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Results of Characterization and Retrieval Testing on Tank 241-C-109 Heel Solids

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EXECUTIVE SUMMARY

Eight samples of heel solids from tank 241-C-109 were delivered to the 222-S Laboratory for characterization and dissolution testing. After being drained thoroughly, one-half to two-thirds of the solids were off-white to tan solids that, visually, were fairly evenly graded in size from coarse silt (30-60 µm) to medium pebbles (8-16 mm). The remaining solids were mostly strongly cemented aggregates ranging from coarse pebbles (16-32 mm) to fine cobbles (6-15 cm) in size.

Solid phase characterization and chemical analysis indicated that the air-dry heel solids contained ≈58 wt% gibbsite [$Al(OH)_3$] and ≈37 wt% natrophosphate [$Na_7F(PO_4)_2 \cdot 19H_2O$]. The strongly cemented aggregates were mostly fine-grained gibbsite cemented with additional gibbsite.

Dissolution testing was performed on two test samples. One set of tests was performed on large pieces of aggregate solids removed from the heel solids samples. The other set of dissolution tests was performed on a composite sample prepared from well-drained, air-dry heel solids that were crushed to pass a ¼-in. sieve. The bulk density of the composite sample was 2.04 g/mL.

The dissolution tests included water dissolution followed by caustic dissolution testing. In each step of the three-step water dissolution tests, a volume of water approximately equal to 3 times the initial volume of the test solids was added. In each step, the test samples were gently but thoroughly mixed for approximately 2 days at an average ambient temperature of 25 °C.

The caustic dissolution tests began with the addition of sufficient 49.6 wt% NaOH to the water dissolution residues to provide ≈3.1 moles of OH for each mole of Al estimated to have been present in the starting composite sample and ≈2.6 moles of OH for each mole of Al potentially present in the starting aggregate sample. Metathesis of gibbsite to sodium aluminate was then allowed to proceed over 10 days of gentle mixing of the test samples at temperatures ranging from 26-30 °C. The metathesized sodium aluminate was then dissolved by addition of volumes of water approximately equal to 1.3 times the volumes of caustic added to the test slurries. Aluminate dissolution was allowed to proceed for 2 days at ambient temperatures of ≈29 °C.

Overall, the sequential water and caustic dissolution tests dissolved and removed 80.0 wt% of the tank 241-C-109 crushed heel solids composite test sample. The 20 wt% of solids remaining after the dissolution tests were 85-88 wt% gibbsite. If the density of the residual solids was approximately equal to that of gibbsite, they represented ≈17 vol% of the initial crushed solids composite test sample.

In the water dissolution tests, addition of a volume of water ≈6.9 times the initial volume of the crushed solids composite was sufficient to dissolve and recover essentially all of the natrophosphate present. The ratio of the weight of water required to dissolve the natrophosphate solids to the estimated weight of natrophosphate present was 8.51. The Environmental Simulation Program (OLI Systems, Inc., Morris Plains, New Jersey) predicts that an 8.36 w/w ratio would be required to dissolve the estimated weight of natrophosphate present in the absence

of other components of the heel solids. Only minor amounts of Al-bearing solids were removed from the composite solids in the water dissolution tests.

The caustic metathesis/aluminate dissolution test sequence, executed at temperatures ranging from 27-30 °C, dissolved and recovered ≈69 wt% of the gibbsite estimated to have been present in the initial crushed heel solids composite. This level of gibbsite recovery is consistent with that measured in previous scoping tests on the dissolution of gibbsite in strong caustic solutions.

Overall, the sequential water and caustic dissolution tests dissolved and removed 80.3 wt% of the tank 241-C-109 aggregate solids test sample. The residual solids were 92-95 wt% gibbsite. Only a minor portion (≈4.5 wt%) of the aggregate solids was dissolved and recovered in the water dissolution test. Other than some smoothing caused by continuous mixing, the aggregates were essentially unaffected by the water dissolution tests. During the caustic metathesis/aluminate dissolution test sequence, ≈81 wt% of the gibbsite estimated to have been present in the aggregate solids was dissolved and recovered. The pieces of aggregate were significantly reduced in size but persisted as distinct pieces of solids. The increased level of gibbsite recovery, as compared to that for the crushed heel solids composite, suggests that the way the gibbsite solids and caustic solution are mixed is a key determinant of the overall efficiency of gibbsite dissolution and recovery.

The liquids recovered after the caustic dissolution tests on the crushed solids composite and the aggregate solids were observed for 170 days. No precipitation of gibbsite was observed.

The distribution of particle sizes in the residual solids recovered following the dissolution tests on the crushed heel solids composite was characterized. Wet sieving indicated that 21.4 wt% of the residual solids were >710 µm in size, and laser light scattering indicated that the median equivalent spherical diameter in the <710-µm solids was 35 µm.

The settling behavior of the residual solids following the large-scale dissolution tests was also studied. When dispersed at a concentration of ≈1 vol% in water, ≈24 wt% of the residual solids settled at a rate >0.43 in./s; ≈68 wt% settled at rates between 0.02 and 0.43 in./s; and ≈7 wt% settled slower than 0.02 in./s.

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LIST OF TERMS

Abbreviations and Acronyms

222-S	WRPS 222-S Laboratory
A	acid digestion sample preparation
AEA	Alpha energy analysis
ATL	Advanced Technologies and Laboratories International, Inc.
C-109	single-shell waste tank 241-C-109
C_D	coefficient of drag
C-Liq	centrifuged liquid(s)
C-Slds	centrifuged solids
d	particle diameter
d##	particle diameter at a specified (##) %-undersize cut point
$D_{##}$	density at ## °C
D_{Bulk}	bulk density, i.e., the overall density of a sample including solids, liquids, and occluded void space.
D_{Sld}	solid density
D_{Liq}	liquid density
DSR	Data Summary Report
η	viscosity
ESP	Environmental Simulation Program (OLI Systems, Inc., Morris Plains, NJ)
$F_{D\#}$	fraction of particles removed from settling column after # decantings
$F_{R\#}$	fraction of particles remaining in settling column after # decantings
g	acceleration due to gravity
GEA	Gamma energy analysis
HISI	Hanford Information System Inventory
I&C	Integration and Control group at the 222-S Laboratory
IC	ion chromatography
ICP-AES	inductively coupled plasma/atomic emission spectrometry
ICP-MS	Inductively coupled plasma-mass spectrometry
IDMS	Integrated Document Management System
k	extinction coefficient
λ	wavelength
n	real part of the complex refractive index of a substance
N	complex refractive index of a substance
N_R	Reynolds number
PMP	polymethylpentene
PSD	particle size distribution
φ	particle shape factor (used in settling rate calculations)
RPD	relative percent difference
RRI	relative refractive index
SAP	sampling and analysis plan
SEM/EDX	scanning electron microscopy/energy dispersive X-ray spectrometry
SPC	solid phase characterization

Abbreviations and Acronyms (continued)

t	temperature in °C
TGA	thermogravimetric analysis
TIC	total inorganic carbon
TOC	total organic carbon
ΔTOC	total organic carbon after removal of carbon associated with analyzed organic acid anions
$V_{Settling}$	settling velocity
V_{Stokes}	Stokes' settling velocity
$V_{Terminal}$	terminal settling velocity
vol	volume
W	water leach sample preparation
wt	weight
XRD	powder X-ray diffraction or diffractometry
Z	fusion sample preparation in zirconium crucible

Units

cm	centimeter
cm/s	centimeters per second
cm/s^2	centimeters per second per second
ft^3	cubic feet
°C	degrees Celsius
gal	gallon(s)
g	gram(s)
g/cm·s	grams per centimeter second (Poise)
g/cm^3	grams per cubic centimeter
g/mL	grams per milliliter
hr(s)	hour(s)
in.	inch(es)
in./hr	inches per hour
in./s	inches per second
kg	kilogram
kHz	kilohertz
L	liter(s)
µCi/g	microcuries per gram
µCi/mL	microcuries per milliliter
µg	microgram
µg/g	micrograms per gram
µg/mL	micrograms per milliliter
µm	micrometer
mg	milligram(s)
mL	milliliter
mL/s	milliliters per second

Units (continued)

mm	millimeter
min	minute(s)
M	molar (moles/L)
nm	nanometer(s)
oz	ounce (U.S. liquid)
rpm	revolution(s) per minute
s	second(s)
vol%	volume percent
W	watt(s)
wt%	weight percent
w/w	weight for weight

1.0 INTRODUCTION

In 2010, waste retrieval from single-shell tank 241-C-109 (C-109) using modified sluicing was completed. An estimated 7,800 gal (≈1040 ft^3) of waste, primarily heel solids, remained in the tank after completion of the retrieval operations. This residual waste volume exceeds the residual waste volume requirements found in the *Hanford Federal Facility Agreement and Consent Order* (Ecology et al. 1989): <360 ft^3 (≈2,700 U.S. liquid gallons) for 100 series single-shell tanks.

A sampling and analysis plan (SAP) directing retrieval of samples of the residual waste in C-109 was issued in October 2010 (RPP-PLAN-47927, *Sampling and Analysis Plan for Waste Solids in Tank 241-C-109 After Sluicing*); it was modified in April 2011 (Characterization Change Notice 11-CCN-02 issued April 21, 2011) and in May 2011 (Characterization Change Notice 11-CCN-04 issued May 25, 2011). (The Characterization Change Notices and other electronic correspondence are attached to the initial analytical report issued by Advanced Technologies and Laboratories International, Inc. [ATL] [RPP-RPT-50883, *Final Report for the Analysis of Waste Solids in Tank 241-C-109, 2011*].) Additional modifications/guidance was provided by verbal direction in meetings with the client(s) on June 7, October 6, and October 25, 2011. The final SAP directed that a test program be developed to provide data needed to evaluate retrieval technologies that could be used to remove more of the residual heel solids from C-109. At a minimum, the test program was required to produce the following information:

- The chemical and radiochemical composition of the C-109 heel solids
- The bulk density of the heel solids
- The mineralogical properties (i.e., the solid phase composition) of the heel solids
- The mode and strength of grain adhesion in solid aggregates, if present
- The amount of heel solids retrievable by addition(s) of water to the heel solids at ≈25 °C
- The amount of heel solids retrievable by addition of 50 wt% caustic at ≈25 °C at a ratio of 3 moles OH:1 mole Al
- The chemical and radiochemical composition of any solids remaining at the end of the dissolution test(s)
- The bulk density of any residual solids
- The particle size distribution in any residual solids
- The settling behavior and rate(s) of any residual solids in water

A laboratory test plan presenting a strategy to acquire the required information was issued in December 2011 (LAB-PLN-11-00006, *Test Plan for Tank 241-C-109 Heel Solids Characterization and Dissolution Testing*). The test plan was prepared by members of the Integration and Control group (I&C) at the 222-S Laboratory (222-S) and was reviewed and approved by the client(s).

2.0 TEST SAMPLES

2.1 TANK SAMPLES

Tank C-109 went into service in 1948 and was declared inactive in 1977. During its service life, the tank was used to store waste from several sources including PUREX aluminum cladding waste, Hot Semi-Works waste, in-farm ferrocyanide waste, and bismuth phosphate first-cycle waste. The tank was interim stabilized in November 2003.

Most of the waste in C-109 was transferred to a double-shell tank using modified sluicing. Post-sluicing videos showed that most of the remaining "heel" solids were in three large mounds: two located on the east and west sides of the tank and one located near the center of the tank. The remaining solids appeared to form a thin layer covering most of the tank bottom except near the north and south walls where the sluicers were located. The solids in the large piles near the east and west walls appeared to be mostly large particles or aggregates while the remaining heel solids appeared to be composed of mostly smaller particles (RPP-PLAN-47927).

Eight samples of the post sluicing waste in C-109 were collected using the Off-Riser Sampling System. Five were retrieved on April 12, 2011, and transferred to the 222-S 11A hot cells on April 13; three were collected on April 18, 2011, and transferred to the hot cells on April 19. The samples were initially collected in 250-mL fluoro-polymer sample bottles. Photographs and qualitative descriptions of the eight as-received samples are included in the ATL analysis report (RPP-RPT-50883).

2.2 HEEL SAMPLE LIQUIDS

Free liquid was decanted from six of the eight C-109 heel samples during the period April 26-28, 2011 (no liquid was present in samples C109-10-01A and -02D). All decanted liquids, entrained fine solids, and accidentally transferred solids from the heel samples were combined in a single container and set aside. The composite liquids sample was initially identified as sample S11T0056714. The sample number was changed to S12R000008 following transfer to 222-S I&C.

2.3 HEEL SAMPLE SOLIDS

On May 3, 2011, the drained contents of each heel sample were emptied on a clean tray and spread in a uniform layer. Photographs were taken and observations of physical characteristics such as grain size, color, and consistency recorded (RPP-RPT-50883).

Following the direction of client representatives, small subsamples were taken from six of the eight heel solids samples and submitted for solid phase characterization (SPC) to identify the solid phases present. Powder X-ray diffraction (XRD) (ATS-LT-507-101, "222-S Laboratory

X-ray Diffractometry [XRD]") and scanning electron microscopy/energy dispersive X-ray spectrometry (SEM/EDX) (ATS-LT-161-100, "222-S Laboratory Sample Preparation and Operating Procedure for Scanning Electron Microscopes") were used. The results of the SPC measurements are presented in Section 3.1.

The heel samples were returned to their original containers until the SPC analyses were completed. After evaluating the results of the SPC measurements, the client determined that the tests, measurements, and analyses described in this report would be performed on:

a. The composite prepared from the liquids drained from the C-109 heels solids samples,
b. Large pieces of solid aggregate selected from two of the heel solids samples, and
c. A composite prepared from all the remaining C-109 heel solids.

2.4 HEEL SOLIDS AGGREGATES

The largest piece of aggregate solids was removed from each of two of the C-109 heel samples on June 14, 2011, and transferred to labeled sample bottles. The 31.8-g solid aggregate from sample C109-10-1C was assigned sample number S11T009120; the 31.5-g solid aggregate from sample C109-10-2A was assigned sample number S11T009121.

2.5 HEEL SOLIDS COMPOSITE

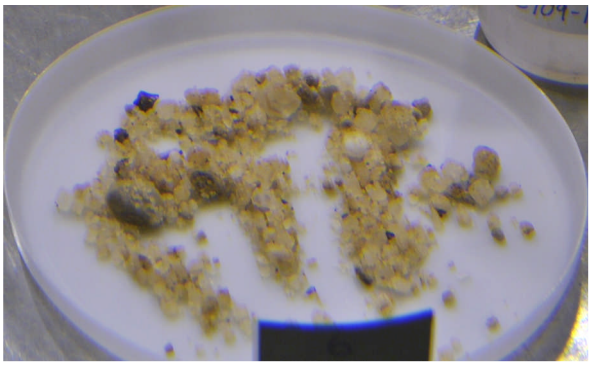
During June 14-17, 2011, the material remaining in each C-109 heel sample was emptied onto a sample tray and photographed. Representative photographs of the well-drained heel solids are presented in Figure 2-01. The material in each sample was then crushed in a mortar and pestle until all the solids just passed through a 1/4-in. sieve. All the crushed solids were transferred to a compositing container. When the crushed solids from each of the eight samples had been added to the container, the composite was mixed by stirring thoroughly with a spatula. At this point the initial crushed solids composite (sample number S11T009479) contained ≈1070 g of solids.

On June 17, ≈100 g of the crushed solids composite was transferred to a separate container. These solids were homogenized by ATL personnel in the 222-S 11A hot cells using a tissue homogenizer. The homogenized solids were assigned sample number S11T009481. A small (≈3 g) subsample of the homogenized solids (S11T009482) was collected and submitted for SPC analysis. Subsamples required for the chemical and radiochemical analyses specified in Table 4-2 of the SAP (RPP-PLAN-47927) were also collected from the homogenized solids by ATL personnel. The subsamples of "homogenized" composite solids were loaded out of the hot cells on June 20, 2011. The remaining ≈959 g of the crushed solids composite, identified as sample number S11T009480-A, was transferred to 222-S I&C personnel on June 30, 2011.

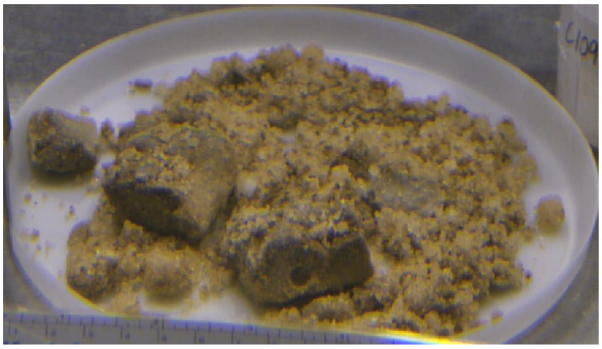
Figure 2-01. Drained Tank 241-C-109 Heel Solids Samples.

(Sample trays are 17.5 cm (6.9 in.) in diameter)

(a) C109-10-1A



(b) C109-10-1B



(c) C109-10-1C

(d) C109-10-1D

(e) C109-10-2A

(f) C109-10-2B

(g) C109-10-2C

(h) C109-10-2D

The results of the analyses of sample S11T009481 performed by ATL were presented in RPP-RPT-50883. As a result of unrepresentative sampling of composite S11T009479 and/or inadequate homogenization of sample S11T009481, the ATL analytical results did not provide an estimated composition of the heel solids of sufficient accuracy for project requirements.

On December 28, 2011, the crushed solids in composite S11T009480-A were thoroughly but gently mixed by stirring with a spatula. Subsample S11T009480, containing 450.69 g of solids, was transferred to a large glass jar, covered with a fluted watch glass, and set aside. Over the following eight days the subsample solids were periodically mixed with a spatula and allowed to dry at hot cell ambient temperature, 23-24 °C. After drying, the weight of the subsample of the crushed solids composite decreased to 446.68 g. On January 8, 2012, 50.2 g of the air-dry crushed solids were transferred to a small container and thoroughly homogenized with a tissue homogenizer by 222-S I&C personnel. These homogenized solids were identified as sample S12R000001. Two subsamples of these homogenized solids, S12R000002 and S12R000005, weighing a combined 18.7 g, were submitted to 222-S for analysis. An additional subsample of the S12R000001 homogenized solids, weighing 6.1 g, was collected for a weight loss measurement.

The results of the analyses of samples S12R000002 and S12R000005 are the basis of the estimated composition of the C-109 heel solids developed in Section 3.0 of this report. With the exceptions noted in Section 3.3.1, none of the results of the analyses of the first subsample of homogenized solids, S11T009481, were used in this test program.

The large-scale retrieval dissolution test series started on February 22, 2012. At that time, the final large-scale dissolution test sample was prepared by adding ≈5 g of homogenized solids from sample S12R000001 to the ≈396 g of air-dry, crushed solids remaining in composite subsample S11T009480. The total weight of the test sample of air-dry, C-109 heel solids at the beginning of the large-scale dissolution test series was 401.47 g. A photograph of the composite solids immediately before the start of test measurements is shown in Figure 4-01.

3.0 ANALYSIS OF TANK 241-C-109 HEEL SOLIDS

3.1 SOLID PHASE CHARACTERIZATION

Solids from six of the eight C-109 heel solids subsamples that were collected on May 3, 2011, (Section 2.3) and from the subsample (S11T009482) taken from the initial homogenized solids composite on June 17 (Section 2.5) were examined to identify the solid phases present. The detailed results of the SPC measurements were documented in a separate report prepared by 222-S I&C personnel (LAB-RPT-11-00009, *Final Report for the Initial Solid Phase Characterization of the 2011 Grab Samples and Composite for the C-109 Hard Heel Study*).

In general, the three specimens of 'stone-like' cobbles from the tank samples that were examined were found to be primarily gibbsite [$Al(OH)_3$]. Electron microscopy showed the cobbles were generally made up of euhedral to subhedral gibbsite crystals, 20-100 µm in size that were cemented primarily (but not exclusively) by additional gibbsite.

The three samples of 'bulk' solids examined consisted of fine to coarse pebble-sized particles (2-20 mm in size). These solids were found to be composed of gibbsite and/or natrophosphate [$Na_7(PO_4)_2F{\cdot}19(H_2O)$]. Sample C109-10-1A was found to be mostly natrophosphate; the solids from samples C109-10-1D and -2C were found to be mixtures of gibbsite and natrophosphate.

Subsample S11T009482 of 'homogenized' composite solids was described as a mixture of ≈84% gibbsite and ≈16% natrophosphate.

Minor to trace quantities of other solid phases were also tentatively identified by SEM/EDX. These included: uranium-rich solids [possibly sodium diuranate, $Na_2U_2O_7$,and/or clarkeite, $(Na,Ca,Pb)(UO_2)O(OH){\cdot}0\text{-}1(H_2O)$]; sodium/aluminum-rich solids [dawsonite, $NaAl(CO_3)(OH)_2$, and/or sodium aluminate, $NaAl(OH)_4$]; sodium/phosphate- and/or sodium/aluminum/ phosphate-rich solids; and a sludge-like phase with variable iron/nickel/lead-rich composition. None of these solids comprised the ≥ 5% of the solids required for definitive identification by XRD.

3.2 CRUSH TESTING

As was noted in Section 2.5, preparation of the primary C-109 heel solids composite began with reduction of the solids to <1/4-in. in size by crushing in a mortar and pestle. During this process, larger pieces of solids that represented all the visually distinct types of solids in the samples were selected, and the relative ease with which the solids were crushed was noted. This entirely qualitative test was intended to indicate whether larger pieces of the heel solids are easily broken up. The term crushed is used in absence of any compressive strength testing that would address this question more quantitatively. The crush test results are summarized as follows:

Solids Type	Crushability
A. Light tan to light gray aggregates of fine- to coarse-sand sized particles. Aggregate sizes ranging from coarse-gravel to moderate-cobbles in size.	Crushed only with great difficulty. Hammering with pestle was required to initially break the aggregates; then the maximum pressing-force possible with the hot cell manipulators was required to complete the crushing to <1/4 in.
B. Rusty brown-red aggregates of fine-grained solids. Generally coarse-gravel to small-cobble in size.	Crushed with difficulty but somewhat more easily than solid aggregates described in A.
C. Small- to medium-sized, angular, white to ivory colored particles.	Crushed easily with moderate, pressing force on the pestle.
D. Tan-to-brown aggregates of medium-grained solids with some darker colored material. Mostly <1 cm in size.	Aggregates crushed very easily (some with squeezing with large plastic tweezers).

In general, the larger pieces in the heel solids samples were very strongly cemented type A and type B aggregates. Solid phase characterization suggests that these solids were aggregates of gibbsite grains cemented, primarily, by additional gibbsite.

The type C solids, some of which were crystalline and/or monolithic solids, crushed with moderate pressure. This 'crushability' is consistent with that previously observed for natrophosphate.

The majority of the other large solids in the heel samples were mixed agglomerates of solids (type D solids) that probably formed randomly as the heel solids dried. These agglomerates were very easily crushed.

All of the solids crushed, with the exception of the type D agglomerates, were fairly uniform in color and grain composition throughout their volume.

Following the testing, all the crushed solids were added to the large-scale test composite.

3.3 PHYSICOCHEMICAL ANALYSES

3.3.1 Analysis Results

A sample of the drained liquid composite (S12R000008) and two separate samples of the re-homogenized large-scale composite (S12R000002 and S12R000005) were submitted to 222-S for chemical analyses. Transcripts of the data summary reports presenting the results of these analyses are presented in Attachment A.

The results of the physicochemical analyses of the drained liquid and large-scale composite samples are summarized in Table 3-01. Analytes for which the results of analyses of all test samples were reported as being less than the measurement detection limit have been omitted from the table. Where duplicate or repeat measurements were performed, average results are presented.

The only radiochemical analysis performed on the test composites for this study was 'Gross Alpha' for the drained liquid composite. The values presented in Table 3-02 for the heel solids are from the initial analysis of the heel solids performed by ATL (RPP-RPT-50883). The values in Table 3-02 have been corrected to an air-dry basis using moisture measurement data from the ATL report.

The concentrations of Si, Ca, Cr, Mn, Ni, and U in the heel solids composite were also taken from RPP-RPT-50883. For Si, Ca, Cr, and Mn, results of inductively coupled plasma-atomic emission spectrometry (ICP-AES) following acid digestion are reported. For Ni, the result of ICP-AES following both acid digestion and fusion in a zirconium crucible is reported. For U, the sum of the isotope concentrations from Table 3-02 is entered. Results of analyses of the samples of the test composite (S12R000002 and S12R000005) for these analytes were all reported as less than the detection limit.

The left-most entry for each analyte for each composite indicates the type of sample preparation used prior to the analytical measurement. The sample preparation codes used in all the results tables in this report follow:

	Measurement performed on unaltered portion of sample
(W)	Measurement performed on liquid specimen prepared by water leaching of a portion of sample solids
(A)	Measurement performed on liquid specimen prepared by acid digestion of a portion of sample solids
(Z)	Measurement performed on liquid specimen prepared by dissolution of the product of alkali-metal hydroxide fusion of a portion of sample solids in a zirconium crucible
(A/Z)	Reported result based on measurements on both acid digest and alkali-metal hydroxide fusion in zirconium crucible sample preparations

In Table 3-01, total inorganic carbon (TIC) analytical results are recalculated as carbonate (CO_3) by multiplying by a gravimetric factor of 4.996. Phosphorous and sulfur concentrations, determined by ICP-AES, are recalculated as phosphate (PO_4) and sulfate (SO_4) by multiplying by gravimetric factors of 3.066 and 2.996, respectively.

Adjusted total organic carbon (TOC) values, ΔTOC, were calculated by subtracting the carbon equivalents of the reported formate, acetate, and oxalate concentrations from the reported TOC concentrations:

$$[\Delta TOC] = [TOC] - [(Formate) \times 0.2668] - [(Acetate) \times 0.4068] - [(Oxalate) \times 0.2729] \qquad (3\text{-}1)$$

Table 3-01. Analyses of Liquid and Heel Solids Composites.

Analyte	Drained Liquid Composite		Heel Solids Composite (Air-dry)		
Specific gravity	1.172			—	
Density	1.188 (23.4 °C)	g/mL		2.044	g/mL
Dissolved solids (95 °C)	20.10	wt%		—	
Wt loss (95 °C)	—			18.26	wt%
TGA (< 200 °C)	76.72	wt%		17.04	wt%
TGA (> 200 °C)	< 1	wt%		20.16	wt%
H_2O (110 °C)	—			19.3	wt%
pH	—		(W)	11.8	
OH from pH				956	µg/g
TIC	12,600	µg/mL		1,755	µg/g
TIC as CO_3	62,951	µg/mL		8,768	µg/g
TOC	2,840	µg/mL		2,175	µg/g
ΔTOC	1,976	µg/mL		1,794	µg/g
Formate	384	µg/mL	(W)	122	µg/g
Acetate	397	µg/mL	(W)	111	µg/g
Oxalate	2,200	µg/mL	(W)	1,111	µg/g
Nitrite	25,000	µg/mL	(W)	4,945	µg/g
Nitrate	12,800	µg/mL	(W)	2,895	µg/g
Fluoride	892	µg/mL	(W)	10,500	µg/g
Phosphate	30,300	µg/mL	(W)	108,000	µg/g
Sulfate	2,710	µg/mL	(W)	621	µg/g
Chloride	266	µg/mL	(W)	57	µg/g
Sodium	91,200	µg/mL	(Z)	93,600	µg/g
Aluminum	< 15	µg/mL	(Z)	193,000	µg/g
Silicon	< 16	µg/mL	(A)	231	µg/g
Phosphorus	10,600	µg/mL	(Z)	30,700	µg/g
P as PO_4	32,502	µg/mL		94,132	µg/g
Sulfur	1,080	µg/mL	(Z)	< 4,540	µg/g
S as SO_4	3,236	µg/mL		< 13,602	µg/g
Potassium	359	µg/mL		—	
Calcium	< 25	µg/mL	(A)	575	µg/g
Chromium	54	µg/mL	(A)	19	µg/g
Manganese	< 2	µg/mL	(A)	9	µg/g
Iron	180	µg/mL	(Z)	1,705	µg/g
Nickel	49	µg/mL	(A/Z)	884	µg/g
Copper	10	µg/mL		—	
Zirconium	—			—	
Molybdenum	13	µg/mL		—	
Uranium	1,920	µg/mL	(Z)	982	µg/g

TGA = thermogravimetric analysis

Table 3-02. Radiochemical Analyses of Test Composites.

Analyte	Drained Liquid Composite	Heel Solids Composite Air-Dry Solids
Gross alpha	2.17E-3 µCi/mL	—
Strontium-89/90	—	(Z) 1.14E+2 µCi/g
Technetium-99	—	(A) 2.68E-1 µg/g
Iodine-129	—	< 6.51E-6 µCi/g
Cesium-137	—	(Z) 1.91E+1 µCi/g
Lead-212	—	—
Lead-214	—	—
Actinium-228	—	—
Uranium-233	—	(Z) < 1.09E-1 µg/g
Uranium-234	—	(Z) 6.03E-2 µg/g
Uranium-235	—	(Z) 6.46E+0 µg/g
Uranium-236	—	(Z) 8.07E-2 µg/g
Neptunium-237	—	(Z) 8.13E-2 µg/g
Uranium-238	—	(Z) 9.76E+2 µg/g
Plutonium-238	—	(Z) 6.79E-4 µCi/g
Plutonium-239/240	—	(Z) 1.86E-2 µCi/g
Americium-241	—	(Z) 1.03E-2 µCi/g
Curium-243/244	—	—

3.3.2 Liquid Density Measurements

'Specific gravity' values reported for liquid samples are the results of density measurements performed at 222-S by ATL. The reference water temperature was assigned a value of 4 °C for convenience; the temperature of the test liquid was not reported.

All density values reported for liquid samples (D_{Liq}) in this report are the results of gravimetric measurements performed in the 11A hot cells. For each measurement, two of three Class-A, 25-mL volumetric flasks were filled with test liquid and weighed. The temperature of the test liquid in each flask was measured immediately after the weight was recorded. The D_{Liq} in each flask was then calculated using the nominal 25-mL flask volumes (See Section 8.2.4). The average D_{Liq} and liquid temperature of the two measurements are reported here.

3.3.3 Water Content Measurements

'Dissolved solids' and 'Wt loss' values are the results of gravimetric measurements performed in the 11A hot cells. A portion of a test sample was transferred to a sample vial, and then dried to constant weight in a small oven at 90 to 100 °C. Several grams of sample were used in each measurement.

'TGA' values are the results of thermogravimetric analyses (TGA) performed by ATL personnel. The weight loss measured by TGA, usually but not always at temperatures <200 °C, is commonly reported as 'Percent Water'. For the drained liquid composite, the specimen sizes were ≈14 mg, and the observed weight loss was essentially complete at 110 °C.

The TGA measurements for the heel solids composite samples used ≈30 mg samples. The solids also showed an initial weight loss that was essentially complete at 110 °C (reported as TGA [<200 °C]); however, additional weight loss began at ≈200 °C and was nearly complete at 310 °C (reported as TGA [>200 °C]). The initial weight loss is probably dominated by loss of the 19 waters of hydration from natrophosphate [$Na_7F(PO_4)_2 \cdot 19(H_2O)$]. The second loss of weight can be attributed, in part, to the decomposition of gibbsite:

$$2\ Al(OH)_3 \rightarrow 2\ AlOOH + 2\ H_2O(\uparrow) \rightarrow Al_2O_3 + H_2O(\uparrow) \qquad (3\text{-}2)$$

'H_2O' values are the results of water content analyses using the Computrac VaporPro Moisture Analyzer in room 1D of 222-S (ATS-LT-564-101, "222-S Laboratory Percent Water Content Analysis Using the Computrac Vapor Pro Mositure Analyzer"). This instrument directly measures the weight of water vapor evolved as a sample is heated in a stream of dry air. All reported measurements in this report were completed at a temperature of 110 °C. Specimen sizes for the test composite ranged from 630 to 780 mg.

3.3.4 Anomalous Results

The calculated ΔTOC values (Section 3.3.1) for the drained liquid and the heel solids composites are 70% and 82%, respectively, of the total TOC measured in the composites. The relative magnitude of contributions of water-soluble but unanalyzed and/or water-insoluble organic components of the C-109 wastes to the ΔTOC values is unknown. During the retrieval dissolution tests, many samples formed foam layers when vigorously agitated or formed bubbles when expelled from a pipette. In the same samples, small amounts of very fine-grained solids would float in a thin layer at the surface of liquid samples even after extended periods of centrifugation. These observations suggest that a small quantity of some organic liquid may have been associated with the heel solids samples.

The stoichiometric ratio of phosphate to fluoride in natrophosphate is 10:1 w/w. Ion chromatography (IC) results for water soluble anions in the heel solids composite indicate the presence of a 2.9% excess of phosphate over that required to combine with the reported fluoride to form natrophosphate. However, phosphorus analysis by ICP-AES following fusion of the heel solids indicates a 10.3% deficit in the amount of phosphorus needed to combine with fluoride to form natrophosphate and a 12.8% shortfall in the phosphorus required to form the reported quantity of phosphate. The presence of a minor, non-natrophosphate phosphate component in the heel solids is possible but is not directly supported by the chemical analyses.

3.4 ESTIMATED SOLIDS COMPOSITION

Based on the SPC results (Section 3.1) and the analytical data in Table 3-01, an estimate of the solid phase composition of the C-109 heel solids composite can be derived. One version of such an estimate is shown in Table 3-03. All the aluminum found in the heel solids is assigned to gibbsite. The amount of natrophosphate present is estimated by multiplying the reported fluoride concentration by the gravimetric factor 37.49. All TIC in the heel solids is assumed to have been in the form of a carbonate species.

Table 3-03. Estimated Solid Phase Composition of Heel Solids Composite.

C-109 Heel Solids Composite (Air-dry)			
Analyte		**Calculated As**	
Aluminum	193,000 µg/g	$Al(OH)_3$	55.80 wt%
Fluoride	10,500 µg/g	$Na_7F(PO_4)_2 \cdot 19\ H_2O$	39.36 wt%
TIC as CO_3	8,768 µg/g	$Na_2CO_3 \cdot H_2O$	1.81 wt%
Nitrite	4,945 µg/g	$NaNO_2$	0.74 wt%
Nitrate	2,895 µg/g	$NaNO_3$	0.40 wt%
Iron	1,705 µg/g	Fe_2O_3	0.24 wt%
Hydroxide	956 µg/g	NaOH	0.22 wt%
ΔTOC	1,794 µg/g	Organic C	0.18 wt%
Oxalate	1,111 µg/g	Na-oxalate	0.17 wt%
Uranium	982 µg/g	$Na_2U_2O_7$	0.13 wt%
Nickel	884 µg/g	NiO	0.11 wt%
Sulfate	621 µg/g	Na_2SO_4	0.09 wt%
Calcium	575 µg/g	CaO	0.08 wt%
Silicon	231 µg/g	SiO_2	0.05 wt%
Formate	122 µg/g	Na-formate	0.02 wt%
Acetate	111 µg/g	Na-acetate	0.02 wt%
Chloride	57 µg/g	NaCl	0.01 wt%
		Total	99.44 wt%
		H_2O from solids	19.2 wt%
		Wt loss (95 °C)	18.3 wt%
		H_2O (110 °C)	19.3 wt%
		Total Sodium Calculated	10.16 wt%
		Total Sodium Analyzed	9.36 wt%

The calculated 19.2 wt% of water associated with the proposed heel solids is within the 18.3-19.3 wt% range of the two water content measurements. Over 98% of this water is associated with the natrophosphate solids. As fluoride is converted to natrophosphate and then to hydration water, any error in the initial fluoride concentration results in an error in hydration water concentration that is ≈18 times larger. Given this potential error multiplier and the ease with which natrophosphate begins to lose hydration water even at near-ambient temperatures, the

degree of agreement between the measured and calculated water content of the air-dry solids is considered to be quite good.

The calculated total sodium content associated with the proposed solid phase composition, 10.16 wt%, is 8.5% greater than the reported analytical result of 9.36 wt%. As an error of ±20% is considered to be acceptable for sodium analysis by ICP-AES on fusion preparations, this difference does not indicate that the proposed solid phase composition is unreasonable. The initial analysis of the damp heel solids composite by ATL (RPP-RPT-50883) indicated that the potential natrophosphate content was slightly less than that indicated in Table 3-03. If so, the difference between the calculated and analyzed sodium inventories would decrease.

As noted in Section 2.4, two relatively large pieces of aggregate solids weighing 63.3 g were removed from the C-109 heel solids samples before the 1070-g crushed solids composite was prepared. Analysis by XRD and SEM/EDX indicated that similar pieces of aggregate solids were predominantly gibbsite (Section 3.1). If 63.3 g of gibbsite is added to the solid phase composition in Table 3-03, the estimated gibbsite concentration in the as-received heel solids samples increases to

$$\left\{\frac{[(55.80\ wt\%)(1070\ g)] + 63.3\ g}{1070\ g + 63.3\ g}\right\} \times 100 = 58.3\ wt\% \tag{3-3}$$

and the estimated natrophosphate concentration decreases to

$$\left[\frac{(39.36\ wt\%)(1070\ g)}{1070\ g + 63.3\ g}\right] \times 100 = 37.2\ wt\% \tag{3-4}$$

3.5 BULK DENSITY

3.5.1 Bulk Density Measurement

The bulk density (D_{Bulk}) of the C-109 heel solids composite was measured by water displacement. In this report, D_{Bulk} is the overall density of a sample consisting of subdivided solids, any interstitial and/or adsorbed liquids, and any void spaces not penetrated by the added water.

A special lid was fabricated for a wide-mouthed, 16-oz sample jar (Figure 4-01) that allowed it to be consistently filled with liquid to a known volume. This effectively converted the sample jar into a pycnometer. The calibrated volume of the pycnometer jar was 485.06 ± 0.17 mL (Section 8.2.4).

The D_{Bulk} measurement was performed as part of the first water contact in the large-scale dissolution test series. At 08:00 on February 22, 2012, the entire 401.47 g test sample of air-dry, crushed composite solids was transferred to the pycnometer jar. The jar+lid+solids were then weighed. Portions of cold reagent water, taken from an ice-water mixture, were added to the jar until the solids were completely submerged. As the water was added, the solids and added water were periodically mixed gently with a spatula to ensure that significant volumes of air were not trapped in the solids. The pycnometer lid was then firmly attached and filling to the calibration

mark was completed. The final weighing of the filled pycnometer was completed by 09:00. During the D_{Bulk} measurement the hot cell ambient temperature ranged from 22.8-22.9 °C. During the final stages of filling, the liquid temperature in the pycnometer jar ranged from 11.8-14.0 °C.

3.5.2 Heel Solids Bulk Density

The weight of the cold reagent water added to the heel solids composite was converted to a volume using the reference density of water at 12.9 °C, the average temperature of the liquid in the pycnometer jar during the final filling. The volume of the composite solids was then calculated as the difference between the pycnometer jar volume and the volume of the added water. The data recorded during the measurement and the calculation of D_{Bulk} follow.

Wt pycnometer	339.79	±	0.03	g
Wt (pycnometer + composite)	741.26	±	0.07	g
Wt composite solids	401.47	±	0.08	g
Wt (pycnometer + composite + H_2O)	1029.74	±	0.10	g
Wt H_2O	288.48	±	0.13	g
Reference density of H_2O at 11.8 °C	0.999529			g/mL
at 14.0 °C	0.999244			g/mL
Calculation H_2O density at 12.9 °C	0.999389			g/mL
Vol H_2O	288.66	±	0.13	mL
Vol pycnometer	485.06	±	0.17	mL
Vol composite solids	196.40	±	0.21	mL
D_{Bulk} of air-dry, crushed C-109 heel solids composite	2.044	±	0.003	g/mL

The measured D_{Bulk} of the crushed solids composite can be compared to the value predicted for the solid phase composition of the C-109 heel solids composite presented in Table 3-03. The partial weight of each proposed solid phase in the composite is first calculated using the wt% concentration data in Table 3-03. Using the reference density for each solid phase, the partial volume occupied by each phase is then calculated. After summing the partial weights and partial volumes, the D_{Bulk} is calculated. This calculation is presented in Table 3-04.

