

Key Words:

Sodium Bearing Waste
Steam Reforming
Durability Testing
THOR Treatment Technologies®

Retention:

Permanent

**DURABILITY TESTING OF FLUIDIZED BED STEAM REFORMER
(FBSR) WASTE FORMS FOR SODIUM BEARING WASTE (SBW) AT
IDAHO NATIONAL LABORATORY (INL) (U)**

**Charles L. Crawford
Carol M. Jantzen**

AUGUST 2007

Washington Savannah River Company
Savannah River Site
Aiken, SC 29808

Prepared for the U.S. Department of Energy
Under Contract Number DEAC09-96-SR18500



SRNL
SAVANNAH RIVER NATIONAL LABORATORY

DISCLAIMER

This report was prepared for the United States Department of Energy under Contract No. DE-AC09-96SR18500 and is an account of work performed under that contract. Neither the United States Department of Energy, nor WSRC, nor any of their employees makes any warranty, expressed or implied, or assumes any legal liability or responsibility for accuracy, completeness, or usefulness, of any information, apparatus, or product or process disclosed herein or represents that its use will not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, name, manufacturer or otherwise does not necessarily constitute or imply endorsement, recommendation, or favoring of same by Washington Savannah River Company or by the United States Government or any agency thereof. The views and opinions of the authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

Printed in the United States of America

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

Key Words:

Sodium Bearing Waste
Steam Reforming
Durability Testing
THOR Treatment Technologies®

Retention:

Permanent

**DURABILITY TESTING OF FLUIDIZED BED STEAM REFORMER
(FBSR) WASTE FORMS FOR SODIUM BEARING WASTE (SBW) AT
IDAHO NATIONAL LABORATORY (INL)**

**Charles L. Crawford
Carol M. Jantzen**

AUGUST 2007

Washington Savannah River Company
Savannah River Site
Aiken, SC 29808

Prepared for the U.S. Department of Energy
Under Contract Number DEAC09-96-SR18500



SRNL
SAVANNAH RIVER NATIONAL LABORATORY

REVIEWS AND APPROVALS

AUTHOR(S):

Charles L. Crawford, Process Science and Engineering	Date
--	------

Carol M. Jantzen, Process Science and Engineering	Date
---	------

TECHNICAL REVIEWER:

John M. Pareizs, Process Science and Engineering	Date
--	------

APPROVERS:

David A. Crowley, Process Science and Engineering	Date
---	------

Richard E. Edwards, Manager, Process Science and Engineering	Date
--	------

EXECUTIVE SUMMARY

Fluidized Bed Steam Reforming (FBSR) processing of Sodium Bearing Waste simulants was performed in December 2006 by THORsm Treatment Technologies LLC (TTT). The testing was performed at the Hazen Research Inc. (HRI) pilot plant facilities in Golden, CO. FBSR products from these pilot tests on simulated waste representative of the SBW at the Idaho Nuclear Technology and Engineering Center (INTEC) were subsequently transferred to the Savannah River National Laboratory (SRNL) for characterization and leach testing. Four as-received Denitration and Mineralization Reformer (DMR) granular/powder samples and four High Temperature Filter (HTF) powder samples were received by SRNL. FBSR DMR samples had been taken from the “active” bed, while the HTF samples were the fines collected as carryover from the DMR. The process operated at high fluidizing velocities during the mineralization test such that nearly all of the product collected was from the HTF. Active bed samples were collected from the DMR to monitor bed particle size distribution.

Characterization of these crystalline powder samples shows that they are primarily Al, Na and Si, with > 1 wt% Ca, Fe and K. The DMR samples contained less than 1 wt% carbon and the HTF samples ranged from 13 to 26 wt% carbon. X-ray diffraction analyses show that the DMR samples contained significant quantities of the Al_2O_3 startup bed. The DMR samples became progressively lower in starting bed alumina with major Na/Al/Si crystalline phases (nepheline and sodium aluminosilicate) present as cumulative bed turnover occurred but 100% bed turnover was not achieved. The HTF samples also contained these major crystalline phases.

Durability testing of the DMR and HTF samples using the ASTM C1285 Product Consistency Test (PCT) 7-day leach test at 90°C was performed along with several reference glass samples. Comparison of the normalized leach rates for the various DMR and HTF components was made with the reference glasses and the Low Activity Waste (LAW) specification for the Hanford Waste Treatment and Vitrification Plant (WTP). Normalized releases from the DMR and HTF samples were all less than 1 g/m². For comparison, normalized release from the High-Level Waste (HLW) benchmark Environmental Assessment (EA) glass for Si, Li, Na and B ranges from 2 to 8 g/m². The normalized release specification for LAW glass for the Hanford WTP is 2 g/m².

The Toxicity Characteristic Leach Test (TCLP) was performed on DMR and HTF as-received samples and the tests showed that these products meet the criteria for the EPA RCRA Universal Treatment Standards for all of the constituents contained in the starting simulants such as Cr, Pb and Hg (RCRA characteristically hazardous metals) and Ni and Zn (RCRA metals required for listed wastes).

TABLE OF CONTENTS

EXECUTIVE SUMMARY	iii
LIST OF FIGURES	v
LIST OF TABLES	v
LIST OF ACRONYMS	vi
1.0 INTRODUCTION AND BACKGROUND	1
2.0 OPERATIONAL SUMMARY	2
3.0 EXPERIMENTAL	8
3.1 CHARACTERIZATION	8
3.2 DURABILITY TESTING	8
3.3 TCLP	9
4.0 RESULTS AND DISCUSSION	11
4.1 CHEMICAL COMPOSITION	11
4.2 CRYSTALLINE PHASES	15
4.3 DURABILITY TESTING	16
4.3.1 Comparison of Normalized Release to High-Level Waste	22
4.3.2 Comparison of Normalized Release to Low Activity Waste	22
4.3.3 Comparison of 2007 FBSR Product Durability Testing to Previous Work	25
4.4 TOXICITY CHARACTERISTIC LEACHING PROCEDURE	28
5.0 QUALITY ASSURANCE	30
6.0 CONCLUSIONS	31
ACKNOWLEDGEMENTS	32
7.0 REFERENCES	34
APPENDIX A - XRD SPECTRA OF DMR AND HTF SAMPLES	36
APPENDIX B – ACCURA REPORT	45

LIST OF FIGURES

Figure 2-1. Time Line of FBSR.....	4
Figure 2-2. Ternary Plot Showing DMR and HTF Samples	5
Figure 2-3. Ternary Plot Showing Adjusted DMR Compositions Relative to the Target Compositions Defined by the Starting Simulant and the Starting Clay by Adjustment for Unreacted Al_2O_3	7
Figure 4-1. Normalized Release for DMR vs. EA Glass.....	23
Figure 4-2. Normalized Release for HTF vs. EA Glass	23
Figure 4-3. Normalized Release for DMR vs. LRM Glass and Hanford Contract LAW.....	24
Figure 4-4. Normalized Release for HTF vs. LRM Glass and Hanford Contract LAW	24
Figure 4-5. Linearity of Alkali (NL(Na)) and Alumina Released to Solution.....	25
Figure 4-6. Linearity of Alkali (NL(Cs)) and Alumina Released to Solution.	26
Figure 4-7. Release of Re, S, and Si to the PCT Leachates as a Function of the pH of the Final Leachate.	27
Figure 4-8. Relation of Re to S, Re to Si, and S to Si in the PCT Leachates of the FBSR Products Tested	28
Figure A- 1. INL 4504 DMR (Sieved).....	37
Figure A- 2. INL 4531 DMR (Sieved).....	38
Figure A- 3. INL-4637-DMR (Sieved).....	39
Figure A- 4. INL-4726-DMR (Sieved).....	40
Figure A- 5. INL-4508-HTF (Not Sieved)	41
Figure A- 6. INL-4546-HTF (Not Sieved)	42
Figure A- 7. INL-4649-HTF (Not Sieved)	43
Figure A- 8. INL-4728-HTF (Not Sieved)	44

LIST OF TABLES

Table 2-1. Operational Summary of FBSR.....	3
Table 2-2. MINCALC Results for DMR Adjustments for Unreacted Al_2O_3	6
Table 4-1. Elemental Composition – Wt%.....	12
Table 4-2. Oxide Composition – Wt%	13
Table 4-3. XRD Crystalline Phases	15
Table 4-4. BET and Geometric Surface Area Data	17
Table 4-5. PCT Leachate Standards and Blanks (ppm).....	18
Table 4-6. PCT Leachate Standards and Blanks (g/L)	19
Table 4-7. PCT Leachate Concentrations (ppm)	20
Table 4-8. Normalized PCT Leachate Release (g/m^2) Using BET Surface Area.....	21
Table 4-9. Toxicity Characteristic Leaching Procedure (TCLP) Results	29

LIST OF ACRONYMS

ARM-1	Approved Reference Material - 1
ASTM	American Society for Testing and Materials
DMR	Denitration and Mineralization Reformer
DOE	Department of Energy
EA	Environmental Assessment
EPA	Environmental Protection Agency
FBSR	Fluidized Bed Steam Reformer
HLW	High Level Waste
HRI	Hazen Research Inc.
HTF	High Temperature Filter
ICP-AES	Inductively Coupled Plasma – Atomic Emission Spectroscopy
ICP-MS	Inductively Coupled Plasma – Mass Spectroscopy
INTEC	Idaho Nuclear Technology and Engineering Center
LAW	Low Activity Waste
LRM	Low-activity Reference Material
NAS	Sodium Aluminosilicates
PCT	Product Consistency Testing
RCRA	Resource Conservation and Recovery Act
SA/V	Surface Area to Volume
SBW	Sodium Bearing Waste
SRNL	Savannah River National Laboratory
TCLP	Toxicity Characteristic Leaching Procedure
TRU	Transuranic
TTT	THOR sm Treatment Technologies
UTS	Universal Treatment Standards
WAPS	Waste Acceptance Product Specifications
WFO	Work for Others
WIPP	Waste Isolation Pilot Plant
XRD	X-ray Diffraction

1.0 INTRODUCTION AND BACKGROUND

Fluidized Bed Steam Reforming (FBSR) has been selected by the United States Department of Energy (DOE) as the preferred treatment technology for the Sodium Bearing Waste (SBW) at the Idaho Nuclear Technology and Engineering Center (INTEC) [Fed. Register 2005]. The FBSR application for treatment of SBW simulants has been reported in previous studies [Mason 2006, Burket 2005, Soelberg 2004a, Soelberg 2004b]. Characterization and durability testing of FBSR bed and fines material produced at pilot scale facilities have been reported [Pareizs et al., 2005, Jantzen et al, 2006a, Jantzen et al, 2006b]. Durability testing of those materials in monolithic form was also studied [Jantzen 2006c].

Recent FBSR processing of simulated Idaho SBW was performed in December of 2006 at the Hazen Research Inc. (HRI) Facility in Golden, CO [Pilot Plant Report 2007]. Bed product materials and filter fines were produced and transferred to the Savannah River National Laboratory (SRNL) for characterization and durability testing as part of a Work for Others (WFO) project. This report presents the results of characterization, durability testing, and toxicity characteristic leaching procedure (TCLP) testing on these materials.

2.0 OPERATIONAL SUMMARY

The HRI facility tests were performed December 12-21, 2006. Figure 2-1 and Table 2-1 show operational details that were provided by TTT to SRNL. A total of four as-received Denitration and Mineralization Reformer (DMR) samples and four High Temperature Filter (HTF) samples were received by SRNL.

The DMR samples were taken from the “active” bed, and HTF are the fines collected downstream of the DMR. The process operated at high fluidization during the mineralization test such that nearly all of the product collected was from the HTF. Active bed samples were collected from the DMR to monitor bed particle size distribution.

The major difference in the samples (and tests) is the quantity of clay that was combined with the liquid waste to “mineralize” the granular solid product that was produced in the DMR and HTF. Table 2-1 shows which samples are associated with the test condition (clay addition). The numerical value of the sample log numbers reflects the time line. The test was started with 228, then 276, then 339, and finally 200 g clay/liter SBW.

A ternary plot showing the DMR and HTF ‘as-received’ sample compositions is shown in Figure 2-2. Note that as-received sample compositions will be presented in detail later in this report. This plot shows progression away from starting alumina bed turnover for the successive DMR samples. The earliest DMR sample ‘DMR 4504’ shown as the light green data point is nearest to the ‘pure’ Al_2O_3 point on the ternary diagram. As testing proceeded, successive DMR product sample compositions got further away from the Al_2O_3 portion of the ternary plot and closer to the targeted composition of the HTF samples.

One can use the software MINCALC process control strategy* to adjust the as-received DMR product compositions to account for the excess starting bed Al_2O_3 . Table 2-2 shows the MINCALC results for calculated portions of starting bed Al_2O_3 and DMR product. These adjusted compositions can be normalized based on the ternary components (Al_2O_3 , $\text{M}_2\text{O} = \text{Na}_2\text{O} + \text{K}_2\text{O} + \text{Cs}_2\text{O}$, and SiO_2), and plotted on the ternary plot shown in Figure 2-3. Figure 2-3 also shows the $\text{Na}_2\text{O}/\text{Al}_2\text{O}_3$ starting point for the SBW simulant as well as the $\text{SiO}_2/\text{Al}_2\text{O}_3$ starting point for the clay. Comparison of the adjusted DMR data shown on Figure 2-3 ternary indicates that the product portion of the as-received DMR samples are indeed close to the target composition as defined by the line connecting the SWB simulant and the clay.

* MINCALC is a spreadsheet that aids in Steam Reforming batch preparations (clay and waste amounts, carbon additions, etc.) by predicting product mineral phases. Further details of MINCALC have been previously presented in Pareizs et al., 2005.

Table 2-1. Operational Summary of FBSR

Sample Log #	Date	Time	Location	Test Condition
4504	12/15/06	0115	DMR	Production Tests P-1 & P-2 (228 g clay / L SBW Lite)
4508	12/15/06	0115, 0145	HTF	(same as above)
4531	12/16/06	0015	DMR	Production Tests P-3 (276 g clay / L SBW Lite)
4546	12/16/06	0215-0245	HTF	(same as above)
4637	12/18/06	0100	DMR	Production Tests P-4 (339 g clay / L SBW Lite)
4649	12/18/06	0100-0140	HTF	(same as above)
4726	12/20/06	0100	DMR	Production Tests P-5A (200 g clay / L SBW Lite)
4728	12/20/06	0030	HTF	(same as above)

ESTD Mineralizing (Production) Time Line

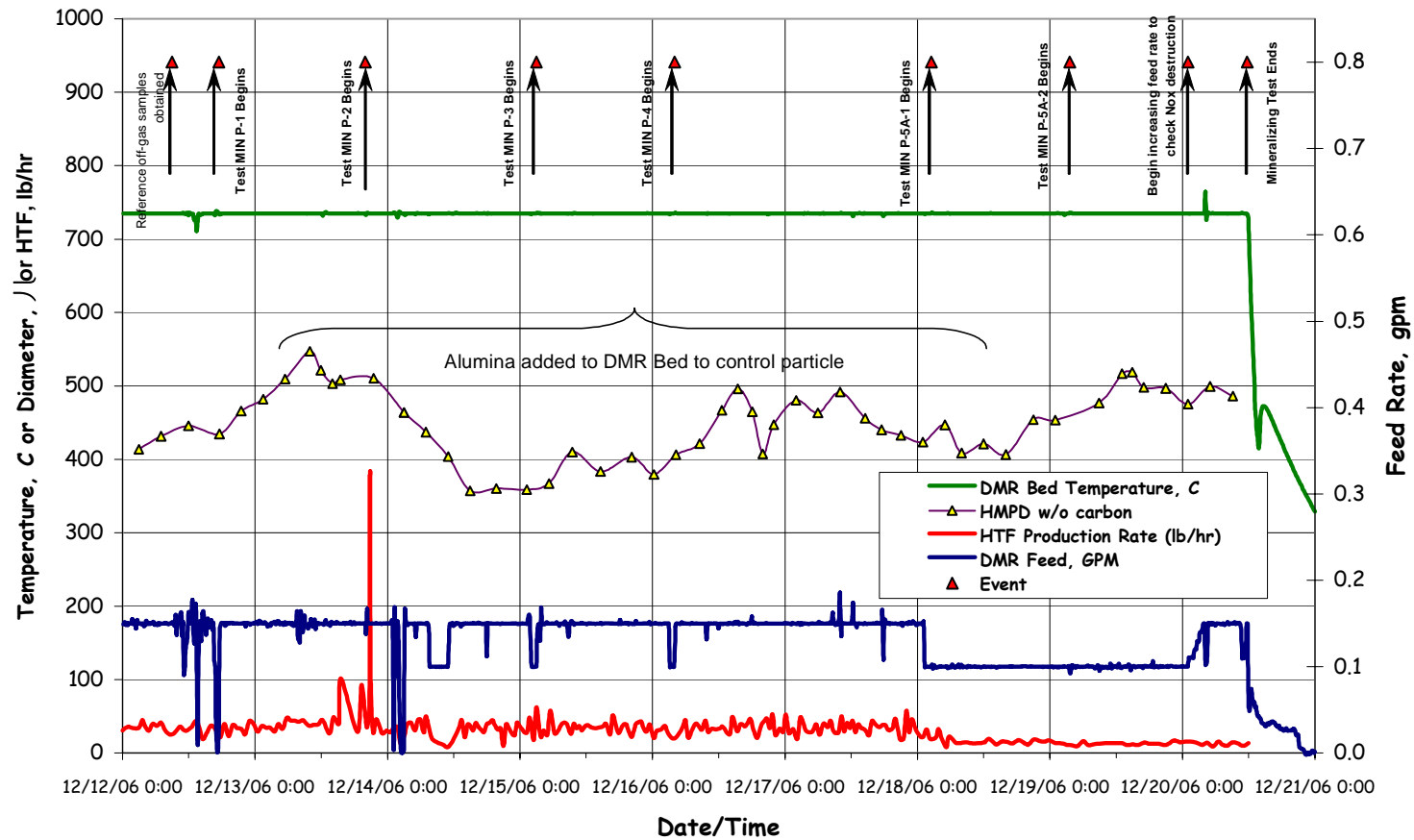
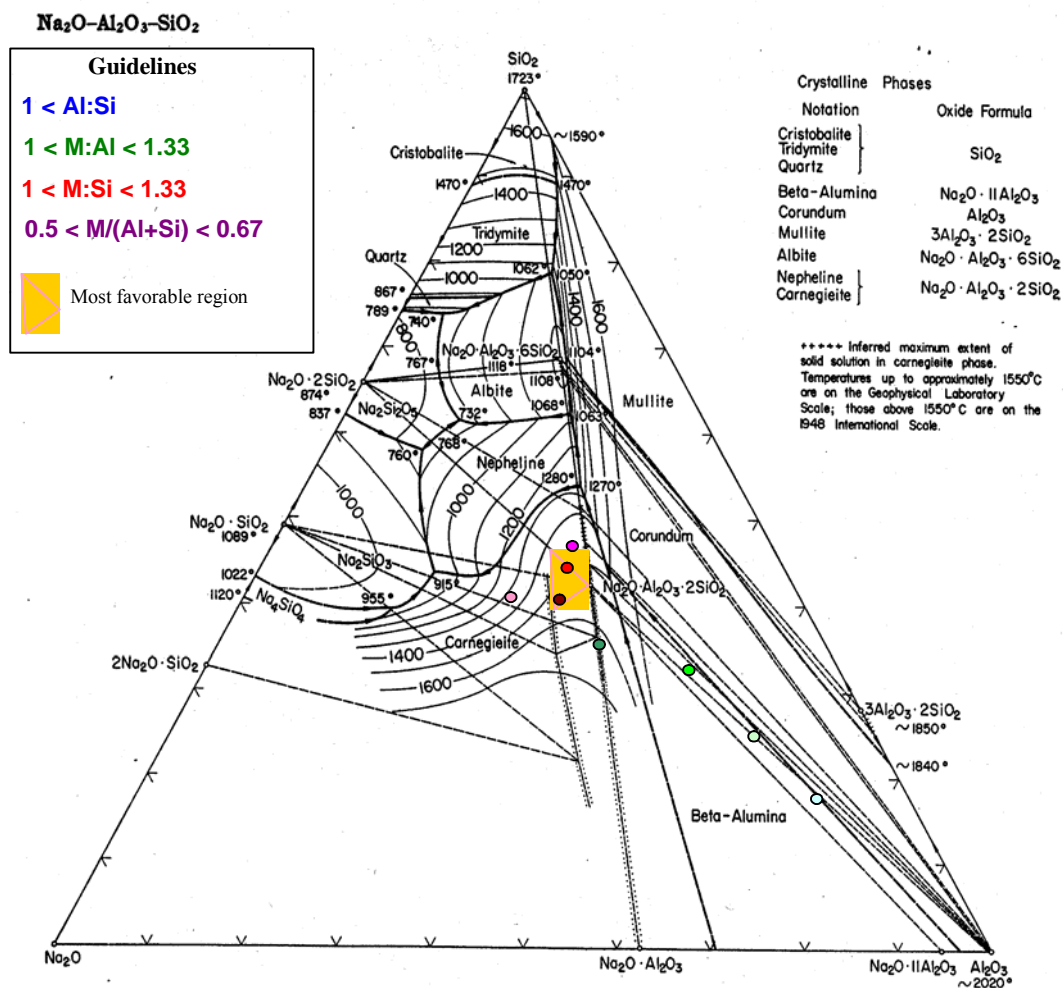


Figure 2-1. Time Line of FBSR

FIG. 501.—System Na₂O-Al₂O₃-SiO₂; composite.

