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**Fluidized Bed,**  
**Steam Reforming,**  
**monolith,**  
**geopolymers,**  
**cements,**  
**Hanford**

**Retention:**  
**Permanent**

**Evaluation of THOR™ Mineralized Waste Forms (Granular and Monolith) for the  
DOE Advanced Remediation Technologies (ART) Phase 2 Project**

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

DECEMBER 2011

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

**Prepared for the U.S. Department of Energy Under  
Contract Number DE-AC09-08SR22470**



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## EXECUTIVE SUMMARY

Fluidized Bed Steam Reforming (FBSR) processing of Hanford Low Activity Waste (LAW) and Waste Treatment Plant Secondary Waste (WTP-SW) simulants was performed in April/May 2008 by THOR Treatment Technologies LLC (TTT). The testing was performed at the Hazen Research Inc. (HRI) Engineering Scale Technology Demonstration (ESTD) pilot plant facilities in Golden, CO. FBSR products from these pilot tests on simulated waste representative of the Hanford LAW and the WTP-SW were subsequently transferred to the Savannah River National Laboratory (SRNL) for characterization, leach testing, and a monolith binder down selection. Initially, bed and fines samples consisting of four granular LAW as-received Product Receiver (PR) bed samples from the Denitration and Mineralization Reformer (DMR), four High Temperature Filter (HTF) fines, two granular WTP-SW as-received PR bed samples from the DMR and two HTF samples were received by SRNL. After the initial bed and fines samples had been received and testing commenced, SRNL received aggregate samples (blends of bed and fines that had been blended at HRI) of both LAW and WTP-SW. Only the aggregate blends were monolithed using a variety of different binders.

FBSR PR samples had been taken from the Product Receiver Tank, while the HTF samples were the fines collected as carryover from the DMR. The process operated with DMR fluidization gas rates set to maintain process and bed health. The amount of material elutriated to the HTF is dependent on the physical dimensions of the DMR, chemistry of the process, and fluidizing gas rate. These conditions contributed to a PR/HTF weight ratio that ranged between about 0.15 and 0.35.

The as-received PR and HTF samples were roasted in air to determine coal content by loss-on-ignition (LOI) at 525°C and to prepare the samples for chemical composition, crystalline analysis, bulk density and durability testing. Characterization of the initial bed and fines crystalline powder samples indicated they were primarily Al, Na and Si, with < 1wt% Fe, K and S present. The PR samples contained less than 2.1 wt% carbon with a nominal bulk density of ~ 1 g/cc, and the carbon content of the HTF samples ranged from 8.6 to 13 wt% carbon with nominal bulk density of 0.65 g/cc.

Crystalline phases observed in the aggregate and blends from the Hazen ESTD testing showed two forms of NaAlSiO<sub>4</sub> (Low-Carnegieite and Nepheline), Nosean (a sulfate containing sodalite) and a halide containing sodalite similar to previous FBSR testing results.

The as-received PR and HTF samples were roasted in air to determine coal content by LOI at 525°C and to prepare the samples for durability testing. Durability testing of the PR and HTF samples using the American Society for Testing and Materials (ASTM) C1285-08 Product Consistency Test (PCT) 7-day leach test at 90°C was performed along with several reference glass samples. Normalized releases from the PR and HTF samples were all less than 0.08 and 0.14 g/m<sup>2</sup>, respectively, with NL<sub>S</sub> giving the highest of the measured elements.

Measured leachate values were normalized using Brunauer-Emmett-Teller – Surface Area (BET SAs) that measured in the range of 3.8 to 5.5 m<sup>2</sup>/g for the 100-200 mesh fractions obtained from the FBSR bed and fines. Note that BET measurements were performed on a

PCT prepared sub-specimen so that the surface area (SA) of the mineral product and not the coal can be used during release calculations.

A blend of the PR and HTF samples from the Production Run P-1B LAW testing were also leach tested with the coal in the sample. The mineral/coal aggregates were used for the monolith studies and down selection criteria amongst binders were based on normalized elemental PCT release with emphasis given to  $NL_{Re}$  as a surrogate for Tc-99, compression testing, and Toxicity Characteristic Leach Procedure (TCLP) performance. The aggregates (blends) contained 1.72 wt% and 11.06 wt% residual carbon, respectively, for the LAW (P-1B) and WTP-SW (P-2B). The TCLP tests performed on the aggregates (blends) indicate that these products met the criteria for the EPA RCRA Universal Treatment Standards (UTS) for all of the constituents except for Cd (LAW) and Sb (both LAW and WTP-SW). However, these elements along with other toxic metals (Ba, Se and Tl) had been added to the LAW FBSR feed simulant at  $\sim 10$ -1,000X the expected concentration to allow for adequate detection in the off-gas during process demonstrations. Therefore, performance at actual waste concentrations is not known.

Monolith studies were initially performed on P-1B LAW aggregate (blend) to make 2" cubes using ordinary Portland cement, high aluminum cements, ceramicrete, a variety of geopolymers and Nu-Cap binder. Based on compression data, PCT and TCLP performance, four monolith recipes from the 2" LAW cubes (two different high aluminum cements, a clay-based geopolymer and a fly ash-based geopolymer) were carried forward into both 3" x 6" cylinders and 6" x 12" cylinders that were made from both LAW and WTP-SW. Curing temperatures were measured for the 6" x 12" cylinders using centerline thermocouples. In addition, 3" x 6" cylinders of a geopolymeric cement (L-TEM) were provided by TTT for comparative testing.

PCT durability testing of the best candidate monolith forms showed average normalized release values for the seven elements measured (Al, Cs, I, Na, Re, S and Si) below  $0.04 \text{ g/m}^2$ . Statistical analysis of the data indicate that the 95% confidence level was less than or equal to  $\sim 0.1 \text{ g/m}^2$ .

Finally, the single geopolymer formulation containing fly ash (GEO-7) that had been optimized for the P-1B LAW aggregate (blend) was chosen to produce 24 replicate WTP-SW P-2B aggregate (blend) monoliths, e.g. 2" x 4" cylinders. The GEO-7 formulation was used for the WTP-SW P-2B blend (3" x 6", 6" x 12" and 2" x 4" cylinders) without reformulation. Considering that the WTP-SW wastes had considerable fluoride over what was present in the LAW P-1B they performed relatively well in durability testing and passed TCLP for all elements.

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## LIST OF ACRONYMS

ANL	Argonne National Laboratory
ARM-1	Approved Reference Material - 1
ART	Advanced Remediation Technologies
ASTM	American Society for Testing and Materials
CEES	Columbia Energy and Environmental Services
COC	Constituents of Concern
DMR	Denitration and Mineralization Reformer
DOE	Department of Energy
ESTD	Engineering Scale Technology Demonstration
BET-SA	Brunauer-Emmett-Teller – Surface Area
EA	Environmental Assessment
EPA	Environmental Protection Agency
ESTD	Engineering Scale Test Demonstration
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
IDF	Integrated Disposal Facility
IOC	Iron Oxide Catalyst
LAW	Low Activity Waste
LDRD	Laboratory Directed Research & Development
LOI	Loss-on-Ignition at 525°C
LOD	Loss-on-Drying at 110°C
LRM	Low-activity Reference Material
MDL	Method Detection Limit
NAS	Sodium AluminoSilicates
OPC	Ordinary Portland Cement
ORP	Office of River Protection
PCT	Product Consistency Testing
PR	Product Receiver (material from DMR bed)
RCRA	Resource Conservation and Recovery Act
RL	Reporting Limit
RPP	River Protection Project
REDOX	REDuction/OXidation
SA	Surface Area
SA/V	Surface Area to Volume
SRNL	Savannah River National Laboratory
SRNS	Savannah River Nuclear Solutions
SRS	Savannah River Site
TC	Thermocouple
TCLP	Toxicity Characteristic Leaching Procedure
TPA	Tri-Party Agreement
TTT	THOR Treatment Technologies, LLC

URS	United Research Services
UTS	Universal Treatment Standards
VSL	Vitreous State Laboratory
WFO	Work for Others
WTP	Waste Treatment and Immobilization Plant
WTP-SW	Waste Treatment and Immobilization Plant – Secondary Waste
XRD	X-ray Diffraction

## 1.0 BACKGROUND AND OPERATIONAL SUMMARY

The U.S. Department of Energy's (DOE) Office of River Protection (ORP) is responsible for the retrieval, treatment, immobilization, and disposal of Hanford's tank waste. Currently there are approximately 56 million gallons of highly radioactive mixed wastes awaiting treatment. A key aspect of the River Protection Project (RPP) cleanup mission is to construct and operate the Waste Treatment and Immobilization Plant (WTP). The WTP will separate the tank waste into high-level and low-activity waste (LAW) fractions, both of which will subsequently be vitrified.

The projected throughput capacity of the WTP LAW Vitrification Facility is insufficient to complete the RPP mission in the time frame required by the Hanford Federal Facility Agreement and Consent Order, also known as the Tri-Party Agreement (TPA), i.e. December 31, 2047. Therefore, Supplemental Treatment is required both to meet the TPA treatment requirements as well as to more cost effectively complete the tank waste treatment mission. The Supplemental Treatment chosen will immobilize that portion of the retrieved LAW that is not sent to the WTP's LAW Vitrification facility into a solidified waste form. The solidified waste will then be disposed on the Hanford site in the Integrated Disposal Facility (IDF). In addition, the WTP LAW Vitrification facility off-gas condensate known as WTP Secondary Waste (WTP-SW) will be generated and enriched in volatile components such as Cs-137, I-129, Tc-99, Cl, F, and SO<sub>4</sub> that volatilize at the vitrification temperature of 1150°C in the absence of a continuous cold cap. The current waste disposal path for the WTP-SW is to recycle it to the supplemental LAW treatment to avoid a large steady state accumulation in the pretreatment-vitrification loop.

Fluidized Bed Steam Reforming (FBSR) offers a moderate temperature (700-750°C) continuous method by which LAW and/or WTP-SW wastes can be processed irrespective of whether they contain organics, nitrates, sulfates/sulfides, chlorides, fluorides, volatile radionuclides or other aqueous components. The FBSR technology can process these wastes into a crystalline ceramic (mineral) waste form. The mineral waste form that is produced by co-processing waste with kaolin clay in an FBSR process has been shown to be as durable as LAW glass. Monolithing of the granular FBSR product, which is one of the objectives of this current study, is being investigated to prevent dispersion during transport or burial/storage but is not necessary for performance.

FBSR testing of a Hanford LAW simulant and a WTP-SW simulant at the pilot scale was performed by THOR Treatment Technologies, LLC at Hazen Research Inc. in April/May 2008.<sup>1</sup> The Hanford LAW simulant was the Rassat<sup>2</sup> 68 tank blend and the target concentrations for the LAW was increased by a factor of 10 for Sb, As, Ag, Cd, and Tl; 100 for Ba and Re (Tc surrogate); 1,000 for I; and 254,902 for Cs based on discussions with the DOE field office and the environmental regulators and an evaluation of the Hanford Tank Waste Envelopes A, B, and C.<sup>3</sup> It was determined through the evaluation of the actual tank waste metals concentrations that some metal levels were not sufficient to achieve reliable detection in the off-gas sampling.<sup>1</sup> Therefore, the identified metals concentrations were increased in the Rassat simulant processed by TTT at HRI to ensure detection and enable calculation of system removal efficiencies, product retention efficiencies, and mass balance closure without regard to potential results of those determinations or impacts on product durability response such as Toxicity Characteristic Leach Procedure (TCLP).<sup>1</sup>

A WTP-SW simulant based on melter off-gas analyses from Vitreous State Laboratory (VSL) was also tested at HRI in the 15" diameter Engineering Scale Test Demonstration (ESTD) dual reformer at HRI in 2008.<sup>1</sup> The target concentrations for the Resource Conservation and Recovery Act (RCRA) metals were increased by 16X for Se, 29X for Tl, 42X for Ba, 48X for Sb, by 100X for Pb and Ni, 1000X for Ag, and 1297X for Cd to ensure detection by the analytic laboratory used for the demonstration and, as with the LAW simulant, without regard to potential impacts on product durability response such as TCLP.

Details of the ESTD testing that was performed in April and May of 2008 are shown in Table 1 as the Operational Summary. The first eight tests were performed on LAW using a Denitration and Mineralization Reformer (DMR) temperature of 725 °C and different ratios of clay:simulant of 675 g or 640 g of OptiKast clay per liter of simulant. The last four tests were performed on WTP-SW (originally referred to as LAW-Recycle) using a DMR temperature in the range of 680 °C to 700 °C with a mixture of 45/55 Sagger/OptiKast clay with 307 g clay/L simulant. The coal feed rates used in the FBSR processing are also shown as noted at the bottom of Table 1. It should be noted that the carbon feed rates are averaged over those periods. Carbon feed rates fluctuated as process conditions dictated, i.e., to maintain H<sub>2</sub> levels. An iron oxide catalyst (IOC) used as a denitration catalyst, was also added as Cr reductant in both the LAW and WTP-SW processing as noted at the bottom of Table 1. The Product Receiver (PR) and High Temperature Filter (HTF) bed products and fines were received by SRNL in June of 2008. Photographs of the mineralized products are shown in Appendix 1. Visible pieces of dark-colored coal residues are apparent in the PR samples.

Simulants used in the ESTD testing are detailed in Table 2. The major components common to both the LAW and WTP-SW feeds are Na, Al, Si, NO<sub>3</sub>/NO<sub>2</sub>, CO<sub>3</sub> and OH. The WTP-SW is higher in F and Cl and lower in SO<sub>4</sub> and PO<sub>4</sub> versus the LAW. The components B, NH<sub>4</sub> and Zn are only in the WTP-SW and it does not contain any organics (C<sub>2</sub>O<sub>4</sub>, CH<sub>3</sub>COO) that are in the LAW. The WTP-SW contains more 'trace' components than the LAW, e.g., 10 trace components present in the WTP-SW at < 0.2 g/L versus only 4 in the LAW present at < 0.2 g/L. A schematic of the SRNL testing 'modules' performed on the ESTD products is shown in Figure 1.

**Table 1. ESTD Operational Summary**

Sample Description	Sample Log #	Date/Time Sample Obtained	Test*	Hours of Operation at Test Conditions before Sample Obtained	DMR Temp.	Simulant	g Moist Clay per Liter Simulant	Clay Type	SO <sub>4</sub> Conc. ( g/L )	Expected Al:Si Mole Ratio
HTF	5280	4/27/2008 21:42	P-1A	17.7	725°C	LAW	675	OptiKast	8.65	1.02
PR	5274	4/28/2008 5:15		25.3						
HTF	5297	4/28/2008 17:28		37.5						
PR	5316	4/29/2008 3:54		47.9						
HTF	5351	4/30/2008 12:00	P-1B	28			640			
HTF	5357	4/30/2008 19:44		35.7						
PR	5359	4/30/2008 22:55		38.9						
PR	5372	5/1/2008 7:00		47						
HTF	5471	5/5/2008 0:20	P-2A	70.3	680°C	WTP-SW	307	45% Sagger/ 55% OptiKast	0.53	1.02
PR	5475	5/5/2008 4:00		74						
HTF	5520	5/6/2008 10:00	P-2B	26.5	700°C					
PR	5522	5/6/2008 10:00		26.5						

\* Coal feed rates for P-1A, P-1B, P-2A, P-2B were 36.7, 32.8, 35.6, 44 lbs/hr, respectively.

\* Total of 105 lbs Fe added (as IOC) during LAW processing, and 107 lbs Fe (as IOC) added during WTP-SW processing.

Table 2. Simulant Compositions

Component	LAW Target Component Concentration [g/L]	LAW Target Component Concentration [mg/L]	LAW Target Component Concentration with 675 g clay/L [g/L]	LAW Target Component Concentration with 640 g clay/L [g/L]	WTP-SW Target Component Concentration [g/L]	WTP-SW Target Component Concentration [mg/L]	WTP-SW Target Component Concentration with 307 g clay/L [g/L]
C <sub>2</sub> O <sub>4</sub>	1.039	1039	0.829	0.838	-	-	-
CH <sub>3</sub> COO	7.794	7794	6.221	6.287	-	-	-
Ag	0.174	174	0.139	0.14	0.092	92	0.083
Al	1.719	1719	102.744	98.519	14.782	14782	61.951
As	0.103	103	0.986	0.949	0.008	8	0.007
B	-	-	-	-	1.432	1432	1.284
Ba	1.031	1031	1.384	1.37	0.003	3	0.003
Ca			0.133	0.127	-	-	0.147
Cd	0.472	472	0.377	0.381	0.098	98	0.088
Cl	1.553	1553	1.239	1.253	3.758	3758	3.37
CO <sub>3</sub>	28.504	28504	22.752	22.993	12.012	12012	10.773
Cr	0.541	541	0.432	0.436	0.315	315	0.282
Cs	1.728	1728	1.379	1.394	1.952	1952	1.751
F	0.6	600	0.479	0.484	4.155	4155	3.727
Fe	-	-	1.948	1.867	-	-	1.261
I	1.65	1650	1.317	1.331	0.1	100	0.09
K	0.485	485	0.387	0.391	0.409	409	0.492
Mg	-	-	0.081	0.078	-	-	0.134
Na	115.319	115319	92.048	93.022	61.334	61334	55.715
NH <sub>4</sub>	-	-	-	-	5.272	5272	4.728
Ni	0.622	622	0.497	0.502	0.269	269	0.241
NO <sub>2</sub>	19.506	19506	15.57	15.735	1.675	1675	1.502
NO <sub>3</sub>	160.275	160275	127.932	129.285	123.447	123447	110.716
OH	12.585	12585	10.046	10.152	26.593	26593	23.85
Pb	1.256	1256	1.002	1.013	0.272	272	0.244
PO <sub>4</sub>	4.673	4673	4.188	4.208	0.699	699	0.755
Re	0.317	317	0.253	0.255	0.211	211	0.189
Sb	0.528	528	0.422	0.426	0.195	195	0.175
Se	0.097	97	0.078	0.078	0.195	195	0.175
Si	-	-	105.902	101.473	0.506	506	61.429
SO <sub>4</sub>	8.646	8646	6.901	6.974	0.525	525	0.471
Ti	-	-	5.07	4.858	-	-	1.884
Tl	0.413	413	0.33	0.333	0.195	195	0.175
Zn	-	-	-	-	0.477	477	0.428



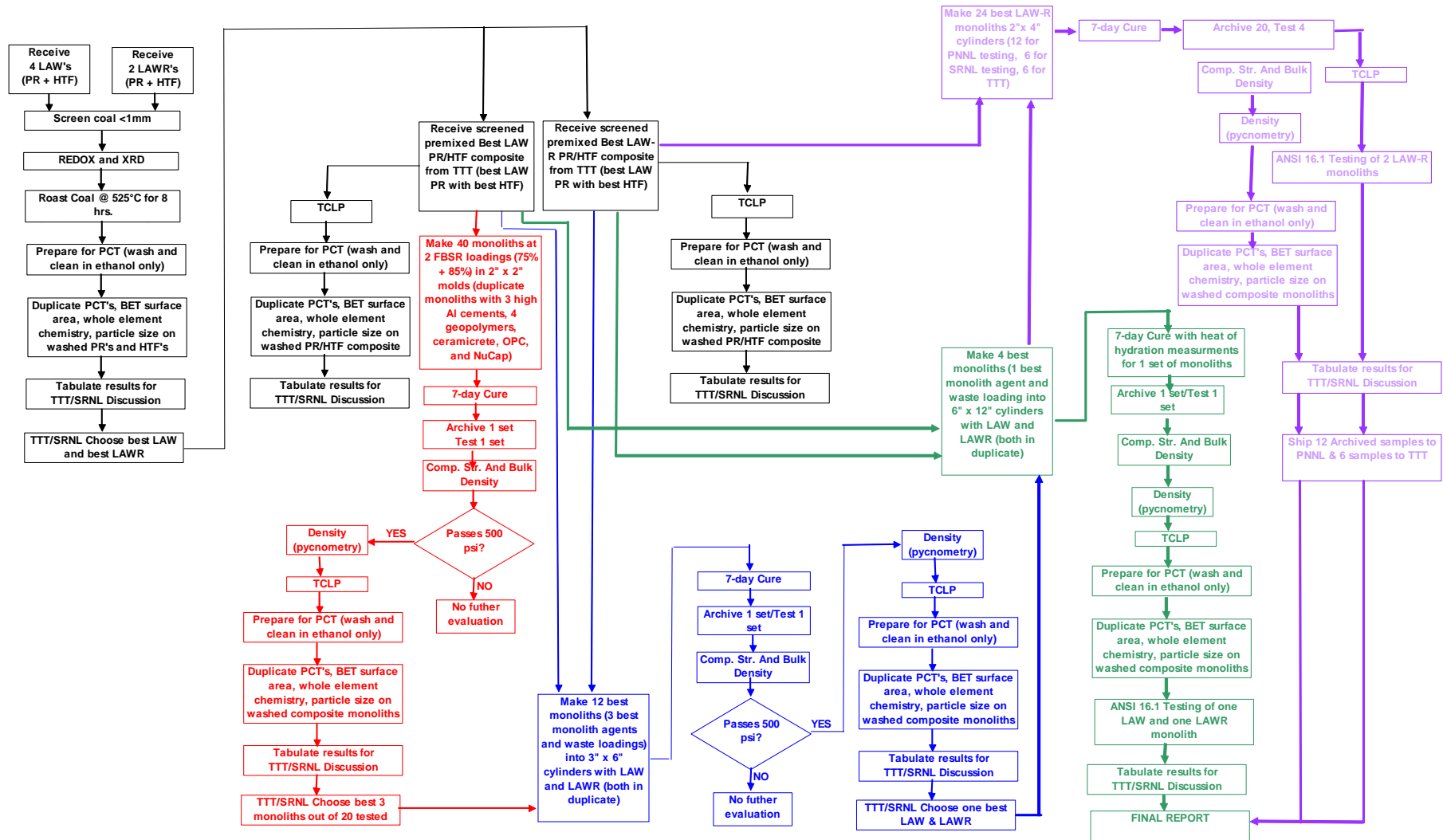


Figure 1. Flow chart of monolith down selection for Hanford LAW

The modules depicted in Figure 1 are listed below along with key objectives in the chronological order performed, followed by a detailed description of each testing phase.

- Module 1 (black text in Figure 1)  
Granular LAW and WTP-SW product bed and fines durability tested with PCT with coal roasted from the product. Granular LAW and WTP-SW blend/aggregates durability tested with PCT with coal present. Note: subsequent durability testing in 2010/2011 allowed for determination of roasted blend/aggregate data for comparison to the roasted product bed and fines data.  
▶ The initial objective of this module was to compare the individual bed products and fines PCT data to select the best LAW and WTP-SW production run from the ESTD testing. Blends from the best production run were then premixed at Hazen and sent to SRNL for further testing (latter phase of Module 1).
- Module 2 (red text in Figure 1)  
Granular LAW product with coal is monolithed in 2" cubes (8 binders and 2 FBSR loadings of 75 wt% and 85 wt%)  
▶ The objective of this module was to down-select three different monolith recipes to carry forward into scale-up monolith testing. A fourth monolith recipe using fly ash in geopolymers was also included in the downselect.
- Module 3 (blue text in Figure 1)  
Granular LAW product with coal is monolithed in 3" x 6" cylinders (3 best binders and FBSR loadings for LAW and WTP-SW from Module 2 testing)  
▶ The objective of this module was to compare performance data from the scaled up 3" x 6" cylinders versus the previous best 2" cubes. The original intent was to use only the three best monolith recipes, but the fly ash geopolymers was also included.
- Module 4 (green text in Figure 1)  
Granular product with coal is monolithed in 6" x 12" cylinders (best binder and FBSR loading for LAW and WTP-SW from Module 3 testing)  
▶ The objective of this module was to compare performance data from the scaled up 6" x 12" cylinders versus the previous 2" cubes and 3" x 6" cylinders. Another objective of this module was to obtain centerline curing temperatures from the monoliths.
- Module 5 (purple text in Figure 1)  
WTP-SW granular product with coal is monolithed in 2" x 4" cylinders (best binder and FBSR loading from Modules 3 and 4 testing). Twenty four (24) replicate samples are made of which 12 were shipped to Pacific Northwest National Laboratory (PNNL) for subsequent testing.  
▶ The Objective of this module was to produce a large replicate set of 2" x 4" cylinders made with WTP-SW using the fly ash geopolymers recipe that was judged as one of the best performers (PCT, TCLP and compression testing) from previous modules.

## 1.1 CHARACTERIZATION OF BED AND FINES SAMPLES (INITIAL PART OF MODULE 1)

A total of six as-received PR samples and six HTF samples were received by SRNL. The scope of work to be performed on the FBSR bed and fines samples included whole element chemical characterization and performance testing using ASTM C1285 PCT durability test. Samples were roasted to 525 °C before both PCT and chemical composition determination. The crystal structure was measured by XRD and residual coal was measured by Loss on Ignition at 525 °C. The REDuction/OXidation (REDOX) equilibria, as expressed by the  $\text{Fe}^{2+}/\Sigma\text{Fe}$ , was also measured.

The PR samples were taken from the “active” DMR bed. Bed material is augured out of the DMR from the defluidized area in the bottom. Then it is pneumatically transferred to the PR. It is collected from the PR into stainless steel “milk” cans, weighed, and sampled. The HTF fines are the fines collected downstream of the DMR. The process operated with DMR fluidization gas rates set to maintain process and bed health. The amount of material elutriated to the HTF is dependent on the physical dimensions of the DMR, chemistry of the process, and fluidizing gas rate. These conditions contributed to a PR/HTF weight ratio that ranged between about 0.15 and 0.35.

The main objective of this initial part of the Module 1 testing was to decide which production run (P-1A or P-1B from LAW testing, or P-2A or P-2B from WTP-SW testing) would be used for further testing with blended aggregates and latter monolith testing. This decision was based on durability results from the PCT.

## 1.2 CHARACTERIZATION OF AGGREGATES (BLENDS OF PR AND HTF) – LATTER PART OF MODULE 1

Table 3 shows the blend ratios of the mixed PR and HTF blends that were sent to SRNL from the HRI testing. The initial 7-kg bags of LAW P-1A and LAW P-1B were sent in early July 2008 for scope testing with monolith development. Later in July 2008 after it was decided to use the P-1B blend for all monolithing, five separate ~ 5-gallon buckets of LAW P-1B blend were received. The blend ratio of these 5-gallon bucket samples were slightly different, but similar to the initial LAW P-1B blend received in early July, 2008. A 55-gallon drum of the WTP-SW P-2B material was received later in September of 2008. A final 6<sup>th</sup> bucket of LAW P-1B was also shipped in late October of 2008 to provide enough material to complete testing at the larger monolith scale.

Appendix 2 shows photographs of the LAW P-1B and the WTP-SW P-2B blends received at SRNL. All of these blends produced at HRI had been screened to < 1mm before shipment to SRNL.

All of the testing initially performed on the bed and fines samples were repeated with the LAW P-1B and WTP-SW P-2B blends. The blended aggregate samples were not roasted prior to testing. Blended aggregate samples were also tested by TCLP to provide RCRA metal leaching data that could later be compared to similar TCLP testing on the monoliths made with the blended aggregate minerals.

**Table 3. Blend Ratios Sent to SRNL from Hazen ESTD Testing**

SAMPLE	DATES	BLEND
7kg, LAW, P-1A	7/3/08	123 lb HTF; 27 lb PR total with PR = 23.6 lb (-)10mesh and 3.4 lb (+) 10mesh $PR/(PR+HTF) \text{ ratio} = 27/(123+27) = 0.18$ $PR/HTF \text{ ratio} = 27/123 = 0.22$ $wt\% \text{ PR} = 23.6/(123+23.6) * 100 = 16.1 \text{ wt}\%$ $wt\% \text{ HTF} = 123/(123 + 23.6) = 83.9 \text{ wt}\%$
7kg, LAW, P-1B	7/3/08	112.5 lb HTF; 37.5 lb PR total with PR = 34.7 lb (-)10mesh and 2.8 lb (+) 10mesh $PR/(PR+HTF) \text{ ratio} = 37.5/(112.5+37.5) = 0.25$ $PR/HTF \text{ ratio} = 37.5/112.5 = 0.31$ $wt\% \text{ PR} = 34.7/(112.5+34.7) * 100 = 23.6 \text{ wt}\%$ $wt\% \text{ HTF} = 112.5/(112.5 + 34.7) = 76.4 \text{ wt}\%$
5 buckets LAW P-1B	7/22/08	20 lb HTF; 5 lb PR all screened to < 1 mm $PR/(PR+HTF) \text{ ratio} = 5/(20 + 5) = 0.20$ $PR/HTF \text{ ratio} = 5/20 = 0.25$ $wt\% \text{ PR} = 5/(20+5) * 100 = 20 \text{ wt}\%$ $wt\% \text{ HTF} = 20/(20+5) = 80 \text{ wt}\%$
1 drum WTP-SW, P-2B	9/24//08	100.5 lb HTF; 49.5 lb. PR total with PR = 45.5 lb (-)10mesh and 4 lb (+) 10mesh all screened to < 1 mm $PR/(PR+HTF) \text{ ratio} = 49.5/(100.5 + 49.5) = 0.33$ $PR/HTF \text{ ratio} = 49.5/100.5 = 0.49$ $wt\% \text{ PR} = 45.5/(100.5 + 45.5) * 100 = 31.2 \text{ wt}\%$ $wt\% \text{ HTF} = 100.5/(100.5 + 45.5) = 68.8 \text{ wt}\%$
1 extra bucket LAW P-1B (6 <sup>th</sup> bucket)	10/30/08	20 lb HTF; 5 lb PR all screened to < 1 mm $PR/(PR+HTF) \text{ ratio} = 5/(20 + 5) = 0.20$ $PR/HTF \text{ ratio} = 5/20 = 0.25$ $wt\% \text{ PR} = 5/(20+5) * 100 = 20 \text{ wt}\%$ $wt\% \text{ HTF} = 20/(20+5) = 80 \text{ wt}\%$

### 1.3 MONOLITH DOWN-SELECTION – MODULE 2 TESTING

Testing and characterization of a minimum of eight monolithic media chosen by agreement between TTT and SRNL were fabricated using FBSR target dry basis loadings of >70 wt%. The initial monoliths were only 2” cubes. Bulk density, skeletal density, durability using ASTM C1285, compressive strength, and Environmental Protection Agency (EPA) TCLP were performed (these test methods are described below in detail in the Experimental Section of this report). The EPA-TCLP testing was subcontracted to an EPA certified laboratory. The whole element chemistry of the granular products and the monoliths were all measured, including the REDuction/OXidation (REDOX) equilibria, so that the durability response of the granular and monolithic specimens could be compared.

#### **1.4 LARGER SCALE MONOLITH TESTING MODULES 3 - 5**

Once the best monolithing agents and waste loadings were agreed upon by SRNL and TTT, scale up was demonstrated by SRNL (3" x 6" cylinders and 6" x 12" cylinders) to complete Modules 3 and 4 per Figure 1. The scaled up products were also durability tested using ASTM C1285 and compared to the smaller scale tests and to the durability of the granular product. A further test objective for Module 4 was to obtain centerline curing temperature data for the 6" x 12" monoliths produced in this program.

Overall project testing was performed in chronological order of the 5 modules as shown by the colors in Figure 1. Module 1 (black text) preceded Module 2 (red text). Then Module 3 (blue text) was performed followed by Module 4 (green text). Module 5 (purple text) was the final testing phase.

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## 2.0 QUALITY ASSURANCE

The waste form testing and monolith fabrication conducted at the Savannah River National Laboratory (SRNL) was conducted in accordance with the ASME NQA-1 based quality assurance program. Monolith formulations are readily available in the open literature and the literature referred to is cited in the body of this report. The L-TEM geopolymeric cements were fabricated by Columbia Energy and Environmental Services (CEES) and tested at SRNL. The Global Matriarchs NuCap™ polymer was formulated at SRNL by a representative of Global Matriarchs. The scope of work is outlined in the Work for Others (WFO) documentation<sup>4</sup> and in the Technical Task and Quality Assurance Plan (TTQAP), SRNL-PSE-2008-00147.<sup>5</sup> The data is recorded in the following notebook: WSRC-NB-2008-00070. A preliminary summary of the SRNL work has been previously published in the 2010 ACS Symposium Series 1046.<sup>6</sup> This current 2011 technical report serves to report a more detailed and up to date interpretation of the data obtained over the course of this project.

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### 3.0 EXPERIMENTAL

#### 3.1 CHARACTERIZATION

Elemental and anion compositions of the steam reforming aggregate PR and HTF materials were measured for as received samples after heating of the samples at 525 °C in air overnight in a muffle furnace. The <200 mesh (<74 µm) PCT-prepared powders were used for the dissolutions in all testing (aggregate and all various monoliths). Elemental and anion analyses were performed on lithium tetraborate fusion (1000 °C open vessel) and sodium peroxide fusion (650 °C open vessel), respectively. These methods used nominally 0.1 g of powder solid sample to 0.1 L of dissolved solution, and 0.15 g of powder solid to 0.250 L of dissolved solution, respectively. The methods have been described in detail previously (Pareizs et al., 2005).<sup>7</sup> The digestion methods for elemental (cation) analysis involve the use of acids for dissolution. A KOH fusion method with water uptake was used for sample dissolution to obtain anion analyses using typically 0.5 g of solid powder to 0.05 L of dissolved solution. All elemental cation concentrations (except for I, Re and Cs) were determined by Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP-AES). The Re, Cs and I were measured by Inductively Coupled Plasma Mass Spectroscopy (ICP-MS). Anions were measured by Ion Chromatography. After all data was gathered for this report it was discovered that the peroxide fusion method performed at lower temperature than the lithium tetraborate fusion method gives better recovery for Re. Accordingly, all data shown in this report gives Re on a peroxide fusion analysis basis. Also, it has been determined in more recent BSR mineral dissolution testing that sealed vessel, i.e., aqua regia method, dissolutions provide better recovery of sulfur versus the open vessel digestion methods used in this study.<sup>8</sup> Accordingly, it is expected that the sulfur values reported in this work are likely low bias, which would result in potentially high biased normalized release calculations.

Table 4 shows the nominal instrument detection limits (IDLs) for the various analyses and the solid to liquid ratios for the various dissolution methods (PerFus = sodium peroxide fusion; Li-TetraBorate = Lithium Tetraborate fusion; KOH = potassium hydroxide fusion). The reported method uncertainty for the ICP-AES and IC-Anion is +/- 10%, while the reported method uncertainty for the ICP-MS is +/- 20%. These data shows that the lowest detection level for cations in the FBSR solids were in the range of 0.01 to 0.017 wt% for ICP-AES. Most of the ICP-MS data was obtained on instrumentation using nominal IDLs of 1 – 10 ppb, which give expected Cs, Re and I detection limits in the range of 0.0001 to 0.001 wt%. The last 2” x 4” monolith samples were analyzed with ICP-MS with lower IDLs of ~ 0.05 ppb giving a detection limit of ~ 0.00001 wt%. The IC-anion method had a higher IDL of ~ 1,000 ppb (1 mg/L) or about 0.04 wt% in the dissolved solids.

**Table 4. Nominal Instrument Detection Limits**

Analytical Method	IDL ug/L (ppb)	Digestion Method	Elements Measured	Ratio gram:Liter	Detection Level (wt%)
ICP-AES	100	PerFus	Cations (no Na)	0.15/0.25	0.017
		Li-TetraBorate	Cations (no Li, B)	0.1/0.1	0.010
ICP-MS	1 - 10	PerFus	Cs, Re	0.15/0.25	0.00017 – 0.002
		Li-TetraBorate		0.1/0.1	0.00010 – 0.001
	0.05	PerFus		0.15/0.25	0.00001
		Li-TetraBorate		0.1/0.1	0.00001
IC-Anion	1,000	KOH	Anions	0.25/0.1	0.040
ICP-MS - Iodide	1	KOH	Iodide	0.25/0.1	4E-05

REDOX (iron (II) to total iron ratio) was determined on samples that were not subjected to carbon removal in air, using a dissolution and absorption spectroscopy method.<sup>9</sup> 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 PCT ASTM procedure C 1285-08 [ASTM 2008].<sup>10</sup> Prior to sizing and washing the sample, carbon was removed from the aggregate PR and HTF material by heating overnight at 525°C. The PR and HTF product samples were sized between (-) 100 and (+) 200 mesh ( $< 149 \mu\text{m}$  and  $> 74 \mu\text{m}$ ), which is the same size fraction used to express glass waste form performance. The sized material was washed six times (2 with rinse/decant, and 4 with rinse/sonication/decant) with 100% ethanol to remove electrostatic fines, followed by overnight drying in an oven at 90°C. Water was not used for washing to avoid removing water soluble phases prior to leaching as cautioned by the ASTM C1285-08 procedure. No analyses were performed on the ethanol rinses to determine if any elements contained in the FBSR minerals or monoliths were solubilized in the rinse ethanol since most phases are not soluble in ethanol but may be soluble in water. Portions of the washed and dried PR and HTF powders were analyzed using Microtrac – S3000 instrumentation for particle size analysis by laser light scattering. Brunauer-Emmett-Teller – Surface Area BET-SA surface area measurements via nitrogen gas adsorption was also performed on the sieved/washed/dried portions of the powders used for PCT. In this study the initial bed and fines samples were pre-roasted before any PCT preparation. Thus the BET surface areas for these samples were from roasted samples.

For all other materials (blended aggregates and all crushed monoliths) the PCT preparations were performed on non-roasted samples, i.e., samples that contained residual FBSR coal. Thus the BET surface area for the blended aggregates and monoliths are on a non-roasted basis. The BET SA determinations on these residual coal-containing samples were often difficult and time-consuming due to the BET method requirement to obtain a stable vacuum signal before the actual gas adsorption is performed. In many cases the PCT prepared powders that contained residual coal were difficult to degas. The degassing temperature was increased numerous times to try to remove the residual coal from the powders. The starting temperature for the degassing step was between 100 - 105°C, and was increased by 50°C to a maximum temperature of 300°C. Even if the powder samples passed the degassing step at lower temperature, some of the powders failed in the evacuation steps prior to measuring the surface area. Typically, a powder sample that contained residual coal, i.e., all the aggregate blend PCT prepared powders and all the monolith PCT prepared powders, would take several attempts with each attempt corresponding to a day.

Both a low and high surface area standard were typically run in parallel for the BET surface area instrument. The low SA standard used NIST-traceable silicon nitride powder with a certified  $2.05 \pm 0.09 \text{ m}^2/\text{g}$  SA. The high SA standard used silica – alumina from Micromeritics with a certified  $214 \pm 6 \text{ m}^2/\text{g}$  SA. These uncertainties in the BET SA of the standards are about 3% (%RSD), which is similar to previously reported uncertainties in the BET SA measurements of primary silicate minerals of about  $\pm 5\%$  for measured surface areas  $> 0.1 \text{ m}^2/\text{g}$ .<sup>11</sup> For all samples, ASTM Type I water was used as the leachant, a constant leachate to sample ratio of  $10 \text{ cm}^3/\text{g}$  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. In this program the original aggregate PR and HTF powders were all heat-treated to remove carbon before performing the PCT. All subsequent PCTs

(blended aggregates and monoliths) were performed on materials that did not receive any heat-treatment, i.e., residual FBSR carbon material remained in the samples that were chemically analyzed and leach-tested with PCT.

The PCT results can be expressed per ASTM C-1285-08 as a normalized concentration ( $NC_i$ ) which have units of  $g_{\text{waste form}}/L_{\text{leachant}}$ , or as a normalized release ( $NL_i$ ) in  $g_{\text{waste form}}/m^2$ . Examples of the calculations are given in Equations 1 and 2.

$$NC_i = \frac{c_i(\text{sample})}{f_i} \quad (\text{Equation 1})$$

where  $NC_i$  = normalized concentration ( $g_{\text{waste form}}/L_{\text{leachant}}$ )  
 $c_i(\text{sample})$  = concentration of element "i" in the solution ( $g_i/L$ )  
 $f_i$  = fraction of element "i" in the unleached waste form

$$NL_i = \frac{c_i(\text{sample})}{(f_i) \bullet (SA/V)} \quad (\text{Equation 2})$$

where  $NL_i$  = normalized release ( $g_{\text{waste form}}/m^2$ )  
 $c_i(\text{sample})$  = concentration of element "i" in the solution ( $g_i/L$ )  
 $f_i$  = fraction of element "i" in the unleached waste form  
 $SA/V$  = surface area of the waste form divided by the leachate volume in  $m^2/L$

Due to the high surface roughness of ceramic/mineral waste forms such as FBSR the method for  $SA/V$  determination for this work 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 of known mass 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/ gram powder, by the leachant volume via Equation 3.

$$SA/V_{\text{BET}} = (SA_{\text{BET}} / \text{gram sample}) / (\text{gram sample}/V) \quad (\text{Equation 3})$$

Due to the presence of coal in the sample, several adjustments have to be made in these equations to express the leaching of a particular element on a coal free basis as described in Pareizs et al.<sup>7</sup> This is done because the coal does not contain any of the constituents of concern (COC) structurally as a separate mineral phase and so it is considered a diluent in the sample when it cannot be removed manually. First, the  $f_i$  term in Equation 1 and 2 must be expressed on a coal free basis. The sample is sent for dissolution and analysis with the coal content in it. Moisture is measured as loss-on-drying (LOD) at 110°C and the coal as LOI at 525°C. The elementals are converted to an oxide basis and mass balanced with the LOD and LOI. The sums should be within the 100±5% which ensures that the chemical analyses mass balance. Then the oxide data is adjusted for the LOD and LOI as shown in Table 17 and Table 26 through Table 30. Coal also contributes to the BET surface area as shown in Pareizs, et al.<sup>7</sup> and would cause an abnormally high BET surface area in the denominator of Equation 2.

Therefore, a subset of the PCT prepared sample meshed to -100 to +200 and ethanol washed is roasted at 525°C to get a "coal free" BET surface area but this subset is not used in the leach testing.

### 3.3 TCLP

The Hanford LAW 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].<sup>12</sup> The Land Disposal Restrictions (LDRs) will apply to shallow land burial at Hanford.

All blended aggregate samples and crushed monoliths were evaluated for retention of the hazardous metals by the EPA TCLP, Method 1311 [TCLP Method 1311, 1986].<sup>13</sup> Greater than 100 g samples (< 9.5 mm size) of as-received blended aggregate material containing residual coal from the FBSR process were submitted to GEL Laboratories, LLC of Charleston, SC, an EPA-certified laboratory. In the leaching procedure, 100 g samples of < 9.5 mm diameter are extracted by a buffered acetic acid 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, Ni, Sb, Tl and Zn. 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. FBSR blends from the LAW and WTP-SW tests, as well as the monoliths formed from the blends were also submitted for TCLP analyses. It is important to note that all TCLP testing in this program used 'as-received' materials, i.e., none of the materials submitted for TCLP were heat-treated to remove carbon. Since TCLP results are reported on a 'mg/L' basis for comparison to the UTS limits, no normalization of the TCLP leachate data was performed, i.e., normalization similar to what is performed for PCT using elemental fractions and measured surface areas. There can be excessive variability in data derived from TCLP testing due to the lack of particle size control used in this test, i.e., controlling the particle size controls the surface area exposed to the leachate. When the particle size is not controlled, particles can range from <9.5 mm down to any potential size including submicron sizes.

### 3.4 MONOLITH FORMATION AND COMPRESSION TESTING

Monolith studies involved the various cements and other binders shown in Table 5. All binder recipes requiring water used ASTM-I water (resistivity >18 Mohm-cm at 25°C). Geopolymers also used Fisher reagent grade sodium hydroxide (NaOH) solutions and D<sup>TM</sup> Sodium Silicate solution from the PQ Corporation (Valley Forge, PA). The ordinary Portland cement composition is made up of various calcium silicates, aluminates and aluminoferrites. The high alumina-containing 'calcium aluminate cements' are made up of various calcium aluminates along with calcium aluminum silicates and calcium aluminum ferrites. The high alumina cement binders were supplied from Kerneos Inc. of Chesapeake, VA.

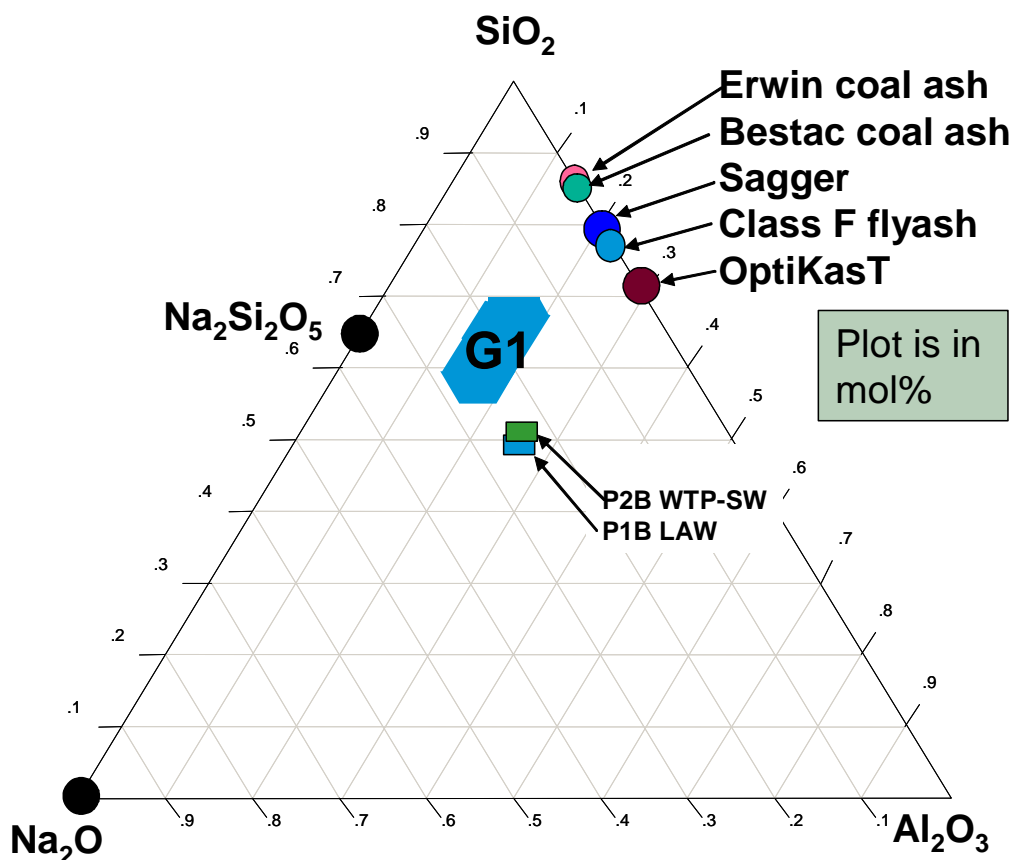
Ceramicrete is a blend of MgO and monopotassium phosphate (KH<sub>2</sub>PO<sub>4</sub>). The blend is mixed with a stoichiometric amount of water according to the formulation:



The reaction product on the right hand side of the equation is Ceramicrete, a rapid setting phosphate ceramic.<sup>14</sup> Ceramicrete ingredients were obtained from Argonne National Laboratory (ANL) and consist

of MgO and  $\text{KH}_2\text{PO}_4$ . No fly ash was used in the ceramicrete for these studies although it can be added as a minor ingredient. Some ceramicrete formulations also used trace boric acid powder as set retarder.

Geopolymer formulations for this project were based on earlier SRNL Laboratory Directed Research & Development (LDRD) studies completed in 2007.<sup>15</sup> Troy metakaolin clays were the main clays used in all geopolymer recipes. Later recipes used a Barden heat-treated clay, and a different set of geopolymer recipes used fly ash as additive in place of the clay. A recent review of fly ash uses in geopolymers for waste stabilization is available in the open literature.<sup>16</sup> Geopolymers were chosen due to their performance in the SRNL LDRD program and because of the similarity of the chemistry of the binder to that of the FBSR product (Figure 2).



**Figure 2.  $\text{Na}_2\text{O}$ - $\text{SiO}_2$ - $\text{Al}_2\text{O}_3$  (mol%) diagram showing region of geopolymer formation (G1) and the composition of the FBSR products analyzed in this study for WTP-SW and LAW**

The NuCap<sup>TM</sup> binder system consists of various liquid additives detailed in Table 5. NuCap<sup>TM</sup> is supplied by GLOBAL MATRECHS, INC. out of Ridgefield, CT. It is advertised as a ‘silicon-based geocomposite radiation resistant coating/foam used to encapsulate and contain radioactive materials’ (<http://www.globalmatrechs.com/>). A thick viscous liquid ‘binder paste’ was added at either 1 part binder to 1 part FBSR by mass, or at 1.2 part binder to 1 part FBSR by mass. Other organic liquids were then added in much smaller amounts (catalyst, thinner and boric acid paste).

Samples of the L-TEM monolith type were provided by CEES, Pasco, WA from Hazen ESTD FBSR material sent to CEES from TTT. However, because neither TTT nor SRNL could validate or confirm the formulation used in making the monoliths, only limited tests were performed, and thus key comparisons couldn't be made.

**Table 5. Cements and Binders Used for Monoliths**

Monolith System	Additives	Comments	Fabricated By
Ordinary Portland Cement	OPC binder	SiO <sub>2</sub> (19-23wt%), Al <sub>2</sub> O <sub>3</sub> (3-7wt%), Fe <sub>2</sub> O <sub>3</sub> (1.5-4.5wt%), CaO (63-67wt%), other metal (Mg,K,Na,S)-oxides trace	SRNL
High Alumina Cements	Tradenames: Ciment Fondu® Secar® 51 Secar® 71	CaO•Al <sub>2</sub> O <sub>3</sub> CaO•2Al <sub>2</sub> O <sub>3</sub> 12CaO•7Al <sub>2</sub> O <sub>3</sub> 2CaO•Al <sub>2</sub> O <sub>3</sub> •SiO <sub>2</sub> 4CaO•Al <sub>2</sub> O <sub>3</sub> •Fe <sub>2</sub> O <sub>3</sub>	SRNL
Ceramicrete	MgO and KH <sub>2</sub> PO <sub>4</sub>	MgO + KH <sub>2</sub> PO <sub>4</sub> + 5 H <sub>2</sub> O = MgKPO <sub>4</sub> •6H <sub>2</sub> O	SRNL with input by ANL
Geopolymer	Troy and Barden heat-treated clays; fly ash; sodium silicate; NaOH solution	Kaolin type clay (Al <sub>2</sub> O <sub>3</sub> •SiO <sub>2</sub> ) or fly ash with NaOH and sodium silicate to make an amorphous Na-Al-Si (NAS) binder	SRNL
NuCap™	One slurry paste and three different liquid additives -see 'Comments' column to this table for descriptions	<b>Slurry Paste:</b> GLOBALMATRECHS NUCAP™ PASTE A = Silicone Paste, 40.0 - 70.0 % Limestone; <1.0 % Quartz  <b>Liquid Additive 1:</b> OS-2000 = Vinyl Oximino Silane (VOS)  <b>Liquid Additive 2:</b> DOW CORNING(R) A-47 ADDITIVE = Silicone compound containing 40.0 - 70.0 %Boric acid  <b>Liquid Additive 3:</b> DOW CORNING(R) Q1-3563 FLUID = Silicone containing 1.0 - 5.0 %Octamethylcyclotetrasiloxane	Global Matrechs at SRNL
L-TEM geopolymeric cement	Unknown	a geopolymeric cement that contains heat treated kaolin and/or bentonite clay, microcrystalline silica, calcium and magnesium additives which appear high in sulfate once analyzed and cement components such as C <sub>3</sub> S (3CaO•SiO <sub>2</sub> )	Columbia Energy and Environmental Services, Pasco, WA

The 2" cubes were formulated by mixing the solid and liquid ingredients by hand in plastic bowls with a spatula for mixing. The final fresh blend was transferred from the plastic mix bowl to the cube mold with the spatula since these blends were typically thick and not easily poured. These monoliths were stored in capped cube molds from American Cube Mold, Inc. of Hinckley, Ohio. The cube molds are made of polyethylene plastic.

All of the 2" x 4" and 3" x 6" cylinders were formulated using a Hobart (Troy, OH) mixer mill (Model N-50, 3-speed, 1/6<sup>th</sup> HP) fitted with an ~ 5 quart stainless steel bowl. A larger Hobart mixer mill (Model D-300) fitted with an ~ 30 quart stainless steel bowl was used for the 6" x 12" cylinder formulations. The Hobart mixers were fitted with a flat beater multipurpose agitator. All cylinder monoliths made in this project used standard plastic molds with fitted plastic caps. No heat treatment was applied to any of the monoliths fabricated in this project.

Duplicate 6" x 12" cylinders were formulated and mixed at ambient laboratory temperatures of  $20^{\circ}\text{C} \pm 5^{\circ}\text{C}$ . A single sample from each duplicate set was fitted with a K-type thermocouple (TC) located on the centerline of the cylinder with the TC tip placed in the vertical center. All the 6" x 12" cylinders were allowed to cure in a constant temperature and humidity room that was maintained at  $23^{\circ}\text{C} \pm 2^{\circ}\text{C}$  and 95% minimum humidity. These are the ASTM-specified conditions for curing temperature and humidity for hydraulic cements and concretes per ASTM C31 and ASTM C511.