The calculated D_{Bulk} for the proposed solid phase composition is ≈2% greater than the measured D_{Bulk} of the crushed heel solids composite. Though this degree of agreement may be somewhat fortuitous, it does suggest that the proposed solid phase composition is not unreasonable.

Table 3-04. Calculated Bulk Density of Estimated Composite Composition.

Solid Phase	Wt%	Wt of Phase	Phase Density	Vol of Phase
$Al(OH)_3$	55.80 wt%	224.00 g	2.42 g/mL [a]	92.56 mL
$Na_7F(PO_4)_2 \cdot 19\ H_2O$	39.36 wt%	158.02 g	1.71 g/mL [b]	92.41 mL
$Na_2CO_3 \cdot H_2O$	1.81 wt%	7.27 g	2.25 g/mL [a]	3.23 mL
$NaNO_2$	0.74 wt%	2.98 g	2.17 g/mL [a]	1.37 mL
$NaNO_3$	0.40 wt%	1.59 g	2.26 g/mL [a]	0.70 mL
Fe_2O_3	0.24 wt%	0.98 g	5.24 g/mL [a]	0.19 mL
NaOH	0.22 wt%	0.90 g	2.13 g/mL [a]	0.42 mL
$Na_2U_2O_7$	0.13 wt%	0.53 g	5.6 g/mL [c]	0.09 mL
NiO	0.11 wt%	0.45 g	6.67 g/mL [a]	0.07 mL
Na-oxalate	0.17 wt%	0.68 g	2.34 g/mL [a]	0.29 mL
Na_2SO_4	0.09 wt%	0.37 g	2.68 g/mL [a]	0.14 mL
CaO	0.08 wt%	0.32 g	3.25 g/mL [a]	0.10 mL
SiO_2	0.05 wt%	0.20 g	2.64 g/mL [a]	0.08 mL
Na-formate	0.02 wt%	0.07 g	1.92 g/mL [a]	0.04 mL
Na-Acetate	0.02 wt%	0.06 g	1.53 g/mL [a]	0.04 mL
NaCl	0.01 wt%	0.04 g	2.16 g/mL [a]	0.02 mL
Calculated: Total	99.44 wt%	399.21 g	—	191.76 mL
D_{Bulk}	—	—	2.082 g/mL	—
Measured: Total	—	401.47 g	—	196.40 mL
D_{Bulk}	—	—	2.044 g/mL	—

[a] Phase densities from *CRC Handbook of Chemistry and Physics*, 65th Edition.
[b] Phase density from *Handbook of Mineralogy*, http://www.handbookofmineralogy.org.
[c] Phase density from Table 4-4 of RPP-RPT-51652, *One System Evaluation of Waste Transferred to the Waste Treatment Plant*.

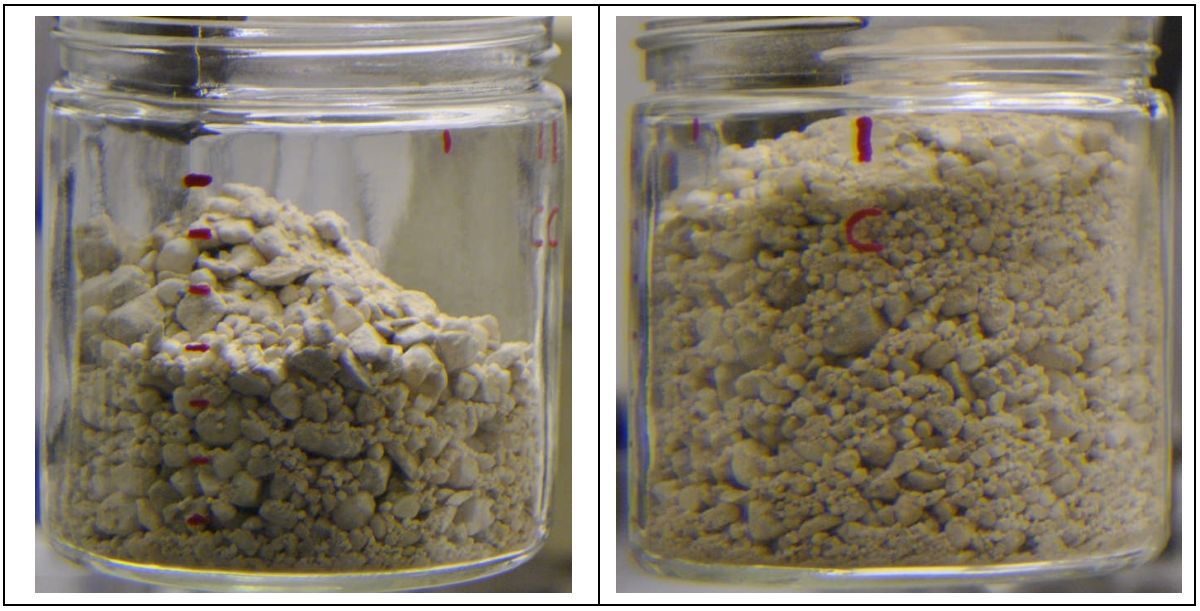
4.0 LARGE-SCALE RETRIEVAL TESTING

4.1 LARGE-SCALE TEST SAMPLE

The test sample of C-109 air-dry, crushed heel solids passed directly from the D_{Bulk} measurement (Section 3.5.1) to the large-scale dissolution test series. The total weight of the crushed solids test sample at the beginning of the D_{Bulk} measurement was 401.47 g. Photographs of the large-scale test sample as it was loaded into the pycnometer jar for the D_{Bulk} measurement are shown in Figure 4-01.

Figure 4-01. Heel Solids Composite Test Sample.

(Outside diameter of jar is 9.0 cm)



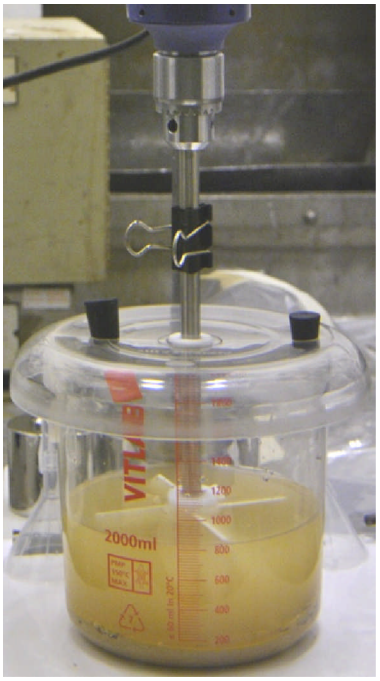
4.2 APPARATUS

A photograph of the test apparatus (at the beginning of Contact #1 of the water dissolution test sequence) is shown in Figure 4-02.

The primary vessel used for the large-scale retrieval dissolution tests was fabricated from a 2-L polymethylpentene (PMP) beaker. PMP is transparent, stable at temperatures up to 130 °C, and highly resistant to a wide range of liquids including highly concentrated caustic solutions. The top of the beaker was removed to create a cylindrical vessel 15.5 cm in height with an inner diameter increasing from 12.6 cm near the bottom to 13.2 cm at the top.

A cover for the vessel was fabricated by cutting the bottom from a 5-L PMP beaker. Three holes were drilled in the cover to allow insertion of a stirrer and a thermometer and to allow periodic withdrawal of small samples of test liquids.

Figure 4-02. Large-scale Dissolution Testing Apparatus.



A four-bladed, fluoro-polymer impeller was affixed to a 10-mm diameter stainless steel shaft that passed through a central 12-mm hole in the test vessel cover. A 25-mm diameter Teflon®[1] washer that slid on the stirrer shaft with minimal clearance rested on the top of the cover and acted as a partial vapor seal. The diameter of the impeller was 10.0 cm. The flat blades were pitched at 60° generating a downward flow of liquid. In operation, the impeller was consistently lowered until the top of the blades were just submerged in the test liquid, which placed the bottom of the blades at a depth of 16 mm. The bottom of the impeller was at least 2 cm above the surface of the test solids at all times.

A low-torque laboratory stirrer motor was used to drive the impeller. The stirrer was operated at the slowest speed that could be set with the manual control on the motor. The speed ranged from 27 to 36 rpm and was typically 31-34 rpm. At this speed, very fine solids were mobilized and maintained in suspension in the test liquids. The bulk of the test solids remained in the bottom of the test vessel throughout each test sequence.

4.3 WATER DISSOLUTION

4.3.1 Test Narrative

Chemical and SPC analysis of the test composite indicated the presence of 39.4 wt% of natrophosphate, a double salt with low water solubility, as well as small quantities of other

[1] Teflon is a registered trademark of I. E. du Pont de Nemours and Company, Wilmington, Delaware.

moderately to highly soluble sodium salts (Section 3.4). A water dissolution test was performed to examine the efficacy with which these solids can be dissolved and removed from tank C-109.

Each 'Contact' in the water dissolution test began with the addition of a volume of water equal to approximately three times the starting volume of the test composite: 3 × 196.4 mL = 589.2 mL (or 587.5 g of water at 25 °C). Slow stirring at hot cell ambient temperatures was then continued until periodic D_{Liq} measurements indicated the contact liquid was saturated.

The D_{Liq} measurements were performed in the same manner throughout the dissolution test series. Stirring in the test vessel was stopped, and the test sample was allowed to settle for approximately 30 min. Approximately 50 mL of the test liquid was then transferred to a centrifuge cone and centrifuged for 10-15 min. Two 25-mL volumetric flasks were then filled with the centrifuged liquid (C-Liq) and weighed. The temperature of the liquid in each flask was measured immediately after weighing. The D_{Liq} for each measurement and the average of the two were then calculated (Sections 3.3.2 and 8.2.4). The liquid and any solids in the centrifuge cone (C-Slds) were then returned to the test vessel. For each D_{Liq} measurement, an average of ≈0.45 g of liquid was lost and not returned to the test vessel.

4.3.1.1 Water Contact #1: All the composite solids and liquid in the pycnometer jar at the end of the D_{Bulk} measurement (Section 3.5) were transferred to the dissolution test vessel. During the D_{Bulk} measurement, 288.48 g of water were added to the test solids. An additional 291.34 g of water were added at the start of Contact #1 for a total of 579.82 g or 581.54 mL at 25 °C. The ratio of the volume of water added in Contact #1 to the initial volume of the composite solids was 2.96.

After stirring for 3 hrs, D_{Liq} was 1.057 g/mL at 22.9 °C.

Stirring in the test sample was resumed and continued for an additional 19 hrs (22 hrs total). The D_{Liq} increased to 1.063 g/mL at 23.4 °C.

Stirring in the test sample was resumed and continued for an additional 7 hrs (29 hrs total). The D_{Liq} was unchanged at 1.064 g/mL at 24.3 °C.

After recovering the final D_{Liq} specimens, stirring was not resumed, and the contents of the test vessel settled overnight. At the beginning of the next work shift, 48 hrs after the start of Contact #1, removal of the supernatant liquid in the test vessel began using a large volume pipette. In pairs, four 50-mL centrifuge cones were repeatedly filled with liquid from the test vessel and centrifuged for 15 min at ≈1000 × *g*. All the portions of clear supernatant C-Liq were decanted to a single holding container. A loss of ≈1 mL of liquid during the decanting and liquid transfers was assumed.

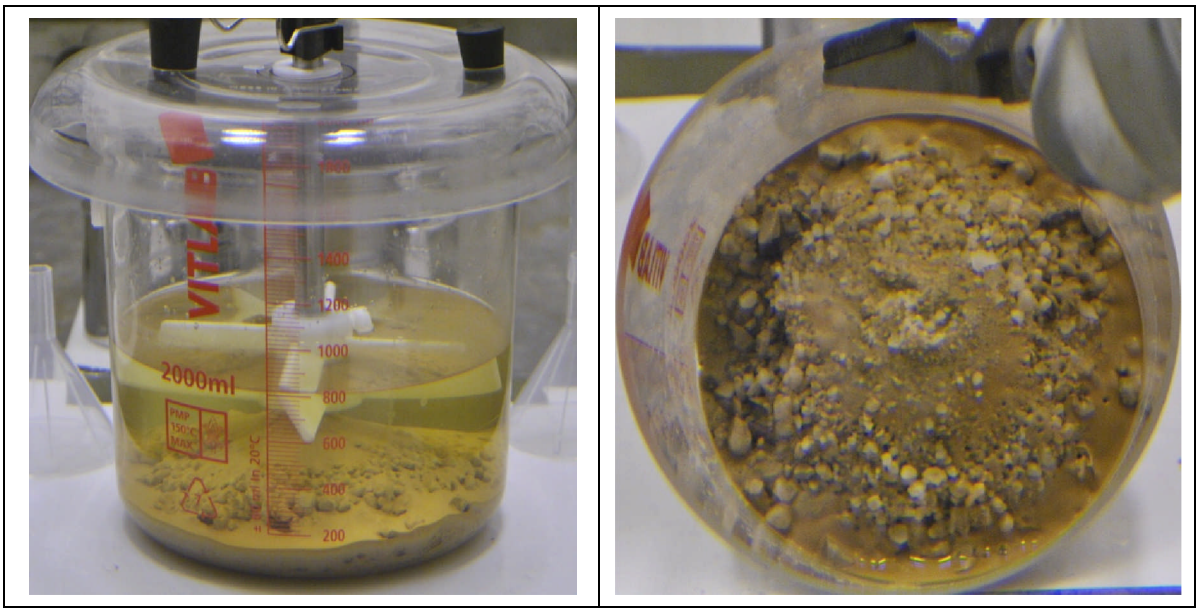
A total of 575.60 g of liquid was decanted from the test vessel. The D_{Liq} of the decanted Contact #1 liquor was 1.064 g/mL at 25.6 °C. The temperature of the liquid in the test vessel ranged from 22.9-25.2 °C during Contact #1. The liquid temperature when the supernatant decant was started was 25.2 °C.

A sample of the decanted Contact #1 liquid (S12R000149) was submitted to 222-S for analysis. Prior to load-out from the 11A hot cells, the sample was diluted 24.93% w/w with deionized water: [(Wt Sample Liquid + Wt H_2O) / Wt Sample Liquid = 1.2493].

The wt% dissolved solids in the decanted liquid was measured by evaporating a 5.496 g portion to dryness at 90-100 °C. The decanted Contact #1 liquor contained 6.60 wt% dissolved solids.

The total weight of the wet heel solids remaining in the test vessel and the damp final C-Slds in the four centrifuge cones at the conclusion of Contact #1 of the large-scale water dissolution test was 402.40 g. Photographs of the contents of the test vessel are shown in Figure 4-03.

Figure 4-03. Heel Solids at Conclusion of Water Dissolution Contact #1.



4.3.1.2 H_2O Contact #2: For Contact #2 of the large-scale water dissolution test, 587.52 g of deionized water (589.26 mL at 25 °C) was added to the wet solids remaining after Contact #1. The final Contact #1 C-Slds were recovered and returned to the test vessel as the Contact #2 water was added. The ratio of the volume of water added in Contact #2 to the initial volume of the composite solids was 3.00.

After stirring for 6 hrs, D_{Liq} was 1.032 g/mL at 25.6 °C.

Stirring in the test sample was resumed and continued for an additional 16.5 hrs (22.5 hrs total). The D_{Liq} increased to 1.053 g/mL at 25.0 °C.

Stirring in the test sample was resumed and continued for an additional 7 hrs (29.5 hrs total). The D_{Liq} increased slightly to 1.056 g/mL at 24.6 °C.

Stirring in the test sample was resumed and continued for an additional 17 hrs (46.5 hrs total). The measured D_{Liq} was unchanged at 1.056 g/mL at 24.2 °C. The Contact #2 supernatant liquid

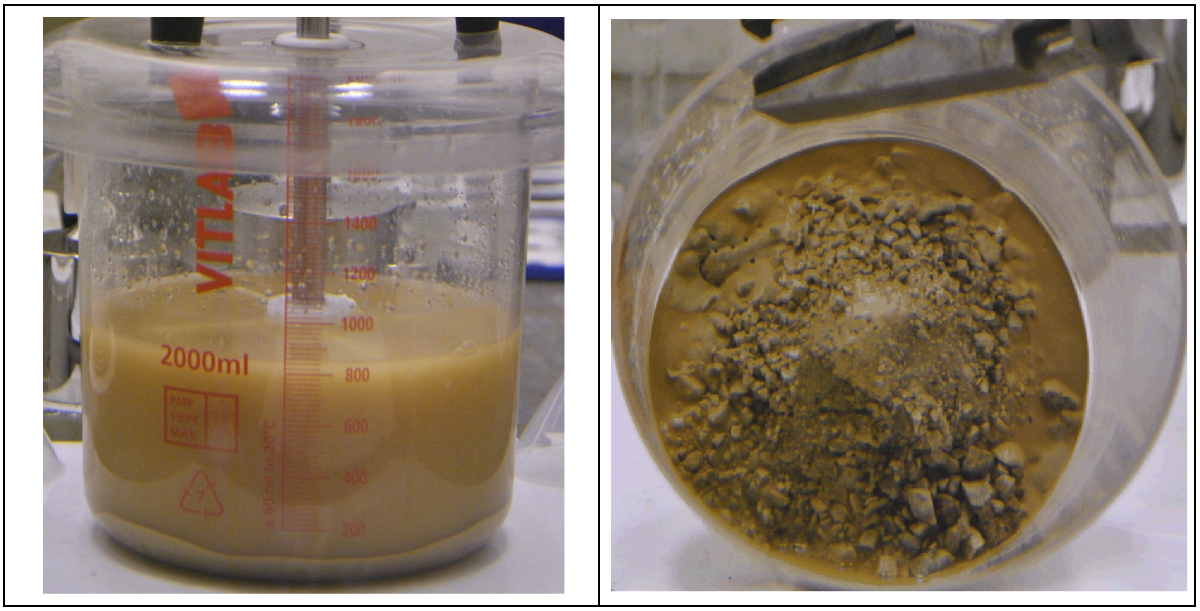
was then decanted, centrifuged, and transferred to a holding container. A total of 661.76 g of liquid was decanted from the test vessel. The temperature of the liquid in the test vessel ranged from 25.3-26.1 °C during Contact #2. The liquid temperature when the supernatant decant was started was 25.3 °C.

A sample of the decanted Contact #2 liquid (S12R000150) was submitted to 222-S for analysis. Prior to load-out from the 11A hot cells, the sample was diluted 25.10% w/w with deionized water.

The wt% dissolved solids was measured by evaporating 5.989 g of the decanted liquid to dryness at 90-100 °C. The decanted Contact #2 liquid contained 5.89 wt% dissolved solids.

The total weight of wet heel solids and damp C-Slds remaining at the conclusion of Contact #2 of the large-scale water dissolution test was 324.36 g. Photographs of the contents of the test vessel at the conclusion of Contact #2 are shown in Figure 4-04.

Figure 4-04. Heel Solids at Conclusion of Water Dissolution Contact #2.



4.3.1.3 Water Contact #3: For Contact #3 of the large-scale water dissolution test, 589.89 g of deionized water (591.64 mL at 25 °C) was added to the wet solids remaining after Contact #2. The final Contact #2 C-Slds were recovered and returned to the test vessel as the Contact #3 water was added. The ratio of the volume of water added in Contact #3 to the initial volume of the composite solids was 3.01.

After stirring for 16 hrs, D_{Liq} was 1.012 g/mL at 24.2 °C.

Stirring in the test sample was resumed and continued for an additional 8 hrs (24 hrs total). The D_{Liq} was unchanged at 1.010 g/mL at 24.1 °C. After recovery of the D_{Liq} specimen, stirring was not resumed. The test sample was left undisturbed over a 3-day weekend.

At the beginning of the following workshift, stirring in the test sample was resumed and continued for an additional 1 hr (25 hrs total). The Contact #3 liquid was then allowed to settle for 2 hrs, decanted, centrifuged, and transferred to a holding container.

A total of 610.84 g of liquid was decanted from the test vessel. The final D_{Liq} of the decanted Contact #3 liquid was 1.014 g/mL at 24.6 °C. The temperature of the liquid in the test vessel ranged from 24.7-25.0 °C during Contact #3. The liquid temperature when the supernatant decant was started was 24.7 °C.

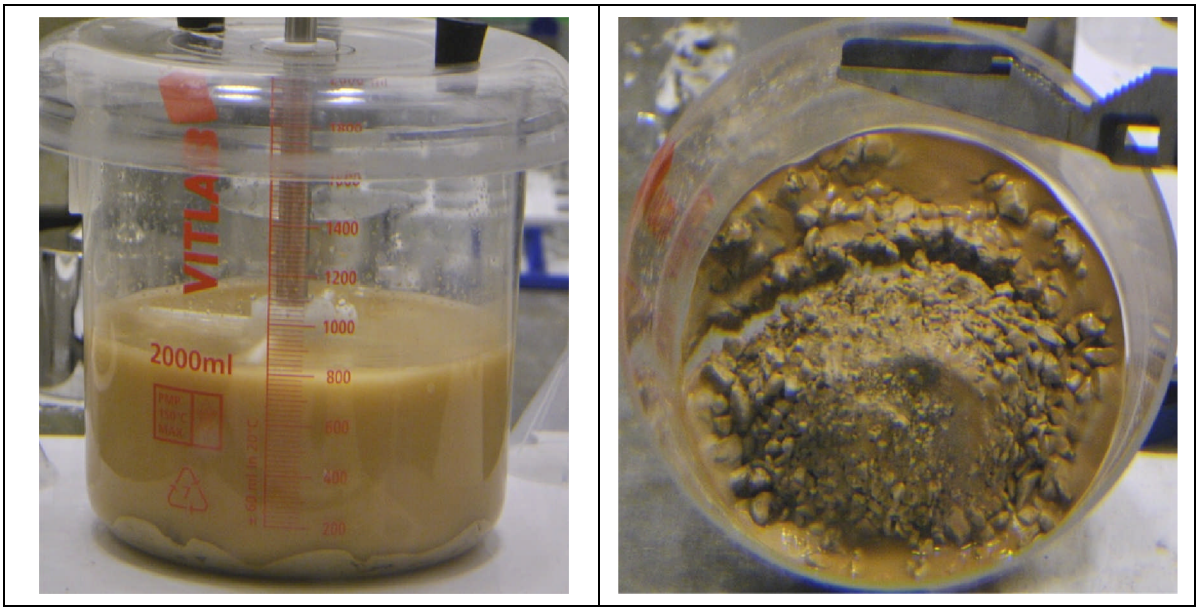
A sample of the decanted Contact #3 liquid (S12R000151) was submitted to 222-S for analysis. Prior to load-out from the 11A hot cells, the sample was diluted 24.83% w/w with deionized water.

The wt% dissolved solids was measured by evaporating 5.948 g of the decanted liquid to dryness at 90-100 °C. The decanted liquid contained 1.92 wt% dissolved solids.

The total weight of wet heel solids remaining at the conclusion of Contact #3 of the large-scale water dissolution test was 304.54 g. Photographs of the contents of the test vessel at the conclusion of Contact #3 are shown in Figure 4-05. After air-drying at hot cell ambient temperatures for 68 hrs, the total weight of the damp solids remaining after the water dissolution test series decreased to 272.11 g.

The decrease in the D_{Liq} and wt% dissolved solids values for the final Contact #3 liquid, relative to those of the first two contacts, indicated that addition of more water would not dissolve and remove a significant additional quantity of water soluble species from the test sample. The large-scale water dissolution test was, therefore, terminated after Contact #3.

Figure 4-05. Heel Solids at Conclusion of Water Dissolution Contact #3.



4.3.2 Sample Analyses

The results of physicochemical analysis of the liquids decanted from the test vessel at the conclusion of each contact in the large-scale water dissolution test are summarized in Table 4-01. The analytical results in the table have been corrected to predilution values. Transcripts of the laboratory data summary reports for the diluted samples and the sample weight and density data used for the dilution corrections are in Attachment A.

Liquid density and dissolved solids measurements were performed in the 11A hot cells by 222-S I&C personnel. All other analyses were performed in 222-S by ATL personnel. The only radiochemical analysis requested for retrieval testing liquid samples was ‘Gross alpha’.

Table 4-01. Analyses of Large-scale Water Dissolution Test Liquids.

Analyte	Units	Large-scale Water Dissolution Test Liquids		
		Contact #1	Contact #2	Contact #3
D_{Liq}	g/mL	1.064 (25.6 °C)	1.056 (24.2 °C)	1.014 (24.6 °C)
Dissolved solids (95 °C)	wt%	6.60	5.89	1.92
TGA (< 200 °C)	wt%	90.47	91.24	98.88
TGA (> 200 °C)		< 1	< 1	< 1
TIC	µg/mL	990	195	101
TIC as CO_3		4,947	974	503
TOC	µg/mL	217	27.3	112
ΔTOC	µg/mL	56.4	0	104
Formate	µg/mL	82	15	4
Acetate	µg/mL	75	27	< 1
Oxalate	µg/mL	395	96	24
Nitrite	µg/mL	2,722	392	52
Nitrate	µg/mL	1,456	216	34
Fluoride	µg/mL	2,887	2,962	933
Phosphate	µg/mL	31,274	30,634	9,744
Sulfate	µg/mL	301	53	11
Chloride	µg/mL	24	5	< 1
Sodium	µg/mL	24,310	21,266	6,512
Aluminum	µg/mL	115	104	65
Silicon	µg/mL	7	8	7
Phosphorus	µg/mL	9,990	9,988	2,943
P as PO_4		30,631	30,624	9,024
Sulfur	µg/mL	108	21	< 13
S as SO_4		324	63	< 38
Potassium	µg/mL	67	< 63	< 63
Calcium	µg/mL	< 6	< 6	< 6
Chromium	µg/mL	5	1	< 1
Manganese	µg/mL	< 1	< 1	< 1
Iron	µg/mL	15	3	< 1
Nickel	µg/mL	< 3	< 3	< 3
Uranium	µg/mL	366	55	< 13
Gross alpha	µCi/mL	3.23E-4	8.16E-5	1.25E-5

4.4 CAUSTIC DISSOLUTION

4.4.1 Test Narrative

Chemical analyses of the test composite used in this study (Section 3.3) indicated that the air-dry, crushed heel solids contained ≈19.3 wt% Al. A caustic dissolution test was performed to examine the degree to which the Al-bearing solids, which represented a major component of the test composite, can be dissolved and removed from C-109.

The only significant Al-bearing species identified in the composite solids by SPC was gibbsite [$Al(OH)_3$] (Section 3.1). If all Al found in the test sample was associated with gibbsite, the composite solids contained ≈55.8 wt% gibbsite[2]. Post-test processing of the results of chemical analyses of the liquids removed from the test vessel during the three-step water dissolution test (Sections 4.3 and 4.6) indicated that only ≈0.2 wt% of the Al in the composite solids was removed. For these reasons, the caustic dissolution test was planned as if the initial test composite sample contained 55.8 wt% gibbsite and that none of this gibbsite was dissolved and removed during the water dissolution test.

The caustic dissolution test was performed in two steps: metathesis of gibbsite to sodium aluminate,

$$Al(OH)_3(s) + n(Na^+ \,\&\, OH^-) + xH_2O \rightarrow NaAl(OH)_4(s) + (n\text{-}1)(Na^+ \,\&\, OH^-) + xH_2O \qquad (4\text{-}1)$$

followed by dissolution and recovery of the sodium aluminate,

$$NaAl(OH)_4(s) + (n\text{-}1)(Na^+ \,\&\, OH^-) + (x+y)H_2O \rightarrow Al(OH)_4^- + nNa^+ + (n\text{-}1)OH^- + (x+y)H_2O \qquad (4\text{-}2)$$

The caustic dissolution testing was performed at hot cell ambient temperatures.

An excess of NaOH was added to drive the gibbsite metathesis step to completion more quickly. As specified by the client, the test target was to add sufficient 50% caustic solution to provide 3 moles of OH for each mole of Al present in the test composite sample.

Solubility within the Na_2O-Al_2O_3-H_2O system at varying NaOH concentrations has been extensively studied (e.g., ARH-ST-133, *Vapor-Liquid-Solid Phase Equilibria of Radioactive Sodium Salt Wastes at Hanford*). The often-cited 'Barney Diagram' is reproduced in Figure 4-06. In the tests described in this report, the target volume of water to be added in the sodium aluminate dissolution step was 102% of the volume of caustic solution added in the metathesis step. If (a) the estimated quantity of gibbsite was present, (b) the 3:1 mole ratio of OH:Al was achieved, and (c) the metathesis reaction was 100% complete, test projections indicated that this volume of water would bring the NaOH concentration just below the level where solids solubility is maximized. In Figure 4-06, this would be just below the junction of the $Al(OH)_3$ and $NaAl(OH)_4$ solubility curves at ≈5.9 M NaOH. At greater NaOH concentrations, dissolution of sodium aluminate is inhibited. At smaller NaOH concentrations, gibbsite can precipitate from solution, albeit quite slowly.

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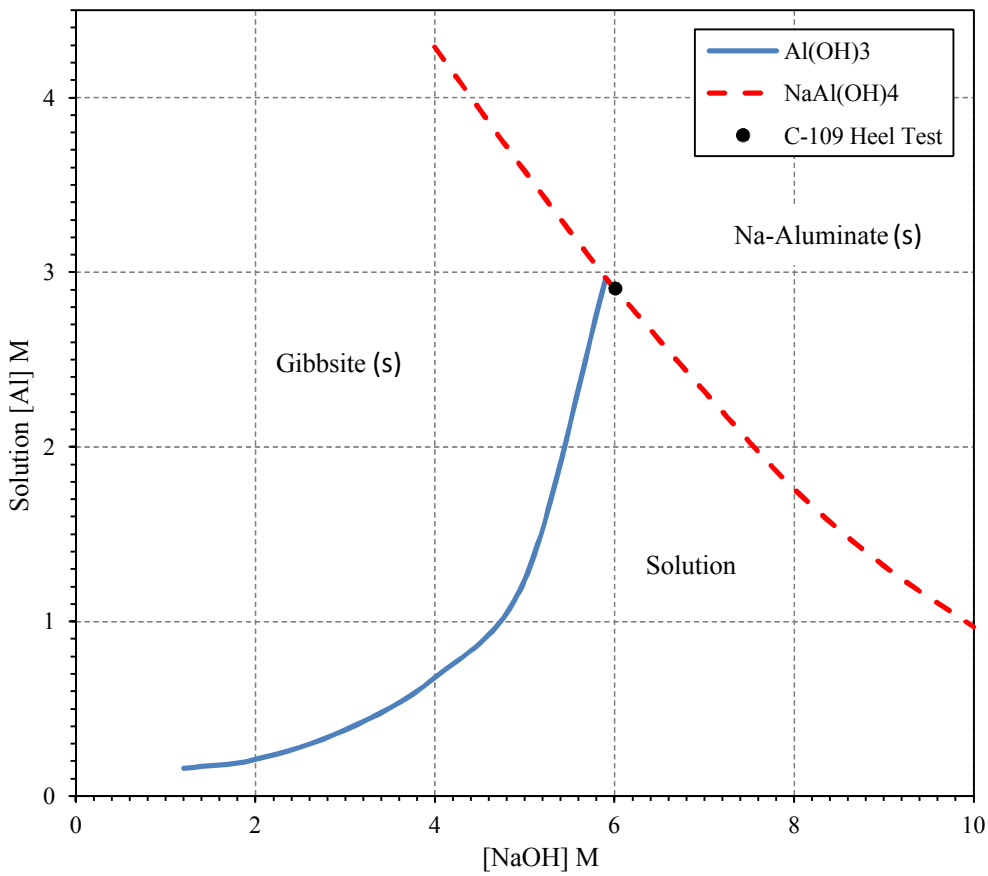
[2] Analyses of the initial composite samples by ATL returned Al concentrations ranging from 15.1-26.6 wt% with an average of 19.7 wt% Al (RPP-RPT-50883). If all Al in the analyzed samples was associated with gibbsite, the initial ATL results indicated that the composite solids contained 43.7-76.9 wt% gibbsite with an average of 56.9 wt%.

Figure 4-06. Solubility in the Na_2O-Al_2O_3-H_2O System at 30 °C.



4.4.1.1 Caustic Preparation: The preparation of the nominal 50% caustic solution used in the caustic dissolution tests is described in Attachment B. The D_{Liq} of the prepared solution was measured as 1.5212 ± 0.0007 g/mL at 22.3 °C. Based on the measured density, the solution was calculated to be 49.644 wt% and 18.875 M NaOH.

4.4.1.2 Gibbsite Metathesis: The test vessel containing the wet solids remaining after completion of the large-scale water dissolution testing passed directly to the caustic dissolution test.

As previously noted, the heel solids were found to contain ≈19.3 wt% Al and assumed to contain 55.8 wt% gibbsite. At these concentrations, the initial 401.47 g of air-dry heel solids composite contained 77.48 g or 2.872 moles of Al (equivalent to 224.00 g or 92.56 mL of gibbsite).

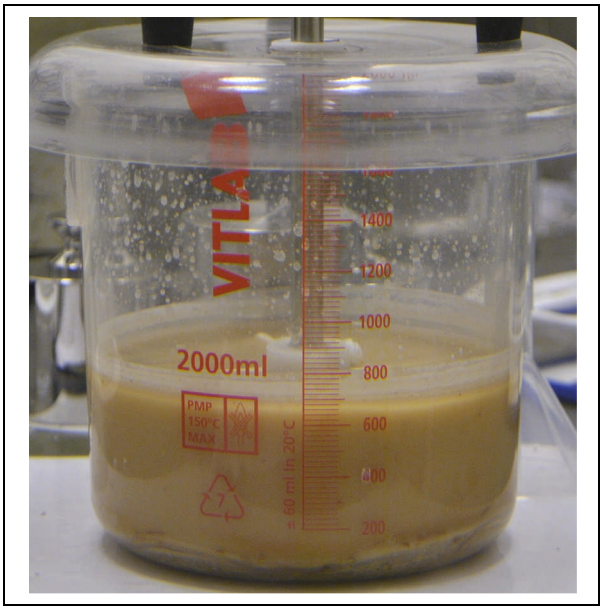
At the beginning of the work shift on March 7, 2012, 706.89 g (≈467 mL at 29 °C) of caustic solution was added to the damp heel solids in the test vessel. This quantity of the caustic solution contained 350.93 g of NaOH providing 8.774 moles of OH. The initial OH:Al mole ratio was 3.055.

Stirring of the caustic slurry was initiated and continued for 10 days. During the gibbsite metathesis period, the hot cell ambient temperature slowly increased from 25.7 to 29.1 °C. The

liquid temperature in the test vessel was 1-2 °C higher. The caustic slurry lost ≈1.5 g in weight over the 10-day metathesis. Based on observations during the water dissolution test, evaporative losses of 10-15 g had been expected. The smaller observed loss could be due, at least in part, to absorption of ambient moisture by the test slurry and/or the reduced partial pressure of water vapor over the concentrated caustic solution.

A photograph of the 993.64 g of caustic slurry in the test vessel at the conclusion of the gibbsite metathesis period is shown in Figure 4-07.

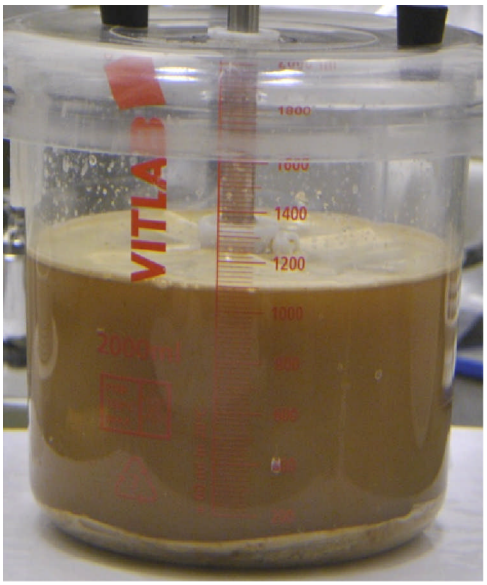
Figure 4-07. Test Slurry at End of Gibbsite Metathesis.



4.4.1.3 Aluminate Dissolution: At 07:45 on March 17, 2012, 470.54 g of 4 °C deionized water was slowly added to the caustic slurry. Addition of cold water counterbalanced the heat generated by dilution of the caustic solution, and the final temperature of the slurry rose to only 31.2 °C. At a temperature of 29 °C, the volume of water added would have been 472.46 mL.

Stirring was restarted and continued until 13:00 on March 19. Over these 2 days, the hot cell ambient temperature ranged from 29.1 to 29.4 °C. The test slurry lost ≈3.0 g over the 53-hr period. Photographs of the 1446.57 g of test slurry at the beginning and end of the aluminate dissolution period are shown in Figure 4-08.

Figure 4-08. Test Slurry at Beginning and End of Aluminate Dissolution.

Beginning 17-Mar-2012, 09:00	End 19-Mar-2012, 08:45
	

Decant of the test liquid began at 14:00 on March 19 and was completed at 11:00 on March 20. Hot cell ambient temperature decreased slightly from 29.4 to 28.9 °C over the 21 hrs. After settling overnight, the supernatant liquid was still semi-opaque and increasing the centrifugation time to 30 min at 1000 × *g* was required to clarify the decanted liquid. All portions of the clear C-Liq were collected in a single large beaker, covered, and set aside.

If all Al in the initial test sample was contained in 55.8 wt% gibbsite and if all the gibbsite was still present at the beginning of the caustic dissolution test, the quantity of caustic solution added provided 3.06 moles of OH for each mole of Al. This value is slightly greater than the target value of 3 moles of OH for each mole of Al. Also, the volume of water added for aluminate dissolution (at a common temperature of 29 °C) was ≈101% of the volume of caustic solution. This value is slightly less than the target value of 102%. Had the metathesis reaction been 100% complete, the composition of the final test liquid would have been shifted slightly from the target to that indicated by the 'C-109 Heel Test' data point in Figure 4-06, and a small portion (≈1.4 g of $NaAl[OH]_4$ out of ≈339 g) may not have been dissolved. Since the metathesis reaction was not 100% complete (Sections 4.5 and 4.6) and any remaining $NaAl(OH)_4$ would have been recovered in the initial rinses of the residual solids (Section 4.4.1.4), the impact of the small deviations from the targeted additions on the results of the dissolution tests would be insignificant.

4.4.1.4 Initial Rinsing of Solids: On March 20, the wet solids that remained following the aluminate dissolution were rinsed three times with 45.75 g, 49.78 g, and 45.60 g of deionized water. In each rinse, the added water was first used to recover any centrifuged solids remaining from previous steps, and then transferred to the test vessel. The walls of the vessel were rinsed using a disposable pipette and then the slurry was gently stirred with a spatula. The slurry was allowed to settle for 3 min, then ≈40 mL of liquid and entrained solids were transferred to a centrifuge cone with a pipette. The liquid was centrifuged until clear, then added to the liquid

collected during the primary decant (Section 4.4.1.3). Centrifuging for 30 min was required for the first rinse, while 15 min was sufficient for the final two.

The liquid decanted from the test vessel combined with the three water rinses weighed 1408 g. The D_{Liq} of the combined liquids was 1.325 g/mL at 30.0 °C. The wt% dissolved solids, measured by evaporating 6.259 g of the liquid to dryness at 90-100 °C, was 33.60 wt%.

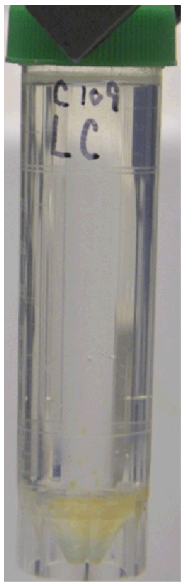
A sample of the final test liquid (S12R000175) was submitted to 222-S for analysis. The sample was not diluted prior to load-out from the 11A hot cells and transfer to 222-S.

A photograph of the wet residual solids after completion of the initial rinses following the aluminate dissolution step is shown in Figure 4-09.

Figure 4-09. Wet Residual Solids Following Initial Rinses.