E. F. Osborn and Arnulf Muan, revised and redrawn "Phase Equilibrium Diagrams of Oxide Systems," Plate 4, published by the American Ceramic Society and the Edward Orton, Jr., Ceramic Foundation, 1960.

DMR Samples	g clay / L		HTF Samples	g clay / L	
○	4504	228	●	4508	228
○	4531	276	●	4546	276
●	4637	339	●	4649	339
●	4726	200	●	4728	200

Figure 2-2. Ternary Plot Showing DMR and HTF Samples

Table 2-2. MINCALC Results for DMR Adjustments for Unreacted Al₂O₃

DMR Sample #	Date	Time		As Received	Adjusted for Unreacted Al₂O₃	Percentage Starting Bed vs. Product
4504	12/15/06	0115	SiO ₂ Alkali Al ₂ O ₃ Sum	17.30 9.85 72.85 100.00	43.17 24.57 32.26 100.00	60% Starting Bed vs. 40% Product
4531	12/16/06	0015	SiO ₂ Alkali Al ₂ O ₃ Sum	24.57 12.68 62.74 100.00	44.13 22.77 33.10 100.00	44% Starting Bed vs. 56% Product
4637	12/18/06	0100	SiO ₂ Alkali Al ₂ O ₃ Sum	32.35 15.83 51.82 100.00	44.72 21.88 33.40 100.00	28% Starting Bed vs. 72% Product
4726	12/20/06	0100	SiO ₂ Alkali Al ₂ O ₃ Sum	35.55 23.87 40.57 100.00	36.93 24.80 38.27 100.00	4% Starting Bed vs. 96% Product

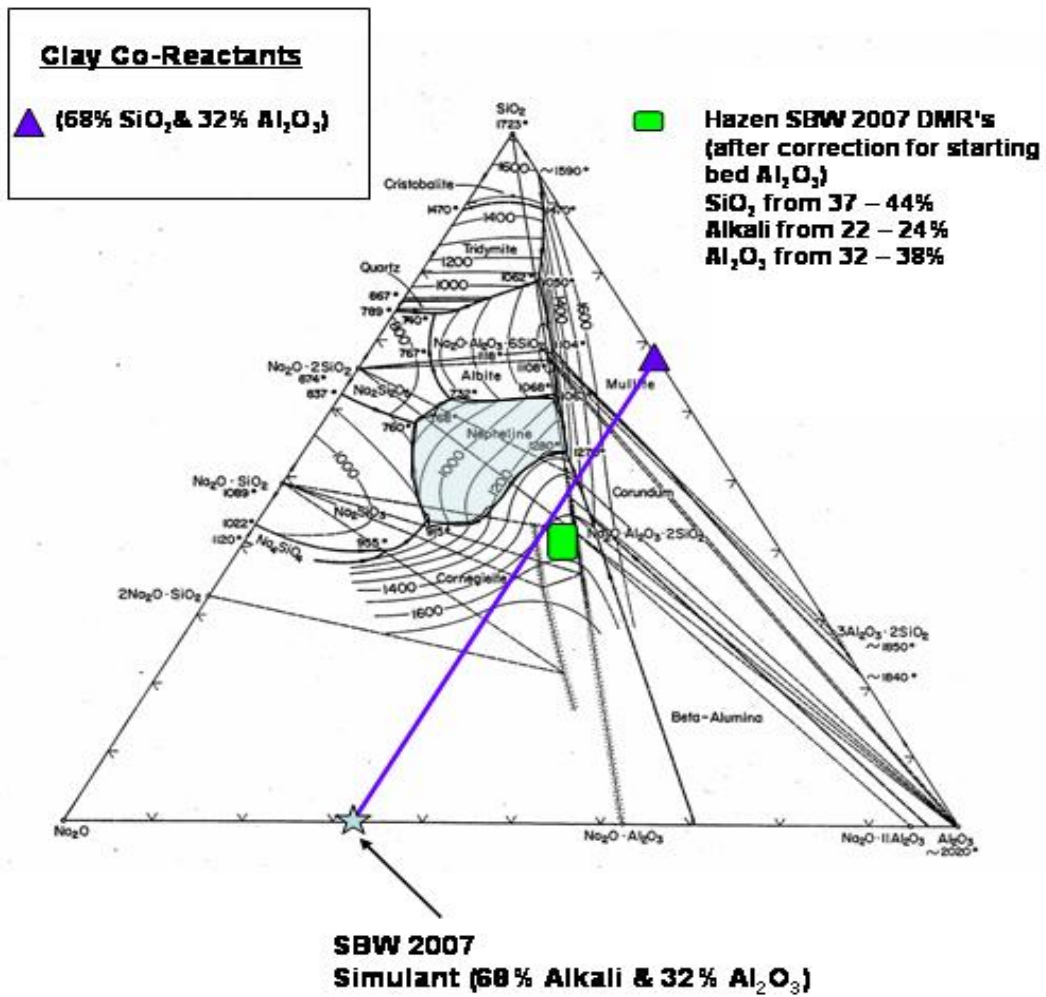


Figure 2-3. Ternary Plot Showing Adjusted DMR Compositions Relative to the Target Compositions Defined by the Starting Simulant and the Starting Clay by Adjustment for Unreacted Al₂O₃

3.0 EXPERIMENTAL

3.1 CHARACTERIZATION

Elemental and anion compositions of the steam reforming materials were measured for as received samples before and after heating of the samples at 525 °C overnight. Elemental and anion analyses were performed on lithium tetraborate fusion (1000 °C) and sodium peroxide digestion (650 °C), respectively. These methods used nominally 0.1 g of powder solid sample to 0.1L of dissolved solution and have been described in detail previously (Pareizs et al., 2005). The digestion methods for elemental analysis involves the use of acids for dissolution. Water is used in place of acid in the sodium peroxide digestion for anion analysis. All elemental concentrations (except for Hg, Re and Cs) were determined by Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP-AES). The Re and Cs were measured by Inductively Coupled Plasma Mass Spectroscopy (ICP-MS). Mercury was measured by performing a separate microwave dissolution, followed by cold vapor atomic absorption spectroscopy. REDOX (iron (II) to total iron ratio) was determined on samples that were not subjected to carbon removal, using a dissolution and absorption spectroscopy method. As received samples were also examined by powder X-ray Diffraction (XRD) to investigate the formation of the mineral phases in the FBSR waste forms.

3.2 DURABILITY TESTING

The chemical durability of the steam reformer products was determined using the Product Consistency Test (PCT) ASTM procedure C 1285-02 [ASTM 2002]. Prior to sizing and washing, carbon was removed from the material by heating overnight at 525°C. The DMR product samples were sized between (-) 100 and (+) 200 mesh (74 µm to 149 µm), which is the same size fraction used to express glass waste form performance. The HTF fines material was sieved to (-) 200 mesh. The sized material was washed six times with 100% ethanol to remove electrostatic fines. Water was not used for washing so no potential water soluble phases would be removed prior to leaching as cautioned by the ASTM C1285-02 procedure. Portions of the washed and dried DMR and HTF powders were analyzed using Microtrac – S3000 instrumentation for particle size analysis by laser light scattering. BET surface area measurements via gas adsorption, and nitrogen gas pycnometry density measurements (Quantachrome Corp.) were also performed on the sieved/washed/dried portions of the powders used for PCT. For all samples, ASTM Type I water was used as leachant, a constant leachant to sample ratio of 10 cm³/g was used, the test temperature was 90°C, and the test duration was seven days. Test duration and temperature are the nominal test conditions used for testing glass waste form performance under the PCT-A [ASTM 2002].

The PCT results can be expressed as a normalized concentration (NC_i) which has units of g waste form/L_{leachant}, or as a normalized release (NL_i) in g waste form/m². Normalized concentrations are calculated using Equation 1 and normalized release is calculated using Equation 2.

$$NC_i = C_i(\text{sample}) / f_i \quad (\text{Equation 1})$$

$$NL_i = NC_i / (SA/V) \quad (\text{Equation 2})$$

Where $C_i(\text{sample})$ is the measured leachate concentration
 f_i is the elemental weight fraction
 SA/V is the surface area to volume ratio

In order to calculate NL_i , the units used to express LAW glass durability, the surface area of the material being tested must either be calculated per ASTM C 1285, Appendix X1, or measured. In this study the SA/V was calculated using the average particle size diameter as determined from the geometric surface area and the powder bulk density via Equation 3.

$$SA/V_{\text{calc}} = 6 / (\rho \cdot d \cdot V) \quad (\text{Equation 3})$$

Where SA/V_{calc} is the calculated surface area to volume ratio based on the average particle diameter and the waste form powder density
 d is the average particle diameter (m)
 ρ is the waste form particle density (g/m^3)
 V is the volume of leachant V per g of waste form (L/g)

The other method for SA/V determination involves a measurement of the surface area by the BET method. In this method, the amount of an inert gas that condenses on a powdered sample is measured at a temperature near the boiling point of the gas. The amount of gas condensed on the sample is measured by the pressure change in the system upon exposure to the sample. This method measures all open pores, inclusions, irregularities, etc. that are penetrable by the inert gas. The SA/V ratio is calculated by dividing the measured BET surface area by the leachant volume via Equation 4.

$$SA/V_{\text{BET}} = SA_{\text{BET}} / V \quad (\text{Equation 4})$$

3.3 TCLP

The INL SBW is a listed waste under the EPA Resource Conservation and Recovery Act (RCRA). When treated, the waste form must retain the hazardous components at the Universal Treatment Standard (UTS) limits [Land Disposal Restrictions 2004]. The Land Disposal Restrictions (LDRs) do not apply to Transuranic (TRU) waste forms disposed of at the Waste Isolation Pilot Plant (WIPP). However, if a mineralized waste form were to be disposed of at the Federal Repository (such as Yucca Mountain), then LDRs (UTS) would apply.

All DMR and HTF samples were evaluated for retention of the hazardous metals by the EPA Toxicity Characteristic Leaching Procedure, Method 1311 (TCLP) [TCLP Method 1311, 1986]. Greater than 100 g samples of as-received material were submitted to Accura Analytical Laboratory, Inc. of Norcross, GA, an EPA-certified laboratory. In the leaching procedure, 100 g samples are extracted by an acidic fluid for 18 hours. The extraction fluid (leachate) is then filtered and analyzed for elements of interest. Since organics are destroyed in the FBSR process, only the following RCRA hazardous inorganic species were measured: As, Ba, Cd, Cr, Pb, Se,

Ag, Hg, Ni, and Zn It should be noted that although all of these constituents were analyzed for in the TCLP, the elements As, Ba, Cd, Se and Ag were not added to the SBW simulant that was processed. If the concentration of a hazardous inorganic species from the simulated waste form is higher than the UTS limits, then it is assumed that a real waste treated in a similar manner would fail the UTS limits and require further remediation.

4.0 RESULTS AND DISCUSSION

4.1 CHEMICAL COMPOSITION

Tables 4-1 and 4-2 show wt% elemental and oxide species, respectively, for the DMR and HTF samples. Data was obtained for both ‘before ashing’ samples (indicated with ‘-B’ labels in Tables 4-1 and 4-2) and for samples that had been ashed at 525°C for ~ 8 hrs until no further mass change (indicated with ‘-A’ labels in Tables 4-1 and 4-2). Boron and fluoride were added to the SBW simulants at ~ 0.2 g/L and 0.6 g/L but were not detected in the dissolution/analysis of the DMR or HTF samples. Zirconium and cerium were also added to the SBW but were not analyzed in this characterization. Table 4-1 data indicates that no detectable mercury was present in any of the samples above the instrument detection limit of 0.004 wt%. Elemental analysis of the samples shows that Al > Si~Na are the main components, with greater than 1 wt% of Ca, Fe and K also present. Table 4-2 shows that no detectable nitrate anions were present in the DMR and HTF samples, and that some samples showed detectable nitrite levels with the HTF samples having > 1 wt% NO₂⁻. The carbon content of the HTF samples was in the range of 13 – 26 wt% and the DMR samples contained < 1 wt% carbon. All of the iron determined in the HTF samples was in the reduced ferrous (Fe²⁺) state, as indicated by the REDOX values of (Fe²⁺/Fe total) equal to one. Summation of all species on an oxide basis including the carbon content gives totals in an acceptable range of 96 – 104 wt%.

Table 4-1. Elemental Composition – Wt%

Sample ID	Al	B*	Ca		Cr	Fe	K	Mg	Mn	Na		Ni	P		Pb		S	Si		Sr	Ti	Zn	Cs	Re	Hg**
4504-DMR-B	35.35	0.1	1.83		0.04	3.43	1.05	0.16	0.14	4.11		0.02	0.07		0.01	<	0.03	5.14		0.02	0.25	0.09	6.1E-03	1.2E-03	0.004
4504-DMR-A	33.70	0.1	2.45		0.05	2.91	1.41	0.18	0.17	5.16		0.02	0.08		0.01		0.04	7.11	<	0.01	0.31	0.11	6.1E-03	1.4E-03	NA
4531-DMR-B	32.30	0.1	2.70		0.05	4.07	1.54	0.20	0.19	5.10		0.02	0.06		0.01	<	0.03	7.48		0.02	0.38	0.14	7.1E-03	1.6E-03	0.004
4531-DMR-A	28.85	0.1	3.26		0.06	3.52	2.19	0.22	0.22	6.11		0.03	0.09		0.02		0.04	9.68	<	0.01	0.43	0.16	6.3E-03	1.7E-03	NA
4637-DMR-B	22.70	0.1	3.42		0.06	2.93	2.53	0.25	0.22	8.42		0.03	0.05		0.02	<	0.03	13.70		0.02	0.65	0.19	1.0E-02	3.1E-03	0.004
4637-DMR-A	24.30	0.1	3.25		0.06	3.15	2.46	0.23	0.21	8.31		0.05	0.04		0.02	<	0.03	13.40		0.01	0.57	0.19	1.1E-02	1.6E-03	NA
4726-DMR-B	19.35	0.1	4.26		0.05	3.20	3.37	0.35	0.33	12.65		0.02	0.08	<	0.01		0.04	14.85		0.02	0.67	0.06	4.0E-03	8.0E-04	0.004
4726-DMR-A	19.25	0.1	4.78		0.05	2.36	3.37	0.40	0.35	12.90		0.02	0.08	<	0.01		0.04	15.00		0.02	0.72	0.07	2.6E-03	9.5E-04	NA
4508-HTF-B	13.35	0.1	5.17		0.06	1.34	2.99	0.29	0.24	9.86		0.03	0.08		0.03		0.68	13.65	<	0.01	0.65	0.10	9.3E-02	6.8E-03	0.004
4508-HTF-A	15.00	0.1	6.33		0.07	1.66	3.82	0.32	0.28	11.85		0.04	0.11		0.04		0.70	16.05		0.02	0.77	0.10	1.1E-01	1.5E-02	NA
4546-HTF-B	11.60	0.1	5.11		0.05	1.14	3.01	0.26	0.22	8.77		0.03	0.08		0.03		0.54	14.30		0.01	0.67	0.07	9.1E-02	6.3E-03	0.004
4546-HTF-A	14.90	0.1	6.14		0.07	1.50	3.41	0.32	0.28	10.90		0.03	0.10		0.05		0.68	18.10		0.02	0.83	0.10	1.0E-01	1.8E-02	NA
4649-HTF-B	10.30	0.1	6.36		0.04	0.61	2.41	0.27	0.20	6.99		0.02	0.06		0.04		0.54	13.50		0.02	0.61	0.06	9.3E-02	3.2E-03	0.004
4649-HTF-A	14.35	0.1	8.59		0.06	0.82	3.33	0.35	0.26	9.50		0.03	0.08		0.05		0.68	18.55		0.03	0.79	0.08	1.3E-01	8.8E-03	NA
4728-HTF-B	10.35	0.1	5.64	<	0.01	0.70	3.74	0.30	0.26	11.35	<	0.01	0.07		0.02		0.76	13.20	<	0.01	0.61	0.02	4.5E-03	1.3E-04	0.004
4728-HTF-A	12.70	0.1	7.13	<	0.01	0.80	4.70	0.34	0.31	13.75	<	0.01	0.09		0.02		0.90	16.25		0.02	0.71	0.03	6.2E-03	2.7E-04	NA

* Note Boron values are 'less than' values; boron was added to SBW simulant at ~ 0.2 g/L;

** Note Hg < 0.004 wt%

NA – Hg analysis not performed on after-ashed samples.

Table 4-2. Oxide Composition – Wt%

Sample ID	Al ₂ O ₃	B ₂ O ₃	CaO		Cr ₂ O ₃	Fe ₂ O ₃	K ₂ O	MgO	MnO ₂	Na ₂ O		NiO	P ₂ O ₅		PbO		SO ₃	SiO ₂		SrO	TiO ₂	ZnO	Cs ₂ O	ReO ₂
4504-DMR-B	66.80	0.32	2.55		0.06	4.90	1.26	0.26	0.22	5.54		0.03	0.15		0.01	<	0.08	10.98		0.02	0.41	0.11	6.5E-03	1.4E-03
4504-DMR-A	63.68	0.32	3.43		0.07	4.16	1.70	0.30	0.27	6.96		0.03	0.19		0.01		0.10	15.21	<	0.01	0.52	0.14	6.5E-03	1.7E-03
4531-DMR-B	61.03	0.32	3.78		0.07	5.81	1.86	0.33	0.30	6.87		0.03	0.14		0.01	<	0.08	15.99		0.02	0.63	0.18	7.5E-03	1.9E-03
4531-DMR-A	54.51	0.32	4.55		0.08	5.03	2.63	0.36	0.34	8.23		0.03	0.19		0.02		0.09	20.71	<	0.01	0.72	0.20	6.6E-03	2.0E-03
4637-DMR-B	42.89	0.32	4.78		0.09	4.19	3.05	0.41	0.36	11.34		0.04	0.12		0.02	<	0.08	29.31		0.03	1.08	0.24	1.1E-02	3.6E-03
4637-DMR-A	45.92	0.32	4.54		0.09	4.50	2.96	0.38	0.33	11.20		0.06	0.10		0.02	<	0.08	28.67		0.01	0.96	0.24	1.2E-02	1.8E-03
4726-DMR-B	36.56	0.32	5.95		0.07	4.57	4.06	0.59	0.52	17.05		0.03	0.18	<	0.01		0.09	31.77		0.02	1.11	0.07	4.3E-03	9.4E-04
4726-DMR-A	36.37	0.32	6.68		0.08	3.37	4.05	0.66	0.55	17.39		0.03	0.19	<	0.01		0.09	32.09		0.02	1.19	0.08	2.8E-03	1.1E-03
4508-HTF-B	25.23	0.32	7.23		0.08	1.92	3.60	0.48	0.37	13.29		0.04	0.18		0.03		1.70	29.20	<	0.01	1.09	0.13	9.8E-02	8.0E-03
4508-HTF-A	28.34	0.32	8.85		0.10	2.37	4.60	0.54	0.44	15.97		0.05	0.25		0.04		1.75	34.33		0.02	1.29	0.13	1.1E-01	1.7E-02
4546-HTF-B	21.92	0.32	7.15		0.08	1.63	3.63	0.43	0.35	11.82		0.03	0.18		0.03		1.34	30.59		0.01	1.12	0.09	9.7E-02	7.3E-03
4546-HTF-A	28.15	0.32	8.58		0.10	2.14	4.10	0.53	0.44	14.69		0.04	0.23		0.05		1.70	38.72		0.02	1.39	0.13	1.1E-01	2.1E-02
4649-HTF-B	19.46	0.32	8.90		0.06	0.87	2.90	0.44	0.32	9.42		0.02	0.14		0.04		1.34	28.88		0.03	1.02	0.07	9.9E-02	3.8E-03
4649-HTF-A	27.12	0.32	12.01		0.08	1.17	4.01	0.57	0.41	12.81		0.04	0.18		0.06		1.70	39.68		0.04	1.31	0.10	1.4E-01	1.0E-02
4728-HTF-B	19.56	0.32	7.88	<	0.01	1.00	4.50	0.49	0.42	15.30	<	0.01	0.16		0.03		1.90	28.24	<	0.01	1.02	0.03	4.8E-03	1.5E-04
4728-HTF-A	24.00	0.32	9.97	<	0.01	1.15	5.66	0.57	0.49	18.53	<	0.01	0.20		0.02		2.25	34.76		0.02	1.18	0.04	6.6E-03	3.1E-04

* Note B₂O₃ values are ‘less than’ values; boron was added to SBW simulant at ~ 0.2 g/L

Table 4-2. Oxide Composition – Wt% - continued

Sample ID		HgO*		F		NaCl		NO ₂		NO ₃	Carbon	Sum wt%	Redox Fe ²⁺ /Fe total
4504-DMR-B	<	0.005	<	0.1	<	0.16		1	<	0.1	0.98	96.33	0.41
4504-DMR-A	<	NA	<	0.1	<	0.16	<	0.1	<	0.1	0	97.79	NA
4531-DMR-B	<	0.005	<	0.1	<	0.16	<	0.1	<	0.1	0.31	98.50	0.27
4531-DMR-A	<	NA	<	0.1	<	0.16		1.16	<	0.1	0	99.83	NA
4637-DMR-B	<	0.005	<	0.1	<	0.16		0.978	<	0.1	0.8	100.76	0.44
4637-DMR-A	<	NA	<	0.1	<	0.16		1.12	<	0.1	0	102.12	NA
4726-DMR-B	<	0.005	<	0.1	<	0.16	<	0.1	<	0.1	0.4	104.12	0.29
4726-DMR-A	<	NA	<	0.1	<	0.16		1.15	<	0.1	0	104.98	NA
4508-HTF-B	<	0.005	<	0.1		0.59		1.99	<	0.1	13.15	101.21	1.00**
4508-HTF-A	<	NA	<	0.1		0.81		1.09	<	0.1	0	101.90	NA
4546-HTF-B	<	0.005	<	0.1		0.47		1.62	<	0.1	20.87	104.25	1.00**
4546-HTF-A	<	NA	<	0.1		0.90		1.07	<	0.1	0	103.93	NA
4649-HTF-B	<	0.005	<	0.1		0.45		1.91	<	0.1	26.47	103.65	1.00**
4649-HTF-A	<	NA	<	0.1		0.89		1.27	<	0.1	0	104.42	NA
4728-HTF-B	<	0.005	<	0.1		0.91		2.52	<	0.1	19.49	104.29	1.00**
4728-HTF-A	<	NA	<	0.1		1.43		1.25	<	0.1	0	102.37	NA

* Note NA – Hg analysis not performed on after-ashed samples.