The 2" cube monolith blocks were compression tested using ASTM C 109-02 and all the cylinders were compression tested using ASTM C39-04A. These compression testing procedures and instruments were the same as used in a previous 2006 SRNL study on monoliths made using FBSR products.<sup>17</sup> Photographs of the compression testing as previously performed on hydroceramic cubes and ceramicrete cylinders are shown in Figure 3. All compression testing was performed at the Savannah River Site (SRS) by William L. Myhre of the United Research Services (URS) Company in the SRS N-Area Civil Engineering Test Facility.



**Figure 3. Photographs of Previous Compression Testing Performed at SRS N-Area Civil Testing Facility. Top photos show ASTM C 109-02 applied to 2" cube, bottom photo shows ASTM C39-04A applied to a cylinder**



## 4.0 RESULTS AND DISCUSSION

### 4.1 PRODUCT RECEIPT AND FILTER FINES PROPERTIES

XRD analyses were performed on the PR and HTF granular powders after screening through an 18-mesh sieve. Table 6 shows the identification of major and minor crystalline species for the initial PR and HTF aggregates as well as for the latter blends.

Appendix 3 shows the XRD spectra for the PR and HTF mineralized products. The phases identified in the LAW and WTP-SW FBSR product are the same as those identified in previous studies at HRI and at the SAIC-STAR facility with LAW wastes and INL SBW wastes. These phase assemblages are given in Table 6 for reference.

The PR and HTF minerals were sieved through an 18-mesh sieve to screen out any particles at  $\sim 1$  mm diameter. The resulting screened material was heat-treated at 525 °C for 8 hours to remove any residual coal remaining in the screened solids. Table 7 shows the mass losses from LOI treatment. Bulk density of the powder aggregate materials were also estimated using a 100-mL graduated cylinder and mass balance. The bulk density values shown in Table 7 are rough estimates of the bulk density since no attempts were made to determine the tap density using ASTM procedures/instrumentation.

Portions of the PR and HTF samples that had not been pre-roasted were also submitted for REDOX and these data are shown in Table 8 and Table 9, respectively. The REDOX data ( $\text{Fe}^{2+} / \text{Fe}_{\text{Total}}$ ) for the PR samples indicates that total iron is about 40 to 60% reduced in the LAW tests and about 40 – 45% reduced in the WTP-SW tests. Similar comparisons for the HTF REDOX data indicate that total iron is about 75 – 90% reduced for the LAW, and about 86 – 90% reduced in the WTP-SW. The previous LOI data indicates that the HTF samples contain between 9 and 13% residual carbon and it is possible that the residual carbon in these samples could alter the REDOX measurements by serving to reduce the iron during the dissolution and absorption measurements. This high bias or ‘increase’ in measured Fe-II has been observed in scope tests that added trace amounts of fresh ground coal to the standard Environmental Assessment (EA) glass that is used in the REDOX procedure. The reported range of the standard EA glass is  $\text{Fe}^{2+} / \text{Fe}_{\text{Total}} = 0.18$  to 0.19.<sup>18</sup> Since the HTF REDOX data showed high reduction values with essentially most of the total iron in the reduced +2 valence, these tests were repeated with nine pre-ethanol rinse/decant steps in attempts to rinse out (float) the carbon based on the lower density of carbon versus the mineral phases. Rinsed HTF samples were dried in ambient air at 60 °C overnight to remove residual ethanol.

Table 10 shows that the rinsed HTF samples give essentially the same REDOX values as those performed without the rinse/decant steps, with all samples showing > 90% iron in +2 reduced valence. It was not obvious that even after nine ethanol rinses that significant amounts of the black fine-grained particles were being successfully removed from the HTF powders. Attempts to remove the carbon by the LOI roasting procedure at 525 °C in air were not attempted due to the suspected alteration in the product REDOX that would be caused from the air-roasting.

**Table 6. Crystalline Phases Identified by XRD**

	<b>Low-Carnegieite</b> $e^f$ Nominally NaAlSiO <sub>4</sub>	<b>Nepheline</b> Nominally NaAlSiO <sub>4</sub> or K <sub>0.25</sub> Na <sub>0.75</sub> AlSiO <sub>4</sub>	<b>Nosean</b> Na <sub>6</sub> [Al <sub>6</sub> Si <sub>6</sub> O <sub>24</sub> ](Na <sub>2</sub> SO <sub>4</sub> ) and/or <b>Sodalite</b> Na <sub>6</sub> [Al <sub>6</sub> Si <sub>6</sub> O <sub>24</sub> ](2NaX where X=Cl,F,I)	<b>Other Minor Components</b>
<b>HANFORD ENVELOPE "C" LAW WASTES (2002) Fe<sup>+2</sup>/□ Fe of Bed = 0.15</b>				
SCT02-098-FM		X	Y	Al <sub>2</sub> O <sub>3</sub> , Fe <sub>2</sub> O <sub>3</sub> , Fe <sub>3</sub> O <sub>4</sub>
Fines PR-01	X	X	Y	Al <sub>2</sub> O <sub>3</sub> , Fe <sub>2</sub> O <sub>3</sub> , Fe <sub>3</sub> O <sub>4</sub>
<b>HANFORD ENVELOPE "A" LAW WASTES (2004) Fe<sup>+2</sup>/□ Fe of Bed = 0.28-0.81</b>				
Bed 1103	X	X	Y	TiO <sub>2</sub>
Bed 1104	X	X	Y	TiO <sub>2</sub>
Fines 1125	X	Y		TiO <sub>2</sub>
<b>INL SBW WASTES (2003-4) Fe<sup>+2</sup>/□ Fe of Bed = 0.51-0.61</b>				
Bed 260	Y	X	TR	Al <sub>2</sub> O <sub>3</sub> and TiO <sub>2</sub>
Bed 272	Y	X	TR	TiO <sub>2</sub>
Bed 277	Y	X	TR	TiO <sub>2</sub>
Bed 1173		X	TR	Al <sub>2</sub> O <sub>3</sub> , SiO <sub>2</sub> , NaAl <sub>11</sub> O <sub>17</sub> , (Ca,Na)SiO <sub>3</sub>
<b>HANFORD RASSAT LAW WASTES (2008) Fe<sup>+2</sup>/□ Fe of Bed = 0.41-0.90</b>				
PR Bed Product 5274 (P-1A)	Y	X	X	Al <sub>2</sub> O <sub>3</sub> ,
PR Bed Product 5316 (P-1A)	Y	X	X	Pyrophyllite*
HTF Fines 5280 (P-1A)	X	Y	TR	NaAl <sub>11</sub> O <sub>17</sub> (Diaoyudaoite), TiO <sub>2</sub>
HTF Fines 5297 (P-1A)	X	Y	X	SiO <sub>2</sub>
PR Bed Product 5359 (P-1B)	Y	X	X	Pyrophyllite*
PR Bed Product 5372 (P-1B)	Y	X	X	Pyrophyllite*
HTF Fines 5351 (P-1B)	X	Y	Y	SiO <sub>2</sub>
HTF Fines 5357 (P-1B)	X	Y	Y	TiO <sub>2</sub>
Composite (P-1A)	X	Y	Y	SiO <sub>2</sub> and TiO <sub>2</sub>
Composite (P-1B)	X	Y	Y	SiO <sub>2</sub> and TiO <sub>2</sub>
<b>HANFORD MELTER OFF-GAS RECYCLE (WTP SW) WASTES (2008) Fe<sup>+2</sup>/□ Fe = 0.41-0.90</b>				
PR 5475 (P-2A)	Y	Y	X	Pyrophyllite*
HTF Fines 5471 (P-2A)	X	X	X	SiO <sub>2</sub>
PR 5522 (P-2B)	Y	Y	X	Pyrophyllite*, TiO <sub>2</sub>
HTF Fines 5520 (P-2B)	X	X	X	SiO <sub>2</sub> and TiO <sub>2</sub>
Composite (P-2B)	Y	X	X	SiO <sub>2</sub>

X = Major constituent ; Y = Minor constituent ; TR = trace constituent

$f$  = the PDF for this phase states it is orthorhombic nepheline and possibly low-carnegieite (PDF 052-1342). Note low-carnegieite also has ring structures that are oval for sequestration of K, Cs, etc.

\*Al<sub>1.333</sub>Si<sub>2.667</sub>O<sub>6.667</sub>(OH)<sub>1.333</sub>

\*\* The XRD method gives information on the specific crystalline phases present by comparison to reference library spectra. Although this method is not used with any internal standards to allow for quantitative measurement of the various crystalline phases, it does provide information as to the 'major' and 'minor' and 'trace' phases present by intercomparison of the main peaks of each crystalline pattern within a given sample.

**Table 7. LOI Data and Bulk Density of Product Receiver (PR) Bed and High Temperature Filter (HTF) Samples**

Campaign	Sample ID	Amount of (-)18 Mesh Material	Mass Loss by Ashing at 525°C	Coal Content*	Bulk Density (g/cc)
<b>PRODUCT RECEIVER (PR) SAMPLES</b>					
<b>P-1A LAW</b>	5274 PR	26.365	0.564	2.14	1.002
	5316 PR	27.505	0.264	0.96	0.976
<b>P-1B LAW</b>	5359 PR	25.123	0.238	0.95	0.998
	5372 PR	28.393	0.105	0.37	0.944
<b>P-2A WTP-SW</b>	5475 PR	29.099	0.240	0.82	0.994
<b>P-2B WTP-SW</b>	5522 PR	26.881	0.297	1.10	0.964
<b>HIGH TEMPERATURE FILTER (HTF) SAMPLES</b>					
<b>P-1A LAW</b>	5280 HTF	34.300	3.416	9.96	0.658
	5297 HTF	33.295	3.333	10.01	0.648
<b>P-1B LAW</b>	5351 HTF	37.995	3.619	9.52	NA
	5357 HTF	29.547	2.548	8.62	0.651
<b>P-2A WTP-SW</b>	5471 HTF	31.644	4.055	12.81	0.651
<b>P-2B WTP-SW</b>	5520 HTF	35.323	4.592	13.00	0.654

\* Ash Mass Loss Divided By Original Mass x 100

Table 8. REDOX Data for Non-roasted PR Samples

Sample	Lab ID	Fe <sup>2+</sup>	Fe <sup>3+</sup>	Fe <sub>(total)</sub>	$\frac{\text{Fe}^{2+}}{\text{Fe}^{3+}}$	$\frac{\text{Fe}^{2+}}{\text{Fe}_{(\text{total})}}$	Test Campaign	Hours of Operation at Test Conditions Before Sample Obtained	DMR Temp. (°C)	g Moist Clay per L Sim.	Clay Type		
EA Glass	--	0.070	0.367	0.437	0.191	0.160	--	--	--	--	--		
EA Glass – Ref. Range	--	--	--	--	0.22 – 0.23	0.18 – 0.19	--	--	--	--	--		
5274 PR (A)	08-1350 A	0.171	0.245	0.416	0.698	0.411	P-1A LAW	25.3	725	675	OptiKast		
5274 PR (B)	08-1350 B	0.170	0.246	0.416	0.691	0.409							
5316 PR (A)	08-1351 A	0.066	0.047	0.113	1.404	0.584		47.9					
5316 PR (B)	08-1351 B	0.066	0.048	0.114	1.375	0.579							
5359 PR (A)	08-1352 A	0.242	0.234	0.476	1.034	0.508	P-1B LAW	38.9	725	640	OptiKast		
5359 PR (B)	08-1352 B	0.242	0.234	0.476	1.034	0.508							
5372 PR (A)	08-1353 A	0.061	0.060	0.121	1.017	0.504		47					
5372 PR (B)	08-1353 B	0.060	0.061	0.121	0.984	0.496							
5475 PR (A)	08-1354 A	0.065	0.094	0.159	0.691	0.409	P-2A WTP-SW	74	680	307	45% Sagger/ 55% OptiKast		
5475 PR (B)	08-1354 B	0.065	0.093	0.158	0.699	0.411							
5522 PR (A)	08-1355 A	0.111	0.130	0.241	0.854	0.461	P-2B WTP-SW	26.5	700				
5522 PR (B)	08-1355 B	0.112	0.129	0.241	0.868	0.465							

Table 9. REDOX Data for Non-roasted HTF Samples

Sample	Lab ID	Fe <sup>2+</sup>	Fe <sup>3+</sup>	Fe <sub>(total)</sub>	$\frac{\text{Fe}^{2+}}{\text{Fe}^{3+}}$	$\frac{\text{Fe}^{2+}}{\text{Fe}_{(\text{total})}}$	Test Campaign	Hours of Operation at Test Conditions Before Sample Obtained	DMR Temp. (°C)	g Moist Clay per L Sim.	Clay Type
EA Glass	--	0.024	0.107	0.131	0.224	0.183	--	--	--	--	--
EA Glass – Ref. Range	--	--	--	--	0.22 – 0.23	0.18 – 0.19	--	--	--	--	--
HTF 5280 (A)	09-0023	0.024	0.008	0.032	2.4	0.750	P-1A LAW	17.7	725	675	OptiKast
HTF 5280 (B)	09-0023	0.023	0.009	0.032	2.3	0.719		37.5			
HTF 5297 (A)	09-0024	0.023	0.003	0.026	2.3	0.885					
HTF 5297 (B)	09-0024	0.023	0.004	0.027	2.3	0.852					
HTF 5351 (A)	09-0025	0.027	0.003	0.030	2.7	0.900	P-1B LAW	28	725	640	OptiKast
HTF 5351 (B)	09-0025	0.026	0.003	0.029	2.6	0.897		35.7			
HTF 5357 (A)	09-0026	0.026	0.003	0.029	2.6	0.897					
HTF 5357 (B)	09-0026	0.027	0.003	0.030	2.7	0.900					
HTF 5471 (A)	09-0027	0.027	0.004	0.031	2.7	0.871	P-2A	70.3	680	307	45% Sagger/ 55% OptiKast
HTF 5471 (B)	09-0027	0.026	0.004	0.030	2.6	0.867	WTP-SW				
HTF 5520 (A)	09-0028	0.023	0.002	0.025	2.3	0.920	P-2B	26.5	700		
HTF 5520 (B)	09-0028	0.023	0.003	0.026	2.3	0.885	WTP-SW				

**Table 10. REDOX Data for Non-roasted HTF Samples After Rinsing**

Sample	Lab ID	Fe <sup>2+</sup>	Fe <sup>3+</sup>	Fe <sub>(total)</sub>	$\frac{\text{Fe}^{2+}}{\text{Fe}^{3+}}$	$\frac{\text{Fe}^{2+}}{\text{Fe}_{(\text{total})}}$	Test Campaign	Hours of Operation at Test Conditions Before Sample Obtained	DMR Temp. (°C)	g Moist Clay per L Sim.	Clay Type
EA Glass	--	0.053	0.223	0.276	0.238	0.192	--	--	--	--	--
EA Glass – Ref. Range	--	--	--	--	0.22 – 0.23	0.18 – 0.19	--	--	--	--	--
HTF 5280 (A)	09-0716	0.094	0.005	0.099	18.80	0.95	P-1A LAW	17.7	725	675	OptiKast
HTF 5280 (B)	09-0716	0.096	0.004	0.100	24.00	0.96		37.5			
HTF 5297 (A)	09-0717	0.086	0.003	0.089	28.67	0.97					
HTF 5297 (B)	09-0717	0.086	0.004	0.090	21.50	0.96					
HTF 5351 (A)	09-0718	0.093	0.002	0.095	46.50	0.98	P-1B LAW	28	725	640	OptiKast
HTF 5351 (B)	09-0718	0.094	0.002	0.096	47.00	0.98		35.7			
HTF 5357 (A)	09-0719	0.077	0.001	0.078	77.00	0.99					
HTF 5357 (B)	09-0719	0.077	0.001	0.078	77.00	0.99					
HTF 5471 (A)	09-0720	0.089	0.002	0.091	44.50	0.98	P-2A WTP-SW	70.3	680	307	45% Sagger/ 55% OptiKast
HTF 5471 (B)	09-0720	0.089	0.003	0.092	29.67	0.97	P-2B WTP-SW	26.5	700		
HTF 5520 (A)	09-0720	0.081	0.005	0.086	16.20	0.94					
HTF 5520 (B)	09-0720	0.081	0.004	0.085	20.25	0.95					

## 4.2 PRODUCT AND FINES CHEMICAL COMPOSITIONS AND PRODUCT CONSISTENCY TESTS

The HTF fines and PR bed samples of mineralized product samples remaining after roasting were prepared for durability testing using the PCT. Thus all data discussed in this section pertain to the roasted fines and bed product samples. Both HTF and PR roasted products were ground and sieved through 100-200 mesh sieves, then washed with ethanol and dried at 90°C. Particle size distributions of the ground/sieved/washed/dried powders were measured via Microtrac instrumentation and the results are presented in Appendix 4. Data shown in Appendix 4 indicate that the particle size of the HTF samples were in the range of 2 to 154 microns with ~ 50% of the particles in the range of 28 to 47 microns. The PR samples ranged from 3 to 173 microns with 50% of the particles in the range of 68 to 89 microns. Both HTF and PR samples display a tailing of smaller particle sizes below the 200 mesh sieve size of 74 microns and both sample sets show that the ‘peak’ particle size in the distribution is below the predicted Gaussian distribution observed for 100-200 mesh sieved mineral products at ~ 112 microns.

Samples of the PCT-prepared powders that passed through the 200 mesh sieve were also submitted for chemical composition dissolution and characterization. The elemental composition of the HTF and PR samples (average of duplicate dissolutions for each sample) are shown in Table 11. Oxide conversions were calculated and presented along with the Cl<sup>-</sup>, F<sup>-</sup> and I<sup>-</sup> anion data in Table 12. The sums of all metal-oxides and anions Cl<sup>-</sup>, F<sup>-</sup>, I<sup>-</sup> and phosphate and sulfate are all near 100% indicating complete dissolution and successful chemical characterization of the powders. It should be noted that no detectable nitrate or nitrite anions were present in the dissolved HTF and PR products (< 0.04 wt%) indicating complete destruction of these components (FBSR feed simulants contained nitrite > 1g/L and nitrate > 100 g/L) in the ESTD processing. Also, since these bed product and fines had been roasted at 525 °C before PCT preparation and dissolution analyses, no corrections were applied to the reported data for presence of residual coal from the ESTD FBSR process.

Comparison of the major elements in the bed and fines (Al, Na and Si) indicate no significant differences between either the LAW versus WTP-SW, or in the PR bed products versus the HTF fines. Both Cl<sup>-</sup> and F<sup>-</sup> were higher in the WTP-SW feeds than the LAW feeds per previous Table 2. These elements were analyzed to be higher concentration in the WTP-SW product samples with Cl<sup>-</sup> predominately in the bed products and F<sup>-</sup> predominately in the fines. Iodide values were higher in the LAW minerals than the WTP-SW as expected from the higher Iodide feed concentrations for LAW versus WTP-SW. Iodide appears to be predominate in the LAW bed product samples versus the fines. The sulfur values from ICP-AES were somewhat evenly distributed across the PR and the HTF for the LAW samples, and they were slightly higher in the WTP-SW PR bed samples versus the WTP-SW HTF samples. The Re values in all samples were similar as were the feed values of Re for LAW and WTP-SW from Table 2. The Re concentrations were somewhat higher for the PR in the range of 0.06 to 0.08 wt% versus the lower Re values in the HTF in the range of 0.02 to 0.05.

Table 11. Elemental Composition (Wt%) of the Bed Products and Fines

(wt%)	FBSR Campaign	Ag	Al	As	B	Ba	Ca	Cd	Cl	Cr	Cs	Cu	F	Fe	I	K
HIF-5280	LAW (P-1A)	0.02	18.80	<0.10	<0.10	0.12	0.07	0.02	<0.02	0.07	0.23	<0.01	0.09	0.56	0.046	0.21
HIF-5297		0.01	18.35	<0.10	<0.10	0.11	0.07	0.02	<0.02	0.07	0.33	<0.01	0.08	0.54	0.035	0.23
HIF-5351	LAW (P-1B)	0.02	17.30	<0.10	<0.10	0.13	0.05	0.06	0.26	0.09	0.14	<0.01	<0.05	1.26	0.090	0.24
HIF-5357		0.01	18.10	<0.10	<0.10	0.13	0.06	0.02	<0.02	0.07	0.24	<0.01	0.09	0.38	0.056	0.19
HIF-5471	WTP-SW (P-2A)	0.01	17.60	<0.10	0.31	0.01	0.15	<0.01	0.37	0.07	0.59	<0.01	0.27	0.60	0.010	0.37
HIF-5520	WTP-SW (P-2B)	0.01	17.45	<0.10	0.30	0.01	0.13	<0.01	0.32	0.08	0.57	<0.01	0.17	0.50	0.008	0.38
PR-5274	LAW (P-1A)	0.03	16.85	<0.10	<0.10	0.09	0.05	0.09	<0.02	0.10	0.07	0.03	0.08	8.05	0.292	0.15
PR-5316		0.03	19.00	<0.10	<0.10	0.09	0.03	0.08	<0.02	0.11	0.08	0.02	0.07	3.77	0.191	0.15
PR-5359	LAW (P-1B)	0.04	16.85	<0.10	<0.10	0.11	0.04	0.07	1.02	0.10	0.10	0.01	0.10	4.34	0.320	0.16
PR-5372		0.04	16.65	<0.10	<0.10	0.12	0.03	0.09	0.08	0.11	0.12	0.01	0.10	4.16	0.225	0.15
PR-5475	WTP-SW (P-2A)	0.04	16.75	<0.10	0.13	0.01	0.05	0.03	0.91	0.11	0.21	0.01	0.08	4.02	0.009	0.43
PR-5522	WTP-SW (P-2B)	0.03	15.80	<0.10	0.13	0.01	0.05	0.03	1.04	0.10	0.16	0.01	0.07	6.58	0.008	0.41

(wt%)	FBSR Campaign	Mg	Mn	Na	Ni	P	Pb	Re	S	Sb	Se	Si	Ti	Tl	Zn
HIF-5280	LAW (P-1A)	0.01	<0.01	13.80	0.05	0.20	<0.10	0.026	0.32	0.07	<0.01	19.70	0.70	<0.01	<0.10
HIF-5297		0.02	<0.01	13.58	0.05	0.19	<0.10	0.024	0.31	0.06	<0.01	19.55	0.70	<0.01	<0.10
HIF-5351	LAW (P-1B)	0.01	<0.01	14.00	0.08	0.23	<0.10	0.049	0.57	0.06	<0.01	18.45	0.69	<0.01	<0.10
HIF-5357		0.01	<0.01	14.84	0.05	0.21	<0.10	0.024	0.32	0.06	<0.01	19.15	0.65	<0.01	<0.10
HIF-5471	WTP-SW (P-2A)	0.05	<0.01	12.86	0.05	0.12	<0.10	0.030	0.19	0.05	<0.01	19.70	0.71	<0.01	0.11
HIF-5520	WTP-SW (P-2B)	0.05	<0.01	13.26	0.05	0.11	<0.10	0.033	0.27	0.05	<0.01	19.90	0.74	<0.01	0.10
PR-5274	LAW (P-1A)	0.02	0.04	13.62	0.08	0.22	<0.10	0.066	0.27	0.07	<0.01	16.35	0.65	<0.01	<0.10
PR-5316		0.01	0.01	14.17	0.08	0.21	<0.10	0.059	0.43	0.06	<0.01	16.25	0.68	<0.01	<0.10
PR-5359	LAW (P-1B)	0.02	0.02	15.14	0.08	0.25	<0.10	0.070	0.50	0.08	<0.01	17.30	0.73	<0.01	<0.10
PR-5372		0.02	0.01	15.31	0.08	0.24	<0.10	0.073	0.65	0.08	<0.01	17.05	0.71	<0.01	<0.10
PR-5475	WTP-SW (P-2A)	0.06	0.02	14.63	0.06	<0.10	<0.10	0.081	0.47	0.04	<0.01	17.85	0.78	<0.01	0.12
PR-5522	WTP-SW (P-2B)	0.06	0.03	14.07	0.06	<0.10	<0.10	0.069	0.34	0.04	<0.01	17.20	0.75	<0.01	0.12



Table 12. Oxide Composition (Wt%) of the Bed Products and Fines

(wt%)	FBSR Campaign	Ag <sub>2</sub> O	Al <sub>2</sub> O <sub>3</sub>	B <sub>2</sub> O <sub>3</sub>	BaO	CaO	CdO	Cl	Cr <sub>2</sub> O <sub>3</sub>	Cs <sub>2</sub> O	CuO	F	Fe <sub>2</sub> O <sub>3</sub>
HTF-5280	LAW(P-1A)	0.02	35.52	<0.32	0.14	0.09	0.03	<0.02	0.10	0.25	<0.01	0.09	0.79
HTF-5297		0.01	34.67	<0.32	0.13	0.10	0.02	<0.02	0.10	0.35	<0.01	0.08	0.78
HTF-5351	LAW(P-1B)	0.02	32.69	<0.32	0.14	0.08	0.06	0.26	0.13	0.15	<0.01	<0.05	1.80
HTF-5357		0.01	34.20	<0.32	0.14	0.08	0.02	<0.02	0.10	0.26	<0.01	0.09	0.54
HTF-5471	WTP-SW(P-2A)	0.01	33.26	0.98	0.01	0.21	<0.01	0.37	0.10	0.63	<0.01	0.27	0.86
HTF-5520	WTP-SW(P-2B)	0.01	32.97	0.97	0.01	0.18	<0.01	0.32	0.12	0.60	<0.01	0.17	0.72
PR-5274	LAW(P-1A)	0.04	31.84	<0.32	0.11	0.06	0.10	<0.02	0.14	0.08	0.04	0.08	11.50
PR-5316		0.04	35.90	<0.32	0.11	0.04	0.09	<0.02	0.16	0.09	0.02	0.07	5.39
PR-5359	LAW(P-1B)	0.04	31.84	<0.32	0.13	0.06	0.08	1.02	0.14	0.11	0.01	0.10	6.20
PR-5372		0.04	31.46	<0.32	0.13	0.04	0.10	0.08	0.15	0.13	0.01	0.10	5.94
PR-5475	WTP-SW(P-2A)	0.04	31.65	0.43	0.01	0.07	0.04	0.91	0.16	0.22	0.02	0.08	5.75
PR-5522	WTP-SW(P-2B)	0.03	29.85	0.42	0.01	0.07	0.04	1.04	0.15	0.17	0.01	0.07	9.41

(wt%)	FBSR Campaign	I	K <sub>2</sub> O	MgO	MnO <sub>2</sub>	Na <sub>2</sub> O	NiO	PO <sub>4</sub>	ReO <sub>2</sub>	SO <sub>4</sub>	SiO <sub>2</sub>	TiO <sub>2</sub>	ZnO	Total
HTF-5280	LAW(P-1A)	0.05	0.25	0.02	<0.02	18.60	0.07	0.62	0.03	1.56	42.14	1.16	<0.12	102.12
HTF-5297		0.03	0.28	0.03	<0.02	18.30	0.07	0.57	0.03	1.47	41.82	1.18	<0.12	100.59
HTF-5351	LAW(P-1B)	0.09	0.29	0.02	<0.02	18.87	0.10	0.71	0.06	2.23	39.47	1.15	<0.12	98.92
HTF-5357		0.06	0.23	0.02	<0.02	20.01	0.07	0.64	0.03	1.71	40.97	1.08	<0.12	100.83
HTF-5471	WTP-SW(P-2A)	0.01	0.45	0.08	<0.02	17.33	0.06	0.35	0.04	0.98	42.14	1.18	0.13	99.56
HTF-5520	WTP-SW(P-2B)	0.01	0.46	0.09	<0.02	17.88	0.06	0.33	0.04	1.25	42.57	1.24	0.13	100.25
PR-5274	LAW(P-1A)	0.29	0.19	0.03	0.06	18.36	0.10	0.67	0.08	1.49	34.98	1.08	<0.12	101.84
PR-5316		0.19	0.18	0.02	0.02	19.10	0.10	0.66	0.07	2.14	34.76	1.13	<0.12	100.82
PR-5359	LAW(P-1B)	0.32	0.19	0.03	0.03	20.41	0.11	0.78	0.08	2.73	37.01	1.21	<0.12	103.17
PR-5372		0.23	0.18	0.03	0.02	20.64	0.11	0.73	0.09	2.55	36.47	1.19	<0.12	100.98
PR-5475	WTP-SW(P-2A)	0.01	0.52	0.10	0.02	19.73	0.08	<0.31	0.10	2.16	38.18	1.30	0.15	102.07
PR-5522	WTP-SW(P-2B)	0.01	0.49	0.10	0.04	18.96	0.08	<0.31	0.08	1.60	36.79	1.26	0.15	101.19

Results of the 7-day PCT performed on the roasted PR bed and HTF fines samples are shown in Table 13. All leachate values are shown in mg/L units for Al, Cs, I, Na, Re, S and Si along with the resulting leachate final pHs. These elements were chosen to be tracked in the PCT to cover the main mineral elements (Al, Na and Si) as well as key trace elements Cs, I and Re to represent radioactive Cs-137, I-129 and Tc-99, respectively. The leachate pHs were similar in the range of 11.2 to 11.6 for all the LAW samples and the WTP-SW leachate pHs were slightly lower in the range of 9.98 to 10.84.

The surface area of the PCT-prepared FBSR mineral powders are also shown from BET measurements in units of  $\text{m}^2/\text{g}$ . All of these BET SA data were obtained using a 4-hr pre-evacuation at 200 °C. The reported uncertainty for these BET SA data were all nominally 0.3 % relative standard deviation (%RSD). These values were determined from dividing the reported  $\pm$  BET SA value by the measured BET SA value and multiplying the ratio by 100X. A sample calculation using the first BET SA shown in Table 13 for the HTF 5280 sample is:

$$\% \text{RSD} = [\pm \text{BET SA} / \text{BET SA}] * 100 = [0.02 \text{ m}^2/\text{g} / 5.28 \text{ m}^2/\text{g}] * 100 = 0.3$$

Note that these reported %RSD values reported for each individual BET SA measurement only give uncertainty related to the various pressures measured within the single analysis. It is likely that the true BET SA uncertainty of the actual powder sample is higher (in the range of  $\pm 5\%$  as reported for replicate measurements of primary silicate mineral samples in previous work).<sup>11</sup> No replicate BET SA measurements were performed on the various mineral samples in this study.

The surface area to volume ratio of the tests in units of  $\text{m}^{-1}$  can be calculated via the following equation:

$$\text{SA/V} = g_{\text{mineral}} / \text{volume leachate (L)} * \text{surface area (m}^2/\text{g)} * (1\text{L}/1000 \text{ cm}^3) * (100\text{cm}/1\text{m})^3$$

$$\text{SA/V for 5280 HTF} = (1.5 \text{ g}/0.015 \text{ L}) * (5.28 \text{ m}^2/\text{g} / 1000) * 100^3 = 528,480 \text{ m}^{-1}$$

Normalized release values for the various elements ( $\text{NL}_i$ ) for the 7-day PCT are also shown in Table 13. The normalized release is calculated using Equation 2:

$$\text{normalized release (NL}_i\text{) in g/m}^2 = [\text{Leachate concentration}] / (\text{SA/V}) / [f_i]$$

For example,

$$\text{NL}_i \text{ for Al for 5280 HTF} = [(192.8 \text{ mg/L} * (1\text{g}/1000 \text{ mg})) / (528,480 \text{ m}^{-1}) / [(18.80/100)) * (1\text{L}/1000 \text{ cm}^3) * (100 \text{ cm}/1 \text{ m})^3] = 1.9\text{E-}03 \text{ g/m}^2$$

It should be noted that a control reference ARM glass<sup>19</sup> and a Low Activity Reference Material (LRM) glass<sup>20</sup> were included in these and all subsequent PCTs described in this project.

The PCT results for these glasses are collected in Appendix 14. The purpose of including the ARM glass is to track the PCT release and compare it to the historical performance of this glass in the PCT. The purpose of including the LRM glass is so that its measured normalized release can be compared to previous PCT round robin testing with this glass. The EA reference glass<sup>18</sup> was inadvertently used in place of the LRM glass for the 3" x 6" cylinder PCTs.

Normalized release data from Table 13 is plotted in Figure 4 to show comparison of the different samples. Normalized release for sulfur was highest for all samples relative to other measured analytes and appears to be higher for the HTF ( $\text{NL}(\text{S})$  in range of 0.07 to 0.13  $\text{g/m}^2$ ) versus the PR samples ( $\text{NL}(\text{S})$  in range of 0.04 to 0.07  $\text{g/m}^2$ ).  $\text{NL}(\text{Re})$  is higher for the HTF fines versus  $\text{NL}(\text{Re})$  in the PR bed products. The  $\text{NL}(\text{Cs})$  is higher for PR bed products versus the HTF fines.  $\text{NL}(\text{Na})$  is about the same

across all bed products and fines and NL(Al) and NL(Si) are less than 0.005 and 0.001 g/m<sup>2</sup>, respectively.

Due to the high surface area of the specimens, one must ask, can the 2 g/m<sup>2</sup> maximum value set for LAW glass<sup>21</sup> even be attained. For example, the PCT-A procedure for typical borosilicate glass involves a 1:10 ratio of 1 g glass (ground to 100-200 mesh) to 10 g water. The geometric calculated surface area for nominal 2.7 g/cc borosilicate glass is 0.02 m<sup>2</sup>/g.<sup>7</sup> Actual BET measured surface area for LRM glass ground to 100-200 mesh supports this calculated surface area, as BET measurements have shown that LRM glass has a measured surface area of 0.04 m<sup>2</sup>/g, which is about 2X higher than that calculated from geometrical average particle size data.<sup>7</sup> The measured BET surface areas for the PR and HTF samples (100-200 mesh) leached in this work are considerably higher in the range of 2 to 5 m<sup>2</sup>/g. The surface area of the leached powders in the PCT-A have a significant effect on the calculated normalized release as shown in Table 14. These data show that the Hanford Immobilized Low Activity Waste (ILAW) specification of 2 g/m<sup>2</sup> for glass corresponds to about 4% total release of a given element in the 7-day PCT using a 1g glass to 10 g leachate ratio if one uses the geometrical surface area. This value increases to 8% release if one uses the BET surface area for glass of 0.04 m<sup>2</sup>/g. These two values are shaded in blue in Table 14. For the much higher surface area FBSR powders, a maximum NL(i) in the range of only 0.05 to 0.5 g/m<sup>2</sup> is attained even if 100% of the powder were to leach into solution. It has been noted in previous mineral leaching studies that surface area has been recognized as an important factor in quantifying mineral dissolution rates.<sup>22</sup> How one treats surface area is one of, if not the most problematic variable, and has been the subject of numerous studies.<sup>11</sup>

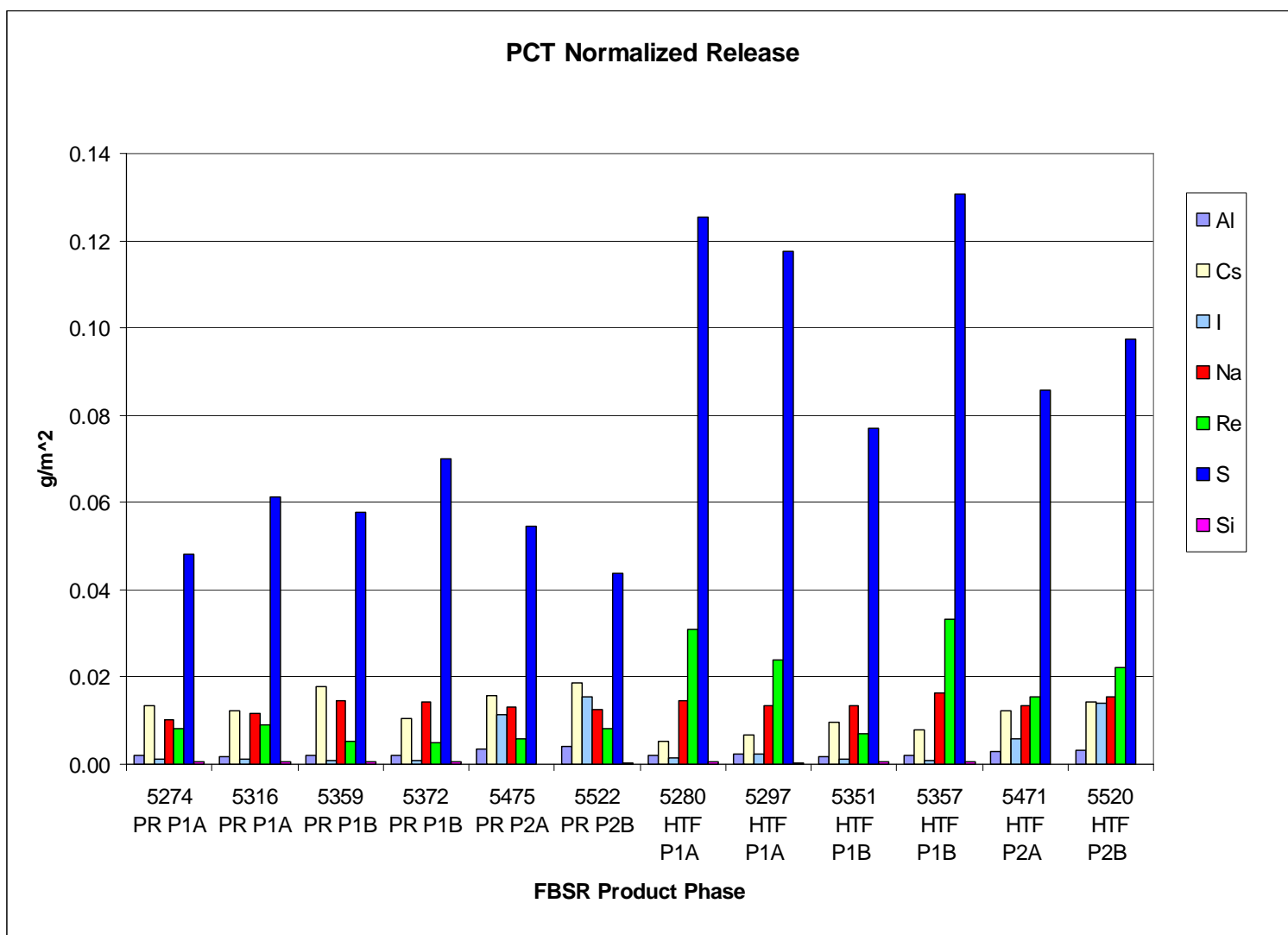
A large degree of uncertainty is associated with measurements of bulk BET surface area, and most critically, the contribution of actual reactive surface area is not always known. Although use of the BET surface area may overestimate the true reactive surface area, the obvious microporosity indicates that use of the geometric surface area will underestimate the true dissolution rate. All dissolution rates reported in this document have been normalized to the BET surface area. The true reactive surface area is probably less than the BET value, but also probably significantly higher than the geometric value. As has been pointed out in previous work, additional work will be required to better constrain the reactive surface area of the FBSR product.<sup>22</sup>

Table 13. PCT Data for the Bed Products and Fines

	FBSR Campaign	As Measured Leachate Concentrations							pH	BET (m <sup>2</sup> /g) roasted	SA/V (m <sup>-1</sup> )	Normalized Releases Calculated from Equations 1 & 2						
		Al (mg/L)	Cs (mg/L)	I (mg/L)	Na (mg/L)	Re (mg/L)	S (mg/L)	Si (mg/L)				NL <sub>Al</sub> (g/m <sup>2</sup> )	NL <sub>Cs</sub> (g/m <sup>2</sup> )	NL <sub>I</sub> (g/m <sup>2</sup> )	NL <sub>Na</sub> (g/m <sup>2</sup> )	NL <sub>Re</sub> (g/m <sup>2</sup> )	NL <sub>S</sub> (g/m <sup>2</sup> )	NL <sub>Si</sub> (g/m <sup>2</sup> )
5280 HTF	LAW (P-1A)	192.8	6.6	0.4	1058.2	4.2	345.0	49.2	11.58	5.28	528,480	1.9E-03	5.4E-03	1.5E-03	1.5E-02	3.1E-02	1.3E-01	4.7E-04
5297 HTF		240.0	12.0	0.4	1002.1	3.2	320.0	34.4	11.48	5.55	554,620	2.4E-03	6.6E-03	2.3E-03	1.3E-02	2.4E-02	1.2E-01	3.2E-04
5351 HTF	LAW (P-1B)	146.1	6.9	0.5	985.8	1.8	283.3	49.8	11.52	5.26	525,904	1.6E-03	9.5E-03	1.1E-03	1.3E-02	7.0E-03	7.7E-02	5.1E-04
5357 HTF		179.7	9.2	0.3	1145.6	3.8	355.0	51.1	11.66	4.76	475,670	2.1E-03	8.0E-03	1.0E-03	1.6E-02	3.3E-02	1.3E-01	5.6E-04
5471 HTF	WTP-SW (P-2A)	285.5	40.3	0.3	956.8	2.6	155.7	12.1	9.98	5.56	555,550	2.9E-03	1.2E-02	5.9E-03	1.3E-02	1.5E-02	8.6E-02	1.1E-04
5520 HTF	WTP-SW (P-2B)	286.4	42.0	0.6	1057.9	3.8	209.2	10.9	9.98	5.15	515,200	3.2E-03	1.4E-02	1.4E-02	1.5E-02	2.2E-02	9.7E-02	1.1E-04
5274 PR P-1A	LAW (P-1A)	132.9	4.0	1.3	567.9	2.2	138.8	43.0	11.28	4.04	404,180	2.0E-03	1.3E-02	1.1E-03	1.0E-02	8.3E-03	4.8E-02	6.5E-04
5316 PR P-1A		151.1	4.8	1.2	777.6	2.6	208.3	46.2	11.43	4.76	476,410	1.7E-03	1.2E-02	1.3E-03	1.2E-02	9.1E-03	6.1E-02	6.0E-04
5359 PR P-1B	LAW (P-1B)	133.7	7.4	1.0	902.0	1.5	216.7	45.1	11.45	4.11	410,570	1.9E-03	1.8E-02	7.6E-04	1.5E-02	5.1E-03	5.8E-02	6.3E-04
5372 PR P-1B		138.1	5.5	0.8	956.7	1.5	260.0	49.4	11.54	4.36	436,000	1.9E-03	1.1E-02	8.6E-04	1.4E-02	4.8E-03	7.0E-02	6.6E-04
5475 PR P-2A	WTP-SW (P-2A)	250.3	14.1	0.4	837.9	2.1	170.8	10.5	10.84	4.35	435,000	3.4E-03	1.6E-02	1.1E-02	1.3E-02	6.0E-03	5.4E-02	1.4E-04
5522 PR P-2B	WTP-SW (P-2B)	237.2	11.7	0.5	676.1	2.2	88.8	12.2	10.64	3.80	380,000	4.0E-03	1.9E-02	1.5E-02	1.3E-02	8.2E-03	4.4E-02	1.9E-04

**Table 14. Calculated NL(i) at Varying % Release for Different Surface Areas (SA)**

	SA= 0.02 m <sup>2</sup> /g	SA= 0.04 m <sup>2</sup> /g	SA= 2 m <sup>2</sup> /g	SA= 20 m <sup>2</sup> /g
% Release	NL(i) (g/m <sup>2</sup> )	NL(i) (g/m <sup>2</sup> )	NL(i) (g/m <sup>2</sup> )	NL(i) (g/m <sup>2</sup> )
2.5	1.3	0.6	0.013	0.001
<b>4.0</b>	<b>2.0</b>	1.0	0.020	0.002
5.0	2.5	1.3	0.025	0.003
<b>8.0</b>	4.0	<b>2.0</b>	0.040	0.004
25.0	12.5	6.3	0.125	0.013
50.0	25.0	12.5	0.250	0.025
75.0	37.5	18.8	0.375	0.038
100.0	50.0	25.0	0.500	0.050

**Figure 4. Normalized PCT Release for HTF and PR Samples**

On conclusion of the PCTs and examination of the data for the PR bed products and HTF fines, the data was used as basis for selecting which LAW Product Runs (P-1A or P-1B) and which WTP-SW Production Runs (P-2A or P-2B) to test in further work that would initially only include the LAW blend/aggregate for monolith studies. The overall data from the LAW P-1B campaign samples for the PR and HTF were judged to be slightly more durable than from the LAW P1A data. For the P-1B PR samples it was noted that the average  $NL_{Re}$  was lower than for the P-1A PR average. This is also true for the P-1B HTF samples versus the P-1A HTF samples if one averages the  $NL_{Re}$  shown in Figure 4. Selection basis focused on Re as a key indicator for the PCT response since this non radioactive element was used in the ESTD FBSR testing as a surrogate for radioactive technetium (Tc-99). Results for the WTP-SW series were similar for the P-2A and P-2B data sets. The LAW P-1B blend was chosen for further testing.

### 4.3 AGGREGATE/BLENDED PR AND HTF PROPERTIES

Original small test blends of both LAW P-1A and P-1B were received in ~ 7-kg bags. These blends were used for initial scope testing for monolithing. Blended PR and HTF products from the HRI testing that were shipped to SRNL were originally characterized for XRD, particle size and LOI. The blends had been prepared and screened at the Hazen facility prior to shipment. As the PCT performance data became available from testing of the PR bed products and HTF fines, and a decision was made to proceed with P-1B for LAW and P-2B for WTP-SW, larger batches of LAW P-1B and WTP-SW P-2B were later received in multiple buckets and a large drum, respectively. The XRD spectra of the blends show similar crystalline phases as were identified in the original aggregate samples as shown in Table 6.

Microtrac particle size measurements were performed on the blends and this data is shown in Table 15. The LAW P-1B particles from the initially received 7 kg bag were broadly distributed over a wide range of  $< 1 \mu m$  up to  $\sim 800 \mu m$ , with a visible peak centered around  $300 \mu m$ . Sieving of this material through a 1 mm sieve did not change the PSD. The PSD for the P-1B bucket blend was similar to the LAW P-1B 7 kg bag blend sample. The PSD for the WTP-SW shows similar distribution to the P-1B LAW blend except that no larger sized 300 micron peak was visible. These particle size distribution data show that the blended aggregates prepared and pre-screened at HRI contained broad size distributions of particles from sub-micron all the way up to  $> 300$  microns.

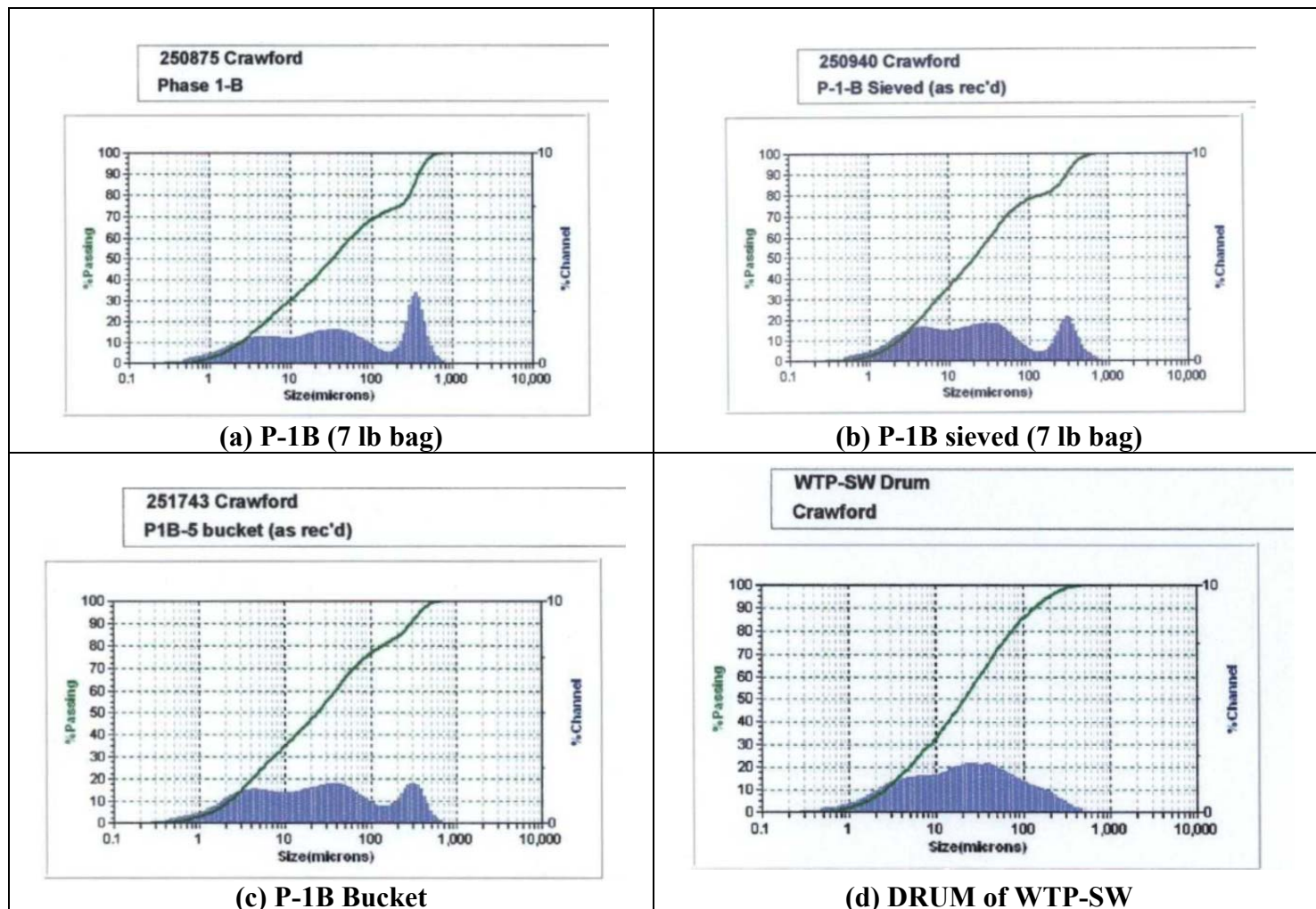
**Table 15. Relative Particle Size Distributions of the LAW and WTP-SW Blends**

Table 16 shows an estimate of the coal content analyzed by LOI data derived from heating the various blends in an oven under air at 525°C overnight. The LAW P1A blend gave higher LOI of ~ 9% versus the LAW P-1B of in the range of 0.8 to 1.7%. The WTP-SW blend showed the highest mass loss with an LOI of ~ 11%. Since all subsequent testing with the blends and monoliths used non heat-treated materials, these data shown in Table 16 were used throughout to adjust chemical compositions for the PCT normalized release calculations (described later in this report) since the major constituents of the coal (C,H,O) do not participate in the leaching.

**Table 16. LOI Data for Aggregates/Blends**

<b>Pilot Scale Campaign</b>	<b>Sample Description</b>	<b>Amount of (-)18 Mesh Material</b>	<b>Mass Loss by Ashing at 525°C</b>	<b>Coal Content*</b>	<b>Skeletal Density (g/cc)</b>
<b>LAW P-1-A</b>	7-kg bag (Dark/Grey powder)	12.152	1.107	9.11	NA
<b>LAW P-1-B</b>	7 kg bag (Tan/Sand-colored)	14.481	0.114	0.79	NA
<b>LAW P-1-B</b>	Bucket (Tan/Sand-colored)	9.858	0.17	1.72	2.39
<b>WTP-SW P-2-B</b>	Drum (Dark/Grey powder)	10.12	1.119	11.06	2.58

\* Ash Mass Loss Divided By Original Mass x 100

#### 4.4 LAW AND WTP-SW BLENDS

All of the PR and HTF aggregates/blends received from Hazen were analyzed for chemical composition. These blends were not roasted prior to dissolution to remove any carbon. Table 17 shows the elemental and calculated oxide weight percent (wt%) compositions on an as-reported basis (average data cells) and on a mass balance adjusted basis using the LOI data from Table 16 (average-adjusted cells) to capture the coal concentration present. Totals of the oxides and halides shown in the final columns of Table 17 indicate complete dissolution was achieved since the totals sum to the range of 99 – 105 wt%. These are typical summation ranges, i.e., 95 to 105%, for dissolution and analysis of complex multiple-component solids such as borosilicate waste glass and minerals given that the analytical uncertainty of the instrumentation (ICP-AES, IC-anions, ICP-MS) is nominally  $\pm 10\%$ . It should be noted that dissolution and analysis for sulfur constituents in these minerals showed similar trends as for the previous roasted bed products and fines. That is, the analyzed sulfate values were not in the expected ratio of the feeds, and the total elemental sulfur from the ICP-AES analyses did not bound sulfur calculated from the sulfate.