4.4.1.5 Caustic Dissolution Liquor: A portion of the combined caustic dissolution/initial rinse liquor was transferred to a 50-mL centrifuge cone immediately after the conclusion of the initial rinses on March 20, 2012. When collected, the liquid was clear, colorless, and free of solids. Within a day, a small amount of a light-brown, floc- or gel-type precipitate formed in the bottom of the cone (and formed a very thin layer covering the bottom of the beaker holding the bulk of the liquid). The nature of the precipitate is unknown but it did not have the appearance of precipitating gibbsite. The sample was observed periodically over 170 days. The only change observed was perhaps a slight increase in the quantity of the precipitate. Photographs of the precipitate are shown in Figure 4-10. A similar, but more voluminous precipitate was observed in the aggregate dissolution tests (Figure 5-07).

Figure 4-10. Large-scale Caustic Dissolution Test Liquids.

(a) March 26, 2012	(b) May 10, 2012	(c) September 7, 2012
		

4.4.2 Sample Analyses

The results of physicochemical analysis of the combination of the liquid decanted from the test vessel at the conclusion of the aluminate dissolution and the liquid recovered after the three initial rinses are summarized in Table 4-02. A transcript of the laboratory data summary report for the analyses is in Attachment A.

Liquid density and dissolved solids measurements were performed in the 11A hot cells by 222-S I&C personnel. All other analyses were performed in 222-S by ATL personnel. The only radiochemical analysis requested for retrieval testing liquid samples was ‘Gross alpha’.

Table 4-02. Analyses of Large-scale Caustic Dissolution Test Liquids.

Analyte	Caustic Dissolution Test Liquid	
Specific gravity	1.319	
D_{Liq}	1.325 (30.0 °C)	g/mL
Dissolved solids (95 °C)	33.60	wt%
TGA (< 200 °C)	68.78	wt%
TGA (> 200 °C)	< 1	wt%
Hydroxide	95,800	µg/mL
TIC	68	µg/mL
TIC as CO_3	342	µg/mL
TOC	245	µg/mL
ΔTOC	242	µg/mL
Formate	10	µg/mL
Acetate	< 1	µg/mL
Glycolate	< 1	µg/mL
Oxalate	< 2	µg/mL
Nitrite	15	µg/mL
Nitrate	24	µg/mL
Fluoride	73	µg/mL
Phosphate	1,160	µg/mL
Sulfate	< 2	µg/mL
Chloride	4	µg/mL
Sodium	156,000	µg/mL
Aluminum	47,000	µg/mL
Silicon	159	µg/mL
Phosphorus	406	µg/mL
P as PO_4	1,245	µg/mL
Sulfur	< 400	µg/mL
S as SO_4	< 1,198	µg/mL
Calcium	< 200	µg/mL
Chromium	< 20	µg/mL
Manganese	< 12	µg/mL
Iron	28	µg/mL
Nickel	< 80	µg/mL
Bismuth	< 400	µg/mL
Uranium	< 400	µg/mL
Gross alpha	1.09E-3	µCi/mL

4.5 RESIDUAL SOLIDS

4.5.1 Supplemental Rinses

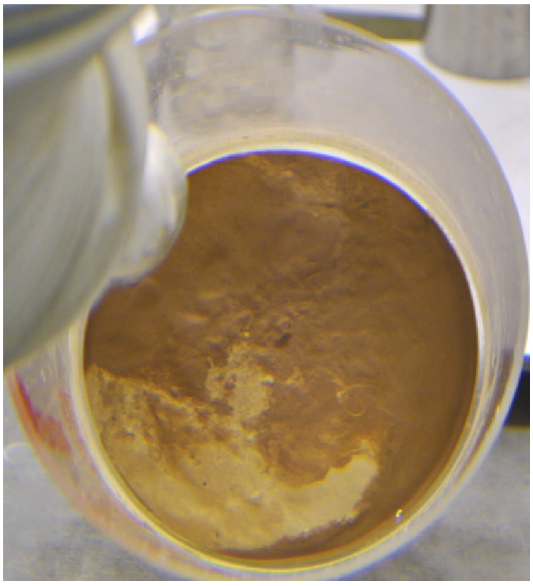
Recovery of the solids remaining at the conclusion of the retrieval dissolution tests was begun on March 21, 2012. The recovery began with a series of supplemental rinses with deionized water. In each rinse, 50 mL of water were added to the test vessel. The slurry was then gently stirred to thoroughly mix the sample and rinse water. After the slurry settled for 5 min, 45-50 mL of liquid were transferred to a pre-weighed centrifuge cone and centrifuged until clear (15-30 min). The cone was then reweighed, and the density of the liquid was estimated. The centrifuged supernatant and any additional clear liquid that could be removed from the test slurry were then disposed of.

The estimated densities of the rinse solutions after five supplemental rinses were: 1.07, 1.06, 1.05, 1.02, and 1.01 g/mL. After the fifth supplemental rinse, the solids in the centrifuge cone were recovered with 10-15 mL of water and returned to the test slurry. After the slurry settled for 30 min, 8-10 mL of clear liquid was pipetted off and disposed of.

The test vessel was then set aside, uncovered, until March 27 to allow the residual solids to dry. Hot cell ambient temperature over this period was 28 °C. The final weight of the air-dry residual solids was 80.36 g. Photographs of the residual solids remaining at the conclusion of the large-scale water and caustic dissolution tests are shown in Figure 4-11.

Figure 4-11. Dry Residual Solids from Large-scale Dissolution Tests.

(a) March 22, 2012



(b) March 26, 2012

4.5.2 Bulk Density

The D_{Bulk} of the air-dry residual solids was measured by water displacement. Two 50-mL centrifuge cones were prepared for the measurement by measuring the volume contained in each when filled to the nominal '45-mL' and '50-mL' marks with reagent water (Section 8.2.4.2).

On March 27, 2012, the solids were divided between the two preweighed cones and weighed. After the cones were partially filled with deionized water, they were gently shaken and vibrated on a vortex mixer to release all visible air bubbles. Each cone was then sequentially filled to the 45-mL and 50-mL marks and weighed. Approximately 10 mL of water was then pipetted from each cone, and the measurement process was repeated. The data collected during the measurement and the D_{Bulk} calculations follow.

		Cone RD1	Cone RD2
Vol Cone at 45-mL mark	(mL)	44.73 ± 0.08	44.51 ± 0.05
Vol Cone at 50-mL mark	(mL)	49.66 ± 0.07	49.38 ± 0.04
Wt Residual solids	(g)	36.938	42.889
First Pass			
t Added H_2O	(°C)	27.6	27.4
Density Added H_2O	(g/mL)	0.996346	0.996402
45-mL Mark			
Wt Added H_2O to 45-mL mark	(g)	27.839	25.331
Vol Added H_2O to 45-mL mark	(mL)	27.941	25.422
Vol Residual solids	(mL)	16.790	19.085
D_{Bulk} Residual solids	(g/mL)	2.200	2.247
50-mL Mark			
Wt Added H_2O to 50-mL mark	(g)	32.451	30.241
Vol Added H_2O to 50-mL mark	(mL)	32.570	30.350
Vol Residual solids	(mL)	17.089	19.031
D_{Bulk} Residual solids	(g/mL)	2.162	2.254
Second Pass			
t Added H_2O	(°C)	27.8	27.3
Density Added H_2O	(g/mL)	0.996290	0.996430
45-mL Mark			
Wt Added H_2O to 45-mL mark	(g)	27.572	24.942
Vol Added H_2O to 45-mL mark	(mL)	27.675	25.031
Vol Residual solids	(mL)	17.056	19.476
D_{Bulk} Residual solids	(g/mL)	2.167	2.202
50-mL Mark			
Wt Added H_2O to 50-mL mark	(g)	32.802	30.018
Vol Added H_2O to 50-mL mark	(mL)	32.924	30.126
Vol Residual solids	(mL)	16.735	19.255
D_{Bulk} Residual solids	(g/mL)	2.207	2.227
Average D_{Bulk}	(g/mL)	2.18 ± 0.08	2.23 ± 0.05
Average D_{Bulk} Residual solids	(g/mL)	2.21 ± 0.09	

4.5.3 Sieve Separation

On March 29, 2012, the residual solids from the large-scale dissolution tests were wet sieved. The sieving operation is described in Section 6.1. The residual solids were found to contain 17.20 g of air-dry solids >710 µm in size and 63.16 g of air-dry solids <710 µm in size.

4.5.4 Sampling

The residual solids >710 µm in size were dried at 90-100 °C after the sieve separation. On April 12, 2012, these solids were combined and ground in a mortar and pestle until they passed through a 425-µm mesh sieve.

A sample of homogenized >710-µm solids (S12R000196) was collected for SPC measurements. Another sample (S12R000197) was collected and submitted to 222-S for physicochemical and radiochemical analyses. All the >710-µm solids were consumed in these two samples.

Three samples were removed from the moderately thin paste of <710-µm solids created in the wet sieving test (Section 6.1.2). The samples were used for a wt% solids measurement, for particle size distribution (PSD) analysis (Section 6.2), and for settling tests (Section 7.0).

The remaining <710-µm residual solids were then air-dried. On April 16, 2012, the dry solids were thoroughly stirred and passed through a 425-µm sieve. Only minimal crushing of a small portion of the dry solids was required to pass through the sieve.

A sample of the dry <710-µm solids (S12R000201) was collected for SPC measurements. Two samples (S12R000200-A and –B) were collected and submitted to 222-S for physicochemical and radiochemical analyses. The remaining dry <710-µm residual solids were set aside for archive.

4.5.5 Solid Phase Characterization

The results of SPC analysis of the residual solids samples are included in Attachment D.

The only solid phase identified by XRD in both the >710-µm and <710-µm fractions of the residual solids was gibbsite. The diffraction patterns did not suggest that significant amounts of unidentified or amorphous solids were present in either sample.

Examination of the solids using SEM/EDX confirmed that gibbsite was the dominant phase present in the residual solids. Minor components of the residual solids were tentatively identified as U-rich solids, Ca/PO_4-rich solids, and Fe-rich solids.

4.5.6 Physicochemical and Radiochemical Analysis

The results of physicochemical analysis of the air-dry <710-µm and >710-µm residual solids recovered at the conclusion of the large-scale dissolution tests are summarized in Table 4-03. The analysis results for the starting crushed solids test composite, from Table 3-01, have been included for comparison. Density, weight loss, and H_2O measurements were performed by 222-S I&C personnel. All other analyses were performed in 222-S by ATL personnel. A transcript of the laboratory data summary report for the ATL analyses is included in Attachment A.

The results of SPC measurements (Section 4.5.5) suggest that most of the Al in the air-dry residual solids from the large-scale dissolution tests was in the form of gibbsite. If all the Al found in the residual solids was in the form of gibbsite, the reported 29.4 wt% Al would indicate that the <710-µm residual solids were ≈85.0 wt% gibbsite. Similarly, the reported 30.3 wt% Al would indicate that the >710-µm residual solids were ≈87.6 wt% gibbsite.

Table 4-03. Physicochemical Analysis of Large-scale Dissolution Residual Solids.

		Crushed Heel Solids Test Composite		Residual Solids			
				< 710 µm		> 710 µm	
D_{Bulk}	g/mL		2.044	2.21			
Wt loss (95 °C)	wt%		18.3		—		—
TGA (<200 °C)	wt%		17.04		< 1		< 1
TGA (>200 °C)	wt%		20.17		29.61		30.00
H_2O (110 °C)	wt%		19.3		0.30		0.24
OH from pH	µg/g	(W)	956		—		—
TIC	µg/g		1,755		347		90
TIC as CO_3	µg/g		8,768		1,734		452
TOC	µg/g		2,175		2,530		537
ΔTOC	µg/g		1,794		2,527		537
Formate	µg/g	(W)	122	(W)	< 9	(W)	< 8
Acetate	µg/g	(W)	111	(W)	< 6	(W)	< 5
Oxalate	µg/g	(W)	1,111	(W)	9	(W)	< 10
Nitrite	µg/g	(W)	4,945	(W)	17	(W)	23
Nitrate	µg/g	(W)	2,895	(W)	53	(W)	61
Fluoride	µg/g	(W)	10,500	(W)	35	(W)	141
Phosphate	µg/g	(W)	108,000	(W)	263	(W)	1,210
Sulfate	µg/g	(W)	621	(W)	19	(W)	17
Chloride	µg/g	(W)	57	(W)	6	(W)	6
Sodium	µg/g	(Z)	93,600	(Z)	8,450	(Z)	7,340
Aluminum	µg/g	(Z)	193,000	(Z)	294,000	(Z)	303,000
Silicon	µg/g	(A)	231	(Z)	3,556	(Z)	2,930
Phosphorus	µg/g	(Z)	30,700	(A)	1,530	(A)	735
P as PO_4	µg/g		94,132		4,691		2,254
Sulfur	µg/g	(Z)	< 4,540	(A)	< 192	(A)	< 154
S as SO_4	µg/g		< 13,602		< 575		< 461
Calcium	µg/g	(A)	575	(A)	3,670	(A)	547
Chromium	µg/g	(A)	19	(Z)	504	(Z)	289
Manganese	µg/g	(A)	9	(A)	55	(A)	37
Iron	µg/g	(Z)	1,705	(A/Z)	9,695	(A/Z)	8,405
Nickel	µg/g	(A/Z)	884	(A)	4,390	(A)	927
Zirconium	µg/g		—	(A)	23	(A)	9
Uranium	µg/g	(Z)	982	(A)	2,440	(A)	398

Results for radiochemical analyses of the <710-µm and >710-µm residual solids are summarized in Table 4-04. The results of the radiochemical analyses of the initial ATL composite solids (RPP-RPT-50883) from Table 3-02 have been included for comparison.

Table 4-04. Radiochemical Analysis of Large-scale Dissolution Residual Solids.

		ATL Composite Air-Dry Heel Solids		Residual Solids			
				< 710 µm		> 710 µm	
Strontium-89/90	uCi/g	(Z)	1.14E+2	(Z)	8.23E+2	(Z)	2.76E+2
Technetium-99	ug/g	(A)	2.68E-1	(A)	< 9.14E-1	(A)	< 7.96E-1
Iodine-129	uCi/g		< 6.51E-6		< 5.99E-5		< 4.25E-5
Cesium-137	uCi/g	(Z)	1.91E+1	(Z)	7.81E-1	(Z)	8.13E-1
Lead-212	uCi/g		—		—	(Z)	5.54E-1
Lead-214	uCi/g		—		—	(Z)	7.83E-1
Actinium-228	uCi/g		—		—		—
Uranium-233	ug/g	(Z)	< 1.09E-1	(Z)	< 1.41E+0	(Z)	< 1.06E+0
Uranium-234	ug/g	(Z)	6.03E-2	(Z)	< 3.54E-2	(Z)	< 2.65E-2
Uranium-235	ug/g	(Z)	6.46E+0	(Z)	1.45E+1	(Z)	2.40E+0
Uranium-236	ug/g	(Z)	8.07E-2	(Z)	< 2.83E-2	(Z)	< 2.12E-2
Neptunium-237	ug/g	(Z)	8.13E-2	(Z)	< 7.07E-1	(Z)	< 5.30E-1
Uranium-238	ug/g	(Z)	9.76E+2	(Z)	2.17E+3	(Z)	3.61E+2
Plutonium-238	uCi/g	(Z)	6.79E-4	(Z)	2.79E-3	(Z)	8.96E-4
Plutonium-239/240	uCi/g	(Z)	1.86E-2	(Z)	8.39E-2	(Z)	2.44E-2
Americium-241	uCi/g	(Z)	1.03E-2	(Z)	8.63E-2	(Z)	3.97E-2
Curium-243/244	uCi/g		—	(Z)	< 1.47E-4	(Z)	< 4.20E-5

4.6 RESULTS SUMMARY

At the beginning of the large-scale dissolution tests, the air-dry, C-109 heel solids composite weighed 401.47 g and had a volume of 196.4 mL. Analysis indicated the solids contained ≈158 g of natrophosphate and ≈224 g (2.87 moles) of gibbsite. The solids passed through a 3-step water dissolution test; in each step a volume of water approximately equal to 3× the initial volume of the solids (≈589 mL) was added. A 10-day gibbsite metathesis step was then initiated by addition of ≈465 mL of 49.6 wt% caustic solution providing 3.05 moles of OH for each mole of Al estimated to have been present in the starting composite sample. Dissolution of the sodium aluminate generated in the metathesis step was initiated by addition of ≈472 mL of water and allowed to proceed for 2 days. After decanting the final test solution, the residual solids were thoroughly rinsed with water and dried.

Overall, the dissolution test sequence removed 80.0 wt% (321.11 g) of the initial 401.47 g of air-dry, C-109 heel solids. The 80.36 g of air-dry residual solids were determined to be mostly gibbsite by XRD; chemical analysis confirmed that the residual solids were up to 85.0-87.6 wt% gibbsite.

4.6.1 Sample Analyses

In Table 4-05(a), the analyses of all the large-scale dissolution test samples have been reformatted in terms of the total weights of analytes (analyte inventories) present throughout the test sequence. The weights, volumes, and densities in the first three rows of the table describe the crushed solids composite, the liquids removed from the system in each dissolution step, and the residual solids. In the remaining rows, the calculated weights of the analytes in the composite solids and remaining in the residual solids are tabulated in the second and seventh columns. The calculated weights of the analytes removed from the system in the dissolution test steps are tabulated in the third through sixth columns.

In Table 4-05(b) the component inventory values are tabulated as cumulative weights removed in the four dissolution steps. In calculating the cumulative weights removed, steps where the base analytical result was '< Detection Limit' were assigned a weight of 0 g. Since most of the Na and OH removed in the caustic dissolution step represented recovery of the added caustic, they were not included in the cumulative weights for the step. The wt% values in the final row of the table are relative to the calculated total weights of the components present in the crushed heel solids composite (172.6 g). Inventories for Si, Ca, Cr, and Mn in the initial composite are based on analyses of acid digest sample preparations reported in RPP-RPT-50883 and may underestimate the amounts of these elements in the heel solids composite.

For eight components—carbonate (TIC), formate, acetate, oxalate, nitrite, nitrate, sulfate, and chloride—sample analyses indicate that the majority of the solids were dissolved/recovered in the water Contact #1. The concentrations/inventories then decrease in the two remaining water contacts and the caustic dissolution as the dissolved components were diluted and recovered. These results would be characteristic of the dissolution and recovery of soluble to highly soluble salts left behind as the liquid component of the C-109 heel evaporated. The uranium component(s) partially follow this trend; however, ≈40 wt% of the estimated inventory remained in the residual solids.

Nearly all of the fluoride and phosphate components were dissolved and recovered in the three water contacts. This is consistent with the assumption that nearly all of the fluoride and phosphate in the crushed heel solids composite was associated with natrophosphate. In addition to the highly soluble sodium salts, ≈1.6 g of fluoride and ≈16.9 g of phosphate were dissolved and recovered in the Contact #1 test liquid (D_{Liq} = 1.064 g/mL). Approximately 1.9 g fluoride and 19.2 g of phosphate were recovered in the Contact #2 test liquid (D_{Liq} = 1.056 g/mL). Nearly all the remaining fluoride (≈0.6 g) and phosphate (≈5.9 g) in the composite solids was dissolved and recovered in the Contact #3 test liquid (D_{Liq} = 1.014 g/mL). The small amounts of fluoride and phosphate recovered in the caustic dissolution test liquid are mostly due to carryover of undecanted water Contact #3 test liquid.

Only 0.2% of the 77.5 g of Al in the crushed heel solids composite was recovered during the three water dissolution contacts. Approximately 64.5% of the Al was dissolved and recovered in the combined metathesis/aluminate dissolution sequence. Approximately 30.8% of the Al in the initial test composite solids remained in the air-dry residual solids.

Table 4-05. Analyte Inventories in the Large-scale Dissolution Tests. (2 sheets)

(a) Weights in composite, removed in dissolution steps, and remaining in residual solids

	Heel Solids Composite	H_2O Contact #1	H_2O Contact #2	H_2O Contact #3	Caustic Dissolution	Residual Solids
Total Wt (g)	401.47	575.60	661.76	610.84	1,408.0	80.36
Total Vol (mL)	196.40	540.72	626.55	602.70	1,062.6	36.36
Density (g/mL)	2.044	1.064	1.056	1.014	1.325	2.210
	Wt (g) In	**Wt (g) Removed**				**Wt (g) In**
Wt Loss (95 °C)	73.29	—	—	—	—	—
Dissolved solids (95 °C)	—	38.02	39.01	11.71	473.1	—
TGA (<200 °C)	68.43	—	—	—	—	< 0.8
TGA (>200 °C)	80.95	—	—	—	—	23.86
H_2O (110 °C)	77.37	—	—	—	—	0.23
Hydroxide	0.38	—	—	—	101.797 [a]	—
TIC as CO_3	3.520	2.675	0.610	0.303	0.363	0.117
ΔTOC	0.720	0.031	< 0.001	0.062	0.257	0.169
Formate	0.049	0.044	0.009	0.003	0.011	< 0.001
Acetate	0.045	0.040	0.017	< 0.001	< 0.001	< 0.001
Oxalate	0.446	0.214	0.060	0.015	< 0.002	0.001
Nitrite	1.985	1.472	0.246	0.032	0.016	0.001
Nitrate	1.162	0.787	0.136	0.021	0.026	0.004
Fluoride	4.215	1.561	1.856	0.562	0.077	0.005
Phosphate	43.359	16.911	19.193	5.873	1.233	0.037
Sulfate	0.249	0.163	0.033	0.007	< 0.002	0.001
Chloride	0.023	0.013	0.003	< 0.001	0.004	0.001
Sodium	37.578	13.145	13.324	3.925	165.766 [a]	0.660
Aluminum	77.484	0.062	0.065	0.039	49.942	23.781
Silicon	0.093	0.004	0.005	0.004	0.169	0.275
Phosphorus	12.325	5.402	6.258	1.774	0.431	0.109
P as PO_4	37.791	16.563	19.187	5.439	1.323	0.335
Sulfur	< 1.823	0.059	0.013	< 0.008	< 0.425	< 0.015
S as SO_4	< 5.461	0.175	0.039	< 0.023	< 1.273	< 0.044
Potassium	—	0.036	< 0.040	< 0.038	—	—
Calcium	0.231	< 0.003	< 0.004	< 0.004	< 0.213	0.241
Chromium	0.008	0.003	0.001	< 0.001	< 0.021	0.037
Manganese	0.004	< 0.001	< 0.001	< 0.001	< 0.013	0.004
Iron	0.685	0.008	0.002	< 0.001	0.030	0.757
Nickel	0.355	< 0.001	< 0.002	< 0.002	< 0.085	0.293
Zirconium	—	—	—	—	—	0.002
Bismuth	< 1.823	< 0.007	< 0.008	< 0.008	< 0.425	< 0.538
Uranium	0.394	0.198	0.035	< 0.008	< 0.425	0.161

[a] Most of the Na and OH removed in the caustic dissolution step represent recovery of the added caustic.

Table 4-05. Analyte Inventories in the Large-scale Dissolution Tests. (2 sheets)

(b) Cumulative weights removed during dissolution tests and remaining in residual solids

	Wt (g) in Heel Solids Composite	Cumulative Wt (g) Removed				Wt (g) in Residual Solids
		H_2O Contact #1	H_2O Contact #2	H_2O Contact #3	Caustic Dissolution	
TIC as CO_3	3.52	2.68	3.28	3.59	3.95	0.12
ΔTOC	0.72	0.031	0.031	0.093	0.35	0.17
Formate	0.049	0.044	0.054	0.056	0.067	0
Acetate	0.045	0.040	0.057	0.057	0.057	0
Oxalate	0.45	0.21	0.27	0.29	0.29	0.001
Nitrite	1.98	1.47	1.72	1.75	1.77	0.001
Nitrate	1.16	0.79	0.92	0.94	0.97	0.004
Fluoride	4.22	1.56	3.42	3.98	4.06	0.005
Phosphate	43.4	16.9	36.1	42.0	43.2	0.34
Sulfate	0.25	0.16	0.20	0.20	0.20	0.001
Chloride	0.023	0.013	0.016	0.016	0.020	0.001
Sodium	37.6	13.1	26.5	30.4	30.4 [b]	0.66
Aluminum	77.5	0.062	0.13	0.17	50.1	23.8
Silicon (A)	0.093	0.004	0.009	0.013	0.18	0.28
Calcium (A)	0.23	0	0	0	0	0.24
Chromium (A)	0.008	0.003	0.003	0.003	0.003	0.037
Manganese (A)	0.004	0	0	0	0	0.004
Iron	0.68	0.008	0.010	0.010	0.040	0.76
Nickel	0.36	0	0	0	0	0.29
Zirconium	—	—	—	—	—	0.002
Uranium	0.39	0.20	0.23	0.23	0.23	0.16
Sums	172.6 g —	37.3 g 21.6 wt%	72.9 g 42.2 wt%	83.8 g 48.5 wt%	135.9 g 78.7 wt%	26.8 g 15.6 wt%

[b] Na and OH recovered in the caustic dissolution test liquid are not included in cumulative wt removed.

The weight losses at temperatures >200 °C reported from TGA analysis of both the heel solids composite and the residual solids were both slightly greater than the estimated Al contents of the solids. These results are consistent with the two-stage dehydration of gibbsite to Al_2O_3 (Equation 3-2) where the weight of water lost is 100.2% of the weight of Al in the gibbsite solids.

Most of the Ca, Mn, Fe, and Ni in the composite solids carried through to the residual solids. This is also probably the case for Si and Cr. Approximately 24 wt% of the ΔTOC was found in the residual solids with ≈36 wt% having been recovered in the caustic dissolution test. As previously noted, ≈41 wt% of the U in the heel composite was found in the residual solids with the remainder having been dissolved and recovered in the water dissolution test.

Examination of the 'Sums' at the bottom of Table 4-05(b) indicates that ≈5.7 wt% of the crushed heel solids composite are unaccounted for based on summation of component inventories. Given that (a) a large number of sampling steps were performed, (b) a large number of analyses of solid and liquid samples were performed, and (c) the liquids from the final water rinses of the residual solids were discarded without analysis, this mass balance is quite acceptable.

Also, the inventory sum for the residual solids in Table 4-05(b), 15.6 wt%, is less than the 20.0 wt% value based on the weights of the test composite and residual solids. This difference could be due to relatively small errors in analytical results for the crushed heel solids and/or the residual solids or to unidentified/misidentified components in one or both of the solid samples.

4.6.2 Discussion and Analysis

In Table 4-06, the analyte inventories (from Table 4-05[b]) are recalculated as possible solid phase components. The same solid phases proposed for the C-109 heel solids composite in Table 3-03 are used (the solid phase composition from Table 3-03 is reproduced in the second column of Table 4-06). Oxides of Si, Ca, Cr, Mn, and Zr have also been included. The cumulative wt%s of each solid phase removed in the four dissolution test steps are tabulated in columns 3 through 6. The wt%s of each solid phase remaining in the residual solids are tabulated in the last column. The wt% values are calculated relative to the 401.47-g starting weight of the crushed heel solids test composite.

As in Table 4-05(b), the 'Sums' at the bottom of Table 4-06 leave ≈4.6 wt% of the crushed heel solids composite unaccounted for based on summation of component solid phase inventories. The summation of the phase inventories for the residual solids, 17.9 wt%, is also less than the 20.0 wt% value based on the weights of the test composite and residual solids. Proposed sources of these relatively small deviations are the same as those noted in Section 4.6.1.

Table 4-06. Solid Phase Inventories during the Large-scale Dissolution Tests.

Estimated wt% of solid phases removed during dissolution tests and remaining in the residual solids (Percentages calculated relative to the initial weight of the test composite)

	Wt% in Heel Solids Composite	Cumulative Wt% Removed				Wt% in Residual Solids
		H_2O Contact #1	H_2O Contact #2	H_2O Contact #3	Caustic Dissolution	
$Al(OH)_3$	55.8	0.045	0.092	0.12	36.1	17.1
$Na_7F(PO_4)_2 \cdot 19H_2O$	39.4	14.6	31.9	37.2	37.9	0.043
$Na_2CO_3 \cdot H_2O$	1.81	1.38	1.69	1.85	2.03	0.060
$NaNO_2$	0.74	0.55	0.64	0.65	0.66	0.001
$NaNO_3$	0.40	0.27	0.32	0.32	0.33	0.001
Fe_2O_3	0.24	0.003	0.004	0.004	0.014	0.27
SiO_2	0.05	0.002	0.005	0.007	0.097	0.15
$Na_2U_2O_7$	0.13	0.066	0.077	0.077	0.077	0.053
Organic C	0.18	0.008	0.008	0.023	0.087	0.042
Na-Oxalate	0.17	0.081	0.104	0.109	0.109	< 0.001
NiO	0.11	0	0	0	0	0.093
CaO	0.08	0	0	0	0	0.084
Na_2SO_4	0.09	0.060	0.072	0.075	0.075	0.001
Na-Formate	0.02	0.017	0.020	0.021	0.025	0
Na-Acetate	0.02	0.014	0.020	0.020	0.020	0
Cr_2O_3	0.003	0.001	0.001	0.001	0.001	0.013
NaCl	0.009	0.005	0.007	0.007	0.008	< 0.001
MnO	0.001	0	0	0	0	0.001
ZrO_2	—	—	—	—	—	0.001
Sums	99.4	17.1	35.0	40.4	77.5	17.9

4.6.2.1 Natrophosphate Dissolution: An estimate of the solid phase composition of the crushed heel solids composite based on SPC measurements and chemical analyses indicated that the heel solids contained ≈158 g or 39.4 wt% of natrophosphate (Section 3.4). Modeling performed using the Environmental Simulation Program[3] (ESP) indicates that 8.355 kg of water are required to dissolve 1 kg of pure natrophosphate at 25 °C[4]. These values indicate that at least 1,320 g (1,324 mL) of water should have been required to dissolve the natrophosphate in the C-109 heel solids composite.

In water Contact #1, 579.8 g of water were added, and 58.5 g of natrophosphate were dissolved and recovered (based on fluoride analysis results). The water:natrophosphate ratio for Contact #1 was 9.91 w/w, i.e., less natrophosphate was dissolved per weight of added water than

[3] OLI systems, Inc., Morris Plains, New Jersey.

[4] Preliminary results of water dissolution testing on heel solids retrieved from tank 241-C-110, which were predominantly natrophosphate, indicated that the ESP predictions were relatively accurate at 15 °C and 45 °C.

predicted by ESP. The reduced natrophosphate solubility was caused by a sodium common-ion effect due to dissolution of other highly soluble sodium salts in Contact #1.

In Contact #2, 587.5 g of water were added, and 69.6 g of natrophosphate were dissolved and recovered. The ratio of water added to natrophosphate recovered was 8.44 w/w. Allowing for analytical errors and recognizing that the weights of natrophosphate recovered in the water contacts were probably smaller than the weights dissolved because the test liquids were not completely removed, the water:natrophosphate ratio in Contact #2 is quite close to the 8.36 value predicted by ESP at 25 °C.

Insufficient natrophosphate remained in the heel solids to produce a saturated solution after the addition of 589.9 g of water in Contact #3. Using the water:natrophosphate ratio from Contact #2, the addition of 177.9 g (178.4 mL) of water in Contact #3 would have been sufficient to dissolve the 21.1 g of natrophosphate recovered. Under the same assumption, a cumulative addition of 1,345 g of water would have been sufficient to dissolve and recover the natrophosphate in the crushed heel solids composite. Because of the noted common-ion effect, the weight of water required to dissolve the natrophosphate in the heel solids was ≈1.8% greater than the weight (from ESP) required to dissolve the same weight of natrophosphate in the absence of other heel solids components.

4.6.2.2 Gibbsite Dissolution: An estimate of the solid phase composition of the test composite based on SPC measurements and chemical analyses indicated that the crushed heel solids contained ≈224 g, or 55.8 wt% gibbsite. The same analyses indicated that the residual solids contained ≈68.8 g, or 85.6 wt% gibbsite. This suggests that ≈155 g, or 69.3 wt%, of the gibbsite present in the heel solids composite was metathesized/dissolved/ recovered in the caustic dissolution test. Previous testing on dissolution of gibbsite in strong caustic solutions (WRPS-0900468, "Gibbsite Dissolution Phase 2 Test Results") showed that under similar test conditions (i.e., 19.4 M NaOH, OH:Al mole ratio of 3:1, and a temperature of 25 °C), the metathesis of gibbsite solids with an average particle size of 56 µm to sodium aluminate was 75% complete after 7 days. Given analytical errors and a C-109 heel composite containing a significant quantity of solids well in excess of 56 µm in size, the observed 69 % gibbsite metathesis/dissolution is in reasonable agreement with the 75% metathesis observed in the scoping tests (WRPS-0900468).

5.0 AGGREGATE DISSOLUTION TESTS

The aggregate dissolution test sequence generally reproduced the large-scale dissolution tests performed on the crushed, C-109 heel composite sample (Section 4.0). The tests were performed (1) to determine if the solids would disaggregate at some point during the test sequence or (2) to determine the degree to which the aggregates were dissolved and retrieved less efficiently than the crushed solids.

5.1 AGGREGATE TEST SAMPLE

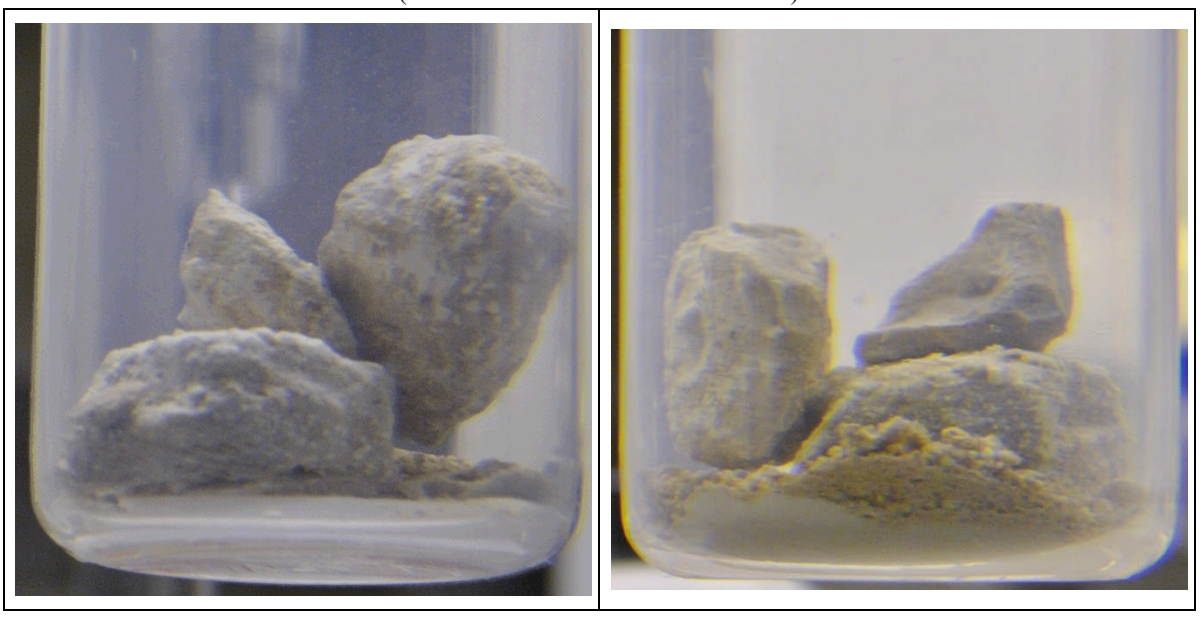
As noted in Section 2.4, the largest piece of aggregate solids was removed from each of two of the C-109 heel samples on June 14, 2011. The piece of aggregate removed from sample C109-10-1C weighed 31.8 g; the piece removed from sample C109-10-2A weighed 31.5 g.

The aggregate solids were not submitted for chemical analysis. However, SPC measurements on similar aggregate solids (Section 3.1) indicated that the test aggregates were probably composed of gibbsite particles cemented, mostly, with additional gibbsite.

The aggregates from both samples were transferred to a single, 250-mL bottle before starting the retrieval dissolution tests. The aggregate from sample C109-10-1C was transferred intact. The aggregate from sample C109-10-2A had to be broken into two large pieces with a few small chips and a small quantity of fines to pass through the 3.1-cm diameter mouth of the test bottle. (Breaking the aggregate sample was very difficult.) After being allowed to air-dry overnight, the total weight of the aggregate solids transferred to the bottle was 58.85 g. A photograph of the final aggregate test sample at the start of the dissolution test sequence is shown in Figure 5-01.

Figure 5-01. Starting Aggregate Dissolution Test Sample.

(Outside diameter of bottle is 6.0 cm)

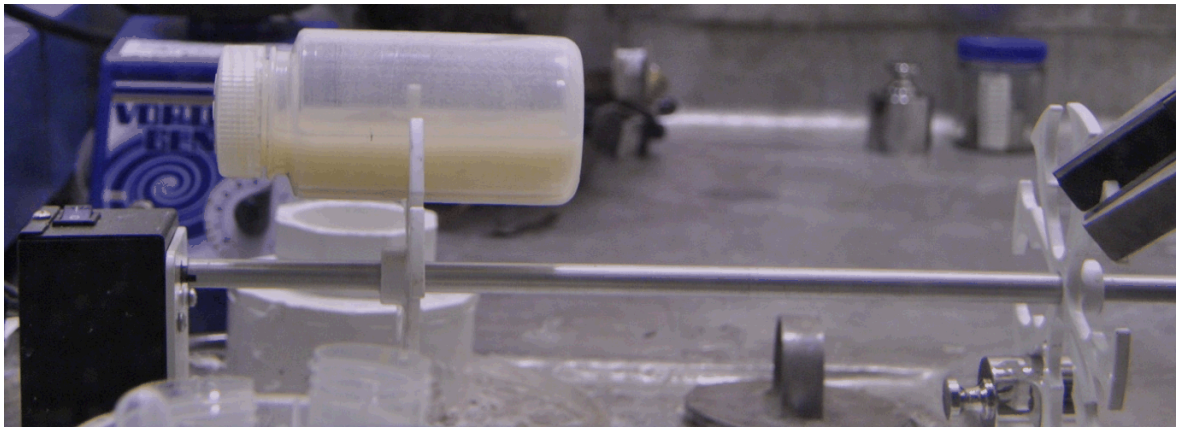


5.2 APPARATUS

The primary vessel used for the aggregate dissolution tests was a 250-mL, wide-mouth PMP bottle. The outer diameter of the bottle was 6.0 cm and the inner diameter of the mouth was 3.1 cm.

Mixing of the test slurries was accomplished by placing the test bottle in a Labquake®[5] Rotisserie mixer. The rotation speed of the rotisserie was ≈8 rpm. A photograph of the aggregate test apparatus (taken during water Contact #2) is shown in Figure 5-02.

Figure 5-02. Aggregate Dissolution Test Apparatus.



5.3 WATER DISSOLUTION

5.3.1 Test Narrative

As in the large-scale tests, the target volume of water to be added in each water contact was 3 times the nominal volume of the aggregate solids. For the water dissolution test, the density of the aggregate solids was assumed to be equal to the D_{Bulk} of the crushed solids composite, 2.044 g/mL. The nominal volume of the 58.85-g aggregate test sample was, therefore, 28.79 mL. The target water addition in each of the three water contacts was 86.37 mL (86.12 g at 25 °C).

The large-scale and aggregate dissolution tests were run concurrently. Each of the three contacts in the aggregate test was terminated at the same time as the corresponding contact in the large-scale test. The density of the aggregate test liquids was not measured during the water contacts, only at the end of each.

[5] Labquake is a registered trademark of Thermo Scientific, Dubuque, Iowa.

5.3.1.1 Water Contact #1: At 10:30 on February 22, 2012, 88.89 g (89.15 mL at 25 °C) of reagent water was added to the aggregate solids. Before mixing began, ≈32 mL of liquid was spilled as the sample was being inserted into the rotisserie mixer. The spilled liquid was colorless and contained only a minute quantity of fine-grained solids. One-half of the spilled liquid was replaced with an additional 16.39 g of water. The test sample was returned to the rotisserie and mixed, without further incident, for 29 hrs. As in the large-scale test, mixing was then stopped, and the sample was allowed to settle overnight.