** Essentially all iron in the reduced +2 state for the HTF samples.

4.2 CRYSTALLINE PHASES

In Table 4-3 the various crystalline phases observed in the coarse-sieved DMR and unsieved HTF samples are summarized. The individual XRD spectrums for each powder sample are collected in an Appendix A to this report. The DMR 4504 sample appears to have major crystalline species corundum attributed to the alumina starting bed and alumina added during processing for particle size control. The DMR 4531 sample appears to also show major corundum, some nepheline and minor phase of quartz (SiO_2). The last two DMR samples appear to be mostly nepheline and sodium aluminum silicate, with only minor phases of corundum and quartz. These XRD spectra and crystalline identification are in agreement with the progression of DMR samples shown previously in the ternary diagram, i.e., progressive ‘turnover’ of the starting alumina (corundum phase) bed in going from DMR-4504 through final DMR-4726. All HTF samples are similar with major nepheline and sodium aluminum silicate phases with minor corundum and quartz phases present. Solid mineral phases produced from using clay as co-reactant in the FBSR are sodium aluminosilicates (NAS) comprised of nepheline group crystals and sodalite group crystals including nosean [Pareizs et al., 2005]. These DMR and HTF samples appear to have mainly the nepheline and sodium aluminum silicate crystals. If the sodalite/nosean crystals are present, they are likely below the nominal ~ 1-2 vol% crystalline content of the powder-XRD method used in this work.

Table 4-3. XRD Crystalline Phases

DMR 4504	(Major) Nepheline – NaAlSiO_4	(Major) Diaoyudaoite – $\text{NaAl}_{11}\text{O}_{17}$	(Major) Corundum – Al_2O_3	-	-
DMR 4531	(Major) Nepheline – NaAlSiO_4	(Major) Diaoyudaoite – $\text{NaAl}_{11}\text{O}_{17}$	(Major) Corundum – Al_2O_3	(Minor) Quartz – SiO_2	-
DMR 4637	(Major) Nepheline – NaAlSiO_4	-	(Minor) Corundum – Al_2O_3	Quartz – SiO_2	(Major) Sodium Aluminum Silicate – $\text{Na}_{1.75}\text{Al}_{1.75}\text{Si}_{0.25}\text{O}_4$
DMR 4726	(Major) Nepheline – NaAlSiO_4	-	(Minor) Corundum – Al_2O_3	(Minor) Quartz – SiO_2	(Major) Sodium Aluminum Silicate – $\text{Na}_{1.75}\text{Al}_{1.75}\text{Si}_{0.25}\text{O}_4$
HTF 4508	(Major) Nepheline – NaAlSiO_4	-	(Minor) Corundum – Al_2O_3	(Minor) Quartz – SiO_2	(Major) Sodium Aluminum Silicate – $\text{Na}_{1.75}\text{Al}_{1.75}\text{Si}_{0.25}\text{O}_4$
HTF 4546	(Major) Nepheline – NaAlSiO_4	-	(Minor) Corundum – Al_2O_3	(Minor) Quartz – SiO_2	(Major) Sodium Aluminum Silicate – $\text{Na}_{1.75}\text{Al}_{1.75}\text{Si}_{0.25}\text{O}_4$
HTF 4649	(Major) Nepheline – NaAlSiO_4	-	(Minor) Corundum – Al_2O_3	(Minor) Quartz – SiO_2	(Major) Sodium Aluminum Silicate – $\text{Na}_{1.75}\text{Al}_{1.75}\text{Si}_{0.25}\text{O}_4$
HTF 4728	(Major) Nepheline – NaAlSiO_4	-	(Minor) Corundum – Al_2O_3	(Minor) Quartz – SiO_2	(Major) Sodium Aluminum Silicate – $\text{Na}_{1.75}\text{Al}_{1.75}\text{Si}_{0.25}\text{O}_4$

4.3 DURABILITY TESTING

Table 4-4 shows the results of surface area to volume ratios calculated from either measured BET surface area or from particle size measurements and density. The HTF samples that were sieved through 200 mesh sieves all showed average particle diameters in the range of 7 – 13 microns. Similar measurements on the DMR samples sieved through 100-200 mesh sieves measured average particle diameters similar to those of glass put through similar sieves, in the range of 106 – 115 microns. The BET surface areas are all greater than corresponding geometric surface areas. The ratio of the BET surface area to geometric surface area is calculated as the ‘surface roughness’ shown in the last column of Table 4-4. The BET surface areas for the HTF samples are 15 to 36 times larger than the geometric surface areas. The BET surface areas for the DMR samples are 135 to 246 times larger than the geometric surface areas.

Table 4-4. BET and Geometric Surface Area Data

	Sample Mass (g)	Leachant Volume (mL)	BET surface area (m ² /g)	SA/V (m ²)		Particle Diameter microns	Particle Density g/mL	Geometric surface area (m ² /g)	SA/V (m ²)	Surface Roughness SA _{BET} /SA _{geo}
HTF4508-1 P76	1.508	15.03	4.526	454116		7.182	2.794	0.2990	29997	15
HTF4508-2 P81	1.498	15.001	4.526	451976		7.182	2.794	0.2990	29855	15
HTF4508-3 P83	1.504	15.072	4.526	451649		7.182	2.794	0.2990	29834	15
Avg				452580					29895	
Stdev				1340					88	
HTF4546-1 P86	1.5	15.018	5.625	561836		13.87	2.748	0.1574	15723	36
HTF4546-2 P92	1.5	14.995	5.625	562698		13.87	2.748	0.1574	15747	36
HTF4546-3 P93	1.498	15.05	5.625	559894		13.87	2.748	0.1574	15669	36
Avg				561476					15713	
Stdev				1436					40	
HTF4649-1 P97	1.507	15.091	6.413	640378		11.74	2.599	0.1966	19635	33
HTF4649-2 P98	1.504	15.004	6.413	642809		11.74	2.599	0.1966	19710	33
HTF4649-3 P99	1.499	15.008	6.413	640501		11.74	2.599	0.1966	19639	33
Avg				641229					19661	
Stdev				1369					42	
HTF4728-1 P100	1.501	15.084	5.1939	516842		12.2	2.646	0.1858	18493	28
HTF4728-2 P100	1.604	15.038	5.1939	553998		12.2	2.646	0.1858	19822	28
HTF4728-3 P100	1.501	15.003	5.1939	519632		12.2	2.646	0.1858	18593	28
Avg				530157					18969	
Stdev				20693					740	
DMR4726-1 P103	1.5	15.029	2.82	281456		104.9	2.736	0.0209	2087	135
DMR4726-2 P104	1.503	15.026	2.82	282075		104.9	2.736	0.0209	2091	135
DMR4726-3 P110	1.505	15.013	2.82	282695		104.9	2.736	0.0209	2096	135
Avg				282075					2091	
Stdev				620					5	
DMR4637-1 P112	1.504	15.06	4.279	427362		115.3	2.986	0.0174	1740	246
DMR4637-2 P113	1.503	15.12	4.279	425383		115.3	2.986	0.0174	1732	246
DMR4637-3 P114	1.502	15.042	4.279	427304		115.3	2.986	0.0174	1740	246
Avg				426683					1737	
Stdev				1126					5	
DMR4531-1 P121	1.503	15.032	3.492	349184		112.4	3.155	0.0169	1692	206
DMR4531-2 P122	1.5	15.044	3.492	348209		112.4	3.155	0.0169	1687	206
DMR4531-3 P170	1.5	15.046	3.492	348162		112.4	3.155	0.0169	1687	206
Avg				348518					1688	
Stdev				577					3	
DMR4504-1 P171	1.501	15.036	2.515	251095		106.5	3.204	0.0176	1755	143
DMR4504-2 P172	1.503	15.029	2.515	251547		106.5	3.204	0.0176	1759	143
DMR4504-3 P178	1.498	15.012	2.515	250994		106.5	3.204	0.0176	1755	143
Avg				251212					1756	
Stdev				294					2	

All DMR and HTF samples were durability tested in triplicate per the PCT procedure. Glass standards were used to ensure test control as required by the PCT procedure. The standard glasses agreed with previous round robin testing of the glasses for the ARM-1 glass [Jantzen et al., 1995], the EA glass [Jantzen et al., 1993], and the LRM glass [Ebert and Wolf, 2000]. Table 4-5 shows pH and concentration results for the standard glasses and Table 4-6 shows the results on a normalized concentration basis.

Table 4-5. PCT Leachate Standards and Blanks (ppm)

Sample ID	pH		Al		B		Li		Na		Si
Blank 1	7.05	<	0.11	<	0.11	<	1.10	<	0.11		0.16
Blank 2	6.28	<	0.11	<	0.11	<	1.10	<	0.11		0.18
ARM 1 -P64	10.38		4.40		18.9		15.9		42.3		68.4
ARM 2 - P65	10.35		4.20		18.9		16.0		42.2		69.9
ARM 3 - P67	10.36		4.15		17.1		15.3		42.4		66.5
Average	10.36		4.25						42.31		68.27
St.Dev.	0.02		0.13						0.07		1.72
Avg.(St.dev.) *	10.17(0.29)		4.85(0.5)						36.22(2.45)		61.23(4.07)
*See Jantzen et al., 1995											
EA-1 P68	11.71	<	2.0		492.0		161.6		1384.9		793.7
EA-2 P69	11.77	<	2.0		493.6		164.3		1428.5		794.5
EA-3 P70	11.75	<	2.0		493.4		159.2		1364.8		787.9
Average	11.74				493.0		161.7		1392.7		792.0
St.Dev.	0.03				0.9		2.6		32.6		3.6
Avg.(St.dev.) **	11.85(0.1)				587(43)		190(14.5)		1662(112)		893(86)
**See Jantzen et al., 1993											
LRM-1 P71	10.96		14.1		25.7	<	2.8		161.2		83.7
LRM-2 P72	10.93		13.2		23.4	<	2.8		158.7		76.2
LRM-3 P74	11.05		14.7		22.7	<	2.8		152.8		106.1
Average	10.98		13.98		23.93				157.58		88.65
St.Dev.	0.06		0.74		1.57				4.34		15.60
Avg.(St.dev.) ***	10.92(0.092)		14.3(2.61)		26.7(1.83)				160(11.5)		82(3.53)
***See Ebert and Wolf, 2000											

Table 4-6. PCT Leachate Standards and Blanks (g/L)

Sample ID	Al	B	Li	Na	Si
ARM 1 -P64	0.149	NA	NA	0.590	0.315
ARM 2 - P65	0.142	NA	NA	0.589	0.322
ARM 3 - P67	0.141	NA	NA	0.591	0.306
Average	0.144	NA	NA	0.590	0.314
St.Dev.	0.004	NA	NA	0.001	0.008
Avg.(St.dev.)*	0.155(0.0172)	NA	NA	0.505(0.0539)	0.282(0.03)
*See Jantzen et al., 1995					
EA-1 P68	NA	14.10	8.21	11.08	3.48
EA-2 P69	NA	14.14	8.35	11.43	3.49
EA-3 P70	NA	14.14	8.09	10.92	3.46
Average	NA	14.13	8.22	11.14	3.48
St.Dev.	NA	0.02	0.13	0.26	0.02
Avg.(St.dev.)**	NA	16.695(1.222)	9.565(0.735)	13.346(0.902)	3.922(0.376)
**See Jantzen et al., 1993					
LRM-1 P71	0.28	1.05	NA	1.08	0.330
LRM-2 P72	0.26	0.96	NA	1.07	0.301
LRM-3 P74	0.29	0.93	NA	1.03	0.419
Average	0.28	0.98	NA	1.06	0.35
St.Dev.	0.01	0.06	NA	0.03	0.06
Avg. ***	0.28	1.10	NA	1.08	0.32
St.Dev. ***	0.05	0.08	NA	0.08	0.01
***See Ebert and Wolf, 2000					

NA = not applicable; the measured normalized concentrations values not shown – reference values are not available for comparison

Triplicate PCT leachate concentration and pH data for the DMR and HTF samples are shown in Table 4-7. All pH values for the DMR and HTF sample leachates were in the range of 12 to 12.7. This pH range is slightly higher than the reported average from the EA glass of ~ 11.8. The leachate concentration data shown in Table 4-7 were normalized using the elemental compositions shown previously in Table 4-1 and the BET surface area shown in Table 4-4, via Equations 1, 2 and 4. The normalized release data (NL_i) are shown in Table 4-8. All of the normalized releases for the DMR and HTF samples were less than 1 g/m². Because the elemental concentration for S in sample DMR-4637 was below detection, the $NL(S)$ values in Table 4-8 are shown as ‘>’ values.

Table 4-7. PCT Leachate Concentrations (ppm)

Sample ID	pH	Al	K	Na	S	Si	Cs	Re
HTF4508-1 P76	12.81	1971.3	574.7	5479.5	958.9	15.4	10.5	30.2
HTF4508-2 P81	12.65	2089.7	573.0	5426.6	1011.2	16.2	11.0	29.6
HTF4508-3 P83	12.79	1916.5	574.9	5413.2	961.6	15.9	10.7	29.6
Avg.	12.75	1992.5	574.2	5439.8	977.2	15.8	10.7	29.8
St. Dev.	0.09	88.6	1.1	35.0	29.4	0.4	0.3	0.4
HTF4546-1 P86	12.37	2232.0	480.1	4087.7	798.1	22.0	19.8	25.5
HTF4546-2 P92	12.38	2173.6	478.4	3916.4	797.3	19.3	20.7	25.5
HTF4546-3 P93	12.39	2090.9	456.0	3781.4	831.4	18.9	21.4	25.4
Avg.	12.38	2165.5	471.5	3928.5	808.9	20.1	20.6	25.5
St. Dev.	0.01	70.9	13.5	153.5	19.4	1.7	0.8	0.0
HTF4649-1 P97	12.12	945.2	266.6	2224.1	581.0	7.6	19.8	22.0
HTF4649-2 P98	12.14	926.4	264.9	2210.6	565.8	7.9	20.0	22.1
HTF4649-3 P99	12.15	929.7	258.4	2192.3	546.7	5.9	20.3	22.3
Avg.	12.14	933.8	263.3	2209.0	564.5	7.1	20.0	22.1
St. Dev.	0.02	10.1	4.4	15.9	17.2	1.1	0.3	0.2
HTF4728-1 P100	12.50	1586.0	895.6	5850.5	1178.4	13.8	0.4	1.3
HTF4728-2 P100	12.88	1624.0	870.4	5784.0	1190.2	13.3	1.6	1.4
HTF4728-3 P100	12.61	1579.5	829.8	5781.3	1286.3	12.7	0.5	1.4
Avg.	12.66	1596.5	865.3	5805.2	1218.3	13.2	0.8	1.4
St. Dev.	0.20	24.0	33.2	39.2	59.2	0.6	0.7	0.0
DMR4726-1 P103	12.40	2611.9	79.7	3417.8	25.3	62.5	0.1	0.2
DMR4726-2 P104	12.58	2546.5	79.2	3376.9	29.1	63.4	0.1	0.2
DMR4726-3 P110	12.57	2595.1	78.2	3449.1	27.5	62.3	0.1	0.2
Avg.	12.52	2584.5	79.0	3414.6	27.3	62.7	0.1	0.2
St. Dev.	0.10	34.0	0.8	36.2	1.9	0.6	0.0	0.0
DMR4637-1 P112	12.01	649.4	20.3	930.9	9.8	43.5	1.4	0.8
DMR4637-2 P113	12.08	651.9	19.7	927.8	9.6	43.5	1.3	0.8
DMR4637-3 P114	12.09	650.2	20.1	971.1	10.7	44.9	1.3	0.8
Avg.	12.06	650.5	20.0	943.2	10.0	44.0	1.3	0.8
St. Dev.	0.04	1.3	0.3	24.2	0.6	0.8	0.0	0.0
DMR4531-1 P121	12.08	908.5	31.8	1237.3	26.8	46.0	0.8	0.7
DMR4531-2 P122	12.06	914.8	33.2	1202.1	27.1	45.2	0.8	0.7
DMR4531-3 P170	12.15	920.3	32.5	1231.7	25.0	44.5	0.8	0.7
Avg.	12.10	914.5	32.5	1223.7	26.3	45.2	0.8	0.7
St. Dev.	0.05	5.9	0.7	19.0	1.1	0.7	0.0	0.0
DMR4504-1 P171	12.27	1292.7	45.2	1820.4	38.0	56.4	1.0	0.5
DMR4504-2 P172	12.32	1314.5	44.0	1913.2	34.5	52.6	0.9	0.6
DMR4504-3 P178	12.25	1311.3	44.0	1823.6	33.7	52.1	0.9	0.5
Avg.	12.28	1306.2	44.4	1852.4	35.4	53.7	1.0	0.5
St. Dev.	0.04	11.8	0.7	52.7	2.3	2.4	0.1	0.0