Table 17. Oxide Composition of Blends

Form	Sample	(wt%)	Ag <sub>2</sub> O	Al <sub>2</sub> O <sub>3</sub>	As <sub>2</sub> O <sub>3</sub>	B <sub>2</sub> O <sub>3</sub>	BaO	CaO	CdO	Cl	Cr <sub>2</sub> O <sub>3</sub>	Cs <sub>2</sub> O	F	Fe <sub>2</sub> O <sub>3</sub>	I	K <sub>2</sub> O	MgO	MnO <sub>2</sub>
7-kg bag	P-1A (A)	08-1712	0.03	31.56	<0.26	<0.32	0.11	0.06	0.04	0.18	0.10	0.27	<0.20	1.29	0.16	0.20	0.02	<0.02
	P-1A (B)	08-1712	0.03	31.37	<0.26	<0.32	0.11	0.07	0.04	0.19	0.10	0.24	<0.20	1.50	0.16	0.20	0.02	<0.02
	Average		0.03	31.46	<0.26	<0.32	0.11	0.06	0.04	0.18	0.10	0.26	<0.20	1.39	0.16	0.20	0.02	<0.02
	Average-Adjust		0.04	34.61	<0.26	<0.35	0.13	0.07	0.05	0.20	0.11	0.28	<0.22	1.53	0.18	0.22	0.02	<0.02
7-kg bag	P-1B (A)	08-1713	0.04	34.39	<0.26	<0.32	0.13	0.06	0.05	0.23	0.10	0.24	<0.20	1.72	0.18	0.23	0.02	<0.02
	P-1B (B)	08-1713	0.04	34.58	<0.26	<0.32	0.13	0.06	0.05	0.20	0.10	0.22	<0.20	1.57	0.18	0.22	0.02	<0.02
	Average		0.04	34.48	<0.26	<0.32	0.13	0.06	0.05	0.22	0.10	0.23	<0.20	1.64	0.18	0.23	0.02	<0.02
	Average-Adjust		0.04	34.76	<0.26	<0.32	0.13	0.06	0.05	0.22	0.10	0.23	<0.20	1.66	0.18	0.23	0.03	<0.02
Bucket	P-1B Bucket 1/5 (A)	08-1714	0.04	34.77	<0.26	<0.32	0.13	0.06	0.05	0.21	0.10	0.18	<0.20	2.29	0.18	0.22	0.02	<0.02
	P-1B Bucket 1/5 (B)	08-1714	0.04	34.96	<0.26	<0.32	0.13	0.06	0.05	0.22	0.10	0.18	<0.20	2.30	0.18	0.23	0.03	<0.02
	Average		0.04	34.86	<0.26	<0.32	0.13	0.06	0.05	0.21	0.10	0.18	<0.20	2.29	0.18	0.22	0.03	<0.02
	Average-Adjust		0.04	35.47	<0.26	<0.33	0.13	0.06	0.05	0.22	0.10	0.18	<0.20	2.33	0.18	0.23	0.03	<0.02
Drum	WTP-SW (A)	08-2055	0.04	29.22	<0.01	1.05	<0.01	0.15	0.02	0.86	0.11	0.35	0.39	4.91	0.02	0.43	0.08	<0.02
	WTP-SW (B)	08-2055	0.03	28.35	<0.01	1.01	<0.01	0.14	0.02	0.86	0.10	0.35	0.39	4.55	0.02	0.42	0.08	<0.02
	Average		0.04	28.78	<0.01	1.03	<0.01	0.15	0.02	0.86	0.11	0.35	0.39	4.73	0.02	0.43	0.08	0.02
	Average-Adjust		0.04	32.36	<0.01	1.16	<0.01	0.16	0.02	0.97	0.12	0.39	0.44	5.32	0.02	0.48	0.09	<0.02

Form	Sample	(wt%)	Na <sub>2</sub> O	NiO	PO <sub>4</sub>	PbO	ReO <sub>2</sub>	SO <sub>4</sub>	Sb <sub>2</sub> O <sub>3</sub>	SeO <sub>2</sub>	SiO <sub>2</sub>	SrO	TiO <sub>2</sub>	Tl	ZnO	Total
7-kg bag	P-1A (A)	08-1712	19.28	0.08	0.72	0.12	0.07	0.91	0.06	<0.01	37.01	<0.01	<1.05	<0.01	<0.01	93.51
	P-1A (B)	08-1712	18.87	0.07	0.72	0.12	0.07	0.91	0.07	<0.01	37.05	<0.01	<1.04	<0.01	<0.01	93.15
	Average		19.07	0.08	0.72	0.12	0.07	0.91	0.06	<0.01	37.03	<0.01	<1.05	<0.01	<0.01	93.33
	Average-Adjust		20.99	0.08	0.79	0.13	0.07	1.00	0.07	<0.01	40.74	<0.01	<1.15	<0.01	<0.01	102.68
7-kg bag	P-1B (A)	08-1713	19.95	0.10	0.81	0.16	0.05	1.55	0.08	<0.01	39.58	<0.01	<1.14	<0.01	<0.01	100.99
	P-1B (B)	08-1713	20.49	0.09	0.81	0.15	0.05	1.50	0.08	<0.01	40.00	<0.01	<1.11	<0.01	<0.01	101.87
	Average		20.22	0.09	0.81	0.16	0.05	1.53	0.08	<0.01	39.79	<0.01	<1.12	<0.01	<0.01	101.43
	Average-Adjust		20.38	0.10	0.81	0.16	0.05	1.54	0.08	<0.01	40.11	<0.01	<1.13	<0.01	<0.01	102.24
Bucket	P-1B Bucket 1/5 (A)	08-1714	20.89	0.10	0.81	0.16	0.04	1.47	0.08	<0.01	40.00	<0.01	<1.12	<0.01	<0.01	103.12
	P-1B Bucket 1/5 (B)	08-1714	20.62	0.10	0.83	0.17	0.04	1.46	0.08	<0.01	40.43	<0.01	<1.15	<0.01	<0.01	103.57
	Average		20.76	0.10	0.82	0.17	0.04	1.47	0.08	<0.01	40.22	<0.01	<1.13	<0.01	<0.01	103.34
	Average-Adjust		21.12	0.10	0.84	0.17	0.04	1.49	0.08	<0.01	40.92	<0.01	<1.15	<0.01	<0.01	105.15
Drum	WTP-SW (A)	08-2055	16.38	0.05	0.26	0.06	0.04	0.98	0.03	<0.01	34.00	<0.01	<1.06	<0.01	0.11	90.50
	WTP-SW (B)	08-2055	15.58	0.06	0.25	0.05	0.04	0.98	0.04	<0.01	32.67	<0.01	<1.02	<0.01	0.11	87.01
	Average		15.98	0.05	0.25	0.06	0.04	0.98	0.03	<0.01	33.34	<0.01	<1.04	<0.01	0.11	88.75
	Average-Adjust		17.96	0.06	0.28	0.06	0.05	1.10	0.04	<0.01	37.48	<0.01	<1.17	<0.01	0.12	99.79

Results of the 7-day PCT performed on the LAW P-1B and WTP-SW P-2B aggregates/ blends are shown in Table 18 along with the previous data from Table 13 for the separate bed and fines products. All as-measured leachate concentrations are shown in mg/L units for Al, C, I, Na, Re, S and Si along with the resulting leachate final pHs. The surface area of the PCT-prepared powders (100 – 200 mesh size) are also shown from BET measurements in units of  $\text{m}^2/\text{g}$ . All SA/V and normalized release data were calculated similarly to the PR and HTF PCT data using Equations 1 and 2. The adjusted elemental compositions for the aggregates/blends (coal-free basis) were used to normalize the PCT release for better comparison to the previous aggregate (coal removed by roasting) PCT data. The PR and HTF P-1B LAW data from Table 13 are green-scale highlighted to indicate the PCT response of the bed and fines and compared to the PCT response of the aggregate/blend (unshaded) for the LAW P-1B blend. Similarly, the PR and HTF P-2B data are blue-highlighted to indicate the individual PCT responses of the bed and fines compared to the aggregate used in the WTP-SW P-2B blend. Recall that the bed and fines had near-Gaussian-shaped particle size distributions with tails extending towards smaller particle sizes (as previously discussed in the 1<sup>st</sup> paragraph of Section 5.2), while the aggregate/blends have a bi-modal distribution. These deviations from normal Gaussian distribution of washed 100-200 mesh PCT powders makes particle size determination of the SA/V term more inaccurate. In addition, the aggregate/blends had coal in them whereas the test response for the bed and fines had the coal roasted out in order to compare the durability mechanism to previous testing.<sup>7</sup> It should be noted that initial BET surface areas measured for the blended aggregate samples were performed on PCT prepared powders with coal present. The BET SA determined for the LAW P-1B aggregate blend used a 3-hour evacuation at 105 °C. The BET SA determined for the WTP-SW P-2B aggregate blend used a 4-hour evacuation at 300 °C. The reported uncertainty for these BET SA data were both nominally 0.2 % relative standard deviation. These data are shown in Table 18 as ‘BET with Coal’. However, at a later date in follow-on programs within SRNL, these blended aggregate LAW and WTP-SW samples were also measured for BET surface area with the coal roasted out. These data are also shown in Table 18 as ‘BET w/out Coal’ for comparison. One can see that the BET SAs for roasted samples give nominally 20% lower SAs when the residual coal is roasted out. Since the SA/V term is in the denominator for Equation 2, these roasted lower BET SAs then increase the calculated normalized release data as shown in Table 18.

Normalized release data from Table 18 are plotted in Figure 5 to show comparison of the LAW and WTP-SW blends with the different aggregate samples. These releases are shown compared to the releases of the PR and HTF samples before aggregation. In general the individual normalized releases from the bed products and fines are similar to the blended aggregates. The  $\text{NL}_\text{S}$  are obviously the highest release in the range of 0.04 to 0.13  $\text{g}/\text{m}^2$ . All  $\text{NL}_\text{Na}$  are less than 0.02  $\text{g}/\text{m}^2$ . The  $\text{NL}_\text{Re}$  appears to be slightly higher for both the HTF fines and the WTP-SW blend. The  $\text{NL}_\text{Cs}$  approaches 0.04  $\text{g}/\text{m}^2$  for the WTP-SW blend and is < 0.02  $\text{g}/\text{m}^2$  for all other samples. One can also see from Figure 5 that the normalized release values for the non-roasted versus the roasted for both the LAW P-1B blend and the WTP-SW P-2B blend are indeed similar due to the ~ 20% difference in the measured (non-roasted versus roasted) BET surface areas.

It is obvious from the normalized release PCT data shown above in Figure 4 and below in Figure 5 that the  $\text{NL}(\text{S})$  values are typically highest compared to all other measured elements for the HTF fines (and the blended LAW and WTP-SW aggregates that were comprised of nominally 80% HTF fines). Also, as mentioned previously, the  $\text{NL}(\text{Re})$  values are higher for the HTF fines versus  $\text{NL}(\text{Re})$  in the PR bed products shown in Figure 4. One explanation for this relatively high release of both S and Re from these minerals is the relatively high REDOX associated with the HTF fines, as shown previously in Tables 8 through 10. At the measured REDOX of  $> 0.73 \text{ Fe}^{2+}/\Sigma\text{Fe}$ , it has been previously published<sup>23</sup> that only about 1% of the available S constituents in the minerals would be in the higher S valence state, i.e.,

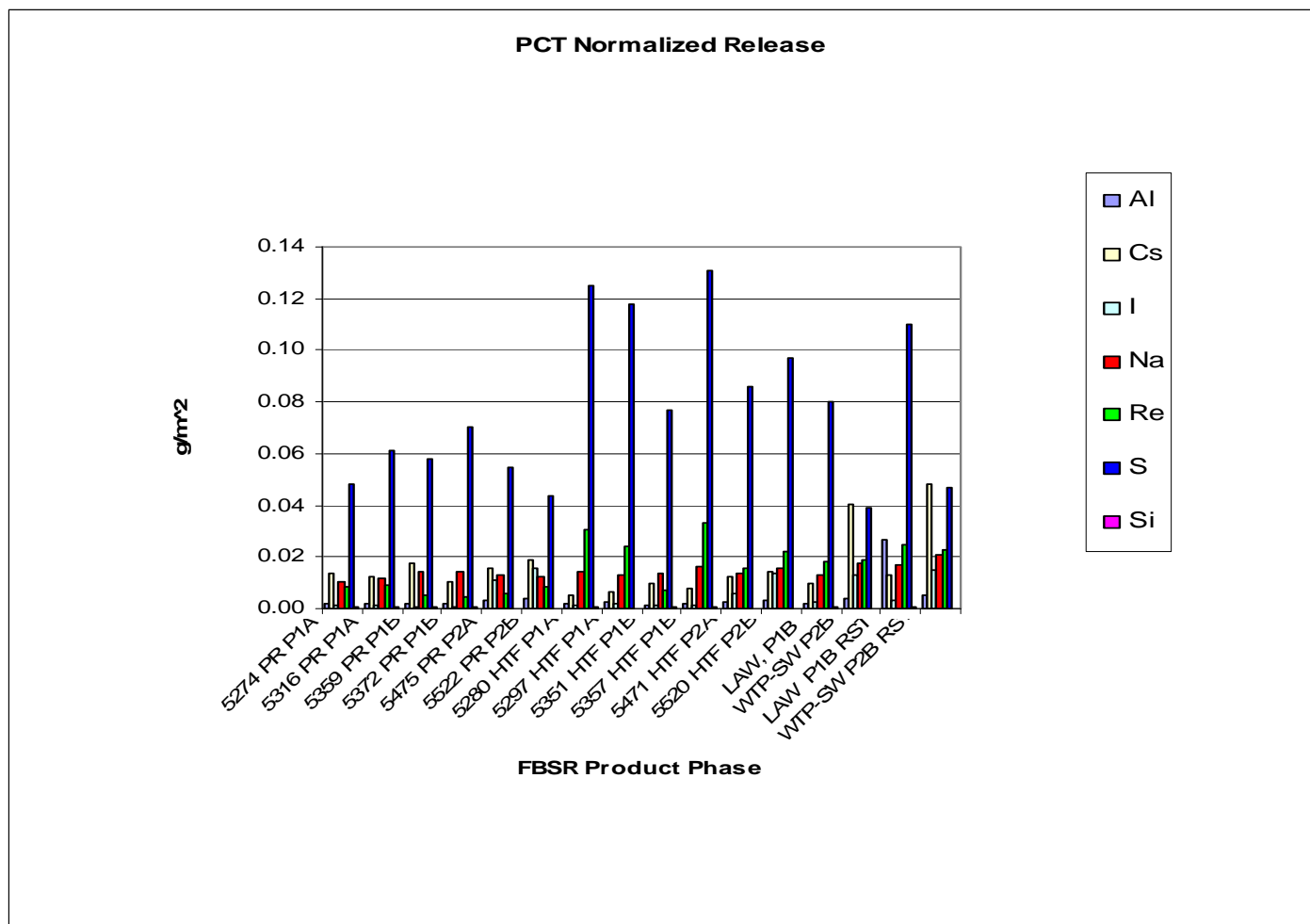
sulfate =  $\text{SO}_4^{2-}$  and only about 2.5% of the available Re constituents in the minerals would be in the higher Re valence state of +7. The correlation of the S and Re valence as well as other applicable elements with measured Fe REDOX derive from the EMF series experimental data measured by Shreiber.<sup>24</sup> The expected percentages of higher valence state S and Re are considerably higher (> 94 and > 86, respectively) for the PR bed products with measured REDOX in the range of 0.4 to 0.5  $\text{Fe}^{2+}/\Sigma\text{Fe}$ . This is further supported by the lack of any sulfate-containing nosean phases (sulfate contained in the mineral cage structure) observed in the XRD analyses for these HTF fines (data from Table 6 indicates that sodalite is present but not nosean). Subsequent BSR testing in SRNL using the WTP-SW simulants and SRNL radioactive feed streams shimmed to represent the WTP-SW matrix<sup>25</sup> as well as simulant and radioactive LAW feed streams<sup>26,27,28</sup> have thus targeted lower REDOX ranges for the product minerals in order to better partition the S as sulfate and to maintain Re at the higher valence of +7.

**Table 18. Normalized PCT Results for Aggregate/Blends Compared to Bed and Fines Tested Separately**

	As Measured Leachate Concentrations							pH	BET w/o COAL (m <sup>2</sup> /g)	BET WITH COAL (m <sup>2</sup> /g)	SA/V w/o COAL (m <sup>-1</sup> )	SA/V WITH COAL	Normalized Releases Calculated from Equations 1 & 2	
	Al (mg/L)	Cs (mg/L)	I (mg/L)	Na (mg/L)	Re (mg/L)	S (mg/L)	Si (mg/L)						NL <sub>Al</sub> (g/m <sup>2</sup> )	NL <sub>Cs</sub> (g/m <sup>2</sup> )
5351 HTF P-1B	146.1	6.9	0.5	985.8	1.8	283.3	49.8	11.52	5.26		525,904		1.6E-03	9.5E-03
5357 HTF P-1B	179.7	9.2	0.3	1145.6	3.8	355.0	51.1	11.66	4.76		475,670		2.1E-03	8.0E-03
5359 PR P-1B	133.7	7.4	1.0	902.0	1.5	216.7	45.1	11.45	4.11		410,570		1.9E-03	1.8E-02
5372 PR P-1B	138.1	5.5	0.8	956.7	1.5	260.0	49.4	11.54	4.36		436,000		1.9E-03	1.1E-02
LAW P-1B AGGREGATE	209.3	11.6	2.3	1138.7	3.7	224.9	64.9	11.77	4.17	5.62	417,000	562,000	2.0E-03 NR* 2.7E-03 R*	9.6E-03 NR 1.3E-02 R
5520 HTF P-2B	286.4	42.0	0.6	1057.9	3.8	209.2	10.9	9.98	5.15		515,200		3.2E-03	1.4E-02
5522 PR P-2B	237.2	11.7	0.5	676.1	2.2	88.8	12.2	10.64	3.80		380,000		4.0E-03	1.9E-02
WTP-SW P-2B AGGREGATE	355.7	73.0	1.3	1164.8	4.1	70.8	11.8	10.10	4.10	4.89	410,000	488,940	4.2E-03 NR 5.0E-03 R	4.1E-02 NR 4.8E-02 R

	Normalized Releases Calculated from Equations 1 & 2				
	NL <sub>I</sub> (g/m <sup>2</sup> )	NL <sub>Na</sub> (g/m <sup>2</sup> )	NL <sub>Re</sub> (g/m <sup>2</sup> )	NL <sub>S</sub> (g/m <sup>2</sup> )	NL <sub>Si</sub> (g/m <sup>2</sup> )
5351 HTF P-1B	1.1E-03	1.3E-02	7.0E-03	7.7E-02	5.1E-04
5357 HTF P-1B	1.0E-03	1.6E-02	3.3E-02	1.3E-01	5.6E-04
5359 PR P-1B	7.6E-04	1.5E-02	5.1E-03	5.8E-02	6.3E-04
5372 PR P-1B	8.6E-04	1.4E-02	4.8E-03	7.0E-02	6.6E-04
LAW P-1B AGGREGATE	2.3E-03 NR 3.1E-03 R	1.3E-02 NR 1.7E-02 R	1.8E-02 NR 2.5E-02 R	8.0E-02 NR 1.1E-01 R	6.0E-04 NR 8.1E-04 R
5520 HTF P-2B	1.4E-02	1.5E-02	2.2E-02	9.7E-02	1.1E-04
5522 PR P-2B	1.5E-02	1.3E-02	8.2E-03	4.4E-02	1.9E-04
WTP-SW P-2B AGGREGATE	1.3E-02 NR 1.5E-02 R	1.8E-02 NR 2.1E-02 R	1.9E-02 NR 2.3E-02 R	3.9E-02 NR 4.7E-02 R	1.4E-04 NR 1.6E-04 R

\* NR = nonroasted; R = roasted



**Figure 5. PCT Normalized Release for Aggregates and Blends**

The blended aggregates for LAW P-1B and WTP-SW P-2B were submitted for off-site certified TCLP testing and the results are shown in Table 19. The aggregates/blends were found to be above Universal Treatment Standard (UTS) limits for two elements; LAW P-1B for Sb and Cd and WTP SW P-2B for Sb. However, in the LAW P-1B sample Sb had been shimmed into the simulant at 10X the level anticipated in the Hanford tanks for off-gas testing purposes while in the WTP-SW P-2B sample the Sb had been doped in 48X the level anticipated in the Hanford tanks in order to be able to analyze for this element in the FBSR product. The LAW P-1B sample also was about 10X above the UTS (0.11 mg/L) limits for Cd which had been shimmed into the simulant at 10X. It should be noted that these Sb and Cd metals, as well as Ba, Se and Tl, were intentionally shimmed into the ESTD simulants at 10-1297X times the expected concentrations in the wastes for these simulants as shown in Table 19.<sup>1</sup>

**Table 19. TCLP Data for the Blends**

Element	P-2B WTP- SW TCLP Response	P-2B-WTP- SW Target (w 307 g clay/L)	P-1B LAW TCLP Response	P-1B LAW Target (w 640 g clay/L)	Reporting Limit (RL)	Method Detection Limit (MDL)	Universal Treatment Standards UTS 40CFR 268.48
	(mg/L)	(g/L)	(mg/L)	(g/L)	(mg/L)	(mg/L)	(mg/L)
Sb	<b>1.61</b>	0.175 (48X)	<b>2.13</b>	0.426 (10X)	0.1	0.03	<b>1.15</b>
As	<MDL	0.007	<MDL	0.949 (10X)	0.15	0.05	5
Ba	0.021 <sup>J</sup>	0.003 (42X)	0.283	1.37 (100X)	0.05	0.01	21
Cd	0.0122 <sup>J</sup>	0.088 (1297X)	<b>1.02</b>	0.381 (10X)	0.05	0.01	<b>0.11</b>
Cr	0.0708	0.282	<MDL	0.436	0.05	0.02	0.6
Pb	<MDL	0.244 (100X)	<MDL	1.013	0.1	0.025	0.75
Se	0.285	0.175 (16X)	0.373	0.078	0.15	0.05	5.7
Ag	<MDL	0.083 (1000X)	<MDL	0.14 (10X)	0.05	0.01	0.14
Hg	<MDL	Not added	<MDL	Not added	0.002	0.0003	0.025
Ni	0.0573	0.241 (100X)	0.567	0.502	0.05	0.01	11
Tl	<MDL	0.175 (29X)	<MDL	0.333 (10X)	0.2	0.05	0.2
Zn	0.0305 <sup>J</sup>	0.428	0.0379 <sup>J</sup>	Not added	0.1	0.02	4.3

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

## 4.5 MONOLITH TESTING WITH 2" CUBES

Analytic and durability test results for the LAW P-1B and WTP-SW P-2B aggregate/blends provided a basis for assessing the results from monoliths prepared by mixing the aggregates/blends with several different binders. Five different binder types were selected to prepare the 2" monolith cubes (see Figure 1) as given below and previously described in detail in Table 5:

- Ordinary Portland Cement (OPC), a very common binder consisting mainly of calcium silicates, calcium aluminates, and calcium aluminoferrate.
- High Alumina Cements, composed mainly of calcium aluminates rather than calcium silicates; these cements usually have less basic pore water than OPC.
- Geopolymers, which are amorphous to semi-crystalline, three-dimensional silico-aluminate polymers.
- Ceramicrete (ceramic cement), which is composed of magnesium phosphate.
- NuCap<sup>TM</sup> material, an advanced silicone geopolymer composite material developed by Global Matrechs, Inc.

Initial scoping tests were performed with LAW P1A and LAW P-1B from the initial 7-kg bags of blended material with different binders using small ~ 2" diameter polybottles and the 2" cubes. Details of all the various scoping tests and final 2" cube tests are shown in Table 20 to Table 24 for OPC, High Alumina Cements, Geopolymers, Ceramicrete and the NuCap<sup>TM</sup> material, respectively. Cure times were a minimum of 7 days. In some cases with duplicate cubes, the initial 7-day cured sample failed compression testing at < 500 psi, whereas the longer cured sample exceeded 500 psi. It should be noted that all data shown in Table 24 for the NuCap<sup>TM</sup> formulations span the size range of the 2" cubes up through the 3"x 6" and 6"x 12" cylinders because all of the NuCap<sup>TM</sup> monoliths were made over the course of only a few days with the NuCap<sup>TM</sup> product vendor at SRNL.

Appendix 5 shows the geopolymer formulations developed initially using 2" diameter polybottles. NuCap<sup>TM</sup> formulation data is shown in Appendix 6. NuCap<sup>TM</sup> Formulation

Data in Table 20 for the OPC monoliths show that the initial samples made with the 7-kg bag blends gave compression strengths above 1,000 psi on a 7-day cure, whereas higher loadings of 83 – 85% showed lower strengths of 500 to 740 psi. Later OPC monolith replicate sets that were tested at both 12 days and 28 days showed values in the range of 1630 to 1862 psi, respectively for the 80% loading, but decreased for similar time breaks down to 470 to 573 psi for the 85% loading. These data support the expected general trends regarding increased compressive strength with longer curing time (for a constant waste loading) and decreased compressive strength with increasingly higher waste loadings (for a constant cure time).

Data in Table 21 for the calcium alumina cements indicate that the binder alone with no FBSR gives compressive strengths of nominally 2,000 psi or greater. For both the SECAR-41 and FONDU systems, the initial lower waste loaded monoliths passed the 500 psi limit and the higher waste loaded monoliths (74 to 80%) failed that limit. The SECAR-71 monoliths passed 500 psi limit at both the 68 and 74% loadings, but failed at the higher 80% FBSR loadings. Data in Table 22 indicate that the Geopolymer monoliths were all above the 500 psi limit except for one of the Troy clay monoliths cured for 11 days and one of the fly ash monoliths cured for 10 days.

All FBSR waste loadings for the various binders are calculated on a wt% solids basis that does not include any water added. The cement binders only used three components comprised of the 1) FBSR powders, 2) the binder powders and 3) water. A sample calculation below shows the calculated wt% FBSR on a dry basis and the corresponding maximum wt% moisture (water) that could be in the monolith assuming no loss/evaporation during curing.

For OPC-1, the mass of FBSR is 132 g and binder is 33 grams and water is 92.81 grams. The wt% FBSR dry basis is thus  $[(132 \text{ g}) / (132 \text{ g} + 33 \text{ g})] \times 100 = 80\%$ . The maximum wt% water is  $[(92.81 \text{ g water}) / (92.81 \text{ g water} + 132 \text{ g FBSR} + 33 \text{ g binder})] \times 100 = 36 \text{ wt}\%$ .

The geopolymer formulations with heat-treated clay (GEO-1 through GEO-6) used FBSR powders, heat-treated clay, Silica-D sodium silicate liquid, sodium hydroxide solution and ASTM Type I water. Geopolymer formulations using fly ash did not commence until after the various geopolymer/clay trials in the 2" cubes had completed. Thus the final GEO-7 recipe used only the initial scoping ~ 2" diameter polybottles, i.e., they were not initially prepared in the 2" cubes. A generalized equation for calculating the wt% FBSR on a dry basis for the geopolymers is:

$$\left[ \frac{(mass_{FBSR})}{(mass_{FBSR} + mass_{clay} + mass_{Na_2O + SiO_2 \text{ from sodium silicate}} + mass_{Na_2O \text{ from NaOH}})} \right] * 100$$

Figure 6 shows the XRD spectra for the geopolymer metakaolin clays (top and middle spectra) or fly ash (bottom spectra) additives.



**Table 20. Ordinary Portland Cement Formulations with LAW**

Sample ID	Monolith Size	FBSR Source	Cure Time (days)	FBSR (g)	Binder (g)	Water (g)	Compression Testing (psi)	Wt% FBSR Dry Basis	Water: Solids Ratio	Water: Cement Ratio	Maximum wt% Moisture
<b>OPC-1</b>	2"x2"x2"	LAW	7	132	33	92.81	1,040	80.0	0.56	2.81	36
<b>OPC-2</b>		P-1A			23.064	83.09	500	85.1	0.54	3.60	35
<b>OPC-ALT</b>		(bag)			25.712	94.639	580	83.7	0.60	3.68	38
<b>OPC-1</b>	2"x2"x2"	LAW	7	132	33	92.81	1270	80.0	0.56	2.81	36
<b>OPC-2</b>		P-1B			23.064	83.09	740	85.1	0.54	3.60	35
<b>OPC-ALT</b>		(bag)			25.712	94.639	590	83.7	0.60	3.68	38
<b>OPC-1</b>	2"x2"x2"	LAW	12	132	33	92.81	1630	80.0	0.56	2.81	36
<b>OPC-1</b>		P-1B (bucket)	28				1862				
<b>OPC-2</b>	2"x2"x2"	LAW	12	132	23.064	83.09	470*	85.1	0.54	3.60	35
<b>OPC-2</b>		P-1B (bucket)	28				573				

\* Sample failed 500 psi compression limit

Table 21. Calcium Aluminate Cement Formulations with LAW

Sample ID	Monolith Size	FBSR Source	Cure Time (days)	FBSR (g)	Binder (g)	Water (g)	Compression Testing (psi)	Wt% FBSR Dry Basis	Water:Solids Ratio	Water: Cement Ratio	Max. wt% Moisture
SECAR-41 BLANK	2"x2"x2"	P-1B (bucket)	8	0	180	86.7	2660	0.0	0.48	0.48	32.5
SECAR-41-ALT			8	118	54	120	190*	68.6	0.70	2.22	41.1
SECAR-41-1			7	118.07	54.01	86.02	672	68.6	0.50	1.59	33.3
SECAR-41-2			7	155.03	54.03	104.53	286*	74.2	0.50	1.93	33.3
SECAR-41-2			15	155.03	54.03	104.53	340*	74.2	0.50	1.93	33.3
SECAR-41-ALT		P-1B (bag)	7	132	33	92.81	120*	80.0	0.56	2.81	36.0
SECAR-41-ALT		P-1B (bucket)	No Set	165	41.25	126.25	No Set	80.0	0.61	3.06	38.0
SECAR-71-ALT	2"x2"x2"	P-1A (bag)	7	165	28.83	113.86	100*	85.1	0.59	3.95	37.0
SECAR-71-BLANK		P-1B (bucket)		0	180	91.55	1900	0.0	0.51	0.51	33.7
SECAR-71-ALT				118	54	101	430*	68.6	0.59	1.87	37.0
SECAR-71-1		P-1B (bucket)	7	118.07	54.08	86.02	1120	68.6	0.50	1.59	33.3
SECAR-71-2		P-1B (bucket)	7	155.02	54.06	104.51	456*	74.1	0.50	1.93	33.3
SECAR-71-2			15				550				
SECAR-71-ALT	2"x2"x2"	P-1B (bag)	7	132	33	92.81	250*	80.0	0.56	2.81	36.0
SECAR-71-ALT		P-1B (bucket)		165	41.25	135	80*	80.0	0.65	3.27	39.6
SECAR-71-ALT		P-1B (bag)	8	132	23.06	83.08	70*	85.1	0.54	3.60	34.9
FONDU-ALT	2"x2"x2"	P-1A (bag)	7	165	28.83	93.09	70*	85.1	0.48	3.23	32.4
FONDU-BLANK		P-1B (bucket)		0	180	74.5	6190	0.0	0.41	0.41	29.3
FONDU -1				118	54	83.14	770	68.6	0.48	1.54	32.6
FONDU -1				118.05	54.05	81.42	840		0.47	1.51	32.1
FONDU -2		7	155.04	54.01	100.5	490*	74.2	0.48	1.86	32.5	
FONDU-ALT		15	155.04	54.01	100.5	460*		0.48	1.86	32.5	
FONDU-ALT		P-1B (bag)	7	132	33	89.09	160*	80.0	0.54	2.70	35.1
FONDU-ALT		P-1B (bucket)		165	41.25	103.12	240*		0.50	2.50	33.3
FONDU-ALT		P-1B (bag)	9	132	23.06	83.08	100*	85.1	0.54	3.60	34.9

\* Samples failed the 500 psi compression limit

**Table 22. Geopolymer Formulations with LAW**

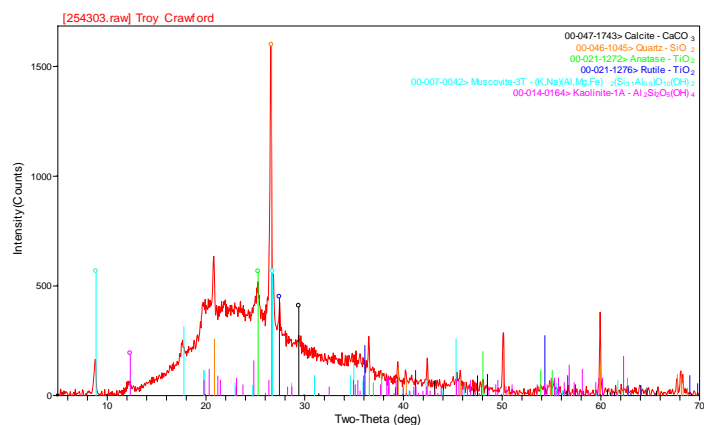
Sampl ID	Monolith Size	FBSR Source	Date Made	Geopolymer Alumino-silicate Source	FBSR (g)	Clay (g)	Silica D (g) / (Na <sub>2</sub> O+SiO <sub>2</sub> ) (g)	NaOH (g) /Na <sub>2</sub> O (g)	Water (g)	Wt% FBSR Dry Basis	Compression Testing (psi)	Cure Time (days)
GEO-1	2x2x2	P-1B (1 of 5)	9/11/2008	Troy Metakaolin (30%)	176.04	34.15	104.1/45.91	12.28/4.76	34.3	<b>67.5</b>	1510	11
GEO-2					176.09	23.53	91.43/40.32	10.82/4.19	34.86	<b>72.1</b>	860	
GEO-3					176.04	24.03	124.43/54.87	14.72/5.70	18.79	<b>67.5</b>	1270	
GEO-4					176.09	11.85	118.1/52.08	14.05/5.44	17.86	<b>71.7</b>	<b>410</b>	
GEO-5			9/22/2008	Barden Metakaolin clay	166.5	27.02	153.84/67.84	10.26/3.18	7.42	<b>62.9</b>	950	7
GEO-6					166.5	27.02	120.63/53.20	8.04/2.49	28.83	<b>66.8</b>	1080	
GEO-7-Z	~ 2" diameter Polybottle	P-1B (2 of 5)	10/9/2008	68.3%, SEFA Class F	100	27.03	45.62/20.12	28.69/8.89	5.41	<b>64.1</b>	1121	14
GEO-7-ZZ				68.8%, SEFA Class F		27.03	37.62/16.59	31.29/9.70	7.78	<b>65.2</b>	1475	
GEO-7-ZZZ			10/13/2008	72.2%, SEFA Class F		27.02	21.62/9.53	20.49/6.35	6.71	<b>70.0</b>	<b>72</b>	10
GEO-7-Z ANL				68.3%, ANL fly ash		27.03	45.62/20.12	29.44/9.12	5.41	<b>64.0</b>	1492	
GEO-7-ZZ ANL				68.8%, ANL fly ash		27.03	37.62/16.59	31.29/9.70	8.76	<b>65.2</b>	2872	
GEO-7-ZZZ ANL				72.2%, ANL fly ash		27.02	21.62/9.53	20.49/6.35	26.71	<b>70.0</b>	3159	

**Table 23. Ceramicrete Formulations with LAW**

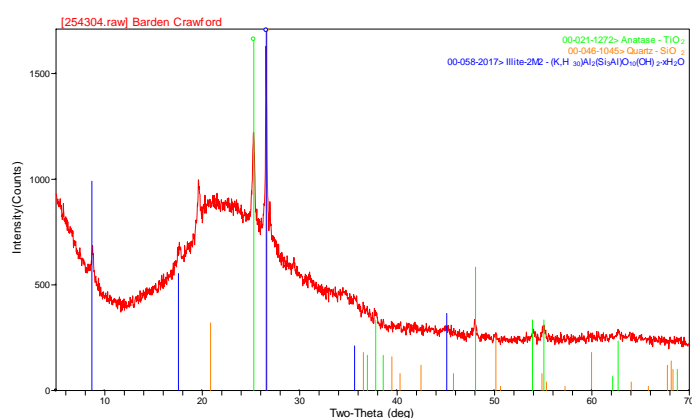
	Monolith	FBSR Source	Cure Time (days)	FBSR (g)	MgO (g)	KH <sub>2</sub> PO <sub>4</sub> (g)	Water (g)	Boric Acid (g)	Compression Testing (psi)	wt% FBSR	wt% FBSR (cal'c w/ Boric Acid)	Maximum wt% Moisture
CER-ALT	2"x2"x2"	P-1B (bucket)	13	176.37	40.31	136.07	90	0	2920	50.0	NA	20
CER-ALT				352.75					510	66.7		15
CER-ALT			8	176.37	40.31	136.07	90	13.2	2500	50.0	48.2	20
CER-1				352.75				17.6	520	66.7	64.5	14
CER-2			7	176.06	15.04	51.01	82.52	12.02	410	72.7	69.3	25
CER-2			14						360			
CER-2			28						550			

**Table 24. NuCap™ Formulations with LAW**

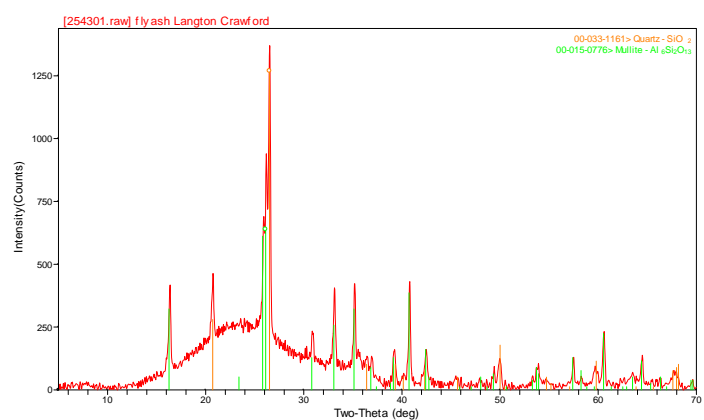
Sample ID	Monolith Size	FBSR Source	Cure Time (days)	Mass Ratio of Binder to FBSR	Compression Testing (psi)	Total Deflection (inches)	Rebound (inches)
NuCaP-ALT	2x2x2	P-1A (bag)	7	1 Binder to 1 FBSR	140	-0.38	0.27
NuCaP -ALT		P-1B (bag)			180	-0.74	0.32
NuCaP -ALT	3x6	P-1B (bag)	53		260	-1.75	1.25
NuCaP -ALT	6x12	P-1B (bucket)	Not Tested		Not Tested (archived)	Not Tested	Not Tested
NuCaP -1	2x2x2	P-1A (bag)	34	1.2 Binder to 1 FBSR	1250	-1.5	0.308
NuCaP -ALT		P-1B (bag)	7		490	-1.5	0.16
NuCaP -ALT	3x6	P-1B (bag)	53		320	-2.125	1.32
NuCaP -ALT	6x12	P-1B (bucket)	Not Tested		Not Tested (archived)	Not Tested	Not Tested



(a)



(b)



(c)

**Figure 6. XRD Spectra of (a) Troy, (b) Barden Heat-Treated Metakaolin Clays, and (c) SEFA Class F Fly ash Used as Geopolymer Starting Materials (Binders)**

Appendix 7 shows the various chemical compositions of the ordinary Portland cement, high alumina cement binders and the Class F fly ash (SEFA Group, Lexington, SC) after dissolution. These elemental values were determined from dissolution of the various binders and an ICP-AES scan of the metals present. The OPC binder contains Ca, Si, Al and Fe, with detectable quantities of K, Mg, S and Ti. The high-aluminum binders are mostly Al and Ca with small amounts of Fe, K, Mg, Si and Ti present. The fly ash contains Al and Si at higher levels than Fe and K, with detectable amounts of Ca, Cr, Mg, Na, P, Sr, Ti and Zr.

Geopolymers were all initially made using Troy metakaolin clay that resulted in the GEO-1 – GEO-4 cubes. A Barden heat-treated clay was also used near the end of geopolymer scope testing that resulted in the GEO-5 and GEO-6 sample.

Appendix 8 shows the mass loss determined from heat-treating the Barden clay.

All scoping formulations and 2” cube monoliths were tested for compressive strength versus the 500 psi limit. Two formulations from each binder type (except for the NuCap™ single formulation) in the 2” cubes were sent forward for further TCLP and PCT testing to include chemical composition.

Table 25 shows the 2” cube formulations along with their respective dry basis FBSR loading, compression test results, cure times, bulk densities, BET SA, skeletal densities and TCLP summary. No 2” cube data are available for the final GEO-7 formulation because it was under development in the 2” polybottle stage as the 2” cube testing series was completed. It can be seen from the compression data that increased FBSR loading resulted in loss of strength for all cement (OPC, FON, S41 and S71) formulations. This same trend of decreasing monolith strength with increased FBSR loading was observed in the Troy clay GEO-1&2 and GEO 3&4 series. Increasing the FBSR loading in the Barden clay GEO 5&6 samples as well as the Ceramicrete 1&2 samples did not significantly change (lower) the measured compressive strengths. Note that many of the curing times were 7-11 days rather than 28 days. Most of the cure times of significantly less than 28 days were necessary due to project time restrictions for moving on to the next larger monolith phase of the testing per Figure 1. It can be seen from Table 25 that a few of the higher waste loading cubes did not pass the 500 psi limit (FON-2, S41-2 and GEO-4). However, these samples were included in the durability test screening along with the other monoliths. The BET SA data shown in Table 25 was determined from the non-roasted PCT powders from these crushed monoliths, and will be discussed in further detail in the PCT data section below.

Detailed TCLP data for these 2” cube monoliths are shown in Appendix 9.

All cement formulations passed the TCLP. The six geopolymer formulations, the higher loaded ceramicrete and the NuCap™ samples all showed higher than UTS limits for certain of the elements Sb and Cd, as was seen with the original blends due to the shimmed levels previously shown in Table 19.

**Table 25. Successful Monolith 2" Cube Data Based on Compressive Strength and TCLP**

Binder Type	Monolith 2" Cube	Nominal Wt% FBSR Loading	Calculated Wt% FBSR Loading	Compressive Strength (psi)	Cure Time (days)	Bulk Monolith Density (g/cc)	BET Surface Area (m <sup>2</sup> /g)	Skeletal Density (g/cc)	TCLP Result
OPC	OPC-1	80%	80.0	1,630	12	1.64	31.5	1.81	Pass
	OPC-2	87%	85.1	573	28	1.61	21.3	2.05	
High Al Cement	FON-1	68%	68.6	770	7	1.77	20.0	2.19	
	FON-2	74.16%	74.2	490	7	1.75	15.5	2.20	
	S41-1	68.6%	68.6	672	7	1.75	12.2	2.18	
	S41-2	74.16%	74.2	340	15	1.70	10.7	2.18	
	S71-1	68.6%	68.6	1,120	7	1.70	13.1	2.11	
	S71-2	74.16%	74.2	550	15	1.65	9.2	2.12	
Geopolymer	GEO-1	67 %	67.5	1,510	11	1.87	15.2	2.21	Note 2
	GEO-2	72%	72.1	860	14 (11)	1.87	17.3	2.11	Note 2
	GEO-3	67%	67.5	1,270	11	1.81	10.9	2.20	Note 3
	GEO-4	71%	71.2	410	11	1.84	6.2	2.27	Note 1
	GEO-5	63%	62.9	950	7	1.88	10.6	2.00	Note 1
	GEO-6	66%	66.8	1,080	7	1.82	10.0	2.22	Note 1
Ceramicrete	CER-1	67%	66.7/64.5	520	8	1.81	32.2	2.14	Note 1
	CER-2	73%	72.7/69.3	550	28	1.81	27.7	2.25	Pass
NuCap <sup>TM</sup>	NUCAP-1	>45.5%*	(see footnote)	1,250	34	1.44	0.09	1.58	Note 2

Notes: 1) Did not pass UTS for Sb

2) Did not pass UTS for Cd

3) Did not pass UTS for Cd and Sb

\* Since the NuCap<sup>TM</sup> binder paste is an insoluble solids-containing liquid, i.e., a 'slurry', then wt% FBSR loading on dry basis is  $> 45.5\text{wt}\% (1 / (1 + (<)1.2)) = > 0.45$

Using post-compression tested 2" cube monoliths, all samples were prepared for PCT by grinding/sieving and ethanol washing followed by overnight drying at 90°C. The resulting dried powders were submitted for chemical composition (duplicate analyses) and PCT leach testing (duplicate leach tests). Portions of the dried powders were also analyzed for non-roasted BET surface area and particle size distribution analysis. Table 26 through Table 30 show the nominal oxide compositions of the monoliths as calculated from the measured elemental concentrations of the dissolved solids.

Portions (chunks and small-size pieces) of the post-compression tested 2" cube monoliths were also oven-dried at ~ 250°C overnight to determine the overall moisture content of the monoliths that could be removed under these conditions as often structural waters are bound to the hydrated minerals and require a slightly higher temperature than just removing pore water, e.g. LOD at 110°C. These data are shown in Table 31. The ceramicretes show 19 – 21 wt% moisture loss, the OPCs show 6 – 22 wt% moisture loss, the calcium aluminate cements show 12 to 21 wt% moisture loss and the geopolymers show 17 to 21 wt% moisture loss. The measured OPC-1 moisture loss appears to be biased low since the waste loading of this recipe was not significantly different from the OPC-2. This suspected low-bias moisture content of OPC-1 results in a low total adjusted oxide sum value of only 80% in Table 26. The measured value for the NuCap<sup>TM</sup> sample is the lowest at only 2 wt% measured moisture loss. Comparison of the measured moisture content of the crushed monolith pieces versus the calculated maximum moisture content shown in the last column of Table 31 indicates that measured values are lower than the calculated maximum moisture obtained from the monolith recipes. However, some of the added water in the monoliths obviously is lost to evaporation during ambient curing of the samples for 7 days or longer.

The measured 250 °C moisture data was then used to adjust the average chemical compositions to a dry-basis (anhydrous) as shown in Table 26 through Table 30. Further adjustment of the monolith chemical compositions was performed to account for the carbon/coal content of the monoliths that was added as part of the FBSR LAW P-1B (LOI previously shown in Table 16 to be 1.72 st%). These data are also

shown in Table 26 to Table 30 as 'FBSR'. Sample calculations for the FONDU-1 sample are shown below.

Calculate wt% of element (i) on dry basis:

Input: measured moisture content of FONDU-1 = 15 wt% (on drying at 250°C overnight)

$X \text{ mg of } i / \text{kg solids} / (1 - (15 \text{ wt\% moisture} / 100)) = X \text{ mg of } i / 0.85 \text{ kg solids dry basis}$

Calculate wt% of element (i) on dry basis and carbon-free basis:

Input: ► calculated wt% loading on dry basis for FONDU-1 = 68.6 wt%

► measured wt% carbon content of LAW P-1B = 1.72 wt% (from loss on ignition (LOI) at 525°C overnight)

$X \text{ mg of } i / 0.85 \text{ kg solids dry/carbon-free basis} =$

$$X \text{ mg of } i / [(0.85 \text{ kg solids} \times 0.686 \times (1 - 0.0172)) + (0.85 \text{ kg solids} \times (1 - 0.686))]$$

In the equation above the first part of the denominator (underlined) represents the dry basis solids portion containing FBSR and the second part of denominator represents all other dry basis solids in the monolith. Since the LAW P-1B only contained 1.72 wt% carbon, the correction to the elemental fraction in the monolith is relatively minor (i.e., relative to the 15 wt% correction for moisture in the FONDU-1 monolith).

Dissolution and chemical composition analyses for the NuCap<sup>TM</sup> monolith material likely resulted in incomplete dissolution of the matrix judging from the extremely low sum of oxides shown in Table 30 of only ~ 38 wt%. All of the major FBSR elements of Al, Na and Si are measured lower than what would be expected in a > 45 wt% dry basis loading in the NuCap<sup>TM</sup> monolith. Another contributing factor could be that the primary slurry additive used to form the NuCap<sup>TM</sup> binder consists mainly of limestone (CaCO<sub>3</sub>). No attempts were made to measure carbonate from the dissolved NuCap<sup>TM</sup> matrix which may account for the low recoveries given in Table 30.



**Table 26. Chemical Composition (wt%) of the 2" Cubes with LAW FBSR and OPC Corrected for Moisture and Coal**

Sample ID	Lab ID	Ag <sub>2</sub> O	Al <sub>2</sub> O <sub>3</sub>	As <sub>2</sub> O <sub>3</sub>	B <sub>2</sub> O <sub>3</sub>	BaO	CaO	CdO	Cl	Cr <sub>2</sub> O <sub>3</sub>	Cs <sub>2</sub> O	F	Fe <sub>2</sub> O <sub>3</sub>	I	K <sub>2</sub> O
	(wt%)														
OPC-1 (A)	08-1827	0.04	19.13	<0.0132	<0.32	0.08	13.25	0.02	0.06	0.06	0.15	0.08	1.51	0.044	0.20
OPC-1 (B)	08-1827	0.04	19.19	<0.0132	<0.32	0.08	13.22	0.02	0.06	0.06	0.15	0.08	1.53	0.044	0.20
	Avg.	0.04	19.16	<0.0132	<0.32	0.08	13.23	0.02	0.06	0.06	0.15	0.08	1.52	0.044	0.20
	Anhyd.	0.04	20.44	<0.0132	<0.34	0.09	14.11	0.02	0.06	0.07	0.16	0.08	1.62	0.047	0.21
	FBSR	0.04	20.72	<0.0132	<0.35	0.09	14.31	0.02	0.06	0.07	0.16	0.08	1.64	0.048	0.22
OPC-2 (A)	08-1828	0.03	23.50	<0.0132	<0.32	0.10	11.59	0.03	0.08	0.08	0.19	0.07	1.27	0.059	0.24
OPC-2 (B)	08-1828	0.03	23.37	<0.0132	<0.32	0.10	11.61	0.03	0.08	0.08	0.19	0.07	1.23	0.059	0.25
	Avg.	0.03	23.44	<0.0132	<0.32	0.10	11.60	0.03	0.08	0.08	0.19	0.07	1.25	0.059	0.25
	Anhyd.	0.04	30.15	<0.0132	<0.41	0.13	14.92	0.04	0.10	0.10	0.24	0.09	1.61	0.076	0.32
	FBSR	0.04	30.60	<0.0132	<0.42	0.13	15.14	0.04	0.11	0.10	0.25	0.09	1.63	0.078	0.32

Sample ID	Lab ID	MgO	MnO <sub>2</sub>	Na <sub>2</sub> O	NiO	PO <sub>4</sub>	PbO	ReO <sub>2</sub>	SO <sub>4</sub>	Sb <sub>2</sub> O <sub>3</sub>	SeO <sub>2</sub>	SiO <sub>2</sub>	TiO <sub>2</sub>	Total
	(wt%)													
OPC-1 (A)	08-1827	0.21	<0.02	10.32	0.06	0.45	0.11	0.018	1.17	0.06	<0.014	27.53	0.77	74.86
OPC-1 (B)	08-1827	0.21	<0.02	10.50	0.05	0.45	0.11	0.018	1.17	0.05	<0.014	27.37	0.77	74.96
	Avg.	0.21	<0.02	10.41	0.05	0.45	0.11	0.018	1.17	0.06	<0.014	27.45	0.77	74.91
	Anhyd.	0.23	<0.02	11.10	0.06	0.48	0.12	0.019	1.25	0.06	<0.014	29.27	0.82	79.89
	FBSR	0.23	<0.02	11.26	0.06	0.48	0.12	0.019	1.27	0.06	<0.014	29.68	0.83	81.00 <sup>a</sup>
OPC-2 (A)	08-1828	0.21	<0.02	12.82	0.07	0.58	0.13	0.020	1.04	0.06	<0.014	31.86	0.89	84.66
OPC-2 (B)	08-1828	0.21	<0.02	12.94	0.08	0.58	0.13	0.020	1.04	0.06	<0.014	31.58	0.90	84.36
	Avg.	0.21	<0.02	12.88	0.07	0.58	0.13	0.020	1.04	0.06	<0.014	31.72	0.89	84.51
	Anhyd.	0.27	<0.02	16.57	0.09	0.75	0.17	0.026	1.34	0.07	<0.014	40.80	1.15	108.71
	FBSR	0.27	<0.02	16.82	0.09	0.76	0.17	0.026	1.36	0.08	<0.014	41.41	1.17	110.33

a) Sum of oxides for OPC-1 likely biased low due to low bias measured wt% moisture in this crushed monolith – see text.