The test sample lost 0.55 g of water to evaporation during Contact #1. Therefore, the total water added in Contact #1 was 88.89 g + 16.39 g – 0.55 g = 104.73 g (105.04 mL at 25 °C). The ratio of the total volume of water added in Contact #1 to the nominal starting volume of the aggregate solids was ≈3.6. Only ≈73 mL of the added water, ≈2.5 times the volume of the aggregate solids, was in contact with the aggregate solids for the entirety of Contact #1.

Approximately 48 hrs after the start of Contact #1, the supernatant liquid in the test bottle was decanted using a large-volume pipette, transferred to two 50-mL centrifuge cones, centrifuged for 15 min at ≈1000 × *g*, and transferred to a holding container. The liquid was completely clear following centrifugation.

A total of 101.74 g (100.71 mL) of liquid was removed from the aggregate test bottle in Contact #1. This total includes the 32.18 g spilled at the beginning of the Contact. The D_{Liq} was 1.010 g/mL at 25.2 °C. The temperature of the liquid was not directly measured during the contact but hot cell ambient temperature ranged from 22.3-25.2 °C.

A sample of the decanted Contact #1 liquid (S12R000146) was submitted to 222-S for analysis. Prior to load-out from the 11A hot cells, the sample was diluted 25.28% w/w with deionized water: [(Wt Sample Liquid + Wt H_2O) / Wt Sample Liquid = 1.2528].

The wt% dissolved solids was measured by evaporating 5.341 g of the decanted liquid to dryness at 90-100 °C. The decanted Contact #1 liquor contained 1.57 wt% dissolved solids.

The total weight of the wet aggregate solids in the test bottle and the damp C-Slds in the two centrifuge cones at the conclusion of Contact #1 of the water dissolution test was 61.80 g. Other than a slight smoothing of the large aggregate pieces and a slight increase in the quantity of fines caused by 29 hrs of slow tumbling, the appearance of the test sample was little changed after Contact #1.

5.3.1.2 Water Contact #2: At 08:00 on February 27, 2012, 86.12 g (86.37 mL at 25 °C) of reagent water was added to the wet solids from Contact #1. The test sample was placed in the rotisserie and mixed for 46.5 hrs.

The test sample lost 2.50 g (to evaporation) during mixing. The corrected Contact #2 water addition was 83.60 g (83.85 mL). The volume of water added was 2.91 times the starting volume of the aggregate solids.

After mixing was stopped, the test sample was allowed to settle for ≈5 hrs. The Contact #2 liquid was then decanted, centrifuged, and transferred to a holding container. Even after centrifuging for 1 hr at ≈1000 × *g,* the Contact #2 liquid still had a very slight white cloudiness.

A total of 82.59 g (82.74 mL) of liquid was removed from the test vessel. The D_{Liq} was 0.998 g/mL at 24.3 °C. Hot cell ambient temperature during Contact #2 ranged from 24.3-25.4 °C.

A sample of the decanted Contact #2 liquid (S12R000147) was submitted to 222-S for analysis. Prior to load-out from the 11A hot cells, this sample was diluted 25.05% w/w with deionized water.

The wt% dissolved solids in the decanted liquid was measured by evaporating 5.489 g to dryness at 90-100 °C. The decanted Contact #2 liquid contained 0.16 wt% dissolved solids.

The total weight of the wet aggregate solids in the test bottle and the damp C-Slds in the two centrifuge cones at the conclusion of Contact #2 of the water dissolution test was 62.90 g. The appearance of the test solids was little changed from Contact #1.

5.3.1.3 Water Contact #3: At 14:00 on February 29, 2012, 86.44 g (86.70 mL at 25 °C) of reagent water was added to the wet solids from Contact #2. The test sample was placed in the rotisserie and mixed for 24 hrs. The test sample was then allowed to sit without mixing over a 3-day weekend. At the beginning of the following workshift, the sample was mixed for an additional 1 hr (25 hrs total).

Over this time, the test sample lost 0.15 g (to evaporation) during mixing. The corrected Contact #3 water addition was 86.29 g (86.55 mL). The volume of water added was 3.01 times the starting volume of the aggregate solids.

After the final weight was recorded, the Contact #3 liquid was decanted, centrifuged, and transferred to a holding container. Even after centrifuging for 1 hr at ≈1000 × g, the Contact #3 liquid still had a very slight white cloudiness.

A total of 86.09 g (86.42 mL) of liquid was removed from the test bottle. The D_{Liq} was 0.996 g/mL at 24.2 °C. Hot cell ambient temperature ranged from 24.2-24.3 °C during Contact #3.

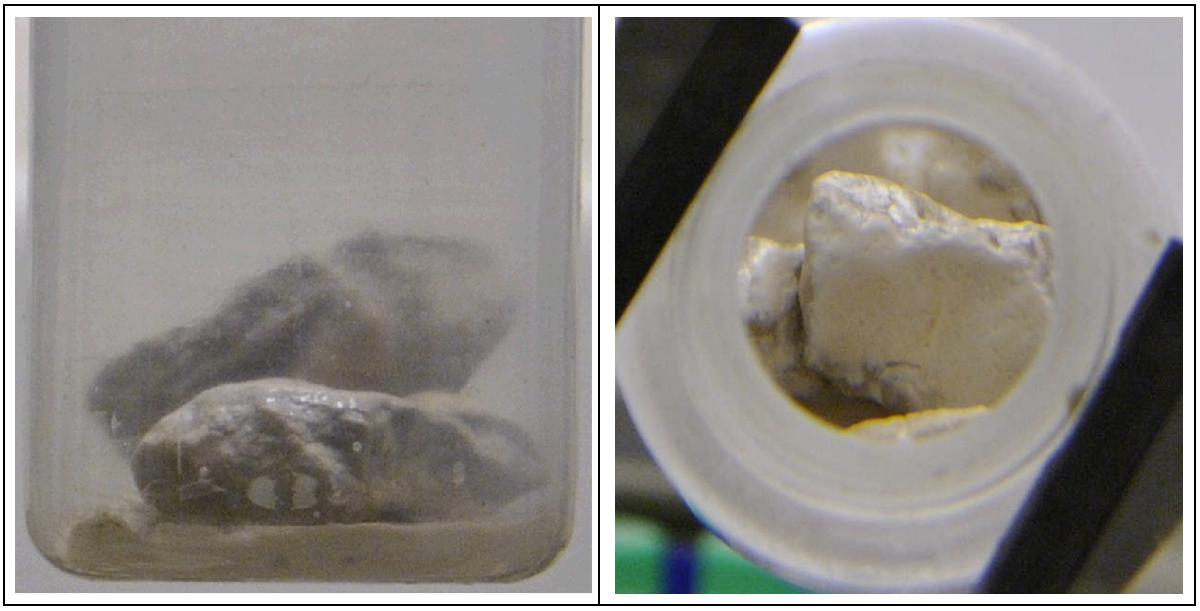
A sample of the decanted Contact #3 liquid (S12R000148) was submitted to 222-S for analysis. Prior to load-out from the 11A hot cells, this sample was diluted 24.82 % w/w with deionized water.

The wt% dissolved solids in the decanted liquid was measured by evaporating 5.753 g to dryness at 90-100 °C. The decanted Contact #3 liquor contained 0.03 wt% dissolved solids.

The total weight of wet aggregate solids in the test bottle and the damp C-Slds in the two centrifuge cones at the conclusion of Contact #3 of the water dissolution test (March 5) was

63.85 g. Photographs of the wet solids in the test bottle are shown in Figure 5-03. After drying for 2 days, prior to initiation of the caustic dissolution test, the total weight of the semi-dry solids decreased to 59.47 g.

Figure 5-03. Aggregate Solids at End of Water Dissolution Contact #3.



5.3.2 Sample Analyses

The results of physicochemical analysis of the liquids removed from the test bottle at the conclusion of each contact in the aggregate water dissolution test are summarized in Table 5-01. The analytical results in the table have been corrected to predilution values. Transcripts of the laboratory data summary reports for the diluted samples and the sample weight and density data used for the dilution corrections are included in Attachment A.

Liquid density and dissolved solids measurements were performed in the 11A hot cells by 222-S I&C personnel. All other analyses were performed in 222-S by ATL personnel. The only radiochemical analysis requested for retrieval testing liquid samples was ‘Gross alpha’.

Table 5-01. Analyses of Aggregate Water Dissolution Test Liquids.

Analyte	Units	Aggregate Water Dissolution Test Liquids		
		Contact #1	Contact #2	Contact #3
D_{Liq}	g/mL	1.010 (25.2 °C)	0.998 (24.3 °C)	0.996 (24.2 °C)
Dissolved solids (95 °C)	wt%	1.57	0.16	0.03
TGA (< 200 °C)	wt%	98.72	99.71	98.13
TGA (> 200 °C)		< 1	< 1	< 1
TIC	µg/mL	926	115	24
TIC as CO_3		4,625	576	120
TOC	µg/mL	127	73.9	< 25
ΔTOC	µg/mL	69.1	67.0	< 25
Formate	µg/mL	30	4	< 1
Acetate	µg/mL	26	4	< 1
Oxalate	µg/mL	143	16	2
Nitrite	µg/mL	1,897	201	24
Nitrate	µg/mL	958	105	21
Fluoride	µg/mL	229	25	6
Phosphate	µg/mL	3,768	445	118
Sulfate	µg/mL	192	25	5
Chloride	µg/mL	21	3	3
Sodium	µg/mL	5,841	748	165
Aluminum	µg/mL	18	12	20
Silicon	µg/mL	< 4	5	4
Phosphorus	µg/mL	1,215	146	48
P as PO_4		3,724	449	147
Sulfur	µg/mL	68	< 12	< 12
S as SO_4		204	< 38	< 38
Potassium	µg/mL	< 63	< 62	< 62
Calcium	µg/mL	< 6	< 6	< 6
Chromium	µg/mL	3	< 1	< 1
Manganese	µg/mL	< 1	< 1	< 1
Iron	µg/mL	11	2	1
Nickel	µg/mL	< 3	< 2	< 2
Uranium	µg/mL	136	20	< 12
Gross alpha	µCi/mL	1.29E-4	6.52E-5	1.16E-4

5.4 CAUSTIC DISSOLUTION

5.4.1 Test Narrative

As in the water dissolution tests, at the beginning of the aggregate caustic dissolution testing the aggregate solids were assumed to have the same chemical composition as the large-scale crushed heel solids composite. The crushed solids composite was found to contain 193,000 µg/g Al and assumed to contain 55.8 wt% gibbsite. Therefore, the 58.85-g aggregate test sample was initially assumed to contain 11.36 g (0.42 moles) of Al.

Halfway through the 10-day caustic metathesis period it was decided that it was more appropriate to treat the aggregate solids as if they were mostly gibbsite (as suggested by SPC analyses). A 58.85-g gibbsite aggregate would have contained 20.36 g (0.75 moles) of Al. This change necessitated the addition of additional caustic solution to attempt to provide 3 moles of OH for each mole of Al assumed to be present in the aggregate solids.

The large-scale and aggregate dissolution tests were run concurrently. The gibbsite metathesis and aluminate dissolution steps in the aggregate test were started and terminated at the same time as the corresponding steps in the large-scale test. The same 49.64 wt% NaOH solution prepared for the large-scale testing (Section 4.4.1.1) was used in the aggregate test.

The aggregate caustic dissolution tests were performed at ambient hot cell temperatures.

5.4.1.1 Gibbsite Metathesis: The 250-mL test bottle containing the wet solids remaining after the aggregate water dissolution testing passed directly to the caustic dissolution test.

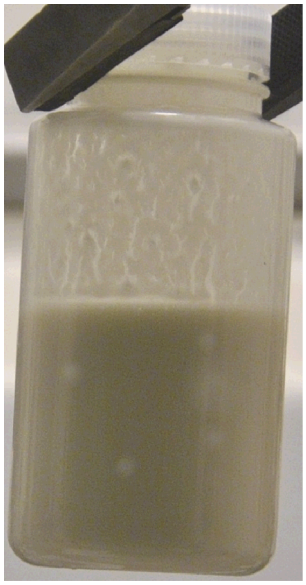
At 08:45 on March 7, 2012, 111.78 g (73.48 mL) of caustic solution was added to the aggregate test bottle. This quantity of caustic solution introduced 55.49 g of NaOH or 1.387 moles of OH to the test sample. If the aggregate solids had the same Al content as the crushed heel solids composite, this would have provided 3.30 moles of OH for each mole of Al in the aggregate solids. The test sample was then mixed continuously for 4.9 days.

At 11:00 on March 12, an additional 48.37 g of caustic solution was added to the test bottle. The total of 160.15 g (105.28 mL) of caustic solution introduced 79.50 g of NaOH or 1.988 moles of OH to the test sample. If the aggregate solids had been 58.85 g of pure gibbsite, this would have provided 2.64 moles of OH for each mole of Al in the gibbsite solids. The volume of the test bottle precluded addition of sufficient caustic solution to provide 3 moles of OH for each mole of Al. The contents of the test bottle were then mixed for an additional 4.8 days.

During the gibbsite metathesis period the hot cell ambient temperature slowly increased from 25.7 to 29.4 °C. The caustic slurry lost ≈0.5 g in weight over the 10 days.

A photograph of the caustic slurry in the test vessel at the conclusion of the gibbsite metathesis period is shown in Figure 5-04.

Figure 5-04. Test Slurry at End of Aggregate Gibbsite Metathesis.



5.4.1.2 Aluminate Dissolution: At 08:10 on March 17, 2012, 105.44 g (≈106 mL) of 4 °C deionized water was added to the caustic slurry in the test bottle. The volume of water added was ≈100% of the total volume of caustic solution added. The test bottle was returned to the mixer and tumbled for 2.0 days. Hot cell ambient temperature ranged from 29.1 to 29.4 °C during the aluminate dissolution. Over the initial 2 hrs of mixing, the appearance of the test slurry changed from off-white and totally opaque (Figure 5-04) to yellow-brown and partially translucent. A photograph of the final test slurry is shown in Figure 5-05.

At 08:00 on March 19, ≈311 g of test liquid was decanted from the test bottle, centrifuged, transferred to a holding container, and set aside.

5.4.1.3 Rinsing of Residual Solids: After the aluminate dissolution test liquid was decanted from the test bottle (on March 19), the remaining wet solids were rinsed three times with 10.59 g, 10.66 g, and 10.61 g of deionized water. In each rinse, the added water was first used to recover any centrifuged solids remaining from previous step(s) and then transferred to the test bottle. The walls of the bottle were rinsed using a disposable pipette and the solids were gently stirred with a spatula. The rinse liquid was allowed to settle for 3-5 min, withdrawn as completely as possible with the pipette, and transferred to a centrifuge cone. The liquid was centrifuged until clear, and then added to the liquid collected during the primary decanting (Section 5.4.1.2).

The liquid decanted from the test vessel combined with the three water rinses weighed 344.37 g. The D_{Liq} of the combined liquids was 1.326 g/mL at 30.0 °C.

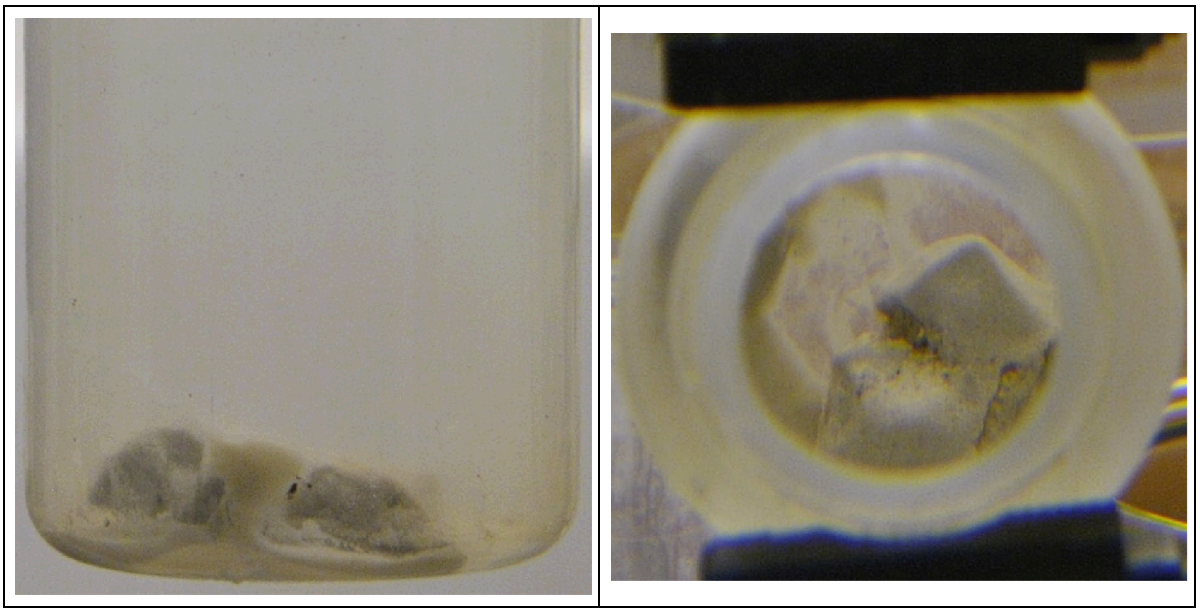
The wt% dissolved solids in the combined liquids was measured by evaporating a 6.329-g portion to dryness at 90-100 °C. The combined liquids contained 35.19 wt% dissolved solids.

Figure 5-05. Aggregate Test Slurry at End of Aluminate Dissolution.

A sample of the final aggregate test liquid (S12R000145) was submitted to 222-S for analysis. The sample was not diluted prior to transfer to 222-S.

Photographs of the wet, rinsed residual solids are shown in Figure 5-06. After these photographs were taken, the uncapped test bottle was set aside to allow the solids to air-dry.

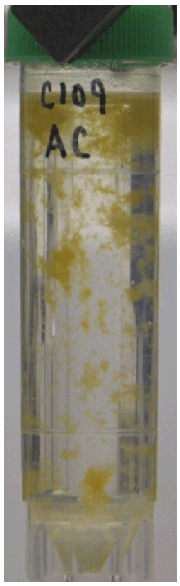
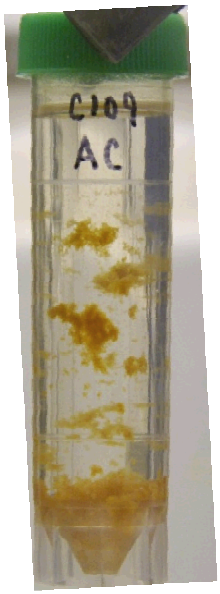
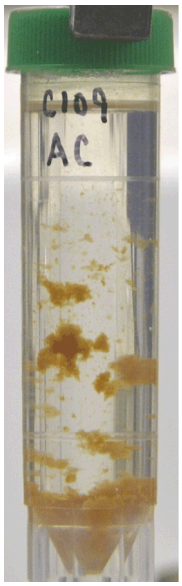
Figure 5-06. Wet, Residual Aggregate Solids Following Rinses.



5.4.1.4 Caustic Dissolution Liquor: A portion of the combined caustic dissolution/rinse liquids was transferred to a 50-mL centrifuge cone immediately after the conclusion of the water rinses on March 19. When collected, the liquid was clear, colorless, and free of solids. Within a

day, a small amount of a reddish-brown, floc- or gel- type precipitate formed in the cone. The precipitate was quite similar in appearance, but more voluminous, than that observed in the large-scale caustic dissolution test liquid (Figure 4-10). The nature of the precipitate is unknown but it did not have the appearance of precipitating gibbsite. The sample was observed periodically over the following 170 days. The only changes observed were a slight increase in the quantity of the precipitate and very slow settling. Photographs of the precipitate in the aggregate caustic dissolution test liquor are shown in Figure 5-07.

Figure 5-07. Aggregate Caustic Dissolution Test Liquid.

(a) March 26, 2012	(b) May 10, 2012	(c) September 7, 2012
		

5.4.2 Sample Analyses

The results of physicochemical analysis of the combination of the liquid decanted from the test bottle at the conclusion of the aluminate dissolution and the liquid recovered after the three water rinses are summarized in Table 5-02. Transcripts of the laboratory data summary reports for the liquid sample are in Attachment A.

Liquid density and dissolved solids measurements were performed in the 11A hot cells by 222-S I&C personnel. All other analyses were performed in 222-S by ATL personnel. No radiochemical analyses were requested for the aggregate caustic dissolution test liquid.

Table 5-02. Analyses of Aggregate Caustic Dissolution Test Liquid.

Analyte	Caustic Dissolution Test Liquid	
Specific gravity	1.326	
D_{Liq}	1.326 (30.0 °C)	g/mL
Dissolved solids (95 °C)	35.19	wt%
TGA (< 200 °C)	—	
TGA (> 200 °C)	—	
Hydroxide	87,800	µg/mL
TIC	45	µg/mL
TIC as CO_3	226	µg/mL
TOC	84	µg/mL
ΔTOC	82	µg/mL
Formate	8	µg/mL
Acetate	< 1	µg/mL
Oxalate	< 2	µg/mL
Nitrite	11	µg/mL
Nitrate	18	µg/mL
Fluoride	2	µg/mL
Phosphate	64	µg/mL
Sulfate	< 2	µg/mL
Chloride	5	µg/mL
Sodium	163,000	µg/mL
Aluminum	56,500	µg/mL
Silicon	< 120	µg/mL
Phosphorus	< 200	µg/mL
P as PO_4	< 613	µg/mL
Sulfur	< 400	µg/mL
S as SO_4	< 1,198	µg/mL
Potassium	—	
Calcium	444	µg/mL
Chromium	< 23	µg/mL
Manganese	< 12	µg/mL
Iron	28	µg/mL
Nickel	< 80	µg/mL
Zirconium	—	
Bismuth	< 400	µg/mL
Uranium	< 400	µg/mL
Gross alpha	—	

5.5 RESIDUAL AGGREGATE SOLIDS

5.5.1 Residual Solids Recovery

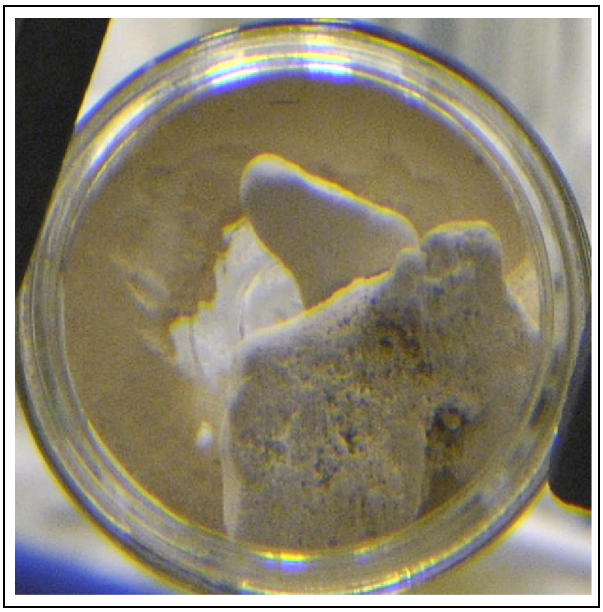
The semi-dry aggregate solids remaining at the conclusion of the retrieval dissolution tests were recovered from the test bottle on March 21, 2012. First, two relatively large solid pieces, weighing 8.67 g and 1.07 g, were removed from the test bottle and set aside.

The fine-grained solids were then transferred, as completely as possible, to a 4-oz jar using a disposable pipette and several small additions of deionized water. After settling for 1 hr, the supernatant liquid over the fine solids was completely clear. Most of this liquid was pipetted from the jar and disposed of. The jar was then covered with a fluted watch glass and set aside to allow the fine solids to dry overnight.

At 08:00 on March 22, the two solid pieces were added to the jar. After drying at hot cell ambient temperature (28 °C) for 5 days, the total weight of the residual aggregate solids was 11.58 g. A photograph of the dry solids remaining at the conclusion of the aggregate dissolution tests is shown in Figure 5-08.

Figure 5-08. Dry Residual Aggregate Solids.

(Inner diameter of mouth of jar is 4.8 cm)



5.5.2 Bulk Density

The D_{Bulk} of the air-dry residual aggregate solids was measured by water displacement following the same procedure used for the large-scale residual solids (Section 4.5.2). On April 9, 2012, the solids were transferred to one of the 50-mL centrifuge cones (RD1) and weighed. The cone was partially filled with deionized water and gently shaken and vibrated on a vortex mixer to release

all visible air bubbles. The cone was then sequentially filled to the 45-mL and 50-mL marks and weighed. The data collected during the measurement and the D_{Bulk} calculations follow.

		Cone RD1
Vol Cone at 45-mL mark	(mL)	44.73 ± 0.08
Vol Cone at 50-mL mark	(mL)	49.66 ± 0.07
Wt Residual solids	(g)	11.461
t Added H_2O	(°C)	26.1
Density Added H_2O	(g/mL)	0.996757
45-mL Mark		
Wt Added H_2O to 45-mL mark	(g)	40.376
Vol Added H_2O to 45-mL mark	(mL)	40.507
Vol Residual solids	(mL)	4.224
D_{Bulk} Residual solids	(g/mL)	2.71 ± 0.08
50-mL Mark		
Wt Added H_2O to 50-mL mark	(g)	44.748
Vol Added H_2O to 50-mL mark	(mL)	44.894
Vol Residual solids	(mL)	4.765
D_{Bulk} Residual solids	(g/mL)	2.40 ± 0.07
D_{Bulk} Residual Aggregate Solids	2.56 ± 0.11 g/mL	

Following the D_{Bulk} measurement, the wet solids were returned to the 4-oz sample jar and the added water was pipetted off as it clarified.

5.5.3 Sampling

On April 11, 2012 the residual aggregate solids were ground in a mortar and pestle until they passed through a 425-µm mesh sieve. The two larger pieces crushed with only moderate pressure (certainly with much greater ease than the starting aggregates).

A sample of the homogenized solids (S12R000191) was collected for SPC measurements. Another sample (S12R000192) was collected and submitted to 222-S for physicochemical and radiochemical analyses. All the residual aggregate solids were consumed in these two samples.

5.5.4 Solid Phase Characterization

The SPC results for the residual aggregate solids sample are included in Attachment D.

Gibbsite was the only solid phase identified in the residual solids by XRD. The diffraction patterns did not suggest that significant amounts of unidentified or amorphous solids were present.

Examination of the solids using SEM/EDX confirmed that gibbsite was the dominant phase present in the residual aggregate solids. Most of the remaining solids were fine-grained Fe/Ni-rich solids (possibly including some Al). Other minor components identified were U-rich solids (without Na), Ca/PO_4-rich solids, Fe-rich, and Fe/Pb-rich solids.

5.5.5 Physicochemical and Radiochemical Analysis

The results of physicochemical analysis of the air-dry residual aggregate solids are summarized in Table 5-03. Transcripts of the laboratory data summary reports for the analyses of the residual aggregate solids are in Attachment A.

Density and H_2O measurements were performed by 222-S I&C personnel. All other analyses were performed in 222-S by ATL personnel.

The duplicate analyses for Al (on specimens prepared by fusion in Zr crucibles) returned concentrations of 320,000 and 328,000 µg/g. Pure gibbsite contains 345,900 µg/g Al (and has a density of 2.42 g/mL). If all of the Al in the residual aggregate solids was associated with gibbsite, the aggregate residues were 92.5 to 94.8 wt% $Al(OH)_3$.

Table 5-03. Physicochemical Analysis of Residual Aggregate Solids.

Analyte	Residual Aggregate Solids		
D_{Bulk}		2.56	g/mL
Wt loss (95 °C)		—	
TGA (< 200 °C)		—	
TGA (>200 °C)		—	
H_2O (110 °C)		0.31	wt%
pH		—	
TIC		228	µg/g
TIC as CO_3		1,139	µg/g
TOC		692	µg/g
ΔTOC		—	
Formate	(W)	—	
Acetate	(W)	—	
Oxalate	(W)	—	
Nitrite	(W)	—	
Nitrate	(W)	—	
Fluoride	(W)	—	
Phosphate	(W)	—	
Sulfate	(W)	—	
Chloride	(W)	—	
Sodium	(Z)	2,933	µg/g
Aluminum	(Z)	324,000	µg/g
Silicon	(Z)	527	µg/g
Phosphorus	(A)	< 107	µg/g
P as PO_4		< 328	µg/g
Sulfur	(A)	< 214	µg/g
S as SO_4		< 641	µg/g
Potassium		—	
Calcium	(A)	532	µg/g
Chromium	(Z)	< 228	µg/g
Manganese	(A)	< 6	µg/g
Iron	(A/Z)	1,340	µg/g
Nickel	(A)	1,650	µg/g
Zirconium	(A)	< 9	µg/g
Bismuth	(A/Z)	< 10,500	µg/g
Uranium	(A)	275	µg/g

Results for radiochemical analyses of the residual aggregate solids are summarized in Table 5-04.

Table 5-04. Radiochemical Analysis of Residual Aggregate Solids.

Analyte	Residual Aggregate Solids		
Strontium-89/90	(Z)	5.08E+2	µCi/g
Technetium-99	(A)	< 9.19E-1	µg/g
Iodine-129		< 6.06E-5	µCi/g
Cesium-137	(Z)	< 4.80E-1	µCi/g
Lead-212	(Z)	—	
Lead-214	(Z)	—	
Actinium-228	(Z)	< 1.25E+0	µCi/g
Uranium-233	(Z)	< 2.10E+0	µg/g
Uranium-234	(Z)	< 5.24E-2	µg/g
Uranium-235	(Z)	1.62E+0	µg/g
Uranium-236	(Z)	< 4.19E-2	µg/g
Neptunium-237	(Z)	< 1.05E+0	µg/g
Uranium-238	(Z)	2.54E+2	µg/g
Plutonium-238	(Z)	1.66E-3	µCi/g
Plutonium-239/240	(Z)	1.29E-2	µCi/g
Americium-241	(Z)	3.02E-2	µCi/g
Curium-243/244	(Z)	< 1.15E-4	µCi/g

5.6 SUMMARY RESULTS

At the beginning of the aggregate dissolution tests, the air-dry aggregate test sample weighed 58.85 g. Solid phase characterization indicated that gibbsite was the dominant solid phase in aggregates with similar appearance and crushability.

The solids passed through a three-step water dissolution test. If D_{Bulk} of the air-dry aggregate solids was close to the density of gibbsite, 2.42 g/mL, the initial volume of the aggregate solids would have been ≈24.3 mL. In that case, the volumes of water added in the three water dissolution test contacts were ≈4.3, ≈3.4, and ≈3.6 times the initial volume of the predominantly-gibbsite aggregate solids.

The larger pieces of solids did not disaggregate during the water dissolution tests. Comparison of Figures 5-01 and 5-03 shows that other than some smoothing caused by gentle but continuous mixing, the larger pieces of aggregate solids were largely unaffected by extended contact with water.

A gibbsite metathesis reaction was then initiated by addition of 111.8 g (73.5 mL) of 49.6 wt% caustic solution to the aggregate solids. After 5 days, an additional 48.4 g (31.8 mL) of caustic was added. If the aggregate solids had been pure gibbsite, the 160.2 g of caustic solution added would have provided 2.64 moles of OH for each mole of Al present in the initial test sample. After 10 days of metathesis, dissolution of the sodium aluminate product was initiated by

addition of 105.4 g of water and allowed to proceed for 2 days. After decanting the final test solution, the residual solids were thoroughly rinsed with water and dried.

Comparison of Figures 5-01 and 5-03 with Figures 5-06 and 5-08 shows that the larger pieces of aggregate solids were significantly reduced in size during the caustic dissolution test but persisted as distinct pieces of solids (i.e., metathesis and dissolution appeared to proceed inward from the surface of the aggregate solids). The largest piece of aggregate solids in the starting test sample weighed 31.8 g whereas the larger of the two solid pieces in the residual solids weighed 8.67 g.

Overall, the water/caustic dissolution test sequence dissolved and removed 80.3 wt% (47.27 g) of the initial 58.85 g of C-109 aggregate solids leaving 19.7 wt% (11.58 g) of residual solids. Chemical and SPC analysis indicated that the residual aggregate solids were ≈94 wt% gibbsite. This overall gravimetric result was very similar to that of the large-scale water/caustic dissolution tests where 80.0 wt% of the initial composite sample was dissolved and removed.

5.6.1 Sample Analyses

In Table 5-05(a), the analyses of all the aggregate dissolution test samples have been reformatted in terms of the total weights of analytes (analyte inventories) throughout the test sequence. The weights, volumes, and densities in the first three rows of the table describe the initial aggregate solids, the liquids removed from the system in each dissolution step, and the final residual solids. In the remaining rows, the calculated weights of the analytes removed from the system in the dissolution test steps are tabulated in the third through sixth columns. The calculated weights of the analytes remaining in the residual aggregate solids are tabulated in the last column. For analytes where the analysis result was reported as '< Detection Limit', a weight of 0 g was entered.

In Table 5-05(b) the inventory values are recalculated as cumulative weights of the components removed in the dissolution steps. Since most of the Na and OH removed in the caustic dissolution step represented recovery of the added caustic, they were not included in the cumulative weights for the step.

Table 5-05. Analyte Inventories in the Aggregate Dissolution Tests. (2 sheets)

(a) Weights removed in dissolution test steps and remaining in residual solids

Analyte	Aggregate Solids	H_2O Contact #1	H_2O Contact #2	H_2O Contact #3	Caustic Dissolution	Residual Solids
Total Wt (g)	58.85	101.74	82.59	86.09	344.37	11.58
Total Vol (mL)	—	100.71	82.74	86.42	259.70	4.52
Density (g/mL)	—	1.010	0.998	0.996	1.326	2.56
	Wt (g) In	**Wt (g) Removed**				**Wt (g) In**
Dissolved solids (95 °C)	—	1.60	0.14	0.03	121.17	—
TGA (<200 °C)	—	100.44	82.35	84.48	—	—
TGA (>200 °C)	—	—	—	—	—	—
H_2O (110 °C)	—	—	—	—	—	0.04
Hydroxide	—	—	—	—	22.801 [a]	—
TIC as CO_3	—	0.466	0.048	0.010	0.059	0.013
ΔTOC	—	0.007	0.006	0	0.021	0.008
Formate	—	0.003	< 0.001	< 0.001	0.002	—
Acetate	—	0.003	< 0.001	< 0.001	0	—
Oxalate	—	0.014	0.001	< 0.001	0	—
Nitrite	—	0.191	0.017	0.002	0.003	—
Nitrate	—	0.097	0.009	0.002	0.005	—
Fluoride	—	0.023	0.002	0.001	0.001	—
Phosphate	—	0.380	0.037	0.010	0.017	—
Sulfate	—	0.019	0.002	< 0.001	0	—
Chloride	—	0.002	< 0.001	< 0.001	0.001	—
Sodium	—	0.588	0.062	0.014	42.330 [a]	0.034
Aluminum	—	0.002	0.001	0.002	14.673	3.752
Silicon	—	0	< 0.001	< 0.001	0	0.006
Phosphorus P as PO_4	—	0.122 0.375	0.012 0.037	0.004 0.013	0	0
Sulfur S as SO_4	—	0.007 0.021	0	0	0	0
Potassium	—	0	0	0	—	—
Calcium	—	0	0	0	0.115	0.006
Chromium	—	< 0.001	< 0.001	0	0	0
Manganese	—	0	0	0	0	0
Iron	—	0.001	< 0.001	< 0.001	0.007	0.016
Nickel	—	0	0	0	0	0.019
Zirconium	—	—	—	—	—	0
Bismuth	—	0	0	0	0	0
Uranium	—	0.014	0.002	0	0	0.003

[a] Most of the Na and OH removed in the caustic dissolution step represents recovery of the added caustic.

Table 5-05. Analyte Inventories in the Aggregate Dissolution Tests. (2 sheets)
(b) Cumulative weights removed in dissolution tests and remaining in residual solids

	Cumulative Wt (g) Removed				Wt (g) in Residual Solids
	H_2O Contact #1	H_2O Contact #2	H_2O Contact #3	Caustic Dissolution	
TIC as CO_3	0.466	0.513	0.524	0.582	0.013
ΔTOC	0.007	0.012	0.012	0.034	0.008
Formate	0.003	0.003	0.003	0.005	—
Acetate	0.003	0.003	0.003	0.003	—
Oxalate	0.014	0.016	0.016	0.016	—
Nitrite	0.191	0.208	0.210	0.213	—
Nitrate	0.097	0.105	0.107	0.112	—
Fluoride	0.023	0.025	0.026	0.026	—
Phosphate	0.380	0.416	0.427	0.443	0
Sulfate	0.019	0.021	0.022	0.022	0
Chloride	0.002	0.002	0.003	0.004	—
Sodium	0.588	0.650	0.664	0.664 [b]	0.034
Aluminum	0.002	0.003	0.005	14.677	3.752
Silicon	0	< 0.001	0.001	0.001	0.006
Calcium	0	0	0	0.115	0.006
Chromium	< 0.001	< 0.001	< 0.001	< 0.001	0
Iron	0.001	0.001	0.001	0.009	0.016
Nickel	0	0	0	0	0.019
Uranium	0.014	0.015	0.015	0.015	0.003
Sums	1.81	2.00	2.04	16.94	3.86

[b] Na and OH recovered in the caustic dissolution test liquid are not included in cumulative wt removed.

As in the large-scale dissolution tests, most of the highly soluble salts—carbonate (TIC as CO_3), formate, acetate, oxalate, nitrite, nitrate, sulfate, and chloride salts—were dissolved/recovered in the first water contact and then diluted/recovered in the last two water contacts. Similarly, some uranium was dissolved in the first water contact but some carried through to the residual solids.

The sodium, fluoride, and phosphate in the aggregate test sample were also essentially completely dissolved/recovered in the first water contact. This suggests that natrophosphate probably joined the highly soluble evaporite salts in a coating on the exterior of the aggregate solids. However, unlike the situation in the large-scale test, dissolution of the other highly soluble salts in the first water addition did not raise the sodium concentration sufficiently to suppress complete dissolution of the small amount of natrophosphate present.

As expected, most of the Al was recovered in the caustic dissolution. The weight of Al recovered in the caustic dissolution test liquid was 3.9 times that found in the residual solids. Most of the Ca and some of the Fe in the aggregate solids were also dissolved and recovered in the gibbsite metathesis/aluminate dissolution sequence.

Most of the Si, Fe, and Ni detected in the aggregate dissolution tests were found in the residual solids. Minor portions of Ca and U were also found in the residual aggregate solids.

5.6.2 Discussion and Analysis

In Table 5-06, the cumulative analyte inventories are recalculated in terms of potential solid phase components. The same solid phases proposed for the C-109 crushed heel solids composite (Table 3-03) are used; oxides of Si, Ca, and Cr have also been included. The cumulative wt% of each solid phase removed in the water and caustic dissolution tests and the wt% remaining in the residual aggregate solids are tabulated. The wt% values are calculated relative to the initial 58.85 g weight of the aggregate test sample.

Table 5-06. Solid Phase Inventories in the Aggregate Dissolution Tests.

Estimated wt%s of solid phases removed during dissolution tests and remaining in the residual solids (relative to the initial weight of the aggregate solids).