Table 4-8. Normalized PCT Leachate Release (g/m²) Using BET Surface Area

Sample ID	Al	K	Na	S	Si	Cs	Re
HTF4508-1 P76	2.89E-02	3.31E-02	1.02E-01	3.02E-01	2.11E-04	2.14E-02	4.59E-01
HTF4508-2 P81	3.08E-02	3.32E-02	1.01E-01	3.20E-01	2.23E-04	2.25E-02	4.52E-01
HTF4508-3 P83	2.83E-02	3.33E-02	1.01E-01	3.05E-01	2.20E-04	2.20E-02	4.54E-01
Avg.	2.94E-02	3.32E-02	1.01E-01	3.09E-01	2.18E-04	2.20E-02	4.55E-01
St.Dev	1.32E-03	1.01E-04	3.54E-04	9.73E-03	6.50E-06	5.66E-04	3.71E-03
HTF4546-1 P86	2.67E-02	2.51E-02	6.67E-02	2.08E-01	2.16E-04	3.38E-02	2.48E-01
HTF4546-2 P92	2.59E-02	2.50E-02	6.39E-02	2.08E-01	1.90E-04	3.55E-02	2.48E-01
HTF4546-3 P93	2.51E-02	2.39E-02	6.20E-02	2.18E-01	1.87E-04	3.68E-02	2.48E-01
Avg.	2.59E-02	2.47E-02	6.42E-02	2.11E-01	1.97E-04	3.53E-02	2.48E-01
St.Dev	8.00E-04	6.47E-04	2.41E-03	5.60E-03	1.61E-05	1.48E-03	2.84E-04
HTF4649-1 P97	1.03E-02	1.25E-02	3.66E-02	1.33E-01	6.39E-05	2.38E-02	3.92E-01
HTF4649-2 P98	1.00E-02	1.24E-02	3.62E-02	1.29E-01	6.65E-05	2.39E-02	3.93E-01
HTF4649-3 P99	1.01E-02	1.21E-02	3.60E-02	1.25E-01	4.93E-05	2.44E-02	3.97E-01
Avg.	1.01E-02	1.23E-02	3.63E-02	1.29E-01	5.99E-05	2.40E-02	3.94E-01
St.Dev	1.25E-04	1.99E-04	2.70E-04	3.94E-03	9.29E-06	3.15E-04	2.89E-03
HTF4728-1 P100	2.42E-02	3.69E-02	8.23E-02	2.53E-01	1.64E-04	1.25E-02	9.61E-01
HTF4728-2 P100	2.31E-02	3.34E-02	7.59E-02	2.38E-01	1.48E-04	4.62E-02	9.19E-01
HTF4728-3 P100	2.39E-02	3.40E-02	8.09E-02	2.75E-01	1.50E-04	1.50E-02	9.94E-01
Avg.	2.37E-02	3.48E-02	7.97E-02	2.55E-01	1.54E-04	2.46E-02	9.58E-01
St.Dev	5.70E-04	1.85E-03	3.36E-03	1.83E-02	8.88E-06	1.87E-02	3.73E-02
DMR4726-1 P103	4.82E-02	8.42E-03	9.41E-02	2.47E-01	1.48E-03	1.83E-02	6.80E-02
DMR4726-2 P104	4.69E-02	8.34E-03	9.28E-02	2.83E-01	1.50E-03	1.93E-02	6.58E-02
DMR4726-3 P110	4.77E-02	8.22E-03	9.46E-02	2.68E-01	1.47E-03	1.45E-02	6.87E-02
Avg.	4.76E-02	8.33E-03	9.38E-02	2.66E-01	1.48E-03	1.74E-02	6.75E-02
St.Dev	6.60E-04	1.03E-04	9.24E-04	1.81E-02	1.45E-05	2.52E-03	1.50E-03
DMR4637-1 P112	6.25E-03	1.93E-03	2.62E-02	> 6.88E-02	7.59E-04	2.90E-02	1.27E-01
DMR4637-2 P113	6.31E-03	1.88E-03	2.62E-02	> 6.73E-02	7.62E-04	2.90E-02	1.26E-01
DMR4637-3 P114	6.26E-03	1.92E-03	2.73E-02	> 7.51E-02	7.85E-04	2.90E-02	1.27E-01
Avg.	6.27E-03	1.91E-03	2.66E-02	> 7.04E-02	7.69E-04	2.90E-02	1.27E-01
St.Dev	2.89E-05	2.35E-05	6.47E-04	4.16E-03	1.38E-05	3.88E-05	6.60E-04
DMR4531-1 P121	9.02E-03	4.16E-03	5.80E-02	2.11E-01	1.36E-03	3.66E-02	1.16E-01
DMR4531-2 P122	9.11E-03	4.36E-03	5.65E-02	2.14E-01	1.34E-03	3.82E-02	1.15E-01
DMR4531-3 P170	9.16E-03	4.28E-03	5.79E-02	1.97E-01	1.32E-03	3.68E-02	1.18E-01
Avg.	9.10E-03	4.27E-03	5.75E-02	2.07E-01	1.34E-03	3.72E-02	1.16E-01
St.Dev	7.27E-05	9.94E-05	8.38E-04	8.93E-03	2.02E-05	8.50E-04	1.90E-03
DMR4504-1 P171	1.53E-02	1.28E-02	1.40E-01	3.94E-01	3.16E-03	6.60E-02	1.47E-01
DMR4504-2 P172	1.55E-02	1.24E-02	1.47E-01	3.58E-01	2.94E-03	6.06E-02	1.53E-01
DMR4504-3 P178	1.55E-02	1.24E-02	1.41E-01	3.50E-01	2.92E-03	5.98E-02	1.50E-01
Avg.	1.54E-02	1.25E-02	1.43E-01	3.67E-01	3.01E-03	6.21E-02	1.50E-01
St.Dev	1.32E-04	2.07E-04	3.90E-03	2.36E-02	1.33E-04	3.38E-03	3.34E-03

4.3.1 Comparison of Normalized Release to High-Level Waste

Normalized sodium and silicon release values for the DMR and HTF samples are shown in comparison to those of the High Level Waste (HLW) reference EA glass in Figure 4-1 and Figure 4-2. The EA glass normalized release values are calculated from the data shown previously in Table 4-6 and using Equations 1, 2 and 3. Normalized release EA data are shown as the average and as the average minus two standard deviations. Comparison of a given HLW waste form normalized release vs. the EA values minus two standard deviations is one method suggested by the Waste Acceptance Product Specifications (WAPS) for showing adequate waste form durability [WAPS 2003]. Figure 4-1 and Figure 4-2 clearly show that the normalized releases for Na and Si from the FBSR products are orders of magnitude lower than the corresponding average EA normalized release values.

4.3.2 Comparison of Normalized Release to Low Activity Waste

Normalized releases for Na and Si and from other elements in the DMR and HTF samples are also compared to the normalized release rate from the Low Activity Waste (LAW) target LRM glass (plotted on both a BET surface area and a geometric surface area basis) in Figure 4-3 and Figure 4-4. The Hanford River Protection Project (RPP) contract specification [WTP Contract 2007] of 2 g/m^2 is also plotted in the figure. Figure 4-3 and Figure 4-4 show that the normalized release for the major FBSR product elements (Al, Na, and Si), as well as the minor elements (K, S, Cs and Re) are all less than half of the 2 g/m^2 limit. Careful comparison of the various normalized release elements (Al, Na, Si, K, S, Cs and Re) for the DMR and HTF samples indicates that NL(S) and NL(Re) appear to be highest. The NL(Re) values for the HTF samples shown in Figure 4-4 also appear higher than the corresponding NL(Re) values for the DMR samples. The oxide forms of both S and Re are known to be REDOX-dependent. For instance sulfur can exist in an oxidized state as Na_2SO_4 or in a reduced state of Na_2S . Similarly, Re can be in the +7 state as Re_2O_7 or NaReO_4 or in a more reduced state of Re_2O . A possible explanation for the higher NL(Re) values for the HTF samples (very reduced REDOX values from Table 4-2) vs. the NL(Re) values for the DMR samples (more oxidized REDOX values from Table 4-2) is that the more oxidized form of NaReO_4 can be incorporated into the cage-like structure of the product minerals but ReO_2 cannot. The durability of these S/Re oxide forms and their incorporation into various mineral phases has been previously considered and discussed by Lorier et al., 2005, in durability testing that used single-pass flowthrough (SPFT) testing.

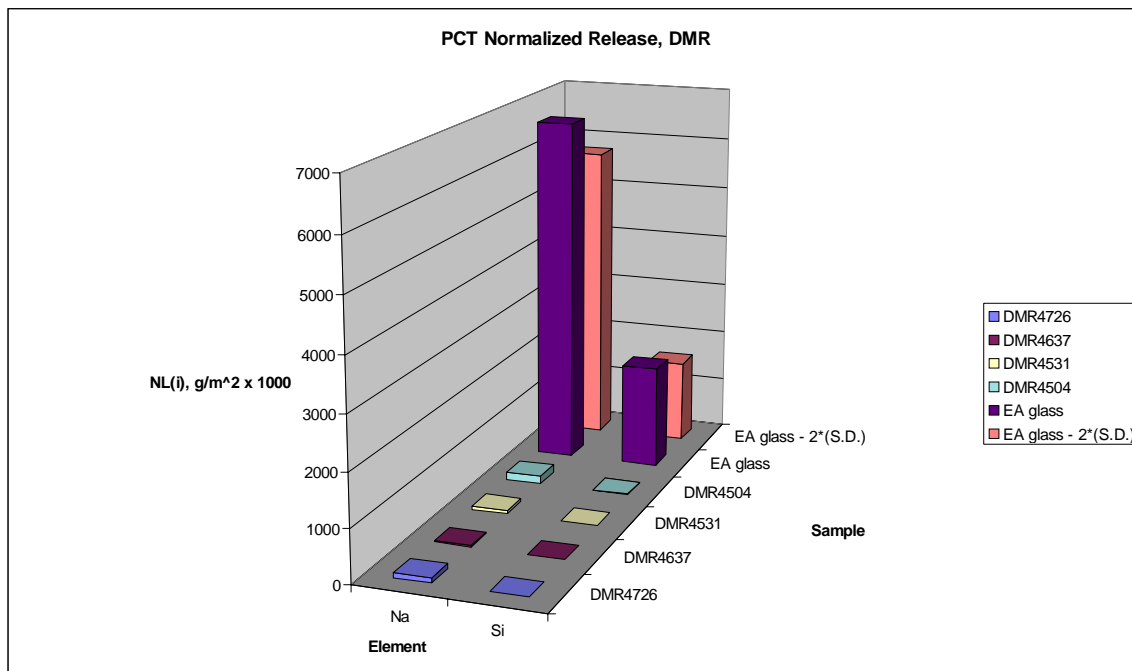


Figure 4-1. Normalized Release for DMR vs. EA Glass

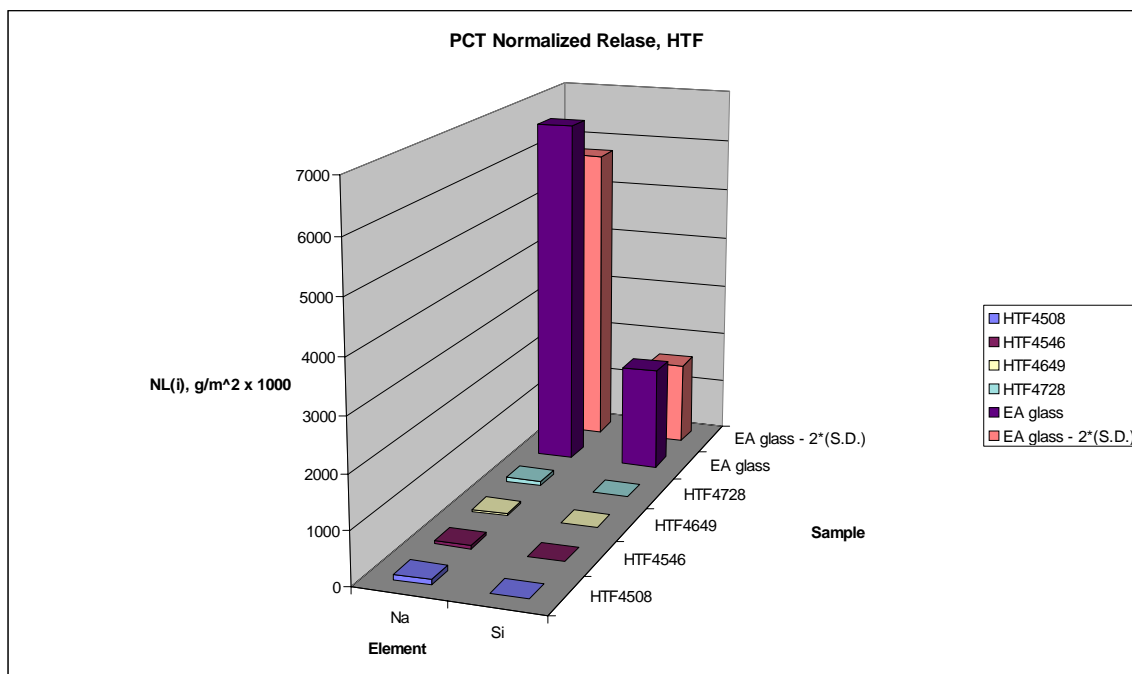


Figure 4-2. Normalized Release for HTF vs. EA Glass

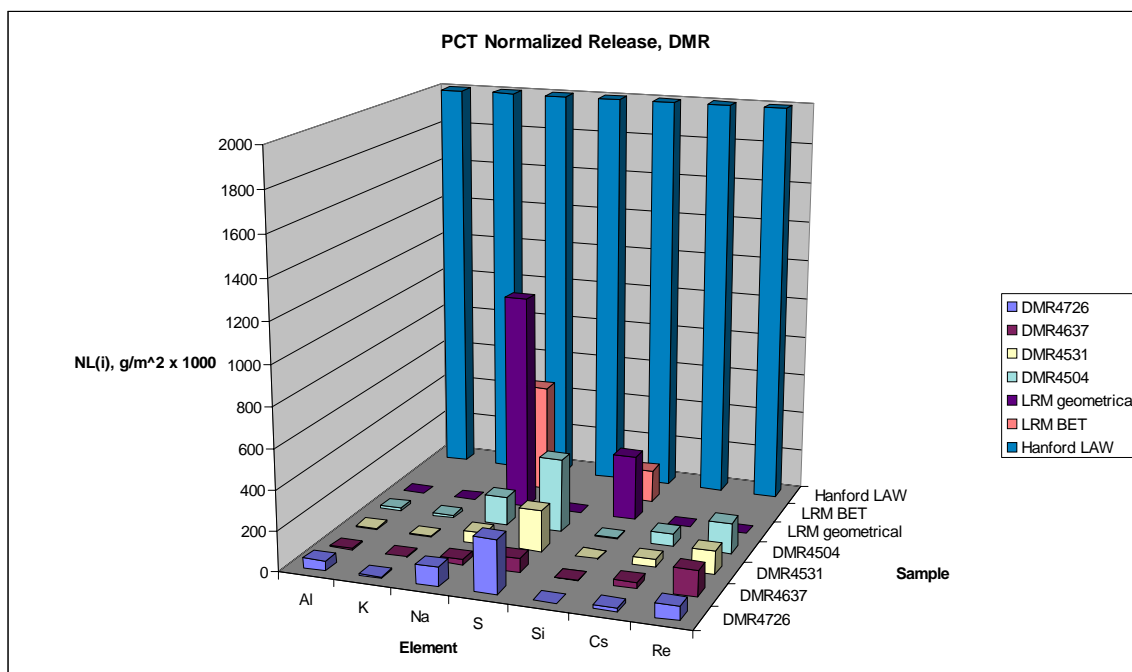


Figure 4-3. Normalized Release for DMR vs. LRM Glass and Hanford Contract LAW

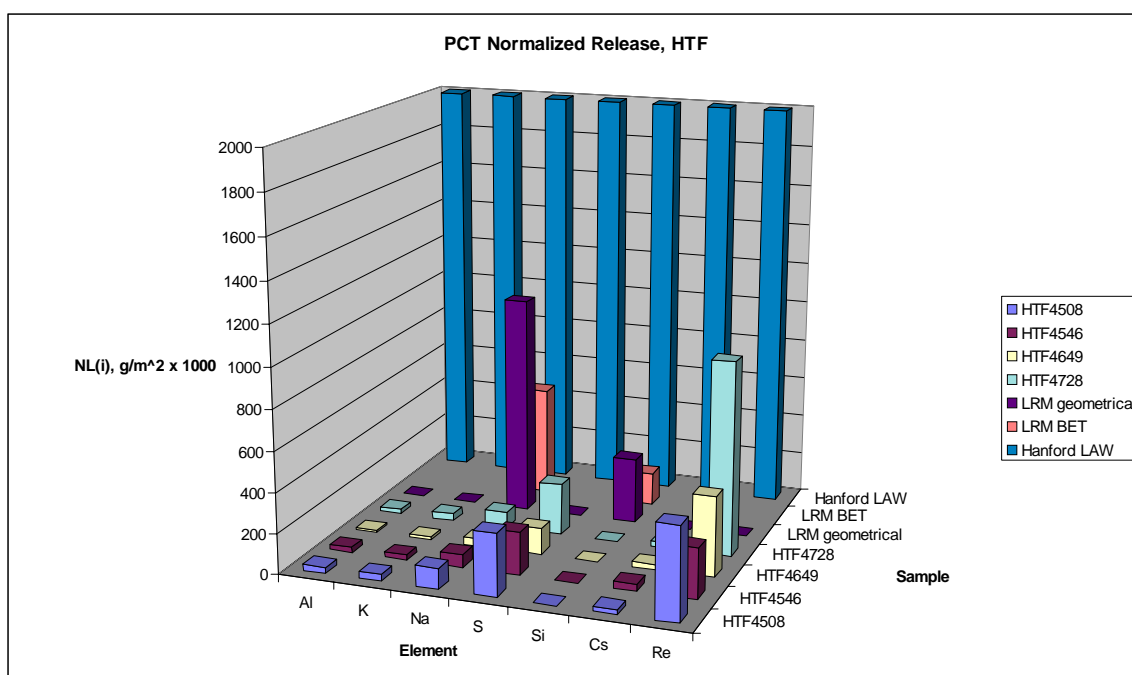


Figure 4-4. Normalized Release for HTF vs. LRM Glass and Hanford Contract LAW

4.3.3 Comparison of 2007 FBSR Product Durability Testing to Previous Work

Previous durability testing of FBSR products compared data sets from various FBSR campaign SBW simulants and LAW simulants (see Table 4-1 of Pareizs et al., 2005). It is therefore instructive to also compare our recent 2007 PCT results from the Hazen 2006 SBW to previous testing. Figures 4-5 and 4-6 show the recent DMR and HTF PCT results for NL(Na) vs. NL(Al) and NL(Cs) vs. NL(Al) overlaid onto the previous comparison plots from Pareizs et al., 2005). The solid line in these figures represents the best fit of the Pareizs et al. data and the dotted lines indicate the 95% confidence interval. Figure 4-5 indicates that all current DMR and HTF values (except for the early processing DMR 4504 and DMR 4531 points) fall within the previous fit of 0.76 slope. These 2 DMR samples not fitting the correlation contained the highest fraction of the startup bed and the beta-alumina phase ($\text{NaAl}_{11}\text{O}_{17}$) shown previously in Table 4-3. Similar conclusions are drawn from the NL(Cs) vs. the NL(Al). Current DMR and HTF values are in good agreement with the correlation that shows a 1.09 slope, except for the single DMR 4726 sample.

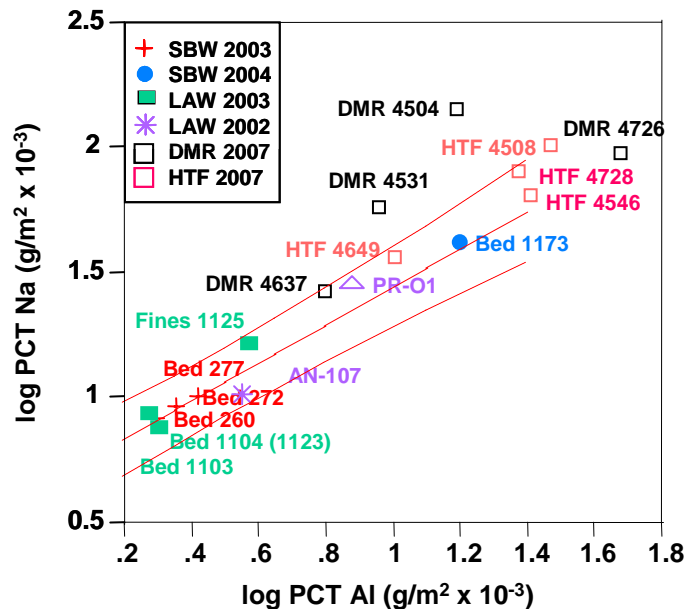


Figure 4-5. Linearity of Alkali (NL(Na)) and Alumina Released to Solution.

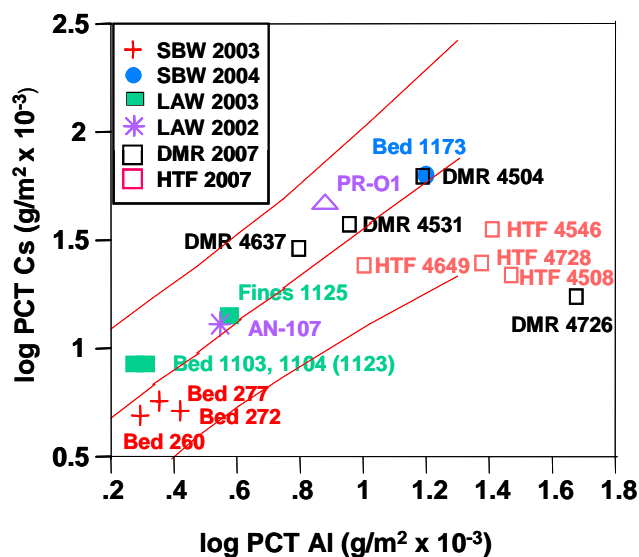


Figure 4-6. Linearity of Alkali (NL(Cs)) and Alumina Released to Solution.

Previous PCT testing indicated that Re, S and Si released to the PCT leachate is a strong function of the leachate final pH as controlled by the aluminosilicate reaction mechanisms discussed in Section 5.3.1 of Pareizs et al., 2005. The plots of Figure 5-11 from Pareizs et al., 2005 have been reproduced here with the recent 2007 PCT data as shown in Figure 4-7. The PCT response for Re vs. pH for the 2007 samples shows that the HTF samples do not correlate well with previous testing and that the DMR samples are only slightly better. Similar comparisons for 2007 PCT data for S vs. pH indicate excellent correlation with previous testing. The Si values from current 2007 testing show good correlation for the DMR samples, but the HTF samples are outside the 95% confidence interval of the previous correlation.