**Table 27. Chemical Composition (wt%) of 2" Cubes with LAW FBSR and Calcium Aluminate Cements Corrected for Moisture and Coal**

Sample ID	Lab ID														
	(wt%)	Ag <sub>2</sub> O	Al <sub>2</sub> O <sub>3</sub>	As <sub>2</sub> O <sub>3</sub>	B <sub>2</sub> O <sub>3</sub>	BaO	CaO	CdO	Cl	Cr <sub>2</sub> O <sub>3</sub>	Cs <sub>2</sub> O	F	Fe <sub>2</sub> O <sub>3</sub>	I	K <sub>2</sub> O
FON-1 (A)	08-1830	0.03	26.28	<0.0132	<0.32	0.08	12.93	0.02	0.06	0.10	0.16	0.07	5.44	0.049	0.16
FON-1 (B)	08-1830	0.03	25.86	<0.0132	<0.32	0.08	12.56	0.02	0.06	0.10	0.16	0.07	5.57	0.049	0.16
	Avg.	0.03	26.07	<0.0132	<0.32	0.08	12.74	0.02	0.06	0.10	0.16	0.07	5.51	0.049	0.16
	Anhyd.	0.04	30.66	<0.0132	<<0.38	0.09	14.99	0.02	0.07	0.12	0.19	0.08	6.48	0.057	0.19
	FBSR	0.04	31.03	<0.0132	<0.38	0.09	15.17	0.02	0.07	0.12	0.19	0.08	6.55	0.058	0.19
FON-2 (A)	08-2057	0.02	25.48	<0.0132	<0.32	0.08	8.95	0.03	0.07	0.08	0.16	<0.05	4.08	0.057	0.19
FON-2 (B)	08-2057	0.02	26.36	<0.0132	<0.32	0.08	9.18	0.03	0.07	0.08	0.16	<0.05	4.20	0.057	0.20
	Avg.	0.02	25.92	<0.0132	<0.32	0.08	9.07	0.03	0.07	0.08	0.16	<0.05	4.14	0.057	0.20
	Anhyd.	0.03	33.15	<0.0132	<0.41	0.10	11.60	0.03	0.08	0.11	0.21	<0.06	5.30	0.073	0.25
	FBSR	0.03	33.58	<0.0132	<0.42	0.10	11.75	0.03	0.09	0.11	0.21	<0.06	5.37	0.074	0.25
S41-1 (A)	08-1831	0.03	29.02	<0.0132	<0.32	0.09	12.30	0.02	<0.05	0.08	0.16	0.10	2.68	0.037	0.18
S41-1 (B)	08-1831	0.03	28.94	<0.0132	<0.32	0.09	12.24	0.02	<0.05	0.08	0.16	0.10	2.72	0.037	0.18
	Avg.	0.03	28.98	<0.0132	<0.32	0.09	12.27	0.02	<0.05	0.08	0.16	0.10	2.70	0.037	0.18
	Anhyd.	0.03	36.49	<0.0132	<0.41	0.11	15.45	0.02	<0.06	0.10	0.21	0.12	3.40	0.047	0.23
	FBSR	0.03	36.93	<0.0132	<0.41	0.11	15.64	0.02	<0.06	0.10	0.21	0.12	3.44	0.047	0.23
S41-2 (A)	08-2056	0.02	27.17	<0.0132	<<0.32	0.08	8.89	0.02	0.06	0.07	0.15	<0.05	2.27	0.068	0.20
S41-2 (B)	08-2056	0.02	28.01	<0.0132	<0.32	0.08	9.17	0.03	0.06	0.07	0.15	<0.05	2.35	0.068	0.22
	Avg.	0.02	27.59	<0.0132	<0.32	0.08	9.03	0.02	0.06	0.07	0.15	<0.05	2.31	0.068	0.21
	Anhyd.	0.03	31.68	<0.0132	<0.37	0.09	10.37	0.03	0.07	0.08	0.18	<0.06	2.65	0.078	0.24
	FBSR	0.03	32.09	<0.0132	<0.37	0.09	10.50	0.03	0.07	0.08	0.18	<0.06	2.68	0.079	0.25
S71-1 (A)	08-1832	0.03	34.09	<0.0132	<0.32	0.08	9.57	0.02	0.05	0.06	0.16	<0.05	0.54	0.043	0.19
S71-1 (B)	08-1832	0.03	34.20	<0.0132	<0.32	0.08	9.71	0.02	0.05	0.06	0.16	<0.05	0.54	0.043	0.19
	Avg.	0.03	34.15	<0.0132	<0.32	0.08	9.64	0.02	0.05	0.06	0.16	<0.05	0.54	0.043	0.19
	Anhyd.	0.03	40.40	<0.0132	<0.38	0.09	11.40	0.02	0.06	0.07	0.19	<0.06	0.64	0.051	0.22
	FBSR	0.03	40.88	<0.0132	<0.39	0.09	11.54	0.02	0.06	0.07	0.19	<0.06	0.65	0.051	0.23
S71-2 (A)	08-2054	0.03	32.56	<0.0132	<0.32	0.08	5.16	0.04	0.13	0.07	0.12	<0.05	1.46	0.107	0.15
S71-2 (B)	08-2054	0.03	33.18	<0.0132	<0.32	0.08	5.32	0.04	0.13	0.07	0.12	<0.05	1.65	0.107	0.16
	Avg.	0.03	32.87	<0.0132	<0.32	0.08	5.24	0.04	0.13	0.07	0.12	<0.05	1.56	0.107	0.15
	Anhyd.	0.04	40.09	<0.0132	<0.39	0.09	6.39	0.04	0.16	0.08	0.15	<0.06	1.90	0.130	0.19
	FBSR	0.04	40.61	<0.0132	<0.40	0.09	6.47	0.04	0.17	0.08	0.15	<0.06	1.92	0.132	0.19

Table 27. Chemical Composition (wt%) of 2" Cubes with LAW FBSR and Calcium Aluminate Cements Corrected for Moisture and Coal, continued

Sample ID	Lab ID	MgO	MnO <sub>2</sub>	Na <sub>2</sub> O	NiO	PO <sub>4</sub>	PbO	ReO <sub>2</sub>	SO <sub>4</sub>	Sb <sub>2</sub> O <sub>3</sub>	SeO <sub>2</sub>	SiO <sub>2</sub>	TiO <sub>2</sub>	Total
	(wt%)													
FON-1 (A)	08-1830	0.27	0.09	10.55	0.06	0.50	0.11	0.021	0.79	0.05	<0.014	23.51	1.18	82.26
FON-1 (B)	08-1830	0.27	0.09	10.52	0.06	0.49	0.11	0.021	0.79	0.05	<0.014	23.62	1.18	81.66
	Avg.	0.27	0.09	10.53	0.06	0.49	0.11	0.021	0.79	0.05	<0.014	23.57	1.18	81.96
	Anhyd.	0.31	0.11	12.39	0.07	0.58	0.13	0.025	0.92	0.06	<0.014	27.72	1.39	96.40
	FBSR	0.32	0.11	12.53	0.07	0.59	0.13	0.025	0.93	0.06	<0.014	28.05	1.40	97.55
FON-2 (A)	08-2057	0.19	<0.02	11.56	0.05	0.47	0.11	0.027	0.74	0.05	<0.014	24.64	1.03	77.92
FON-2 (B)	08-2057	0.19	<0.02	11.82	0.06	0.50	0.12	0.027	0.74	0.05	<0.014	25.36	1.07	80.27
	Avg.	0.19	<0.02	11.69	0.06	0.48	0.12	0.027	0.74	0.05	<0.014	25.00	1.05	79.10
	Anhyd.	0.25	<0.02	14.95	0.07	0.62	0.15	0.035	0.95	0.07	<0.014	31.98	1.34	101.18
	FBSR	0.25	<0.02	15.15	0.07	0.63	0.15	0.036	0.96	0.07	<0.014	32.39	1.36	102.48
S41-1 (A)	08-1831	0.17	0.02	10.39	0.07	0.48	0.11	0.017	0.57	0.05	<0.014	24.57	1.25	82.38
S41-1 (B)	08-1831	0.17	0.02	10.51	0.06	0.48	0.11	0.017	0.57	0.05	<0.014	24.64	1.24	82.46
	Avg.	0.17	0.02	10.45	0.06	0.48	0.11	0.017	0.57	0.05	<0.014	24.60	1.25	82.42
	Anhyd.	0.21	0.03	13.16	0.08	0.61	0.14	0.021	0.72	0.06	<0.014	30.98	1.57	103.78
	FBSR	0.21	0.03	13.32	0.08	0.61	0.14	0.021	0.73	0.06	<0.014	31.35	1.59	105.02
S41-2 (A)	08-2056	0.12	<0.02	11.55	0.06	0.46	0.11	0.026	0.73	0.05	<0.014	25.06	1.08	78.10
S41-2 (B)	08-2056	0.13	<0.02	12.00	0.05	0.47	0.12	0.026	0.73	0.05	<0.014	25.84	1.14	80.63
	Avg.	0.12	<0.02	11.78	0.05	0.47	0.11	0.026	0.73	0.05	<0.014	25.45	1.11	79.37
	Anhyd.	0.14	<0.02	13.53	0.06	0.54	0.13	0.030	0.83	0.06	<0.014	29.23	1.27	91.14
	FBSR	0.14	<0.02	13.70	0.06	0.54	0.13	0.030	0.85	0.06	<0.014	29.60	1.29	92.32
S71-1 (A)	08-1832	0.16	<0.02	10.36	0.08	0.52	0.11	0.017	0.72	0.05	<0.014	22.52	0.66	79.88
S71-1 (B)	08-1832	0.15	<0.02	10.33	0.05	0.52	0.11	0.017	0.72	0.04	<0.014	22.45	0.66	79.97
	Avg.	0.15	<0.02	10.35	0.07	0.52	0.11	0.017	0.72	0.04	<0.014	22.48	0.66	79.92
	Anhyd.	0.18	<0.02	12.24	0.08	0.62	0.13	0.020	0.85	0.05	<0.014	26.60	0.78	94.55
	FBSR	0.18	<0.02	12.38	0.08	0.62	0.13	0.021	0.86	0.05	<0.014	26.92	0.79	95.68
S71-2 (A)	08-2054	0.07	<0.02	11.53	0.05	0.39	0.09	0.052	0.81	0.06	<0.014	22.02	0.64	75.34
S71-2 (B)	08-2054	0.07	<0.02	11.81	0.05	0.41	0.10	0.052	0.81	0.05	<0.014	22.56	0.66	77.24
	Avg.	0.07	<0.02	11.67	0.05	0.40	0.09	0.052	0.81	0.05	<0.014	22.29	0.65	76.29
	Anhyd.	0.08	<0.02	14.23	0.06	0.49	0.11	0.063	0.98	0.06	<0.014	27.19	0.79	93.06
	FBSR	0.08	<0.02	14.41	0.07	0.50	0.11	0.064	0.99	0.07	<0.014	27.54	0.80	94.24

**Table 28. Chemical Composition (wt%) of 2" Cubes with LAW FBSR and Ceramicrete Corrected for Moisture and Coal**

Sample ID	Lab ID															
	(wt%)	Ag <sub>2</sub> O	Al <sub>2</sub> O <sub>3</sub>	As <sub>2</sub> O <sub>3</sub>	B <sub>2</sub> O <sub>3</sub>	BaO	CaO	CdO	Cl	Cr <sub>2</sub> O <sub>3</sub>	Cs <sub>2</sub> O	F	Fe <sub>2</sub> O <sub>3</sub>	I	K <sub>2</sub> O	MgO
CER-1 (A)	08-1829	0.03	16.91	<0.0132	<0.32	0.07	0.19	0.02	<0.05	0.05	0.15	<0.05	0.49	0.043	9.24	7.31
CER-1 (B)	08-1829	0.03	16.80	<0.0132	<0.32	0.07	0.19	0.02	<0.05	0.05	0.15	<0.05	0.57	0.043	9.02	7.34
	Avg.	0.03	16.86	<0.0132	<0.32	0.07	0.19	0.02	<0.05	0.05	0.15	<0.05	0.53	0.043	9.13	7.32
	Anhyd.	0.04	21.04	<0.0132	<0.40	0.09	0.24	0.03	<0.06	0.07	0.19	<0.06	0.67	0.053	11.39	9.14
	FBSR	0.04	21.28	<0.0132	<0.41	0.09	0.24	0.03	<0.06	0.07	0.19	<0.06	0.67	0.054	11.52	9.25
CER-2 (A)	08-2058	0.03	19.31	<0.0132	2.41	0.08	0.12	0.03	0.06	0.05	0.14	<0.05	0.42	0.053	6.08	4.66
CER-2 (B)	08-2058	0.03	19.79	<0.0132	2.36	0.08	0.11	0.03	0.06	0.06	0.14	<0.05	0.44	0.053	6.31	4.80
	Avg.	0.03	19.55	<0.0132	2.38	0.08	0.11	0.03	0.06	0.05	0.14	<0.05	0.43	0.053	6.20	4.73
	Anhyd.	0.03	24.98	<0.0132	3.04	0.10	0.14	0.03	0.08	0.07	0.18	<0.06	0.55	0.068	7.92	6.04
	FBSR	0.03	25.28	<0.0132	3.08	0.10	0.15	0.03	0.08	0.07	0.19	<0.06	0.56	0.069	8.01	6.12

Sample ID	Lab ID												
	(wt%)	MnO <sub>2</sub>	Na <sub>2</sub> O	NiO	PO <sub>4</sub>	PbO	ReO <sub>2</sub>	SO <sub>4</sub>	Sb <sub>2</sub> O <sub>3</sub>	SeO <sub>2</sub>	SiO <sub>2</sub>	TiO <sub>2</sub>	Total
CER-1 (A)	08-1829	<0.02	9.80	0.05	14.76	0.10	0.010	0.70	0.05	<0.014	21.09	0.64	81.62
CER-1 (B)	08-1829	<0.02	10.13	0.05	14.93	0.11	0.010	0.70	0.04	<0.014	21.20	0.65	82.02
	Avg.	<0.02	9.96	0.05	14.85	0.11	0.010	0.70	0.04	<0.014	21.14	0.64	81.82
	Anhyd.	<0.02	12.44	0.06	18.53	0.13	0.013	0.88	0.06	<0.014	26.40	0.80	102.15
	FBSR	<0.02	12.58	0.06	18.74	0.13	0.013	0.89	0.06	<0.014	26.69	0.81	103.30
CER-2 (A)	08-2058	<0.02	11.33	0.05	11.23	0.11	0.023	0.76	0.05	<0.014	23.18	0.67	80.69
CER-2 (B)	08-2058	<0.02	11.71	0.05	11.69	0.11	0.023	0.76	0.05	<0.014	23.72	0.69	82.93
	Avg.	<0.02	11.52	0.05	11.46	0.11	0.023	0.76	0.05	<0.014	23.45	0.68	81.81
	Anhyd.	<0.02	14.72	0.06	14.64	0.14	0.030	0.96	0.06	<0.014	29.96	0.87	101.49
	FBSR	<0.02	14.90	0.07	14.82	0.14	0.030	0.98	0.06	<0.014	30.32	0.88	102.71

**Table 29. Chemical Composition (wt%) of 2" Cubes with LAW FBSR and Geopolymers Corrected for Moisture and Coal**

Sample ID	Lab ID																
	(wt%)	Ag <sub>2</sub> O	Al <sub>2</sub> O <sub>3</sub>	As <sub>2</sub> O <sub>3</sub>	B <sub>2</sub> O <sub>3</sub>	BaO	CaO	CdO	Cl	Cr <sub>2</sub> O <sub>3</sub>	Cs <sub>2</sub> O	F	Fe <sub>2</sub> O <sub>3</sub>	I	K <sub>2</sub> O	MgO	MnO <sub>2</sub>
GEO-1 (A)	08-2048	0.04	21.52	<0.0132	<0.32	0.07	0.13	0.02	0.07	0.06	0.14	<0.05	0.74	0.052	0.25	0.04	<0.02
GEO-1 (B)	08-2048	0.03	21.52	<0.0132	<0.32	0.07	0.15	0.02	0.07	0.06	0.14	<0.05	0.74	0.052	0.26	0.05	<0.02
	Avg.	0.04	21.52	<0.0132	<0.32	0.07	0.14	0.02	0.07	0.06	0.14	<0.05	0.74	0.052	0.26	0.04	<0.02
	Anhyd.	0.04	26.12	<0.0132	<0.39	0.09	0.17	0.03	0.08	0.07	0.17	<0.06	0.90	0.063	0.31	0.05	<0.02
	FBSR	0.04	26.43	<0.0132	<0.40	0.09	0.17	0.03	0.08	0.07	0.17	<0.06	0.91	0.064	0.31	0.06	<0.02
GEO-2 (A)	08-2049	0.03	21.06	<0.0132	<0.32	0.08	0.10	0.02	0.07	0.05	0.14	<0.05	0.58	0.045	0.24	0.03	<0.02
GEO-2 (B)	08-2049	0.03	20.93	<0.0132	<0.32	0.08	0.14	0.02	0.07	0.06	0.14	<0.05	0.59	0.045	0.23	0.03	<0.02
	Avg.	0.03	20.99	<0.0132	<0.32	0.08	0.12	0.02	0.07	0.05	0.14	<0.05	0.59	0.045	0.24	0.03	<0.02
	Anhyd.	0.04	25.44	<0.0132	<0.39	0.09	0.15	0.03	0.08	0.07	0.17	<0.06	0.71	0.055	0.29	0.04	<0.02
	FBSR	0.04	25.76	<0.0132	<0.40	0.09	0.15	0.03	0.08	0.07	0.17	<0.06	0.72	0.056	0.29	0.04	<0.02
GEO-3 (A)	08-2050	0.03	21.03	<0.0132	<0.32	0.07	0.10	0.03	0.15	0.06	0.13	<0.05	0.68	0.067	0.23	0.03	<0.02
GEO-3 (B)	08-2050	0.02	20.03	<0.0132	<0.32	0.07	0.12	0.03	0.15	0.06	0.13	<0.05	0.65	0.067	0.22	0.03	<0.02
	Avg.	0.03	20.53	<0.0132	<0.32	0.07	0.11	0.03	0.15	0.06	0.13	<0.05	0.66	0.067	0.22	0.03	<0.02
	Anhyd.	0.03	24.83	<0.0132	<0.39	0.09	0.14	0.03	0.18	0.07	0.16	<0.06	0.80	0.081	0.27	0.04	<0.02
	FBSR	0.03	25.12	<0.0132	<0.39	0.09	0.14	0.03	0.18	0.07	0.16	<0.06	0.81	0.082	0.27	0.04	<0.02
GEO-4 (A)	08-2051	0.03	20.25	<0.0132	<0.32	0.08	0.13	0.03	0.09	0.06	0.14	<0.05	0.71	0.079	0.20	0.02	<0.02
GEO-4 (B)	08-2051	0.03	19.79	<0.0132	<0.32	0.07	0.09	0.03	0.09	0.06	0.14	<0.05	0.68	0.079	0.20	0.03	<0.02
	Avg.	0.03	20.02	<0.0132	<0.32	0.07	0.11	0.03	0.09	0.06	0.14	<0.05	0.70	0.079	0.20	0.02	<0.02
	Anhyd.	0.04	25.30	<0.0132	<0.41	0.09	0.14	0.04	0.12	0.07	0.18	<0.06	0.88	0.100	0.25	0.03	<0.02
	FBSR	0.04	25.61	<0.0132	<0.41	0.10	0.14	0.04	0.12	0.08	0.18	<0.06	0.89	0.101	0.25	0.03	<0.02
GEO-5 (A)	08-2052	0.02	21.31	<0.0132	<0.32	0.07	0.12	0.02	0.07	0.06	0.13	<0.05	0.61	0.056	0.20	0.02	<0.02
GEO-5 (B)	08-2052	0.02	21.40	<0.0132	<0.32	0.08	0.09	0.03	0.07	0.06	0.13	<0.05	0.61	0.056	0.22	0.02	<0.02
	Avg.	0.02	21.35	<0.0132	<0.32	0.07	0.11	0.02	0.07	0.06	0.13	<0.05	0.61	0.056	0.21	0.02	<0.02
	Anhyd.	0.03	26.48	<0.0132	<0.40	0.09	0.13	0.03	0.09	0.07	0.16	<0.06	0.76	0.070	0.26	0.02	<0.02
	FBSR	0.03	26.77	<0.0132	<0.40	0.09	0.13	0.03	0.09	0.07	0.17	<0.06	0.77	0.070	0.26	0.02	<0.02
GEO-6 (A)	08-2053	0.03	21.35	<0.0132	<0.32	0.07	0.06	0.02	0.07	0.05	0.14	<0.05	0.57	0.045	0.21	0.01	<0.02
GEO-6 (B)	08-2053	0.02	21.07	<0.0132	<0.32	0.07	0.07	0.02	0.07	0.05	0.14	<0.05	0.57	0.045	0.20	0.02	<0.02
	Avg.	0.02	21.21	<0.0132	<0.32	0.07	0.06	0.02	0.07	0.05	0.14	<0.05	0.57	0.045	0.20	0.02	<0.02
	Anhyd.	0.03	25.97	<0.0132	<0.39	0.09	0.08	0.03	0.08	0.07	0.17	<0.06	0.69	0.055	0.25	0.02	<0.02
	FBSR	0.03	26.27	<0.0132	<0.40	0.09	0.08	0.03	0.08	0.07	0.17	<0.06	0.70	0.056	0.25	0.02	<0.02

Table 29. Chemical Composition (wt%) of 2" Cubes with LAW FBSR and Geopolymers Corrected for Moisture and Coal, continued

Sample ID	Lab ID	Na <sub>2</sub> O	NiO	PO <sub>4</sub>	PbO	ReO <sub>2</sub>	SO <sub>4</sub>	Sb <sub>2</sub> O <sub>3</sub>	SeO <sub>2</sub>	SiO <sub>2</sub>	TiO <sub>2</sub>	Total
	(wt%)											
GEO-1 (A)	08-2048	16.97	0.05	0.39	0.10	0.020	0.59	0.05	<0.014	37.74	0.78	79.71
GEO-1 (B)	08-2048	17.22	0.05	0.39	0.10	0.020	0.59	0.05	<0.014	37.99	0.80	80.31
	Avg.	17.09	0.05	0.39	0.10	0.020	0.59	0.05	<0.014	37.86	0.79	80.01
	Anhyd.	20.75	0.06	0.47	0.12	0.024	0.72	0.06	<0.014	45.96	0.96	97.11
	FBSR	20.99	0.06	0.48	0.12	<b>0.024</b>	0.73	0.06	<0.014	46.50	0.97	<b>98.26</b>
GEO-2 (A)	08-2049	16.66	0.04	0.41	0.10	0.021	0.62	0.05	<0.014	35.69	0.77	76.72
GEO-2 (B)	08-2049	16.60	0.05	0.39	0.10	0.021	0.62	0.05	<0.014	35.72	0.77	76.59
	Avg.	16.63	0.04	0.40	0.10	0.021	0.62	0.05	<0.014	35.71	0.77	76.65
	Anhyd.	20.15	0.05	0.49	0.13	0.025	0.75	0.06	<0.014	43.27	0.94	92.89
	FBSR	20.41	0.06	0.49	0.13	<b>0.025</b>	0.76	0.06	<0.014	43.82	0.95	<b>94.06</b>
GEO-3 (A)	08-2050	19.18	0.05	0.40	0.10	0.027	0.69	0.05	<0.014	38.45	0.75	82.12
GEO-3 (B)	08-2050	19.91	0.05	0.46	0.09	0.027	0.69	0.04	<0.014	38.29	0.72	81.72
	Avg.	19.55	0.05	0.43	0.09	0.027	0.69	0.05	<0.014	38.37	0.73	81.92
	Anhyd.	23.64	0.06	0.52	0.11	0.033	0.84	0.06	<0.014	46.40	0.89	99.08
	FBSR	23.92	0.06	0.53	0.12	<b>0.033</b>	0.85	0.06	<0.014	46.95	0.90	<b>100.24</b>
GEO-4 (A)	08-2051	19.39	0.05	0.44	0.10	0.035	0.73	0.05	<0.014	36.73	0.72	79.85
GEO-4 (B)	08-2051	18.78	0.05	0.43	0.10	0.035	0.73	0.04	<0.014	35.78	0.69	77.68
	Avg.	19.09	0.05	0.43	0.10	0.035	0.73	0.05	<0.014	36.26	0.70	78.77
	Anhyd.	24.12	0.06	0.55	0.13	0.044	0.93	0.06	<0.014	45.82	0.89	99.54
	FBSR	24.42	0.06	0.55	0.13	<b>0.044</b>	0.94	0.06	<0.014	46.39	0.90	<b>100.78</b>
GEO-5 (A)	08-2052	19.11	0.05	0.38	0.10	0.029	0.62	0.04	<0.014	40.32	0.75	83.99
GEO-5 (B)	08-2052	19.33	0.04	0.40	0.10	0.029	0.62	0.05	<0.014	40.98	0.77	85.04
	Avg.	19.22	0.04	0.39	0.10	0.029	0.62	0.04	<0.014	40.65	0.76	84.51
	Anhyd.	23.83	0.06	0.48	0.12	0.036	0.77	0.06	<0.014	50.41	0.95	104.81
	FBSR	24.09	0.06	0.49	0.12	<b>0.037</b>	0.77	0.06	<0.014	50.97	0.96	<b>105.96</b>
GEO-6 (A)	08-2053	17.51	0.04	0.38	0.10	0.025	0.60	0.04	<0.014	38.27	0.75	80.25
GEO-6 (B)	08-2053	17.20	0.05	0.39	0.10	0.025	0.60	0.04	<0.014	37.54	0.74	78.94
	Avg.	17.36	0.04	0.39	0.10	0.025	0.60	0.04	<0.014	37.90	0.74	79.59
	Anhyd.	21.25	0.05	0.47	0.12	0.031	0.73	0.05	<0.014	46.40	0.91	97.44
	FBSR	21.50	0.06	0.48	0.12	<b>0.031</b>	0.74	0.05	<0.014	46.94	0.92	<b>98.56</b>

**Table 30. Chemical Composition (wt%) of 2" Cubes with LAW FBSR and NuCap™ Corrected for Moisture and Coal**

Sample ID	Lab ID													
	(wt%)	Ag <sub>2</sub> O	Al <sub>2</sub> O <sub>3</sub>	As <sub>2</sub> O <sub>3</sub>	B <sub>2</sub> O <sub>3</sub>	BaO	CaO	CdO	Cr <sub>2</sub> O <sub>3</sub>	Cs <sub>2</sub> O	Fe <sub>2</sub> O <sub>3</sub>	I	K <sub>2</sub> O	MgO
NUCAP-1 (A)		0.00	12.07	<0.0132	0.67	0.05	10.27	0.01	0.01	0.10	0.85	0.016	0.12	0.93
NUCAP-1 (B)		0.00	11.85	<0.0132	0.74	0.05	10.52	0.02	0.01	0.10	0.86	0.016	0.12	0.95
	Avg.	0.00	11.96	<0.0132	0.71	0.05	10.40	0.02	0.01	0.10	0.86	0.016	0.12	0.94
	Anhyd.	0.00	12.21	<0.0132	0.72	0.05	10.61	0.02	0.01	0.10	0.87	0.017	0.12	0.96
	FBSR	0.00	12.32	<0.0132	0.73	0.05	10.70	0.02	0.01	0.10	0.88	0.017	0.12	0.97

Sample ID	Lab ID												
	(wt%)	MnO <sub>2</sub>	Na <sub>2</sub> O	NiO	PO <sub>4</sub>	PbO	ReO <sub>2</sub>	SO <sub>4</sub>	Sb <sub>2</sub> O <sub>3</sub>	SeO <sub>2</sub>	SiO <sub>2</sub>	TiO <sub>2</sub>	Total
NUCAP-1 (A)		<0.02	7.58	0.02	0.30	0.06	0.014	1.41	0.01	<0.014	4.28	0.04	37.22
NUCAP-1 (B)		<0.02	7.54	0.02	0.30	0.06	0.014	1.42	0.01	<0.014	4.09	0.04	37.03
	Avg.	<0.02	7.56	0.02	0.30	0.06	0.014	1.41	0.01	<0.014	4.18	0.04	37.12
	Anhyd.	<0.02	7.71	0.02	0.30	0.06	0.014	1.44	0.01	<0.014	4.27	0.04	37.90
	FBSR	<0.02	7.78	0.02	0.31	0.06	0.015	1.46	0.01	<0.014	4.31	0.04	38.23

**Table 31. Moisture Data for 2" Cube Monoliths with LAW FBSR Dried at 250°C Overnight**

	<b>Initial Mass Sample (g)</b>	<b>Dry Mass Sample (g)</b>	<b>Dry Mass / Initial Mass</b>	<b>wt% Mass Loss</b>	<b>Maximum Calculated Water Content (wt%)</b>
CER-1	5.51	4.41	0.80	19.9	14
CER-2	5.41	4.24	0.78	21.7	25
OPC-1	5.39	5.052	0.94	6.2	36
OPC-2	7.08	5.504	0.78	22.3	35
FON-1	6.05	5.14	0.85	15.0	33
FON-2	5.61	4.39	0.78	21.8	33
S41-1	5.18	4.113	0.79	20.6	33
S41-2	5.15	4.482	0.87	12.9	33
S71-1	7.81	6.402	0.82	15.5	33
S71-2	7.81	6.402	0.82	18.0	33
NUCAP-1	6.09	5.964	0.98	2.0	NA
GEO-1	5.81	4.785	0.82	17.6	28
GEO-2	6.37	5.257	0.83	17.5	27
GEO-3	6.27	5.182	0.83	17.3	27
GEO-4	7.53	5.96	0.79	20.9	27
GEO-5	6.07	4.892	0.81	19.4	28
GEO-6	5.27	4.303	0.82	18.3	29

Results of the 7-day PCT performed on the non-roasted PCT powders prepared from the 2" cubes are shown in Table 32. All leachate values are shown in mg/L units for Al, Cs, I, Na, Re, S and Si along with the resulting final leachate pHs. The surface area of the non-roasted PCT-prepared powders are also shown from non-roasted BET measurements in units of m<sup>2</sup>/g. The BET SA data was obtained for the OPC samples using a 4 hr evacuation at 300 °C, whereas all the other data was obtained using 3 to 4 hour evacuations at 105 °C. The reported nominal relative standard deviation for the BET SA data is in the range of 0.2 to 0.4 for all samples except for the NuCap<sup>TM</sup> which showed higher %RSD of 1.1.

Particle size information for the 2" cube monolith PCT-prepared powders are shown in Appendix 10. All SA/V and normalized release data were calculated similarly to the PR and HTF fines and aggregate blends PCT data via Equations 1 and 2. As was the case with the aggregate blends, the measured BET SA terms were from non-roasted samples. The adjusted elemental compositions for the 2" cubes (anhydrous, coal-free basis from Table 26 through Table 30 were used to normalize the PCT release for better comparison to the previous aggregate (coal removed by LOI) and blend (coal removed by calculation) PCT data.

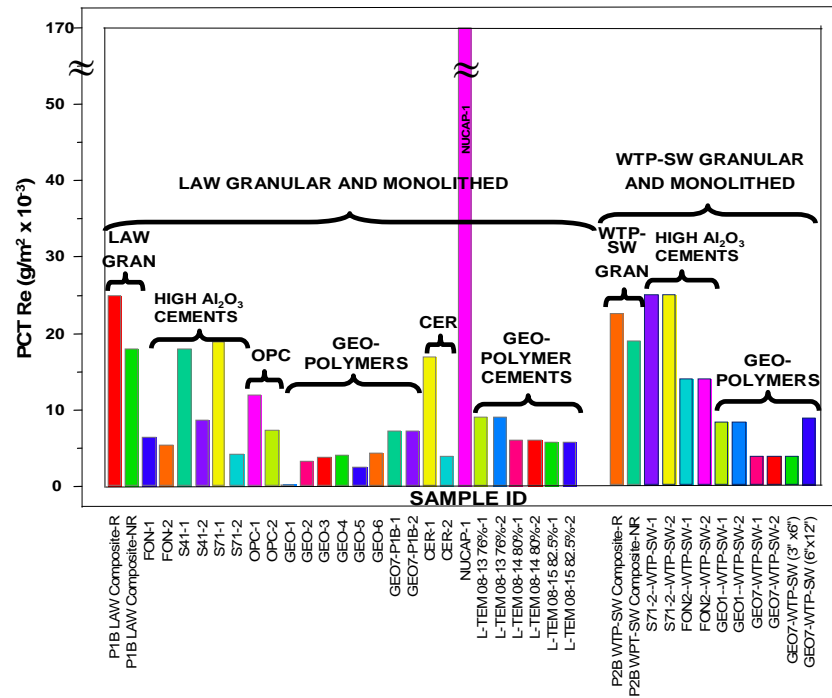


Table 32. PCT Data for 2" Cubes

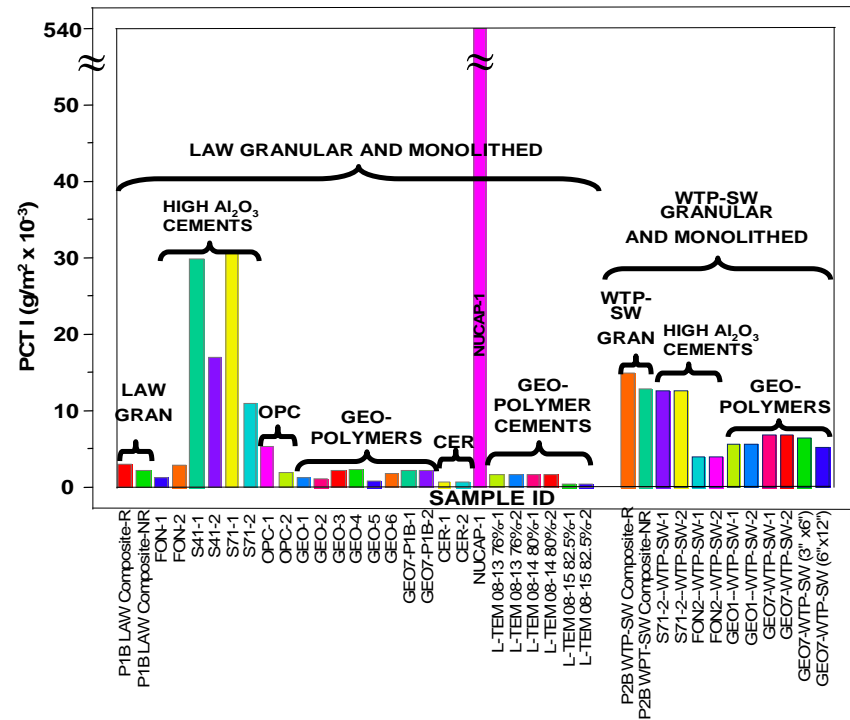
2" Cube Mono.	As Measured Leachate Concentrations							pH	BET non-roasted (m <sup>2</sup> /g)	SA/V (m <sup>2</sup> )	Normalized Releases Calculated from Equations 1 & 2						
	Al (mg/L)	Cs (mg/L)	I (mg/L)	Na (mg/L)	Re (mg/L)	S (mg/L)	Si (mg/L)				NL <sub>Al</sub> (g/m <sup>2</sup> )	NL <sub>Cs</sub> (g/m <sup>2</sup> )	NL <sub>I</sub> (g/m <sup>2</sup> )	NL <sub>Na</sub> (g/m <sup>2</sup> )	NL <sub>Re</sub> (g/m <sup>2</sup> )	NL <sub>S</sub> (g/m <sup>2</sup> )	NL <sub>Si</sub> (g/m <sup>2</sup> )
FON-1	1443.5	21.9	1.5	2300.0	2.8	118.6	4.8	12.08	20.0	2,000,000	4.4E-03	6.2E-03	1.3E-03	1.2E-02	6.5E-03	1.9E-02	1.8E-05
FON-2	1369.9	25.6	3.4	2725.0	2.6	168.0	9.2	12.26	15.5	1,550,000	5.0E-03	8.3E-03	3.0E-03	1.6E-02	5.5E-03	3.4E-02	3.9E-05
S41-1	1775.0	22.8	15.3	2358.3	3.4	125.6	2.9	12.14	12.2	1,220,000	8.5E-03	1.1E-02	3.0E-02	2.2E-02	1.8E-02	4.8E-02	1.8E-05
S41-2	1725.0	7.8	14.4	2741.7	2.4	175.6	5.9	12.19	10.7	1,070,000	9.5E-03	4.3E-03	1.7E-02	2.5E-02	8.7E-03	5.8E-02	4.0E-05
S71-1	1575.5	40.7	20.7	2450.0	4.4	152.2	3.6	12.26	13.1	1,310,000	5.6E-03	1.7E-02	3.1E-02	2.0E-02	1.9E-02	4.0E-02	2.2E-05
S71-2	1593.0	35.6	13.1	2066.7	2.1	136.6	16.0	11.94	9.2	921,000	8.0E-03	2.8E-02	1.1E-02	2.1E-02	4.3E-03	4.5E-02	1.4E-04
OPC-1	292.3	10.6	8.1	2441.7	6.3	485.7	18.9	12.23	31.5	3,145,440	8.5E-04	2.2E-03	5.4E-03	9.3E-03	1.2E-02	3.7E-02	4.3E-05
OPC-2	206.0	4.5	3.4	2408.3	3.5	436.6	37.2	12.37	21.3	2,130,420	6.0E-04	9.1E-04	2.0E-03	9.1E-03	7.4E-03	4.5E-02	9.0E-05
GEO-1	14.6	2.2	1.3	3225.0	0.1	154.0	827.8	12.12	15.2	1,520,000	6.9E-05	8.9E-04	1.3E-03	1.4E-02	3.5E-04	4.2E-02	2.5E-03
GEO-2	14.3	1.5	1.2	3416.7	1.2	165.8	859.7	12.15	17.3	1,730,000	6.1E-05	5.4E-04	1.2E-03	1.3E-02	3.3E-03	3.8E-02	2.4E-03
GEO-3	32.4	2.7	2.0	4233.3	1.2	158.7	1006.3	12.29	10.9	1,090,000	2.2E-04	1.7E-03	2.2E-03	2.2E-02	3.9E-03	5.1E-02	4.2E-03
GEO-4	41.9	4.0	1.5	4841.7	0.9	160.4	1006.3	12.36	6.2	617,000	5.0E-04	3.7E-03	2.4E-03	4.3E-02	4.1E-03	8.3E-02	7.5E-03
GEO-5	8.4	1.5	0.7	4608.3	0.8	145.6	2508.3	12.30	10.6	1,060,000	5.6E-05	8.8E-04	8.9E-04	2.4E-02	2.5E-03	5.3E-02	9.9E-03
GEO-6	7.9	0.6	1.0	3358.3	1.2	150.3	1563.1	12.27	10.0	1,000,000	5.7E-05	4.0E-04	1.8E-03	2.1E-02	4.4E-03	6.1E-02	7.1E-03
CER-1	39.5	33.2	1.4	1900.0	6.0	299.6	7.8	9.86	32.2	3,220,000	1.1E-04	5.6E-03	8.2E-04	6.3E-03	1.7E-02	3.1E-02	1.9E-05
CER-2	36.9	30.8	1.4	1658.3	2.8	265.1	7.4	9.62	27.7	2,770,000	1.0E-04	6.3E-03	7.3E-04	5.4E-03	4.0E-03	2.9E-02	1.9E-05
NuCap	75.6	3.7	8.1	705.3	2.0	155.9	455.3	10.01	0.1	9,000	1.3E-01	4.3E-01	5.4E+00	1.4E+00	1.7E+00	8.9E+00	2.5E+00

Normalized release data from Table 32 are plotted in Figure 7 to show comparison of the LAW and WTP-SW blends with the different 2" cube monolith samples, as well as some of the larger monoliths that are discussed later in this report. All of the NuCap<sup>TM</sup> data is off-scale on the graphs shown due to the low measured elemental contents of these monoliths and their relatively low measured BET SAs. This figure shows that the normalized release from these monolithed FBSR mineral aggregates (with the exception of NuCap<sup>TM</sup>) are comparable in magnitude to the previously leached LAW and WTP-SW blends. The NL(x) values for Re, I, Cs and Na seem to indicate better performance for the geopolymers versus either the high Al cements or Portland cement monoliths for both the LAW P-1B and WTP-SW P-2B series. Some of the larger scale 3"x 6" and 6" x 12" monolith PCT data are also plotted in Figure 7 for comparison. The NL<sub>S</sub> values appear to be lower for the L-TEM monoliths discussed later in this report.

The initial PCT data obtained for the 2" cube monoliths were used along with the compression test results and TCLP data to select the 'best three' monolith recipes (per Figure 1 scope) to carry forward into larger scale testing with 3"x 6" cylinders and 6"x12" cylinders. It was judged that the higher loaded S71-2 and FON-2 calcium aluminate monoliths and the GEO-1 monolith would be further tested. The S71-2 and GEO-1 results showed the best performance for NL<sub>Re</sub>, and although the S71-2 did not pass compressive strength testing at 490 psi for a 7-day break, it was anticipated that a longer cure time could contribute to a compressive strength that would be above 500 psi. It was also decided to add the GEO-7 monolith recipe made with fly ash for a fourth formulation for larger scale testing based on its initial compression testing results per Table 22.

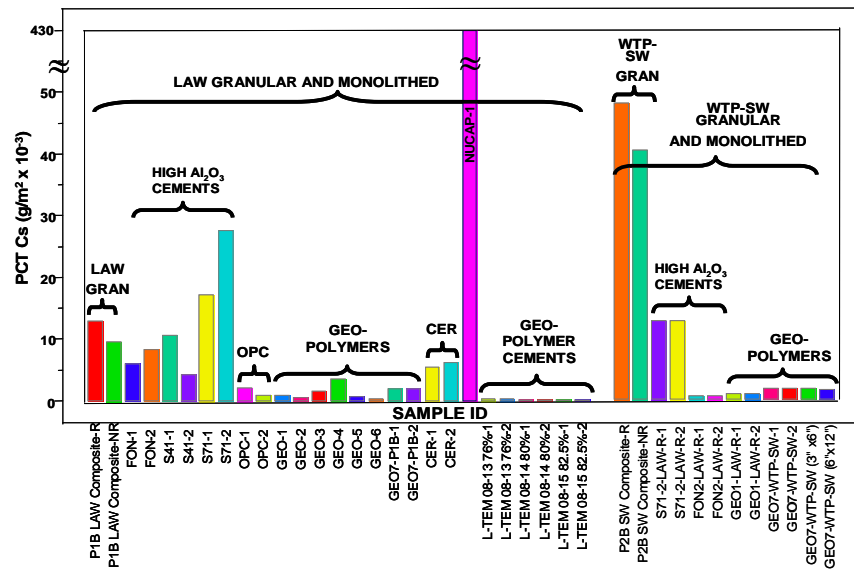


(a)

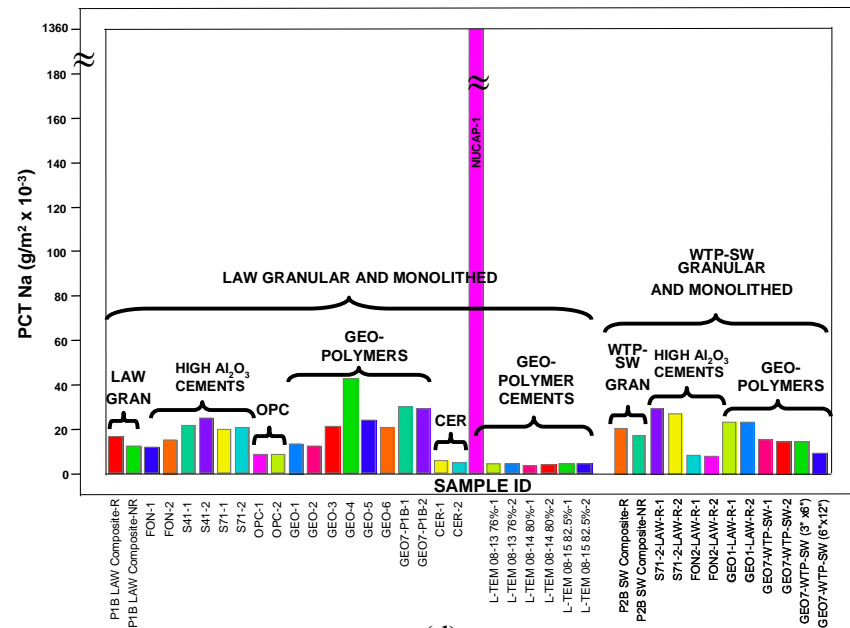


(b)

Figure 7 a,b. Normalized Release for Re and I from Blended Aggregates, 2" Cube Monoliths, and Some Additional Larger (3"x6" and 6"x12") Monoliths

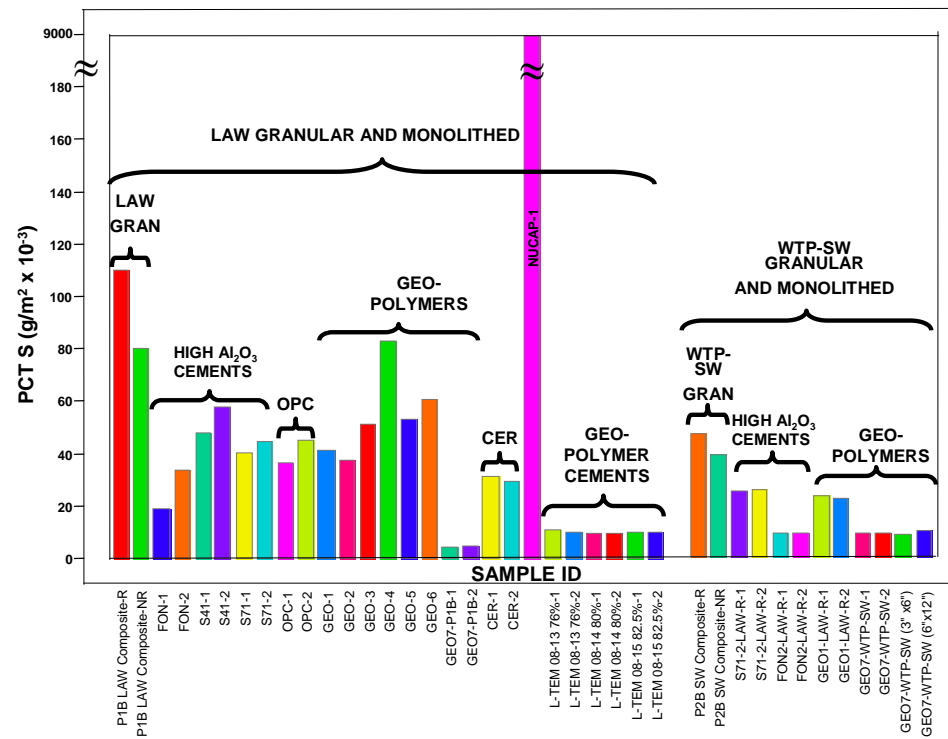


(c)



(d)

Figure 7 c, d. Normalized Release for Cs and Na from Blended Aggregates, 2" Cube Monoliths, and Some Additional Larger (3"x6" and 6"x12") Monoliths



(e)

**Figure 7 e. Normalized Release for Sulfur from Blended Aggregates, 2" Cube Monoliths, and Some Additional Larger (3"x6" and 6"x12") Monoliths**

#### 4.6 MONOLITH TESTING WITH 3" X 6" AND 6" X 12" CYLINDERS

Recipes for the FON-1, S71-2, GEO-1 and GEO-7 were used to make the 3"x 6" cylinders and later to make the 6" x 12" cylinders. These monoliths were made with both the LAW P-1B and the WTP-SW P-2B blended aggregate FBSR products. All of the scoping work (2" cubes) was done and optimized with the P-1B material (LAW). When P-2B material was monolithed, the GEO-7 recipe was used, but that recipe was not optimized for P-2B. The focus was on the LAW material.

All monoliths were submitted for compression testing and TCLP. PCT preparations were performed on the crushed monoliths (grinding/sieving/washing/drying). Table 33 shows a summary of the compressive strength, cure times, monolith bulk densities and the measured PCT-powder non-roasted BET surface areas for comparison. As indicated in the footnotes to Table 33, some of the measured BET SAs that were obtained for smaller monolith PCT prepared powders were also used for calculations in the larger-scale monolith PCT-prepared powders. This was done to conserve project funding and time, as the BET SA method is one of the most time-consuming and therefore, costlier analytical methods. The FONDU formulation shows that 3 of the 5 monoliths do not pass compression testing target of 500 psi even at 28 day cure. All of the other monoliths pass compression testing. All measured (non-roasted) monolith PCT-prepared powder surface areas are in the range of 9 to 30 m<sup>2</sup>/g which are slightly higher than the range of original bed product (PR) and fines (HTF) measured surface areas (with coal roasted out) and blended aggregate surface areas (with residual coal present) in the range of 3 to 5 m<sup>2</sup>/g. This indicates that perhaps there is more porosity associated with the monolith PCT powders versus the PR bed, HTF fines and blended aggregate PCT powders, all of which were ground/sieved to 100-200 mesh. Also shown in Table 33 are data for the L-TEM 3" x 6" cylinders (supplied to SRNL by TTT via CEES, Pasco, WA) that indicate these monoliths passed the compression testing and have measured PCT-prepared powder BET surface areas that are significantly higher (43 – 48 m<sup>2</sup>/g) than the other monoliths formulated at SRNL. Also the waste loadings shown in Table 33 were provided by CEES.

**Table 33. Summary of Monolith Data for 2" Cubes, 3" x 6" Cylinders and 6" x 12" Cylinders**

Binder & Size	Compressive Strength (psi)	Cure Time (days)	Density (g/cc)	Non-roasted BET Surface Area (m <sup>2</sup> /g)
<b>LAW</b>				
FON-2	74.2% waste loading			
2" Cube	490	7	1.75	15.50
3" dia x 6" cylinder	580	28	1.69	15.50 *
6" dia x 12" cylinder	370	28	1.69	15.50 *
S71-2	74.2% waste loading			
2" Cube	550	15	1.65	9.20
3" dia x 6" cylinder	660	17	1.68	9.20 *
6" dia x 12" cylinder	550	19	1.70	9.20 *
GEO-1	67.5% waste loading			
2" Cube	1,510	11	1.87	15.20
3" dia x 6" cylinder	1,690	14	1.85	15.20 *
6" dia x 12" cylinder	1,530	14	1.82	15.20 *
GEO-7	65.2% waste loading			
3" dia x 6" cylinder	2,500	14	1.90	11.70
6" dia x 12" cylinder	1,920	18	NM	11.70**
L-TEM	76% waste loading, LAW P1A, (provided by CEES)			
3" dia x 6" cylinder	1,750	20	1.43	46.52
L-TEM	80% waste loading, LAW P1A, (provided by CEES)			
3" dia x 6" cylinder	1,210	20	1.39	48.77
L-TEM	82.5% waste loading, LAW P1A, (provided by CEES)			
3" dia x 6" cylinder	930	20	1.38	43.20
<b>WTP-SW</b>				
FON-2	74.2% waste loading			
3" dia x 6" cylinder	570	18	1.68	30.83
6" dia x 12" cylinder	420	28	1.67	30.83***
S71-2	74.2% waste loading			
3" dia x 6" cylinder	820	17	1.67	8.99
6" dia x 12" cylinder	660	19	1.66	8.99***
GEO-1	67% waste loading			
3" dia x 6" cylinder	890	14	1.83	12.55
6" dia x 12" cylinder	1,710	19	1.83	12.55***
GEO-7	65.2% waste loading			
3" dia x 6" cylinder	1,980	14	1.83	26.86
6" dia x 12" cylinder	520	28	NM	26.86***

\*These BET SAs were obtained from the 2" cube PCT prepared powders.

\*\* These BET SAs used an average of the GEO-1 to GEO-6 2" cube data.

\*\*\* These BET SAs were obtained from the 3"x 6" cylinder PCT prepared powders.

All TCLP data for the 3" x 6" and 6" x 12" cylinders are shown in Appendix 11. As was the case with the original blends and the 2" cubes, the only elements that showed TCLP leachate response failure were Sb and Cd which had been shimmed in at elevated concentrations for off-gas measurement purposes. Table 34 shows that the FONDU formulations passed TCLP for these and all other elements at all monolith sizes and with both P-1B and WTP-SW P-2B. The S71-2, GEO-1 and GEO-7 LAW monoliths and the S71-2 and GEO-1 WTP-SW monoliths all show cases of TCLP leachate failure for Sb and Cd. However, as was noted previously with TCLP data for the blends and 2" cubes, these elements were added to the ESTD simulant at ~ 1,000X their detection limits. All of the GEO-7 WTP-SW monoliths passed TCLP.

**Table 34. TCLP Data for the 2" Cube, 3" x 6", and 6" x 12" Monoliths**

Binder & Size	Pass/Fail TCLP	
	Sb	Cd
<b>LAW</b>		
<b>FON-2</b>		
2" Cube	YES	YES
3" x 6" cylinder	YES	YES
6" x 12" cylinder	YES	YES
<b>S71-2</b>		
2" Cube	YES	YES
3" x 6" cylinder	YES	YES
6" x 12" cylinder	YES	NO
<b>GEO-1</b>		
2" Cube	YES	NO
3" x 6" cylinder	NO	YES
6" x 12" cylinder	YES	YES
<b>GEO-7</b>		
3" x 6" cylinder	NO	NO
6" x 12" cylinder	YES	YES
<b>WTP SW</b>		
<b>FON-2</b>		
3" x 6" cylinder	YES	YES
6" x 12" cylinder	YES	YES
<b>S71-2</b>		
3" x 6" cylinder	YES	NO
6" x 12" cylinder	YES	NO
<b>GEO-1</b>		
3" x 6" cylinder	NO	YES
6" x 12" cylinder	NO	YES
<b>GEO-7</b>		
3" x 6" cylinder	YES	YES
6" x 12" cylinder	YES	YES

All of the 3" x 6" cylinders and the 6" x 12" cylinders were prepared for chemical composition analysis and PCT in the same manner as the 2" cubes described above. Table 35 shows the measured moisture content of the various 3" x 6" cylinders that used ~ 5 gram portions of the post-compression test pieces heated to 250°C overnight in air. These data were used to normalize both the 3" x 6" and the 6" x 12" data (shown later in this report) to an anhydrous basis. S71-2 and FON-2 cement formulations contained 16-21 wt% moisture, geopolymers contained 19-22 wt% moisture, and the L-TEM samples contained 13-26 wt% moisture. The chemical compositions were further normalized to a coal-free basis by using



the FBSR wt% loading and the measured LOI of either the LAW P-1B (1.72 wt% mass loss on LOI) and the WTP-SW P-2B (11.06 wt% mass loss on LOI).

