Limits (mg/kg)
SRS ID	4Q10	UTS^b
GEL ID	271001001	
benzene	^U ND	10
phenol	1.34	6.2
cyanide (total)	5.74	590
cyanide (amenable)	^U ND	30

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

^U Final concentration of the analyte was found to be below the DL.

^b R.61-79.268.48 “Universal Treatment Standards”.

3.2.2 Quality Assurance

The following subsections include summaries of results from blanks, laboratory control samples, matrix spikes, and matrix spike duplicates. The data package for this task also includes data for calibration verifications, interference checks, and serial dilutions.

3.2.3 Blanks

Blank concentrations are given in Table 3-8. Target and non target analytes were detected in the Method Blank below the reporting limit. Amenable to chlorination cyanide is determined by subtracting the results determined in the chlorinated cyanide test from those determined in the total cyanide test. The Method Blanks analyzed with this Sample Delivery Group (SDG) met the acceptance criteria.

Table 3-8. Method Blank.

Analyte	Method Blank (µg/kg)
benzene	^U ND
phenol	^U ND
cyanide (total)	94.5
cyanide (amenable)	--

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

^U Final concentration of the analyte was found to be below the DL.

ND – Not Detectable

3.2.4 Laboratory Control Samples

Results from the Laboratory Control Sample (LCS) are given in Table 3-9. All LCS recoveries met the vendor laboratory acceptance. Laboratory Control Samples are clean aqueous solutions analyzed to assure integrity of the analytical technique exclusive of matrix effects.

Table 3-9. Laboratory Control Sample.

Analyte	Laboratory Control ($\mu\text{g /kg}$)		Recovery (%)	
	True	Measured		
-			-	
benzene	50.0	50.1	100	
phenol	2500	2580 2730	103	109
cyanide (total)	67900	52500	77.3	
cyanide (amenable)	--	--	--	

3.2.5 Matrix Spikes

Total cyanide is the only method where a matrix spike would be applicable. The result from analysis of the matrix spike (MS) is given in Table 3-10. These results show that the percent recoveries (%R) obtained from the MS analyses met the recommended quality control acceptance criteria for percent recoveries.

Table 3-10. Matrix Spike Results.

Analyte	Initial Concentrations ($\mu\text{g /kg}$)		Spiked Sample ($\mu\text{g /kg}$)	Recovery (%)
	GEL ID 271001001	Spike Added		
-				
cyanide	5740	4960	4200	82.5
(total)	5740	4860	4500	90.1

3.2.6 Calibration Information

- All initial calibration requirements have been met for this sample delivery group (SDG).
- All Contract Required Detection Limit standard(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 4QCY10 saltstone samples and the subsequent TCLP analyses showed that:

- The saltstone waste form disposed of in the Saltstone Disposal Facility in 4QCY10 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 4QCY10 met the SCHWMR R.61-79.261.24(b) RCRA metals requirements for a nonhazardous waste form. The TCLP leachate concentrations were less than 5x the MCLs in SCDHEC Regulations R.61-107.19, Part I C.

The saltstone waste form placed in the Saltstone Disposal Facility in 4QCY10 met the R.61-79.268.48(a) non wastewater treatment standards.

Analyses met all USEPA SW-846 quality assurance requirements. However, due to shipping delays from the Savannah River Site, the hold time for organics analyses was missed. 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

1. Liner, K.R., "Saltstone Grout Sampling (U)," Savannah River Site, ESH-EPG-2004-00318, 2004.
2. "Toxicity Characteristic Leaching Procedure," EPA SW-846, Procedure 1311.
3. Britt, T.E., "Assessment of Regulated Organics Under 40 CFR Part 268, Section 49, Universal Treatment Standards, Relative to SRS Tank Farm Waste," Savannah River Site, LWO-LWE-2007-00052, 2007.
4. Reigel, M.M. and Bibler, N.E., "Tables Containing Results for the Fourth Quarter 2010 Tank 50 WAC Sample: Chemical and Radionuclide Contaminant Results," Savannah River National Laboratory, Aiken, SC, SRNL-L3100-2010-00225 Rev. 0, December 21, 2010.
5. Reigel, M.M., "Saltstone TCLP," Savannah River National Laboratory, SRNL-NB-2009-00076.
6. Liner, K.R., Nov. 28, 2007, Private Communication
7. Reigel, M.M., "Data Package From Vendor for 4QCY10 TCLP Analysis," Savannah River National Laboratory, Aiken, SC, SRNL-L3100-2011-00040, March 3, 2011.