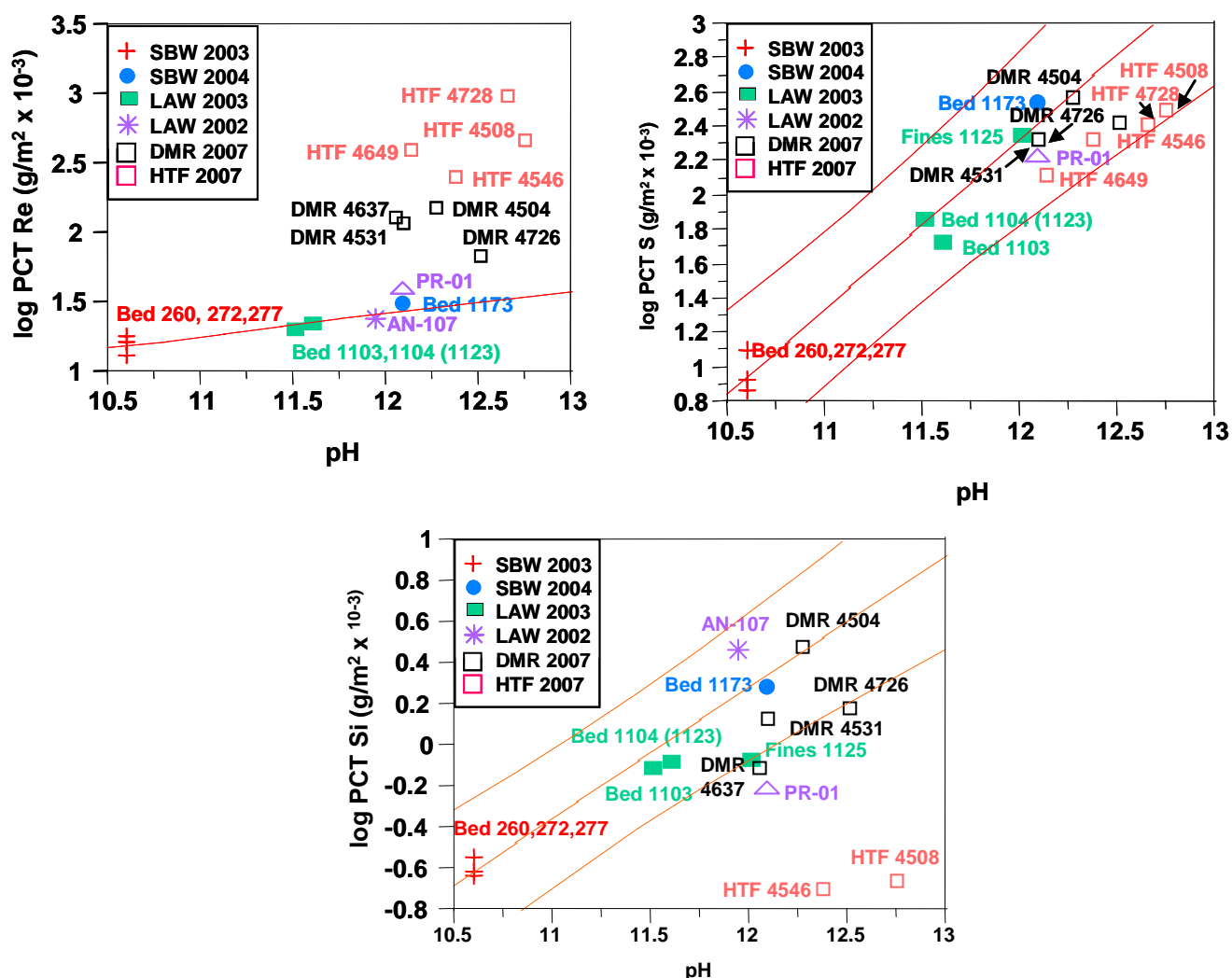


Figure 4-7. Release of Re, S, and Si to the PCT Leachates as a Function of the pH of the Final Leachate.

Previous correlations of Re, S and Si that showed strong correlation to pH also showed correlation to each other as in Figure 5-12 of Pareizs et al, 2005. Those plots are presented below in Figure 4-8 for comparison. In all cases, the current 2007 NL(i) values for all HTF samples (Re vs. S, Re vs. Si, and S vs. Si) correlate higher than previous comparisons. The current 2007 NL(i) values for the DMR samples are all closer to the previous correlations, with the S vs. Si DMR correlation falling within the 95% confidence interval of previous testing.

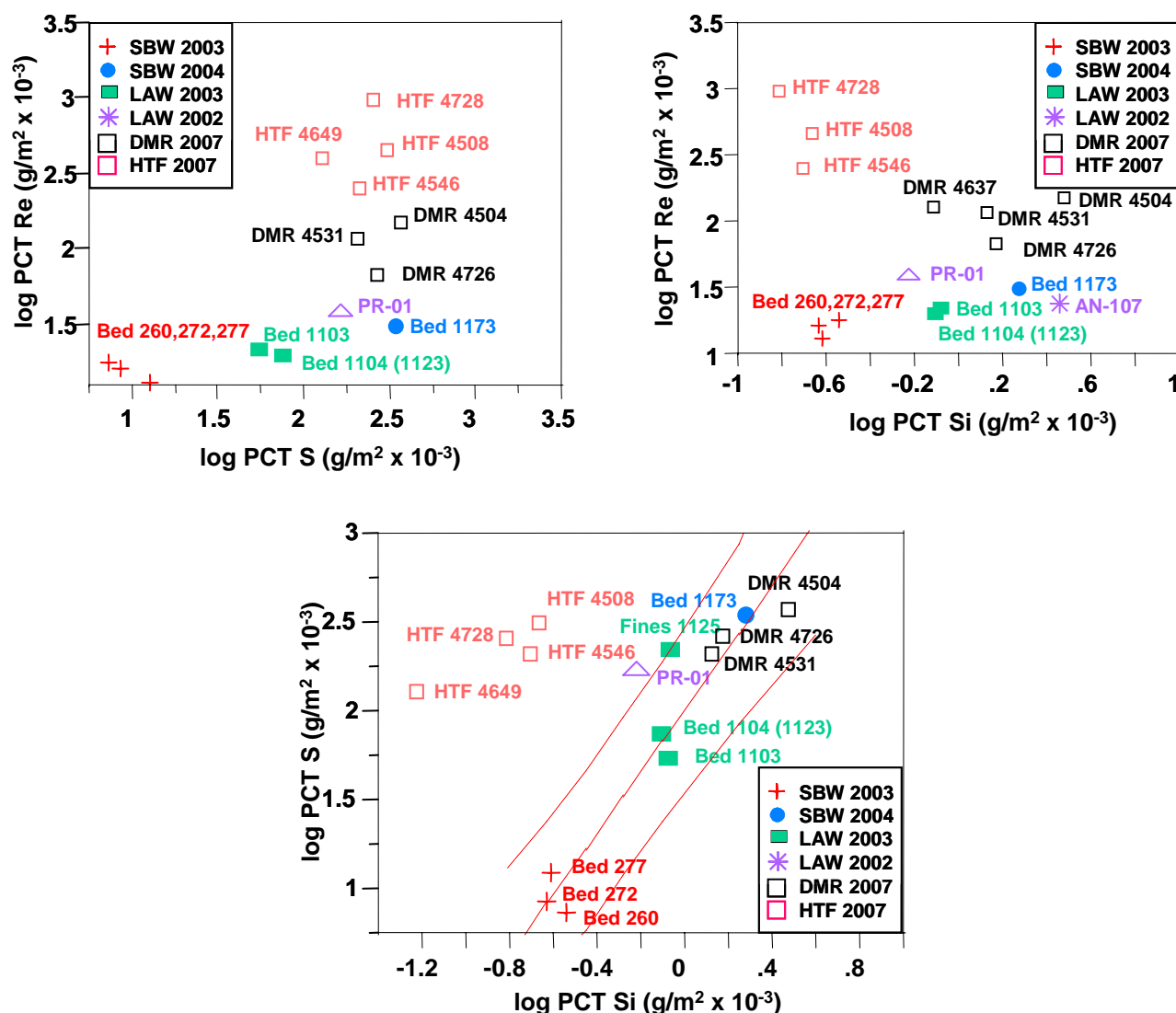


Figure 4-8. Relation of Re to S, Re to Si, and S to Si in the PCT Leachates of the FBSR Products Tested

4.4 TOXICITY CHARACTERISTIC LEACHING PROCEDURE

TCLP results for the DMR and HTF samples are presented in Table 4-9. The samples submitted for TCLP were as-received and were not sized or sieved or ashed to remove carbon. The samples met the criteria for the EPA RCRA Universal Treatment Standards for all of the constituents in the simulants. The TCLP analyses were performed for all eight RCRA metals and the additional UTS metals Ni and Zn. However, only Cr, Pb and Hg RCRA metals were present in the SBW simulants. A complete Analytical Report for the TCLP tests and analyses is presented in Appendix B to this report.

Table 4-9. Toxicity Characteristic Leaching Procedure (TCLP) Results

Element	DMR4504	DMR4531	DMR4637	DMR4726	HTF4508	HTF4546	HTF4649	HTF4728	Reporting Limit	Method Detection Limit	Characteristic RCRA Limits *	UTS **
	(mg/L)	(mg/L)	(mg/L)	(mg/L)	(mg/L)	(mg/L)	(mg/L)	(mg/L)	(mg/L)	(mg/L)	(mg/L)	(mg/L)
As	0 ^U	0 ^U	0.011 ^J	0.014 ^J	0 ^U	0 ^U	0 ^U	0 ^U	1	0.0086	5	5
Ba	0.23 ^J	0.2 ^J	0.29 ^J	0.27 ^J	0.29 ^J	0.19 ^J	0.34 ^J	0.26 ^J	1	0.0016	100	21
Cd	0 ^U	0 ^U	0 ^U	0 ^U	0 ^U	0 ^U	0 ^U	0 ^U	1	0.0022	1	0.11
Cr	0.19 ^J	0.15 ^J	0.44 ^J	0.055 ^J	0.11 ^J	0.016 ^J	0.012 ^J	0 ^U	1	0.004	5	0.6
Pb	0.12 ^J	0.074 ^J	0.2 ^J	0.024 ^J	0.02 ^J	0.011 ^J	0.013 ^J	0.016 ^J	1	0.006	5	0.75
Se	0.13 ^{JB}	0.13 ^{JB}	0.12 ^{JB}	0.1 ^{JB}	0.13 ^{JB}	0.12 ^{JB}	0.11 ^{JB}	0.14 ^{JB}	1	0.023	1	5.7
Ag	0 ^U	0 ^U	0 ^U	0 ^U	0 ^U	0 ^U	0 ^U	0 ^U	1	0.0038	5	0.14
Hg	0 ^U	0 ^U	0 ^U	0 ^U	0 ^U	0 ^U	0 ^U	0 ^U	0.02	0.00086	0.2	0.025
Ni	0.37 ^J	0.34 ^J	0.37 ^J	0.046 ^J	0.0044 ^J	0.01 ^J	0.005 ^J	0 ^U	20	0.0016	-	11
Zn	0.4 ^J	0.57 ^J	0.76 ^J	0.1 ^{JB}	0.018 ^{JB}	0.017 ^{JB}	0.019 ^{JB}	0 ^U	40	0.012	-	4.3

*40 CFR 261.24 – “Toxicity Characteristic”

**40 CFR 268.48 – “Universal Treatment Standards (UTS)”

U = results less than MDL, or “U” flag

J = results below Reporting Limit but above the MDL, or “J” flag

JB = results below Reporting Limit but above the MDL, and detectable amounts in blank (Se = 0.078 mg/L and Zn = 0.024 mg/L), or “JB” flag

5.0 QUALITY ASSURANCE

This work was completed in accordance with WSRC and SRNL quality assurance requirements. The waste form characterization and testing was conducted in accordance with DOE/RW-0214, DOE/RW-0333P, and ASME NQA-1 based quality assurance programs. The data is recorded in the following notebook: WSRC-NB-2007-00096.

6.0 CONCLUSIONS

The FBSR process was performed on INTEC simulated SBW in a recent pilot-scale demonstration that produced representative DMR and HTF samples. These powdered samples were analyzed and leach tested in this task. The following conclusions can be drawn from the testing.

- Elemental compositional analysis of the DMR and HTF samples indicates that they contain primarily Al, Si and Na, with > 1wt% of Ca, Fe and K also present. No detectable mercury was observed. The DMR samples all contained less than 1% carbon. The HTF samples containing carbon in the range of 13-26 wt% had detectable nitrite anion levels and their REDOX values show that they were completely reduced with $\text{Fe}^{2+}/\text{Fe}_{\text{total}}$ ratios of one.
- Crystalline phases observed in the coarse-sieved DMR samples support increasing alumina bed turnover in the FBSR processing, with the final two DMR samples comprised mostly of nepheline group mineral phases. All of the HTF samples contained nepheline.
- PCT durability testing of the DMR and HTF samples along with appropriate HLW and LAW reference or benchmark glasses shows that the FBSR products are orders of magnitude more durable than the HLW EA glass, and at least 2X more durable than the current Hanford RPP WTP limit for LAW product disposal.
- REDOX results indicate that the HTF samples were overly reduced to form the desired Re host phase. This likely influenced the durability of the Re and S oxide components in these samples, contributing to slightly higher normalized release when compared to other components in the HTF products.
- The TCLP results performed on the FBSR samples by an independent off-site EPA-certified laboratory indicate that these products meet the criteria for the EPA RCRA Universal Treatment Standards for all of the constituents (Cr, Pb and Hg) in the simulants.

ACKNOWLEDGEMENTS

The authors wish to acknowledge many colleagues who contributed to the completion of this effort. Sherry Vissage prepared the samples for all compositional analysis and leach testing. Dave Best and Pat Toole performed the DMR/HTF chemical analyses and REDOX using ICP-AES in the SRNL Process Science Analytical Laboratory (PSAL). Several different SRNL Analytical Development personnel contributed. Art Jurgensen and David Missimer performed the XRD analyses. Curtis Johnson directed the ICP-MS analyses and performed the Hg dissolutions and analyses.

The authors would also like to thank Kevin Ryan of WGI and Arlin L. Olson, Idaho Cleanup Project, CH2M WG Idaho, LLC for technical advice and fruitful discussions regarding this work.

Finally, the authors would like to thank Mimi Jones for her support in the technical editing of this report.

Distribution:

Savannah River National Laboratory

J. E. Marra, 773-A
R. E. Edwards, 773-A
D. A. Crowley, 999-W
A. B. Barnes, 999-W
C. C. Herman, 999-W
D. B. Burns, 786-5A
N. E. Bibler, 773-A
C. M. Jantzen, 773-A
C. L. Crawford, 773-41A
J. C. Marra, 773-42A
J. R. Harbour, 773-42A
G. G. Wicks, 773-A
J. M. Pareizs, 773-A
D. K. Peeler, 999-W
W. E. Daniel, 999-W
M. R. Williams, 786-5A
P. R. Burket, 773-42A
M. E. Smith, 999-W
A. D. Cozzi, 999-W

THOR Treatment Technologies®:

Kevin Ryan, kevin.ryan@wgint.com
Brad Mason, brad.mason@wgint.com
Brent Evans, brent.evans@wgint.com
Vishal Vora, vishal.vora@wgint.com

Idaho Cleanup Project, CH2M WG Idaho, LLC:

Arlin Olson, Arlin.Olson@icp.doe.gov
Scott Roesener, W.Roesener@icp.doe.gov
John Tadlock, John.Tadlock@icp.doe.gov

Eldredge Engineering:

Brad Eldredge, brad@eldeng.com

U.S. Department of Energy:

Joel Case, CASEJT@ID.DOE.GOV
Bill Owca, OWCAWA@ID.DOE.GOV

7.0 REFERENCES

ASTM C1285. Annual Book of ASTM Standards, Vol. 12.01: **Standard Test Methods for Determining Chemical Durability of Nuclear, Hazardous, and Mixed Waste Glasses and Multiphase Glass Ceramics: The Product Consistency Test (PCT)**, (2002).

Burket, P.R., Marra, J.C., Pareizs, J.M. and Jantzen, C.M., **“Evaluation of Fluidized Bed Steam Reforming (FBSR) Technology for Sodium Bearing Wastes From Idaho and Hanford Using the Bench-Top Steam Reformer (BSR)”**, U.S. DOE Report WSRC-TR-2004-00560 (Jan 2005).

Ebert, W. L. and S. F. Wolf, **“An Interlaboratory Study of a Standard Glass for Acceptance Testing of Low-activity Waste Glass,”** J. of Nuc. Materials, 282, pp. 112-124, 2000.

Federal Register, Vol. 70, No. 148, August 3, 2005 / Notices, pp 44598 – 44600.

Jantzen, C. M. et al., **“Characterization of the Defense Waste Processing Facility (DWPF) Environmental Assessment (EA) Glass Standard Reference Material,”** US DOE Report WSRC-TR-92-346, Rev. 1, WSRC, Aiken, SC, 1993.

Jantzen, C. M. et al., **“Process/Product Models for the Defense Waste Processing Facility (DWPF): Part II. Appendix G,”** US DOE Report WSRC-TR-93-0672, p. 464, 1995.

Jantzen, C.M., Pareizs, J.M., Lorier, T.H. and Marra, J.C. **“Durability Testing of Fluidized Bed Steam Reforming (FBSR) Products,”** Ceramic Transactions V. 176, 121-137 (2006a).

Jantzen, C.M., Lorier, T.H., Marra, J.C., and Pareizs, J.M., **“Durability Testing of Fluidized Bed Steam Reforming (FBSR) Waste Forms”** WM06 Paper #6373 (February 2006b).

Jantzen, C. M., **“Fluidized Bed Steam Reformer (FBSR) Product: Monolith Formation and Characterization,”** U.S. DOE Report WSRC-STI-2006-00033, Rev. 0, (June 2006c).

Land Disposal Restrictions. **Code of Federal Regulations**, Part 268, Title 40, 2004.

Lorier, T.H., Pareizs, J.M. and Jantzen, C.M., **“Single-Pass Flow Through (SPFT) Testing of Fluidized Bed Steam Reforming (FBSR) Waste Forms,”** U.S. DOE Report WSRC-TR-2005-00124 (October 2005).

Mason, J. B. et al., THOR Treatment Technologies, LLC and B. Landman, CH2M WG Idaho, LLC, **“Steam Reforming Application for Treatment of DOE Sodium Bearing Tank Wastes at Idaho National Laboratory for Idaho Cleanup Project,”** Waste Management '06, Tucson, AZ, 2006.

Office of Solid Waste, US EPA. **TCLP Method 1311**, Test Methods for Evaluating Solid Waste; Physical/Chemical Methods, Third Edition; EPA 530/SW-846; National Technical Information Service: Washington, DC, 1986.

Pareizs, J. M., C. M. Jantzen and T. H. Lorier, “**Durability Testing of Fluidized Bed Steam Reformer (FBSR) Waste Forms for High Sodium Wastes at Hanford and Idaho (U)**,” U.S. DOE Report WSRC-TR-2005-00102 (May 2005).

Pilot Plant Report for Treating SBW Simulants: Mineralizing Flowsheet, Report Number 28266-RT-002, Rev. 0, THOR Treatment Technologiessm, July 2007.

Soelberg, N. R. et al., “Phase 2 THOR Steam Reforming Tests for Sodium-Bearing Waste Treatment,” INEEL/EXT-04-01493, Idaho National Engineering and Environmental Lab, Bechtel BWXT Idaho, LLC, January 2004a.

Soelberg, N. R. et al., “Phase 2 TWR Steam Reforming Tests for Sodium-Bearing Waste Treatment,” INEEL/EXT-04-01494, Idaho National Engineering and Environmental Lab, Bechtel BWXT Idaho, LLC, January 2004b.

WAPS-Waste Acceptance Product Specifications for Vitrified High Level Waste Forms, U. S. Department of Energy, Office of Environmental Restoration and Waste Management, USDOE Document EM-WAPS, Washington, DC (February, 2003).