**Table 35. Moisture Content of 3" x 6" Cylinders at 250°C**

	<b>Initial Sample Mass</b>	<b>Heated Sample Mass</b>	<b>Mass Loss after Heating</b>	<b>Heated Mass/ Initial Mass</b>	<b>wt% Loss</b>
S71-2 LAW P-1B	5.203	4.290	0.913	0.825	17.55
S71-2 WTP-SW	5.789	4.556	1.233	0.787	21.30
FON-2 LAW P-1B	5.062	4.160	0.902	0.822	17.82
FON-2 WTP-SW	5.372	4.470	0.902	0.832	16.79
GEO-1 LAW P-1B	5.795	4.564	1.231	0.788	21.24
GEO-1 WTP-SW	5.256	4.102	1.154	0.780	21.96
GEO-7 LAW P-1B	5.529	4.433	1.096	0.802	19.82
GEO-7 WTP-SW	5.381	4.312	1.069	0.801	19.87
L-TEM 76 (WTP-SW)	5.329	3.960	1.369	0.743	25.69
L-TEM 80 (WTP-SW)	5.084	3.800	1.284	0.747	25.26
L-TEM 82.5 (WTP-SW)	5.708	4.935	0.773	0.865	13.54

All chemical composition data for the 3" x 6" cylinders are shown below in Table 36 to Table 38 as nominal oxide concentrations calculated from the measured elemental concentrations from the dissolved solids. Fully normalized chemical composition data (normalized for moisture and residual coal content) are shown as grey-scale in these tables. These fully normalized chemical compositions were used to normalize the PCT release data discussed later.

Table 36. Chemical Composition of 3" x 6" Cylinders (LAW and WTP-SW) with S71 and FONDU

Sample ID	Lab ID														
	(wt%)	Ag <sub>2</sub> O	Al <sub>2</sub> O <sub>3</sub>	As <sub>2</sub> O <sub>3</sub>	B <sub>2</sub> O <sub>3</sub>	BaO	CaO	CdO	Cl	Cr <sub>2</sub> O <sub>3</sub>	Cs <sub>2</sub> O	F	Fe <sub>2</sub> O <sub>3</sub>	I	K <sub>2</sub> O
S71 P-1B (A)	08-2412	<0.01	34.39	<0.0134	<0.32	0.08	8.30	0.02	0.06	0.06	0.14	<0.05	0.70	0.045	0.16
S71 P-1B (B)	08-2412	<0.01	34.39	<0.0134	<0.32	0.08	8.40	0.02	0.06	0.05	0.14	<0.05	0.66	0.045	0.17
	Avg.	<0.01	34.39	<0.0134	<0.32	0.08	8.35	0.02	0.06	0.06	0.14	<0.05	0.68	0.045	0.16
	Anhyd.	<0.01	41.71	<0.0134	<0.39	0.09	10.12	0.02	0.07	0.07	0.17	<0.06	0.82	0.054	0.20
	FBSR	<0.01	42.24	<0.0134	<0.40	0.10	10.25	0.02	0.08	0.07	0.17	<0.06	0.83	0.055	0.20
S71 WTP-SW (A)	08-2419	<0.01	28.91	<0.0134	<0.32	0.00	8.19	<0.01	0.28	0.05	0.24	0.17	0.80	0.012	0.24
S71 WTP-SW (B)	08-2419	<0.01	28.91	<0.0134	<0.32	0.00	8.03	<0.01	0.28	0.05	0.24	0.17	0.80	0.012	0.25
	Avg.	<0.01	28.91	<0.0134	<0.32	0.00	8.11	<0.01	0.28	0.05	0.24	0.17	0.80	0.012	0.24
	Anhyd.	<0.01	36.73	<0.0134	<0.41	0.00	10.30	<0.01	0.35	0.06	0.30	0.22	1.01	0.015	0.31
	FBSR	<0.01	40.03	<0.0134	<0.45	0.00	11.23	0.02	0.38	0.07	0.33	0.24	1.11	0.017	0.34
FON P-1B (A)	08-2414	<0.01	27.97	<0.0134	<0.32	0.08	10.21	0.02	0.05	0.08	0.15	<0.05	4.29	0.041	0.23
FON P-1B (B)	08-2414	<0.01	27.78	<0.0134	<0.32	0.09	10.38	0.02	0.05	0.09	0.15	<0.05	4.20	0.041	0.20
	Avg.	<0.01	27.87	<0.0134	<0.32	0.09	10.30	0.02	0.05	0.08	0.15	<0.05	4.25	0.041	0.22
	Anhyd.	<0.01	33.91	<0.0134	<0.39	0.10	12.53	0.02	0.06	0.10	0.19	<0.06	5.17	0.050	0.26
	FBSR	<0.01	34.35	<0.0134	<0.40	0.11	12.69	0.02	0.06	0.10	0.19	<0.06	5.23	0.051	0.27
FON-WTP-SW (A)	08-2415	<0.01	24.00	<0.0134	<0.32	0.00	10.27	<0.01	0.37	0.09	0.21	<0.05	5.66	0.009	0.28
FON-WTP-SW (B)	08-2415	<0.01	23.81	<0.0134	<0.32	0.01	10.20	<0.01	0.37	0.09	0.21	<0.05	5.52	0.009	0.28
	Avg.	<0.01	23.90	<0.0134	<0.32	0.00	10.24	<0.01	0.37	0.09	0.21	<0.05	5.59	0.009	0.28
	Anhyd.	<0.01	28.73	<0.0134	<0.39	0.01	12.30	<0.01	0.45	0.11	0.26	<0.06	6.72	0.011	0.34
	FBSR	<0.01	31.30	<0.0134	<0.42	0.01	13.40	<0.01	0.49	0.12	0.28	<0.07	7.32	0.012	0.37

Table 36. Chemical Composition of 3" x 6" Cylinders (LAW and WTP-SW) with S71 and FONDU, continued

Sample ID	Lab ID	MgO	MnO <sub>2</sub>	Na <sub>2</sub> O	NiO	PO <sub>4</sub>	PbO	ReO <sub>2</sub>	SO <sub>4</sub>	Sb <sub>2</sub> O <sub>3</sub>	SeO <sub>2</sub>	SiO <sub>2</sub>	TiO <sub>2</sub>	Tl	ZnO	Total
	(wt%)															
<b>S71 P-1B (A)</b>	<b>08-2412</b>	0.08	<0.02	11.32	0.05	0.37	0.11	0.025	0.98	0.04	<0.014	24.17	0.71	<0.01	<0.01	81.47
<b>S71 P-1B (B)</b>	<b>08-2412</b>	0.07	<0.02	11.24	0.04	0.27	0.10	0.025	0.92	0.03	<0.014	24.17	0.56	<0.01	<0.01	81.15
	Avg.	0.07	<0.02	11.28	0.05	0.32	0.10	0.025	0.95	0.04	<0.014	24.17	0.64	<0.01	<0.01	81.31
	Anhyd.	0.09	<0.02	13.68	0.06	0.39	0.13	0.030	1.15	0.04	<0.014	29.32	0.77	<0.01	<0.02	98.61
	FBSR	0.09	<0.02	13.86	0.06	0.39	0.13	0.030	1.17	0.04	<0.014	29.69	0.78	<0.01	<0.02	99.87
<b>S71 WTP-SW (A)</b>	<b>08-2419</b>	0.10	<0.02	8.49	0.04	0.12	0.02	0.010	0.53	0.02	<0.014	21.05	0.61	<0.01	0.05	69.54
<b>S71 WTP-SW (B)</b>	<b>08-2419</b>	0.09	<0.02	8.51	0.04	0.15	0.03	0.010	0.55	0.01	<0.014	21.03	0.61	<0.01	0.05	69.41
	Avg.	0.10	<0.02	8.50	0.04	0.13	0.02	0.010	0.54	0.02	<0.014	21.04	0.61	<0.01	0.05	69.48
	Anhyd.	0.12	<0.02	10.80	0.05	0.17	0.03	0.013	0.68	0.02	<0.014	26.73	0.78	<0.01	0.07	88.28
	FBSR	0.13	<0.02	11.77	0.05	0.19	0.03	0.015	0.75	0.02	<0.014	29.13	0.85	<0.01	0.07	96.19
<b>FON P-1B (A)</b>	<b>08-2414</b>	0.20	0.06	11.61	0.06	0.46	0.11	0.026	0.89	0.04	<0.014	26.53	1.14	<0.01	<0.01	83.98
<b>FON P-1B (B)</b>	<b>08-2414</b>	0.21	0.06	11.47	0.07	0.47	0.12	0.026	0.89	0.04	<0.014	26.10	1.17	<0.01	<0.01	83.36
	Avg.	0.21	0.06	11.54	0.06	0.46	0.12	0.026	0.89	0.04	<0.014	26.31	1.15	<0.01	<0.01	83.67
	Anhyd.	0.25	0.08	14.04	0.08	0.56	0.14	0.031	1.08	0.05	<0.014	32.02	1.40	<0.01	<0.02	101.81
	FBSR	0.26	0.08	14.22	0.08	0.57	0.14	0.032	1.09	0.05	<0.014	32.43	1.42	<0.01	<0.02	103.11
<b>FON-WTP-SW (A)</b>	<b>08-2415</b>	0.22	0.07	8.71	0.04	0.21	0.02	0.023	0.62	0.02	<0.014	23.32	1.04	<0.01	0.05	74.86
<b>FON-WTP-SW (B)</b>	<b>08-2415</b>	0.24	0.07	8.55	0.04	0.17	0.03	0.023	0.62	0.02	<0.014	22.46	1.06	<0.01	0.05	73.48
	Avg.	0.23	0.07	8.63	0.04	0.19	0.02	0.023	0.62	0.02	<0.014	22.89	1.05	<0.01	0.05	74.17
	Anhyd.	0.28	0.08	10.37	0.05	0.23	0.03	0.027	0.74	0.02	<0.014	27.51	1.26	<0.01	0.06	89.14
	FBSR	0.30	0.09	11.29	0.05	0.25	0.03	0.030	0.81	0.02	<0.014	29.95	1.38	<0.01	0.07	89.14

Table 37. Chemical Composition Data for 3" x 6" Cylinders (LAW and WTP-SW) with GEO-1 and GEO-7

Sample ID	Lab ID															
	(wt%)	Ag <sub>2</sub> O	Al <sub>2</sub> O <sub>3</sub>	As	B <sub>2</sub> O <sub>3</sub>	BaO	CaO	CdO	Cl	Cr <sub>2</sub> O <sub>3</sub>	Cs <sub>2</sub> O	F	Fe <sub>2</sub> O <sub>3</sub>	I	K <sub>2</sub> O	MgO
GEO-1 P-1B (A)	08-2416	<0.01	21.92	0.01	<0.32	0.08	0.17	0.02	0.06	0.05	0.13	<0.05	0.60	0.054	0.42	0.03
GEO-1 P-1B (B)	08-2416	<0.01	22.49	0.01	<0.32	0.08	0.16	0.02	0.06	0.05	0.13	<0.05	0.58	0.054	0.28	0.03
	Avg.	<0.01	22.20	0.01	<0.32	0.08	0.16	0.02	0.06	0.05	0.13	<0.05	0.59	0.054	0.35	0.03
	Anhyd.	<0.01	28.19	0.01	<0.41	0.10	0.21	0.02	0.08	0.07	0.17	<0.06	0.74	0.069	0.44	0.04
	FBSR	<0.01	28.52	0.01	<0.41	0.10	0.21	0.02	0.08	0.07	0.17	<0.06	0.75	0.069	0.45	0.04
GEO-1 WTP-SW (A)	08-2417	<0.01	18.63	0.01	<0.32	0.01	0.14	<0.01	0.29	0.05	0.21	0.36	0.62	0.009	0.35	0.05
GEO-1 WTP-SW (B)	08-2417	<0.01	18.44	0.01	<0.32	0.01	0.14	<0.01	0.29	0.05	0.21	0.36	0.66	0.009	0.35	0.05
	Avg.	<0.01	18.54	0.01	<0.32	0.01	0.14	<0.01	0.29	0.05	0.21	0.36	0.64	0.009	0.35	0.05
	Anhyd.	<0.01	23.75	0.01	<0.41	0.01	0.18	<0.01	0.37	0.06	0.27	0.46	0.82	0.011	0.45	0.07
	FBSR	<0.01	25.67	0.01	<0.45	0.01	0.19	<0.02	0.40	0.07	0.29	0.50	0.89	0.012	0.49	0.07
GEO-7 P-1B (A)	08-2418	<0.01	21.16	0.01	<0.32	0.09	0.29	0.02	0.08	0.06	0.11	<0.05	1.99	0.055	0.76	0.20
GEO-7 P-1B (B)	08-2418	<0.01	21.35	0.01	<0.32	0.09	0.29	0.02	0.08	0.06	0.11	<0.05	1.92	0.055	0.77	0.20
	Avg.	<0.01	21.26	0.01	<0.32	0.09	0.29	0.02	0.08	0.06	0.11	<0.05	1.95	0.055	0.77	0.20
	Anhyd.	<0.01	26.51	0.01	<0.40	0.11	0.36	0.02	0.10	0.07	0.14	<0.06	2.43	0.069	0.96	0.25
	FBSR	<0.01	26.81	0.01	<0.41	0.11	0.37	0.02	0.10	0.07	0.14	<0.06	2.46	0.069	0.97	0.25
GEO-7 WTP-SW (A)	08-2413	<0.01	18.61	0.01	<0.32	0.02	0.48	<0.01	0.31	0.06	0.18	0.25	2.85	0.009	0.81	0.21
GEO-7 WTP-SW (B)	08-2413	<0.01	18.46	0.01	<0.32	0.02	0.49	<0.01	0.32	0.06	0.18	0.26	2.82	0.009	0.83	0.21
	Avg.	<0.01	18.54	0.01	<0.32	0.02	0.49	<0.01	0.31	0.06	0.18	0.26	2.83	0.009	0.82	0.21
	Anhyd.	<0.01	23.13	0.01	<0.40	0.03	0.61	<0.01	0.39	0.07	0.23	0.32	3.53	0.012	1.02	0.26
	FBSR	<0.01	24.94	0.01	<0.43	0.03	0.65	<0.02	0.42	0.08	0.25	0.35	3.81	0.013	1.10	0.28

Table 37. Chemical Composition Data for 3" x 6" Cylinders (LAW and WTP-SW) with GEO-1 and GEO-7, continued

Sample ID	Lab ID	MnO <sub>2</sub>	Na <sub>2</sub> O	NiO	PO <sub>4</sub>	PbO	ReO <sub>2</sub>	SO <sub>4</sub>	Sb <sub>2</sub> O <sub>3</sub>	Se	SiO <sub>2</sub>	TiO <sub>2</sub>	Tl	Zn	ZnO	Total
	(wt%)															
<b>GEO-1 P-1B (A)</b>	<b>08-2416</b>	<0.02	16.18	0.04	0.39	0.10	0.031	0.85	0.04	<0.014	38.72	0.83	<0.01	<0.01	<0.01	80.43
<b>GEO-1 P-1B (B)</b>	<b>08-2416</b>	<0.02	16.45	0.04	0.34	0.09	0.031	0.84	0.03	<0.014	39.79	0.77	<0.01	<0.01	<0.01	82.07
	Avg.	<0.02	16.31	0.04	0.37	0.10	0.031	0.85	0.04	<0.014	39.25	0.80	<0.01	<0.01	<0.01	81.25
	Anhyd.	<0.02	20.71	0.06	0.47	0.12	0.040	1.07	0.05	<0.014	49.84	1.02	<0.01	<0.01	<0.02	103.17
	FBSR	<0.02	20.95	0.06	0.47	0.12	0.040	1.09	0.05	<0.014	50.42	1.03	<0.01	<0.01	<0.02	104.36
<b>GEO-1 WTP-SW (A)</b>	<b>08-2417</b>	<0.02	14.42	0.02	0.15	0.02	0.013	0.48	0.01	<0.014	36.15	0.69	<0.01	0.04	0.05	72.37
<b>GEO-1 WTP-SW (B)</b>	<b>08-2417</b>	<0.02	14.42	0.02	0.14	0.02	0.013	0.49	0.01	<0.014	35.94	0.70	<0.01	0.04	0.05	72.02
	Avg.	<0.02	14.42	0.02	0.14	0.02	0.013	0.49	0.01	<0.014	36.05	0.70	<0.01	0.04	0.05	72.20
	Anhyd.	<0.02	18.48	0.03	0.18	0.02	0.017	0.62	0.01	<0.014	46.19	0.89	<0.01	0.05	0.06	92.51
	FBSR	<0.02	19.98	0.03	0.20	0.02	0.018	0.67	0.02	<0.014	49.93	0.97	<0.01	0.06	0.07	100.00
<b>GEO-7 P-1B (A)</b>	<b>08-2418</b>	<0.02	16.45	0.06	0.32	0.08	0.023	0.76	0.03	<0.014	35.08	0.74	<0.01	<0.01	<0.01	78.06
<b>GEO-7 P-1B (B)</b>	<b>08-2418</b>	<0.02	16.45	0.05	0.36	0.08	0.023	0.79	0.03	<0.014	35.08	0.80	<0.01	<0.01	<0.01	78.30
	Avg.	<0.02	16.45	0.05	0.34	0.08	0.023	0.77	0.03	<0.014	35.08	0.77	<0.01	<0.01	<0.01	78.18
	Anhyd.	<0.02	20.51	0.06	0.43	0.10	0.029	0.96	0.04	<0.014	43.76	0.96	<0.01	<0.01	<0.02	97.51
	FBSR	<0.02	20.74	0.07	0.43	0.10	0.029	0.97	0.04	<0.014	44.25	0.97	<0.01	<0.01	<0.02	98.60
<b>GEO-7 WTP-SW (A)</b>	<b>08-2413</b>	<0.02	14.56	0.03	0.15	0.02	0.020	0.62	0.01	<0.014	32.09	0.76	<0.01	0.05	0.06	71.76
<b>GEO-7 WTP-SW (B)</b>	<b>08-2413</b>	<0.02	14.83	0.04	0.14	0.02	0.020	0.65	0.01	<0.014	32.09	0.73	<0.01	0.04	0.05	71.85
	Avg.	<0.02	14.69	0.03	0.14	0.02	0.020	0.63	0.01	<0.014	32.09	0.75	<0.01	0.04	0.06	71.81
	Anhyd.	<0.02	18.34	0.04	0.18	0.03	0.025	0.79	0.02	<0.014	40.04	0.93	<0.01	0.06	0.07	89.61
	FBSR	<0.02	19.77	0.04	0.19	0.03	0.027	0.85	0.02	<0.014	43.17	1.01	<0.01	0.06	0.08	96.60

Table 38. Chemical Composition Data for 3" x 6" Cylinders with L-TEM

Sample ID	Lab ID															
	(wt%)	Ag <sub>2</sub> O	Al <sub>2</sub> O <sub>3</sub>	As <sub>2</sub> O <sub>3</sub>	B <sub>2</sub> O <sub>3</sub>	BaO	CaO	CdO	Cl	Cr <sub>2</sub> O <sub>3</sub>	Cs <sub>2</sub> O	F	Fe <sub>2</sub> O <sub>3</sub>	I	K <sub>2</sub> O	MgO
L-TEM, 08-13, 76% (A)	08-2420	<0.01	17.84	<0.013	<0.32	0.06	13.68	0.02	0.11	0.05	0.10	<0.05	0.74	0.080	0.24	0.15
L-TEM, 08-13, 76% (B)	08-2420	<0.01	18.03	<0.013	<0.32	0.06	13.74	0.01	0.11	0.05	0.10	<0.05	0.68	0.080	0.24	0.15
	Avg.	<0.01	17.93	<0.013	<0.32	0.06	13.71	0.01	0.11	0.05	0.10	<0.05	0.71	0.080	0.24	0.15
	Anhyd.	<0.01	24.13	<0.013	<0.43	0.08	18.45	0.02	0.15	0.07	0.13	<0.07	0.95	0.107	0.32	0.20
	FBSR	<0.02	26.33	<0.013	<0.47	0.09	20.14	0.02	0.16	0.08	0.15	<0.07	1.04	0.117	0.35	0.22
L-TEM, 08-14, 80% (A)	08-2421	<0.01	17.97	<0.013	<0.32	0.07	13.89	0.01	0.11	0.06	0.11	<0.05	0.60	0.056	0.21	0.13
L-TEM, 08-14, 80% (B)	08-2421	<0.01	18.05	<0.013	0.32	0.07	13.94	0.02	0.11	0.05	0.11	<0.05	0.61	0.056	0.23	0.13
	Avg.	<0.01	18.01	<0.013	<0.32	0.07	13.92	0.01	0.11	0.06	0.11	<0.05	0.60	0.056	0.22	0.13
	Anhyd.	<0.01	24.09	<0.013	<0.43	0.09	18.62	0.02	0.14	0.07	0.14	<0.07	0.81	0.075	0.30	0.17
	FBSR	<0.02	26.42	<0.013	<0.47	0.10	20.41	0.02	0.16	0.08	0.16	<0.07	0.89	0.082	0.32	0.19
L-TEM, 08-15, 82.5% (A)	08-2422	<0.01	19.84	<0.013	<0.32	0.07	10.89	0.02	0.12	0.06	0.13	<0.05	0.55	0.071	0.22	0.13
L-TEM, 08-15, 82.5% (B)	08-2422	<0.01	20.22	<0.013	<0.32	0.07	11.21	0.02	0.12	0.06	0.13	<0.05	0.54	0.071	0.21	0.12
	Avg.	<0.01	20.03	<0.013	<0.32	0.07	11.05	0.02	0.12	0.06	0.13	<0.05	0.55	0.071	0.22	0.12
	Anhyd.	<0.01	23.17	<0.013	<0.37	0.08	12.78	0.02	0.14	0.07	0.15	<0.06	0.63	0.082	0.25	0.14
	FBSR	<0.01	25.50	<0.013	<0.41	0.09	14.07	0.02	0.15	0.08	0.16	<0.06	0.70	0.091	0.28	0.16

Sample ID	Lab ID													
	(wt%)	MnO <sub>2</sub>	Na <sub>2</sub> O	NiO	PO <sub>4</sub>	PbO	ReO <sub>2</sub>	SO <sub>4</sub>	Sb <sub>2</sub> O <sub>3</sub>	SeO <sub>2</sub>	SiO <sub>2</sub>	TiO <sub>2</sub>	ZnO	Total
L-TEM, 08-13, 76% (A)	08-2420	<0.02	9.98	0.04	0.35	0.06	0.015	1.58	0.03	<0.014	25.03	0.64	<0.01	69.71
L-TEM, 08-13, 76% (B)	08-2420	<0.02	9.66	0.03	0.36	0.06	0.015	1.64	0.02	<0.014	25.03	0.65	<0.01	69.46
	Avg.	<0.02	9.82	0.03	0.36	0.06	0.015	1.61	0.02	<0.014	25.03	0.64	<0.01	69.59
	Anhyd.	<0.02	13.21	0.05	0.48	0.08	0.020	2.17	0.03	<0.014	33.68	0.86	<0.02	93.64
	FBSR	<0.02	14.42	0.05	0.52	0.09	0.022	2.36	0.03	<0.014	36.75	0.94	<0.02	102.18
L-TEM, 08-14, 80% (A)	08-2421	<0.02	10.49	0.03	0.40	0.07	0.008	1.41	0.01	<0.014	25.03	0.68	<0.01	70.29
L-TEM, 08-14, 80% (B)	08-2421	<0.02	10.45	0.04	0.39	0.07	0.008	1.47	0.03	<0.014	25.24	0.68	<0.01	70.78
	Avg.	<0.02	10.47	0.03	0.40	0.07	0.008	1.44	0.02	<0.014	25.14	0.68	<0.01	70.54
	Anhyd.	<0.02	14.00	0.05	0.53	0.09	0.011	1.93	0.03	<0.014	33.63	0.91	<0.02	94.37
	FBSR	<0.02	15.36	0.05	0.58	0.10	0.012	2.11	0.03	<0.014	36.87	1.00	<0.02	103.48
L-TEM, 08-15, 82.5% (A)	08-2422	<0.02	11.03	0.03	0.35	0.07	0.016	1.50	0.02	<0.014	26.31	0.66	<0.01	70.97
L-TEM, 08-15, 82.5% (B)	08-2422	<0.02	10.96	0.04	0.41	0.07	0.016	1.47	0.02	<0.014	26.53	0.68	<0.01	71.95
	Avg.	<0.02	10.99	0.04	0.38	0.07	0.016	1.49	0.02	<0.014	26.42	0.67	<0.01	71.46
	Anhyd.	<0.02	12.71	0.04	0.44	0.08	0.018	1.72	0.02	<0.014	30.56	0.77	<0.01	82.65
	FBSR	<0.02	14.00	0.05	0.48	0.09	0.020	1.89	0.03	<0.014	33.64	0.85	<0.02	90.98

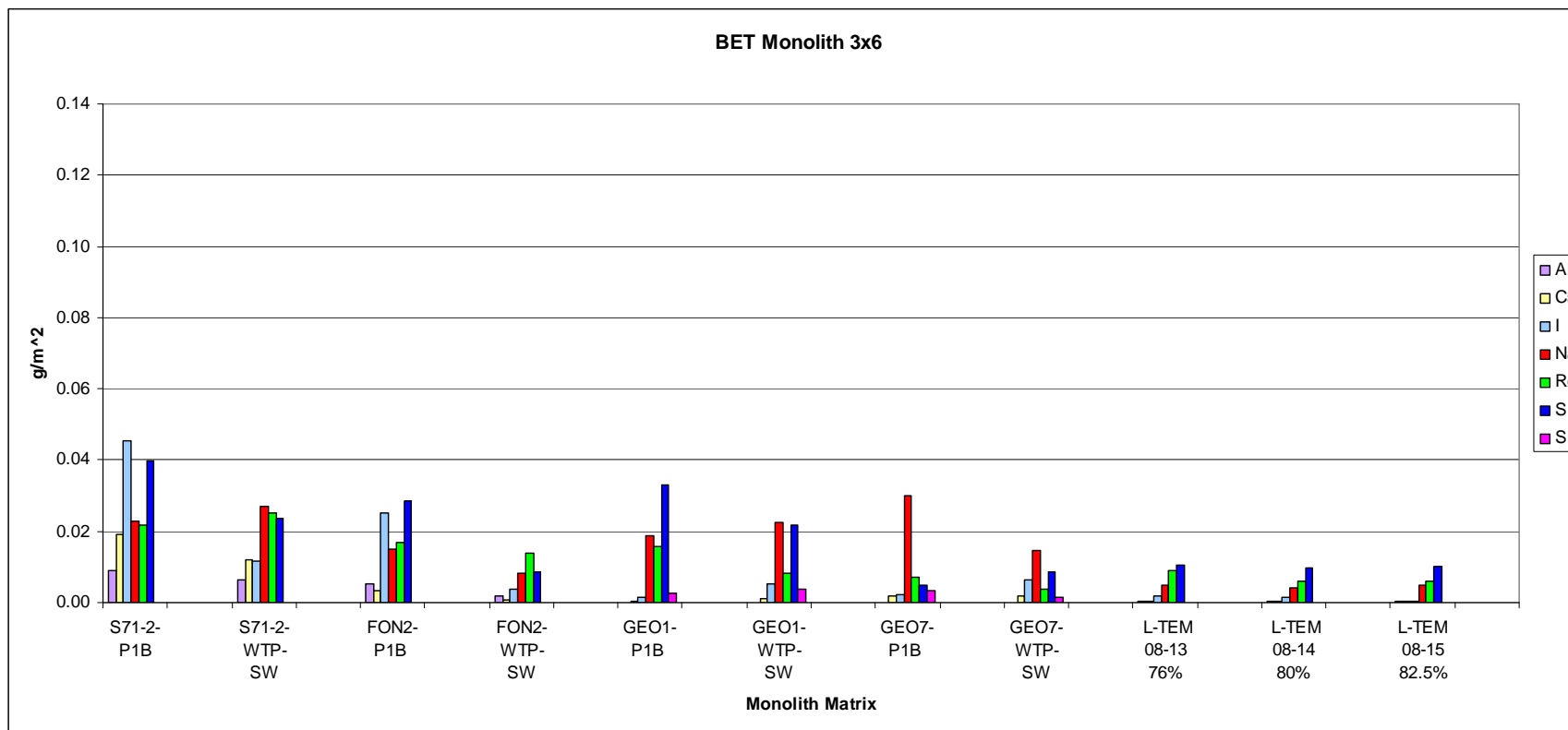
Results of the 7-day PCT performed on the 3" x 6" cylinders are shown in Table 39. All leachate values are shown in mg/L units for Al, Cs, I, Na, Re, S and Si along with the resulting final leachate pHs. The surface area of the non-roasted PCT-prepared powders are also shown from BET measurements in units of m<sup>2</sup>/g. All BET surface area data for the 3" x 6" cylinders containing P-1B use previous BET measurements that were performed on the 2" cubes. The BET surface area shown for the WTP-SW containing 3" x 6" cylinders were actually measured from the 3" x 6" cylinder PCT prepared powders. The BET SA data for the WTP-SW containing monolith powders required pre-BET SA evacuation temperatures of 300 °C heating overnight. The BET SA data for the LTEM samples required 90 °C heating overnight. The longer heating times in the evacuation of the WTP-SW containing monolith powders were required presumably due to the relatively higher levels of residual carbon in the FBSR blends that went into making these monoliths. Microtrac particle size data for the 3" x 6" WTP-SW cylinders and the L-TEM samples are shown in Appendix 12. Microtrac data shown in Appendix 12 indicates that the WTP-SW monolith PCT powders show particle size distributions centered around 85 to 121 microns. However, for the FON2-WTP-SW and the GEO-7 WTP-SW, the size distributions indicate a significant 'tail' of fines around the 10 micron region. Similar data for the L-TEM samples indicate particle size distributions centered around 109 to 124 microns with the single L-TEM 08-13 76%-1 sample showing a significant tail in the 10 micron region.

All SA/V and normalized release data were calculated similarly to the 2" cube data by mathematically removing the moisture and the coal from the measured elemental composition data. The adjusted elemental compositions for the 3" x 6" cylinders (anhydrous, coal-free basis) were used to normalize the PCT release for better comparison to the previous aggregate (coal removed by LOI) and blend (coal removed by calculation) PCT data. Normalized release data are shown in Figure 8. The y-axis of Figure 8 is shown as maximum of 0.14 g/m<sup>2</sup> to show the same scale as previous PCT data for bed/fines, blended aggregates and 2" cube data.

Table 39. PCT Data for 3" x 6" Cylinders

As Measured Leachate Concentrations										
	Al (mg/L)	Cs (mg/L)	I (mg/L)	Na (mg/L)	Re (mg/L)	S (mg/L)	Si (mg/L)	pH	BET (m <sup>2</sup> /g) Non-roasted	SA/V (m <sup>-1</sup> )
S71-2 P-1B	1841.7	28.0	22.9	2183.3	4.2	143.3	12.2	11.92	9.2	921,000
S71-2-WTP-SW	1228.4	33.1	1.8	2116.7	5.2	53.1	16.1	11.52	9.0	898,650
FON2 P-1B	1446.9	9.1	19.7	2481.1	3.5	162.3	5.0	12.19	15.5	1,550,000
FON2-WTP-SW	924.1	4.8	1.4	2100.0	3.8	71.0	8.0	11.96	30.8	3,082,520
GEO1 P-1B	5.9	0.7	1.6	4416.7	2.8	181.4	895.8	12.09	15.2	1,520,000
GEO1-WTP-SW	6.8	3.3	0.8	4183.3	2.3	61.1	1073.0	11.98	12.5	1,254,860
GEO7 P-1B	9.5	3.1	1.8	5416.7	2.1	17.9	835.1	12.18	11.7	1,169,500
GEO7-WTP-SW	0.9	11.1	2.2	5758.3	2.4	65.8	902.5	11.97	26.9	2,685,530
L-TEM 08-13 76%-1	307.7	3.1	9.5	2416.6	8.2	389.3	16.6	12.44	46.5	4,652,270
L-TEM 08-14 80%-1	296.0	2.0	6.6	2341.7	7.1	335.2	17.2	12.36	48.8	4,877,360
L-TEM 08-15 82.5%-1	228.5	1.6	2.1	2166.7	6.8	279.6	32.5	12.34	43.2	4,319,500
Normalized Releases Calculated from Equations 1 & 2										
	NL <sub>Al</sub> (g/m <sup>2</sup> )	NL <sub>Cs</sub> (g/m <sup>2</sup> )	NL <sub>I</sub> (g/m <sup>2</sup> )	NL <sub>Na</sub> (g/m <sup>2</sup> )	NL <sub>Re</sub> (g/m <sup>2</sup> )	NL <sub>S</sub> (g/m <sup>2</sup> )	NL <sub>Si</sub> (g/m <sup>2</sup> )			
S71-2 P-1B	8.9E-03	1.9E-02	4.5E-02	2.3E-02	2.2E-02	4.0E-02	9.5E-05			
S71-2-WTP-SW	6.5E-03	1.2E-02	1.2E-02	2.7E-02	2.5E-02	2.4E-02	1.3E-04			
FON2 P-1B	5.1E-03	3.3E-03	2.5E-02	1.5E-02	1.7E-02	2.9E-02	2.1E-05			
FON2-WTP-SW	1.8E-03	5.9E-04	3.8E-03	8.1E-03	1.4E-02	8.5E-03	1.8E-05			
GEO1 P-1B	2.6E-05	2.8E-04	1.5E-03	1.9E-02	1.6E-02	3.3E-02	2.5E-03			
GEO1-WTP-SW	4.0E-05	9.8E-04	5.3E-03	2.3E-02	8.4E-03	2.2E-02	3.7E-03			
GEO7 P-1B	5.7E-05	2.0E-03	2.2E-03	3.0E-02	7.3E-03	4.7E-03	3.5E-03			
GEO7-WTP-SW	2.4E-06	1.8E-03	6.5E-03	1.5E-02	3.9E-03	8.7E-03	1.7E-03			
L-TEM 08-13 76%-1	4.7E-04	4.9E-04	1.7E-03	4.9E-03	9.1E-03	1.1E-02	2.1E-05			
L-TEM 08-14 80%-1	4.3E-04	2.8E-04	1.7E-03	4.2E-03	6.1E-03	9.7E-03	2.0E-05			
L-TEM 08-15 82.5%-1	3.9E-04	2.5E-04	5.4E-04	4.8E-03	5.9E-03	1.0E-02	4.8E-05			





**Figure 8. PCT Release for 3" x 6" Cylinders**

All chemical composition data for the 6" x 12" cylinders are shown below in Table 40 for the high aluminate binders and Table 41 for the geopolymers. Nominal oxide compositions are shown that are calculated from the measured elemental concentrations from the dissolved solids.

**Table 40. Chemical Composition for 6" x 12" Cylinders with S71 and FONDU**

Sample ID	Lab ID															
	(wt%)	Ag <sub>2</sub> O	Al <sub>2</sub> O <sub>3</sub>	As <sub>2</sub> O <sub>3</sub>	B <sub>2</sub> O <sub>3</sub>	BaO	CaO	CdO	Cl	Cr <sub>2</sub> O <sub>3</sub>	Cs <sub>2</sub> O	F	Fe <sub>2</sub> O <sub>3</sub>	I	K <sub>2</sub> O	MgO
S71 P-1B (A)	08-2412	<0.01	32.84	<0.013	<0.32	0.09	7.79	0.02	0.09	0.06	0.14	0.07	0.52	0.043	0.18	0.10
S71 P-1B (B)	08-2412	<0.01	32.48	<0.013	<0.32	0.09	7.72	0.02	0.09	0.06	0.14	0.07	0.53	0.043	0.18	0.10
	Avg.	<0.01	32.66	<0.013	<0.32	0.09	7.75	0.02	0.09	0.06	0.14	0.07	0.52	0.043	0.18	0.10
	Anhyd.	<0.01	39.61	<0.013	<0.39	0.11	9.40	0.03	0.11	0.08	0.17	0.08	0.64	0.052	0.22	0.12
	FBSR	<0.01	40.12	<0.013	<0.40	0.11	9.52	0.03	0.11	0.08	0.18	0.08	0.64	0.053	0.22	0.12
S71 WTP-SW (A)	08-2419	<0.01	27.76	<0.013	0.88	<0.01	8.37	<0.01	0.70	0.06	0.22	0.39	0.64	0.008	0.23	0.14
S71 WTP-SW (B)	08-2419	<0.01	28.10	<0.013	0.75	<0.01	8.49	<0.01	0.70	0.06	0.22	0.39	0.66	0.008	0.23	0.14
	Avg.	<0.01	27.93	<0.013	0.81	<0.01	8.43	<0.01	0.70	0.06	0.22	0.39	0.65	0.008	0.23	0.14
	Anhyd.	<0.01	35.49	<0.013	1.03	<0.01	10.71	<0.01	0.89	0.08	0.28	0.50	0.83	0.011	0.29	0.17
	FBSR	<0.01	38.63	<0.013	1.13	<0.02	11.66	0.02	0.97	0.09	0.30	0.54	0.90	0.012	0.32	0.19
FON P-1B (A)	08-2414	<0.01	24.68	<0.013	<0.32	0.09	9.14	0.02	0.13	0.10	0.14	0.08	4.18	0.033	0.19	0.21
FON P-1B (B)	08-2414	<0.01	25.04	<0.013	<0.32	0.09	9.26	0.02	0.13	0.09	0.14	0.08	4.22	0.033	0.20	0.22
	Avg.	<0.01	24.86	<0.013	<0.32	0.09	9.20	0.02	0.13	0.09	0.14	0.08	4.20	0.033	0.19	0.21
	Anhyd.	<0.01	30.25	<0.013	<0.39	0.11	11.19	0.03	0.16	0.11	0.17	0.09	5.11	0.040	0.24	0.26
	FBSR	<0.01	30.63	<0.013	<0.40	0.11	11.33	0.03	0.16	0.11	0.17	0.09	5.17	0.040	0.24	0.26
FON- WTP-SW (A)	08-2415	0.02	20.54	<0.013	0.35	<0.01	9.55	<0.01	0.80	0.09	0.20	0.40	4.22	0.009	0.24	0.24
FON- WTP-SW (B)	08-2415	0.02	21.11	<0.013	0.35	<0.01	9.77	<0.01	0.80	0.08	0.20	0.40	4.30	0.009	0.24	0.25
	Avg.	0.02	20.82	<0.013	0.35	<0.01	9.66	<0.01	0.80	0.09	0.20	0.40	4.26	0.009	0.24	0.25
	Anhyd.	0.02	25.02	<0.013	0.42	<0.01	11.61	<0.01	0.96	0.10	0.24	0.48	5.12	0.011	0.29	0.30
	FBSR	0.03	27.25	<0.013	0.46	<0.01	12.64	<0.01	1.04	0.11	0.26	0.52	5.58	0.012	0.32	0.32

Table 40. Chemical Composition for 6" x 12" Cylinders with S71 and FONDU, continued

Sample ID	Lab ID	MnO <sub>2</sub>	Na <sub>2</sub> O	NiO	PO <sub>4</sub>	PbO	ReO <sub>2</sub>	SO <sub>4</sub>	Sb <sub>2</sub> O <sub>3</sub>	SeO <sub>2</sub>	SiO <sub>2</sub>	TiO <sub>2</sub>	ZnO	Total
	(wt%)													
<b>S71 P-1B (A)</b>	08-2412	<0.02	12.56	0.06	0.48	0.11	0.011	1.87	0.04	<0.014	25.18	0.78	<0.01	83.39
<b>S71 P-1B (B)</b>	08-2412	<0.02	12.43	0.05	0.48	0.11	0.011	1.87	0.03	<0.014	25.16	0.78	<0.01	82.81
	Avg.	<0.02	12.49	0.06	0.48	0.11	0.011	1.87	0.04	<0.014	25.17	0.78	<0.01	83.10
	Anhyd.	<0.02	15.15	0.07	0.58	0.14	0.013	2.27	0.04	<0.014	30.52	0.95	<0.02	100.79
	FBSR	<0.02	15.35	0.07	0.59	0.14	0.013	2.30	0.04	<0.014	30.91	0.96	<0.02	102.07
<b>S71 WTP-SW (A)</b>	08-2419	<0.02	9.61	0.04	0.18	0.02	0.008	0.80	0.02	<0.014	20.58	0.67	0.07	71.43
<b>S71 WTP-SW (B)</b>	08-2419	<0.02	9.73	0.04	0.18	0.02	0.008	0.80	0.01	<0.014	20.50	0.67	0.07	71.80
	Avg.	<0.02	9.67	0.04	0.18	0.02	0.008	0.80	0.02	<0.014	20.54	0.67	0.07	71.62
	Anhyd.	<0.02	12.29	0.05	0.23	0.03	0.011	1.01	0.02	<0.014	26.10	0.85	0.08	91.00
	FBSR	<0.02	13.38	0.06	0.25	0.03	0.011	1.10	0.02	<0.014	28.42	0.93	0.09	99.07
<b>FON P-1B (A)</b>	08-2414	0.07	11.03	0.06	0.51	0.12	0.014	1.26	0.04	<0.014	23.66	1.14	<0.01	77.22
<b>FON P-1B (B)</b>	08-2414	0.11	11.24	0.08	0.51	0.12	0.014	1.26	0.04	<0.014	23.40	1.15	<0.01	77.77
	Avg.	0.09	11.13	0.07	0.51	0.12	0.014	1.26	0.04	<0.014	23.53	1.15	<0.01	77.49
	Anhyd.	0.11	13.54	0.08	0.62	0.15	0.017	1.53	0.05	<0.014	28.63	1.39	<0.02	94.29
	FBSR	0.11	13.72	0.09	0.63	0.15	0.017	1.55	0.05	<0.014	29.00	1.41	<0.02	95.50
<b>FON- WTP-SW (A)</b>	08-2415	0.07	8.91	0.05	0.21	0.02	0.011	1.28	0.02	<0.014	19.89	1.03	0.06	68.21
<b>FON- WTP-SW (B)</b>	08-2415	0.07	8.97	0.05	0.21	0.02	0.011	1.28	0.02	<0.014	20.46	1.04	0.06	69.74
	Avg.	0.07	8.94	0.05	0.21	0.02	0.011	1.28	0.02	<0.014	20.18	1.03	0.06	68.97
	Anhyd.	0.08	10.74	0.06	0.25	0.02	0.013	1.54	0.02	<0.014	24.25	1.24	0.07	82.89
	FBSR	0.09	11.70	0.06	0.27	0.03	0.014	1.68	0.02	<0.014	26.40	1.35	0.08	90.26

**Table 41. Chemical Composition Data for 6" x 12" Cylinders with GEO-1 and GEO-7**

Sample ID	Lab ID															
	(wt%)	Ag <sub>2</sub> O	Al <sub>2</sub> O <sub>3</sub>	As <sub>2</sub> O <sub>3</sub>	B <sub>2</sub> O <sub>3</sub>	BaO	CaO	CdO	Cl	Cr <sub>2</sub> O <sub>3</sub>	Cs <sub>2</sub> O	F	Fe <sub>2</sub> O <sub>3</sub>	I	K <sub>2</sub> O	MgO
GEO-1 P-1B (A)	08-2416	<0.01	22.07	<0.013	<0.32	0.09	0.13	0.03	0.13	0.06	0.12	0.05	1.04	0.059	0.25	0.04
GEO-1 P-1B (B)	08-2416	<0.01	22.18	<0.013	<0.32	0.09	0.13	0.03	0.13	0.07	0.12	0.05	1.05	0.059	0.26	0.04
	Avg.	<0.01	22.13	<0.013	<0.32	0.09	0.13	0.03	0.13	0.07	0.12	0.05	1.05	0.059	0.26	0.04
	Anhyd.	<0.01	28.09	<0.013	<0.41	0.11	0.17	0.03	0.16	0.09	0.15	0.06	1.33	0.075	0.32	0.05
	FBSR	<0.01	28.42	<0.013	<0.41	0.12	0.17	0.03	0.17	0.09	0.15	0.06	1.34	0.076	0.33	0.05
GEO-1 WTP-SW (A)	08-2417	<0.01	18.80	<0.013	<0.32	<0.01	0.16	<0.01	0.71	0.06	0.17	0.72	1.07	0.011	0.32	0.07
GEO-1 WTP-SW (B)	08-2417	<0.01	18.60	<0.013	<0.32	<0.01	0.17	<0.01	0.71	0.06	0.17	0.72	1.03	0.011	0.34	0.07
	Avg.	<0.01	18.70	<0.013	<0.32	<0.01	0.16	<0.01	0.71	0.06	0.17	0.72	1.05	0.011	0.33	0.07
	Anhyd.	<0.01	23.96	<0.013	<0.41	<0.01	0.21	<0.01	0.91	0.08	0.22	0.92	1.34	0.014	0.42	0.09
	FBSR	<0.01	25.88	<0.013	<0.45	<0.02	0.23	<0.02	0.98	0.08	0.24	1.00	1.45	0.015	0.46	0.09
GEO-7 P-1B (A)	08-2418	<0.01	19.73	<0.013	<0.32	0.09	0.27	0.02	0.11	0.05	0.10	0.08	2.05	0.045	0.72	0.20
GEO-7 P-1B (B)	08-2418	<0.01	19.88	<0.013	<0.32	0.09	0.27	0.02	0.11	0.06	0.10	0.08	2.02	0.045	0.72	0.20
	Avg.	<0.01	19.80	<0.013	<0.32	0.09	0.27	0.02	0.11	0.06	0.10	0.08	2.03	0.045	0.72	0.20
	Anhyd.	<0.01	24.70	<0.013	<0.40	0.11	0.34	0.02	0.13	0.07	0.12	0.09	2.54	0.056	0.89	0.25
	FBSR	<0.01	24.98	<0.013	<0.41	0.11	0.34	0.02	0.13	0.07	0.12	0.10	2.57	0.057	0.90	0.25
GEO-7 WTP-SW (A)	08-2413	<0.01	18.04	<0.013	<0.32	0.03	0.33	<0.01	0.71	0.06	0.17	0.35	2.74	0.009	0.83	0.24
GEO-7 WTP-SW (B)	08-2413	<0.01	18.05	<0.013	<0.32	0.03	0.33	<0.01	0.71	0.06	0.17	0.35	2.73	0.009	0.83	0.24
	Avg.	<0.01	18.04	<0.013	<0.32	0.03	0.33	<0.01	0.71	0.06	0.17	0.35	2.74	0.009	0.83	0.24
	Anhyd.	<0.01	22.52	<0.013	<0.40	0.03	0.41	<0.01	0.89	0.08	0.21	0.44	3.42	0.011	1.04	0.30
	FBSR	<0.01	24.25	<0.013	<0.43	0.03	0.44	<0.02	0.95	0.08	0.23	0.47	3.68	0.012	1.12	0.32

Table 41. Chemical Composition Data for 6" x 12" Cylinders with GEO-1 and GEO-7, continued

Sample ID	Lab ID	MnO <sub>2</sub>	Na <sub>2</sub> O	NiO	PO <sub>4</sub>	PbO	ReO <sub>2</sub>	SO <sub>4</sub>	Sb <sub>2</sub> O <sub>3</sub>	SeO <sub>2</sub>	SiO <sub>2</sub>	TiO <sub>2</sub>	ZnO	Total
	(wt%)													
GEO-1 P-1B (A)	08-2416	<0.02	17.19	0.05	0.43	0.10	0.020	5.25	0.04	<0.013	39.45	0.94	<0.01	87.88
GEO-1 P-1B (B)	08-2416	<0.02	17.34	0.06	0.43	0.10	0.020	5.25	0.03	<0.013	39.36	0.94	<0.01	88.09
	Avg.	<0.02	17.26	0.06	0.43	0.10	0.020	5.25	0.04	<0.013	39.40	0.94	<0.01	87.98
	Anhyd.	<0.02	21.92	0.07	0.54	0.13	0.026	6.67	0.05	<0.013	50.03	1.19	<0.02	111.72
	FBSR	<0.02	22.18	0.07	0.55	0.13	0.026	6.74	0.05	<0.013	50.61	1.20	<0.02	113.01
GEO-1 WTP-SW (A)	08-2417	<0.02	16.35	0.04	0.19	0.02	0.011	0.91	0.01	<0.013	37.03	0.80	0.06	77.89
GEO-1 WTP-SW (B)	08-2417	<0.02	16.06	0.04	0.18	0.03	0.011	0.91	0.01	<0.013	36.73	0.78	0.06	77.05
	Avg.	<0.02	16.20	0.04	0.19	0.03	0.011	0.91	0.01	<0.013	36.88	0.79	0.06	77.47
	Anhyd.	<0.02	20.76	0.05	0.24	0.03	0.015	1.17	0.01	<0.013	47.25	1.01	0.08	99.27
	FBSR	<0.02	22.43	0.05	0.26	0.04	0.016	1.26	0.02	<0.013	51.05	1.09	0.08	107.23
GEO-7 P-1B (A)	08-2418	<0.02	17.41	0.05	0.44	0.08	0.012	0.98	0.03	<0.013	33.48	0.82	<0.01	77.10
GEO-7 P-1B (B)	08-2418	<0.02	17.60	0.05	0.44	0.09	0.012	0.98	0.03	<0.013	33.74	0.82	<0.01	77.68
	Avg.	<0.02	17.50	0.05	0.44	0.08	0.012	0.98	0.03	<0.013	33.61	0.82	<0.01	77.39
	Anhyd.	<0.02	21.83	0.06	0.55	0.11	0.014	1.22	0.04	<0.013	41.92	1.03	<0.02	96.52
	FBSR	<0.02	22.08	0.06	0.56	0.11	0.015	1.23	0.04	<0.013	42.39	1.04	<0.02	97.60
GEO-7 WTP-SW (A)	08-2413	<0.02	15.53	0.04	0.20	0.02	0.009	1.76	0.01	<0.013	32.58	0.84	0.06	74.91
GEO-7 WTP-SW (B)	08-2413	<0.02	15.65	0.04	0.20	0.02	0.009	1.76	0.01	<0.013	32.52	0.84	0.06	74.98
	Avg.	<0.02	15.59	0.04	0.20	0.02	0.009	1.76	0.01	<0.013	32.55	0.84	0.06	74.94
	Anhyd.	<0.02	19.46	0.05	0.25	0.03	0.012	2.20	0.02	<0.013	40.62	1.04	0.07	93.52
	FBSR	0.02	20.96	0.05	0.27	0.03	0.012	2.37	0.02	<0.013	43.75	1.13	0.08	100.75

The 6" x 12" cylinders were fabricated in duplicate in the N-Area Civil Testing Facility at SRS. Mixing and loading of the cylinder molds were performed in the test facility laboratory in temperatures of  $\sim 20 \pm 3$  °C. A K-type thermocouple was placed in a single cylinder of each duplicate set and the cylinders were promptly moved and allowed to cure in a constant temperature ( $23 \pm 2$  °C) and humidity curing room. The thermocouple tips were placed near the center of the cylinders on the centerline by using a small drilled centerline hole in the 6" x 12" mold caps. Figure 9 and Figure 10 show the prompt temperature increases observed for the geopolymers that reached maximum temperatures in the 35 – 45 °C range. The GEO-1 LAW cylinders were prepared on the afternoon of 12/16/08 and the GEO-1 WTP-SW cylinders were prepared the next morning. Figure 9 data indicates that the initial ambient temperature data point for the GEO-1 LAW monolith was not recorded but that the prompt maximum temperature attained was  $\sim 35$  °C. Figure 9 data also shows that the morning preparation of the GEO-1 WTP-SW contained the relatively low starting temperature of about 20 °C with prompt temperature increase to 35 °C. The GEO-7 WTP-SW cylinders were prepared on the afternoon of 12/17/08 and show a starting temperature of about 24 °C and the GEO-7 LAW cylinders were prepared the next morning and show a relatively cooler starting temperature of 20 °C. The rapid temperature increase on mixing of these four formulations was noticeable from brief handling of the cylinders immediately after preparation. This temperature increase derives from the exothermic nature of the reactions during setting of the geopolymer matrix.

Figure 11 and Figure 12 show relatively slower temperature increases observed for the high alumina binder monoliths that also reached maximum temperatures in the 35 °C to 45 °C range. These temperature increases are presumably due to the hydration reactions of the water and cement binders. The S71-2 LAW cylinders were prepared on the afternoon of 12/18/09 and the S71-2 WTP-SW cylinders were prepared on the morning of 12/23/08. The FON-2 LAW cylinders were prepared on the afternoon of 12/18/08 and the FON-2 WTP-SW cylinders were prepared on the morning of 12/23/08.