Solid Phase	Cumulative Wt% Removed		Wt% in Residual Solids
	Water Dissolution	Caustic Dissolution	
$Al(OH)_3$	0.02	72.1	18.4
$Na_2CO_3 \cdot H_2O$	1.84	2.04	0.05
$Na_7F(PO_4)_2 \cdot 19\ H_2O$	1.63	1.67	< 0.001
$NaNO_2$	0.54	0.54	—
CaO	< 0.001	0.27	0.015
$NaNO_3$	0.25	0.26	—
Organic C	0.021	0.057	0.014
Fe_2O_3	0.003	0.021	0.04
Na_2SO_4	0.055	0.055	< 0.001
$Na_2U_2O_7$	0.035	0.035	0.007
Na-Oxalate	0.041	0.041	—
NiO	< 0.001	< 0.001	0.04
SiO_2	0.003	0.003	0.02
Na-Formate	0.009	0.014	—
NaCl	0.007	0.011	—
Na-Acetate	0.007	0.007	—
Cr_2O_3	0.001	0.001	< 0.001
Sums	4.5	77.1	18.6

The 'Sums' row at the bottom of Table 5-06 shows that the calculated amount of material dissolved and removed based on analysis of the test liquids, 77.1 wt%, is 3.2 wt% less than the 80.3 wt% based on the weights of the initial sample and residual solids. The calculated weight of material in the residual solids based on the solid phase inventory, 18.6 wt%, is 1.1 wt% less than the 19.7 wt% gravimetric value. Given that (a) several sample handling steps were performed, (b) several analyses of solid and liquid matrices were performed, (c) only one of the

proposed solid phases was identified by direct measurement, and (d) all water and any dissolved solids used in the final processing of the residual aggregate solids were discarded without analysis, this mass balance is quite reasonable.

5.6.2.1 Water Dissolution: Table 5-06 indicates that 4.5 wt% (≈2.6 g) of the aggregate solids were removed in the water dissolution tests. Dissolution and removal of the small amounts of highly soluble salts and natrophosphate left on the aggregate solids as the liquid component of the C-109 heel wastes evaporated probably account for the majority of this loss.

5.6.2.2 Caustic Dissolution: Table 5-06 indicates that the water and caustic dissolution tests removed ≈4.5 wt% (≈2.6 g) of non-gibbsite solids from the aggregates. This implies that ≈56.2 g of gibbsite were present when the caustic dissolution test began. Approximately 10.8 g of gibbsite (18.4 wt%) remained in the residual solids. These values suggest that ≈81 wt% (≈45.4 g) of the gibbsite in the aggregates was dissolved and recovered in the caustic dissolution test. This is somewhat greater than the 69 wt% of gibbsite solids recovered in the large-scale caustic dissolution test even though the ≈2.6 OH:Al mole ratio used was smaller than the 3.0 ratio used in the large-scale test. The ≈81 wt% recovery is also greater than the 75% recovery at 25 °C predicted in WRPS-0900468. Hot cell temperatures approached 30 °C during the caustic dissolution tests on the C-109 heel solids. This increase would be expected to increase the rate and degree of completion of the metathesis/dissolution of gibbsite solids (though it would also have enhanced the recovery in the large-scale test). It is plausible that the slow tumbling of the aggregates in the caustic solution was very effective at removing sodium aluminate from the surface of the solids as it was formed, thereby continuously exposing fresh gibbsite for metathesis. The two-step caustic addition in the aggregate dissolution test (Section 5.4.1.1) may also have enhanced the overall efficiency of the metathesis reaction.

6.0 PARTICLE SIZE OF RESIDUAL SOLIDS

The particle sizes in the residual solids from the large-scale C-109 heel solids dissolution tests were measured in two steps. The residual solids were first separated into three size fractions using 1.4-mm and 710-µm sieves. The PSD in the <710-µm fraction of residual solids was then measured by a light-scattering-based technique.

It should be noted that the distribution of particle sizes in the residual solids was strongly influenced by the procedures used to prepare the test composite and to execute the dissolution tests. The results presented here may have little relationship to the distribution of particle sizes characterizing solids remaining in C-109 after actual retrieval operations in the field.

6.1 SIEVE ANALYSIS

6.1.1 Apparatus

Two sieves were used in the sieve analysis: a U.S. Standard No. 14 sieve and a U.S. Standard No. 25 sieve. Each sieve was 3 in. in diameter with stainless steel frame and wire cloth. Key dimensions of the sieve wire cloths taken from ASTM E11, *Standard Specification for Wire Cloth and Sieves for Testing Purposes* are:

Sieve	Nominal Opening	Variation in Average Opening	Opening Dimension Exceeded by <5% of Openings	Maximum Opening Dimension
U.S. Standard No.14	1.4 mm	± 0.050 mm	1.505 mm	1.565 mm
U.S. Standard No. 25	710 µm	± 30 µm	775 µm	815 µm

The inner diameter of the neck of a 16-oz wide-mouth jar (the receiver jar) was widened slightly so that the bottom of the 710-µm test sieve slid into it easily and was supported on the top rim of the jar.

A 125-mL plastic wash bottle was used to direct a stream of deionized water on the solids to facilitate the sieve separation.

6.1.2 Sieving Operations

The sieve analysis was begun on March 28, 2012 in the 11A hot cells. At the conclusion of the D_{Bulk} measurements on the residual solids (Section 4.5.2), all the solids (80.36 g) and the water used in the density measurements were contained in two 50-mL centrifuge cones. The sieves were weighed and then the 710-µm sieve was placed on the receiver jar and the 1.4-mm sieve was stacked on the 710-µm sieve.

The residual solids were transferred to the wire cloth of the 1.4-mm sieve in small portions. When all the residual solids had been transferred, the liquid from the density measurement was used to begin washing the solids through the wire cloth of the sieves. When the density measurement liquid was exhausted, the solids were gently manipulated on the 1.4-mm sieve cloth with a small spatula while rinsing frequently with a stream of water from the 125-mL wash bottle. When all <1.4-mm solids had been washed through the 1.4-mm sieve, it was carefully removed from the stack and set aside on a plastic dish to air-dry. This process was then repeated for the solids retained on the 710-µm sieve. The receiver jar containing the <710-µm solids and sieve liquid was capped and set aside.

After drying overnight (March 29), the sieves and retained solids were weighed. The dry solids on each sieve were then recovered and transferred to separate centrifuge cones. A total of 16.22 g of solids were retained on the 1.4-mm sieve and 16.06 g were transferred to a cone. A total of 0.98 g was retained on the 710-µm sieve and 0.84 g was transferred to a cone.

On April 2, the supernatant sieve liquid in the receiver jar was still opaque. Most of the liquid was transferred to centrifuge cones and centrifuged for 1-2.5 hrs at ≈1000 × *g* until clear. The clear liquid was transferred to a jar and set aside. Near the end of the liquid recovery, transfer of the <710-µm solids to a pre-weighed 4-oz jar (Jar 20172) was begun. When liquid was needed to facilitate the solids transfer, the centrifuged sieving liquid was used. After the transfer was completed on April 3, the sample jar contained 90.1 g of the <710-µm solids slurry. A fluted watch glass was then placed on the sample jar so the wet solids could begin to dry.

On April 4, no free liquid was observed in Jar 20172, and the surface of the solids was just beginning to show signs of dryness. The jar was capped and set aside. On April 9, the wet solids, which had the consistency of a thin paste, were thoroughly mixed by gentle stirring with a small spatula. A 2.122 g portion of the wet solids was transferred to a sample vial for determination of total solids. Another 2-g portion of the wet solids, sample S12R000199, was transferred to a sample vial for PSD analysis (Section 6.2). The sample jar was then recapped.

Evaporation of the 2.122-g portion of wet <710-µm solids to dryness indicated that the paste contained 65.98 wt% solids. Based on this solids content, 18.767 g of the wet <710-µm residual solids were transferred to a sample vial (S12R000198) for settling tests (Section 7.0).

The jar containing the remaining wet <710-µm residual solids was then covered with a fluted watch glass until the solids dried to a constant weight at hot cell ambient temperature. On April 16, 2012, samples of the air-dry solids were taken for SPC measurements and laboratory analysis as described in Section 4.5.4. The remaining <710-µm residual solids were set aside for archive.

6.1.3 Sieve Separation Results

Photographs of the air-dry >1.4-mm and >710-µm solids and of the <710-µm solids just before the PSD sample was withdrawn are shown in Figure 6-01.

The weights of the dry solids retained on the 1.4-mm and 710-µm sieves were measured directly as described in Section 6.1.2. The total weight of the <710-µm solids reported was determined by difference. The weight of material retained in the D_{Bulk} cones and on the sieves following recovery of the retained solids was <0.05 g; these solids were included in the <710-µm size fraction. The results for the sieve analysis are summarized in Table 6-01.

Figure 6-01. Residual Solids Sieve Fractions.

(Inside diameter of sieves is 7.5 cm. Outside diameter of jar is 6 cm.)

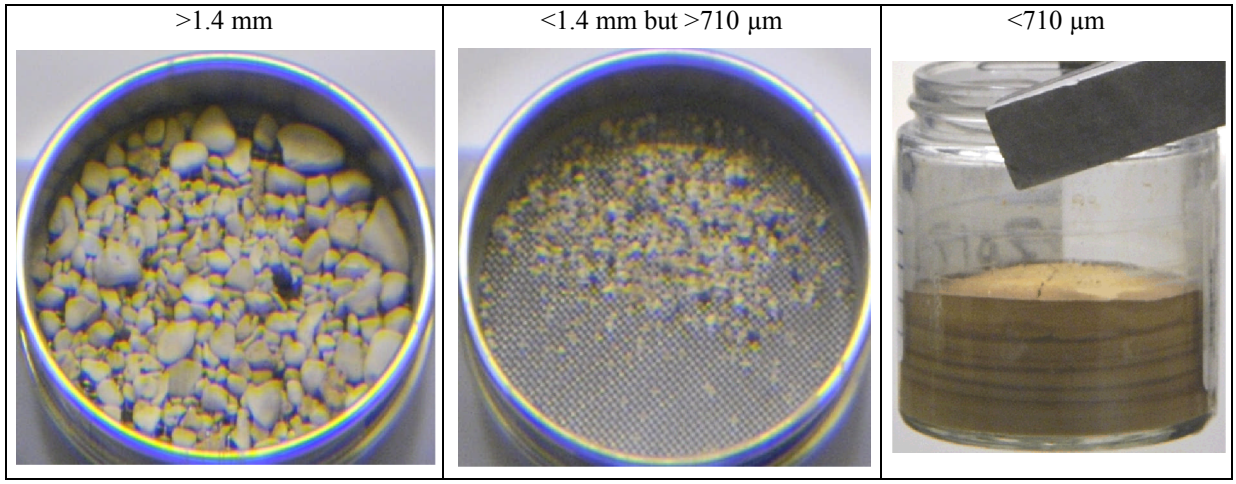


Table 6-01. Results of Sieve Analysis on Residual Solids.

Size Class	Wt (g)	Wt%
>1.4 mm	16.22	20.18
<1.4 mm but >710 µm	0.98	1.22
<710 µm	63.16	78.60
Total residual solids	80.36	100.00

6.2 LIGHT-SCATTERING MEASUREMENTS

The PSD within the <710-µm sieve fraction of the C-109 residual solids was measured using a light-scattering-based technique. The full details of the measurement, complete data results including raw data files, relevant limitations, and quality control measurement results are presented in Attachment C.

6.2.1 Light-Scattering Particle Size Distribution Measurements

The PSDs in the <710-µm residual solids from the large-scale dissolution tests on the C-109 crushed heel solids composite were measured in triplicate. Each of the triplicate PSD specimens was a separate portion removed from sample S12R000199 (Section 6.1.2). Each of the triplicate PSD measurements consisted of a set of three consecutive PSD runs. Run 1 was initiated as quickly as possible after transfer of the sample solids to 210 mL of reagent water circulating in the PSD analyzer. The minimum circulation pump and stirrer speeds consistent with reliable PSD measurements were utilized. The Run 1 results represent PSDs measured in the minimum time and under the lowest shear conditions possible.

In Run 2, initiated immediately following completion of Run 1, the speed settings of both the circulation pump and stirrer in the analyzer were kept constant. The specimen slurry was circulated through the analyzer for 2 min prior to initiation of the second light-scattering measurement. The Run 2 results represent PSDs where dissolution or dispersion resulting from extended exposure to water under relatively low shear forces may be evidenced.

In Run 3, initiated immediately following the completion of Run 2, the pump and stirrer speeds were unchanged but the specimen slurry underwent low-power ultrasonic treatment (sonication) for 2 min prior to initiation of the third (and final) light-scattering measurement. The Run 3 results represent PSDs after exposure of the specimen solids to mild-to-moderate shear forces.

6.2.2 Histograms and %-Undersize Curves

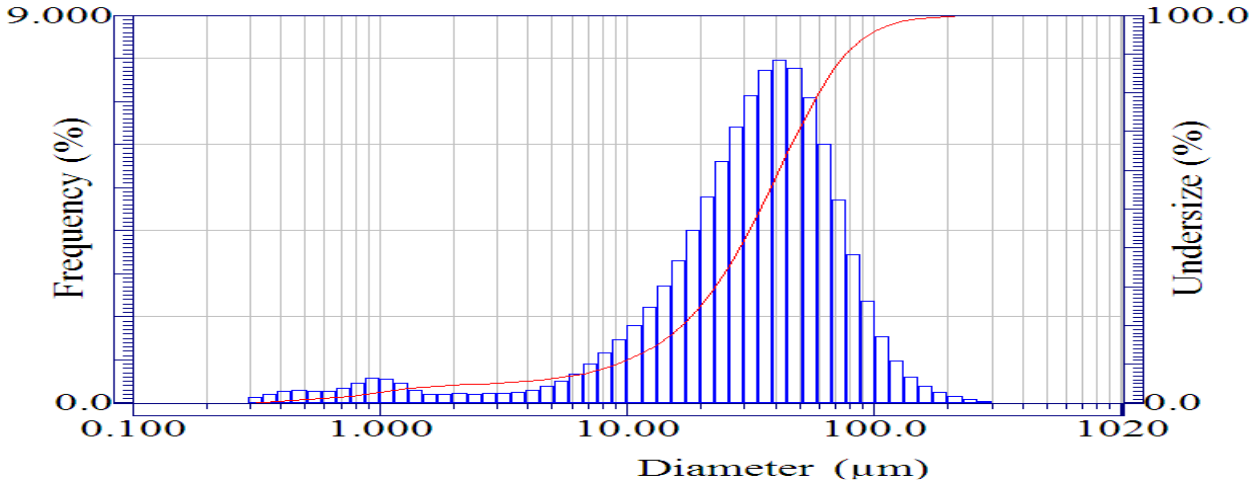
The volume-based histograms and cumulative %-undersize curves displaying the results of the PSD measurements on the <710-µm C-109 residual solids are presented in Figure 6-02. The data files for corresponding PSD runs on the triplicate specimens were combined into 'average' data files. This averaged data is displayed in the figure.

The header for each PSD run in Figure 6-02 displays the stirrer speed setting, the circulation pump speed setting, whether the specimen slurry was sonicated for 2 min, and the average duration of the run (in minutes). For Run 1, the average duration includes the approximate time required to introduce the PSD specimen into the analyzer sample tank.

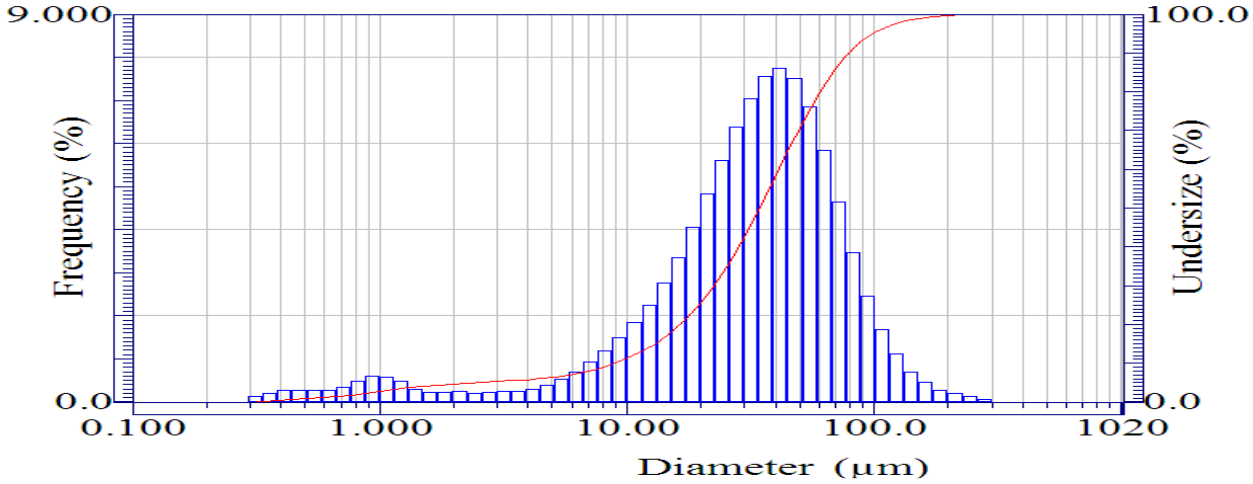
(The analyzer software allows the primary volume-based distributions to be recalculated as number-based distributions. The converted number-based distributions are included in Attachment C for comparison with the volume-based data.)

Figure 6-02. Volume-based Particle Size Distributions in <710-µm Residual Solids.

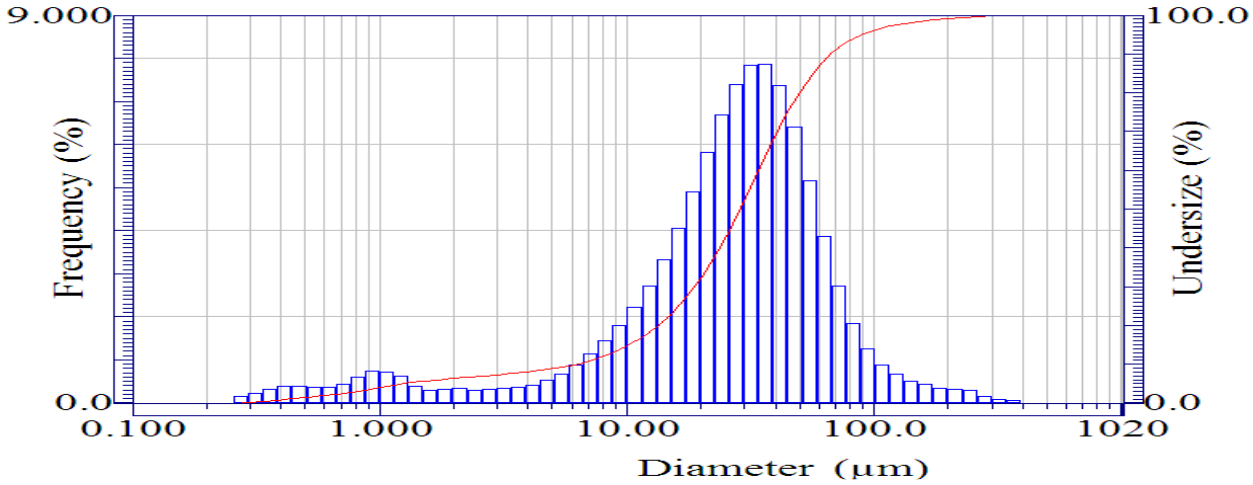
Run 1 — Stir Speed: S1-3 Pump Speed: P3-5 Sonication: None Duration: 2.4 min



Run 2 — Stir Speed: S3 Pump Speed: P5 Sonication: None Duration: 3.2 min



Run 3 — Stir Speed: S3 Pump Speed: P5 Sonication: 2.0 min Duration: 3.4 min



6.2.3 Cumulative %-Undersize Data

The particle diameters associated with five pre-selected points on the cumulative %-undersize curves describing the volume-based PSDs for the <710-µm C-109 residual solids are presented in Table 6-02. The particle diameters associated with 10-, 25-, 50-, 75-, and 90 %-undersize cut points are presented in the table. (These values are commonly reported as d10, d25, d50, d75, and d90 particle diameters, respectively.)

Table 6-02. Diameter vs. %-Undersize for <710-µm Residual Solids.

PSD Run	Diameters of Particles (µm) at Volume-based %-Undersize Cut Points				
	10%	25%	50%	75%	90%
1	9	20	35	54	77
2	9	20	35	54	79
3	6	16	29	45	66

Comparison of the PSD histograms and the diameter vs. %-undersize data for PSD Run 1 and Run 2 indicate that significant quantities of loosely bound agglomerates were not present in the <710-µm residual solids (i.e., the PSDs are virtually identical). A small shift to smaller particle diameters is observed in the Run 3 data suggesting that a small portion of the particulates in the 10-100 µm size range may have been weakly bound aggregates.

The PSD data can also be formatted as %-undersize vs. diameter data where the cumulative percentages of sample particles with diameters less than selected spherical equivalent particle diameters are presented. The volume-based PSD data for the <710-µm C-109 residual solids is presented in this format in Table 6-03[6]. The particle diameters selected in Table 6-03 are related to settling rate cut points used in the settling tests discussed in Section 7.0.

Table 6-03. %-Undersize vs. Diameter for <710-µm Residual Solids.

PSD Run	Vol% of Solids from Particles with Diameters Smaller than Cut Points				
	11.4 µm	25.8 µm	56.4 µm	139 µm	710 µm
1	12.6	34.9	77.1	98.7	100.0
2	12.9	35.4	76.7	98.4	100.0
3	16.8	43.9	84.9	98.0	100.0

[6] The PSD measurements indicated that the residual solids contained essentially no particulates with equivalent spherical diameters ranging from ≈300 to 710 µm. As seen in Figure 7-03(e), the fraction of the solids that settled faster than 0.43 in./s (with estimated diameters >139 µm) that were isolated in the settling rate test appear to contain particles in this size range. The settling test sample was two to three orders of magnitude larger than the PSD measurement specimens and would be expected to sample this size class of particles more representatively. Even so, particulates with diameters ranging from 300-710 µm appear to represent ≈1%, or less, of the residual solids.

6.3 COMBINED PARTICLE SIZE DISTRIBUTION

Sieve analysis is nominally a particle-counting technique based on an actual particle size dimension, the minimum cross section. Light scattering, on the other hand, is an ensemble technique based on particle volume and is reported in terms of an equivalent spherical diameter (Attachment C). For these reasons, combination of the two data sets is generally inadvisable.

However, SPC analysis and, to a lesser extent, chemical analysis indicated that the residual solids from the C-109 dissolution tests were predominantly gibbsite; therefore, the wt% sieve analysis data and the vol% light-scattering data should be approximately equivalent. Microscopy (Attachment D) also tended to show that the particles in the residual solids, while certainly not spherical, were generally not greatly elongated or flattened; therefore, the cross section sieve data and the equivalent spherical diameter light-scattering data are also approximately equivalent. If it is assumed that all the particulates in the <710-µm residual solids have approximately the same density and scatter light in approximately the same manner, the two particle size data sets can be combined. With the proviso that the result is only a semi-quantitative description, the sieving results (Table 6-01) and light-scattering results (Table 6-03) have been combined in the calculated particle size distribution shown in Table 6-04.

Table 6-04. Combined Particle Size Distribution in Residual Solids.

Particle Size Class	Wt%	
> 1.4 mm	20.2	
< 1.4 mm but > 710 µm	1.2	
	Before Sonication	**After Sonication**
< 710 µm but > 139 µm	1.2	1.6
< 139 µm but > 56.4 µm	17.0	10.3
< 56.4 µm but > 25.8 µm	32.5	32.2
< 25.8 µm but > 11.4 µm	17.7	21.3
< 11.4 µm	10.1	13.2

The before-sonication distribution is based on the light-scattering Run 2 results; the after-sonication distribution is based on the light-scattering Run 3 results.

7.0 SETTLING BEHAVIOR OF RESIDUAL SOLIDS

A set of settling tests was performed on a subsample of the wet <710-µm C-109 residual solids recovered at the conclusion of the large-scale dissolution tests.

The first test was a settling behavior test. A slurry containing ≈1 vol% of the residual solids in water was prepared and then allowed to settle for 22 hrs. The qualitative appearance of the test slurry was observed and documented in a series of photographs.

A series of four settling rate tests were then performed, starting with the same sample slurry. The purpose of the rate tests was to determine the weights of the C-109 residual solids that settled from a well-mixed aqueous slurry with an initial ≈1 vol% solids at rates exceeding 0.004 in./s, 0.02 in./s, 0.1 in./s, and 0.5 in./s.

As for the particle size measurements, it should be noted that the settling behavior of the residual solids was strongly influenced by the procedures used to prepare the test composite and to execute the dissolution and settling tests. The results presented here may have little relationship to the settling behavior exhibited by solids remaining in C-109 after actual retrieval operations in the field.

7.1 SETTLING TEST PROCEDURE

7.1.1 Settling Test Sample

The settling test sample (S12R000198) was taken from the wet paste of <710-µm residual solids collected during wet sieving (Sections 4.5.4 and 6.1.2). The weight of the sample of wet solids transferred to the sample vial was 18.767 g. The paste/slurry contained 65.98 wt% solids.

The settling test sample contained 18.767 × 0.6598 = 12.382 g of dry residual solids. At the measured density of the residual solids, 2.21 g/mL (Section 4.5.2), the test sample contained 5.60 mL of dry solids.

The volume of the test slurry prepared for the settling tests was 544.1 mL. Quantitative transfer of the solids in the test sample to the settling column resulted in a test slurry containing 1.03 vol% solids (2.25 wt% solids).

7.1.2 Test Apparatus

A glass settling column was used in the settling tests. The total length of the column was 50 cm; the internal diameter was 4 cm. The settling column had screw-cap closures on both ends to facilitate introduction and recovery of test samples. On the bottom of the column, the screw cap backed up a rubber plug that sealed the column and facilitated controlled recovery of settled

solids. The column was mounted in a stand and clamp system that allowed it to be rotated end-over-end about an axis perpendicular to the long axis of the column.

The distance from the '0.0-cm' mark ≈5 cm down from the top of the column to the top of the plug sealing the bottom of the column was 44.5 cm. The volume of the 44.5-cm working depth of the column was 544.1 mL. A settling distance scale, extending 43 cm down from the 0.0-cm mark, was inscribed on the outside of the column.

An additional mark was applied to the column 39.4 cm down from the 0.0-cm mark. The volume of the settling column below this mark was 62.2 mL. Finally, a series of thick (≈5 mm high) black marks were applied to the side of the settling column opposite the settling distance scale at 5-cm intervals to provide a high-contrast reference to aid in estimating the opacity of the test slurries.

A vacuum aspiration system was set up with the settling column. This system allowed the rapid transfer of liquid from the settling column to a capture flask. The suction wand inserted into the settling column during aspiration facilitated control of the time-to-depth of liquid removal.

7.1.3 Settling Test Procedure

The settling tests were performed as follows:

Settling Behavior

1. The bottom plug and screw cap were assembled on the bottom of the settling column.
2. All the solids in the test sample were quantitatively transferred into the column using rinses with reagent water as required.
3. The column was filled to the 0.0-cm mark with reagent water, and the top screw cap was attached.
4. The column was rotated end-over-end several times until the sample solids were as well dispersed as possible. The final half-rotation brought the top of the settling column from the 6 o'clock to the 12 o'clock position.
5. Immediately after bringing the column upright (column top to 12 o'clock) a test timer was started.
6. The settling solids were observed and photographed periodically for 22 hrs.

Settling Rates

7. The settling behavior test slurry was remixed by rotating the column end-over-end several times.
8. Immediately after bringing the settling column upright, the test timer was started and the top screw cap was removed.
9. The upper 39.4 cm of the test slurry was removed by vacuum aspiration. The rate of removal was controlled so that the 39.4-cm depth was reached after 1 hr:4 min:38 s, the time interval defined by a settling rate of 0.004 in./s.

Care was taken that the liquid removal rate did not exceed the target solids settling rate at any time/depth during the aspiration. The actual final aspiration depth and time were recorded.

10. The water and suspended solids aspirated from the column were transferred to a labeled, pre-weighed container and set aside.
11. The column was refilled to the 0.0-cm mark with reagent water (for a second time), and the top screw cap was attached.
12. Steps 7-10 were repeated. The aspirated water and suspended solids were combined with those from Step 10.
13. The column was refilled to the 0.0-cm mark with reagent water (for a third time), and the top screw cap was attached.
14. Steps 7-10 were repeated. The aspirated water and suspended solids were combined with those from Step 12.
15. The three cycle fill/mix/settle/decant set in Steps 7-14 was repeated. In this set, the target settling period preceding slurry aspiration in each cycle was 12 min:56 s, the time interval defined by a settling rate of 0.02 in./s.
16. The three cycle fill/mix/settle/decant set in Steps 7-14 was repeated. In this set, the target settling period preceding slurry aspiration in each cycle was 2 min:35 s, the time interval defined by a settling rate of 0.10 in./s.
17. The three cycle fill/mix/settle/decant set in Steps 7-14 was repeated for a final time. In this set, the target settling period preceding slurry aspiration in each cycle was 31 s, the time interval defined by a settling rate of 0.5 in./s.
18. The bottom screw cap was removed, and the bottom of the column was oriented within a labeled, pre-weighed container.
19. The bottom plug was carefully dislodged from the column and captured in the container along with ≈62 mL of water and fast settling sample solids. Recovery of the solids was facilitated by rinsing the inner walls of the column with water from a squeeze bottle.
20. The plug was retrieved from the sample jar, and any recovered solids were rinsed back into the jar with water.
21. The sample of fast settling solids was set aside.
22. After settling for several days, most of the clear supernatant liquid in the four aspirated slurry samples and the sample of fast settling solids was carefully pipetted off and disposed of as waste.
23. The five samples of solids segregated by settling rate were transferred to a laboratory oven and dried at ≈60 °C.
24. The five fractions of dry solids were weighed and photographed.

The average temperature near the face of the hood in 222-S/Room 1C where the settling tests were performed was 22 °C.

While attempting to recover the dry solids for temporary archive, the fume hood where the settling tests were performed was contaminated. As part of the recovery from the contamination event, the five samples of solids were disposed of as waste.

7.2 SETTLING BEHAVIOR TEST RESULTS

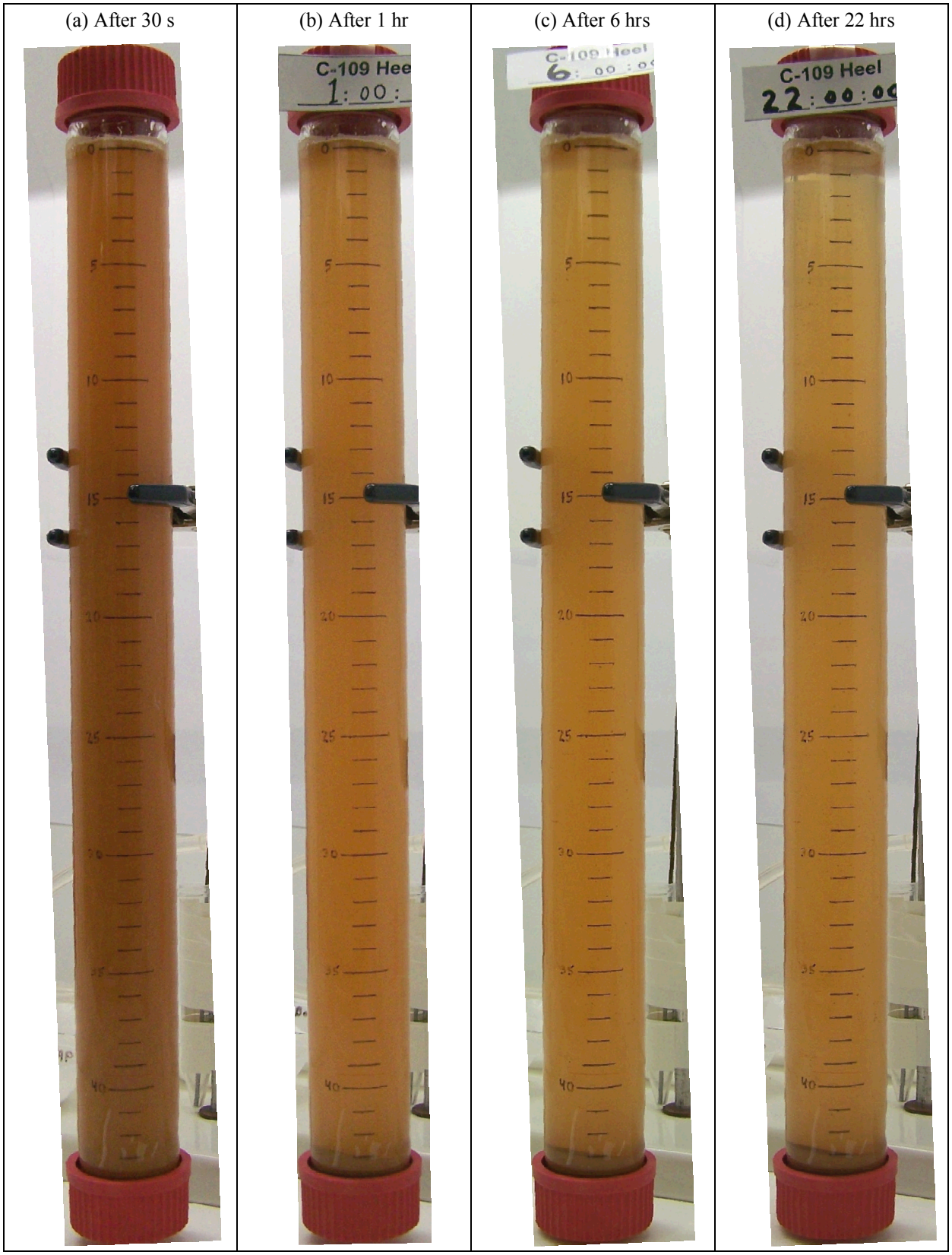
Photographs of the settling column at the beginning of the test (after ≈30 s) and after 1, 6, and 22 hrs of settling are shown in Figure 7-01.

The test slurry was completely opaque at the beginning of the test. After 1 hr, the solids loading in the slurry had visibly decreased, and an accumulating layer of settled solids was visible at the bottom of the column. For the remaining 21 hrs of the test, the solids loading continued to decrease slowly and uniformly throughout the length of the column. After 22 hrs, the solids loading in the slurry was significantly less than in the starting slurry, but some of the <710-µm residual solids remained in suspension. A 2-3 mm layer of clear liquid was observed at the top of the column, and the solids concentration appeared to increase uniformly with depth in the remainder of the column.

Calculations of the predicted settling velocities ($V_{Settling}$) for the C-109 residual solids in 1.03 vol% suspension in water at 22 °C are shown in Attachment E.1. The 2.21 g/mL D_{Bulk} for the residual solids (Section 4.5.2) was assumed to hold for all the solids. Calculated Stokes settling velocities (V_{Stokes}) indicate that particles with diameters ≥ 2.7 µm would settle ≥ 38.8 cm in the test slurry in 22 hrs while particles with diameters of 1 µm would only settle up to 5.3 cm in 22 hrs. The PSD measurements (Section 6.2) indicated that 5-7 vol% of the particulates in the <710-µm residual solids had diameters <2.7 µm; 3-4 vol% had diameters <1 µm.

The second set of decantings in the settling rate tests (Section 7.3) were completed at 1500 hrs on May 17. At that time, 1.18 g (9.5 wt%) of the residual solids had been removed from the test slurry. The column was refilled with water and the 11.20 g of remaining solids were redispersed; the suspension immediately after agitation appeared as shown in Figure 7-02(c). After ≈17 hrs (08:00 on May 18), the liquid in the upper 42 cm of the column was clear and colorless. This settling behavior indicated that less than 10% of the residual solids settled from suspension at a rate slower than ≈1 in./hr.

Figure 7-01. Settling of <710-µm Residual Solids in Water.



The settling behavior observations showed that while the smallest particles in the residual solids settled very slowly from the 1.03 vol% aqueous slurry, ≈90% of the solids settled at rates ≥ 1 in./hr. The distinct interface between settling solids and supernatant liquid characteristic of hindered settling[7] was not observed in the test slurry nor was there any visible evidence of flocculation. In general, the observed behavior was that of small, low-to-medium density solids settling from a suspension with weak (if any) interparticle or particle-liquid interactions.

7.3 SETTLING RATE TEST RESULTS

The settling rate tests were intended to determine the quantity of C-109 residual solids that settled from aqueous suspension at specified rates. The 1.03 vol% solids slurry observed in the settling behavior test passed directly to the settling rate test series.

The tests were based on the method of classification by decantation or fractional decantation. As described in Section 7.1.3, 4 sets of 3 decant cycles each were performed. In each cycle, the C-109 residual solids were suspended in water, allowed to settle for a set time, and then decanted to a set depth.

A different target settling time was used in each set. The target settling times were the times required for particulates starting at the surface of the aqueous slurry and settling at rates of 0.004 in./s (0.01 cm/s), 0.02 in./s (0.05 cm/s), 0.1 in./s (0.25 cm/s), or 0.5 in./s (1.27 cm/s) to reach a depth of 39.4 cm.

The average temperature near the face of the hood in 222-S/Room 1C where the settling tests were performed was 22 °C.

7.3.1 Settling Rate Test Results

Photographs of the test sample slurry at the beginning of each set of 3 decant cycles are shown in Figure 7-02.

Photographs of the solids recovered during the settling rate tests are shown in Figure 7-03. Photographs (a)-(d) show the solids recovered from the liquids that were decanted from the settling column in the four sets of decantings. Photograph (e) shows the solids that were recovered from the bottom 5.1 cm of the settling column after the final decant cycle (Decant 12). All the solids were allowed to air dry in a fume hood prior to being photographed. The samples were then dried in a small laboratory oven at ≈60 °C for several hours prior to being weighed.

[7] The terms 'hindered settling' or 'mass subsidence' generally describe the settling of an initially homogeneous and relatively concentrated suspension of particles at a constant rate with a well-defined interface between supernatant fluid and settling particles. All the particles settle at the same rate, regardless (within limits) of size, and the suspension does not change in concentration until the top of the accumulating layer of settled solids is reached.

Figure 7-02. Test Slurries During Settling Rate Tests.

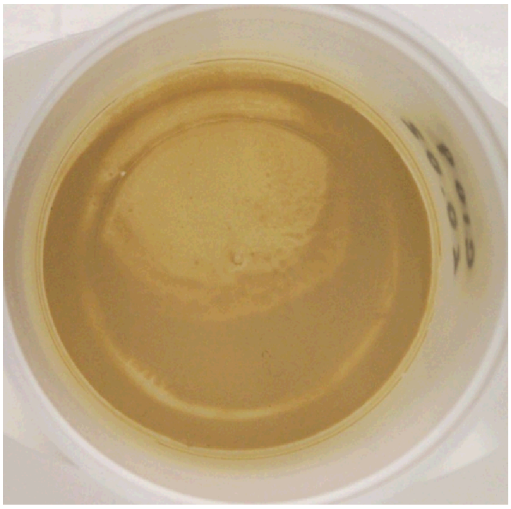
(a) Before Decant 1 12.38 g solids	(b) Before Decant 4 12.02 g solids	(c) Before Decant 7 11.20 g solids	(d) Before Decant 10 3.48 g solids

Figure 7-03. Solids Recovered in Settling Rate Tests.

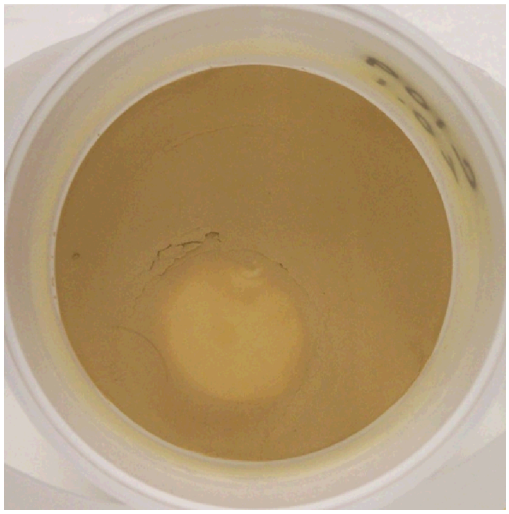
(The inside bottom diameter of the sample containers is 6.5 cm)

(a)

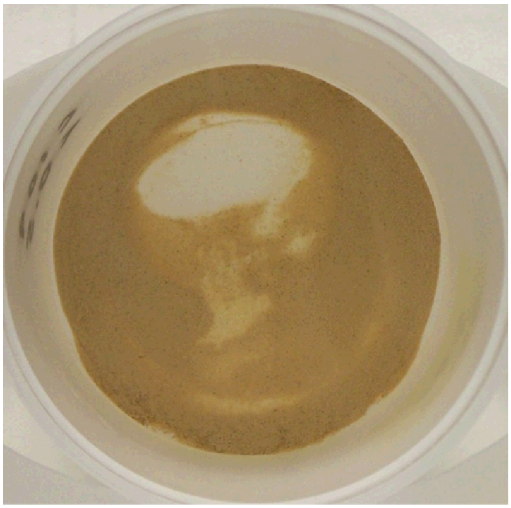
(b)



(c)



(d)



(e)

(a) <0.0039 in./s (0.366 g)

(b) >0.0039 but <0.020 in./s (0.811 g)

(c) >0.020 but <0.096 in./s (7.724 g)

(d) >0.0.096 but <0.43 in./s (3.009 g)

(e) >0.43 in./s (0.471 g)

Quantitative observations recorded during the settling rate tests and the calculated test results are presented in Table 7-01.