WTP Contract, Section C, Statement of Work, Contract No. DE-AC27-01RV14136, Modification No. M076, 2007

APPENDIX A - XRD SPECTRA OF DMR AND HTF SAMPLES

DMR 4504
DMR 4531
DMR 4637
DMR 4726
HTF 4508
HTF 4546
HTF 4649
HTF 4728

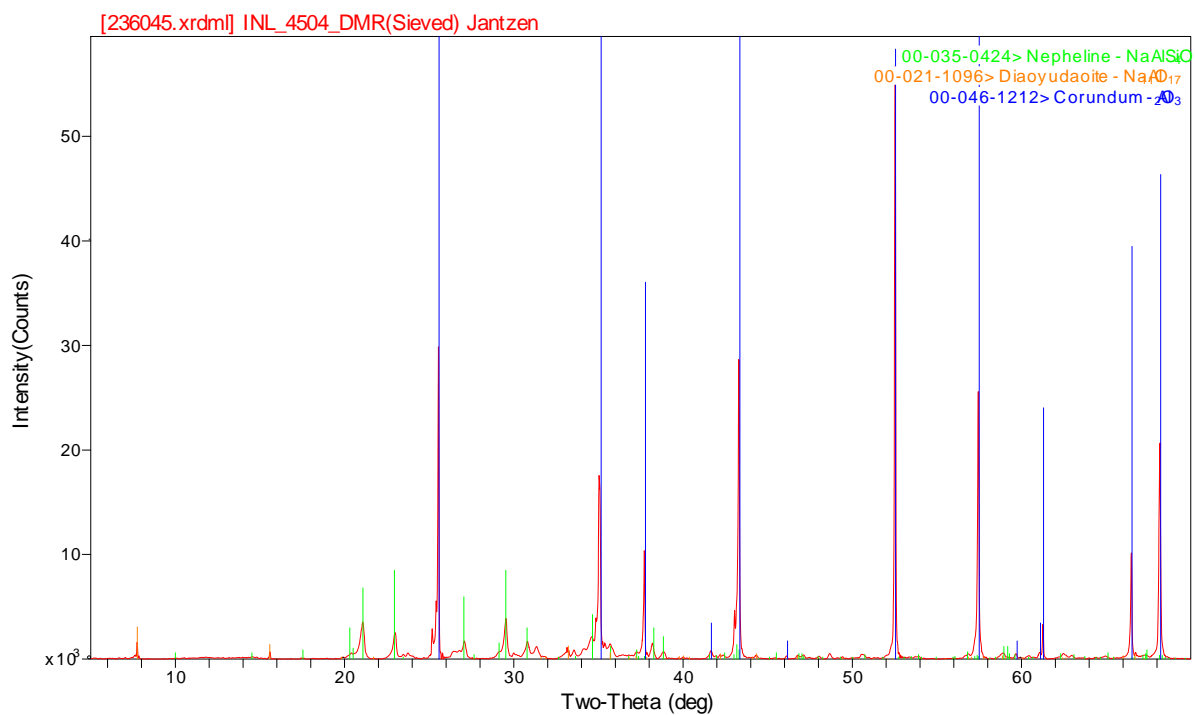


Figure A- 1. INL 4504 DMR (Sieved)

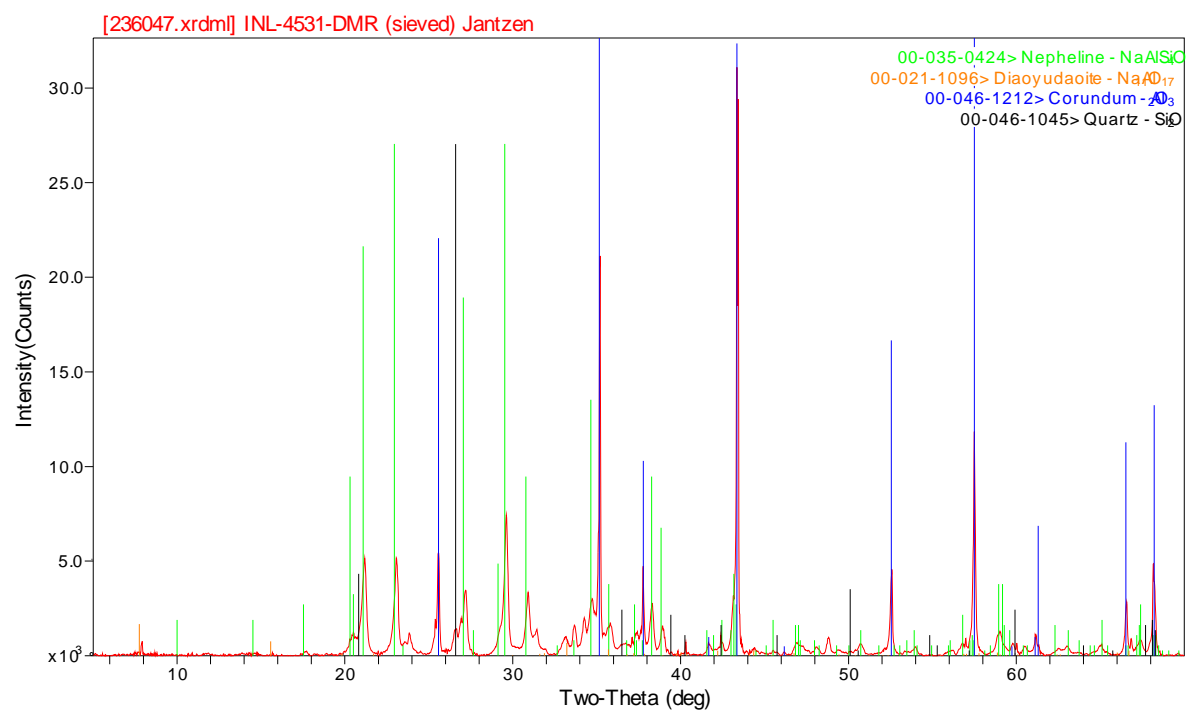


Figure A- 2. INL 4531 DMR (Sieved)

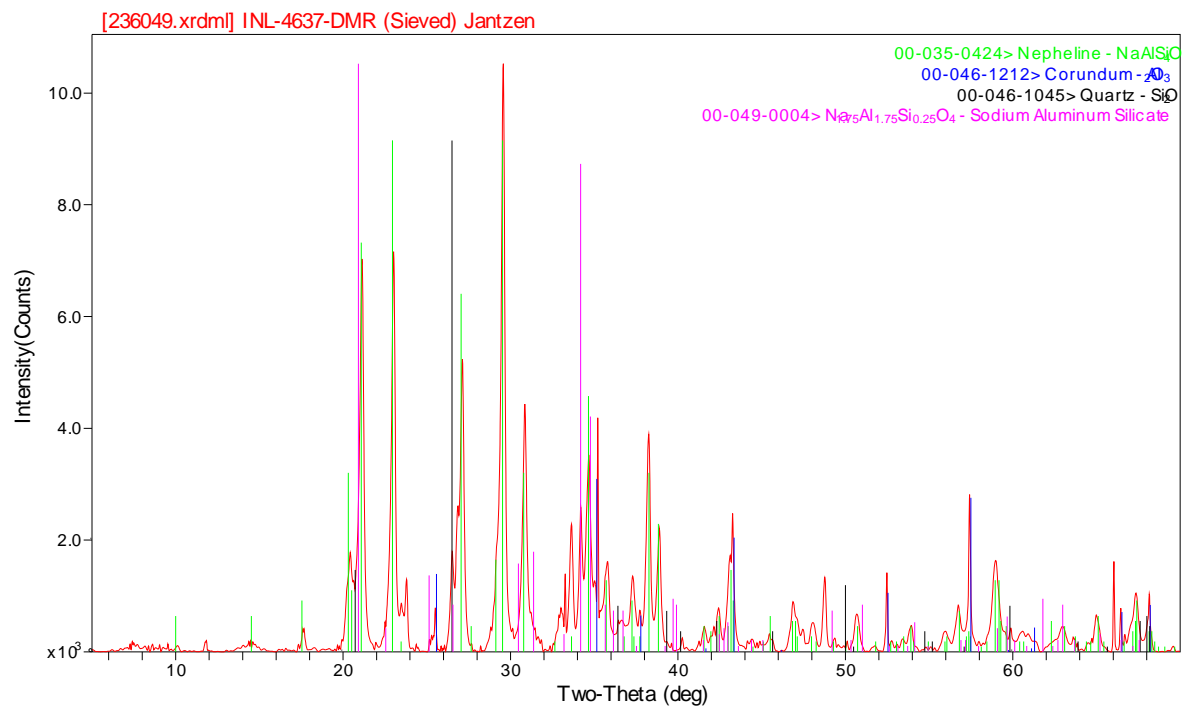


Figure A- 3. INL-4637-DMR (Sieved)

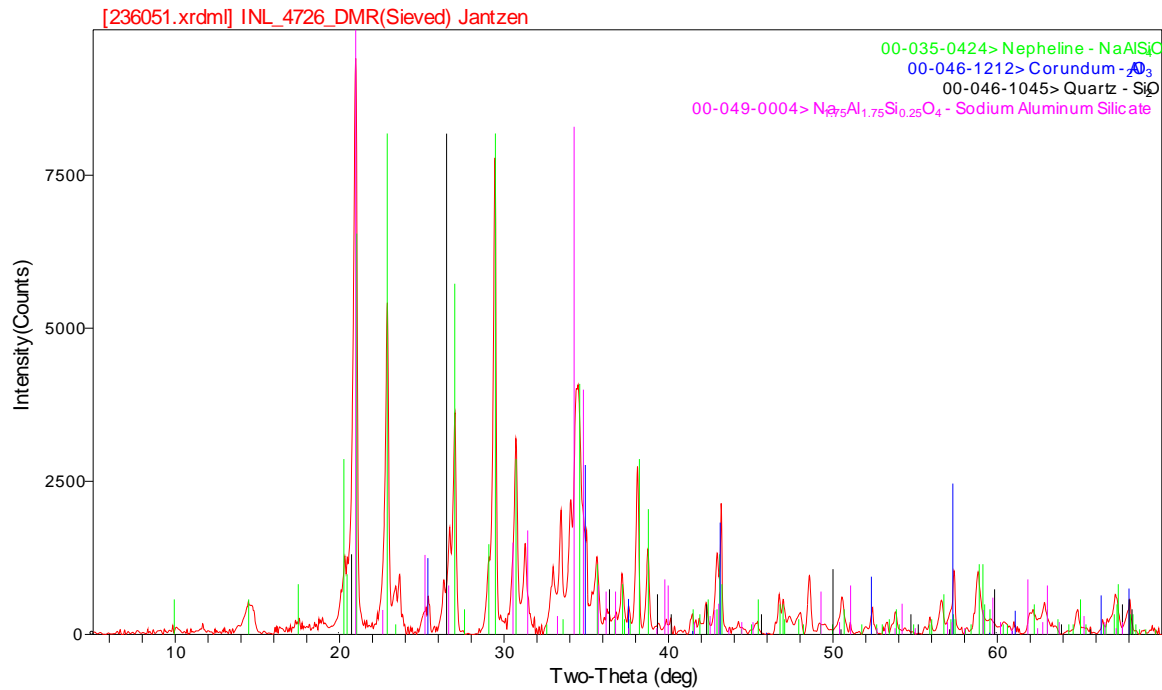


Figure A- 4. INL-4726-DMR (Sieved)

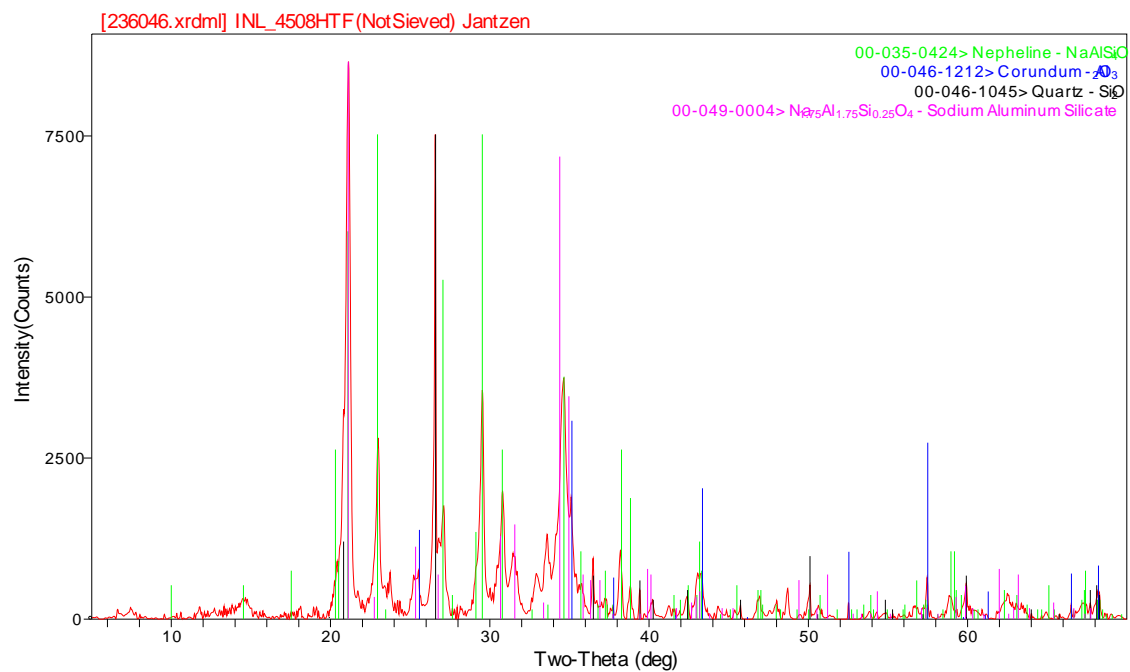


Figure A- 5. INL-4508-HTF (Not Sieved)

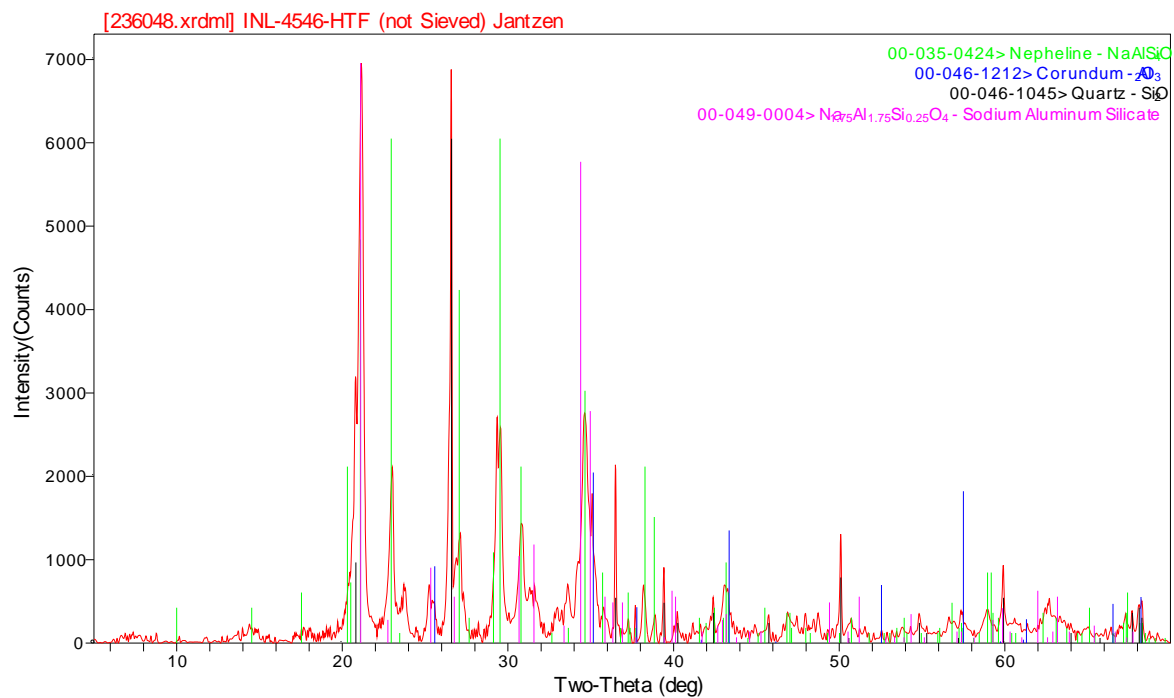


Figure A- 6. INL-4546-HTF (Not Sieved)

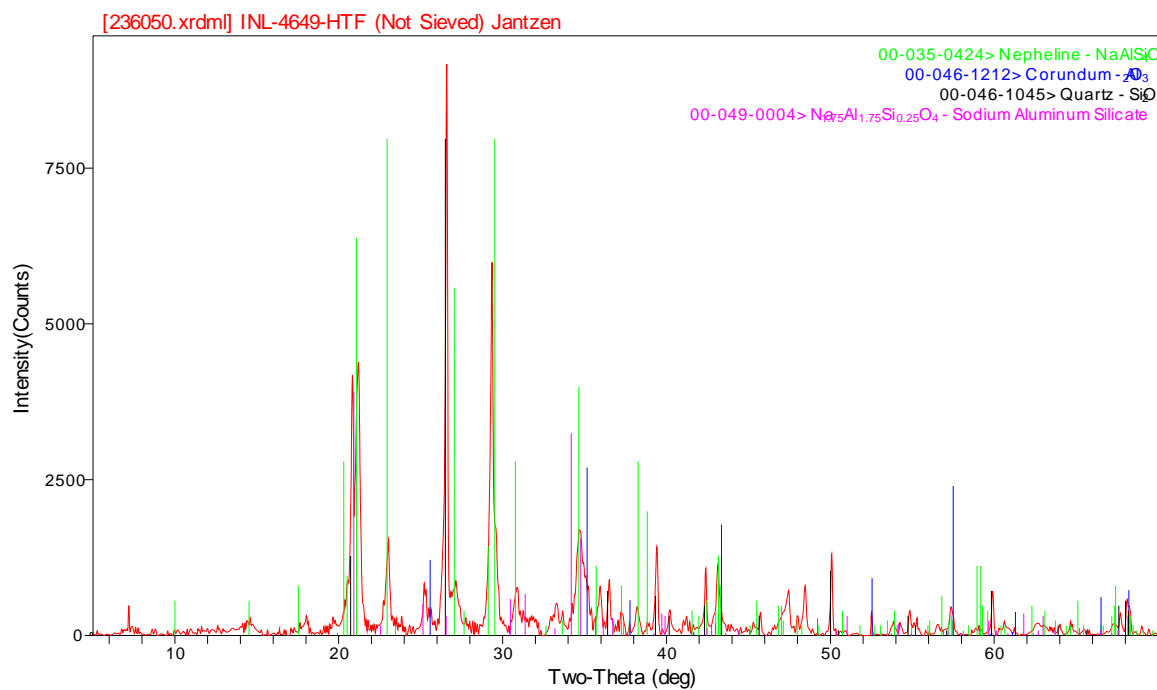


Figure A- 7. INL-4649-HTF (Not Sieved)

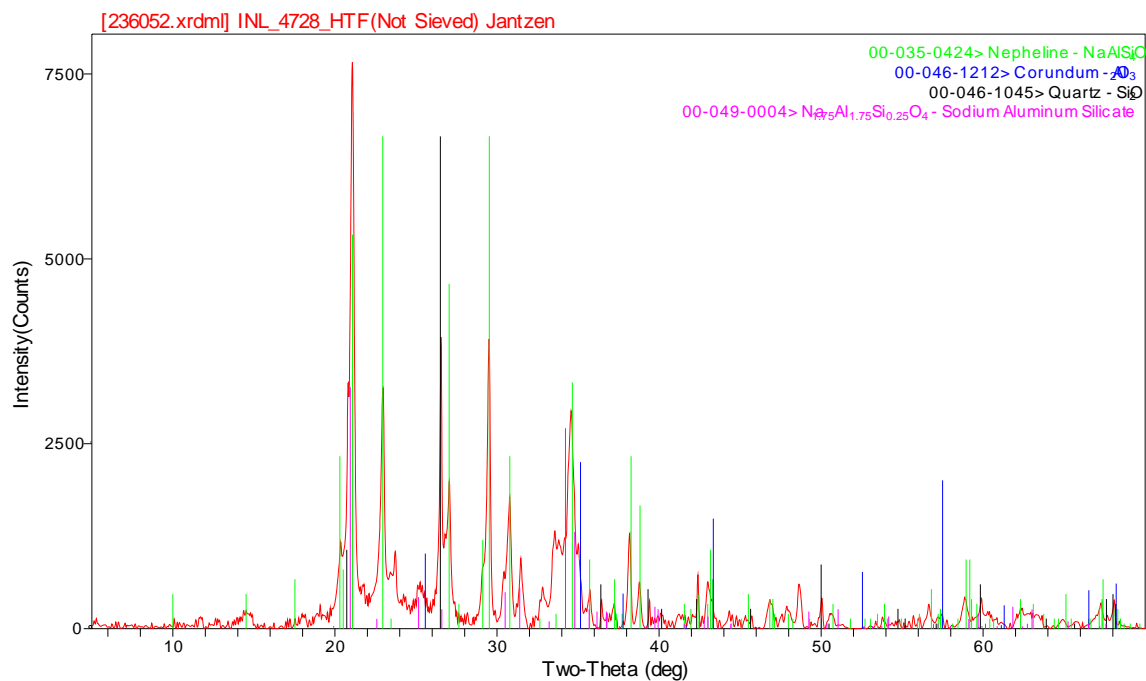


Figure A- 8. INL-4728-HTF (Not Sieved)

APPENDIX B – ACCURA REPORT

Analytical Report for: Washington Savannah River Co.

Project Name: 07079

Project ID: 07079

Project Manager: Larry Dewitt (WSRC)

Project Location: South Carolina

Lab. Work Order #: 11733

April 12, 2007

ACCURA Analytical Laboratory, Inc.

6017 Financial Drive, Norcross, GA 30071

The entire report from ACCURA Analytical Laboratory, Inc. follows.

Analytical Report for:
Washington Savannah River Co.