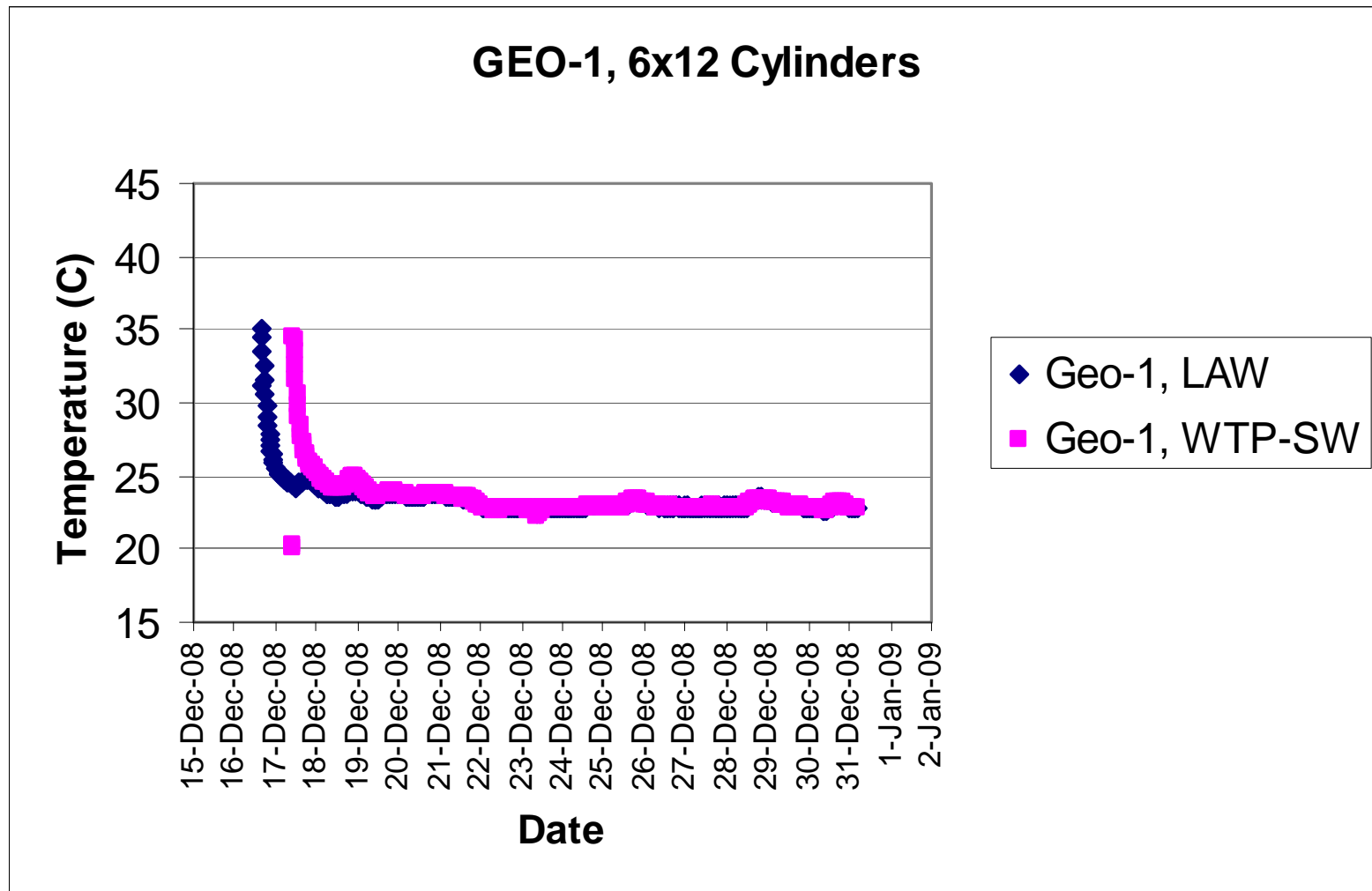


Figure 9. Cure Temperatures for GEO-1 Cylinders

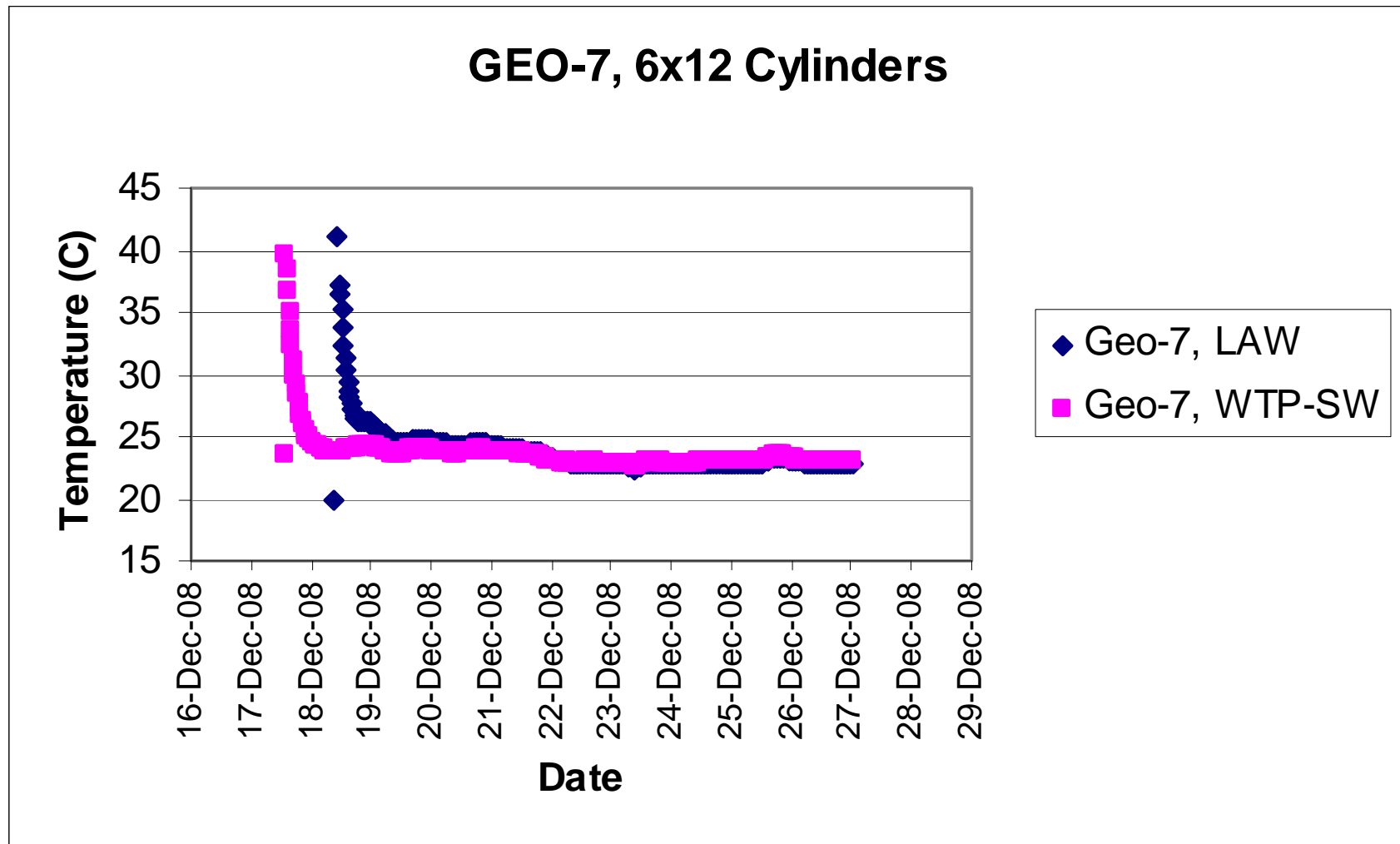


Figure 10. Cure Temperatures for GEO-7 Cylinders



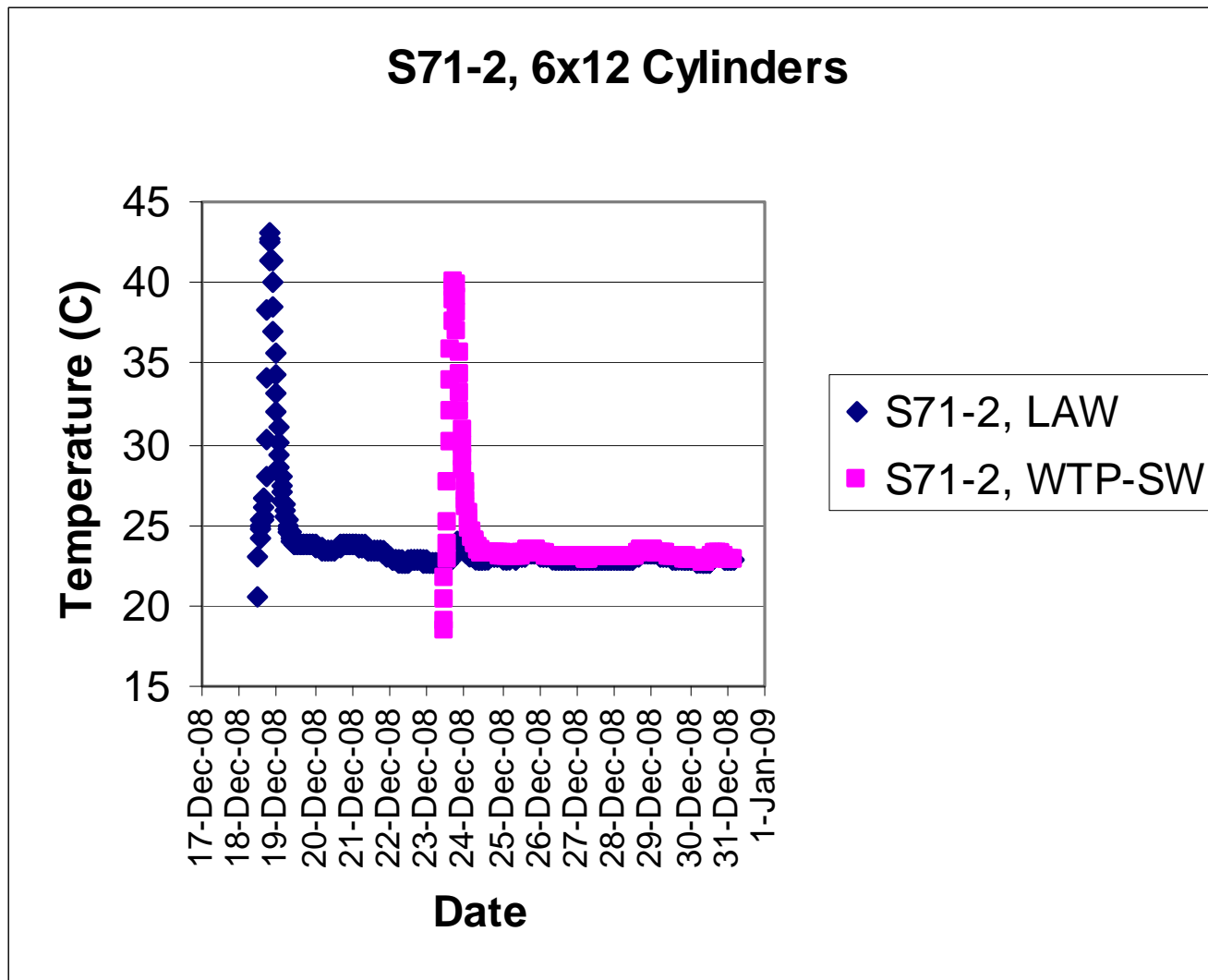


Figure 11. Cure Temperatures for S71-2 Cylinders

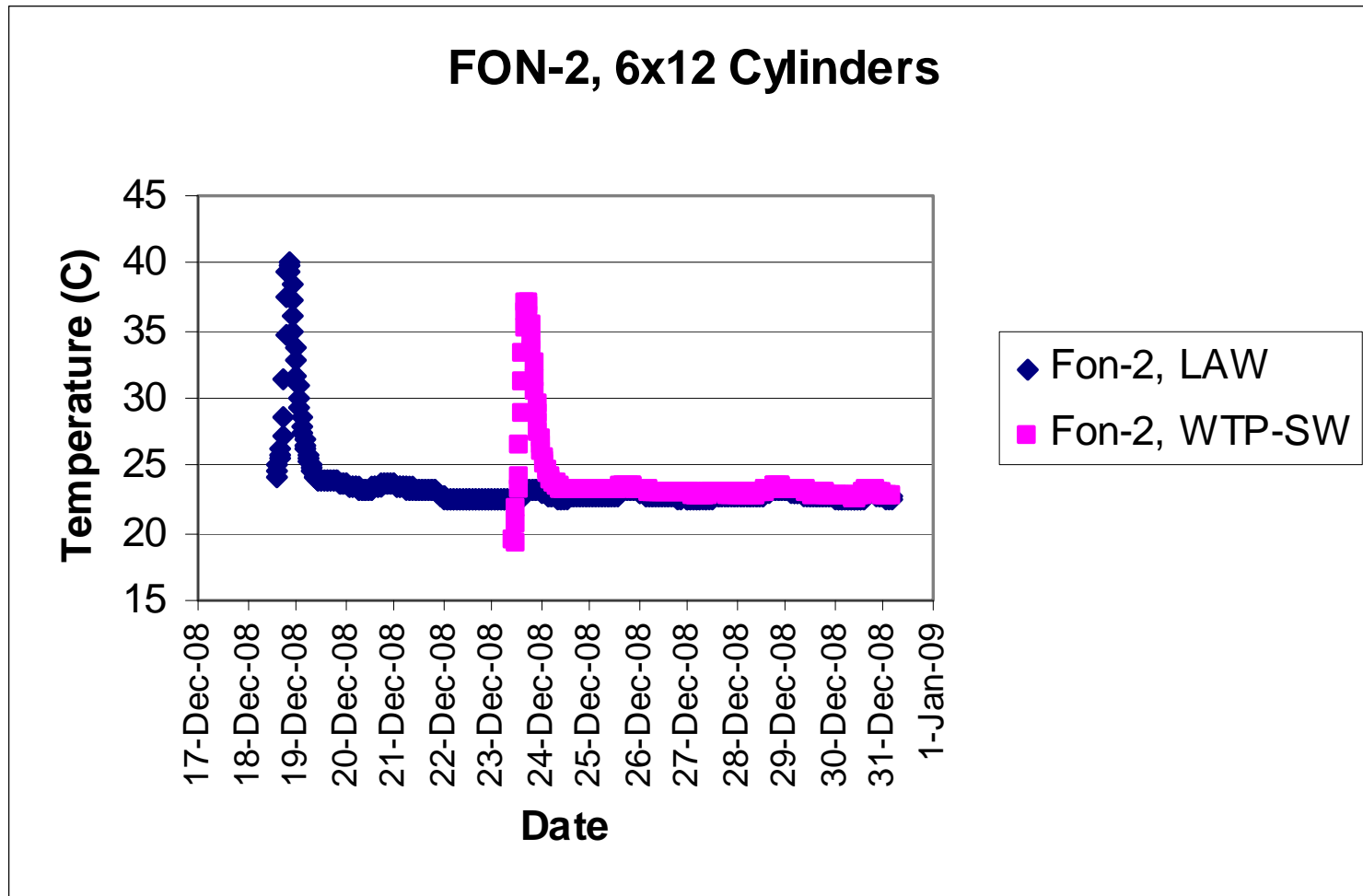
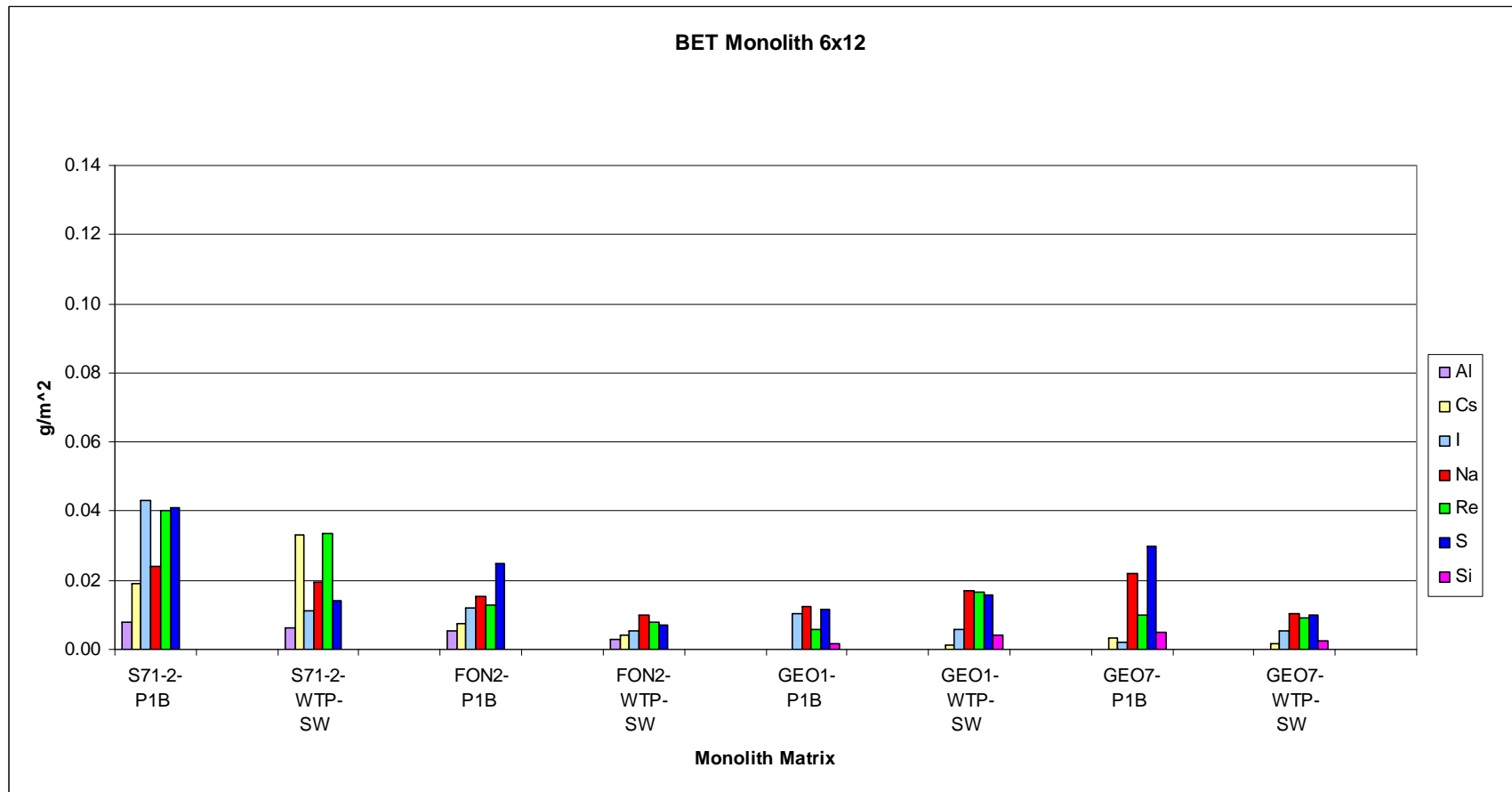


Figure 12. Cure Temperatures for FON-2 Cylinders

Results of the 7-day PCT performed on the 6" x 12" cylinders are shown in Table 42. All leachate values are shown in mg/L units for Al, Cs, I, Na, Re, S and Si along with the resulting final leachate pHs. The measured non-roasted BET surface area of the PCT-prepared powders for the 3" x 6" cylinders (previously shown in Table 39) were also used for the 6" x 12" cylinders. All SA/V and normalized release data were calculated similarly to the 2" cube data by mathematically removing the moisture and the coal from the measured elemental composition data. The adjusted elemental compositions for the 6" x 12" cylinders (anhydrous, coal-free basis) were used to normalize the PCT release for better comparison to the previous aggregate (coal removed by LOI) and blend (coal removed by calculation) PCT data. Normalized PCT release data for the 6" x 12" cylinders are shown in Figure 13. These PCT normalized data are shown again with the y-axis of 0.14 g/m<sup>2</sup> for direct comparison to earlier normalized PCT data in this report.

**Table 42. PCT Data for 6" x 12" Cylinders**

As Measured Leachate Concentrations										
	Al (mg/L)	Cs (mg/L)	I (mg/L)	Na (mg/L)	Re (mg/L)	S (mg/L)	Si (mg/L)	pH	BET (m <sup>2</sup> /g) Non-roasted	SA/V (m <sup>2</sup> )
S71-2 P-1B	1504.5	29.1	20.8	2500.0	5.0	289.0	2.9	12.21	9.21	921,000
S71-2 WTP-SW	1136.9	85.7	1.2	1733.3	3.5	45.8	3.4	12.58	8.99	898,650
FON2 P-1B	1384.5	19.1	7.4	2391.7	3.3	198.4	4.1	12.62	15.50	1,550,000
FON2 WTP-SW	1243.8	31.1	2.0	2633.3	3.5	123.0	3.8	12.46	30.83	3,082,520
GEO1 P-1B	3.1	0.4	11.8	3141.7	2.3	390.6	612.4	12.51	15.20	1,520,000
GEO1- WTP-SW	6.9	3.2	1.1	3525.0	3.3	83.9	1218.4	12.22	12.55	1,254,860
GEO7 P-1B	11.7	4.5	1.3	4233.5	1.7	142.9	1196.2	12.71	11.70	1,169,500
GEO7- WTP-SW	4.6	9.8	1.7	4333.3	3.0	214.2	1424.0	12.71	26.86	2,685,530
Normalized Releases Calculated from Equations 1 & 2										
	NL <sub>Al</sub> (g/m <sup>2</sup> )	NL <sub>Cs</sub> (g/m <sup>2</sup> )	NL <sub>I</sub> (g/m <sup>2</sup> )	NL <sub>Na</sub> (g/m <sup>2</sup> )	NL <sub>Re</sub> (g/m <sup>2</sup> )	NL <sub>S</sub> (g/m <sup>2</sup> )	NL <sub>Si</sub> (g/m <sup>2</sup> )			
S71-2 P-1B	7.7E-03	1.9E-02	4.3E-02	2.4E-02	4.0E-02	4.1E-02	2.1E-05			
S71-2 WTP-SW	6.2E-03	3.3E-02	1.1E-02	1.9E-02	3.4E-02	1.4E-02	2.8E-05			
FON2 P-1B	5.5E-03	7.6E-03	1.2E-02	1.5E-02	1.3E-02	2.5E-02	1.9E-05			
FON2 WTP-SW	2.8E-03	4.1E-03	5.5E-03	9.8E-03	7.9E-03	7.1E-03	1.0E-05			
GEO1 P-1B	1.4E-05	1.7E-04	1.0E-02	1.3E-02	5.8E-03	1.1E-02	1.7E-03			
GEO1- WTP-SW	4.0E-05	1.1E-03	5.8E-03	1.7E-02	1.7E-02	1.6E-02	4.1E-03			
GEO7 P-1B	7.6E-05	3.3E-03	2.0E-03	2.2E-02	9.8E-03	3.0E-02	5.2E-03			
GEO7- WTP-SW	1.3E-05	1.7E-03	5.3E-03	1.0E-02	8.9E-03	1.0E-02	2.6E-03			



**Figure 13. Normalized PCT Release for 6" x 12" Cylinders with BET Surface Area Normalization**

#### 4.7 MONOLITH TESTING WITH 2" X 4" CYLINDERS

The final task in this project was to produce a batch of 24 replicate 2" x 4" cylinders containing the WTP-SW with the optimal recipe as determined from previous monolith data (compression testing, TCLP and PCT). It was decided to use the fly-ash-containing GEO-7 recipe for these final monoliths. The 2" x 4" cylinders were batched using the same recipe as applied to the previous 3"x6" and 6"x12" cylinders. The batching was done to produce four replicate cylinders per mixing-batch in the mixer that was previously used for the 3"x6" cylinders. Table 43 shows the additives and the sequencing to produce 6 different replicate batches of four 2" x 4" cylinders. Three batches were produced on 6/1/09 and another three batches were produced on 6/3/09. These cylinders were allowed to cure in the plastic molds for ~ 30 days.

**Table 43. Batching Information for 2" x 4" Cylinders**

Batch / Date	Additives	Amounts (g)
B#1 / 6/1/09	WTP-SW	698.29
	Fly ash (SEFA)	188.72
	Silica-D	262.71
	14 M NaOH	218.47
	ASTM-I water	74.28
B#2 / 6/1/09	WTP-SW	714.70
	Fly ash (SEFA)	193.17
	Silica-D	268.88
	14 M NaOH	223.61
	ASTM-I water	76.02
B#3 / 6/1/09	Same as B#2	Same as B#2
B#4 / 6/3/09	Same as B#2	Same as B#2
B#5 / 6/3/09	Same as B#2	Same as B#2
B#6 / 6/3/09	Same as B#2	Same as B#2

Densities for the 2" x 4" cylinders were measured after batching and at 1-week and 4-week intervals. Densities were simply calculated by the total mass of the monolith and cure mold (minus the empty mass of cure mold) divided by the calculated overall volume of a 2"x 4" mold. Very recent measurements from July 2011 on remaining intact monoliths are also included in Table 44. The remaining intact monoliths had been stored in the original cure molds without caps for the 105 weeks. The data in Table 44 shows that the average density of 1.78 g/cc for the monoliths was constant throughout the initial curing time of the 1<sup>st</sup> four weeks. The 4<sup>th</sup> sample from Batch #1 shown as blue-scale bold text was not included in the averages since this mold was not completely filled during monolith formation. The recent latter 105-week density data performed on monoliths that remained after others were tested shows that the densities have decreased to 1.71 g/cc. The final column in Table 44 shows actual de-molded monolith measured densities using the measured monolith masses and electronic caliper measured geometric height and diameter.

**Table 44. Measured Densities for 2" x 4" Cylinders**

Sample	Empty Mold	Full Day 1	Bulk Density	Full		Full		Full		Bulk Density (Mass/Meas. Vol.)
	(g)	(g)	g/cc	1 Week	Bulk Density	4 Week	Bulk Density	105 Week	Bulk Density	g/cc
1	23.33	384.99	1.76	384.99	1.76	384.97	1.76			
2	23.96	386.03	1.76	386.01	1.76	385.87	1.76			
3	23.60	386.24	1.76	386.24	1.76	386.22	1.76			
4	<b>23.93</b>	<b>363.73</b>	<b>1.65</b>	<b>363.74</b>	<b>1.65</b>	<b>363.73</b>	<b>1.65</b>	<b>349.81</b>	<b>1.58</b>	<b>1.67</b>
5	23.68	390.36	1.78	390.35	1.78	390.27	1.78			
6	23.92	386.75	1.76	386.75	1.76	386.66	1.76			
7	23.36	389.62	1.78	389.61	1.78	389.52	1.78			
8	24.53	394.11	1.80	394.09	1.80	394.04	1.80	386.43	1.76	1.68
9	23.68	386.59	1.76	386.57	1.76	386.48	1.76			
10	24.01	389.59	1.78	389.57	1.78	389.45	1.78			
11	24.23	392.3	1.79	392.24	1.79	392.17	1.79			
12	24.64	393.94	1.79	393.92	1.79	393.17	1.79	384.44	1.75	1.72
13	23.74	390.8	1.78	390.86	1.78	390.82	1.78			
14	23.29	392.57	1.79	392.59	1.79	392.59	1.79			
15	24.30	394.25	1.80	394.27	1.80	394.33	1.80			
16	23.57	386.95	1.77	386.98	1.77	386.97	1.77	377.65	1.72	1.73
17	24.44	390.43	1.78	390.41	1.78	390.35	1.78			
18	24.14	396.99	1.81	396.96	1.81	396.9	1.81			
19	23.55	388.84	1.77	388.8	1.77	388.73	1.77			
20	23.89	386.71	1.76	386.68	1.76	386.59	1.76	376.25	1.71	1.74
21	22.86	391.31	1.79	391.28	1.79	391.21	1.79			
22	22.77	388.34	1.78	388.33	1.78	388.24	1.78			
23	22.63	391.39	1.79	391.36	1.79	391.3	1.79	375.46	1.71	1.70
24	22.60	385.56	1.76	385.54	1.76	385.48	1.76	374.82	1.71	1.72
-	<b>Avg.</b>	<b>389.77</b>	<b>1.78</b>	<b>389.76</b>	<b>1.78</b>	<b>389.67</b>	<b>1.78</b>		<b>1.73</b>	<b>1.71</b>
-	<b>St.Dev.</b>	<b>3.28</b>	<b>0.01</b>	<b>3.27</b>	<b>0.01</b>	<b>3.25</b>	<b>0.01</b>		<b>0.02</b>	<b>0.02</b>
-	<b>%rsd</b>	<b>0.84</b>	<b>0.84</b>	<b>0.84</b>	<b>0.84</b>	<b>0.83</b>	<b>0.84</b>		<b>1.19</b>	<b>1.41</b>

Compression data for a single cylinder taken from four of the batches are shown in Table 45. These data indicate that the compressive strengths of these cylinders in the range of 2,300 to 2,400 psi are ~ 4X the magnitude of the lower compressive strength limit of 500 psi. These data also indicate a very good precision of the different batches with average and standard deviation compressive strength of  $2340 \pm 93$  psi.

**Table 45. Compression Data for 2" x 4" Cylinders**

Sample ID	Date Cast	Date Tested	Sample Size	End Area, sq. in.	Total Load, lbs	Unit load, psi
B#2, No. 5	6-01-09	7-06-09	2" X 4"	3.14	7296	2230
B#3, No. 9					7271	2320
B#4, No. 13	6-03-09				7085	2260
B#5, No. 17					7651	2440

All TCLP data for the 2" x 4" cylinders are shown in Table 46. These data showing all TCLP leachate levels below the UTS limits are in agreement with previous TCLP data shown for the GEO-7 recipe used for WTP-SW monolithing that used 3"x 6" and 6" x 12" cylinders shown previously in Table 34. All chemical composition data for the 2"x 4" cylinders are shown below in Table 47.

**Table 46. TCLP Data for 2"x4" Cylinders**

Element	GEO-7#5 WTP- SW-P-2B	GEO-7#9 WTP- SW -P- 2B	GEO- 7#17 WTP- SW -P- 2B	UTS *	(w 307 g clay/L) P-2B WTP-SW Target	Reporting Detection Limit (RDL)	Method Detection Limit (MDL)
	(mg/L)	(mg/L)	(mg/L)	(mg/L)	(g/L)	(mg/L)	(mg/L)
Sb	1.1	1.1	1.1	1.15	0.175	<b>0.1</b>	0.06
As	0.30	0.28	0.33	5	0.007	<b>0.1</b>	0.05
Ba	1.4	1.5	1.5	21	0.003	<b>0.2</b>	0.004
Cd	<0.05 <sup>U</sup>	<0.05 <sup>U</sup>	<0.05 <sup>U</sup>	0.11	0.088	<b>0.05</b>	0.008
Cr	0.25	0.27	0.23	0.6	0.282	<b>0.1</b>	0.014
Pb	0.18	0.13	0.13	0.75	0.244	<b>0.1</b>	0.008
Se	1.7	1.7	1.7	5.7	0.175	<b>0.1</b>	0.15
Ag	< 0.10 <sup>U</sup>	< 0.10 <sup>U</sup>	< 0.10 <sup>U</sup>	0.14	0.083	<b>0.1</b>	0.014
Hg	<0.002 <sup>U</sup>	<0.002 <sup>U</sup>	<0.002 <sup>U</sup>	0.025	0	<b>0.002</b>	0.0004
Ni	0.2	0.22	<0.2 <sup>U</sup>	11	0.241	<b>0.2</b>	0.01
Tl	0.15	0.14	0.15	0.2	0.175	<b>0.04</b>	0.08
Zn	0.5	0.57	0.47	4.3	0.428	<b>0.2</b>	0.004

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

U = results less than Reporting Detection Limit, or “U” flag

Table 47. Chemical Composition for the 2" x 4" Cylinders

Sample ID	Lab ID															
	(wt%)	Ag <sub>2</sub> O	Al <sub>2</sub> O <sub>3</sub>	As <sub>2</sub> O <sub>3</sub>	B <sub>2</sub> O <sub>3</sub>	BaO	CaO	CdO	Cl	Cr <sub>2</sub> O <sub>3</sub>	Cs <sub>2</sub> O	F	Fe <sub>2</sub> O <sub>3</sub>	I	K <sub>2</sub> O	MgO
GEO7-WTP-SW-5	09-0745	<0.01	17.10	<0.013	0.87	0.03	0.32	<0.11	0.25	0.06	0.21	0.38	2.41	0.006	0.82	0.25
	Anhyd.	<0.01	21.34	<0.013	1.09	0.04	0.40	<0.14	0.31	0.07	0.26	0.47	3.00	0.008	1.02	0.32
	FBSR	<0.01	22.99	<0.013	1.17	0.04	0.43	<0.15	0.33	0.08	0.28	0.50	3.24	0.009	1.10	0.34
GEO7-WTP-SW-9	09-0746	<0.01	17.29	<0.013	0.80	0.03	0.34	<0.11	0.25	0.05	0.22	0.40	2.47	0.007	0.79	0.26
	Anhyd.	<0.01	21.58	<0.013	1.00	0.04	0.42	<0.14	0.31	0.07	0.27	0.49	3.09	0.008	0.99	0.32
	FBSR	<0.01	23.25	<0.013	1.07	0.04	0.46	<0.15	0.34	0.07	0.29	0.53	3.33	0.009	1.07	0.35
GEO7-WTP-SW-17	09-0747	<0.01	16.90	<0.013	0.78	0.03	0.28	<0.11	0.25	0.05	0.20	0.38	2.27	0.007	0.79	0.25
	Anhyd.	<0.01	21.09	<0.013	0.97	0.04	0.35	<0.14	0.32	0.07	0.25	0.48	2.84	0.008	0.98	0.32
	FBSR	<0.01	22.72	<0.013	1.05	0.04	0.38	<0.15	0.34	0.07	0.27	0.51	3.05	0.009	1.06	0.34

Sample ID	Lab ID														
	(wt%)		MnO <sub>2</sub>	Na <sub>2</sub> O	NiO	PO <sub>4</sub>	PbO	ReO <sub>2</sub>	SO <sub>4</sub>	Sb <sub>2</sub> O <sub>3</sub>	SeO <sub>2</sub>	SiO <sub>2</sub>	TiO <sub>2</sub>	ZnO	Total
GEO7-WTP-SW-5	09-0745	<	0.16	14.65	0.03	0.22	0.03	0.017	0.40	0.04	<0.10	33.05	0.80	0.06	72.28
	Anhyd.	<	0.20	18.29	0.04	0.27	0.03	0.021	0.50	0.06	<0.12	41.24	1.00	0.07	90.20
	FBSR	<	0.21	19.70	0.05	0.29	0.03	0.022	0.54	0.06	<0.13	44.43	1.08	0.08	97.17
GEO7-WTP-SW-9	09-0746	<	0.16	14.59	0.03	0.22	0.02	0.015	0.42	0.06	<0.10	33.37	0.79	0.06	72.77
	Anhyd.	<	0.20	18.20	0.04	0.27	0.03	0.019	0.52	0.08	<0.12	41.64	0.99	0.08	90.82
	FBSR	<	0.21	19.61	0.04	0.29	0.03	0.021	0.56	0.08	<0.13	44.86	1.06	0.09	97.83
GEO7-WTP-SW-17	09-0747	<	0.16	14.44	0.03	0.20	0.02	0.016	0.41	0.03	<0.10	32.67	0.79	0.05	71.15
	Anhyd.	<	0.20	18.02	0.04	0.26	0.03	0.020	0.52	0.04	<0.12	40.76	0.99	0.07	88.78
	FBSR	<	0.21	19.41	0.05	0.27	0.03	0.022	0.56	0.04	<0.13	43.91	1.06	0.07	95.64



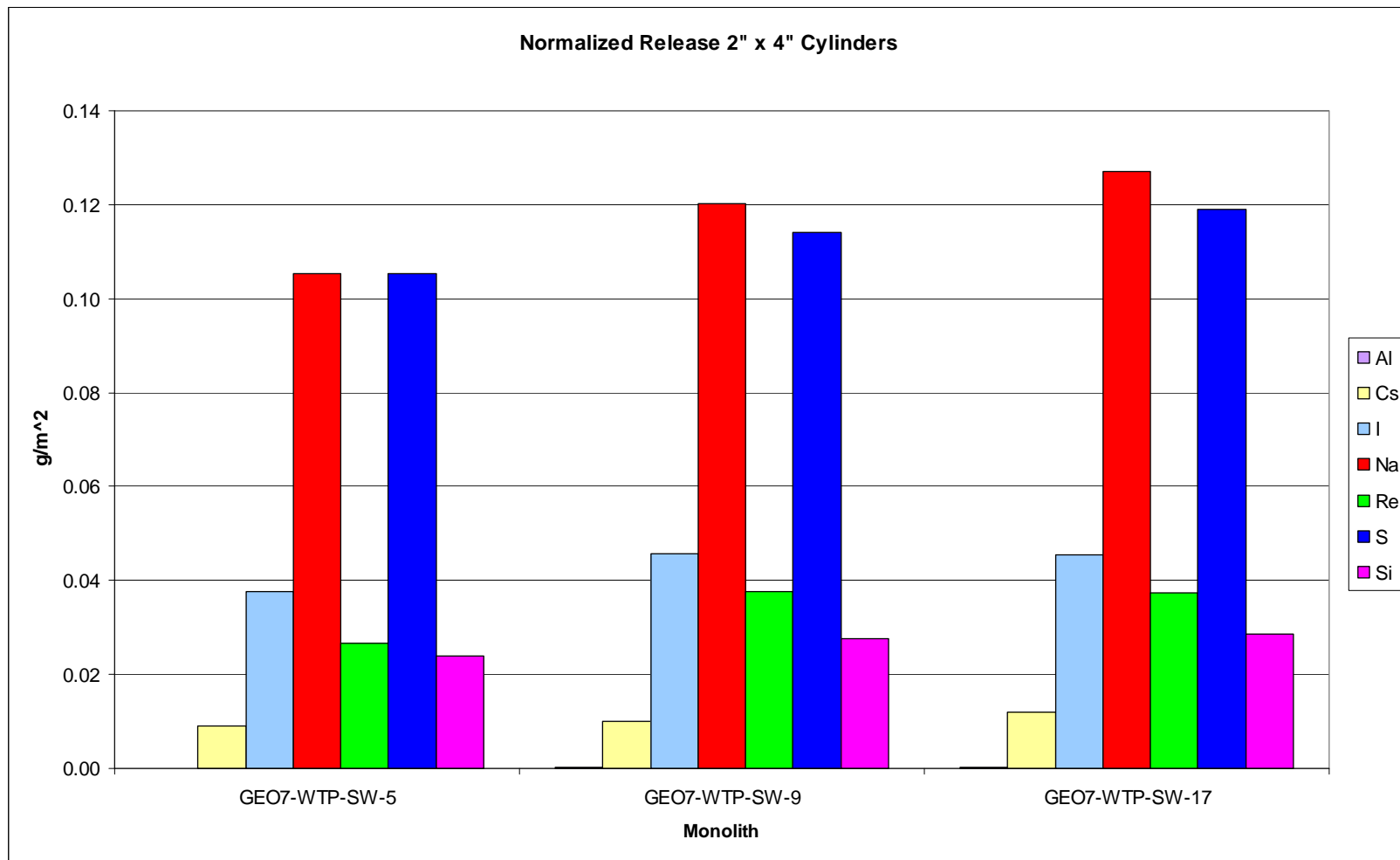
Results of the 7-day PCT performed on the 2" x 4" cylinders are shown in Table 48. All leachate values are shown in mg/L units for Al, Cs, I, Na, Re, S and Si along with the resulting final leachate pHs. The surface area of these triplicate 2" x 4" PCT-prepared powders were measured to be ~ 10X lower than previous BET SA measurements on the 3" x 6" GEO-7 WTP-SW cylinder. Particle size distributions for these powders are presented in Appendix 13 and they show that the powders prepared for PCT for these 2" x 4" crushed monoliths have peak diameters in the range of 93 to 117 microns, with smaller particle 'tails' that range down to below 10 microns. All SA/V and normalized release data were calculated similarly to the 2" cube data by mathematically removing the moisture and the coal from the measured elemental composition data. The adjusted elemental compositions for the 2" x 4" cylinders (anhydrous, coal-free basis) were used to normalize the PCT release for better comparison to the previous aggregate (coal removed by LOI) and blend (coal removed by calculation) PCT data. Normalized PCT release data for the 2" x 4" cylinders are shown in Figure 14. The overall normalized release for the 2" x 4" cylinders shows that  $NL_{Na}$  and  $NL_S$  are highest at less than  $0.13 \text{ g/m}^2$ , with all other  $NL(i)$  values below  $0.05 \text{ g/m}^2$ . Comparison of Figure 14 data to previous GEO-7 WTP-SW PCT data for either the 3" x 6" monoliths (Figure 8) or the 6" x 12" monoliths (Figure 13) shows that the normalized release values are higher (up to  $0.13 \text{ m}^2/\text{g}$ ) for these 2" x 4" crushed monoliths. The 10X lower measured BET SAs for these 2" x 4" cylinder monoliths are responsible for the calculated higher normalized release values via Equation 2. As has been previously mentioned in this report, the GEO-7 geopolymer formulation was developed out of the LAW initial studies and was not optimized for the WTP-SW aggregate blend. One concern for the WTP-SW blend and subsequent monolith durability is the relatively higher F and Cl present versus the LAW. Release of these weak base anion constituents into solution during leaching could produce strong conjugate acid species (HF and HCl) that could attack the leached monolith surface. Measurements of fluoride PCT release were not included in this study, however, more recent testing to be reported in the Module A technical report from SRNL in 2011 has shown significant F release on monolith ASTM 1308 testing.<sup>25</sup> Such data suggests that indeed the monolith formulations could possibly be better optimized for the WTP-SW aggregate blends as discussed in the recent SRNL Monolith document.<sup>29</sup>

All of the normalized PCT data for the monoliths brought forward from the original 2" cube testing (the S71-2, FON-2, GEO-1 and GEO-7 for 2" cubes, 3'x6" cylinders, 6"x12" cylinders, and 2" x 4" cylinders) were averaged. Figure 15 shows a plot of the average normalized release along with successive plots of average + one standard deviation, and average + two standard deviations. These data indicate that the overall normalized release for the monoliths trends with  $(NL_{Na}, NL_{Re}, NL_S) > (NL_I) > (NL_{Al}, NL_{Cs}, NL_{Si})$ . Appendix 14 shows all of the PCT data for this project collected for the blanks and the reference glasses. The data shown in Appendix 14 suggest that all PCTs performed in this project were properly controlled, i.e., time, temperature, duration and leachate preparations for analyses, due to good agreement with past data for both the ARM control glass and the LRM reference glass.

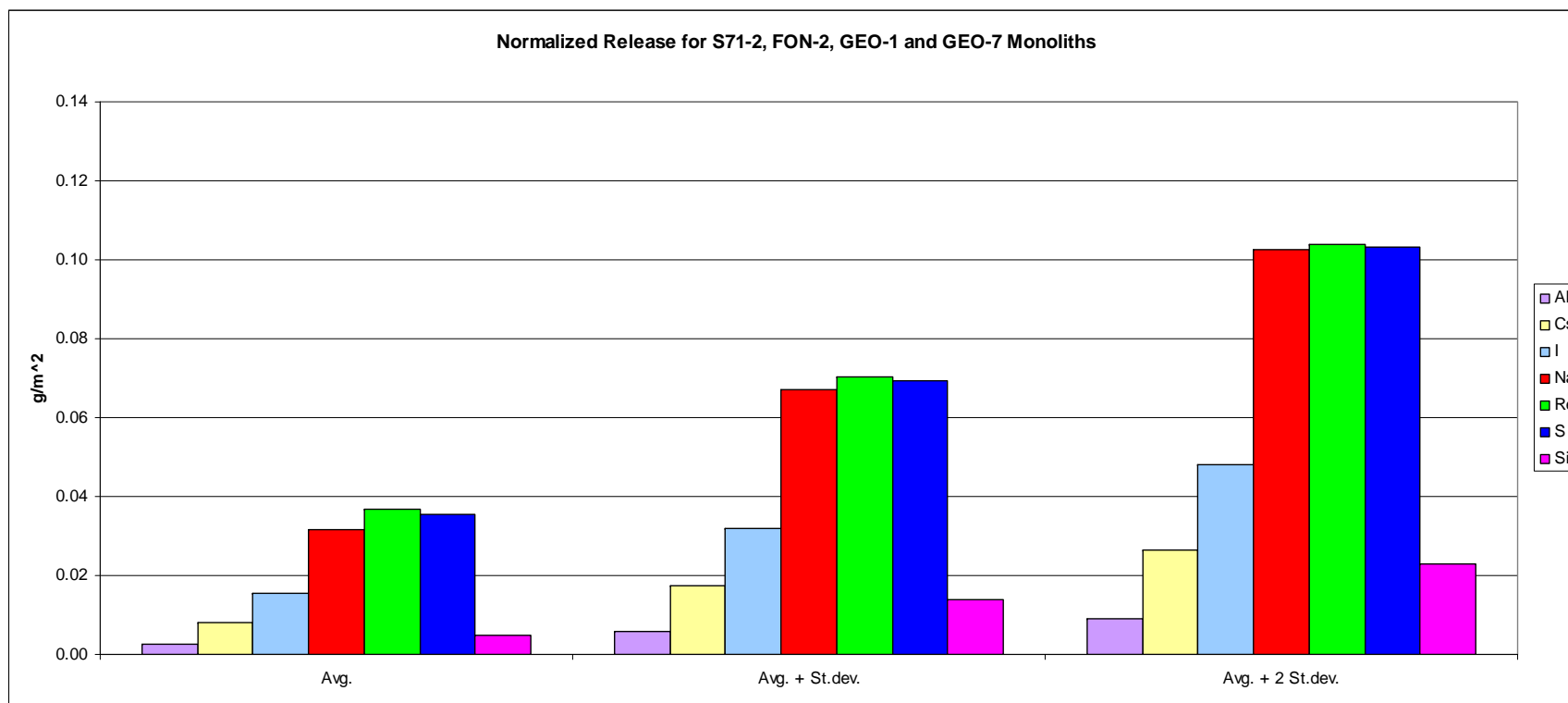
**Table 48. PCT Results for the 2" x 4" Cylinders**

	As Measured Leachate Concentrations							pH	BET (m <sup>2</sup> /g) Non-roasted	SA/V (m <sup>-1</sup> )
	Al (mg/L)	Cs (mg/L)	I (mg/L)	Na (mg/L)	Re (mg/L)	S (mg/L)	Si (mg/L)			
GEO7-WTP-SW-5	4.6	7.6	1.0	4966.7	1.9	61.8	1604.2	12.17	3.23	323,000
GEO7-WTP-SW-9	4.7	8.0	1.2	5050.0	2.2	62.2	1666.7	12.21	2.89	289,000
GEO7-WTP-SW-17	4.8	8.3	1.1	5100.0	2.3	61.7	1642.5	12.16	2.79	279,000

	Normalized Releases Calculated from Equations 1 & 2						
	NL <sub>Al</sub> (g/m <sup>2</sup> )	NL <sub>Cs</sub> (g/m <sup>2</sup> )	NL <sub>I</sub> (g/m <sup>2</sup> )	NL <sub>Na</sub> (g/m <sup>2</sup> )	NL <sub>Re</sub> (g/m <sup>2</sup> )	NL <sub>S</sub> (g/m <sup>2</sup> )	NL <sub>Si</sub> (g/m <sup>2</sup> )
GEO7-WTP-SW-5	1.2E-04	9.0E-03	3.8E-02	1.1E-01	2.7E-02	1.1E-01	2.4E-02
GEO7-WTP-SW-9	1.3E-04	9.9E-03	4.6E-02	1.2E-01	3.8E-02	1.1E-01	2.7E-02
GEO7-WTP-SW-17	1.4E-04	1.2E-02	4.5E-02	1.3E-01	3.7E-02	1.2E-01	2.9E-02



**Figure 14. Normalized Release PCT Data for 2" x 4" Cylinders**



**Figure 15. Normalized Release PCT Data (Average and Standard Deviations) for S71-2, FON-2, GEO-1 and GEO-7 Monoliths Made with Both LAW and WTP-SW with BET Surface Area Normalization**

## 5.0 CONCLUSIONS

The FBSR process was performed on simulated Hanford WTP LAW and WTP-SW in a recent pilot-scale demonstration at the ESTD that produced representative DMR(PR) and HTF samples. These mineralized bed and fines samples were roasted to remove carbon and analyzed and leach tested in this task. The mineralized aggregate blends were also analyzed and leach tested. These first two sample sets (bed products/fines and blended aggregates) used samples from both LAW and WTP-SW. Blended aggregate LAW P-1B was monolithed using various binders and cements at the 2" cube scale. The monoliths were then compression tested, leach tested using ASTM C-1285-08 and the EPA TCLP procedure. Further monolith work involved the optimal monoliths from the LAW P-1B applied to both LAW P-1B and WTP-SW at the 3"x6" and 6"x12" cylinder scale. Finally, the GEO-7 geopolymer recipe was applied to 24 samples of the WTP-SW at the 2" x 4" scale. The following conclusions can be drawn from testing on the LAW P-1B and WTP-SW samples:

- Elemental compositional analysis of the PR and HTF samples indicates they contain primarily Al, Si and Na, with < 1wt% of K and S also present. The PR bed products contain higher Fe in the range of 4-8 wt% for both LAW and WTP-SW. This iron is derived from either the clay additives or the IOC used in the FBSR process, as no iron is present in the simulants. The PR LAW samples contained 0.4 to 2.1 wt% carbon and the PR WTP-SW samples contained 0.8 wt% carbon. The HTF LAW samples contain carbon in the range of 8 - 10 wt% and the HTF WTP-SW samples contain carbon in the range of 12-13 wt%. REDOX measurements for the PR bed products show  $\text{Fe}^{2+}/\text{Fe}_{\text{Total}}$  in the range of 0.4 to 0.6, whereas HTF fines show higher reduced values of 0.7 to 0.92 although this measurement is likely biased high due to the large amount of coal in the HTF fines samples for both LAW and WTP-SW.
- Crystalline phases observed in the bed products/fines and aggregate blends from the Hazen ESTD testing on LAW samples show low-carnegieite and nepheline as the primary phases. Nosean, the  $\text{SO}_4$  containing sodalite phase only appears when a waste is high in sulfate. It may be present at  $\leq 5$  wt% and just not in sufficient concentration to be observed in the XRD patterns.
- Similar analysis for the WTP-SW samples revealed increased concentrations of nosean and a chloride/fluoride/iodide type sodalite structure in addition to the low-carnegieite and nepheline. All of these crystalline phase structures are similar to previous FBSR testing results.
- PCT durability testing of the best candidate monolith forms made with both LAW P-1B and WTP-SW showed average normalized release values below  $0.04 \text{ g/m}^2$ . Statistical analysis of the data indicated that the 95% confidence level ( $\sim 2$  sigma) was less than or equal to  $\sim 0.1 \text{ g/m}^2$ .
- All of the best candidate monoliths with both LAW P-1B and WTP-SW P-2B FBSR dry-basis loadings in the range of 65 to 74 wt% passed the target lower compressive strength limit of 500 psi except for certain of the FON-2 formulations.
- The TCLP results performed on the LAW P-1B FBSR and the WTP-SW FBSR aggregate blends indicated that Sb was above the UTS limit for LAW and both Sb and Cd were above the UTS limit

for WTP-SW. However, as indicated in the text, these metals were added in the ESTD testing at artificially high levels (10 – 48X) in order to help detection in the final products and off-gas.

- The initial monolith 2” cube series made with LAW P-1B also showed some Sb and Cd failures for the geopolymers, ceramicrete and NuCap<sup>TM</sup> monoliths, with the OPC and high Al cements giving TCLP results below the UTS limit for all analytes. These wastes were also shimmed with elevated concentrations of Sb and Cd.
- In the optimized monolith studies conducted on 3”x 6” and 6” x 12” monoliths made with LAW P-1B, all of the FON-2 samples passed TCLP and certain monoliths in the S71-2, GEO-1 and GEO-7 series again showed some failures for Sb and Cd, which were believed to be due to the excessive shimming of the feeds.
- Similar TCLP testing on the 3”x 6” and 6” x 12” monoliths made with WTP-SW P-2B indicated that both the FON-2 and GEO-7 samples passed TCLP whereas the S71-2 and GEO-1 samples again showed some failures for both Sb and Cd with the excessive shimming of the feeds.
- The GEO-7 formulation which was optimized for LAW wastes was used for the WTP-SW P-2B blend (3” x 6”, 6” x 12” and 2” x 4” cylinders) without reformulation. Considering that the WTP-SW wastes had considerable fluoride over what was present in the LAW P-1B, they performed relatively well in durability testing and passed TCLP for all elements.