Table 7-01. Settling Rate Test Results.

	Target $V_{Settling}$ = 0.004 in./s			Target $V_{Settling}$ = 0.02 in./s		
	Decant 1	**Decant 2**	**Decant 3**	**Decant 4**	**Decant 5**	**Decant 6**
Decant depth (cm)	39.4	39.5	39.3	39.4	39.5	39.3
Decant Time (s)	3,878	3,876	4,101	777.2	776.4	778.2
$V_{Settling}$ (cm/s)	0.0102	0.0102	0.00958	0.0507	0.0509	0.0505
Average $V_{Settling}$ (cm/s)	0.00998			0.0507		
(in./s)	0.0039			0.020		
Decanted Solids (g)	0.366			0.811		
Adjusted (g)	0.367			0.812		
Solids wt%	2.96			6.56		

	Target $V_{Settling}$ = 0.1 in./s			Target $V_{Settling}$ = 0.5 in./s			
	Decant 7	**Decant 8**	**Decant 9**	**Decant 10**	**Decant 11**	**Decant 12**	**Final Solids**
Decant depth (cm)	39.6	39.5	39.4	39.4	39.5	39.4	
Decant Time (s)	166.3	160.9	156.8	41.3	37.2	31.7	
$V_{Settling}$ (cm/s)	0.238	0.246	0.251	0.954	1.06	1.24	
Average $V_{Settling}$ (cm/s)	0.245			1.09			
(in./s)	0.096			0.43			
Decanted Solids (g)	7.724			3.009			
Adjusted (g)	7.734			3.002			
Recovered Solids (g)	—			—			0.471
Adjusted (g)							0.466
Solids wt%	62.47			24.25			3.77

In decant cycles 3, 10, 11, and 12 more time was taken to aspirate the supernatant suspensions to the target depth than was defined by the target $V_{Settling}$. In cycles 3 and 10, the extended times were due to procedural errors; in cycles 11 and 12, the extended times were due to experimental difficulties in completing the aspirations in 31 s.

In Table 7-01, 'Decanted Solids' are the dry weights of the solids that were recovered from the aspirated suspensions (Figures 7-03[a]-[d]); 'Recovered Solids' is the dry weight of the solids that remained in the column after the final decanting (Figure 7-03[e]). As discussed in Attachment E.2, a portion of the solids settling slower than the target $V_{Settling}$ will always be left in the column because they start each decant cycle below the 39.4-cm decant depth. These small corrections, which are relatively independent of the actual properties of the test sample, have been applied in the 'Adjusted' decanted and recovered solids weights. The adjusted weights were used to calculate the wt%s of solids in the five $V_{Settling}$ defined classes.

As discussed in Section 6.1, the residual solids from the large-scale dissolution tests were separated into three size classes by sieving. The sieve separation indicated that 21.4 wt% of the

residual solids were >710 µm in size. Only the 78.6 wt% of the residual solids that were <710 µm in size were sampled for the settling tests. A sample of the residual solids containing 12.382 g of <710-µm solids (as the settling test sample did) would have also contained 3.371 g of >710-µm solids. These solids would have been recovered in the class of particulates settling at >0.43 in./s. Incorporation of these coarse solids into the results of the settling rate tests presented in Table 7-01 yields the overall settling test results shown in Table 7-02.

Table 7-02. Settling Rates of Residual Solids from Large-scale Dissolution Tests.

$V_{Settling}$ Class	Wt (g)	Wt%
< 0.0039 in./s	0.367	2.33
> 0.0039 in./s but < 0.020 in./s	0.812	5.15
> 0.020 in./s but < 0.096 in./s	7.734	49.10
> 0.096 in./s but < 0.43 in./s	3.002	19.06
> 0.43 in./s	3.837	24.36

7.3.2 Measurement Bias

As discussed in Attachment E.2, a large number of decant cycles might be required before all particulates settling slower than a target $V_{Settling}$ are removed from a sample (certainly more than 3 decantings). For example, only 24% of any particulates settling at 90% of a target $V_{Settling}$ would be removed from a test sample in 3 decant cycles. It is not possible to predict the amount of slower settling solids that are not recovered in a limited number of decantings (but are corecovered with faster settling solids) without additional sample information. However, observations of the settling test suspensions, the recovered solids, and the general settling behavior allow the general measurement bias to be estimated.

Solids settling at <0.0039 in./s: Biased low. Some of these solids (perhaps a relatively large amount) were almost certainly left in the column after the completion of Decant 3.

Solids settling at >0.0039 in./s but <0.020 in./s: Probably biased low. Because the $V_{Settling}$ targets were separated by a factor of five, nearly all the particulates settling at <0.0039 in./s that were left in the column after Decant 3 were incorporated in the solids recovered in Decants 4-6. However, this positive bias was probably more than offset by the quantity of solids settling <0.020 in./s that were left in the column after the completion of Decant 6.

Solids settling at >0.020 in./s but <0.096 in./s: Probably biased high. Again, nearly all the solids settling at <0.020 in./s left in the column after Decant 6 were incorporated in the solids recovered in Decants 7-9. Note the very similar appearance of the solids recovered in Decants 4-6 (Figure 7-03[b]) and Decants 7-9 (Figure 7-03[c]). In this case, the quantity of solids settling <0.096 in./s that were left in the column after Decant 9 probably did not offset the positive bias. The significant change in appearance of the test suspensions at the beginning of Decant cycles 7 and 10 (Figures 7-02 [c] and [d]) suggest that this was the case.

Solids settling at >0.096 in./s but <0.43 in./s: Neutral bias. The appearance of the test slurry (Figure 7-02[d]) suggests that only a relatively small quantity of solids settling at <0.096 in./s remained in the column at the beginning of Decant 10. The appearance of the recovered solids (Figures 7-03 [d] and [e]) suggests that the particulates settling at <0.43 in./s were significantly finer grained than those settling at >0.43 in./s. This would result in a more complete separation with 3 decantings and a more complete incorporation of the slower settling solids in this class. The overall bias resulting from the combination of these relatively small positive and negative inputs is difficult to estimate.

Solids settling at >0.43 in./s: Biased high. Though the amount is believed to be small, some solids settling at <0.43 in./s were left in the column after Decant 12 and recovered in this class of the fastest settling particulates.

7.3.3 Comparison to Particle Size Results

The PSD and settling test samples were collected from the same parent material at approximately the same time and, presumably, contained solids with similar distributions of phase, size, and density. The results of the settling rate tests and the PSD measurements (Section 6.2) can be compared if (a) nearly all of the particulates in the <710-µm residual solids have approximately the same density, (b) the approximate density of the particulates is known, (c) the particulates in the residual solids are approximately spherical, and (d) the particles settled freely (or nearly so) without significant particle-particle and/or particle-liquid chemical or electrostatic interactions.

The residual solids were determined to be mostly gibbsite. The D_{Bulk} values of 2.21 g/mL for the large-scale residual solids and 2.56 g/mL for the aggregate residues bracket the 2.42 g/mL reference density for gibbsite and varied over a relatively small range. Microscopic examination of the residual solids did not indicate the presence of significant amounts of elongated or plate-like solids (i.e., significantly non-spherical particles). Therefore the results of the settling test and the PSD measurements can be compared with caution.

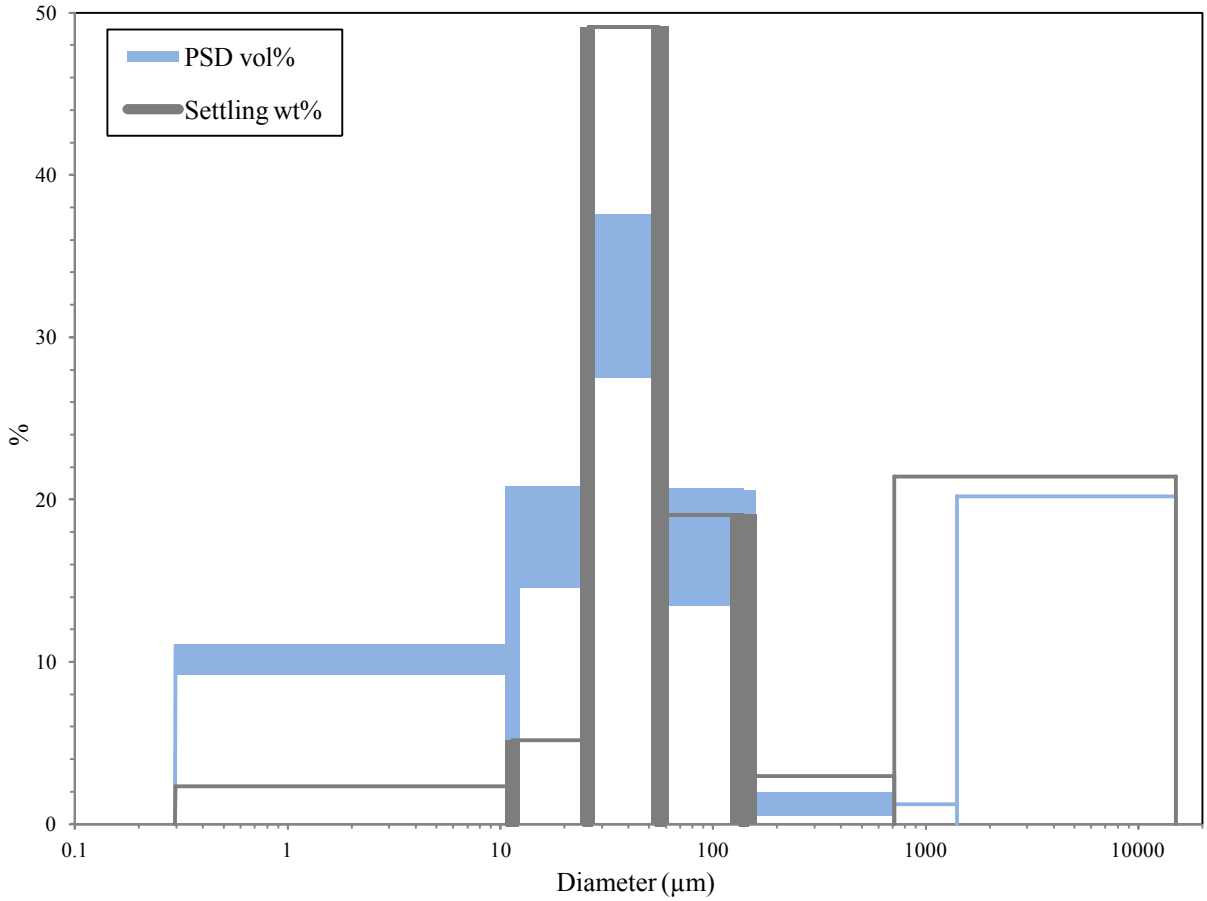
First, the four $V_{Settling}$ cut points in the settling rate tests were used to calculate equivalent spherical particle diameters (Attachment E.1). These calculations were completed using solid densities of 2.21, 2.42, and 2.56 g/mL. The actual settling times and estimated solids concentrations in each decant cycle were used in the calculations. The summarized results of these calculations are:

Average $V_{Settling}$	0.0039 in./s	0.020 in./s	0.096 in./s	0.43 in./s
	0.00998 cm/s	0.0507 cm/s	0.245 cm/s	1.09 cm/s
Equivalent Spherical Diameters				
Low	10.5 µm	24.0 µm	52.2 µm	120 µm
High	12.3 µm	27.4 µm	60.6 µm	158 µm

The settling rate test data combined with the sieve test data is plotted as a six-bin histogram in Figure 7-04. The width of the bin verticals defined by the $V_{Settling}$ cut points display the range of calculated particle diameters associated with each.

The PSD Run 2 measurement (Figure 6-02), where the test slurries were stirred and pumped for several minutes but not sonicated, were selected for comparison. Using the estimated, $V_{Settling}$-based, particle diameter cut points, the averaged PSD Run 2 data combined with the sieve test data was reformatted as a seven-bin histogram and the range of vol%s of the particulate solids belonging in each bin calculated. This is the histogram displayed in light-blue in Figure 7-04.

Figure 7-04. Comparison of Settling Rate and Particle Size Test Results.



The general shapes of the histograms based on the settling rate wt% data and the PSD vol% data are similar. The settling rate wt% bin values and the PSD vol% bin values differ significantly; however, close agreement would probably have been fortuitous and expected only under very specific circumstances. It is of note that the deviations of the settling rate data from the PSD data correspond with the predicted measurement biases discussed in Section 7.3.2. Generally, the PSD measurements and the settling rate tests appear to provide consistent descriptions of the same population of particulate solids.

8.0 QUALITY ASSURANCE

8.1 LABORATORY ANALYSES

Quality control measurements required to ensure the quality of the physicochemical analyses of samples generated during completion of this test program are specified in ATS-MP-1032, *222-S Laboratory Quality Assurance Project Plan*, and in ATL-MP-1011, *ATL Quality Assurance Project Plan for 222-S Laboratory*. The results of all quality control measurements performed in conjunction with the 222-S chemical analyses are included in the transcripts of the analysis results presented in Attachment A.

8.2 CHARACTERIZATION TESTS

8.2.1 Supporting Data

Raw data and test observations supporting this report are recorded in notebook HNF-N-723 1, "222-S Process Chemistry: Tank Waste Testing and Characterization". Test data and observations and other supporting data and documentation will also be maintained in project file 'C109 Heel Solids' until final wrap-up of the project. At that point the project file will be scanned into the Integrated Document Management System (IDMS) and can be accessed in IDMS at:

Enterprise \ Managed Information \ Electronic Records \ Tank Operations/WRPS \ 222S Laboratory \ Analytical Data Packages-Non-Sensitive

Nine, single-use Excel spreadsheets were used to process the test data and generate the test results, tables, and plots presented in this report. The spreadsheets and release forms documenting their verification/validation are registered as System 3478 in the Hanford Information Systems Inventory (HISI).

8.2.2 Repeat Tests

As discussed in Section 3.3.2, all the D_{Liq} values reported for test sample liquids were the average of duplicate measurements.

As discussed in Section 6.2 and in Attachment C, three sets of light-scattering-based PSD measurements were completed on separate portions of the <710-µm residual solids from the C-109 heel large-scale dissolution testing (Sample S12R000199). The PSD data returned by each of the triplicate measurements was very comparable.

8.2.3 Standards

Light-scattering-based measurements of the PSDs of certified particle size standards were performed before sample measurements began and after they were completed. The results of the standard measurements are presented in Section 6.2 of Attachment C. The measured mean particle diameters were within ±10% of the certified values for all standards.

8.2.4 Calibrations

8.2.4.1 Gravimetric: The majority of the quantitative measurements made during the characterization of the C-109 heel solids were gravimetric. The in-tolerance status of each analytical balance used during the test program was verified before use or daily, whichever was less frequent (ATS-LO-140-008, "222-S Laboratory Routine Use and Quality Assurance for Analytical Balances at 222-S Laboratory Complex"). The calibration verification measurements were recorded on Balance Calibration Verification Check Sheets. Copies of the check sheets are included in the project file.

8.2.4.2 Volumetric: Accurate volumes were key components of four measurement results presented in this report.

D_{Bulk} of Composite Solids: The total volume of the pycnometer jar fabricated to measure D_{Bulk} of the C-109 heel solids (Section 3.5) was measured by repeated filling with reagent water and weighing. Sixteen calibration measurements were completed using water at temperatures ranging from 5.9-37.5 °C.

The calculated average volume of the pycnometer used in the D_{Bulk} measurement of the air-dry C-109 heel solids composite was 485.06 ± 0.17 mL.

D_{Liq}: The densities of test liquids were measured by filling two of three Class-A, 25-mL volumetric flasks with test liquid and then weighing. The reported Class-A tolerance for the volume of these flasks is ±0.03 mL at 20 °C. The nominal 25-mL flask volumes were used to calculate the reported D_{Liq} values.

Flasks A and B were paired for most of the D_{Liq} measurements. The density of reagent water was measured 17 times in each flask at temperatures ranging from 18-46 °C. For Flask A, the difference between the calculated density and the reference density for water ranged from –0.0043 g/mL to –0.0019 g/mL and averaged –0.0031 ± 0.0008 g/mL. For Flask B, the difference between the calculated and reference densities of water ranged from –0.0035 g/mL to +0.0011 g/mL and averaged –0.0005 ± 0.0013 g/mL.

Flask C replaced Flask B when it was broken late in the test program and was paired with Flask A in the last three D_{Liq} measurements. The density of reagent water was measured three times in Flask C at temperatures ranging from 22-23 °C. The difference between the calculated and reference densities of water ranged from –0.0015 g/mL to –0.0013 g/mL.

Based on the check measurements using reagent water, the average D_{Liq} values in this report have an average error of –0.2 %. This estimated error was not used to adjust the calculated and reported values.

D_{Bulk} Residual Solids: The D_{Bulk} of the residual solids from the dissolution tests were measured by water displacement in two 50-mL polypropylene centrifuge cones (Sections 4.5.2 and 5.5.2). In each measurement, the cones containing the test solids were sequentially filled to 45- and 50-mL volume marks with reagent water at a known temperature.

The actual volumes of the empty cones at the two fill marks were measured by filling with reagent water and weighing. Three sets of measurements were made for each cone using water at temperatures ranging from 22.3 to 22.7 °C. The calculated average cone volume at each fill mark was used to calculate a volume displaced by a test sample. The average of the two displaced volumes was then used to calculate the reported D_{Bulk} values.

The calculated cone volumes at the 45- and 50-mL marks for Cone RD1 were 44.73 ± 0.08 mL and 49.66 ± 0.07 mL. The calculated cone volumes at the 45- and 50-mL marks for Cone RD2 were 44.51 ± 0.05 mL and 49.38 ± 0.04 mL.

Settling Test Column: The column used in the settling tests was fabricated for a previous study (74A10-WSC-08-152, "Results of Testing Performed to Characterize Tank 241-S-112 Heel Solids"). The working volume of the settling column was determined at that time in a fashion similar to that described for the pycnometer jar. Three calibration measurements using reagent water at 21.7 °C established the working volumes of the settling column to be:

0.0-cm mark to bottom of column	544.1 mL
39.4-cm mark to bottom of column	62.2 mL

8.3 DEVIATIONS FROM TEST PLAN

The C-109 heel solids characterization and retrieval testing described in this report was executed, in general, as described in the client-approved test plan (LAB-PLN-11-00006). However, the following deviations from the test plan or quality issues were identified during the testing.

a. No photographs of the as-received C-109 heel solids samples were taken (or retained). This deficiency was caused by poor definition/control of the interface/transition between ATL and Washington River Protection Solutions early in the test program. Impact on results of the test program was minimal.

b. The contents of the aggregate test bottle leaked/spilled during the water dissolution test. The attempt to execute the aggregate dissolution tests in a hot cell with only one operational manipulator did not allow the tightness of the lid to be verified and actually contributed to its loosening. Impact on the results of the aggregate dissolution testing was minimal.

c. The aggregate dissolution testing (particularly the caustic dissolution test) was very loosely defined. The specifics of this test, which was a late 'what-if' addition to the test scope, changed continuously throughout execution. The last requested change (assuming the aggregate solids were nearly pure gibbsite) could not be fully addressed given the physical dimensions of the test apparatus. Though (a) the displayed flexibility in the test planning was desirable, (b) the client communications were quite good, and (c) the basic goals of the testing were addressed, considerable extra time and effort were required.

d. In three of the twelve settling rate decanting cycles, the time taken to aspirate test liquids from the settling column was significantly longer than specified. In one case, poor focus on work activity was the root cause; in the other two, a late personnel change at a critical test stage was the root cause. Though the target settling rates in the test plan were not attained, the overall goals of the settling rate testing were met.

e. No SEM/EDX data was returned from SPC measurements on the <710-µm residual solids (sample S12R000201). This sample represented the majority of the dissolution test residues. The combination of chemical analysis and XRD was able to tentatively identify that 85-88% of the residual solids were gibbsite. Tentative identification of the remaining 12-15 % of the solids is unsupported by documented measurements.

f. Completion and issue of this report was not timely. This report will be issued after the recent completion of the retrieval of the C-109 heel solids. Several presentations of interim test results over the course of the testing ameliorated the impact of this delay on field operations.

9.0 REFERENCES

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ATTACHMENT A

LABORATORY ANALYSIS DATA

ATTACHMENT A – LABORATORY ANALYSIS DATA

Transcripts of the data summary reports (DSRs) for the analyses of liquid and solid samples generated during the characterization and testing of the C-109 heel solids are presented in this attachment. All analyses were performed at 222-S by ATL personnel.

Table A-1. Contents.

Test Sample	Sample Group	Sample number(s) (S12R000-)	Page A-
Drained Liquids Composite	20120059	-008	6
Crushed Solids Composite – Sample 1		-002, -003, -004	8
Crushed Solids Composite – Sample 2		-005, -006, -007	9
Large-scale Dissolution Tests			
Water Dissolution - Contact 1 Liquid	20120383	-149	10
Water Dissolution - Contact 2 Liquid		-150	12
Water Dissolution - Contact 3 Liquid		-151	14
Caustic Dissolution - Final Liquid	20120471	-175	16
Residual Solids - <710 µm	20120602	-200, -206, -207, -208, -209	17
Residual Solids - >710 um		-197, -202, -203, -204, -205	19
Aggregate Dissolution Tests			
Water Dissolution - Contact 1 Liquid	20120383	-146	21
Water Dissolution - Contact 2 Liquid		-147	23
Water Dissolution - Contact 3 Liquid		-148	25
Caustic Dissolution - Final Liquid	20120471	-174	27
Residual Solids	20120602	-192, -193, -194, -195	28

The measurements and analyses performed were specified in the SAP (RPP-PLAN-47927) and the laboratory test plan (LAB-PLN-11-00006). The analyses performed are summarized in Table A-2.

Table A-2. Sample Identifications and Analyses Completed. (2 sheets)

Test Sample(s)	Sample Preparations	Analyses
Drained Liquids Composite	None – Direct analysis	Specific gravity TGA (percent water) TIC/TOC IC (inorganic and organic acid anions) ICP-AES (total metals) Gross alpha
Heel Solids Composite Sample 1 Sample 2	None – Direct analysis	TGA (percent water) TIC/TOC
	Water leach	pH (10:1 w/w H_2O:sample) IC (inorganic and organic acid anions)
	KOH fusion – Zr crucible	ICP-AES (total metals)
Large-scale Water Dissolution Contact 1 Liquid Contact 2 Liquid Contact 3 Liquid	None – Direct analysis	TGA (percent water) TIC/TOC IC (inorganic and organic acid anions) ICP-AES (total metals) Gross alpha
Large-scale Caustic Dissolution Final Liquid	None – Direct analysis	Specific gravity TGA (percent water) Hydroxide TIC/TOC IC (inorganic and organic acid anions) ICP-AES (total metals) Gross alpha
Large-scale Dissolution <710-µm Residual Solids >710-µm Residual Solids	None – Direct analysis	TGA (percent water) TIC/TOC GEA (^{129}I)
	Water leach	IC (inorganic and organic acid anions)
	Acid digest	ICP-AES (total metals)
	Acid digest	ICP-MS (^{99}Tc)
	KOH fusion – Zr crucible	ICP-AES (total metals) $^{89/90}$Sr (separation and β-counting) GEA (^{137}Cs and opportunistic isotopes) ICP-MS (Actinides: ^{233}U, ^{234}U, ^{235}U, ^{236}U, ^{237}Np, and ^{238}U) AEA (^{238}Pu, $^{239/240}$Pu, ^{241}Am)

Table A-2. Sample Identifications and Analyses Completed. (2 sheets)

Test Sample(s)	Sample Preparations	Analytes
Aggregate Water Dissolution Contact 1 Liquid Contact 2 Liquid Contact 3 Liquid	None – Direct analysis	TGA (percent water) TIC/TOC IC (inorganic and organic acid anions) ICP-AES (total metals) Gross alpha
Aggregate Caustic Dissolution Final Liquid	None – Direct analysis	Specific gravity Hydroxide TIC/TOC IC (inorganic and organic acid anions) ICP-AES (total metals)
Aggregate Dissolution Residual Solids	Direct	TIC/TOC GEA (^{129}I)
	Acid digest	ICP-AES (total metals)
	Acid digest	ICP-MS (^{99}Tc)
	KOH fusion – Zr crucible	ICP-AES (total metals) $^{89/90}$Sr (separation and β-counting) GEA (^{137}Cs and opportunistic isotopes) ICP-MS (Actinides: ^{233}U, ^{234}U, ^{235}U, ^{236}U, ^{237}Np, and ^{238}U) AEA (^{238}Pu, $^{239/240}$Pu, ^{241}Am)

Table Notes:

AEA	Alpha energy analysis
GEA	Gamma energy analysis
IC	Ion chromatography
ICP-AES	Inductively coupled plasma-atomic emission spectrometry
ICP-MS	Inductively coupled plasma-mass spectrometry
TGA	Thermogravimetric analysis
TIC/TOC	Total inorganic carbon/total organic carbon by silver-catalyzed persulfate oxidation

Abbreviations and codes used throughout the data summary tables are summarized in Table A-3.

Table A-3. Abbreviations and Codes in Data Summary Tables.

Header	A	Sample Preparation Code
	Std %-Rec	Standard percent recovery
	RPD	Relative Percent Difference (between duplicate measurement results)
	Spk %-Rec	Spike percent recovery
	Qual Flags	Codes for qualifiers for reported results
Sample Preparation Codes	(Blank)	Direct analysis of sample
	W	Water leach
	A	Acid digest
	Z	KOH/KNO_3 fusion in Zr crucible
Qualifier Codes	a	Laboratory Control Sample outside range
	b	Matrix Spike/Matrix Spike Duplicate outside range
	B	Found in blank
	c	RPD outside range
	I	Indirect calibration
	J	Estimated
	U	Less than detection limit
	Y	Comment in downloaded data (included at end of DSR)

C-109 Heel Solids: Drained Liquid Composite (2 sheets) **Group: 20120059** **DSR: 26-Jan-2012 13:48:46**

Sample Number	A	Analyte	Units	Std %-Rec	Blank	Result	Duplicate	Average	RPD	Spk %-Rec	Detection Limit	Count %-Error	Qual Flags
S12R000008		Gross alpha	uCi/mL	100	<2.39E-05	2.19E-03	2.16E-03	2.17E-03	1.57	80.3	1.23E-05	6.45	
S12R000008		Fluoride	ug/mL	105	<1.61E-03	891	893	892	0.213	104.7	1.61	n/a	
S12R000008		Glycolate	ug/mL	105	<9.37E-03	<9.37	<9.37	n/a	n/a	106.3	9.37	n/a	U
S12R000008		Acetate	ug/mL	105	<6.04E-03	396	398	397	0.327	108.1	6.04	n/a	
S12R000008		Formate	ug/mL	105	<4.67E-03	384	385	384	0.385	106.7	4.67	n/a	
S12R000008		Chloride	ug/mL	104	<9.98E-03	266	266	266	0.0527	106.5	9.98	n/a	
S12R000008		Nitrite	ug/mL	104	<0.0192	2.50E+04	2.50E+04	2.50E+04	0.172	103	48.0	n/a	
S12R000008		Sulfate	ug/mL	105	<0.0187	2.72E+03	2.70E+03	2.71E+03	0.642	104.9	18.7	n/a	
S12R000008		Oxalate	ug/mL	105	<0.0231	2.21E+03	2.20E+03	2.20E+03	0.549	103.6	23.1	n/a	
S12R000008		Bromide	ug/mL	103	<0.0580	<58.0	<58.0	n/a	n/a	104.1	58.0	n/a	U
S12R000008		Nitrate	ug/mL	105	<0.0208	1.28E+04	1.28E+04	1.28E+04	0.0234	104.6	20.8	n/a	
S12R000008		Phosphate	ug/mL	105	<0.0167	3.03E+04	3.02E+04	3.03E+04	0.324	104	41.8	n/a	
S12R000008		Aluminum	ug/mL	93.8	<0.0300	<15.0	<15.0	n/a	n/a	96.5	15.0	n/a	U
S12R000008		Bismuth	ug/mL	105	<0.100	<50.0	<50.0	n/a	n/a	107	50.0	n/a	U
S12R000008		Calcium	ug/mL	100	<0.0500	<25.0	<25.0	n/a	n/a	97.3	25.0	n/a	U
S12R000008		Chromium	ug/mL	98.7	<5.00E-03	54.2	53.6	53.9	1.02	98.4	2.50	n/a	
S12R000008		Copper	ug/mL	101	<5.00E-03	10.5	10.3	10.4	2.79	101	2.50	n/a	J
S12R000008		Iron	ug/mL	99.1	<5.00E-03	182	177	180	2.48	96.9	2.50	n/a	
S12R000008		Potassium	ug/mL	106	<0.500	398	320	359	21.6	94.9	250	n/a	J
S12R000008		Manganese	ug/mL	98.2	<3.00E-03	<1.50	<1.50	n/a	n/a	98.7	1.50	n/a	U
S12R000008		Molybdenum	ug/mL	98.3	<0.0200	13.5	12.0	12.7	12.0	98.2	10.0	n/a	J
S12R000008		Sodium	ug/mL	103	<0.100	8.80E+04	9.44E+04	9.12E+04	7.00	101	100	n/a	
S12R000008		Nickel	ug/mL	99.8	<0.0200	50.6	48.0	49.3	5.38	98.6	10.0	n/a	BJ

C-109 Heel Solids: Drained Liquid Composite (2 sheets)

Sample Number	A	Analyte	Units	Std %-Rec	Blank	Result	Duplicate	Average	RPD	Spk %-Rec	Detection Limit	Count %-Error	Qual Flags
S12R000008		Phosphorus	ug/mL	100	<0.0500	1.05E+04	1.06E+04	1.06E+04	0.465	102	50.0	n/a	
S12R000008		Sulfur	ug/mL	99.5	<0.100	1.09E+03	1.08E+03	1.08E+03	1.14	96.8	50.0	n/a	
S12R000008		Silicon	ug/mL	97.7	<0.0300	<15.0	15.5	n/a	n/a	97.9	15.0	n/a	U
S12R000008		Tin	ug/mL	102	<0.0300	21.6	<15.0	n/a	n/a	99.8	15.0	n/a	J
S12R000008		Thorium	ug/mL	94.0	<0.0500	<25.0	<25.0	n/a	n/a	91.9	25.0	n/a	U
S12R000008		Uranium	ug/mL	95.7	<0.100	1.95E+03	1.89E+03	1.92E+03	3.02	88.8	50.0	n/a	
S12R000008		Specific gravity	None	101.6	n/a	1.172	1.172	1.172	0.0	n/a	1.00E-03	n/a	
S12R000008		Percent water	%	95.9	n/a	72.6	80.8	76.7	10.7	n/a	0.01	n/a	
S12R000008		Total organic carbon	ug/mL	98.2	<20.0	2.84E+03	n/a	n/a	n/a	n/a	100	n/a	
S12R000008		Total inorganic carbon	ug/mL	98.0	<7.00	1.26E+04	n/a	n/a	n/a	n/a	35.0	n/a	

C-109 Heel Solids: Solids Composite: Sample 1 **Group: 20120059** **DSR: 26-Jan-2012 13:48:46**

Sample Number	A	Analyte	Units	Std %-Rec	Blank	Result	Duplicate	Average	RPD	Spk %-Rec	Detection Limit	Count %-Error	Qual Flags
S12R000002		pH	None	n/a	n/a	11.8	11.8	11.8	0.0	n/a	0.01	n/a	
S12R000002		Percent water	%	100	n/a	18.0	n/a	n/a	n/a	n/a	0.01	n/a	
S12R000002		Total organic carbon	ug/g	105	<20.0	2.15E+03	2.06E+03	2.10E+03	4.28	111	124	n/a	
S12R000002		Total inorganic carbon	ug/g	98.2	<7.00	1.76E+03	1.73E+03	1.74E+03	1.72	98.0	43.4	n/a	
S12R000003	Z	Aluminum	ug/g	93.8	<1.36E+03	1.94E+05	1.87E+05	1.91E+05	3.83	94.4	1.36E+03	n/a	
S12R000003	Z	Bismuth	ug/g	105	<4.54E+03	<4.54E+03	<4.36E+03	n/a	n/a	105	4.54E+03	n/a	U
S12R000003	Z	Calcium	ug/g	100	<2.27E+03	<2.27E+03	<2.18E+03	n/a	n/a	99.4	2.27E+03	n/a	U
S12R000003	Z	Chromium	ug/g	98.7	<227	<227	<218	n/a	n/a	99.5	227	n/a	U
S12R000003	Z	Iron	ug/g	99.1	<227	1.68E+03	1.74E+03	1.71E+03	3.64	98.6	227	n/a	J
S12R000003	Z	Manganese	ug/g	98.2	<136	<136	<131	n/a	n/a	99.7	136	n/a	U
S12R000003	Z	Sodium	ug/g	103	<4.54E+03	9.22E+04	8.75E+04	8.99E+04	5.24	92.2	4.54E+03	n/a	
S12R000003	Z	Nickel	ug/g	99.8	<908	<9.08E+02	1.29E+03	n/a	n/a	100	908	n/a	U
S12R000003	Z	Phosphorus	ug/g	100	<2.27E+03	3.14E+04	3.00E+04	3.07E+04	4.68	100	2.27E+03	n/a	
S12R000003	Z	Sulfur	ug/g	99.5	<4.54E+03	<4.54E+03	<4.36E+03	n/a	n/a	99.9	4.54E+03	n/a	U
S12R000003	Z	Silicon	ug/g	97.7	<1.36E+03	<1.36E+03	<1.31E+03	n/a	n/a	97.1	1.36E+03	n/a	U
S12R000003	Z	Thorium	ug/g	94.0	<2.27E+03	<2.27E+03	<2.18E+03	n/a	n/a	93.9	2.27E+03	n/a	U
S12R000003	Z	Uranium	ug/g	95.7	<4.54E+03	<4.54E+03	<4.36E+03	n/a	n/a	96.2	4.54E+03	n/a	U
S12R000004	W	Fluoride	ug/g	106	<1.61E-03	1.07E+04	1.09E+04	1.08E+04	1.91	-1946	13.0	n/a	
S12R000004	W	Glycolate	ug/g	105	<9.37E-03	<1.90	<1.88	n/a	n/a	89.6	1.90	n/a	U
S12R000004	W	Acetate	ug/g	106	<6.04E-03	142	97.1	120	37.6	88.8	1.22	n/a	c
S12R000004	W	Formate	ug/g	106	<4.67E-03	126	117	121	7.60	103.5	0.945	n/a	
S12R000004	W	Chloride	ug/g	105	<9.98E-03	57.8	55.6	56.7	3.90	106.2	2.02	n/a	
S12R000004	W	Nitrite	ug/g	107	0.0577	4.97E+03	4.95E+03	4.96E+03	0.422	94.6	3.88	n/a	
S12R000004	W	Sulfate	ug/g	106	<0.0187	626	616	621	1.64	104.1	3.78	n/a	
S12R000004	W	Oxalate	ug/g	106	<0.0231	1.03E+03	9.33E+02	9.82E+02	9.92	86.6	4.67	n/a	
S12R000004	W	Bromide	ug/g	104	<0.0580	<11.7	11.9	11.8	1.77	104.4	11.7	n/a	U
S12R000004	W	Nitrate	ug/g	106	<0.0208	2.89E+03	2.86E+03	2.88E+03	0.808	102.2	4.21	n/a	
S12R000004	W	Phosphate	ug/g	106	<0.0167	1.10E+05	1.13E+05	1.11E+05	2.06	-2285	135	n/a	

C-109 Heel Solids: Solids Composite: Sample 2 **Group: 20120059** **DSR: 26-Jan-2012 13:48:46**

Sample Number	A	Analyte	Units	Std %-Rec	Blank	Result	Duplicate	Average	RPD	Spk %-Rec	Detection Limit	Count %-Error	Qual Flags
S12R000005		pH	None	n/a	n/a	11.7	n/a	n/a	n/a	n/a	0.01	n/a	
S12R000005		Percent water	%	100	n/a	17.4	14.7	16.0	17.0	n/a	0.01	n/a	
S12R000005		Total organic carbon	ug/g	105	<20.0	2.25E+03	n/a	n/a	n/a	n/a	137	n/a	
S12R000005		Total inorganic carbon	ug/g	98.2	<7.00	1.77E+03	n/a	n/a	n/a	n/a	47.5	n/a	
S12R000006	Z	Aluminum	ug/g	93.8	<1.36E+03	1.95E+05	n/a	n/a	n/a	n/a	1.27E+03	n/a	
S12R000006	Z	Bismuth	ug/g	105	<4.54E+03	<4.24E+03	n/a	n/a	n/a	n/a	4.24E+03	n/a	U
S12R000006	Z	Calcium	ug/g	100	<2.27E+03	<2.12E+03	n/a	n/a	n/a	n/a	2.12E+03	n/a	U
S12R000006	Z	Chromium	ug/g	98.7	<227	<212	n/a	n/a	n/a	n/a	212	n/a	U
S12R000006	Z	Iron	ug/g	99.1	<227	1.70E+03	n/a	n/a	n/a	n/a	212	n/a	J
S12R000006	Z	Manganese	ug/g	98.2	<136	<127	n/a	n/a	n/a	n/a	127	n/a	U
S12R000006	Z	Sodium	ug/g	103	<4.54E+03	9.73E+04	n/a	n/a	n/a	n/a	4.24E+03	n/a	
S12R000006	Z	Nickel	ug/g	99.8	<908	<847	n/a	n/a	n/a	n/a	847	n/a	U
S12R000006	Z	Phosphorus	ug/g	100	<2.27E+03	3.07E+04	n/a	n/a	n/a	n/a	2.12E+03	n/a	
S12R000006	Z	Sulfur	ug/g	99.5	<4.54E+03	<4.24E+03	n/a	n/a	n/a	n/a	4.24E+03	n/a	U
S12R000006	Z	Silicon	ug/g	97.7	<1.36E+03	<1.27E+03	n/a	n/a	n/a	n/a	1.27E+03	n/a	U
S12R000006	Z	Thorium	ug/g	94.0	<2.27E+03	<2.12E+03	n/a	n/a	n/a	n/a	2.12E+03	n/a	U
S12R000006	Z	Uranium	ug/g	95.7	<4.54E+03	<4.24E+03	n/a	n/a	n/a	n/a	4.24E+03	n/a	U
S12R000007	W	Fluoride	ug/g	106	<1.61E-03	1.02E+04	n/a	n/a	n/a	n/a	12.9	n/a	
S12R000007	W	Glycolate	ug/g	105	<9.37E-03	<1.88	n/a	n/a	n/a	n/a	1.88	n/a	U
S12R000007	W	Acetate	ug/g	106	<6.04E-03	102	n/a	n/a	n/a	n/a	1.21	n/a	
S12R000007	W	Formate	ug/g	106	<4.67E-03	122	n/a	n/a	n/a	n/a	0.938	n/a	
S12R000007	W	Chloride	ug/g	105	<9.98E-03	56.7	n/a	n/a	n/a	n/a	2.01	n/a	
S12R000007	W	Nitrite	ug/g	107	0.0577	4.93E+03	n/a	n/a	n/a	n/a	3.86	n/a	
S12R000007	W	Sulfate	ug/g	106	<0.0187	621	n/a	n/a	n/a	n/a	3.76	n/a	
S12R000007	W	Oxalate	ug/g	106	<0.0231	1.24E+03	n/a	n/a	n/a	n/a	4.64	n/a	
S12R000007	W	Bromide	ug/g	104	<0.0580	14.0	n/a	n/a	n/a	n/a	11.7	n/a	J
S12R000007	W	Nitrate	ug/g	106	<0.0208	2.91E+03	n/a	n/a	n/a	n/a	4.18	n/a	
S12R000007	W	Phosphate	ug/g	106	<0.0167	1.05E+05	n/a	n/a	n/a	n/a	134	n/a	