Project Name : 07079
Project ID : 07079
Project Manager: Larry Dewitt
Project Location : South Carolina

Lab. Work Order # : 11733



April 12, 2007

ACCURA Analytical Laboratory, Inc.
6017 Financial Drive Norcross, GA 30071
Phone: 770-449-8800 Fax: 770-449-5477

Labora

07079

Customer Name: Crawford, Charles

Contract Number
AC39040N

Customer Department: WTT/ITS

Customer Address: 773-41A Rm180

Customer Phone/Beeper	5-8049	18277
-----------------------	--------	-------

**Washington Savannah River Company
Aiken, SC 29808**

**Environmental Services Section
Waste Sample Management Group**

COC creation date.	3/12/07
--------------------	---------

Matrix: S=Soil, SO=Solid, SL=Sludge, O=Organic, A=Aqueous, SM=Smear
Sample Analysis Requested

TCLP, Metals (Prep & Analysis) (33)

TCLP, Metals - Report Nickel (106)

TCLP, Metals - Report Zinc (141)

11733-001	11733-002	11733-003
-----------	-----------	-----------

28 Dav TAT

STR Authorization *Larry DeWitt*

1 Relinquished by:

Received by:

Date/Time

2 Relinquished by:

Date/Time

Received by:

(Print) Charles L. Cawford

Date	3/15/07	(Print)	Grace Miller
------	---------	---------	--------------

(Print) Grace Miller

(Print) Minnie Lightner

3 Relinquished by:

Received by:

Date/Time

4 Relinquished by:

Date/Time

Received by:

(Print) **2** Winnie Lightfoot

3/15/07	(Print)	C/S
---------	---------	-----

(Print) **FED EX**

(Print) Boome, Michael

26

SAMPLE RECEIPT CHECKLIST VERSION 6

Client Code: 2321 AAL Project Mgr: DCF

Client Project Name: 07079 ACCURA Work Order#: 11733

Are there **EnCores**, tests with **< 48Hr hold times**, or **RUSH IAT's** requested? YES ☒ NO ☒

If YES, you must communicate RUSH analyses to the appropriate analyst(s) immediately!!! / or preserve EnCores (see #16 below)!!!

Preliminary Examination: Initials: 3m Date received: 3/16/07 Date cooler was opened: 3/16/07

1. Did cooler/package come with a shipping slip (airbill, Etc.)? YES ☒ NO ☐
If YES, enter carrier name and airbill number here: FedEx - 7916 5185 5227
Describe type of packing in cooler: Bubble Wrap / Ice
*****If cooler was hand delivered, CIRCLE HERE, skip to item #5*****
2. Were custody seals on outside of cooler? YES ☐ NO ☒
If YES, how many: _____ seal dated: _____ seal name: _____
3. Were custody seals unbroken and intact at the date and time of arrival? YES ☐ N/A ☒ NO ☐
4. Were custody papers sealed in a plastic bag to prevent damage to chain of custody? ☒ YES ☐ NO
5. If required, was enough ice used? (Internal cooler temperature, 4°C) ☒ YES ☐ N/A ☐ NO
6. Did you sign custody papers in the appropriate place? ☒ YES ☐ NO
7. Was project identifiable from custody papers? ☒ YES ☐ NO
If YES, enter project name at the top.

Complete project file with green sheet, proper file tag, and shipping documentation. Line up samples following chain. Complete Container Receipt Verification form (include extra containers for dissolved metals filtrates). Complete login in XENCO and generate AAL ID Labels.

8. Did all containers arrive unbroken and were labels in good condition? ☒ YES ☐ NO
9. Were custody papers filled out properly and did all labels agree with custody papers? ☒ YES ☐ NO
10. Were correct containers and sufficient amount of sample sent for the test indicated? ☒ YES ☐ NO
11. All samples collected within three days of date received for these analyses
(Reactive Cn & S, Solids in H₂O, Sulfide, Sulfite, IALL! Extractable Organic Waters)? YES ☐ N/A ☒ NO
If NO, coordinate with the project manager to ensure that no samples go out of hold!!!
12. No residual chlorine found in waters for these analyses:
(Cyanide, PAH, SVOC, Pesticides, PCB's, Herbicides)? YES ☐ N/A ☒ NO
Checked by: _____ (Initials)
13. Were samples properly chemically preserved, if required, upon receipt? YES ☐ N/A ☒ NO
(For example: pH checked for waters for all Metals, Wet Chemistry, Pesticides, PCB's, Herbicides, and VOC/BTEX samples submitted with HCL for waters and in either Encore samplers or NaHSO₄ labeled vials for soils)
Preservation checked by: _____ (Initials)
14. Were air bubbles (>1/4 inch) absent in VOC/BTEX samples? YES ☐ N/A ☒ NO
If NO, list ID # on back and label vials with: Do Not Use Until Notified By Management
15. If there are samples for dissolved metals, were they field filtered? YES ☐ N/A ☒ NO
If NO, list date and time samples were filtered and preserved in lab: _____

16. Were Encore samplers included? YES ☐ NO ☒
If YES, date and time preserved with NaHSO₄: _____ By whom: _____
17. Does this submittal contain soil NaHSO₄ vials for BTEX/GRO/VOC'S? YES ☐ NO ☒
If YES, vials weighed by and entered into vial database by: _____
18. Initials of laboratory personnel responsible for labeling laboratory I.D. numbers on containers: 3m

Keep samples and chain out. Before moving samples to their appropriate location, another person must review the entire project ensuring that information on the AAL ID Barcode label matches the container label, and that all information is consistent with the chain.

Final check and samples logged to locations by: _____ (Initials)

19. Was it necessary to call the assigned project manager in order to proceed with login? YES ☐ NO ☒
If YES, give details on the back of this form.
20. Who was called? 3m By whom? _____ Date/Time: _____

Project Mgr. Review: 3m (Initials) 3/19/07 (Date)

<u>Item #</u>	<u>Discrepancies Noted:</u>
---------------	-----------------------------


<u>Item #</u>	<u>Discrepancies Noted:</u>
---------------	-----------------------------

[illegible]

<u>Item #</u>	<u>Actions Taken:</u>
---------------	-----------------------

Project Mgr. Review: _____ (Initials) _____ (Date)

3/19/07





Accura Analytical Laboratory, Inc.

Sample Inspection and Receipt Survey Form

Date: 3/16/07

Time: 1600hr

Surveyor: MB

Instrument Data

Survey Type: Sample Inspection and Receipt

Client: 2321

Client Job/WO # 07079 WO# 11733

Comments: Scanned for RAD Properties

3/16/07
MB

Mfg. Eberline

Model SAC-4

Serial # 751

Cal Due 08/07/05

Efficiency 33.4

MDA 20 dpm

Mfg. Eberline

Model BC-4

Serial # BC-838

Cal Due 02/07/06

Efficiency 25.6

MDA 346 dpm

Mfg. N.E. Tech

Model Electra

Serial # 1429

Cal Due 08/07/05

Efficiency α 20.4 β 30.5

MDA α 57 β 338

Mfg. Bicorn

Model Micro Rem

Serial # C3036

Cal Due 08/07/05

Efficiency N/A

MDA 17 μ REM/hr

SURVEY RESULTS

SWIPE #	LOCATION	ALPHA	ALPHA	BETA	BETA	Exposure	Other
		Swipe	Frisk	Swipe	Frisk	Micro Rem	
		dpm/100cm ²	dpm/100cm ²	dpm/100cm ²	dpm/100cm ²	μ REM/hr	
1	Cooler Exterior	—	< 57	—	< 338	< 17 μ REM/hr	—
2	Cooler Interior	—	< 57	—	< 338	< 17 μ REM/hr	—
3	Sample Bag #1	—	< 57	—	< 338	< 17 μ REM/hr	—
4	Sample Bag #2	—	< 57	—	< 338	—	—
5	Sample Bubble Wrap #	—	< 57	—	< 338	—	—
6	Sample # 5	—	< 57	—	< 338	—	—
7	Sample Bubble Wrap #	—	—	—	—	—	—
8	Sample # 6	—	< 57	—	< 338	—	—
9	Sample Bubble Wrap #	—	—	—	—	—	—
10	Sample # 7	—	< 57	—	< 338	—	—
11	Sample Bubble Wrap #	—	—	—	—	—	—
12	Sample # 8	—	< 57	—	< 338	—	—
13	Sample Bubble Wrap #	—	—	—	—	—	—
14	Sample #	—	—	—	—	—	—
15	Background	—	< 57	—	< 338	< 17 μ REM/hr	—

Reviewed by RSO: [Signature]

Date: 3/16/07

Time: 1720



ACCURA ANALYTICAL LABORATORY, INC

A Multi-Service Corporation.

6017 Financial Drive, Norcross, Georgia 30071, 770-449-8800 (ph), 770-449-5477 (fax)

CASE NARRATIVE REPORT

for

Washington Savannah River Site

Subcontract No. AC39040N

WSRC Job #: 07079

AAL WO #: 11733

Date: April 12, 2007

Laboratory Identification: Accura Analytical Laboratory, Inc.

Summary:

Sample Receipt:

Eight solid samples from the Washington Savannah River Site arrived at Accura Analytical Laboratory, Inc. on March 16, 2007 for analysis. The sample listed on the chain arrived to the laboratory via FedEx. A twenty-eight day turnaround was requested on the chain of custody.

The sample was stored properly according to SW-846 procedures and Laboratory Standard Operating Procedures (SOP).

The laboratory received the following samples:

<u>Client Sample ID</u>	<u>Laboratory ID</u>	<u>Matrix</u>
07079-4504-DMR	11733-001	Solid
07079-4508-HTF	11733-002	Solid
07079-4531-DMR	11733-003	Solid
07079-4546-HTF	11733-004	Solid
07079-4637-DMR	11733-005	Solid
07079-4649-HTF	11733-006	Solid
07079-4726-DMR	11733-007	Solid
07079-4728-HTF	11733-008	Solid

Case Narrative

Sample analyses were conducted using methodology as outlined in Accura Analytical Laboratory's Standard Operating Procedures. Any technical or administrative problems during analysis, data review, and reduction are written by analytical fraction in the enclosed narratives.

Data Package:

The enclosed data package contains the following sections: Case Narrative, Certificate of



ACCURA ANALYTICAL LABORATORY, INC

A Multi-Service Corporation.

6017 Financial Drive, Norcross, Georgia 30071, 770-449-8800 (ph), 770-449-5477 (fax)

Analysis, Surrogate information, Quality Control Results, Sample Receipt Checklist, Rad Sample Receipt Checklist (as appropriate), Chain of Custody, and Nonconformance Reports.

The Certificate of Analysis contains the following headings:

Sample ID:	Sample Identification
Lab ID:	This is the laboratory identification number
Matrix:	Sample matrix
Date Collected:	Date of sample collection
Date Received:	Date of sample receipt by the laboratory
Priority:	NA
Collector:	Party responsible for sample collection

The detail on the Certificate includes the following:

Parameter:	Analyte or characteristic tested for in the sample
Qualifier:	Qualifier used for data interpretation
Result:	Final result for each parameter
DL:	Method Detection Limit (adjusted)
RL:	Reporting Limit (adjusted)
Units:	Units of final result
DF:	Dilution Factor
Analyst:	Initials of analyst who performed test
Date:	Date of analysis
Time:	Time of analysis
Batch:	Analytical batch in which the sample was analyzed
Method:	Analytical method used for the analysis of the sample. Identified on the report numerically with a corresponding table.
Surrogate Recovery:	Provided for organic analysis only. Surrogate compound identified.
Test:	Analytical test associated with surrogate compound.
Percent %:	Surrogate percent recovery
Acceptable Limits:	Limits established for surrogate recoveries based upon the method requirements.

The QC Summary Report contains the following headings:

Sample ID:	Analyte or characteristic tested for in the QC sample.
Batch:	Analytical batch in which the QC sample was analyzed
Spike Amt:	Nominal concentration of the spiking compound
Sample Result:	Amount of compound found in the sample associated with the QC sample.
__Result:	Amount of compound found in the QC sample



ACCURA ANALYTICAL LABORATORY, INC

A Multi-Service Corporation.

6017 Financial Drive, Norcross, Georgia 30071, 770-449-8800 (ph), 770-449-5477 (fax)

RPD:	Relative percent difference between LCS/LCS dup, MS/MSD, and/or Sample/Sample duplicate
Recovery:	Recovery for the control samples
Reference Range:	Acceptance limits for control samples

Types of QC samples that may be found on the QC Summary Report and/or Certificate of Analysis are:

Blank:	Results of the blank analysis for the sample batch
LCS:	Lab control sample
LCS dup:	Lab control sample duplicate
MD:	Duplicate analysis of sample
MS:	Matrix spike
MSD:	Matrix spike duplicate
PS:	Post-Digestion Spike
PSD:	Post-Digestion Spike Duplicate
SDILT:	Serial Dilution

The following are definitions of reporting limits used at Accura Analytical Laboratory:

DL Detection Limit: The minimum level of an analyte that can be determined (identified not quantified) with 99% confidence. The values are normally achieved by preparing and analyzing seven aliquots of laboratory water spiked 1 to 5 times the estimated MDL, taking the standard deviation and multiplying it against the one-tailed t-statistic at 99%.

The detection limit is the minimum concentration of a substance that can be identified, measured, and reported with 99% confidence that the analyte concentration is above zero. It answers the question "Is it present".

QL Quantitation Limit: The lowest concentration that can be reliably achieved within specified limits of precision and accuracy during routine laboratory operating conditions. The QL is generally 5 to 10 times the MDL. However, it may be nominally chosen within these guidelines to simplify data reporting. For many analytes the QL analyte concentration is selected as the slowest non-zero standard in the calibration curve. Sample QL's are highly matrix-dependent. Sample specific preparation and dilution factors are applied to these limits when they are reported.

The QL is always > DL

RL Reporting Limit: Same as QL except where driven by contract or client specifications. If the sample specific preparation and dilution factors cause the QL to be elevated above the RL, then the QL is used as the RL.



ACCURA ANALYTICAL LABORATORY, INC

A Multi-Service Corporation.

6017 Financial Drive, Norcross, Georgia 30071, 770-449-8800 (ph), 770-449-5477 (fax)

The quantitation limit is the lowest level at which a chemical may be accurately and reproducibly quantitated. It answers the question "How much is present".

Interpretation of RESULT column on the Certificate of Analysis:

If the final concentration in the sample was found to be above the RL, then the value reported is reported without a flag;

If the final concentration in the sample was found to be below the RL but above the DL, then the value reported is flagged with a "J";

If the final concentration in the sample was found to be below the DL, the value reported is flagged with a "U".

If the final concentration in the sample was found in the corresponding method blank, the value reported is flagged with a "B".

If the final concentration for an analyte in the sample was found to be exceeding the upper level of the calibration range, the value reported is flagged with a "E".

If the final concentration for an analyte in the sample is reported from the dilution run, the value reported is flagged with a "D".

A combination of "JD" and "JB" flags are also used with the above definitions.

Quality Control Flags

Accura Analytical Laboratory maintains acceptance criteria for QC samples through use of statistical process control (SPC). The SPC limits are used to qualify data usability. The flagging criterion identified in WSRC AN98 Format does not necessarily coincide with the laboratory SPC criteria. There may be instances where the Electronic Data Deliverable (EDD) has flagged data based on the AN98 criteria and the lab has not identified the data to be outside of established control limits.

Those instances where the QC has not met laboratory SPC established criteria will be noted in the section case narratives that are included in this package.

This data package, to the best of my knowledge, is in compliance with technical and administrative requirements.

David C. Fuller

Project Manager - WSRC



ACCURA ANALYTICAL LABORATORY, INC

A Multi-Service Corporation.

6017 Financial Drive, Norcross, Georgia 30071, 770-449-8800 (ph), 770-449-5477 (fax)

**Case Narrative for WSRC
Work Order # 11733
Metals Analysis by ICP
Mercury Analysis by CVAA**

Sample Analysis

The following sample was prepared and analyzed for "TCLP Metals", according to the methods referenced in the "Method / Analysis Information" section of this narrative:

<u>Client Sample ID</u>	<u>Laboratory ID</u>
07079-4504-DMR	11733-001
07079-4508-HTF	11733-002
07079-4531-DMR	11733-003
07079-4546-HTF	11733-004
07079-4637-DMR	11733-005
07079-4649-HTF	11733-006
07079-4726-DMR	11733-007
07079-4728-HTF	11733-008

TCLP – Metals

302349 BLK (Blank)	Method Blank (MB)
302349 BKS (Blank Spike)	Laboratory Control Sample (LCS)
302349 BSD (Blank Spike Duplicate)	Laboratory Control Sample Duplicate(LCSD)
07079-4649-HTF MD	Matrix Sample Duplicate - 11733-006 MD
07079-4649-HTF MS	Matrix Spike - 11733-006 MS
07079-4649-HTF MSD	Matrix Spike Duplicate - 11733-006 MSD

TCLP – Mercury

302366 BLK (Blank)	Method Blank (MB)
302366 BKS (Blank Spike)	Laboratory Control Sample (LCS)
302366 BSD (Blank Spike Duplicate)	Laboratory Control Sample Duplicate(LCSD)
07079-4728-HTF MD	Matrix Sample Duplicate - 11733-008 MD
07079-4728-HTF MS	Matrix Spike - 11733-008 MS
07079-4728-HTF MSD	Matrix Spike Duplicate - 11733-008 MSD

Method/Analysis Information:

TCLP Metals

Analysis Batch: 35559
Prep Batch #: 302349
Procedure: TCLP-ICP-6010
Analytical Method: SW846 1311/6010
Prep Method: SW846 3050B



ACCURA ANALYTICAL LABORATORY, INC

A Multi-Service Corporation.

6017 Financial Drive, Norcross, Georgia 30071, 770-449-8800 (ph), 770-449-5477 (fax)

TCLP Mercury

Analysis Batch: 35601
Prep Batch #: 302366
Procedure: TCLP-CVAA-7470A
Analytical Method: SW846 1311/7470A
Prep Method: SW846 1311/7470A

System Configuration

Metals

The analysis was performed on an inductively coupled plasma mass spectrometer. The instrument is equipped with a Meinhardt nebulizer, cyclonic spray chamber, and uses lithium, scandium, yttrium, indium and bismuth as internal standards. Operating conditions for the ICP/MS are set at a power level of 1000 watts. The instrument has a peristaltic pump flow rate of 24 RPM (2.0 mL/min sample uptake rate), argon gas flow of 15L/min for the torch and 0.5L/min for the plasma (torch tip), and a pressure setting of 20 PSI for the nebulizer.

Mercury

Mercury analysis was performed on a Flow Injection Mercury System (FIMS-400) automated mercury analyzer. The instrument consists of a cold vapor atomic absorption spectrometer set to detect mercury at a wavelength of 254nm. Sample introduction through the flow injection system is performed via a peristaltic pump at 9-mL/min and nitrogen carrier gas rate of 5 L/min.

Sample Preparation

The samples were prepared in accordance with the referenced SW-846 procedures.

Calibration Information

Initial Calibration

Instrument calibrations were conducted using method and instrument manufacturer's specification. All initial calibration requirements have been met for this analysis.

CRDL Standard

All CRDL standard recoveries met the advisory limits.

ICSA/ICSAB Requirements

All interference check samples (ICSA and ICSAB) associated with this work order met the established acceptance criteria.

Continuing Calibration Blanks (CCB) Requirements

All continuing calibration blanks (CCB) bracketing sample analyses associated with this work order met the established acceptance criteria.



ACCURA ANALYTICAL LABORATORY, INC

A Multi-Service Corporation.

6017 Financial Drive, Norcross, Georgia 30071, 770-449-8800 (ph), 770-449-5477 (fax)

Continuing Calibration Verification (CCV) Requirements

All continuing calibration verification (CCV) standards bracketing sample analyses associated with this work order met the recovery acceptance criteria.

Quality Control (QC) Information

Method Blank Acceptance

The method blanks analyzed with this job did not contain analytes of interest at concentrations greater than the reporting limit (RL).

LCS/LCSD Recovery Statement

All LCS and LCSD spike recoveries for this job were within the required acceptance limits.

QC Sample Designation

Sample 07079-4649-HTF from WSRC Job 07079 was designated as the quality control sample for the TCLP Metals ICP batch and sample 07079-4728-HTF was designated QC sample for the TCLP Mercury CVAA batch.

The ICP and the CVAA batch included a Laboratory Control Sample (LCS), Laboratory Control Sample Duplicate(LCSD), Matrix Spike (MS) and Matrix Spike Duplicate (MSD).

MS/MSD Recovery Statement

The percent recoveries (%R) obtained from the matrix spike (MS) and matrix spike duplicate (MSD) analyses were evaluated when the sample concentration was less than four times (4X) the spike concentration added. All applicable elements in the MS and MSD analyses met the established recovery limits.

MS/MSD RPD Statement

The relative percent difference (RPD) between the MS and MSD recoveries were within the acceptance limits for all the analytes for this work order.

Serial Dilution % Difference Statement

The serial dilution is used to access interference caused by matrix suppression or enhancement. Raw element concentrations that are at least 50X the instrument detection limit (IDL) for ICP analyses are applicable for serial dilution assessment.

All applicable elements met the established criteria for serial dilution evaluations, percent differences values <10.

Technical Information

Holding Time Specification

The samples associated with this work order met the specified holding time requirements.



ACCURA ANALYTICAL LABORATORY, INC

A Multi-Service Corporation.

6017 Financial Drive, Norcross, Georgia 30071, 770-449-8800 (ph), 770-449-5477 (fax)

Sample Dilution

Dilutions are performed to minimize matrix interference resulting from elevated mineral elements concentrations present in samples and/or to bring over range target analyte concentrations into the linear calibration range of this instruments. No sample dilution was required in this work order:

LQCF (NCR) Documentation

Laboratory Quality Communication Forms (LQCFs) are generated to document procedural anomalies that may deviate from referenced SOPs or Contractual documents. No LQCF was necessary for this work order.

Additional Comments

The additional comments field is used to address special issues associated with each analysis, clarify method/contractual issues pertaining to the analysis and to list any report documents generated as a result of sample analysis or review. No additional comments were required for this work order.

Review / Validation

The laboratory requires all analytical data to be verified by a qualified data validator.