## 6.0 ACKNOWLEDGEMENTS

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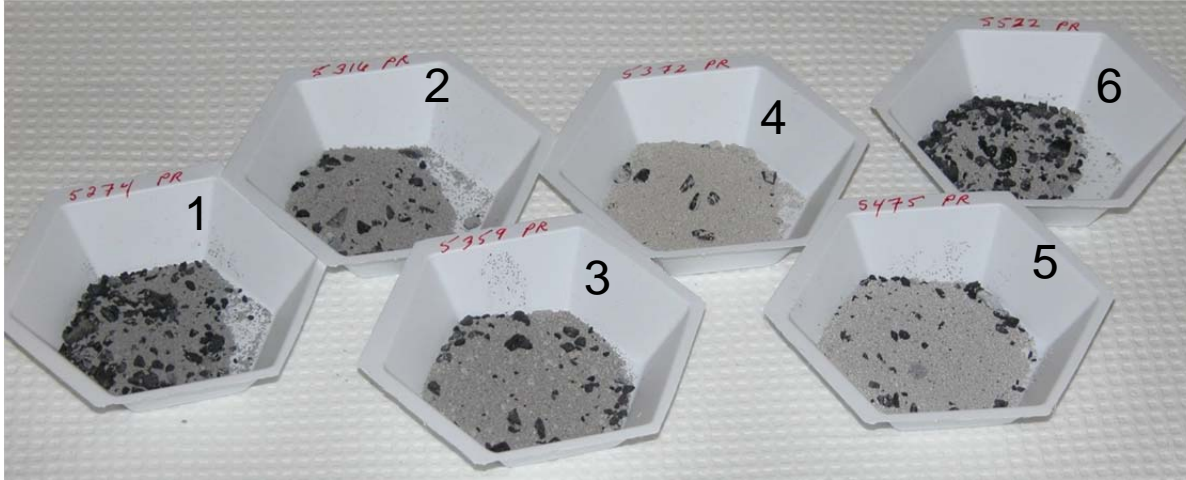
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# APPENDIX 1. AS RECEIVED PR BED PRODUCT AND HTF FINES PHOTOGRAPHS



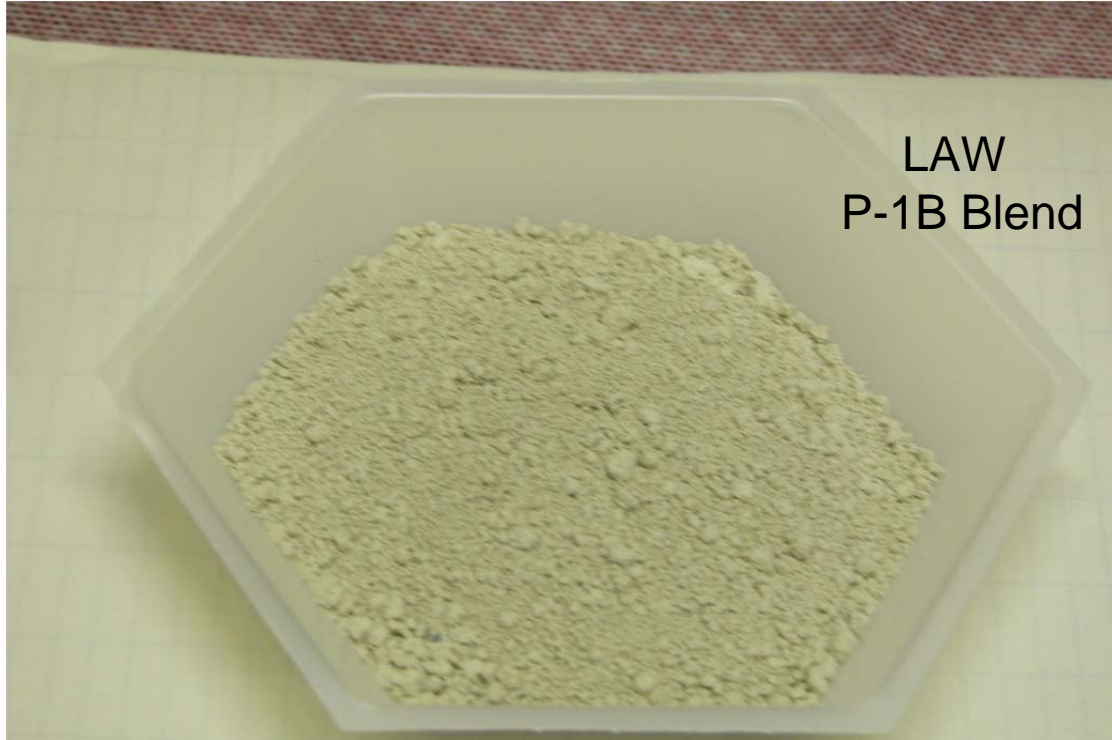
(1) PR	5274	4/28/2008 5:15	P-1A
(2) PR	5316	4/29/2008 3:54	P-1A
(3) PR	5359	4/30/2008 22:55	P-1B
(4) PR	5372	5/1/2008 7:00	P-1B
(5) PR	5475	5/5/2008 4:00	P-2A
(6) PR	5522	5/6/2008 10:00	P-2B



(1) HTF	5280	4/27/2008 21:42	P-1A
(2) HTF	5297	4/28/2008 17:28	P-1A
(3) HTF	5351	4/30/2008 12:00	P-1B
(4) HTF latter sample	5357	4/30/2008 19:44	P-1B
(5) HTF initial sample	5357	4/30/2008 19:44	P-1B
(6) HTF	5471	5/5/2008 0:20	P-2A
(7) HTF	5520	5/6/2008 10:00	P-2B

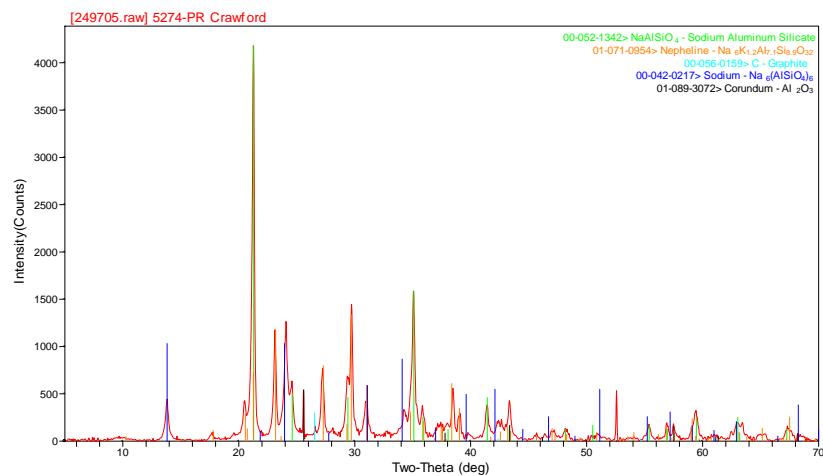
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**APPENDIX 2. LAW P-1B AND WTP-SW P-2B AGGREGATE BLEND  
PHOTOGRAPHS**

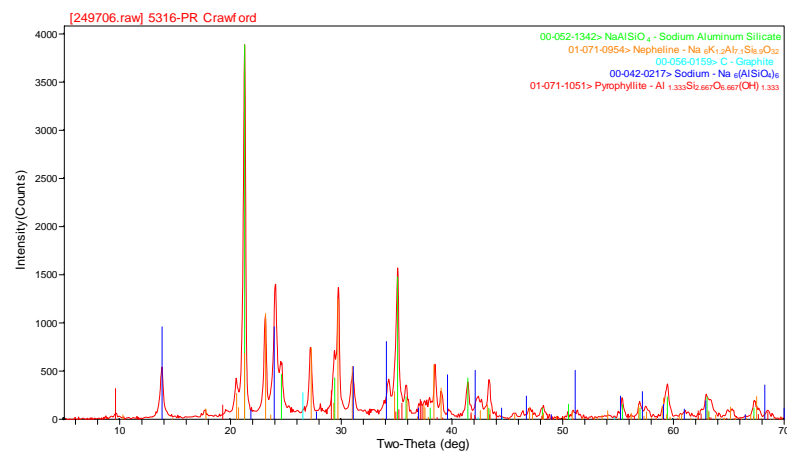


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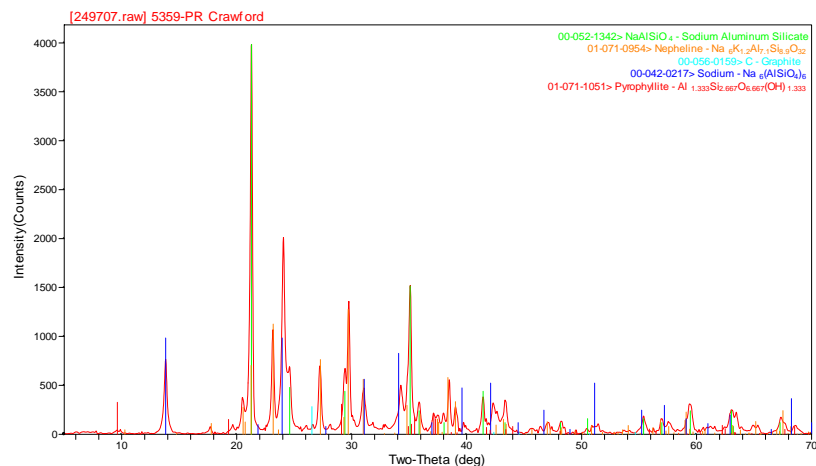
## APPENDIX 3. XRD SPECTRA OF PR BED PRODUCTS AND HTF FINES



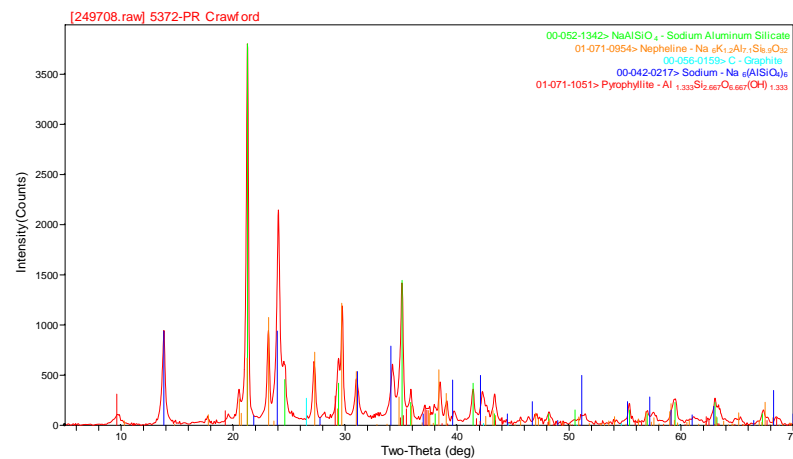
**XRD Spectra of Bed Product 5274 PR**



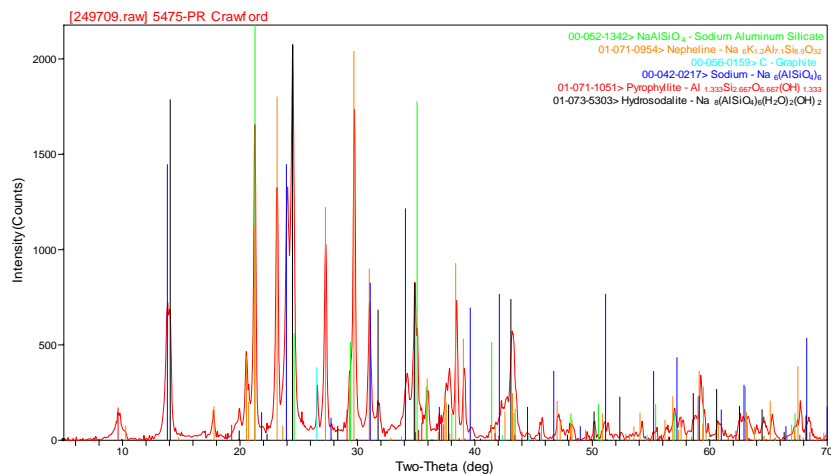
**XRD Spectra of Bed Product 5316 PR**



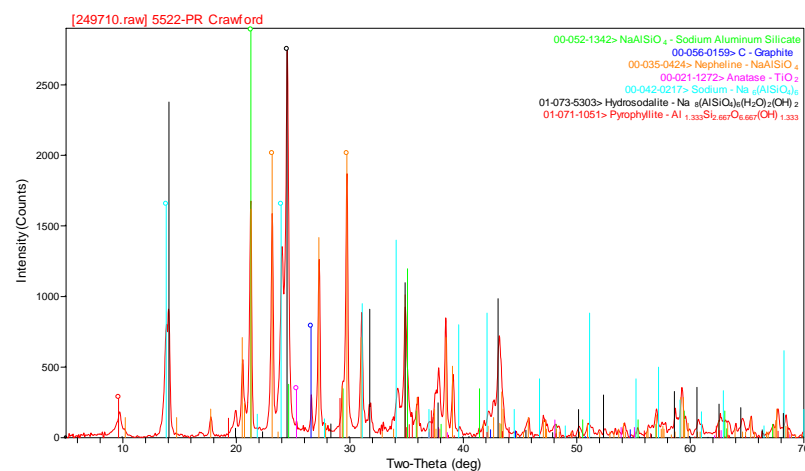
**XRD Spectra of Bed Product 5359 PR**



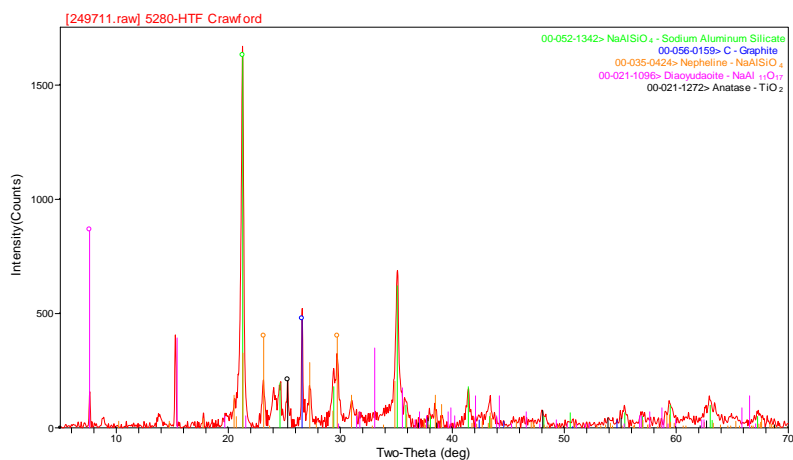
**XRD Spectra of Bed Product 5372 PR**



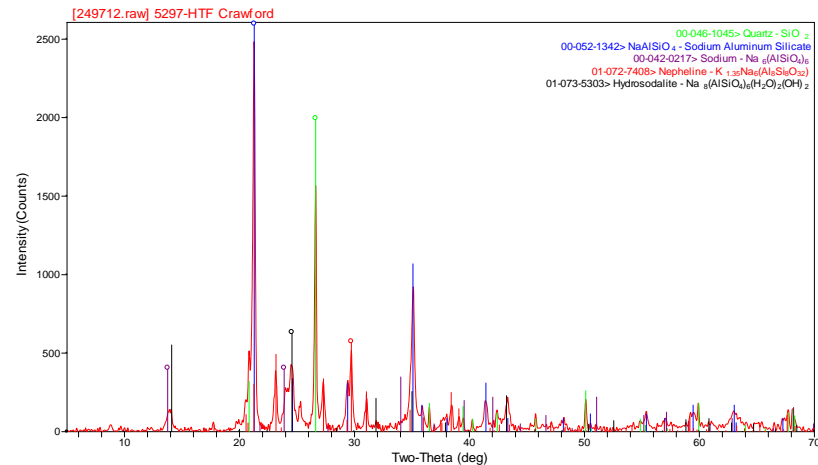
XRD Spectra of Bed Product 5475 PR



XRD Spectra of Bed Product 5522 PR

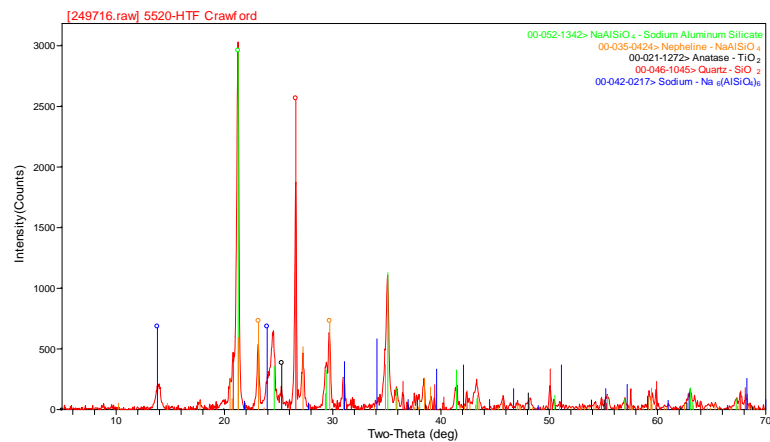


XRD Spectra of Fines 5280 HTF

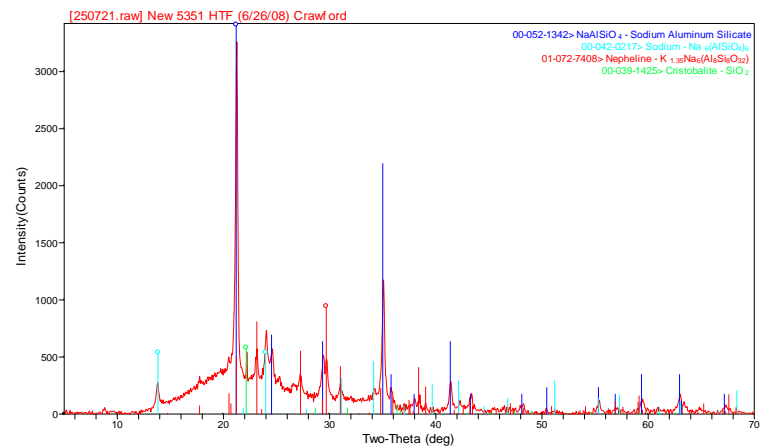


XRD Spectra of Fines 5297 HTF

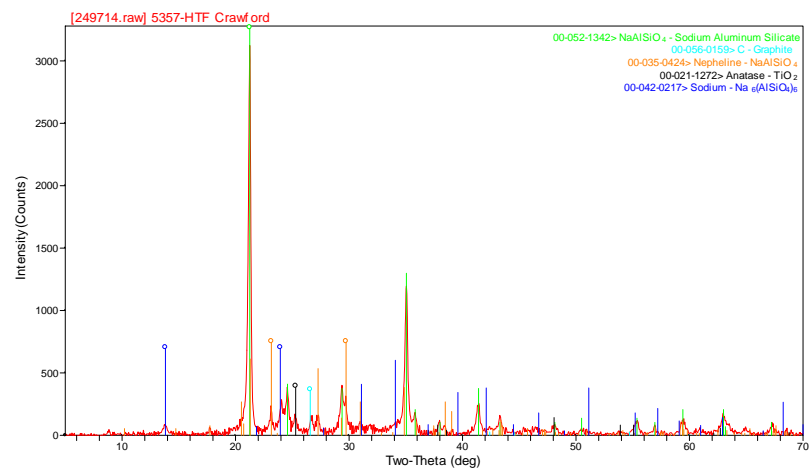




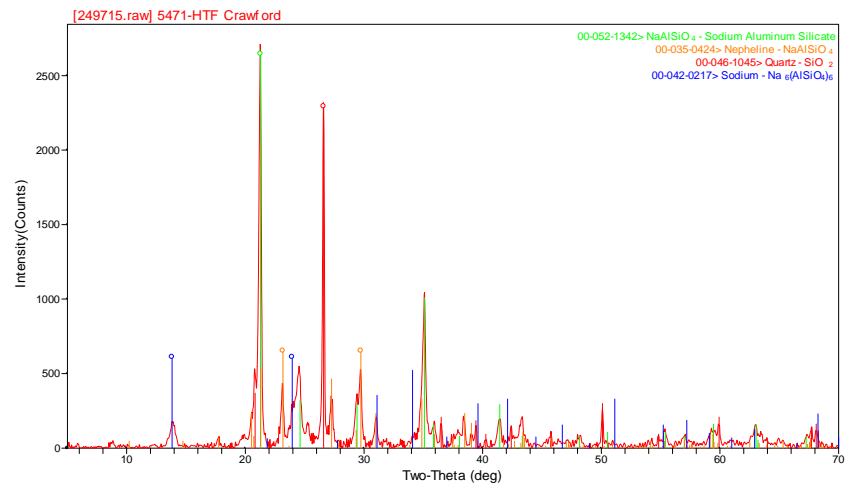
**XRD Spectra of Fines 5520 HTF**



**XRD Spectra of Fines 5351 HTF**



**XRD Spectra of Fines 5357 HTF**



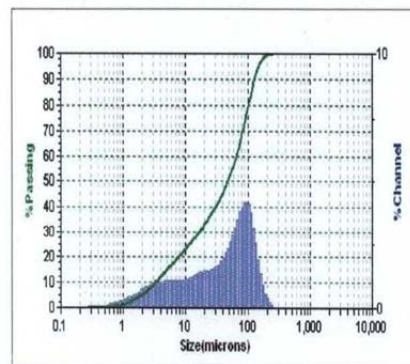
**XRD Spectra of Fines 5471 HTF**

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# APPENDIX 4. MICROTRAC PARTICLE SIZE DISTRIBUTION OF PR BED PRODUCTS AND HTF FINES PREPARED FOR PCT

250182  
5280 HTF Washed

Data Acquired: 06/19/2008 - 08:24  
Calculated: 06/19/2008 - 08:24



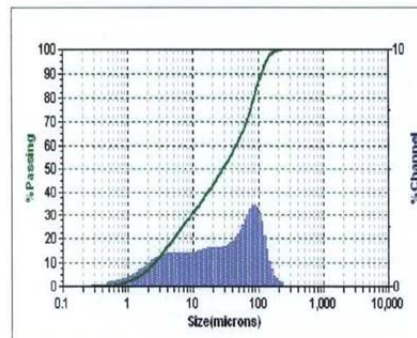
## Peak Summary

Dia	Vol%	Width
47.88	100.0	106.10

PSD for Fines 5280 HTF

250183 Crawford  
5297 HTF Washed

Data Acquired: 06/19/2008 - 08:38  
Calculated: 06/19/2008 - 08:38



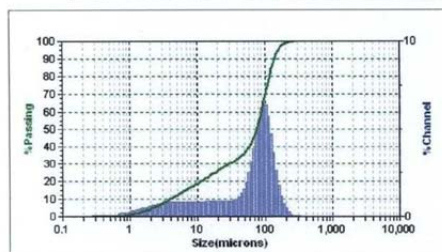
## Peak Summary

Dia	Vol%	Width
28.16	100.0	91.72

PSD for Fines 5297 HTF

250184 Crawford  
5351 HTF Washed

Data Acquired: 06/19/2008 - 08:48  
Calculated: 06/19/2008 - 08:48



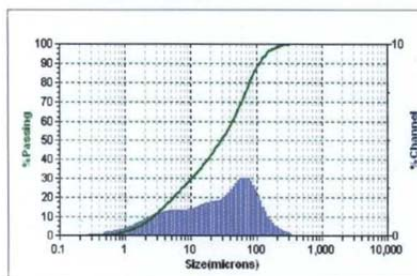
## Peak Summary

Dia	Vol%	Width
75.32	100.0	116.80

PSD for Fines 5351 HTF

250185 Crawford  
5357 HTF Washed

Data Acquired: 06/19/2008 - 09:34  
Calculated: 06/19/2008 - 09:34



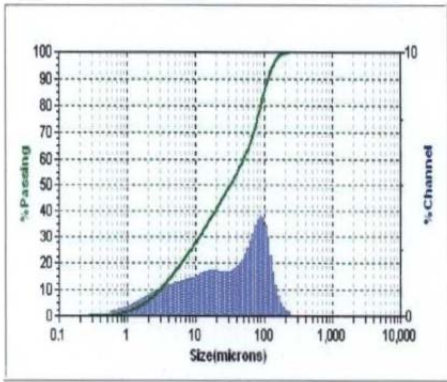
## Peak Summary

Dia	Vol%	Width
28.71	100.0	84.98

PSD for Fines 5357 HTF

250186 Crawford  
5471 HTF Washed

Data Acquired: 06/19/2008 - 09:40  
Calculated: 06/19/2008 - 09:40



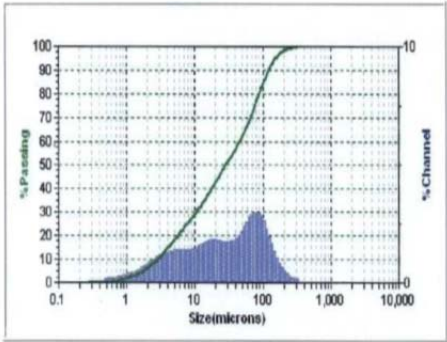
Peak Summary

Dia	Vol%	Width
31.65	100.0	94.21

PSD for Fines 5471 HTF

250187 Crawford  
5520 HTF Washed

Data Acquired: 06/19/2008 - 09:50  
Calculated: 06/19/2008 - 09:50



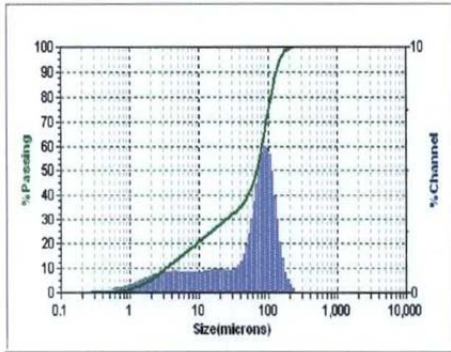
Peak Summary

Dia	Vol%	Width
28.33	100.0	94.90

PSD for Fines 5520 HTF

250189 Crawford  
5316 PR Washed

Data Acquired: 06/19/2008 - 10:12  
Calculated: 06/19/2008 - 10:12



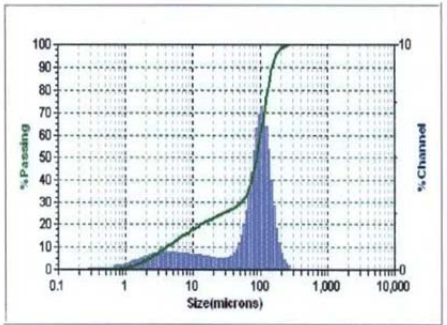
Peak Summary

Dia	Vol%	Width
68.09	100.0	110.50

PSD for Bed 5316 PR

250188 Crawford  
5274 PR Washed

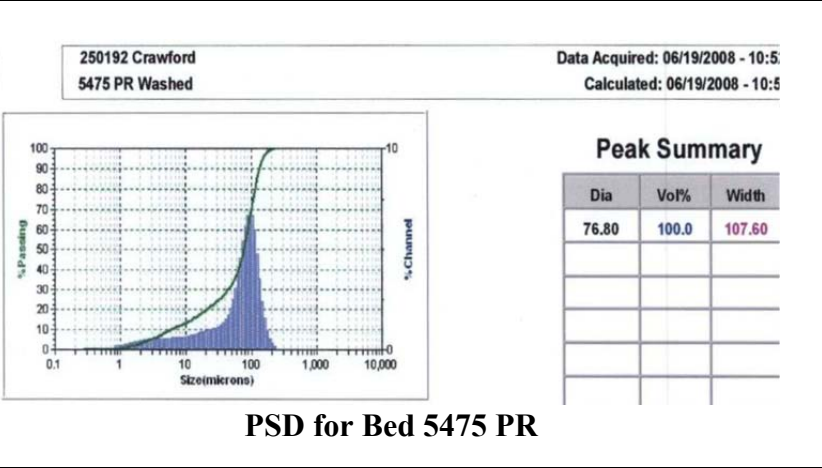
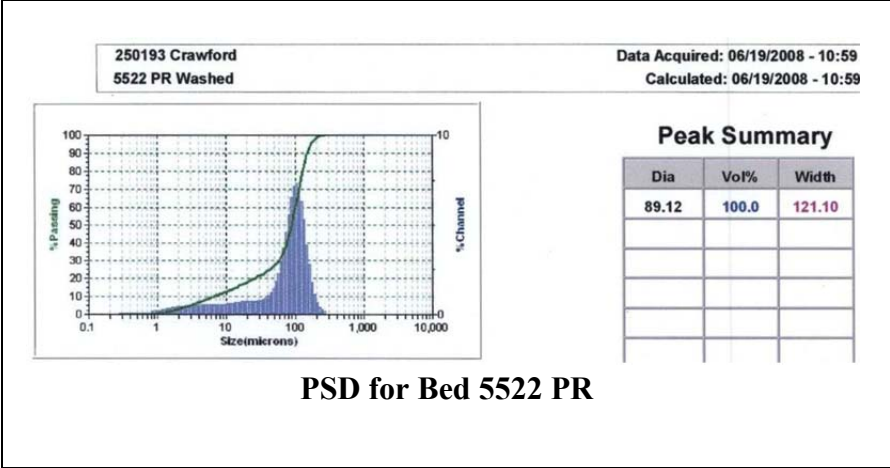
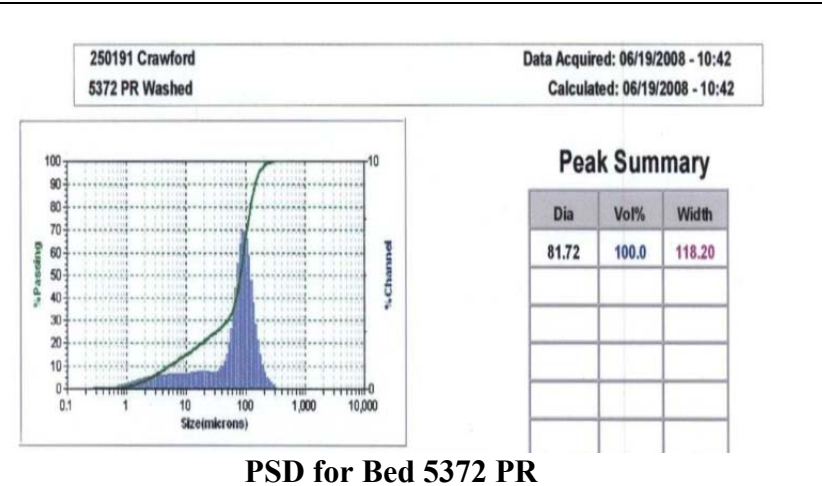
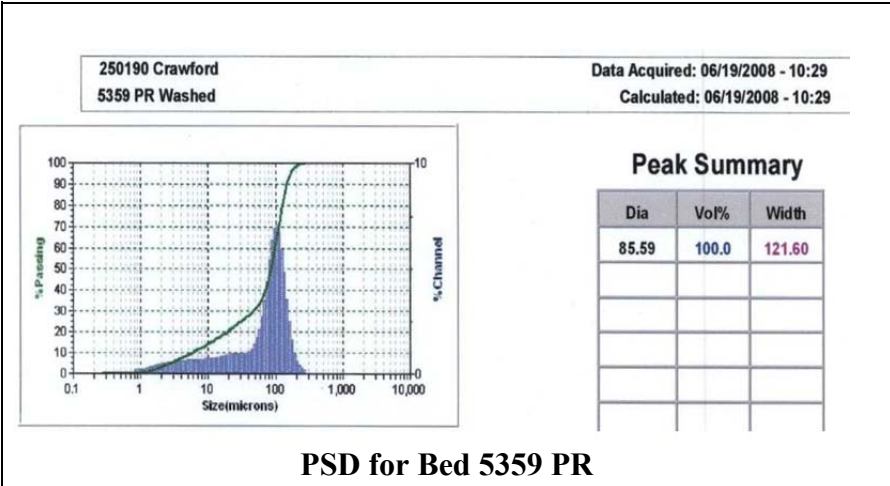
Data Acquired: 06/19/2008 - 10:00  
Calculated: 06/19/2008 - 10:00



Peak Summary

Dia	Vol%	Width
97.38	86.5	101.50
2.783	13.5	3.31

PSD for Bed 5274 PR



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## APPENDIX 5. DEVELOPMENTAL GEOPOLYMER FORMULATIONS WITH TROY CLAY

Polybottle	P1B (1 of 5)	7/31/2008	Troy, 40%clay/FBSR	82.5	7.54	74.43	0.98	0.49	-	150	6
						32.82	0.38		66.9		
Polybottle	P1B (1 of 5)	7/31/2008	Troy, 40%clay/FBSR	82.5	3.77	67.51	0.89	0.45	-	No set	NA
						29.77	0.34		70.9		
Polybottle	P1B (1 of 5)	7/31/2008	Troy, 40%clay/FBSR	55	7.51	38.85	4.6	3.74	-	2680	6
						17.13	1.78		67.5		
Polybottle	P1B (1 of 5)	7/31/2008	Troy, 40%clay/FBSR	112.5	7.51	69.2	8.18	6.66	-	2110	6
						30.52	3.17		73.2		
Polybottle	P1B (1 of 5)	8/5/2008	Troy, 20%clay/FBSR	105.14	15.11	66.29	0.86	21.77	-	90	7
						29.23	0.33		70.2		
Polybottle	P1B (1 of 5)	8/5/2008	Troy, 20%clay/FBSR	165.17	15.01	88.52	1.15	37.03	-	70	7
						39.04	0.45		75.2		
Polybottle	P1B (1 of 5)	8/5/2008	Troy, 20%clay/FBSR	77.56	15.05	36.57	4.36	22.34	-	230	7
						16.13	1.69		70.2		
Polybottle	P1B (1 of 5)	8/5/2008	Troy, 20%clay/FBSR	112.52	15.07	44.97	5.38	34.93	-	90	7
						19.83	2.08		75.3		
Polybottle	P1B (1 of 5)	8/6/2008	Troy, 30%clay/FBSR	105.14	15.11	85.52	1.14	8.93	-	390	7
						37.71	0.44		66.4		
Polybottle	P1B (1 of 5)	8/6/2008	Troy, 30%clay/FBSR	165.17	15.01	119	1.58	18.88	-	120	7
						52.48	0.61		70.8		
Polybottle	P1B (1 of 5)	8/6/2008	Troy, 30%clay/FBSR	77.56	15.05	45.9	5.41	14.67	-	530	7
						20.24	2.10		67.5		
Polybottle	P1B (1 of 5)	8/6/2008	Troy, 30%clay/FBSR	112.52	15.07	58.44	6.97	23.53	-	300	7
						25.77	2.70		72.1		
Polybottle	P1B (1 of 5)	8/6/2008	Troy, 40%clay/FBSR	105.14	15.09	104.79	1.4	1.4	-	230	7
						46.21	0.54		63.0		
Polybottle	P1B (1 of 5)	8/6/2008	Troy, 40%clay/FBSR	165.17	15.06	149.43	1.98	1.97	-	120	7
						65.90	0.77		66.9		
Polybottle	P1B (1 of 5)	8/6/2008	Troy, 40%clay/FBSR	78.49	15.08	55.15	6.53	10.63	-	690	7
						24.32	2.53		65.2		
Polybottle	P1B (1 of 5)	8/6/2008	Troy, 40%clay/FBSR	112.57	15.13	71.97	8.5	14.88	-	700	7
						31.74	3.29		69.2		

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## APPENDIX 6. NUCAP™ FORMULATION

<b>2x2 Cubes</b>					
1 Part Binder: 1 Part FBSR by weight					
<b>Additive</b>	<b>Mass(g)</b>				
FBSR (LAW, P-1A)	165				
Binder Paste	168.3				
Catalyst	10.09	(6% of Binder mass)			
Q1-3563 Thinner	21.37	(12% of Binder + Catalyst mass)			
Boric Acid paste	10.93	(3% of FBSR/Binder/Catalyst/Thinner mass)			
<b>2x2 Cubes</b>					
1.2 Part Binder: 1 Part FBSR by weight					
<b>Additive</b>	<b>Mass(g)</b>				
FBSR (LAW, P-1A)	165				
Binder Paste	202.6				
Catalyst	12.16	(6% of Binder mass)			
Q1-3563 Thinner	25.77	(12% of Binder + Catalyst mass)			
Boric Acid paste	12.16	(3% of FBSR/Binder/Catalyst/Thinner mass)			
<b>2x2 Cubes</b>					
1 Part Binder: 1 Part FBSR by weight					
<b>Additive</b>	<b>Mass(g)</b>				
FBSR (LAW, P-1B)	165				
Binder Paste	165.7				
Catalyst	9.42	(6% of Binder mass)			
Q1-3563 Thinner	21.07	(12% of Binder + Catalyst mass)			
Boric Acid paste	10.85	(3% of FBSR/Binder/Catalyst/Thinner mass)			
<b>2x2 Cubes</b>					
1.2 Part Binder: 1 Part FBSR by weight					
<b>Additive</b>	<b>Mass(g)</b>				
FBSR (LAW, P-1B)	165				
Binder Paste	203.6				
Catalyst	12.21	(6% of Binder mass)			
Q1-3563 Thinner	25.9	(12% of Binder + Catalyst mass)			
Boric Acid paste	12.2	(3% of FBSR/Binder/Catalyst/Thinner mass)			
<b>3 x 6 Cylinders</b>					
1 Part Binder: 1 Part FBSR by weight					
<b>Additive</b>	<b>Mass(g)</b>				
FBSR (LAW, P-1B)	500				
Binder Paste	500				
Catalyst	30	(6% of Binder mass)			
Q1-3563 Thinner	63.6	(12% of Binder + Catalyst mass)			
Boric Acid paste	32.81	(3% of FBSR/Binder/Catalyst/Thinner mass)			
<b>3 x 6 Cylinders</b>					
1.2 Part Binder: 1 Part FBSR by weight					
<b>Additive</b>	<b>Mass(g)</b>				
FBSR (LAW, P-1B)	500				
Binder Paste	600				
Catalyst	36	(6% of Binder mass)			
Q1-3563 Thinner	76.32	(12% of Binder + Catalyst mass)			
Boric Acid paste	36.36	(3% of FBSR/Binder/Catalyst/Thinner mass)			
<b>6 x 12 Cylinders</b>					
1 Part Binder: 1 Part FBSR by weight					
<b>Additive</b>	<b>Mass(g)</b>				
FBSR (LAW, P-1B)	4000				
Binder Paste	4000				
Catalyst	240	(6% of Binder mass)			
Q1-3563 Thinner	508.8	(12% of Binder + Catalyst mass)			
Boric Acid paste	262.46	(3% of FBSR/Binder/Catalyst/Thinner mass)			
<b>6 x 12 Cylinders</b>					
1.2 Part Binder: 1 Part FBSR by weight					
<b>Additive</b>	<b>Mass(g)</b>				
FBSR (LAW, P-1B)	4000				
Binder Paste	4800				
Catalyst	288	(6% of Binder mass)			
Q1-3563 Thinner	610.56	(12% of Binder + Catalyst mass)			
Boric Acid paste	290.95	(3% of FBSR/Binder/Catalyst/Thinner mass)			

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**APPENDIX 7. CHEMICAL COMPOSITIONS OF CEMENT BINDERS AND FLY ASH**

Sample ID	Lab ID									
	(wt%)	Al	Ca	Fe	K	Mg	Na	S	Si	Ti
OPC	08-1661	2.45	47.60	2.44	0.29	0.50	<0.10	0.92	9.98	0.15
FONDU	08-1663	20.38	27.45	9.89	0.08	0.44	<0.10	<0.10	1.90	0.90
SECAR 41	08-1665	25.15	28.50	5.14	0.18	0.24	<0.10	<0.10	2.20	1.05
SECAR 71	08-1667	37.05	22.25	0.06	<0.10	0.17	<0.10	<0.10	<0.10	<0.10

Sample ID	Lab ID													
	(wt%)	Al	Ca	Cr	Fe	K	Mg	Na	P	S	Si	Sr	Ti	Zr
Fly Ash	08-1659	14.85	0.93	0.02	5.26	2.76	0.56	0.34	0.12	<0.100	25.20	0.11	0.72	0.02

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**APPENDIX 8. BARDEN CLAY HEAT TREATMENT**

KT Barden Calcining						
Sample ID	Empty Crucible Weight	Amount of KT Barden	Crucible weight and sample (before calcining)	Weight of crucible and contents (after calcining)	Amount of KT Barden after calcine	Percent Mass Loss
KT Barden #1	182.296	107.950	290.248	276.032	93.736	13.17
KT Barden #2	196.266	114.387	310.655	296.007	99.741	12.80
KT Barden #3	202.137	111.367	313.506	298.764	96.627	13.24
KT Barden #4	202.859	104.596	307.455	294.030	91.171	12.84

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## APPENDIX 9. TCLP DATA FOR 2" CUBE LAW MONOLITHS

### TCLP Data for 2" Cube LAW Monoliths (Fon, S41, S71, OPC)

Element	FON-1 (mg/L)	FON-2 (mg/L)	S41-1 (mg/L)	S41-2 (mg/L)	S71-1 (mg/L)	S71-2 (mg/L)	OPC-1 (mg/L)	OPC-2 (mg/L)	UTS *	P-1B LAW Target (g/L) (w 640 g clay/L)
Sb	0.182	0.271	0.118 <sup>UV</sup>	0.185	0.305	0.154	0.75	0.827	1.15	0.426
As	0.075 <sup>J</sup>	0.0144 <sup>U</sup>	< 0.05 <sup>U</sup>	< 0.05 <sup>U</sup>	< 0.05 <sup>U</sup>	< 0.05 <sup>U</sup>	0.196	0.0742 <sup>J</sup>	5	0.949
Ba	0.0202 <sup>J</sup>	0.492	0.053	0.11	0.447	0.145	0.00498 <sup>U</sup>	0.0258 <sup>J</sup>	21	1.37
Cd	0.00284 <sup>U</sup>	< 0.01 <sup>U</sup>	< 0.01 <sup>U</sup>	< 0.01 <sup>U</sup>	< 0.01 <sup>U</sup>	0.000079 <sup>U</sup>	< 0.01 <sup>U</sup>	< 0.01 <sup>U</sup>	0.11	0.381
Cr	0.159	0.123	0.0284 <sup>J</sup>	0.0567	0.00728 <sup>U</sup>	0.0224 <sup>J</sup>	0.565	0.264	0.6	0.436
Pb	< 0.025 <sup>U</sup>	0.023 <sup>U</sup>	< 0.025 <sup>U</sup>	0.0135 <sup>U</sup>	< 0.025 <sup>U</sup>	0.00109 <sup>U</sup>	0.0185	0.0162 <sup>U</sup>	0.75	1.013
Se	0.024 <sup>U</sup>	0.166	0.0899 <sup>J</sup>	0.129 <sup>J</sup>	< 0.05 <sup>U</sup>	0.112 <sup>J</sup>	0.332	0.123 <sup>J</sup>	5.7	0.078
Ag	< 0.01 <sup>U</sup>	< 0.01 <sup>U</sup>	< 0.01 <sup>U</sup>	< 0.01 <sup>U</sup>	< 0.01 <sup>U</sup>	< 0.01 <sup>U</sup>	< 0.01 <sup>U</sup>	< 0.01 <sup>U</sup>	0.14	0.14
Ni	0.0078 <sup>U</sup>	0.00437 <sup>U</sup>	0.000382 <sup>U</sup>	0.00642 <sup>U</sup>	< 0.01 <sup>U</sup>	0.00443 <sup>U</sup>	0.00671 <sup>U</sup>	< 0.01 <sup>U</sup>	11	0.502
Tl	0.0782 <sup>J</sup>	< 0.05 <sup>U</sup>	0.0163 <sup>U</sup>	0.0112 <sup>U</sup>	< 0.05 <sup>U</sup>	0.000441 <sup>U</sup>	0.0373 <sup>U</sup>	< 0.05 <sup>U</sup>	0.2	0.333
Zn	0.00811 <sup>U</sup>	0.0341 <sup>UJV</sup>	0.0669 <sup>UJV</sup>	0.0357 <sup>UJV</sup>	0.0754 <sup>UJV</sup>	0.0251 <sup>J</sup>	0.00482 <sup>U</sup>	0.0645 <sup>UJV</sup>	4.3	0

\*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

UV = Positive blank result and sample result > MDL

UJV = Positive blank result and sample result below RL but above MDL

**TCLP Data for 2" Cube LAW Monoliths (GEO, CER, NuCap)**

Element	GEO-1 (mg/L)	GEO-2 (mg/L)	GEO-3 (mg/L)	GEO-4 (mg/L)	GEO-5 (mg/L)	GEO-6 (mg/L)	CER-1 (mg/L)	CER-2 (mg/L)	NuCap (mg/L)	UTS *	P-1B LAW Target (g/L) (w 640 g clay/L)
Sb	0.733	0.99	1.43	3.29	2.22	1.38	1.55	1.09	0.171	1.15	0.426
As	0.0891 <sup>J</sup>	0.113 <sup>J</sup>	0.328	2.13	1.71	1.63	1.68	0.513	< 0.05 <sup>U</sup>	5	0.949
Ba	0.137	0.151	0.195	0.251	0.401	0.457	0.0168 <sup>J</sup>	0.508	0.0832	21	1.37
Cd	0.314	0.288	0.141	0.0484 <sup>J</sup>	0.0424 <sup>J</sup>	0.0131 <sup>J</sup>	< 0.01 <sup>U</sup>	0.00312 <sup>U</sup>	0.127	0.11	0.381
Cr	0.03 <sup>J</sup>	0.0395 <sup>J</sup>	0.0535	0.162	0.0559	0.031 <sup>J</sup>	0.0246 <sup>J</sup>	0.0555	0.0134 <sup>U</sup>	0.6	0.436
Pb	0.0927 <sup>J</sup>	0.146	0.135	0.0861 <sup>J</sup>	0.109	0.0603 <sup>J</sup>	0.030 <sup>J</sup>	0.0537 <sup>J</sup>	0.0203 <sup>U</sup>	0.75	1.013
Se	0.334	0.395	0.415	0.607	0.475	0.455	0.478	0.538	0.0386 <sup>U</sup>	5.7	0.078
Ag	< 0.01 <sup>U</sup>	< 0.01 <sup>U</sup>	< 0.01 <sup>U</sup>	< 0.01 <sup>U</sup>	< 0.01 <sup>U</sup>	< 0.01 <sup>U</sup>	< 0.01 <sup>U</sup>	< 0.01 <sup>U</sup>	< 0.01 <sup>U</sup>	0.14	0.14
Ni	0.168	0.168	0.104	0.0215 <sup>J</sup>	0.0337 <sup>J</sup>	0.0127 <sup>J</sup>	0.0146 <sup>J</sup>	0.0702	0.0609	11	0.502
Tl	0.0473 <sup>U</sup>	0.0633 <sup>J</sup>	0.108 <sup>J</sup>	0.000881 <sup>U</sup>	0.0181 <sup>U</sup>	0.00251 <sup>U</sup>	< 0.05 <sup>U</sup>	0.0151 <sup>U</sup>	< 0.05 <sup>U</sup>	0.2	0.333
Zn	0.0838 <sup>J</sup>	0.0489 <sup>J</sup>	0.0327 <sup>J</sup>	0.0324 <sup>UV</sup>	0.0291 <sup>UV</sup>	0.0269 <sup>UV</sup>	0.0112 <sup>U</sup>	0.00608 <sup>U</sup>	0.135	4.3	0

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

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

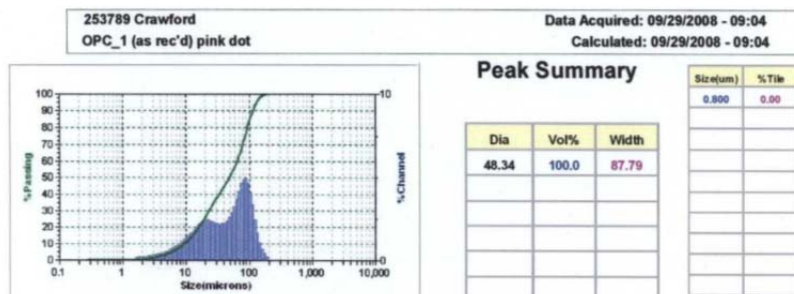
J = results below Reporting Limit but above the MDL, or “J” flag

UV = Positive blank result and sample result > MDL

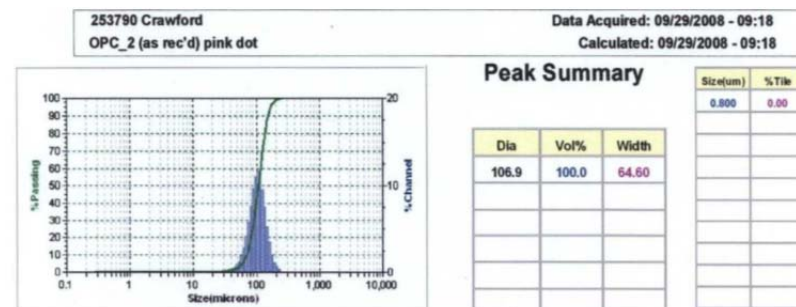
UVJ = Positive blank result and sample result below RL but above MDL



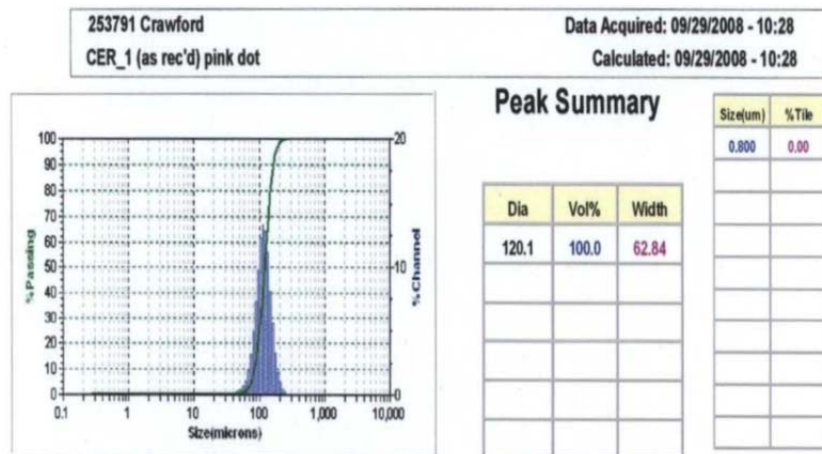
# APPENDIX 10. PARTICLE SIZE DISTRIBUTION FOR 2" CUBE MONOLITHS FOR 100 - 200 MESH POWDERS PREPARED FOR PCT