C-109 Heel Solids: Large-scale Water Dissolution: Diluted[a] Contact 1 Liquid (2 sheets) **Group: 20120383** **DSR: 29-Jun-2012 15:09:36**

Sample Number	A	Analyte	Units	Std %-Rec	Blank	Result	Duplicate	Average	RPD	Spk %-Rec	Detection Limit	Count %-Error	Qual Flags
S12R000149		Gross alpha	uCi/mL	102	<4.63E-06	2.55E-04	n/a	n/a	n/a	n/a	3.28E-06	8.693	
S12R000149		Fluoride	ug/mL	97.0	<1.61E-03	2.28E+03	n/a	n/a	n/a	n/a	1.61	n/a	
S12R000149		Glycolate	ug/mL	102	<9.37E-03	<6.56	n/a	n/a	n/a	n/a	6.56	n/a	U
S12R000149		Acetate	ug/mL	103	<6.04E-03	59	n/a	n/a	n/a	n/a	4.23	n/a	J
S12R000149		Formate	ug/mL	106	<4.67E-03	64.7	n/a	n/a	n/a	n/a	3.27	n/a	J
S12R000149		Chloride	ug/mL	109	<9.98E-03	18.8	n/a	n/a	n/a	n/a	6.99	n/a	J
S12R000149		Nitrite	ug/mL	107	<0.0192	2.15E+03	n/a	n/a	n/a	n/a	13.4	n/a	
S12R000149		Sulfate	ug/mL	107	<0.0187	238	n/a	n/a	n/a	n/a	13.1	n/a	
S12R000149		Oxalate	ug/mL	107	<0.0231	312	n/a	n/a	n/a	n/a	16.2	n/a	
S12R000149		Bromide	ug/mL	107	<0.0580	<40.6	n/a	n/a	n/a	n/a	40.6	n/a	U
S12R000149		Nitrate	ug/mL	108	<0.0208	1.15E+03	n/a	n/a	n/a	n/a	14.6	n/a	
S12R000149		Phosphate	ug/mL	101	<0.0167	2.47E+04	n/a	n/a	n/a	n/a	16.7	n/a	
S12R000149		Aluminum	ug/mL	97.3	<0.0300	90.7	n/a	n/a	n/a	n/a	3.00	n/a	
S12R000149		Bismuth	ug/mL	105	<0.100	<10.0	n/a	n/a	n/a	n/a	10.0	n/a	U
S12R000149		Calcium	ug/mL	100	<0.0500	<5.00	n/a	n/a	n/a	n/a	5.00	n/a	U
S12R000149		Chromium	ug/mL	101	<5.00E-03	3.68	n/a	n/a	n/a	n/a	0.500	n/a	J
S12R000149		Iron	ug/mL	101	5.79E-03	11.9	n/a	n/a	n/a	n/a	0.500	n/a	
S12R000149		Potassium	ug/mL	107	<0.500	52.9	n/a	n/a	n/a	n/a	50.0	n/a	J
S12R000149		Manganese	ug/mL	100	<3.00E-03	<0.300	n/a	n/a	n/a	n/a	0.300	n/a	U
S12R000149		Sodium	ug/mL	100	<0.100	1.92E+04	n/a	n/a	n/a	n/a	10.0	n/a	
S12R000149		Nickel	ug/mL	101	<0.0200	<2.00	n/a	n/a	n/a	n/a	2.00	n/a	U
S12R000149		Phosphorus	ug/mL	101	<0.0500	7.89E+03	n/a	n/a	n/a	n/a	5.00	n/a	
S12R000149		Sulfur	ug/mL	102	<0.100	85.5	n/a	n/a	n/a	n/a	10.0	n/a	J

C-109 Heel Solids: Large-scale Water Dissolution: Diluted[a] Contact 1 Liquid (2 sheets)

Sample Number	A	Analyte	Units	Std %-Rec	Blank	Result	Duplicate	Average	RPD	Spk %-Rec	Detection Limit	Count %-Error	Qual Flags
S12R000149		Silicon	ug/mL	99.5	<0.0300	5.31	n/a	n/a	n/a	n/a	3.00	n/a	J
S12R000149		Thorium	ug/mL	95.4	<0.0500	<5.00	n/a	n/a	n/a	n/a	5.00	n/a	U
S12R000149		Uranium	ug/mL	99.2	<0.100	289	n/a	n/a	n/a	n/a	10.0	n/a	
S12R000149		Percent water	%	97.6	n/a	93.0	91.7	92.4	1.36	n/a	0.01	n/a	
S12R000149		Total inorganic carbon	ug/mL	102	<7.00	782	n/a	n/a	n/a	n/a	7.00	n/a	
S12R000149		Total organic carbon	ug/mL	98.4	<20.0	171	n/a	n/a	n/a	n/a	20.0	n/a	J

[a] Large-scale Water Dissolution: 1st Contact

	Wt (g)	*t* (°C)	D_{Liq} (g/mL)	Vol (mL)
Test Liquid	15.396	25.6	1.065	14.463
Added H_2O	3.838	25.2	0.9970	3.850
Dilution Factor	1.2662			

C-109 Heel Solids: Large-scale Water Dissolution: Diluted[a] Contact 2 Liquid (2 sheets) **Group: 20120383** **DSR: 29-Jun-2012 15:09:36**

Sample Number	A	Analyte	Units	Std %-Rec	Blank	Result	Duplicate	Average	RPD	Spk %-Rec	Detection Limit	Count %-Error	Qual Flags
S12R000150		Gross alpha	uCi/mL	102	<4.63E-06	6.45E-05	n/a	n/a	n/a	n/a	3.28E-06	18.221	
S12R000150		Fluoride	ug/mL	103	<1.61E-03	2.34E+03	n/a	n/a	n/a	n/a	1.61	n/a	
S12R000150		Glycolate	ug/mL	103	<9.37E-03	<0.937	n/a	n/a	n/a	n/a	0.937	n/a	U
S12R000150		Acetate	ug/mL	105	<6.04E-03	21.3	n/a	n/a	n/a	n/a	0.604	n/a	J
S12R000150		Formate	ug/mL	108	<4.67E-03	11.7	n/a	n/a	n/a	n/a	0.467	n/a	J
S12R000150		Chloride	ug/mL	108	<9.98E-03	3.94	n/a	n/a	n/a	n/a	0.998	n/a	J
S12R000150		Nitrite	ug/mL	108	<0.0192	310	n/a	n/a	n/a	n/a	1.92	n/a	
S12R000150		Sulfate	ug/mL	108	<0.0187	42.2	n/a	n/a	n/a	n/a	1.87	n/a	
S12R000150		Oxalate	ug/mL	108	<0.0231	76.2	n/a	n/a	n/a	n/a	2.31	n/a	
S12R000150		Bromide	ug/mL	108	<0.0580	<5.80	n/a	n/a	n/a	n/a	5.80	n/a	U
S12R000150		Nitrate	ug/mL	109	<0.0208	171	n/a	n/a	n/a	n/a	2.08	n/a	
S12R000150		Phosphate	ug/mL	108	<0.0167	2.42E+04	n/a	n/a	n/a	n/a	16.7	n/a	
S12R000150		Aluminum	ug/mL	97.3	<0.0300	82.5	n/a	n/a	n/a	n/a	3.00	n/a	
S12R000150		Bismuth	ug/mL	105	<0.100	<10.0	n/a	n/a	n/a	n/a	10.0	n/a	U
S12R000150		Calcium	ug/mL	100	<0.0500	<5.00	n/a	n/a	n/a	n/a	5.00	n/a	U
S12R000150		Chromium	ug/mL	101	<5.00E-03	0.726	n/a	n/a	n/a	n/a	0.500	n/a	J
S12R000150		Iron	ug/mL	101	5.79E-03	2.21	n/a	n/a	n/a	n/a	0.500	n/a	BJ
S12R000150		Potassium	ug/mL	107	<0.500	<50.0	n/a	n/a	n/a	n/a	50.0	n/a	U
S12R000150		Manganese	ug/mL	100	<3.00E-03	<0.300	n/a	n/a	n/a	n/a	0.300	n/a	U
S12R000150		Sodium	ug/mL	100	<0.100	1.68E+04	n/a	n/a	n/a	n/a	10.0	n/a	
S12R000150		Nickel	ug/mL	101	<0.0200	<2.00	n/a	n/a	n/a	n/a	2.00	n/a	U
S12R000150		Phosphorus	ug/mL	101	<0.0500	7.89E+03	n/a	n/a	n/a	n/a	5.00	n/a	
S12R000150		Sulfur	ug/mL	102	<0.100	16.6	n/a	n/a	n/a	n/a	10.0	n/a	J

C-109 Heel Solids: Large-scale Water Dissolution: Diluted[a] Contact 2 Liquid (2 sheets)

Sample Number	A	Analyte	Units	Std %-Rec	Blank	Result	Duplicate	Average	RPD	Spk %-Rec	Detection Limit	Count %-Error	Qual Flags
S12R000150		Silicon	ug/mL	99.5	<0.0300	6.22	n/a	n/a	n/a	n/a	3.00	n/a	J
S12R000150		Thorium	ug/mL	95.4	<0.0500	<5.00	n/a	n/a	n/a	n/a	5.00	n/a	U
S12R000150		Uranium	ug/mL	99.2	<0.100	43.5	n/a	n/a	n/a	n/a	10.0	n/a	J
S12R000150		Percent water	%	97.6	n/a	93.0	n/a	n/a	n/a	n/a	0.01	n/a	
S12R000150		Total inorganic carbon	ug/mL	102	<7.00	154	n/a	n/a	n/a	n/a	7.00	n/a	
S12R000150		Total organic carbon	ug/mL	98.4	<20.0	21.6	n/a	n/a	n/a	n/a	20.0	n/a	J

[a] Large-scale Water Dissolution: 2nd Contact

	Wt (g)	*t* (°C)	D_{Liq} (g/mL)	Vol (mL)
Test Liquid	15.027	24.2	1.056	14.227
Added H_2O	3.772		0.9972	3.782
Dilution Factor	1.2659			

C-109 Heel Solids: Large-scale Water Dissolution: Diluted[a] Contact 3 Liquid (2 sheets) **Group: 20120383** **DSR: 29-Jun-2012 15:09:36**

Sample Number	A	Analyte	Units	Std %-Rec	Blank	Result	Duplicate	Average	RPD	Spk %-Rec	Detection Limit	Count %-Error	Qual Flags
S12R000151		Gross alpha	uCi/mL	102	<4.63E-06	9.99E-06	n/a	n/a	n/a	n/a	3.28E-06	60.332	J
S12R000151		Fluoride	ug/mL	103	<1.61E-03	745	n/a	n/a	n/a	n/a	0.805	n/a	
S12R000151		Glycolate	ug/mL	103	<9.37E-03	<0.937	n/a	n/a	n/a	n/a	0.937	n/a	U
S12R000151		Acetate	ug/mL	105	<6.04E-03	<0.604	n/a	n/a	n/a	n/a	0.604	n/a	U
S12R000151		Formate	ug/mL	108	<4.67E-03	3.54	n/a	n/a	n/a	n/a	0.467	n/a	J
S12R000151		Chloride	ug/mL	108	<9.98E-03	<0.998	n/a	n/a	n/a	n/a	0.998	n/a	U
S12R000151		Nitrite	ug/mL	108	<0.0192	41.9	n/a	n/a	n/a	n/a	1.92	n/a	
S12R000151		Sulfate	ug/mL	108	<0.0187	9.13	n/a	n/a	n/a	n/a	1.87	n/a	J
S12R000151		Oxalate	ug/mL	108	<0.0231	19.4	n/a	n/a	n/a	n/a	2.31	n/a	J
S12R000151		Bromide	ug/mL	108	<0.0580	<5.80	n/a	n/a	n/a	n/a	5.80	n/a	U
S12R000151		Nitrate	ug/mL	109	<0.0208	27.4	n/a	n/a	n/a	n/a	2.08	n/a	J
S12R000151		Phosphate	ug/mL	108	<0.0167	7.78E+03	n/a	n/a	n/a	n/a	8.35	n/a	
S12R000151		Aluminum	ug/mL	97.3	<0.0300	52.1	n/a	n/a	n/a	n/a	3.00	n/a	
S12R000151		Bismuth	ug/mL	105	<0.100	<10.0	n/a	n/a	n/a	n/a	10.0	n/a	U
S12R000151		Calcium	ug/mL	100	<0.0500	<5.00	n/a	n/a	n/a	n/a	5.00	n/a	U
S12R000151		Chromium	ug/mL	101	<5.00E-03	<0.500	n/a	n/a	n/a	n/a	0.500	n/a	U
S12R000151		Iron	ug/mL	101	5.79E-03	<0.500	n/a	n/a	n/a	n/a	0.500	n/a	U
S12R000151		Potassium	ug/mL	107	<0.500	<50.0	n/a	n/a	n/a	n/a	50.0	n/a	U
S12R000151		Manganese	ug/mL	100	<3.00E-03	<0.300	n/a	n/a	n/a	n/a	0.300	n/a	U
S12R000151		Sodium	ug/mL	100	<0.100	5.20E+03	n/a	n/a	n/a	n/a	10.0	n/a	
S12R000151		Nickel	ug/mL	101	<0.0200	<2.00	n/a	n/a	n/a	n/a	2.00	n/a	U
S12R000151		Phosphorus	ug/mL	101	<0.0500	2.35E+03	n/a	n/a	n/a	n/a	5.00	n/a	
S12R000151		Sulfur	ug/mL	102	<0.100	<10.0	n/a	n/a	n/a	n/a	10.0	n/a	U

C-109 Heel Solids: Large-scale Water Dissolution: Diluted[a] Contact 3 Liquid (2 sheets)

Sample Number	A	Analyte	Units	Std %-Rec	Blank	Result	Duplicate	Average	RPD	Spk %-Rec	Detection Limit	Count %-Error	Qual Flags
S12R000151		Silicon	ug/mL	99.5	<0.0300	5.67	n/a	n/a	n/a	n/a	3.00	n/a	J
S12R000151		Thorium	ug/mL	95.4	<0.0500	<5.00	n/a	n/a	n/a	n/a	5.00	n/a	U
S12R000151		Uranium	ug/mL	99.2	<0.100	<10.0	n/a	n/a	n/a	n/a	10.0	n/a	U
S12R000151		Percent water	%	98.5	n/a	99.9	98.3	99.1	1.62	n/a	0.01	n/a	
S12R000151		Total inorganic carbon	ug/mL	102	<7.00	80.4	n/a	n/a	n/a	n/a	7.00	n/a	
S12R000151		Total organic carbon	ug/mL	98.4	<20.0	89.0	n/a	n/a	n/a	n/a	20.0	n/a	J

[a] Large-scale Water Dissolution: 3rd Contact

	Wt (g)	*t* (°C)	D_{Liq} (g/mL)	Vol (mL)
Test Liquid	14.964	24.6	1.014	14.765
Added H_2O	3.716		0.9971	3.727
Dilution Factor	1.2524			

C-109 Heel Solids: Large-scale Caustic Dissolution: Final Test Liquid **Group: 20120471** **DSR: 12-Apr-2012 13:08:14**

Sample Number	A	Analyte	Units	Std %-Rec	Blank	Result	Duplicate	Average	RPD	Spk %-Rec	Detection Limit	Count %-Error	Qual Flags
S12R000175		Gross alpha	uCi/mL	98.3	<1.61E-04	9.98E-04	1.18E-03	1.09E-03	17.0	82.4	1.31E-04	34.635	J
S12R000175		Fluoride	ug/mL	91.4	<1.61E-03	69.3	76.1	72.7	9.42	88.3	0.161	n/a	
S12R000175		Glycolate	ug/mL	91.8	<9.37E-03	<0.937	<0.937	n/a	n/a	115	0.937	n/a	U
S12R000175		Acetate	ug/mL	95.9	<6.04E-03	<0.604	<0.604	n/a	n/a	120	0.604	n/a	U
S12R000175		Formate	ug/mL	96.3	<4.67E-03	10.1	10.2	10.1	1.28	113	0.467	n/a	J
S12R000175		Chloride	ug/mL	94.5	<9.98E-03	4.03	3.93	3.98	2.61	100	0.998	n/a	J
S12R000175		Nitrite	ug/mL	98.3	<0.0192	15.3	15.3	15.3	0.359	100	1.92	n/a	J
S12R000175		Sulfate	ug/mL	96.2	<0.0187	<1.87	<1.87	n/a	n/a	96.6	1.87	n/a	U
S12R000175		Oxalate	ug/mL	96.2	<0.0231	<2.31	<2.31	n/a	n/a	81.5	2.31	n/a	U
S12R000175		Bromide	ug/mL	95.0	<0.0580	<5.80	<5.80	n/a	n/a	99.6	5.80	n/a	U
S12R000175		Nitrate	ug/mL	96.7	<0.0208	24.2	24.4	24.3	0.765	97.5	2.08	n/a	J
S12R000175		Phosphate	ug/mL	96.9	<0.0167	1.17E+03	1.15E+03	1.16E+03	1.91	99.1	1.67	n/a	
S12R000175		Aluminum	ug/mL	91.9	<0.0300	4.70E+04	n/a	n/a	n/a	n/a	120	n/a	
S12R000175		Bismuth	ug/mL	106	<0.100	<400	n/a	n/a	n/a	n/a	400	n/a	U
S12R000175		Calcium	ug/mL	109	<0.0500	<200	n/a	n/a	n/a	n/a	200	n/a	U
S12R000175		Chromium	ug/mL	97.4	<5.00E-03	<20.0	n/a	n/a	n/a	n/a	20.0	n/a	U
S12R000175		Iron	ug/mL	98.0	<5.00E-03	28.1	n/a	n/a	n/a	n/a	20.0	n/a	J
S12R000175		Manganese	ug/mL	96.8	<3.00E-03	<12.0	n/a	n/a	n/a	n/a	12.0	n/a	U
S12R000175		Sodium	ug/mL	91.6	<0.100	1.56E+05	n/a	n/a	n/a	n/a	400	n/a	
S12R000175		Nickel	ug/mL	97.9	<0.0200	<80.0	n/a	n/a	n/a	n/a	80.0	n/a	U
S12R000175		Phosphorus	ug/mL	95.0	<0.0500	406	n/a	n/a	n/a	n/a	200	n/a	J
S12R000175		Sulfur	ug/mL	97.0	<0.100	<400	n/a	n/a	n/a	n/a	400	n/a	U
S12R000175		Silicon	ug/mL	93.0	<0.0300	159	n/a	n/a	n/a	n/a	120	n/a	J
S12R000175		Uranium	ug/mL	93.9	<0.100	<400	n/a	n/a	n/a	n/a	400	n/a	U
S12R000175		Hydroxide	ug/mL	94.4	<41.7	9.58E+04	n/a	n/a	n/a	n/a	1.11E+03	n/a	
S12R000175		Specific gravity	None	100.5	n/a	1.319	n/a	n/a	n/a	n/a	1.00E-03	n/a	
S12R000175		Percent water	%	98.9	n/a	68.7	68.9	68.8	0.218	n/a	0.01	n/a	
S12R000175		Total inorganic carbon	ug/mL	106	<7.00	68.4	n/a	n/a	n/a	n/a	7.00	n/a	BJ
S12R000175		Total organic carbon	ug/mL	100	<20.0	245	n/a	n/a	n/a	n/a	20.0	n/a	

C-109 Heel Solids: Large-scale Dissolution: <710 um Residual Solids (2 sheets) **Group: 20120602** **DSR: 22-May-2012 [a] 15:37:49**

Sample Number	A	Analyte	Units	Std %-Rec	Blank	Result	Duplicate	Average	RPD	Spk %-Rec	Detection Limit	Count %-Error	Qual Flags
S12R000200		Iodine-129	uCi/g	97.3	<2.48E-05	<5.99E-05	n/a	n/a	n/a	n/a	5.99E-05	n/a	U
S12R000200		Percent water	%	95.3	n/a	29.6	n/a	n/a	n/a	n/a	0.01	n/a	Y
S12R000200		Total inorganic carbon	ug/g	100	<7.00	347	n/a	n/a	n/a	n/a	33.8	n/a	
S12R000200		Total organic carbon	ug/g	96.7	<20.0	2.53E+03	n/a	n/a	n/a	n/a	96.5	n/a	
S12R000206	W	Fluoride	ug/g	101	<5.00E-03	35.2	n/a	n/a	n/a	n/a	1.11	n/a	
S12R000206	W	Glycolate	ug/g	101	<0.0160	<3.55	n/a	n/a	n/a	n/a	3.55	n/a	U
S12R000206	W	Acetate	ug/g	101	<0.0280	<6.21	n/a	n/a	n/a	n/a	6.21	n/a	U
S12R000206	W	Formate	ug/g	102	<0.0420	<9.31	n/a	n/a	n/a	n/a	9.31	n/a	U
S12R000206	W	Chloride	ug/g	104	<0.0160	6.43	n/a	n/a	n/a	n/a	3.55	n/a	J
S12R000206	W	Nitrite	ug/g	95.4	0.0840	17.0	n/a	n/a	n/a	n/a	3.24	n/a	BJ
S12R000206	W	Sulfate	ug/g	101	<0.0460	18.6	n/a	n/a	n/a	n/a	10.2	n/a	J
S12R000206	W	Oxalate	ug/g	102	<0.0260	9.31	n/a	n/a	n/a	n/a	5.76	n/a	J
S12R000206	W	Bromide	ug/g	101	<0.0150	<3.32	n/a	n/a	n/a	n/a	3.32	n/a	U
S12R000206	W	Nitrate	ug/g	101	<0.0630	52.5	n/a	n/a	n/a	n/a	14.0	n/a	J
S12R000206	W	Phosphate	ug/g	97.5	<0.0167	263	n/a	n/a	n/a	n/a	2.82	n/a	
S12R000207	Z	Curium-243/244	uCi/g	n/a	<1.64E-04	<1.47E-04	n/a	n/a	n/a	n/a	1.47E-04	n/a	U
S12R000207	Z	Americium-241	uCi/g	96.4	2.16E-04	0.0863	n/a	n/a	n/a	n/a	2.22E-04	1.74	
S12R000207	Z	Cesium-137	uCi/g	105	<0.268	0.781	n/a	n/a	n/a	n/a	0.274	11.44	
S12R000207	Z	Aluminum	ug/g	89.7	<3.14E+03	2.94E+05	n/a	n/a	n/a	n/a	2.12E+03	n/a	
S12R000207	Z	Bismuth	ug/g	100	<1.05E+04	<7.07E+03	n/a	n/a	n/a	n/a	7.07E+03	n/a	U
S12R000207	Z	Calcium	ug/g	110	<5.24E+03	3.72E+03	n/a	n/a	n/a	n/a	3.54E+03	n/a	J
S12R000207	Z	Chromium	ug/g	98.3	529	<354	n/a	n/a	n/a	n/a	354	n/a	U
S12R000207	Z	Iron	ug/g	98.9	<524	8.99E+03	n/a	n/a	n/a	n/a	354	n/a	
S12R000207	Z	Manganese	ug/g	98.0	<314	<212	n/a	n/a	n/a	n/a	212	n/a	U
S12R000207	Z	Sodium	ug/g	94.7	<1.05E+04	8.45E+03	n/a	n/a	n/a	n/a	7.07E+03	n/a	J
S12R000207	Z	Nickel	ug/g	98.4	<2.10E+03	4.12E+03	n/a	n/a	n/a	n/a	1.41E+03	n/a	J
S12R000207	Z	Phosphorus	ug/g	99.4	<5.24E+03	<3.54E+03	n/a	n/a	n/a	n/a	3.54E+03	n/a	U
S12R000207	Z	Sulfur	ug/g	98.5	<1.05E+04	<7.07E+03	n/a	n/a	n/a	n/a	7.07E+03	n/a	U
S12R000207	Z	Silicon	ug/g	95.6	<3.14E+03	<2.12E+03	n/a	n/a	n/a	n/a	2.12E+03	n/a	U

C-109 Heel Solids: Large-scale Dissolution: <710 um Residual Solids (2 sheets)

Sample Number	A	Analyte	Units	Std %-Rec	Blank	Result	Duplicate	Average	RPD	Spk %-Rec	Detection Limit	Count %-Error	Qual Flags
S12R000207	Z	Uranium	ug/g	95.2	<1.05E+04	<7.07E+03	n/a	n/a	n/a	n/a	7.07E+03	n/a	U
S12R000207	Z	Uranium-233	ug/g	101	<2.10	<1.41	n/a	n/a	n/a	n/a	1.41	n/a	U
S12R000207	Z	Uranium-234	ug/g	n/a	<0.0524	<0.0354	n/a	n/a	n/a	n/a	0.0354	n/a	IU
S12R000207	Z	Uranium-235	ug/g	98.8	<0.105	14.5	n/a	n/a	n/a	n/a	0.0707	n/a	
S12R000207	Z	Uranium-236	ug/g	n/a	<0.0419	<0.0283	n/a	n/a	n/a	n/a	0.0283	n/a	IU
S12R000207	Z	Neptunium-237	ug/g	103	<1.05	<0.707	n/a	n/a	n/a	n/a	0.707	n/a	U
S12R000207	Z	Uranium-238	ug/g	98.3	<5.24	2.17E+03	n/a	n/a	n/a	n/a	3.54	n/a	
S12R000207	Z	Plutonium-239/240	uCi/g	104	<4.78E-04	0.0839	n/a	n/a	n/a	n/a	5.00E-04	2.15	
S12R000207	Z	Plutonium-238	uCi/g	n/a	<3.11E-04	2.79E-03	n/a	n/a	n/a	n/a	3.84E-04	20.72	
S12R000207	Z	Strontium-89/90	uCi/g	96.3	0.157	823	n/a	n/a	n/a	n/a	0.0973	0.631	
S12R000208	A	Technetium-99	ug/g	109	<6.00E-03	<0.914	n/a	n/a	n/a	n/a	0.914	n/a	U
S12R000209	A	Aluminum	ug/g	82.7	<1.50	1.26E+05	n/a	n/a	n/a	n/a	53.4	n/a	
S12R000209	A	Bismuth	ug/g	85.3	<5.00	<192	n/a	n/a	n/a	n/a	192	n/a	U
S12R000209	A	Calcium	ug/g	75.7	<2.50	3.67E+03	n/a	n/a	n/a	n/a	95.9	n/a	a
S12R000209	A	Chromium	ug/g	83.3	0.392	33.8	n/a	n/a	n/a	n/a	9.59	n/a	BJ
S12R000209	A	Iron	ug/g	85.8	<0.250	1.04E+04	n/a	n/a	n/a	n/a	9.59	n/a	b
S12R000209	A	Manganese	ug/g	83.0	<0.150	55.3	n/a	n/a	n/a	n/a	5.75	n/a	J
S12R000209	A	Sodium	ug/g	83.4	<5.00	3.64E+03	n/a	n/a	n/a	n/a	192	n/a	b
S12R000209	A	Nickel	ug/g	84.4	<1.00	4.39E+03	n/a	n/a	n/a	n/a	38.4	n/a	b
S12R000209	A	Phosphorus	ug/g	87.3	<2.50	1.53E+03	n/a	n/a	n/a	n/a	95.9	n/a	
S12R000209	A	Sulfur	ug/g	83.9	<5.00	<192	n/a	n/a	n/a	n/a	192	n/a	U
S12R000209	A	Silicon	ug/g	79.7	<1.50	1.08E+03	n/a	n/a	n/a	n/a	53.4	n/a	
S12R000209	A	Uranium	ug/g	91.6	<5.00	2.44E+03	n/a	n/a	n/a	n/a	192	n/a	
S12R000209	A	Zirconium	ug/g	84.7	<0.250	23.3	n/a	n/a	n/a	n/a	9.59	n/a	J

[a] Analysis results for IC analysis of water leach (W) specimen preparations were recalculated on September 10, 2013. The revised results are tabulated.

Comment: Sample S12R000200 – Percent Water
"The weight loss at this temperature [225-305 °C] has a high probability of NOT being water. It is probably due to the decomposition of a salt."

C-109 Heel Solids: Large-scale Dissolution: >710 um Residual Solids (2 sheets) **Group: 20120602** **DSR: 22-May-2012 [a] 15:37:49**

Sample Number	A	Analyte	Units	Std %-Rec	Blank	Result	Duplicate	Average	RPD	Spk %-Rec	Detection Limit	Count %-Error	Qual Flags
S12R000197		Iodine-129	uCi/g	97.3	<2.48E-05	<4.25E-05	n/a	n/a	n/a	n/a	4.25E-05	n/a	U
S12R000197		Percent water	%	95.3	n/a	30.0	30.0	30.0	0.200	n/a	0.01	n/a	Y
S12R000197		Total inorganic carbon	ug/g	100	<7.00	90.4	n/a	n/a	n/a	n/a	41.3	n/a	J
S12R000197		Total organic carbon	ug/g	96.7	<20.0	537	n/a	n/a	n/a	n/a	118	n/a	J
S12R000202	W	Fluoride	ug/g	101	<5.00E-03	135	147	141	8.16	147	0.926	n/a	b
S12R000202	W	Glycolate	ug/g	101	<0.0160	<2.96	<2.90	n/a	n/a	103	2.96	n/a	U
S12R000202	W	Acetate	ug/g	101	<0.0280	<5.18	<5.07	n/a	n/a	102	5.18	n/a	U
S12R000202	W	Formate	ug/g	102	<0.0420	<7.77	<7.60	n/a	n/a	102	7.77	n/a	U
S12R000202	W	Chloride	ug/g	104	<0.0160	5.74	5.79	5.77	0.942	105	2.96	n/a	J
S12R000202	W	Nitrite	ug/g	95.4	0.0840	23.3	23.5	23.4	0.849	94.6	3.90	n/a	BJ
S12R000202	W	Sulfate	ug/g	101	<0.0460	17.6	16.3	16.9	7.63	101	8.52	n/a	J
S12R000202	W	Oxalate	ug/g	102	<0.0260	9.63	<4.71	n/a	n/a	99.9	4.81	n/a	J
S12R000202	W	Bromide	ug/g	101	<0.0150	<2.78	<2.72	n/a	n/a	100	2.78	n/a	U
S12R000202	W	Nitrate	ug/g	101	<0.0630	58.1	63.5	60.8	8.90	101	11.7	n/a	
S12R000202	W	Phosphate	ug/g	97.5	<0.0167	1.38E+03	1.04E+03	1.21E+03	27.8	99.2	3.39	n/a	
S12R000203	Z	Curium-243/244	uCi/g	n/a	<1.64E-04	<4.20E-05	n/a	n/a	n/a	n/a	4.20E-05	n/a	U
S12R000203	Z	Americium-241	uCi/g	96.4	2.16E-04	0.0397	n/a	n/a	n/a	n/a	6.76E-05	1.38	
S12R000203	Z	Cesium-137	uCi/g	105	<0.268	0.813	n/a	n/a	n/a	n/a	0.169	7.46	
S12R000203	Z	Lead-212	uCi/g	n/a	0.845	0.554	n/a	n/a	n/a	n/a	0.258	14.96	B
S12R000203	Z	Lead-214	uCi/g	n/a	1.34	0.783	n/a	n/a	n/a	n/a	0.364	18.43	B
S12R000203	Z	Aluminum	ug/g	89.7	<3.14E+03	3.03E+05	n/a	n/a	n/a	n/a	1.59E+03	n/a	
S12R000203	Z	Bismuth	ug/g	100	<1.05E+04	<5.30E+03	n/a	n/a	n/a	n/a	5.30E+03	n/a	U
S12R000203	Z	Calcium	ug/g	110	<5.24E+03	<2.65E+03	n/a	n/a	n/a	n/a	2.65E+03	n/a	U
S12R000203	Z	Chromium	ug/g	98.3	529	289	n/a	n/a	n/a	n/a	265	n/a	BJ
S12R000203	Z	Iron	ug/g	98.9	<524	8.52E+03	n/a	n/a	n/a	n/a	265	n/a	
S12R000203	Z	Manganese	ug/g	98.0	<314	<159	n/a	n/a	n/a	n/a	159	n/a	U
S12R000203	Z	Sodium	ug/g	94.7	<1.05E+04	7.34E+03	n/a	n/a	n/a	n/a	5.30E+03	n/a	J
S12R000203	Z	Nickel	ug/g	98.4	<2.10E+03	<1.06E+03	n/a	n/a	n/a	n/a	1.06E+03	n/a	U
S12R000203	Z	Phosphorus	ug/g	99.4	<5.24E+03	<2.65E+03	n/a	n/a	n/a	n/a	2.65E+03	n/a	U

C-109 Heel Solids: Large-scale Dissolution: >710 um Residual Solids (2 sheets)

Sample Number	A	Analyte	Units	Std %-Rec	Blank	Result	Duplicate	Average	RPD	Spk %-Rec	Detection Limit	Count %-Error	Qual Flags
S12R000203	Z	Sulfur	ug/g	98.5	<1.05E+04	<5.30E+03	n/a	n/a	n/a	n/a	5.30E+03	n/a	U
S12R000203	Z	Silicon	ug/g	95.6	<3.14E+03	2.93E+03	n/a	n/a	n/a	n/a	1.59E+03	n/a	J
S12R000203	Z	Uranium	ug/g	95.2	<1.05E+04	<5.30E+03	n/a	n/a	n/a	n/a	5.30E+03	n/a	U
S12R000203	Z	Uranium-233	ug/g	101	<2.10	<1.06	n/a	n/a	n/a	n/a	1.06	n/a	U
S12R000203	Z	Uranium-234	ug/g	n/a	<0.0524	<0.0265	n/a	n/a	n/a	n/a	0.0265	n/a	IU
S12R000203	Z	Uranium-235	ug/g	98.8	<0.105	2.40	n/a	n/a	n/a	n/a	0.0530	n/a	
S12R000203	Z	Uranium-236	ug/g	n/a	<0.0419	<0.0212	n/a	n/a	n/a	n/a	0.0212	n/a	IU
S12R000203	Z	Neptunium-237	ug/g	103	<1.05	<0.530	n/a	n/a	n/a	n/a	0.530	n/a	U
S12R000203	Z	Uranium-238	ug/g	98.3	<5.24	361	n/a	n/a	n/a	n/a	2.65	n/a	
S12R000203	Z	Plutonium-239/240	uCi/g	104	<4.78E-04	0.0244	n/a	n/a	n/a	n/a	1.29E-04	2.08	
S12R000203	Z	Plutonium-238	uCi/g	n/a	<3.11E-04	8.96E-04	n/a	n/a	n/a	n/a	8.95E-05	18.92	
S12R000203	Z	Strontium-89/90	uCi/g	96.3	0.157	276	n/a	n/a	n/a	n/a	0.0317	0.623	
S12R000204	A	Technetium-99	ug/g	109	<6.00E-03	<0.796	n/a	n/a	n/a	n/a	0.796	n/a	U
S12R000205	A	Aluminum	ug/g	82.7	<1.50	1.62E+05	n/a	n/a	n/a	n/a	40.1	n/a	
S12R000205	A	Bismuth	ug/g	85.3	<5.00	<154	n/a	n/a	n/a	n/a	154	n/a	U
S12R000205	A	Calcium	ug/g	75.7	<2.50	547	n/a	n/a	n/a	n/a	76.8	n/a	Ja
S12R000205	A	Chromium	ug/g	83.3	0.392	19.4	n/a	n/a	n/a	n/a	7.68	n/a	BJ
S12R000205	A	Iron	ug/g	85.8	<0.250	8.29E+03	n/a	n/a	n/a	n/a	7.68	n/a	b
S12R000205	A	Manganese	ug/g	83.0	<0.150	37.4	n/a	n/a	n/a	n/a	4.61	n/a	
S12R000205	A	Sodium	ug/g	83.4	<5.00	3.35E+03	n/a	n/a	n/a	n/a	154	n/a	b
S12R000205	A	Nickel	ug/g	84.4	<1.00	927	n/a	n/a	n/a	n/a	30.7	n/a	b
S12R000205	A	Phosphorus	ug/g	87.3	<2.50	735	n/a	n/a	n/a	n/a	76.8	n/a	J
S12R000205	A	Sulfur	ug/g	83.9	<5.00	<154	n/a	n/a	n/a	n/a	154	n/a	U
S12R000205	A	Silicon	ug/g	79.7	<1.50	890	n/a	n/a	n/a	n/a	40.1	n/a	J
S12R000205	A	Uranium	ug/g	91.6	<5.00	398	n/a	n/a	n/a	n/a	154	n/a	J
S12R000205	A	Zirconium	ug/g	84.7	<0.250	9.29	n/a	n/a	n/a	n/a	7.68	n/a	J

[a] Analysis results for IC analysis of water leach (W) specimen preparations were recalculated on September 10, 2013. The revised results are tabulated.

Comment: Sample S12R000197 – Percent Water
"The weight loss at this temperature [225-305 °C] has a high probability of NOT being water. It is probably due to the decomposition of a salt."