The following data validator verified the data presented in this job.

Reviewer: 

Date: April 13, 2007



Certificate of Analytical Results 11733

Washington Savannah River Co., Aiken, SC

07079

Sample Id: 07079-4504-DMR		Matrix: SOLID		% Moisture:	
Lab Sample Id: 11733-001		Date Collected: Mar-15-07 09:05		Date Received: Mar-16-07 10:11	
Sample Depth:					
Analytical Method: TCLP Mercury by SW1311/7470A				Prep Method: SW7470A_DIG	
Date Analyzed: Apr-03-07 16:39		Analyst: MSN01		Date Prep: Apr-02-07 15:00	
		Seq Number: 35601		Tech: MSN01	
Parameter	Cas Number	Result	Rep Limit	MDL	Units Flag Dil
Mercury	7439-97-6	0.00U	0.0200	0.000860	mg/L U 1
Analytical Method: TCLP Metals by SW1311/6010B (33)				Prep Method: SW3010A	
Date Analyzed: Mar-30-07 13:56		Analyst: VHB01		Date Prep: Mar-29-07 13:00	
		Seq Number: 35559		Tech: MSN01	
Parameter	Cas Number	Result	Rep Limit	MDL	Units Flag Dil
Arsenic	7440-38-2	0.00U	1.0	0.0086	mg/L U 1
Barium	7440-39-3	0.23	1.0	0.0016	mg/L J 1
Cadmium	7440-43-9	0.00U	1.0	0.0022	mg/L U 1
Chromium	7440-47-3	0.19	1.0	0.0040	mg/L J 1
Lead	7439-92-1	0.12	1.0	0.0060	mg/L J 1
Nickel	7440-02-0	0.37	20	0.0016	mg/L J 1
Selenium	7782-49-2	0.13	1.0	0.023	mg/L JB 1
Silver	7440-22-4	0.00U	1.0	0.0038	mg/L U 1
Zinc	7440-66-6	0.40	40	0.012	mg/L J 1

*



Certificate of Analytical Results 11733

Washington Savannah River Co., Aiken, SC

07079

Sample Id: 07079-4508-HTF		Matrix: SOLID		% Moisture:	
Lab Sample Id: 11733-002		Date Collected: Mar-15-07 09:06		Date Received: Mar-16-07 10:11	
Sample Depth:					
Analytical Method: TCLP Mercury by SW1311/7470A				Prep Method: SW7470A_DIG	
Date Analyzed: Apr-03-07 16:43		Analyst: MSN01		Date Prep: Apr-02-07 15:00	
		Seq Number: 35601		Tech: MSN01	
Parameter	Cas Number	Result	Rep Limit	MDL	Units Flag Dil
Mercury	7439-97-6	0.00U	0.0200	0.000860	mg/L U 1
Analytical Method: TCLP Metals by SW1311/6010B (33)				Prep Method: SW3010A	
Date Analyzed: Mar-30-07 14:02		Analyst: VHB01		Date Prep: Mar-29-07 13:00	
		Seq Number: 35559		Tech: MSN01	
Parameter	Cas Number	Result	Rep Limit	MDL	Units Flag Dil
Arsenic	7440-38-2	0.00U	1.0	0.0086	mg/L U 1
Barium	7440-39-3	0.29	1.0	0.0016	mg/L J 1
Cadmium	7440-43-9	0.00U	1.0	0.0022	mg/L U 1
Chromium	7440-47-3	0.11	1.0	0.0040	mg/L J 1
Lead	7439-92-1	0.020	1.0	0.0060	mg/L J 1
Nickel	7440-02-0	0.0044	20	0.0016	mg/L J 1
Selenium	7782-49-2	0.13	1.0	0.023	mg/L JB 1
Silver	7440-22-4	0.00U	1.0	0.0038	mg/L U 1
Zinc	7440-66-6	0.018	40	0.012	mg/L JB 1

*



Certificate of Analytical Results 11733

Washington Savannah River Co., Aiken, SC

07079

Sample Id: 07079-4531-DMR		Matrix: SOLID		% Moisture:	
Lab Sample Id: 11733-003		Date Collected: Mar-15-07 09:07		Date Received: Mar-16-07 10:11	
Sample Depth:					
Analytical Method: TCLP Mercury by SW1311/7470A				Prep Method: SW7470A_DIG	
Date Analyzed: Apr-03-07 16:47		Analyst: MSN01		Date Prep: Apr-02-07 15:00	
		Seq Number: 35601		Tech: MSN01	
Parameter	Cas Number	Result	Rep Limit	MDL	Units Flag Dil
Mercury	7439-97-6	0.00U	0.0200	0.000860	mg/L U 1
Analytical Method: TCLP Metals by SW1311/6010B (33)				Prep Method: SW3010A	
Date Analyzed: Mar-30-07 15:13		Analyst: VHB01		Date Prep: Mar-29-07 13:00	
		Seq Number: 35559		Tech: MSN01	
Parameter	Cas Number	Result	Rep Limit	MDL	Units Flag Dil
Arsenic	7440-38-2	0.00U	1.0	0.0086	mg/L U 1
Barium	7440-39-3	0.20	1.0	0.0016	mg/L J 1
Cadmium	7440-43-9	0.00U	1.0	0.0022	mg/L U 1
Chromium	7440-47-3	0.15	1.0	0.0040	mg/L J 1
Lead	7439-92-1	0.074	1.0	0.0060	mg/L J 1
Nickel	7440-02-0	0.34	20	0.0016	mg/L J 1
Selenium	7782-49-2	0.13	1.0	0.023	mg/L JB 1
Silver	7440-22-4	0.00U	1.0	0.0038	mg/L U 1
Zinc	7440-66-6	0.57	40	0.012	mg/L J 1

*



Certificate of Analytical Results 11733

Washington Savannah River Co., Aiken, SC

07079

Sample Id: 07079-4546-HTF		Matrix: SOLID		% Moisture:	
Lab Sample Id: 11733-004		Date Collected: Mar-15-07 09:08		Date Received: Mar-16-07 10:11	
Sample Depth:					
Analytical Method: TCLP Mercury by SW1311/7470A				Prep Method: SW7470A_DIG	
Date Analyzed: Apr-03-07 16:51		Analyst: MSN01		Date Prep: Apr-02-07 15:00	
		Seq Number: 35601		Tech: MSN01	
Parameter	Cas Number	Result	Rep Limit	MDL	Units Flag Dil
Mercury	7439-97-6	0.00U	0.0200	0.000860	mg/L U 1
Analytical Method: TCLP Metals by SW1311/6010B (33)				Prep Method: SW3010A	
Date Analyzed: Mar-30-07 15:18		Analyst: VHB01		Date Prep: Mar-29-07 13:00	
		Seq Number: 35559		Tech: MSN01	
Parameter	Cas Number	Result	Rep Limit	MDL	Units Flag Dil
Arsenic	7440-38-2	0.00U	1.0	0.0086	mg/L U 1
Barium	7440-39-3	0.19	1.0	0.0016	mg/L J 1
Cadmium	7440-43-9	0.00U	1.0	0.0022	mg/L U 1
Chromium	7440-47-3	0.016	1.0	0.0040	mg/L J 1
Lead	7439-92-1	0.011	1.0	0.0060	mg/L J 1
Nickel	7440-02-0	0.010	20	0.0016	mg/L J 1
Selenium	7782-49-2	0.12	1.0	0.023	mg/L JB 1
Silver	7440-22-4	0.00U	1.0	0.0038	mg/L U 1
Zinc	7440-66-6	0.017	40	0.012	mg/L JB 1

*



Certificate of Analytical Results 11733

Washington Savannah River Co., Aiken, SC

07079

Sample Id: 07079-4637-DMR		Matrix: SOLID		% Moisture:	
Lab Sample Id: 11733-005		Date Collected: Mar-15-07 09:09		Date Received: Mar-16-07 10:11	
Sample Depth:					
Analytical Method: TCLP Mercury by SW1311/7470A				Prep Method: SW7470A_DIG	
Date Analyzed: Apr-03-07 16:55		Analyst: MSN01		Date Prep: Apr-02-07 15:00	
		Seq Number: 35601		Tech: MSN01	
Parameter	Cas Number	Result	Rep Limit	MDL	Units Flag Dil
Mercury	7439-97-6	0.00U	0.0200	0.000860	mg/L U 1
Analytical Method: TCLP Metals by SW1311/6010B (33)				Prep Method: SW3010A	
Date Analyzed: Mar-30-07 15:23		Analyst: VHB01		Date Prep: Mar-29-07 13:00	
		Seq Number: 35559		Tech: MSN01	
Parameter	Cas Number	Result	Rep Limit	MDL	Units Flag Dil
Arsenic	7440-38-2	0.011	1.0	0.0086	mg/L J 1
Barium	7440-39-3	0.29	1.0	0.0016	mg/L J 1
Cadmium	7440-43-9	0.00U	1.0	0.0022	mg/L U 1
Chromium	7440-47-3	0.44	1.0	0.0040	mg/L J 1
Lead	7439-92-1	0.20	1.0	0.0060	mg/L J 1
Nickel	7440-02-0	0.37	20	0.0016	mg/L J 1
Selenium	7782-49-2	0.12	1.0	0.023	mg/L JB 1
Silver	7440-22-4	0.00U	1.0	0.0038	mg/L U 1
Zinc	7440-66-6	0.76	40	0.012	mg/L J 1

*



Certificate of Analytical Results 11733

Washington Savannah River Co., Aiken, SC

07079

Sample Id: 07079-4649-HTF	Matrix: SOLID	% Moisture:
Lab Sample Id: 11733-006	Date Collected: Mar-15-07 09:10	Date Received: Mar-16-07 10:11
Sample Depth:		

Analytical Method: TCLP Mercury by SW1311/7470A	Prep Method: SW7470A_DIG
Date Analyzed: Apr-03-07 17:06	Analyst: MSN01
Seq Number: 35601	Date Prep: Apr-02-07 15:00
	Tech: MSN01

Parameter	Cas Number	Result	Rep Limit	MDL	Units	Flag	Dil
Mercury	7439-97-6	0.00U	0.0200	0.000860	mg/L	U	1

Analytical Method: TCLP Metals by SW1311/6010B (33)	Prep Method: SW3010A
Date Analyzed: Mar-30-07 13:31	Analyst: VHB01
Seq Number: 35559	Date Prep: Mar-29-07 13:00
	Tech: MSN01

Parameter	Cas Number	Result	Rep Limit	MDL	Units	Flag	Dil
Arsenic	7440-38-2	0.00U	1.0	0.0086	mg/L	U	1
Barium	7440-39-3	0.34	1.0	0.0016	mg/L	J	1
Cadmium	7440-43-9	0.00U	1.0	0.0022	mg/L	U	1
Chromium	7440-47-3	0.012	1.0	0.0040	mg/L	J	1
Lead	7439-92-1	0.013	1.0	0.0060	mg/L	J	1
Nickel	7440-02-0	0.0050	20	0.0016	mg/L	J	1
Selenium	7782-49-2	0.11	1.0	0.023	mg/L	JB	1
Silver	7440-22-4	0.00U	1.0	0.0038	mg/L	U	1
Zinc	7440-66-6	0.019	40	0.012	mg/L	JB	1

*



Certificate of Analytical Results 11733

Washington Savannah River Co., Aiken, SC

07079

Sample Id: 07079-4726-DMR		Matrix: SOLID		% Moisture:	
Lab Sample Id: 11733-007		Date Collected: Mar-15-07 09:11		Date Received: Mar-16-07 10:11	
Sample Depth:					
Analytical Method: TCLP Mercury by SW1311/7470A				Prep Method: SW7470A_DIG	
Date Analyzed: Apr-03-07 17:10		Analyst: MSN01		Date Prep: Apr-02-07 15:00	
		Seq Number: 35601		Tech: MSN01	
Parameter	Cas Number	Result	Rep Limit	MDL	Units Flag Dil
Mercury	7439-97-6	0.00U	0.0200	0.000860	mg/L U 1
Analytical Method: TCLP Metals by SW1311/6010B (33)				Prep Method: SW3010A	
Date Analyzed: Mar-30-07 15:28		Analyst: VHB01		Date Prep: Mar-29-07 13:00	
		Seq Number: 35559		Tech: MSN01	
Parameter	Cas Number	Result	Rep Limit	MDL	Units Flag Dil
Arsenic	7440-38-2	0.014	1.0	0.0086	mg/L J 1
Barium	7440-39-3	0.27	1.0	0.0016	mg/L J 1
Cadmium	7440-43-9	0.00U	1.0	0.0022	mg/L U 1
Chromium	7440-47-3	0.055	1.0	0.0040	mg/L J 1
Lead	7439-92-1	0.024	1.0	0.0060	mg/L J 1
Nickel	7440-02-0	0.046	20	0.0016	mg/L J 1
Selenium	7782-49-2	0.10	1.0	0.023	mg/L JB 1
Silver	7440-22-4	0.00U	1.0	0.0038	mg/L U 1
Zinc	7440-66-6	0.10	40	0.012	mg/L JB 1

*



Certificate of Analytical Results 11733

Washington Savannah River Co., Aiken, SC

07079

Sample Id: 07079-4728-HTF		Matrix: SOLID		% Moisture:	
Lab Sample Id: 11733-008		Date Collected: Mar-15-07 09:12		Date Received: Mar-16-07 10:11	
Sample Depth:					
Analytical Method: TCLP Mercury by SW1311/7470A				Prep Method: SW7470A_DIG	
Date Analyzed: Apr-03-07 16:24		Analyst: MSN01		Date Prep: Apr-02-07 15:00	
		Seq Number: 35601		Tech: MSN01	
Parameter	Cas Number	Result	Rep Limit	MDL	Units Flag Dil
Mercury	7439-97-6	0.00U	0.0200	0.000860	mg/L U 1
Analytical Method: TCLP Metals by SW1311/6010B (33)				Prep Method: SW3010A	
Date Analyzed: Mar-30-07 15:33		Analyst: VHB01		Date Prep: Mar-29-07 13:00	
		Seq Number: 35559		Tech: MSN01	
Parameter	Cas Number	Result	Rep Limit	MDL	Units Flag Dil
Arsenic	7440-38-2	0.00U	1.0	0.0086	mg/L U 1
Barium	7440-39-3	0.26	1.0	0.0016	mg/L J 1
Cadmium	7440-43-9	0.00U	1.0	0.0022	mg/L U 1
Chromium	7440-47-3	0.00U	1.0	0.0040	mg/L U 1
Lead	7439-92-1	0.016	1.0	0.0060	mg/L J 1
Nickel	7440-02-0	0.00U	20	0.0016	mg/L U 1
Selenium	7782-49-2	0.14	1.0	0.023	mg/L JB 1
Silver	7440-22-4	0.00U	1.0	0.0038	mg/L U 1
Zinc	7440-66-6	0.00U	40	0.012	mg/L U 1

*



Sample Duplicate Recovery

Project Name: 07079

Work Order #: 11733

Report Date: 04/12/07 12:44

Lab Batch #: 35601

Project ID: 07079

QC- Sample ID: 11733-008 MD

Batch #: 1

Matrix: Sd

Reporting Units: mg/L

SAMPLE / SAMPLE DUPLICATE RECOVERY

TCLP Mercury by SW1311/7470A	Parent Sample Result [A]	Sample Duplicate Result [B]	RPD	Control Limits %RPD	Flag
Analyte					
Mercury	0.000	0.000	NC	20	

Lab Batch #: 35559

QC- Sample ID: 11733-006 MD

Batch #: 1

Matrix: Sd

Reporting Units: mg/L

SAMPLE / SAMPLE DUPLICATE RECOVERY

TCLP Metals by SW1311/6010B	Parent Sample Result [A]	Sample Duplicate Result [B]	RPD	Control Limits %RPD	Flag
Analyte					
Arsenic	0.000	0.000	NC	20	
Barium	0.34	0.34	1	20	
Cadmium	0.000	0.000	NC	20	
Chromium	0.012	0.013	8	20	
Lead	0.013	0.015	14	20	
Nickel	0.0050	0.0042	17	20	
Selenium	0.11	0.11	4	20	
Silver	0.000	0.000	NC	20	
Zinc	0.019	0.018	7	20	

Spike Relative Difference $RPD = 200 * |(B-A)/(B+A)|$

All Results are based on MDL and validated for QC purposes.

F = RPD exceeded the laboratory control limits



Form 3 - MS / MSD Recoveries

Project Name: 07079

Report Date: 04/12/07 12:44

Work Order #: 11733

Lab Batch ID: 35601

Reporting Units: mg/L

Project ID: 07079

QC-Sample ID: 11733-008 MS

Batch #: 1 Matrix: Sd

MATRIX SPIKE / MATRIX SPIKE DUPLICATE RECOVERY STUDY													
Reporting Units: mg/L	TCLP Mercury by SW1311/7470A	Analytes	Parent Sample Result [A]	Spike Added [B]	Spiked Sample Result [C]	Spiked Sample %R [D]	Spike Added [E]	Duplicate Spiked Sample Result [F]	Spiked Dup. %R [G]	RPD %	Control Limits %R	Control Limits %RPD	Flag
			0.000	0.0250	0.0256	102	0.0250	0.0252	101	1	85-115	20	
Mercury													

Lab Batch ID: 35559

Reporting Units: mg/L

QC-Sample ID: 11733-006 MS

Batch #: 1 Matrix: Sd

MATRIX SPIKE / MATRIX SPIKE DUPLICATE RECOVERY STUDY												
TCLP Metals by SW1311/6010B Analytes	Parent Sample Result [A]	Spike Added [B]	Spiked Sample Result [C]	Spiked Sample %R [D]	Spike Added [E]	Duplicate Spiked Sample Result [F]	Spiked Dup. %R [G]	RPD %	Control Limits %R	Control Limits %RPD	Flag	
	Arsenic	0.000	4.0	4.0	100	4.0	4.1	103	3	75-125	20	
	Barium	0.34	4.0	3.8	87	4.0	3.9	89	2	75-125	20	
	Cadmium	0.000	4.0	3.8	95	4.0	3.8	95	0	75-125	20	
	Chromium	0.012	4.0	3.6	90	4.0	3.7	92	2	75-125	20	
	Lead	0.013	4.0	3.8	95	4.0	3.8	95	0	75-125	20	
	Nickel	0.0050	4.0	3.8	95	4.0	3.9	97	2	75-125	20	
	Selenium	0.11	4.0	4.1	100	4.0	4.1	100	0	75-125	20	
	Silver	0.000	4.0	3.7	93	4.0	3.7	93	0	75-125	20	
	Zinc	0.019	4.0	3.9	97	4.0	4.0	100	3	75-125	20	



BS / BSD Recoveries

Project Name: 07079

Report Date 04/12/07 12:44

Project ID: 07079

Work Order #: 11733

Lab Batch ID: 35601

Sample: 302366 BKS

Batch #: 1

Matrix: Sd

Units: mg/L

Units: mg/L		BLANK /BLANK SPIKE / BLANK SPIKE DUPLICATE RECOVERY STUDY										
		Blank Sample Result [A]	Spike Added [B]	Blank Spike Result [C]	Blank Spike %R [D]	Spike Added [E]	Blank Spike Duplicate Result [F]	Blk. Spk Dup. %R [G]	RPD %	Control Limits %R	Control Limits %RPD	Flag
TCLP Mercury by SW1311/7470A												
Analytes												
Mercury		0.000	0.0250	0.0264	106	0.0250	0.0254	102	4	85-115	20	

Lab Batch ID: 35559

Sample: 302349 BKS

Batch #: 1

Matrix: Sd

Units: mg/L

Units: mg/L		BLANK /BLANK SPIKE / BLANK SPIKE DUPLICATE RECOVERY STUDY										
TCLP Metals by SW1311/6010B		Blank Sample Result [A]	Spike Added [B]	Blank Spike Result [C]	Blank Spike %R [D]	Spike Added [E]	Blank Spike Duplicate Result [F]	Blk. Spk Dup. %R [G]	RPD %	Control Limits %R	Control Limits %RPD	Flag
Analytes												
	Arsenic	0.000	4.0	4.0	100	4.0	4.0	100	0	75-125	20	
	Barium	0.0018	4.0	3.6	90	4.0	3.6	90	0	75-125	20	
	Cadmium	0.000	4.0	3.9	98	4.0	3.9	98	0	75-125	20	
	Chromium	0.000	4.0	3.6	90	4.0	3.6	90	0	75-125	20	
	Lead	0.000	4.0	3.8	95	4.0	3.8	95	0	75-125	20	
	Nickel	0.000	4.0	3.8	95	4.0	3.8	95	0	75-125	20	
	Selenium	0.078	4.0	4.1	103	4.0	4.1	103	0	75-125	20	
	Silver	0.000	4.0	3.7	93	4.0	3.7	93	0	75-125	20	
	Zinc	0.024	4.0	4.0	100	4.0	4.0	100	0	75-125	20	

Relative Percent Difference RPD = $200 * (D - G) / (D + G)$

Blank Spike Recovery [D] = $100 * (C) / [B]$

Blank Spike Duplicate Recovery [G] = $100 * (F) / [E]$

All results are based on MDL and Validated for QC Purposes

F = RPD exceeded the laboratory control limits