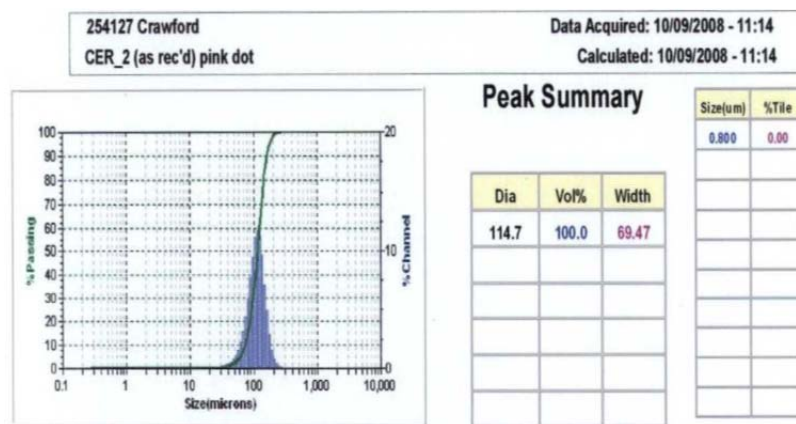
PSD for OPC-1



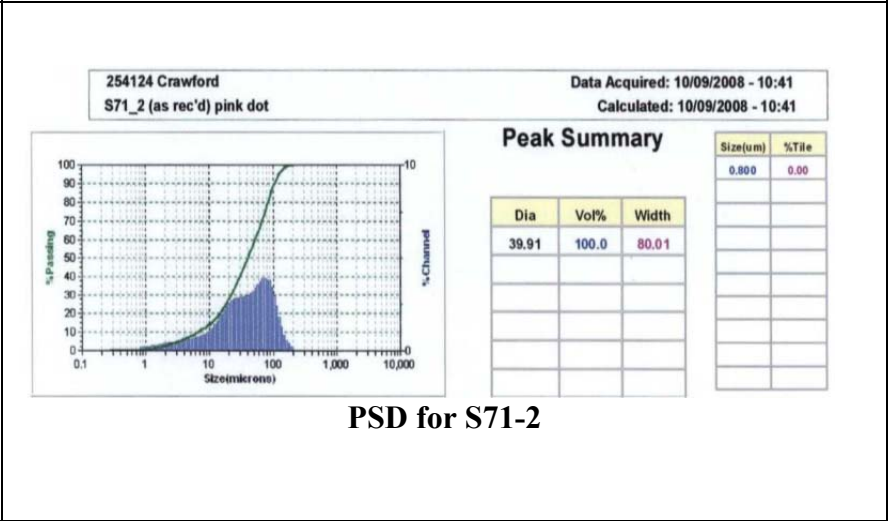
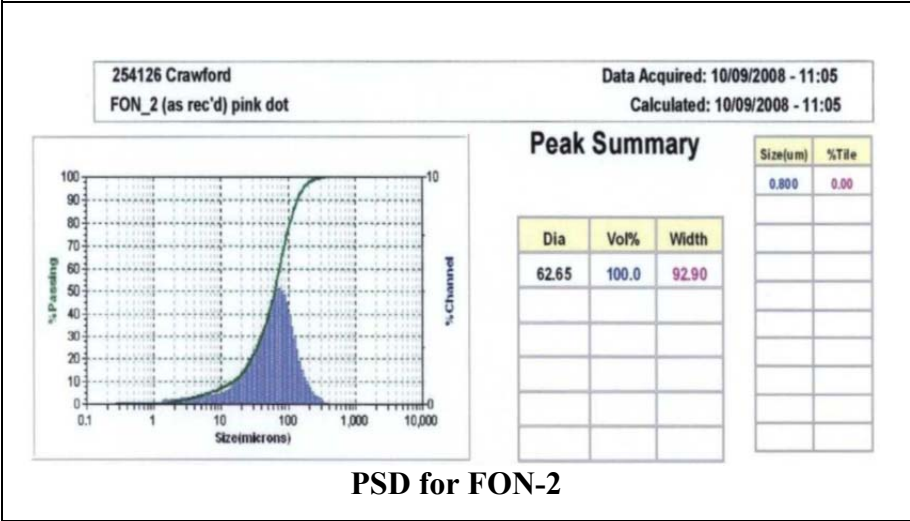
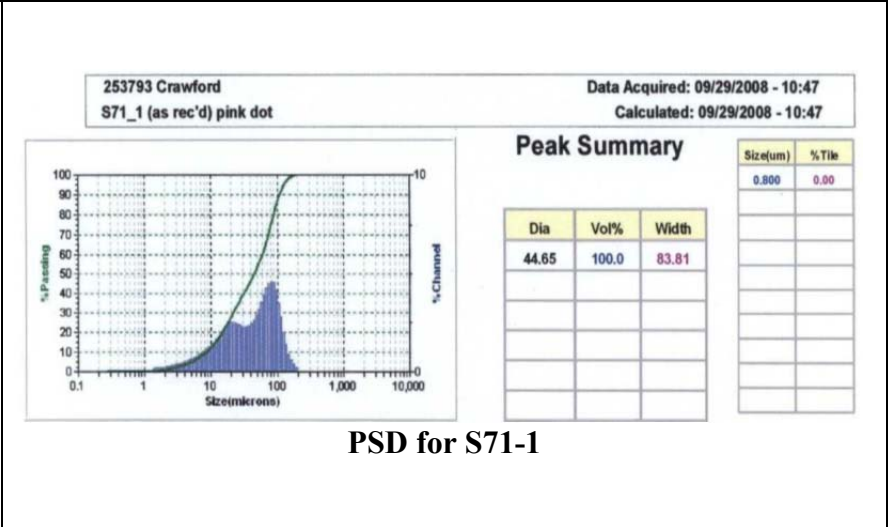
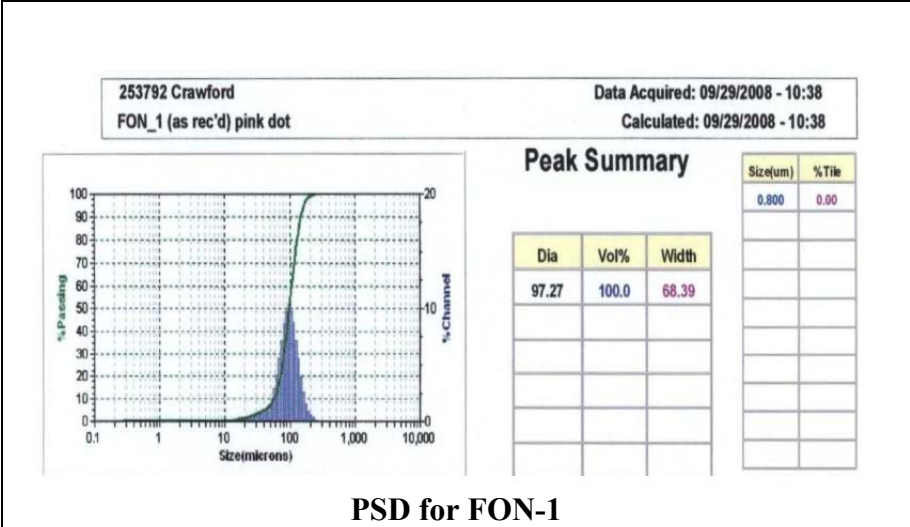
PSD for OPC-2

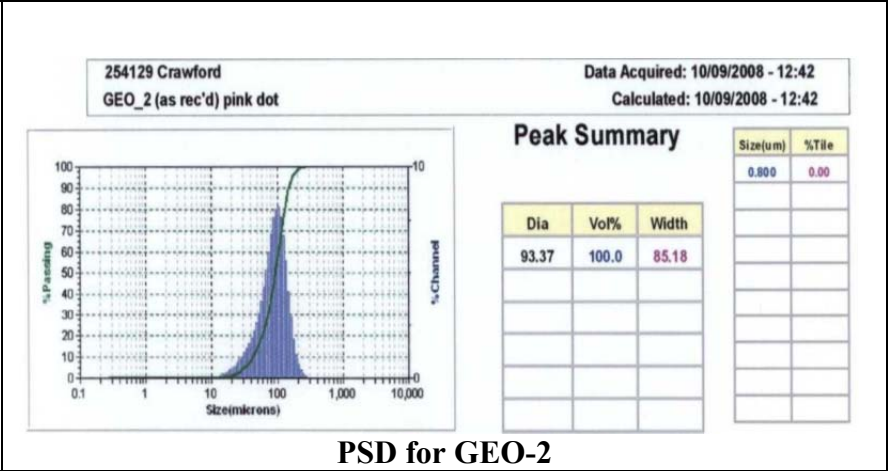
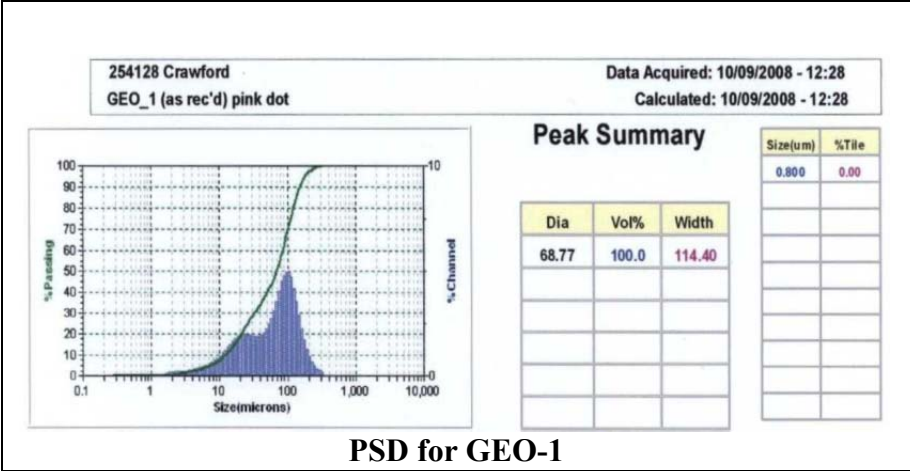
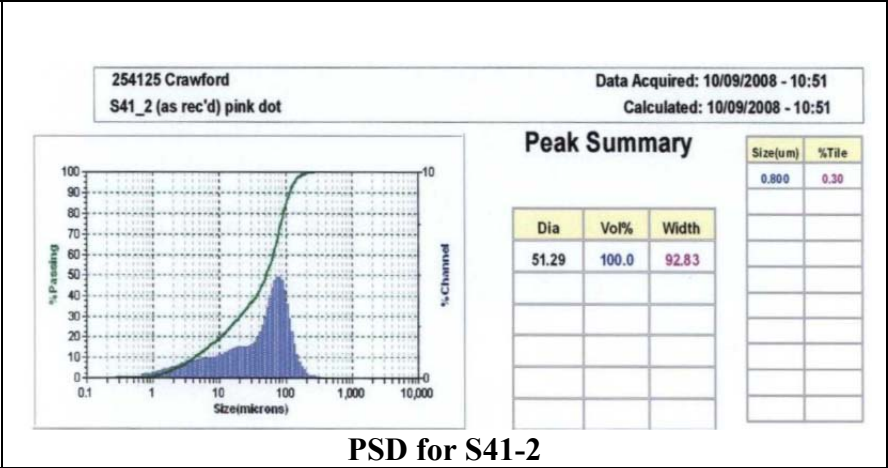
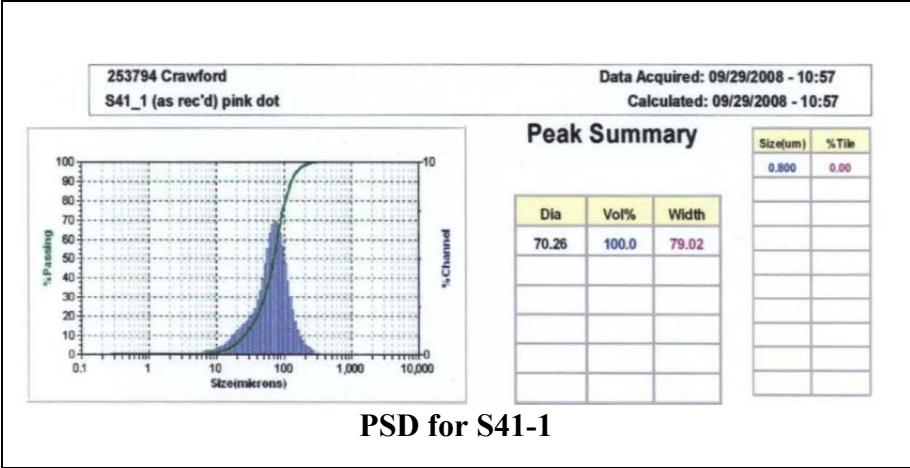


PSD for CER-1

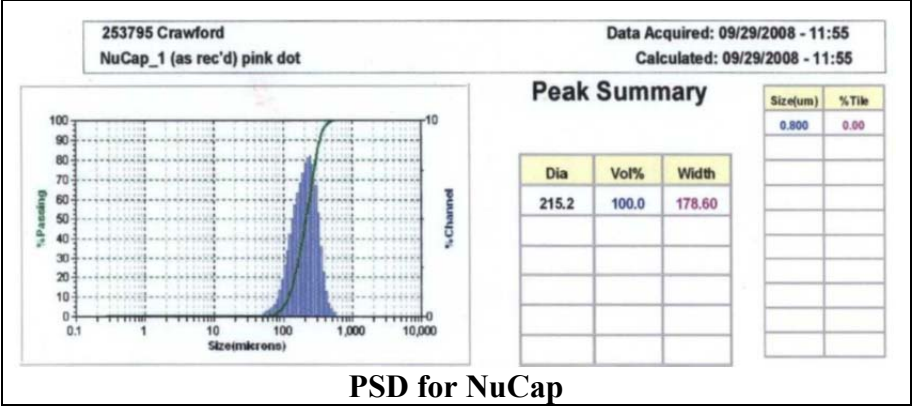
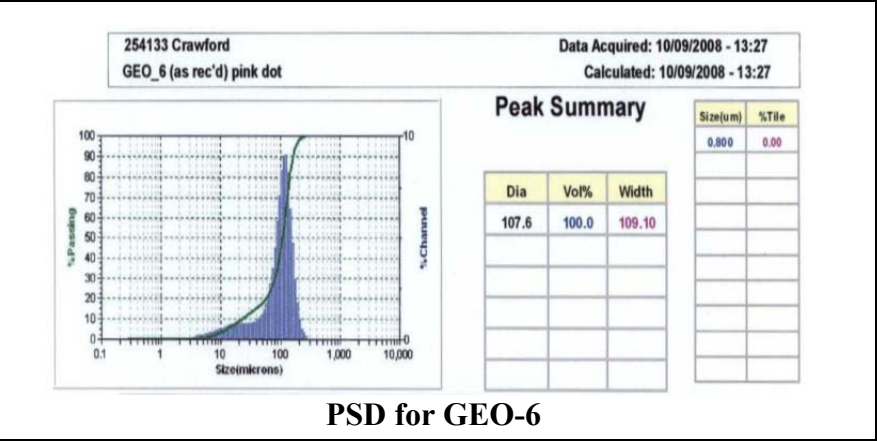
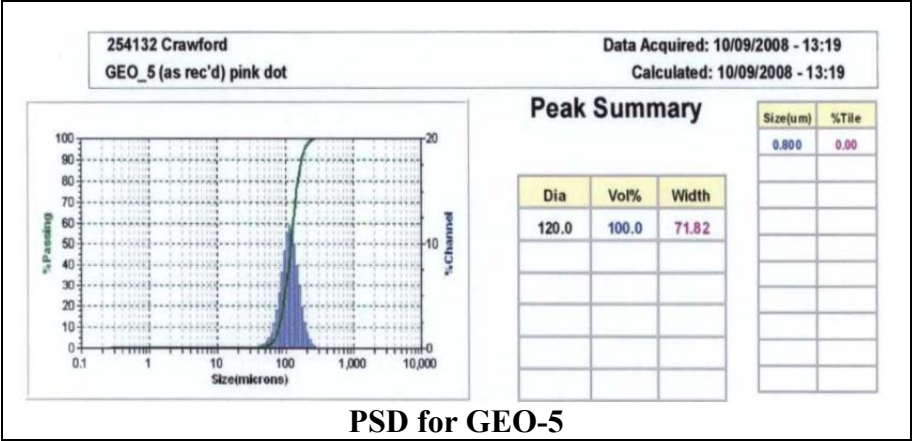
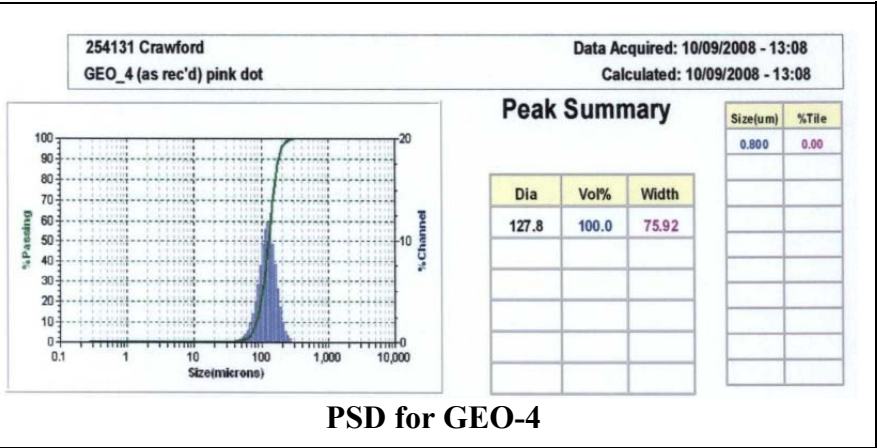
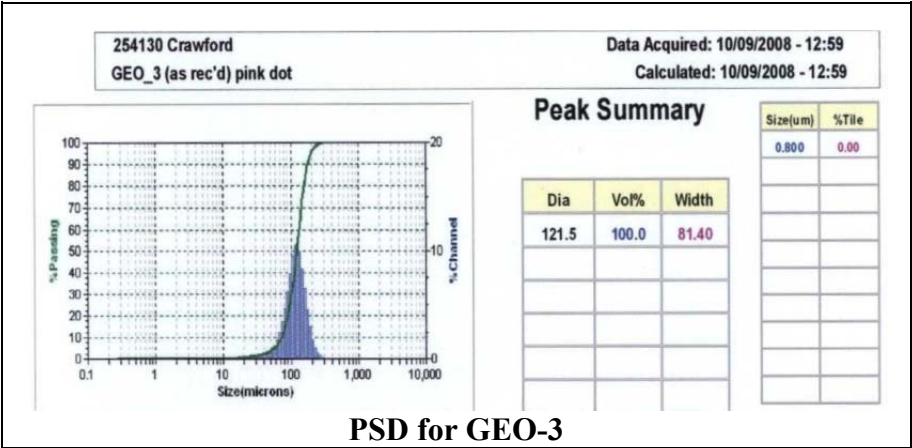


PSD for CER-2









## APPENDIX 11. TCLP DATA FOR 3" X 6" AND 6" X 12" CYLINDER MONOLITHS

3" X 6" Cylinders													
Element	FON-2 LAW-P-1B	FON-2 WTPSW- P-2B	S71-2 LAW-P- 1B	S71-2 WTPSW- P-2B	GEO-1 LAW-P- 1B	GEO-1 WTPSW - P-2B	GEO-7 LAW-P- 1B	GEO-7 WTPSW - P-2B	UTS *	(w 640 g clay/L) P-1B LAW Target	(w 307 g clay/L) P-2B WTP-SW Target	Reporting Limit (RL)	Method Detection Limit (MDL)
	(mg/L)	(mg/L)	(mg/L)	(mg/L)	(mg/L)	(mg/L)	(mg/L)	(mg/L)	(mg/L)	(g/L)	(g/L)	(mg/L)	(mg/L)
Sb	0.272	0.179	0.382	0.944	1.71	1.76	2.32	0.312	1.15	0.426	0.175	0.1	0.03
As	0.0637 <sup>J</sup>	0.042 <sup>U</sup>	0.0621 <sup>J</sup>	1.21	1.99	0.313	3.07	0.0641 <sup>J</sup>	5	0.949	0.007	0.15	0.05
Ba	0.237	0.234	0.316	0.558	0.369	0.363	0.601	0.231	21	1.37	0.003	0.05	0.01
Cd	0.00276 <sup>U</sup>	0.00296 <sup>U</sup>	0.00326 <sup>U</sup>	0.236	0.0213 <sup>J</sup>	0.00567 <sup>U</sup>	0.134	0.00351 <sup>U</sup>	0.11	0.381	0.088	0.05	0.01
Cr	0.165	0.372	0.0406 <sup>J</sup>	0.445	0.0414 <sup>J</sup>	0.166	0.112	0.0598	0.6	0.436	0.282	0.05	0.02
Pb	0.000745 <sup>U</sup>	0.0109 <sup>U</sup>	< 0.025 <sup>U</sup>	0.485	0.107	0.0572 <sup>J</sup>	0.703	0.00126 <sup>U</sup>	0.75	1.013	0.244	0.1	0.025
Se	0.0466 <sup>U</sup>	0.415	0.0695 <sup>J</sup>	1.59	0.483	0.844	0.692	0.148 <sup>J</sup>	5.7	0.078	0.175	0.15	0.05
Ag	< 0.01 <sup>U</sup>	< 0.01 <sup>U</sup>	< 0.01 <sup>U</sup>	0.000068 <sup>U</sup>	< 0.01 <sup>U</sup>	< 0.01 <sup>U</sup>	< 0.01 <sup>U</sup>	< 0.01 <sup>U</sup>	0.14	0.14	0.083	0.05	0.01
Ni	0.00986 <sup>U</sup>	0.0109 <sup>J</sup>	0.0127 <sup>J</sup>	0.337	0.0312 <sup>J</sup>	0.0398 <sup>J</sup>	0.0845	0.0147 <sup>J</sup>	11	0.502	0.241	0.05	0.01
Tl	0.0136 <sup>J</sup>	0.0338 <sup>U</sup>	0.0766 <sup>J</sup>	0.0732 <sup>J</sup>	0.0378 <sup>U</sup>	-0.0131 <sup>U</sup>	-0.0052 <sup>U</sup>	0.0496 <sup>U</sup>	0.2	0.333	0.175	0.2	0.05
Zn	0.0476 <sup>UJV</sup>	0.0438 <sup>UJV</sup>	0.0439 <sup>UJV</sup>	0.673	0.0437 <sup>J</sup>	0.115	0.0694 <sup>UJV</sup>	0.046 <sup>UJV</sup>	4.3	0	0.428	0.1	0.02

\*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; UV = Positive blank result and sample result > MDL

UJV = Positive blank result and sample result below RL but above MDL

3" x 6" Cylinders							
Element	L-TEM-76%, P1A	L-TEM-80%, P1A	L-TEM-82%, P1A	UTS *	(w 675 g clay/L) P-1B LAW Target	Reporting Limit (RL)	Method Detection Limit (MDL)
	(mg/L)	(mg/L)	(mg/L)	(mg/L)	(g/L)	(mg/L)	(mg/L)
Sb	0.816	1.05	1.07	1.15	0.422	0.1	0.03
As	0.056 <sup>J</sup>	0.0801 <sup>J</sup>	0.0767 <sup>J</sup>	5	0.986	0.15	0.05
Ba	0.185	0.118	0.0868	21	1.384	0.05	0.01
Cd	0.00127 <sup>U</sup>	0.000601 <sup>U</sup>	0.00597 <sup>U</sup>	0.11	0.377	0.05	0.01
Cr	0.096	0.109	0.0787	0.6	0.432	0.05	0.02
Pb	0.0028 <sup>U</sup>	0.0163 <sup>U</sup>	0.00597 <sup>U</sup>	0.75	1.002	0.1	0.025
Se	0.0787 <sup>J</sup>	0.113 <sup>J</sup>	0.0457 <sup>U</sup>	5.7	0.078	0.15	0.05
Ag	< 0.01 <sup>U</sup>	< 0.01 <sup>U</sup>	< 0.01 <sup>U</sup>	0.14	0.139	0.05	0.01
Ni	0.0118 <sup>J</sup>	0.0142 <sup>J</sup>	0.0163 <sup>J</sup>	11	0.497	0.05	0.01
Tl	0.0115 <sup>U</sup>	0.00122 <sup>U</sup>	0.0286 <sup>U</sup>	0.2	0.330	0.2	0.05
Zn	0.0377 <sup>UJV</sup>	0.0475 <sup>UJV</sup>	0.0479 <sup>UJV</sup>	4.3	0	0.1	0.02

\*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; UV = Positive blank result and sample result > MDL

UJV = Positive blank result and sample result below RL but above MDL

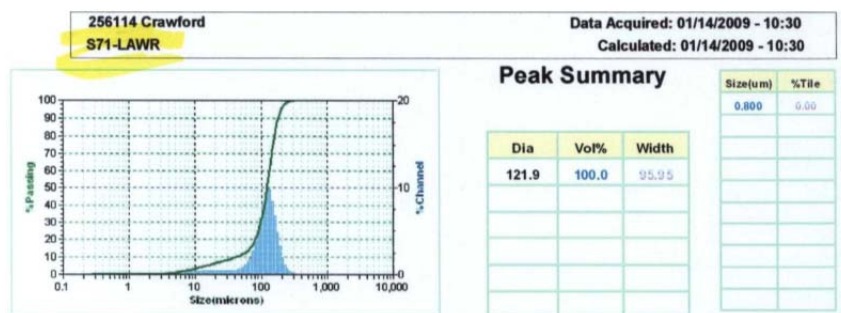
6" x 12" Cylinders														
Element	FON-2 LAW-P- 1B	FON-2 WTPSW - P-2B	S71-2 LAW-P- 1B	S71-2 WTPSW - P-2B	GEO-1 LAW-P- 1B	GEO-1 WTPSW - P-2B	GEO-7 LAW- P-1B	GEO-7 WTPSW - P-2B		UTS *	(w 640 g clay/L) P-1B LAW Target	(w 307 g clay/L) P-2B WTP-SW Target	Reporting Limit (RL)	Method Detection Limit (MDL)
	(mg/L)	(mg/L)	(mg/L)	(mg/L)	(mg/L)	(mg/L)	(mg/L)	(mg/L)		(mg/L)	(g/L)	(g/L)	(mg/L)	(mg/L)
Sb	0.419	0.366	1	0.862	1.09	1.79	1.05	0.866		1.15	0.426	0.175	0.1	0.03
As	< 0.05 <sup>U</sup>	< 0.05 <sup>U</sup>	0.336	< 0.05 <sup>U</sup>	2.03	3.52	< 0.05 <sup>U</sup>	0.113 <sup>J</sup>		5	0.949	0.007	0.15	0.05
Ba	0.226	0.332	2.01	0.55	0.525	0.251	0.456	0.421		21	1.37	0.003	0.05	0.01
Cd	< 0.01 <sup>U</sup>	< 0.01 <sup>U</sup>	0.526	0.158	0.0201 <sup>J</sup>	0.058	0.0665	0.0299 <sup>J</sup>		0.11	0.381	0.088	0.05	0.01
Cr	0.437	0.198	0.155	0.59	0.0784	0.091	0.184	0.262		0.6	0.436	0.282	0.05	0.02
Pb	0.0113 <sup>U</sup>	< 0.025 <sup>U</sup>	0.0899 <sup>J</sup>	0.0767 <sup>J</sup>	0.0699 <sup>J</sup>	0.386	0.0438 <sup>J</sup>	0.143		0.75	1.013	0.244	0.1	0.025
Se	0.16	0.371	0.111 <sup>J</sup>	0.188	0.547	0.791	1.09	1.22		5.7	0.078	0.175	0.15	0.05
Ag	0.0122 <sup>J</sup>	0.0123 <sup>J</sup>	0.0145 <sup>J</sup>	0.0414 <sup>J</sup>	0.00512 <sup>U</sup>	0.00309 <sup>U</sup>	0.00157 <sup>U</sup>	0.0148 <sup>J</sup>		0.14	0.14	0.083	0.05	0.01
Ni	0.0136 <sup>J</sup>	0.0175 <sup>J</sup>	0.285	0.634	0.044 <sup>J</sup>	0.0524	0.142	0.21		11	0.502	0.241	0.05	0.01
Tl	< 0.05 <sup>U</sup>	< 0.05 <sup>U</sup>	< 0.05 <sup>U</sup>	0.0198 <sup>U</sup>	< 0.05 <sup>U</sup>	< 0.05 <sup>U</sup>	0.00723 <sup>U</sup>	0.0583 <sup>J</sup>		0.2	0.333	0.175	0.2	0.05
Zn	0.0486 <sup>UVJ</sup>	0.0506 <sup>UVJ</sup>	0.0755 <sup>UVJ</sup>	0.538	0.0548 <sup>UVJ</sup>	0.0533 <sup>UVJ</sup>	0.195	0.356		4.3	0	0.428	0.1	0.02

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

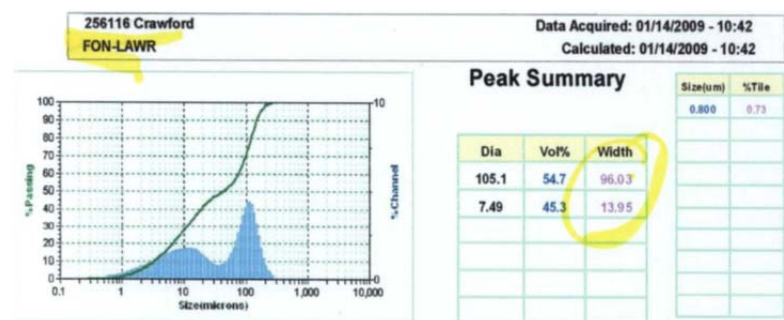
U = results less than MDL, or “U” flag; J = results below Reporting Limit but above the MDL, or “J” flag; UV = Positive blank result and sample result > MDL

UJV = Positive blank result and sample result below RL but above MDL

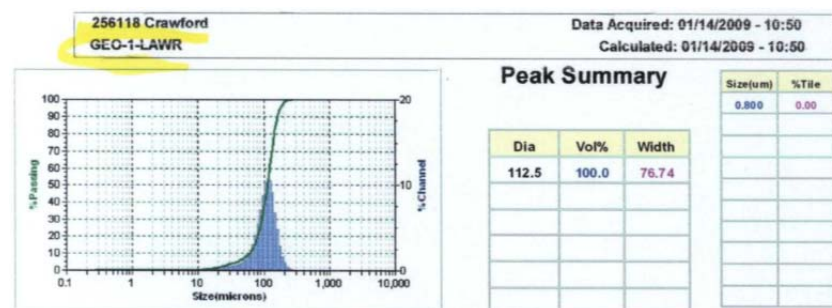
## APPENDIX 12. MICROTRAC PARTICLE SIZE DATA FOR 3" X 6" CYLINDERS CONTAINING WTP-SW FOR 100 - 200 MESH POWDERS PREPARED FOR PCT



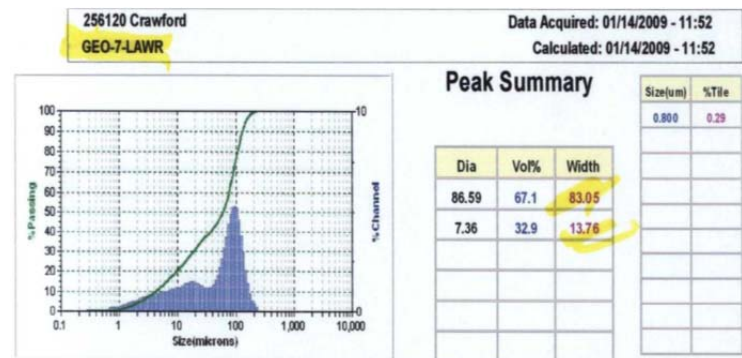
PSD for S71 WTP-SW



PSD for FON WTP-SW

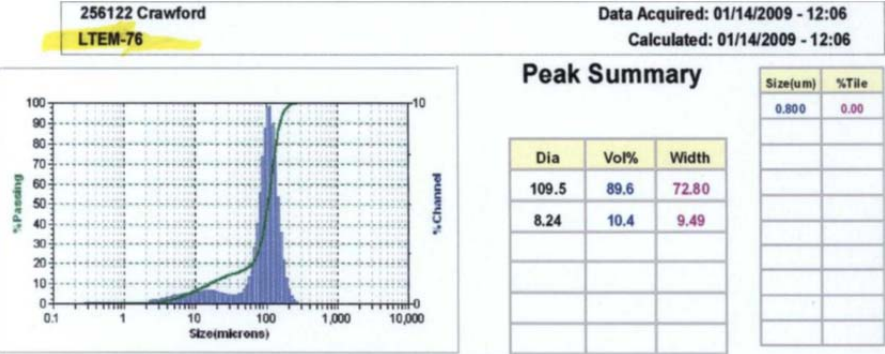


PSD for GEO-1 WTP-SW

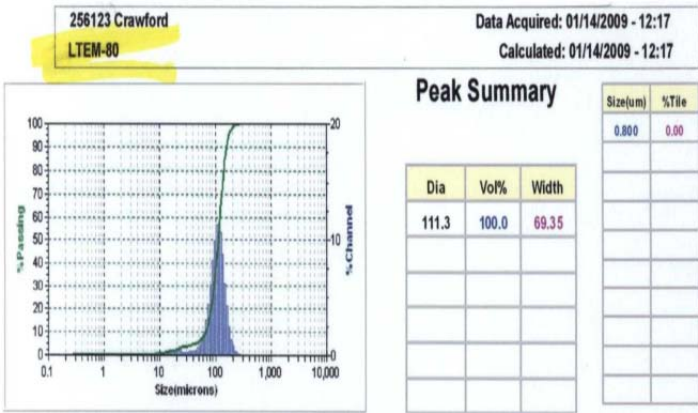


PSD for GEO-7 WTP-SW

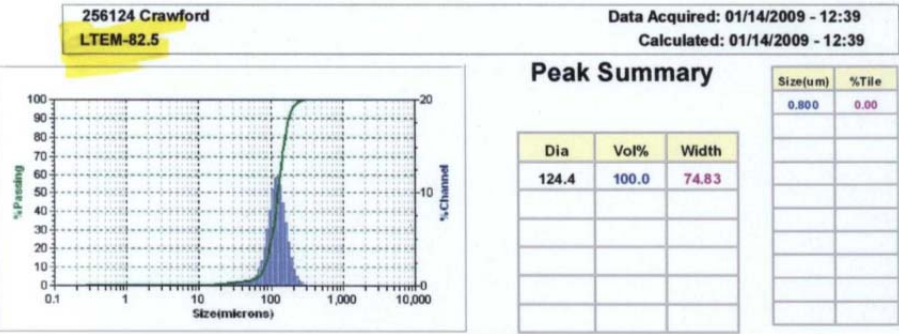




PSD for LTEM-76



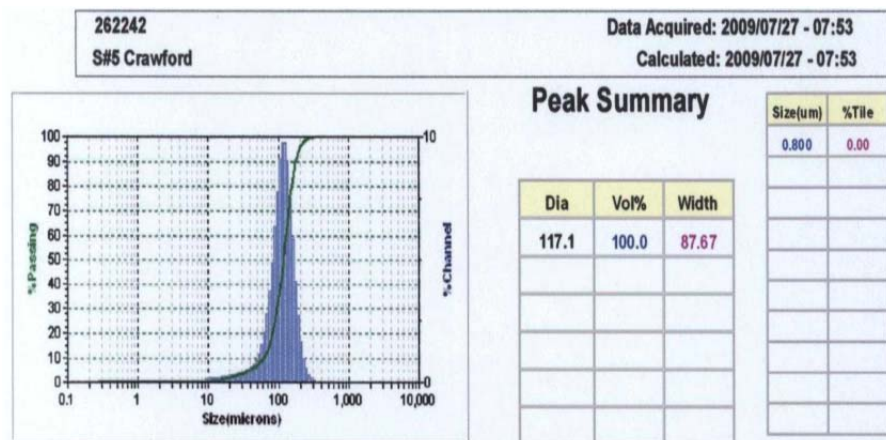
PSD for LTEM-80



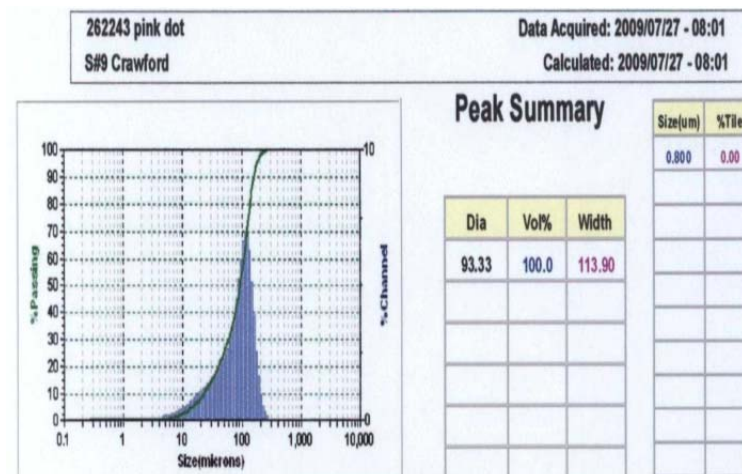
PSD for LTEM-82.5



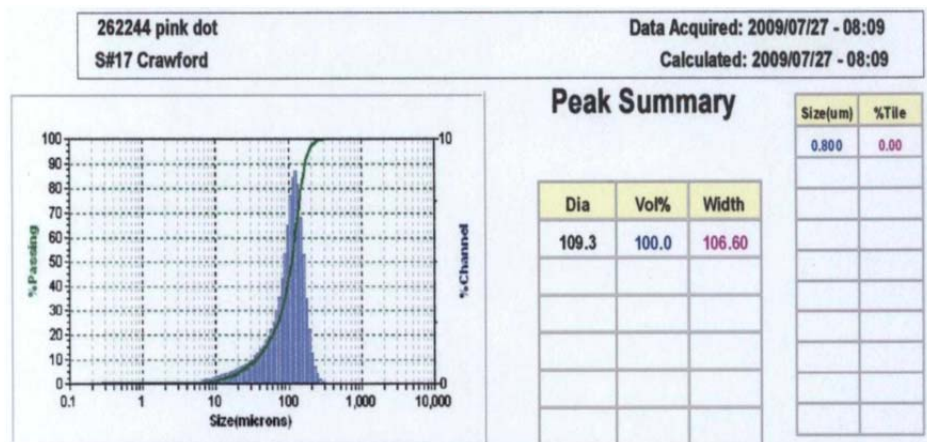
# APPENDIX 13. MICRTOTRAC PARTICLE SIZE DISTRIBUTION OF 2" X 4" CYLINDERS FOR 100 - 200 MESH POWDERS PREPARED FOR PCT



PSD for S#5 GEO-7 WTP-SW



PSD for S#9 GEO-7 WTP-SW



PSD for S#17 GEO-7 WTP-SW

BLANK PAGE

APPENDIX 14. PCT DATA FOR BLANKS AND REFERENCE GLASSES<sup>18,19,20</sup>

## PCT Data for Blanks and Reference Glasses from PR and HTF Samples

PR & HTF Bed and Fines									Measured / Avg.					Avg. All
Leachate			Al		B		Na		Si	pH	Al	B	Na	Si
Concentration	pH		(mg/L)		(mg/L)		(mg/L)		(mg/L)					
Blank 1	6.98	<	0.10	<	0.10	<	0.10	<	0.10					
Blank 2	6.95	<	0.10	<	0.10	<	0.10	<	0.10					
ARM-1	10.07		4.3		18.7		36.0		60.0					
ARM-2	10.03		4.2		18.0		35.5		58.8					
ARM-3	10.06		4.1		17.9		35.4		58.6					
Average	10.05		4.19		18.22		35.62		59.11					
St.Dev.	0.02		0.07		0.44		0.29		0.79					
Avg.(St.dev.)	10.17 (0.29)		4.85 (0.5)				36.22 (2.45)		61.23 (4.07)	0.99	0.86		0.98	0.97
LRM-1	10.7		13.1		23.0		138.0		68.9					
LRM-2	10.72		12.9		22.9		136.9		68.8					
LRM-3	10.75		13.3		23.3		138.4		69.8					
Average	10.72		13.1		23.1		137.8		69.1					
St.Dev.	0.03		0.20		0.20		0.76		0.56					
Avg.(St.dev.)	10.92 (0.092)		14.3 (2.61)		26.7 (1.83)		160 (11.5)		82 (3.53)	0.98	0.92	0.86	0.86	0.84
Normalized			Al		B		Na		Si					
Concentration			(g/L)		(g/L)		(g/L)		(g/L)					
ARM-1			0.136		NA		0.501		0.276					
ARM-2			0.133		NA		0.495		0.271					
ARM-3			0.132		NA		0.494		0.270					
Average			0.134		NA		0.497		0.272					
St.Dev.			0.002		NA		0.004		0.004					
Avg.(St.dev.)			0.155 (0.017)		NA		0.505 (0.054)		0.282 (0.03)		0.86		0.98	0.97
LRM-1			0.260		0.943		0.929		0.272					
LRM-2			0.256		0.939		0.922		0.271					
LRM-3			0.264		0.955		0.931		0.275					
Average			0.260		0.946		0.927		0.273					
St.Dev.			0.004		0.008		0.005		0.002					
Avg.			0.284		1.095		1.077		0.324		0.92	0.86	0.86	0.84
St.Dev.			0.052		0.075		0.077		0.014					
Normalized			Al		B		Na		Si					
Release			(g/m <sup>2</sup> )		(g/m <sup>2</sup> )		(g/m <sup>2</sup> )		(g/m <sup>2</sup> )					
2,000 m-1														
LRM-1			0.130		0.472		0.465		0.136					
LRM-2			0.128		0.469		0.461		0.136					
LRM-3			0.132		0.477		0.466		0.138					
Average			0.130		0.473		0.464		0.136					
St.Dev.			0.002		0.004		0.003		0.001					
Avg.			0.142		0.548		0.538		0.162		0.92	0.86	0.86	0.84
St.Dev.			0.026		0.038		0.039		0.007					
Normalized			Al		B		Na		Si					
Release			(g/m <sup>2</sup> )		(g/m <sup>2</sup> )		(g/m <sup>2</sup> )		(g/m <sup>2</sup> )					
4,000 m-1														
LRM-1			0.065		0.236		0.232		0.068					
LRM-2			0.064		0.235		0.230		0.068					
LRM-3			0.066		0.239		0.233		0.069					
Average			0.065		0.236		0.232		0.068					
St.Dev.			0.001		0.002		0.001		0.001					
Avg.			0.071		0.274		0.269		0.081		0.92	0.86	0.86	0.84
St.Dev.			0.013		0.019		0.019		0.003					

## PCT Data for Blanks and Reference Glasses from 2" Cube Samples

2" Cube Monoliths		Measured / Avg.										Avg. All			
Leachate			Al		B		Na		Si	pH	Al	B	Na	Si	
Concentration	pH		(mg/L)		(mg/L)		(mg/L)		(mg/L)						
Blank 1	6.83	<	0.10	<	0.10	<	0.10	<	0.10						
Blank 2	6.79	<	0.10	<	0.10	<	0.10	<	0.10						
ARM-1	9.95		4.3		17.6		33.9		59.9						
ARM-2	9.95		4.3		17.4		33.3		59.3						
ARM-3	9.95		4.3		17.9		34.4		61.0						
Average	9.95		4.31		17.64		33.86		60.07						
St.Dev.	0.00		0.03		0.26		0.55		0.89						
Avg.(St.dev.)	10.17 (0.29)		4.85 (0.5)				36.22 (2.45)		61.23 (4.07)	0.98	0.89		0.93	0.98	0.89
LRM-1	10.54		12.7		20.7		127.1		69.3						
LRM-2	10.54		12.8		21.4		129.1		70.3						
LRM-3	10.54		12.8		19.7		125.5		68.0						
Average	10.54		12.76		20.56		127.24		69.17						
St.Dev.	0.00		0.03		0.85		1.84		1.13						
Avg.(St.dev.)	10.92 (0.092)		14.3 (2.61)		26.7 (1.83)		160 (11.5)		82 (3.53)	0.97	0.89	0.77	0.80	0.84	
Normalized			Al		B		Na		Si						
Concentration			(g/L)		(g/L)		(g/L)		(g/L)						
ARM-1			0.138		NA		0.472		0.276						
ARM-2			0.137		NA		0.464		0.273						
ARM-3			0.139		NA		0.480		0.281						
Average			0.138		NA		0.472		0.277						
St.Dev.			0.001		NA		0.008		0.004						
Avg.(St.dev.)			0.155 (0.017)		NA		0.505 (0.054)		0.282 (0.03)		0.89		0.93	0.98	0.87
LRM-1			0.253		0.847		0.855		0.273						
LRM-2			0.254		0.876		0.869		0.277						
LRM-3			0.254		0.807		0.844		0.268						
Average			0.253		0.843		0.856		0.273						
St.Dev.			0.001		0.035		0.012		0.004						
Avg.			0.284		1.095		1.077		0.324		0.89	0.77	0.80	0.84	
St.Dev.			0.000		0.000		0.000		0.000						
Normalized			Al		B		Na		Si						
Release			(g/m²)		(g/m²)		(g/m²)		(g/m²)						
2,000 m-1															
LRM-1			0.126		0.424		0.428		0.137						
LRM-2			0.127		0.438		0.435		0.139						
LRM-3			0.127		0.403		0.422		0.134						
Average			0.127		0.422		0.428		0.137						
St.Dev.			0.000		0.017		0.006		0.002						
Avg.			0.142		0.548		0.538		0.162		0.89	0.77	0.80	0.84	0.83
St.Dev.			0.000		0.000		0.000		0.000						
Normalized			Al		B		Na		Si						
Release			(g/m²)		(g/m²)		(g/m²)		(g/m²)						
4,000 m-1															
LRM-1			0.063		0.212		0.214		0.068						
LRM-2			0.064		0.219		0.217		0.069						
LRM-3			0.063		0.202		0.211		0.067						
Average			0.063		0.211		0.214		0.068						
St.Dev.			0.000		0.009		0.003		0.001						
Avg.			0.071		0.274		0.269		0.081		0.89	0.77	0.80	0.84	0.83
St.Dev.			0.000		0.000		0.000		0.000						

## PCT Data for Blanks and Reference Glasses from 3" x 6" Cylinder Samples

3" x 6" Cylinders									Measured / Avg.					Avg. All
Leachate			Al		B		Na		Si	pH	Al	B	Na	Si
Concentration	pH		(mg/L)		(mg/L)		(mg/L)		(mg/L)					
Blank 1	6.79	<	0.10	<	0.10	<	0.10	<	0.10					
Blank 2	6.72	<	0.10	<	0.10	<	0.10	<	0.10					
ARM-1	10.2		4.0		16.1		31.2		56.3					
ARM-2	10.18		4.1		17.0		34.4		59.9					
ARM-3	10.19		4.2		17.0		34.5		59.9					
Average	10.19		4.10		16.69		33.34		58.69					
St.Dev.	0.01		0.13		0.52		1.89		2.10					
Avg.(St.dev.)	10.17 (0.29)		4.85 (0.5)				36.22 (2.45)		61.23 (4.07)	1.00	0.85		0.92	0.96 0.94
LRM-1	10.83		14.3		23.4		147.9		78.9					
LRM-2	10.82		13.5		22.2		138.6		73.4					
LRM-3	10.82		14.9		24.1		150.5		78.0					
Average	10.82		14.23		23.26		145.67		76.77					
St.Dev.	0.01		0.74		0.95		6.26		2.99					
Avg.(St.dev.)	10.92 (0.092)		14.3 (2.61)		26.7 (1.83)		160 (11.5)		82 (3.53)	0.99	1.00	0.87	0.91	0.94
Normalized			Al		B		Na		Si					
Concentration			(g/L)		(g/L)		(g/L)		(g/L)					
ARM-1			0.127		NA		0.434		0.259					
ARM-2			0.132		NA		0.479		0.276					
ARM-3			0.135		NA		0.481		0.276					
Average			0.131		NA		0.465		0.270					
St.Dev.			0.004		NA		0.026		0.010					
Avg.(St.dev.)			0.155 (0.017)		NA		0.505 (0.054)		0.282 (0.03)		0.85		0.92	0.96 0.92
LRM-1			0.285		0.961		0.995		0.312					
LRM-2			0.267		0.912		0.933		0.290					
LRM-3			0.296		0.989		1.013		0.308					
Average			0.283		0.954		0.980		0.303					
St.Dev.			0.015		0.039		0.042		0.012					
Avg.			0.284		1.095		1.077		0.324		1.00	0.87	0.91	0.94
St.Dev.			0.000		0.000		0.000		0.000					
Normalized			Al		B		Na		Si					
Release			(g/m <sup>2</sup> )		(g/m <sup>2</sup> )		(g/m <sup>2</sup> )		(g/m <sup>2</sup> )					
2,000 m-1														
LRM-1			0.142		0.481		0.498		0.156					
LRM-2			0.134		0.456		0.466		0.145					
LRM-3			0.148		0.495		0.507		0.154					
Average			0.141		0.477		0.490		0.152					
St.Dev.			0.007		0.020		0.021		0.006					
Avg.			0.142		0.548		0.538		0.162		1.00	0.87	0.91	0.94 0.93
St.Dev.			0.000		0.000		0.000		0.000					
Normalized			Al		B		Na		Si					
Release			(g/m <sup>2</sup> )		(g/m <sup>2</sup> )		(g/m <sup>2</sup> )		(g/m <sup>2</sup> )					
4,000 m-1														
LRM-1			0.071		0.240		0.249		0.078					
LRM-2			0.067		0.228		0.233		0.072					
LRM-3			0.074		0.247		0.253		0.077					
Average			0.071		0.239		0.245		0.076					
St.Dev.			0.004		0.010		0.011		0.003					
Avg.			0.071		0.274		0.269		0.081		1.00	0.87	0.91	0.94 0.93
St.Dev.			0.000		0.000		0.000		0.000					

## PCT Data for Blanks and Reference Glasses from 6" x 12" Cylinder Samples

6" x 12" Cylinders									Measured / Avg.					Avg. All
Leachate			Al		B		Na		Si	pH	Al	B	Na	Si
Concentration	pH		(mg/L)		(mg/L)		(mg/L)		(mg/L)					
Blank 1	6.8	<	0.10	<	0.10	<	0.10	<	0.10					
Blank 2	6.6	<	0.10	<	0.10	<	0.10	<	0.10					
ARM-1	10.49		4.9		16.6		34.0		58.9					
ARM-2	10.44		5.0		16.7		34.3		59.6					
ARM-3	10.44		5.0		16.8		33.8		59.5					
Average	10.46		4.96		16.68		34.04		59.37					
St.Dev.	0.03		0.02		0.12		0.27		0.36					
Avg.(St.dev.)	10.17 (0.29)		4.85 (0.5)				36.22 (2.45)		61.23 (4.07)	1.03	1.02		0.94	0.97
EA-1	12.00	<	0.2		590.5		1537.1		910.9					
EA-2	12.00	<	0.2		631.1		1600.1		951.5					
EA-3	11.97	<	0.2		622.7		1567.6		961.5					
Average	11.99		0.17		614.76		1568.3		941.32					
St.Dev.	0.02		0.00		21.46		31.52		26.80					
Avg.(St.dev.)	11.85 (0.1)				587 (43)		1662 (112)		893 (86)	1.01		1.05	0.94	1.05
Normalized			Al		B		Na		Si					
Concentration			(g/L)		(g/L)		(g/L)		(g/L)					
ARM-1			0.158		NA		0.474		0.271					
ARM-2			0.160		NA		0.479		0.275					
ARM-3			0.158		NA		0.471		0.274					
Average			0.159		NA		0.475		0.273					
St.Dev.			0.001		NA		0.004		0.002					
Avg.(St.dev.)			0.155 (0.0172)		NA		0.505 (0.0539)		0.282 (0.03)		1.02		0.94	0.97
EA-1			NA		16.92		12.30		4.00					
EA-2			NA		18.08		12.80		4.18					
EA-3			NA		17.84		12.54		4.22					
Average			NA		17.61		12.55		4.13					
St.Dev.			NA		0.61		0.25		0.12					
Avg.			NA		16.695 (1.222)		13.346 (0.902)		3.922 (0.376)			1.06	0.94	1.05
St.Dev.			NA		0.000		0.000		0.000					

## PCT Data for Blanks and Reference Glasses from 2" x 4" Cylinder Samples

2" x 4" Cylinders									Measured / Avg.					Avg. All
Leachate			Al		B		Na		Si	pH	Al	B	Na	Si
Concentration	pH		(mg/L)		(mg/L)		(mg/L)		(mg/L)					
Blank 1	6.8	<	0.10	<	0.10	<	0.10	<	0.10					
Blank 2	6.88	<	0.10	<	0.10	<	0.10	<	0.10					
ARM-1	10.18		5.5		18.7		39.0		61.8					
ARM-2	10.2		5.4		18.8		40.0		61.0					
ARM-3	10.2		5.6		18.8		40.5		61.8					
Average	10.19		5.47		18.78		39.83		61.56					
St.Dev.	0.01		0.09		0.10		0.76		0.48					
Avg.(St.dev.)	10.17 (0.29)		4.85 (0.5)				36.22 (2.45)		61.23 (4.07)	1.00	1.13		1.10	1.01
LRM-1	10.87		15.3		24.2		153.8		75.7					
LRM-2	10.86		14.6		22.7		147.7		70.5					
LRM-3	10.85		14.7		23.2		146.8		72.5					
Average	10.86		14.9		23.3		149.4		72.9					
St.Dev.	0.01		0.40		0.76		3.82		2.61					
Avg.(St.dev.)	10.92 (0.092)		14.3 (2.61)		26.7 (1.83)		160 (11.5)		82 (3.53)	0.99	1.04	0.87	0.93	0.89
Normalized			Al		B		Na		Si					
Concentration			(g/L)		(g/L)		(g/L)		(g/L)					
ARM-1			0.174		NA		0.544		0.285					
ARM-2			0.173		NA		0.558		0.281					
ARM-3			0.178		NA		0.565		0.285					
Average			0.175		NA		0.555		0.283					
St.Dev.			0.003		NA		0.011		0.002					
Avg.(St.dev.)			0.155 (0.0172)		NA		0.505 (0.0539)		0.282 (0.03)		1.13		1.10	1.01
LRM-1			0.305		0.991		1.035		0.299					
LRM-2			0.290		0.930		0.994		0.278					
LRM-3			0.292		0.950		0.988		0.286					
Average			0.296		0.957		1.006		0.288					
St.Dev.			0.008		0.031		0.026		0.010					
Avg.			0.284		1.095		1.077		0.324		1.04	0.87	0.93	0.89
St.Dev.			0.052		0.075		0.077		0.014					
Normalized			Al		B		Na		Si					
Release			(g/m <sup>2</sup> )		(g/m <sup>2</sup> )		(g/m <sup>2</sup> )		(g/m <sup>2</sup> )					
2,000 m-1														
LRM-1			0.152		0.496		0.518		0.149					
LRM-2			0.145		0.465		0.497		0.139					
LRM-3			0.146		0.475		0.494		0.143					
Average			0.148		0.479		0.503		0.144					
St.Dev.			0.004		0.016		0.013		0.005					
Avg.			0.142		0.548		0.538		0.162		1.04	0.87	0.93	0.89
St.Dev.			0.026		0.038		0.039		0.007					
Normalized			Al		B		Na		Si					
Release			(g/m <sup>2</sup> )		(g/m <sup>2</sup> )		(g/m <sup>2</sup> )		(g/m <sup>2</sup> )					
4,000 m-1														
LRM-1			0.076		0.248		0.259		0.075					
LRM-2			0.073		0.232		0.248		0.070					
LRM-3			0.073		0.238		0.247		0.072					
Average			0.074		0.239		0.251		0.072					
St.Dev.			0.002		0.008		0.006		0.003					
Avg.			0.071		0.274		0.269		0.081		1.04	0.87	0.93	0.89
St.Dev.			0.013		0.019		0.019		0.003					

**Distribution:**

A. B. Barnes, 999-W  
D. A. Crowley, 773-43A  
A. P. Fellingner, 773-42A  
S. D. Fink, 773-A  
B. J. Giddings, 786-5A  
C. C. Herman, 999-W  
S. L. Marra, 773-A  
F. M. Pennebaker, 773-42A  
W. R. Wilmarth, 773-A