C-109 Heel Solids: Aggregate Water Dissolution: Diluted[a] Contact 1 Liquid (2 sheets) **Group: 20120383 DSR: 27-Mar-2012 14:06:38**

Sample Number	A	Analyte	Units	Std %-Rec	Blank	Result	Duplicate	Average	RPD	Spk %-Rec	Detection Limit	Count %-Error	Qual Flags
S12R000146		Gross alpha	uCi/mL	102	<4.63E-06	1.05E-04	1.02E-04	1.03E-04	3.22	88.2	3.27E-06	14.07	
S12R000146		Fluoride	ug/mL	104	<1.61E-03	182	182	182	0.295	102.3	0.322	n/a	
S12R000146		Glycolate	ug/mL	102	<9.37E-03	<0.937	<0.937	n/a	n/a	107.3	0.937	n/a	U
S12R000146		Acetate	ug/mL	103	<6.04E-03	21.3	20.9	21.1	1.87	108.9	0.604	n/a	J
S12R000146		Formate	ug/mL	106	<4.67E-03	23.3	24.1	23.7	3.35	109.3	0.467	n/a	J
S12R000146		Chloride	ug/mL	107	<9.98E-03	16.9	16.5	16.7	2.23	109.3	0.998	n/a	
S12R000146		Nitrite	ug/mL	107	<0.0192	1.51E+03	1.52E+03	1.51E+03	0.481	103.3	1.92	n/a	
S12R000146		Sulfate	ug/mL	107	<0.0187	155	151	153	2.49	108.4	1.87	n/a	
S12R000146		Oxalate	ug/mL	108	<0.0231	114	115	114	0.467	109.3	2.31	n/a	
S12R000146		Bromide	ug/mL	107	<0.0580	<5.80	<5.80	n/a	n/a	104.3	5.80	n/a	U
S12R000146		Nitrate	ug/mL	108	<0.0208	762	765	763	0.457	105.6	2.08	n/a	
S12R000146		Phosphate	ug/mL	109	0.0830	3.01E+03	3.00E+03	3.00E+03	0.420	100.5	3.34	n/a	
S12R000146		Aluminum	ug/mL	97.3	<0.0300	13.8	14.7	14.2	6.44	95.4	3.00	n/a	J
S12R000146		Bismuth	ug/mL	105	<0.100	<10.0	<10.0	n/a	n/a	106	10.0	n/a	U
S12R000146		Calcium	ug/mL	100	<0.0500	<5.00	<5.00	n/a	n/a	102	5.00	n/a	U
S12R000146		Chromium	ug/mL	101	<5.00E-03	2.12	2.31	2.22	8.72	102	0.500	n/a	J
S12R000146		Iron	ug/mL	101	5.79E-03	8.59	8.67	8.63	0.928	102	0.500	n/a	B
S12R000146		Potassium	ug/mL	107	<0.500	<50.0	n/a	n/a	n/a	86.5	50.0	n/a	U
S12R000146		Manganese	ug/mL	100	<3.00E-03	<0.300	<0.300	n/a	n/a	102	0.300	n/a	U
S12R000146		Sodium	ug/mL	100	<0.100	4.75E+03	4.55E+03	4.65E+03	4.27	109.8	10.0	n/a	
S12R000146		Nickel	ug/mL	101	<0.0200	<2.00	2.57	n/a	n/a	103	2.00	n/a	U
S12R000146		Phosphorus	ug/mL	101	<0.0500	966	969	967	0.293	103	5.00	n/a	
S12R000146		Sulfur	ug/mL	102	<0.100	54.6	54.1	54.3	0.900	102	10.0	n/a	J

C-109 Heel Solids: Aggregate Water Dissolution: Diluted[a] Contact 1 Liquid (2 sheets)

Sample Number	A	Analyte	Units	Std %-Rec	Blank	Result	Duplicate	Average	RPD	Spk %-Rec	Detection Limit	Count %-Error	Qual Flags
S12R000146		Silicon	ug/mL	99.5	<0.0300	<3.00	<3.00	n/a	n/a	99.0	3.00	n/a	U
S12R000146		Thorium	ug/mL	95.4	<0.0500	<5.00	n/a	n/a	n/a	95.3	5.00	n/a	U
S12R000146		Uranium	ug/mL	99.2	<0.100	106	110	108	3.63	98.0	10.0	n/a	
S12R000146		Percent water	%	98.0	n/a	99.1	98.8	99.0	0.293	n/a	0.01	n/a	
S12R000146		Total inorganic carbon	ug/mL	102	<7.00	738	736	737	0.271	96.4	7.00	n/a	
S12R000146		Total organic carbon	ug/mL	98.4	<20.0	106	95.8	101	10.1	98.6	20.0	n/a	J

[a] Aggregate Water Dissolution: 1st Contact

	Wt (g)	*t* (°C)	D_{Liq} (g/mL)	Vol (mL)
Test Liquid	13.036	25.2	1.010	12.904
Added H_2O	3.295		0.9970	3.305
Dilution Factor	1.2561			

C-109 Heel Solids: Aggregate Water Dissolution: Diluted[a] Contact 2 Liquid (2 sheets) **Group: 20120383 DSR: 29-Jun-2012 15:09:36**

Sample Number	A	Analyte	Units	Std %-Rec	Blank	Result	Duplicate	Average	RPD	Spk %-Rec	Detection Limit	Count %-Error	Qual Flags
S12R000147		Gross alpha	uCi/mL	102	<4.63E-06	5.21E-05	n/a	n/a	n/a	n/a	3.27E-06	22.564	
S12R000147		Fluoride	ug/mL	97.0	<1.61E-03	20.3	n/a	n/a	n/a	n/a	0.0322	n/a	
S12R000147		Glycolate	ug/mL	102	<9.37E-03	<0.0937	n/a	n/a	n/a	n/a	0.0937	n/a	U
S12R000147		Acetate	ug/mL	103	<6.04E-03	3.02	n/a	n/a	n/a	n/a	0.0604	n/a	
S12R000147		Formate	ug/mL	106	<4.67E-03	2.91	n/a	n/a	n/a	n/a	0.0467	n/a	
S12R000147		Chloride	ug/mL	109	<9.98E-03	2.03	n/a	n/a	n/a	n/a	0.0998	n/a	
S12R000147		Nitrite	ug/mL	107	<0.0192	161	n/a	n/a	n/a	n/a	0.192	n/a	
S12R000147		Sulfate	ug/mL	107	<0.0187	19.7	n/a	n/a	n/a	n/a	0.187	n/a	
S12R000147		Oxalate	ug/mL	107	<0.0231	12.9	n/a	n/a	n/a	n/a	0.231	n/a	
S12R000147		Bromide	ug/mL	107	<0.0580	<0.580	n/a	n/a	n/a	n/a	0.580	n/a	U
S12R000147		Nitrate	ug/mL	108	<0.0208	83.7	n/a	n/a	n/a	n/a	0.208	n/a	
S12R000147		Phosphate	ug/mL	101	<0.0167	356	n/a	n/a	n/a	n/a	0.334	n/a	
S12R000147		Aluminum	ug/mL	97.3	<0.0300	9.5	n/a	n/a	n/a	n/a	3.00	n/a	J
S12R000147		Bismuth	ug/mL	105	<0.100	<10.0	n/a	n/a	n/a	n/a	10.0	n/a	U
S12R000147		Calcium	ug/mL	100	<0.0500	<5.00	n/a	n/a	n/a	n/a	5.00	n/a	U
S12R000147		Chromium	ug/mL	101	<5.00E-03	0.509	n/a	n/a	n/a	n/a	0.500	n/a	J
S12R000147		Iron	ug/mL	101	5.79E-03	1.49	n/a	n/a	n/a	n/a	0.500	n/a	BJ
S12R000147		Potassium	ug/mL	107	<0.500	<50.0	n/a	n/a	n/a	n/a	50.0	n/a	U
S12R000147		Manganese	ug/mL	100	<3.00E-03	<0.300	n/a	n/a	n/a	n/a	0.300	n/a	U
S12R000147		Sodium	ug/mL	100	<0.100	598	n/a	n/a	n/a	n/a	10.0	n/a	
S12R000147		Nickel	ug/mL	101	<0.0200	<2.00	n/a	n/a	n/a	n/a	2.00	n/a	U
S12R000147		Phosphorus	ug/mL	101	<0.0500	117	n/a	n/a	n/a	n/a	5.00	n/a	
S12R000147		Sulfur	ug/mL	102	<0.100	<10.0	n/a	n/a	n/a	n/a	10.0	n/a	U

C-109 Heel Solids: Aggregate Water Dissolution: Diluted[a] Contact 2 Liquid (2 sheets)

Sample Number	A	Analyte	Units	Std %-Rec	Blank	Result	Duplicate	Average	RPD	Spk %-Rec	Detection Limit	Count %-Error	Qual Flags
S12R000147		Silicon	ug/mL	99.5	<0.0300	4.11	n/a	n/a	n/a	n/a	3.00	n/a	J
S12R000147		Thorium	ug/mL	95.4	<0.0500	<5.00	n/a	n/a	n/a	n/a	5.00	n/a	U
S12R000147		Uranium	ug/mL	99.2	<0.100	15.6	n/a	n/a	n/a	n/a	10.0	n/a	J
S12R000147		Percent water	%	99.5	n/a	99.8	99.8	99.8	0.0401	n/a	0.01	n/a	
S12R000147		Total inorganic carbon	ug/mL	102	<7.00	92.2	n/a	n/a	n/a	n/a	7.00	n/a	
S12R000147		Total organic carbon	ug/mL	98.4	<20.0	59.1	n/a	n/a	n/a	n/a	20.0	n/a	J

[a] Aggregate Water Dissolution: 2nd Contact

	Wt (g)	*t* (°C)	D_{Liq} (g/mL)	Vol (mL)
Test Liquid	14.833	24.3	0.998	14.860
Added H_2O	3.715		0.9972	3.725
Dilution Factor	1.2507			

C-109 Heel Solids: Aggregate Water Dissolution: Diluted[a] Contact 3 Liquid (2 sheets) **Group: 20120383 DSR: 29-Jun-2012 15:09:36**

Sample Number	A	Analyte	Units	Std %-Rec	Blank	Result	Duplicate	Average	RPD	Spk %-Rec	Detection Limit	Count %-Error	Qual Flags
S12R000148		Gross alpha	uCi/mL	102	<4.63E-06	9.29E-05	n/a	n/a	n/a	n/a	3.28E-06	15.53	
S12R000148		Fluoride	ug/mL	97.0	<1.61E-03	4.77	n/a	n/a	n/a	n/a	8.05E-03	n/a	
S12R000148		Glycolate	ug/mL	102	<9.37E-03	<9.37E-03	n/a	n/a	n/a	n/a	9.37E-03	n/a	U
S12R000148		Acetate	ug/mL	103	<6.04E-03	0.52	n/a	n/a	n/a	n/a	6.04E-03	n/a	
S12R000148		Formate	ug/mL	106	<4.67E-03	0.542	n/a	n/a	n/a	n/a	4.67E-03	n/a	
S12R000148		Chloride	ug/mL	109	<9.98E-03	2.2	n/a	n/a	n/a	n/a	9.98E-03	n/a	
S12R000148		Nitrite	ug/mL	107	<0.0192	19.2	n/a	n/a	n/a	n/a	0.0192	n/a	
S12R000148		Sulfate	ug/mL	107	<0.0187	3.75	n/a	n/a	n/a	n/a	0.0187	n/a	
S12R000148		Oxalate	ug/mL	107	<0.0231	2	n/a	n/a	n/a	n/a	0.0231	n/a	
S12R000148		Bromide	ug/mL	107	<0.0580	<0.0580	n/a	n/a	n/a	n/a	0.0580	n/a	U
S12R000148		Nitrate	ug/mL	108	<0.0208	16.9	n/a	n/a	n/a	n/a	0.0208	n/a	
S12R000148		Phosphate	ug/mL	101	<0.0167	94.4	n/a	n/a	n/a	n/a	0.0835	n/a	
S12R000148		Aluminum	ug/mL	97.3	<0.0300	16.1	n/a	n/a	n/a	n/a	3.00	n/a	J
S12R000148		Bismuth	ug/mL	105	<0.100	<10.0	n/a	n/a	n/a	n/a	10.0	n/a	U
S12R000148		Calcium	ug/mL	100	<0.0500	<5.00	n/a	n/a	n/a	n/a	5.00	n/a	U
S12R000148		Chromium	ug/mL	101	<5.00E-03	<0.500	n/a	n/a	n/a	n/a	0.500	n/a	U
S12R000148		Iron	ug/mL	101	5.79E-03	1.19	n/a	n/a	n/a	n/a	0.500	n/a	BJ
S12R000148		Potassium	ug/mL	107	<0.500	<50.0	n/a	n/a	n/a	n/a	50.0	n/a	U
S12R000148		Manganese	ug/mL	100	<3.00E-03	<0.300	n/a	n/a	n/a	n/a	0.300	n/a	U
S12R000148		Sodium	ug/mL	100	<0.100	132	n/a	n/a	n/a	n/a	10.0	n/a	
S12R000148		Nickel	ug/mL	101	<0.0200	<2.00	n/a	n/a	n/a	n/a	2.00	n/a	U
S12R000148		Phosphorus	ug/mL	101	<0.0500	38.5	n/a	n/a	n/a	n/a	5.00	n/a	J
S12R000148		Sulfur	ug/mL	102	<0.100	<10.0	n/a	n/a	n/a	n/a	10.0	n/a	U

C-109 Heel Solids: Aggregate Water Dissolution: Diluted[a] Contact 3 Liquid (2 sheets)

Sample Number	A	Analyte	Units	Std %-Rec	Blank	Result	Duplicate	Average	RPD	Spk %-Rec	Detection Limit	Count %-Error	Qual Flags
S12R000148		Silicon	ug/mL	99.5	<0.0300	3.56	n/a	n/a	n/a	n/a	3.00	n/a	J
S12R000148		Thorium	ug/mL	95.4	<0.0500	<5.00	n/a	n/a	n/a	n/a	5.00	n/a	U
S12R000148		Uranium	ug/mL	99.2	<0.100	<10.0	n/a	n/a	n/a	n/a	10.0	n/a	U
S12R000148		Percent water	%	98.1	n/a	98.6	98.4	98.5	0.234	n/a	0.01	n/a	
S12R000148		Total inorganic carbon	ug/mL	102	<7.00	19.2	n/a	n/a	n/a	n/a	7.00	n/a	J
S12R000148		Total organic carbon	ug/mL	98.4	<20.0	<20.0	n/a	n/a	n/a	n/a	20.0	n/a	U

[a] Aggregate Water Dissolution: 3rd Contact

	Wt (g)	*t* (°C)	D_{Liq} (g/mL)	Vol (mL)
Test Liquid	14.534	24.2	0.996	14.589
Added H_2O	3.607		0.9972	3.617
Dilution Factor	1.2479			

C-109 Heel Solids: Aggregate Caustic Dissolution: Final Test Liquid **Group: 20120471** **DSR: 12-Apr-2012 13:08:14**

Sample Number	A	Analyte	Units	Std %-Rec	Blank	Result	Duplicate	Average	RPD	Spk %-Rec	Detection Limit	Count %-Error	Qual Flags
S12R000174		Fluoride	ug/mL	91.4	<1.61E-03	2.49	n/a	n/a	n/a	n/a	0.161	n/a	J
S12R000174		Glycolate	ug/mL	91.8	<9.37E-03	<0.937	n/a	n/a	n/a	n/a	0.937	n/a	U
S12R000174		Acetate	ug/mL	95.9	<6.04E-03	<0.604	n/a	n/a	n/a	n/a	0.604	n/a	U
S12R000174		Formate	ug/mL	96.3	<4.67E-03	8.08	n/a	n/a	n/a	n/a	0.467	n/a	J
S12R000174		Chloride	ug/mL	94.5	<9.98E-03	5.36	n/a	n/a	n/a	n/a	0.998	n/a	J
S12R000174		Nitrite	ug/mL	98.3	<0.0192	10.8	n/a	n/a	n/a	n/a	1.92	n/a	J
S12R000174		Sulfate	ug/mL	96.2	<0.0187	<1.87	n/a	n/a	n/a	n/a	1.87	n/a	U
S12R000174		Oxalate	ug/mL	96.2	<0.0231	<2.31	n/a	n/a	n/a	n/a	2.31	n/a	U
S12R000174		Bromide	ug/mL	95.0	<0.0580	<5.80	n/a	n/a	n/a	n/a	5.80	n/a	U
S12R000174		Nitrate	ug/mL	96.7	<0.0208	17.7	n/a	n/a	n/a	n/a	2.08	n/a	J
S12R000174		Phosphate	ug/mL	96.9	<0.0167	63.8	n/a	n/a	n/a	n/a	1.67	n/a	
S12R000174		Aluminum	ug/mL	91.9	<0.0300	5.71E+04	5.58E+04	5.65E+04	2.39	91.3	120	n/a	
S12R000174		Bismuth	ug/mL	106	<0.100	<400	<400	n/a	n/a	104	400	n/a	U
S12R000174		Calcium	ug/mL	109	<0.0500	525	363	444	36.4	108	200	n/a	J
S12R000174		Chromium	ug/mL	97.4	<5.00E-03	<20.0	22.6	n/a	n/a	96.9	20.0	n/a	U
S12R000174		Iron	ug/mL	98.0	<5.00E-03	25.2	31.1	28.1	21.2	96.6	20.0	n/a	J
S12R000174		Manganese	ug/mL	96.8	<3.00E-03	<12.0	<12.0	n/a	n/a	97.4	12.0	n/a	U
S12R000174		Sodium	ug/mL	91.6	<0.100	1.70E+05	1.56E+05	1.63E+05	8.64	81.2	400	n/a	
S12R000174		Nickel	ug/mL	97.9	<0.0200	<80.0	<80.0	n/a	n/a	96.6	80.0	n/a	U
S12R000174		Phosphorus	ug/mL	95.0	<0.0500	<200	<200	n/a	n/a	94.7	200	n/a	U
S12R000174		Sulfur	ug/mL	97.0	<0.100	<400	<400	n/a	n/a	95.7	400	n/a	U
S12R000174		Silicon	ug/mL	93.0	<0.0300	<120	<120	n/a	n/a	107	120	n/a	U
S12R000174		Uranium	ug/mL	93.9	<0.100	<400	<400	n/a	n/a	93.9	400	n/a	U
S12R000174		Hydroxide	ug/mL	94.4	<41.7	8.68E+04	8.89E+04	8.78E+04	2.44	98.5	1.11E+03	n/a	
S12R000174		Specific gravity	None	100.5	n/a	1.329	1.323	1.326	0.4525	n/a	1.00E-03	n/a	
S12R000174		Total inorganic carbon	ug/mL	106	<7.00	70.5	19.9	45.2	112	84.5	7.00	n/a	B
S12R000174		Total organic carbon	ug/mL	100	<20.0	58.5	110	84.2	61.1	109	20.0	n/a	J

C-109 Heel Solids: Aggregate Dissolution: Residual Solids (2 sheets) **Group: 20120602** **DSR: 22-May-2012 15:37:49**

Sample Number	A	Analyte	Units	Std %-Rec	Blank	Result	Duplicate	Average	RPD	Spk %-Rec	Detection Limit	Count %-Error	Qual Flags
S12R000192		Iodine-129	uCi/g	97.3	<2.48E-05	<2.51E-05	<6.06E-05	n/a	n/a	n/a	2.51E-05	n/a	U
S12R000192		Total inorganic carbon	ug/g	100	<7.00	216	239	228	10.1	90.6	42.3	n/a	J
S12R000192		Total organic carbon	ug/g	96.7	<20.0	635	748	692	16.3	109	121	n/a	J
S12R000193	Z	Curium-243/244	uCi/g	n/a	<1.64E-04	<1.15E-04	<9.60E-05	n/a	n/a	n/a	1.15E-04	n/a	U
S12R000193	Z	Americium-241	uCi/g	96.4	2.16E-04	0.0303	0.0300	0.0302	0.995	n/a	1.21E-04	2.23	
S12R000193	Z	Cesium-137	uCi/g	105	<0.268	0.480	<0.278	n/a	n/a	n/a	0.349	26.42	
S12R000193	Z	Actinium-228	uCi/g	n/a	<1.37	<1.25	0.576	n/a	n/a	n/a	1.25	n/a	U
S12R000193	Z	Aluminum	ug/g	89.7	<3.14E+03	3.28E+05	3.20E+05	3.24E+05	2.69	92.7	3.14E+03	n/a	
S12R000193	Z	Bismuth	ug/g	100	<1.05E+04	<1.05E+04	<6.81E+03	n/a	n/a	108	1.05E+04	n/a	U
S12R000193	Z	Calcium	ug/g	110	<5.24E+03	<5.24E+03	n/a	n/a	n/a	107	5.24E+03	n/a	U
S12R000193	Z	Chromium	ug/g	98.3	529	<524	<340	n/a	n/a	107	524	n/a	U
S12R000193	Z	Iron	ug/g	98.9	<524	1.35E+03	1.45E+03	1.40E+03	7.61	104	524	n/a	J
S12R000193	Z	Manganese	ug/g	98.0	<314	<314	<204	n/a	n/a	106	314	n/a	U
S12R000193	Z	Sodium	ug/g	94.7	<1.05E+04	<1.05E+04	<6.81E+03	n/a	n/a	93.8	1.05E+04	n/a	U
S12R000193	Z	Nickel	ug/g	98.4	<2.10E+03	<2.10E+03	2.32E+03	n/a	n/a	104	2.10E+03	n/a	U
S12R000193	Z	Phosphorus	ug/g	99.4	<5.24E+03	<5.24E+03	<3.40E+03	n/a	n/a	102	5.24E+03	n/a	U
S12R000193	Z	Sulfur	ug/g	98.5	<1.05E+04	<1.05E+04	<6.81E+03	n/a	n/a	101	1.05E+04	n/a	U
S12R000193	Z	Silicon	ug/g	95.6	<3.14E+03	<3.14E+03	n/a	n/a	n/a	99.3	3.14E+03	n/a	U
S12R000193	Z	Uranium	ug/g	95.2	<1.05E+04	<1.05E+04	<6.81E+03	n/a	n/a	94.5	1.05E+04	n/a	U
S12R000193	Z	Uranium-233	ug/g	101	<2.10	<2.10	<1.36	n/a	n/a	98.7	2.10	n/a	U
S12R000193	Z	Uranium-234	ug/g	n/a	<0.0524	<0.0524	<0.0340	n/a	n/a	n/a	0.0524	n/a	IU
S12R000193	Z	Uranium-235	ug/g	98.8	<0.105	1.73	1.52	1.62	12.9	107	0.105	n/a	
S12R000193	Z	Uranium-236	ug/g	n/a	<0.0419	<0.0419	<0.0272	n/a	n/a	n/a	0.0419	n/a	IU
S12R000193	Z	Neptunium-237	ug/g	103	<1.05	<1.05	<0.681	n/a	n/a	98.3	1.05	n/a	U
S12R000193	Z	Uranium-238	ug/g	98.3	<5.24	264	244	254	7.81	99.9	5.24	n/a	
S12R000193	Z	Plutonium-239/240	uCi/g	104	<4.78E-04	0.0133	0.0125	0.0129	6.20	n/a	1.76E-04	4.33	
S12R000193	Z	Plutonium-238	uCi/g	n/a	<3.11E-04	3.88E-04	2.93E-03	1.66E-03	153	n/a	2.12E-04	77.50	J

C-109 Heel Solids: Aggregate Dissolution: Residual Solids (2 sheets)

Sample Number	A	Analyte	Units	Std %-Rec	Blank	Result	Duplicate	Average	RPD	Spk %-Rec	Detection Limit	Count %-Error	Qual Flags
S12R000193	Z	Strontium-89/90	uCi/g	96.3	0.157	528	489	508	7.80	n/a	0.0621	0.629	
S12R000194	A	Technetium-99	ug/g	109	<6.00E-03	<0.919	<0.891	n/a	n/a	99.3	0.919	n/a	U
S12R000195	A	Aluminum	ug/g	82.7	<1.50	1.15E+05	1.24E+05	1.20E+05	7.18	1.96E+4	51.1	n/a	
S12R000195	A	Bismuth	ug/g	85.3	<5.00	<179	<214	n/a	n/a	104	179	n/a	U
S12R000195	A	Calcium	ug/g	75.7	<2.50	518	547	532	5.58	118	89.4	n/a	Ja
S12R000195	A	Chromium	ug/g	83.3	0.392	<8.94	15.3	n/a	n/a	120	8.94	n/a	U
S12R000195	A	Iron	ug/g	85.8	<0.250	1.12E+03	1.45E+03	1.28E+03	25.3	145	8.94	n/a	b
S12R000195	A	Manganese	ug/g	83.0	<0.150	<5.36	<6.41	n/a	n/a	116	5.36	n/a	U
S12R000195	A	Sodium	ug/g	83.4	<5.00	1.32E+03	1.27E+03	1.30E+03	4.11	184	179	n/a	Jb
S12R000195	A	Nickel	ug/g	84.4	<1.00	1.65E+03	n/a	n/a	n/a	159	35.8	n/a	b
S12R000195	A	Phosphorus	ug/g	87.3	<2.50	<89.4	<107	n/a	n/a	119	89.4	n/a	U
S12R000195	A	Sulfur	ug/g	83.9	<5.00	<179	<214	n/a	n/a	118	179	n/a	U
S12R000195	A	Silicon	ug/g	79.7	<1.50	154	166	160	7.36	104	51.1	n/a	J
S12R000195	A	Uranium	ug/g	91.6	<5.00	239	310	275	25.8	122	179	n/a	J
S12R000195	A	Zirconium	ug/g	84.7	<0.250	<8.94	n/a	n/a	n/a	103	8.94	n/a	U

ATTACHMENT B

CAUSTIC SOLUTION

ATTACHMENT B – CAUSTIC SOLUTION

A nominal 50% w/w caustic solution was required for the retrieval caustic dissolution test on the C-109 heel solids. Approximately 600 mL of the solution was prepared on March 2, 2012, 5 days prior to initiation of the caustic dissolution tests.

Approximately 500 g of reagent grade NaOH pellets were dried overnight in an oven at 110 °C. The NaOH pellets were then allowed to cool in a dessicator. The primary solution was then prepared as:

Dry NaOH pellets	442.44	g
Cold reagent H_2O	442.60	g
Solution	885.04	g

A small quantity of fine, white solids were present in the caustic solution. The solids were removed by vacuum filtration. The entire volume of the caustic solution was filtered twice using 3-µm polymer filter disks. The air-dry recovered solids weighed 4-5 g.

The density of the caustic solution was measured gravimetrically. Three 100-mL, Class A volumetric flasks were filled with the solution and weighed. The recorded measurement data is presented in Table B-1.

Table B-1. Gravimetric Measurement of Density of Caustic Solution.

	Units	Flask A	Flask B	Flask C
Wt Flask + H_2O	g	156.932	157.095	156.711
Wt Flask	g	57.409	57.447	57.281
Wt H_2O	g	99.523	99.648	99.430
t H_2O	°C	23.0	23.0	23.0
Reference Density H_2O	g/mL	0.997538	0.997538	0.997538
Vol Flask	mL	99.769	99.894	99.675
Wt Flask + Caustic Solution	g	209.155	209.525	209.023
Wt Flask	g	57.410	57.449	57.282
Wt Caustic Solution	g	151.745	152.076	151.741
t Caustic Solution	°C	22.5	22.3	22.4
Density of Caustic Solution	g/mL	1.52097	1.52237	1.52235
Average	g/mL	1.52190		
1 σ	g/mL	0.00066		
t	°C	22.4		

The density of the caustic solution was also measured using an Anton Paar[8] DMA 35N portable density meter. The DMA 35N meter measures liquid densities using the oscillating U-tube principle. The data for this density measurement is presented in Table B-2.

[8] Anton Paar is a registered trademark of Anton Paar GmbH, Austria, Europe.

Table B-2. Density of Caustic Solution Measured with Density Meter.

Liquid	Measured Density (g/mL)	*t* (°C)	Reference Density (g/mL)	($Density_{Measured}$ − $Density_{Reference}$) (g/mL)	Corrected Density (g/mL)	
Reagent H_2O	0.9983	21.7	0.99784	0.00046	—	
	0.9983	21.7	0.99784	0.00046	—	
	0.9985	21.6	0.99786	0.00064	—	
	0.9981	21.6	0.99786	0.00024	—	
	0.9983	21.6	0.99786	0.00044	—	
Caustic Solution	1.5205	22.1	—	—	1.51999	
	1.5209	22.3	—	—	1.52039	Average 1.52053
	1.5211	22.2	—	—	1.52059	1 σ 0.00032
	1.5213	22.2	—	—	1.52079	22.2 °C
	1.5214	22.1	—	—	1.52089	
Reagent H_2O	0.9984	21.7	0.99784	0.00056	—	
	0.9985	21.6	0.99786	0.00064	—	
	0.9985	21.7	0.99784	0.00066	—	

Reference data for the relationships between density and wt% and between density and molarity (M) were collected from the *CRC Handbook of Chemistry and Physics* and from the *Kirk-Othmer Encyclopedia of Chemical Technology*. This data was then plotted and fitted with 3rd order polynomials yielding the following relationships:

$$\text{Wt\% NaOH} = [42.2216\ (D_{20}^3)] - [143.3969\ (D_{20}^2)] + [(252.4432\ (D_{20})] - 151.1727 \qquad R^2 = 0.999993 \qquad \text{(B-1)}$$

and

$$\text{M NaOH} = [17.5190\ (D_{20}^3)] - [37.3572\ (D_{20}^2)] + [(45.6683\ (D_{20})] - 25.8163 \qquad R^2 = 0.999994 \qquad \text{(B-2)}$$

Based on these measurements and relationships, the prepared caustic solution had a density of 1.5212 g/mL at 22.3 °C, contained 49.644 wt% NaOH, and was 18.875 M in NaOH.

Immediately following the completion of the density measurements, the caustic solution was transferred to three 8-oz plastic bottles, sealed tightly, and transferred to the 11A hot cells.

REFERENCES

CRC Handbook of Chemistry and Physics, *65th Edition*, CRC Press, Inc., Boca Raton, Florida, 1984.

Kirk-Othmer Encyclopedia of Chemical Technology, 3rd Edition, John Wiley & Sons, Inc., New York, New York, 1978, Vol. 1.

ATTACHMENT C

PARTICLE SIZE DISTRIBUTION ANALYSIS

ATTACHMENT C – PARTICLE SIZE DISTRIBUTION ANALYSIS

1.0 INTRODUCTION

Measurement of the particle size distribution (PSD) in the solids remaining after completion of large-scale dissolution testing on a composite sample of tank 241-C-109 (C-109) heel solids was specified in a client-approved test plan (LAB-PLN-11-00006, "Test Plan for Tank 241-C-109 Heel Solids Characterization and Dissolution Testing"). The measurement was made in two steps. First, the residual solids from the large-scale dissolution tests were separated into >710-µm and <710-µm fractions by wet sieving. A subsample (S12R000199) of the <710-µm solids was collected for the light-scattering-based PSD measurements. The light-scattering-based PSD measurements and results are the subject of this attachment.

2.0 SAMPLE

All measurements described in this attachment were made on portions of the <710-µm sieve fraction of the solids remaining after completion of large-scale dissolution testing on the C-109 crushed heel solids composite. The parent sample for all analytical specimens was S12R000199. The multiple sampling and measurement steps leading to the subsample are described in detail in the main body of this report.

3.0 PARTICLE SIZE DISTRIBUTION ANALYSIS

3.1 INSTRUMENTATION

Particle size distribution measurements were performed using the Horiba®[9] LA-910 Laser Scattering Particle Size Distribution Analyzer in hood 2 in room 1F of the 222-S Laboratory (222-S). The LA-910 is an ensemble type, light-scattering-based PSD analyzer; it is not a sensing-zone or image-analysis type of instrument where measurements or observations of individual particles are made. The measurement actually performed defines the light-scattering pattern created when a large number of sample particles scatter the light in the focused beam(s) of the analytical probe(s). Analyzer software, using iterative algorithms based on Mie Scattering Theory, creates a virtual population of optically isotropic and homogeneous, spherical particles with a distribution of diameters that would, given the same experimental parameters, generate a similar light-scattering pattern. The frequencies of occurrence of particles of various sizes in these populations are weighted according to the volumes of the virtual spherical particles. The

[9] Horiba is a registered trademark of Horiba, Ltd. Corporation Japan, Kyoto, Japan

reported particle sizes are the diameters of these virtual spherical particles, i.e., equivalent spherical diameters.

The LA-910 analyzer uses dual light sources: a helium-neon laser (λ = 632.8 nm) and a 40-W, tungsten-halogen, blue-filtered lamp (λ = 450 nm). The dual light sources and physical design of the detector array allow measurement of PSDs for samples with particles ranging from 0.02 μm to 1020 μm in equivalent spherical diameter.

The LA-910 analyzer was operated in flow cell mode for the PSD measurements. In this mode, a dilute slurry of sample solids is continuously circulated through the analyzer during PSD measurements. Key components in the measurement loop include the following.

Sample Tank. Sample solids and suspension liquid are introduced into a stainless-steel sample tank (maximum volume ≈280 mL). Specimen suspensions are generally limited to ≈210 mL to minimize both contamination within the sample tank compartment and the volume of analytical waste generated. Suspensions enter the measurement loop from the bottom of the tank.

Stirrer. A three-bladed, impeller-type, mechanical stirrer (≈1-in. diameter) in the sample tank assists in suspending the sample solids in the suspension liquid and is critical in introducing a uniform sample suspension into the flow loop. The stirrer speed is adjustable in seven step settings, S1 through S7. In distilled water, a stirrer speed setting of S6 represents a stirring speed of ≈1000 rpm.

A maximum stirrer speed setting of S3 is used when the total sample volumes are ≈210 ml of aqueous slurry. A greater speed risks drawing the bottom of the liquid vortex down to the stirrer blades resulting in entrainment of air into the slurry and splattering in the sample tank compartment. Tests have shown that the stirrer speed at the S2 setting is the minimum required to effectively sample spherical particles with density ≈2.5 g/cm^3 and with diameters up to 350 μm in aqueous suspensions.

Ultrasonic Generator. The sample tank is also the chamber of a low-power (40 W, 39 kHz), standing-wave type ultrasonic bath. The ultrasonic bath is provided to facilitate dispersion of sample particulates in the suspension liquid. The power of the bath is fixed. The operator may select whether or not to activate the ultrasonic generator. If activated, the operator may adjust the time the bath operates before the light-scattering measurement begins and the length of any delay period between termination of the ultrasonic treatment and initiation of the light-scattering measurement. The operator may also elect to continue ultrasonic treatment during the PSD measurement. For brevity, ultrasonic treatment of samples hereafter is referred to as "sonication."

Circulation Pump. Located immediately downstream from the sample tank, a variable-occlusion peristaltic pump circulates the sample slurry through the analyzer measurement loop. The pump speed is adjustable in seven step settings, P1 through P7. Pump speed settings of P3

and P5 were used in these measurements. The discharge volume at the P5 setting is ≈10.6 mL/s using Tygon®[10] tubing with an internal diameter of 4.8 mm.

The occlusion of the Masterflex®[11] peristaltic pump head is adjusted so that the pump tubing is not totally compressed (occluded) at any point of the rotor rotation. Performance tests using this occlusion setting and various pump and stirrer speed combinations have shown that a stirrer speed setting of P2 combined with a pump speed setting of S4 is the minimum combination required to satisfactorily reproduce the PSD of a certified standard containing soda-lime glass microspheres (density of 2.4–2.5 g/cm^3) with diameters ranging from 50-350 µm—Whitehouse Scientific[12] standard PS223.

Measurement Cell. The flow cell is located immediately downstream from the circulation pump. The two optical windows of the cell are Tempax®[13] glass (with a nonreflective coating on the exterior surfaces). The dimensions of the interior specimen cavity of the flow cell are 70 mm × 45 mm × 3.5 mm (H × W × D). The analytical light probes traverse paths through the depth of the cell perpendicular to the cell windows. The sample suspension enters at the bottom of the measurement cell, exits at the top of the cell, and returns to the sample tank.

3.2 ANALYSIS SPECIMENS

Each PSD specimen was ≈210 mL of a dilute slurry of the <710-µm C-109 residual solids in reagent water. The optimum volumetric concentration of solids in a specimen is approximately proportional to particle size: about 0.002% for particles with diameters of 0.5 µm and about 1% for particles with diameters of 500 µm (ISO-13320-1, *Particle size analysis – Laser diffraction methods – Part 1: General principles*). In practice, an attempt was made to add an amount of sample solids that resulted in obscuration of the analyzer light beams by at least 5% but not more than 30%.

Subsampling of the <710-µm residual solids was performed using a technique recommended in ISO-13320-1. The PSD sample (S12R000199) had been allowed to settle, undisturbed, for 22 days. Prior to the PSD measurements, all the clear supernatant liquid was carefully removed from the sample vial using a disposable pipette. The sample solids were then gently but thoroughly mixed in the vial by stirring with a microspatula. As noted in ISO-13320-1, the ideal consistency of the samples, which minimizes segregation errors, is one like "honey or toothpaste." After addition of a few drops of reagent water, the consistency of the <710-µm C-109 residual solids was within this range.

At the beginning of each of the triplicate PSD measurements, a 210-mL charge of reagent water was added to the analyzer sample tank, and stirring and pumping were initiated. Immediately prior to each PSD measurement, the contents of the sample vial were remixed by stirring with a

[10] Tygon is a registered trademark of Norton Company, Worcester, Massachusetts.
[11] Masterflex is a registered trademark of the Cole-Parmer Instrument Company, Vernon Hills, Illinois.
[12] Whitehouse Scientific, Waverton, Chester, England.
[13] Tempax is a registered trademark of Schott Glaswerkes, Mainz, Germany.

small spatula. A small portion of the sample paste was then transferred to the water in the sample tank of the PSD analyzer using the spatula.

3.3 MEASUREMENTS

3.3.1 Procedure

Particle size distribution measurements were made on three specimens (primary, duplicate, and triplicate portions) of the <710-µm C-109 residual solids. The PSD measurement on each specimen was actually a set of three sequential PSD runs.

The procedure for completing a set of three PSD runs for a single specimen was as follows:

1. Approximately 210 mL of reagent water was added to the analyzer sample tank.
2. The pump speed was set to P5, the stirrer speed to S3, and the water charge was sonicated for 2 min. (This treatment helps to degas the liquid and ensures the measurement loop is free from particulate contamination and bubbles.)
3. A blank measurement was completed.
4. The pump speed was set to P3 and the stirrer speed was set to S1.
5. Portion(s) of the sample were transferred to the sample tank until transmission of light through the measurement cell was reduced by 5%-30% (i.e., %-transmission values were between 70% and 95%).
6. Run 1 analyzer settings (Table C-1) were established, and the Run 1 measurement sequence was started.
7. When Run 1 was complete and the presence of a saved data file was verified, Run 2 analyzer settings were established, and the Run 2 measurement sequence was started.
8. When Run 2 was complete and the presence of a saved data file was verified, Run 3 analyzer settings were established, and the Run 3 measurement sequence was started.
9. When Run 3 was complete, the presence of a saved data file was verified.
10. The specimen suspension was drained from the analyzer sample loop.
11. The analyzer sample loop was flushed with reagent water until the light-scattering pattern returned to a particulate-free profile.

The analyzer settings and sequence times that were used in each PSD run are presented in Table C-1.

Table C-1. Particle Size Distribution Analysis Run Settings.

	PSD Run			
	Load-In[a]	**1**	**2**	**3**
Specimen Circulation	—	—		—
Pump (circulation) speed setting[b]	P3	P5	P5	P5
Stirrer (agitation) speed setting[b]	S1	S3	S3	S3
Premeasurement	—	—	—	—
Work time (s)	As needed	6	120	120
Ultrasonics ON[c]	No	No	No	Yes
Premeasurement wait (s)[d]	N/A	0	0	6[d]
Measurement	—	—	—	—
Measure cycles (laser/lamp)[e]	N/A	36/36	36/36	36/36
Sonication during measure	N/A	No	No	No

[a] Settings established while specimen solids were being transferred to the sample tank of the PSD analyzer.

[b] Speed settings for the peristaltic pump (P) used to circulate specimen slurry through the analyzer flow loop and the impeller type stirrer (S) used to maintain homogeneity of the specimen slurry. Selectable pump speeds range from 1-7; selectable stirrer speeds range from 0-7.

[c] 'Ultrasonics' or 'sonication' refers to treatment of specimen suspension in a low-power (40 W) standing-wave type ultrasonic bath incorporated in the analyzer flow loop.

[d] When the specimen slurry is sonicated before but not during measurement, a short delay is commonly inserted between termination of the sonication and initiation of the light-scattering measurement.

[e] A composite measurement combining 36 readouts of detectors while illuminating specimen with a He-Ne laser and 36 readouts while illuminating the sample with a blue-filtered lamp. Total measurement time is ≈60 s.

3.3.2 Specimen Load-In

The set of analyzer settings identified as "Load-In" were used while each PSD specimen was being transferred to the analyzer sample tank. The time required to transfer each of the triplicate specimens to the analyzer tank averaged 1 min.

3.3.3 Refractive Indexes

Conversion of the light-scattering patterns measured by the analyzer to PSDs requires input of a relative refractive index (RRI) —

$$\text{RRI} = \text{Refractive index (}\boldsymbol{p}\text{articles) / Refractive index (liquid }\boldsymbol{m}\text{edium).} \qquad \text{(C-1)}$$

The accuracy of this input becomes increasingly important as the diameters of the particles become smaller than ≈25 μm (ISO 13320-1).

The complex refractive index (N) of a substance is defined as

$$N = n - ki, \qquad \text{(C-2)}$$

where the real index, n, represents the degree of refraction of light in the material. The complex (or imaginary) part of the index, ki, is directly proportional to the degree of attenuation

(primarily by absorption) of the probe light beam(s) by a material. Transparent materials have small extinction coefficients (k); opaque and/or highly colored materials have larger coefficients.

In practice, it is assumed that a suspension liquid that strongly absorbs at the wavelength(s) of the analytical probes will not be used and that minor absorption by the liquid will be accounted for by blank measurements. The RRI value actually input is

$$\text{RRI} = (n_p/n_m) - k_p i. \tag{C-3}$$

As previously noted, the liquid medium used in these measurements was pure water. The real refractive index of water, n_m, is 1.333 at λ = 589.3 nm.

Solid phase characterization of the <710-µm C-109 residual solids indicated that gibbsite [$Al(OH)_3$] was the dominant solid phase present. An average value of the real portion of the refractive index for gibbsite, 1.575[14], was input for n_p of the C-109 residual solids.

Within the PSD analyzer software (except for specialized, well-defined applications), spherical particles of transparent materials are assigned an extinction coefficient (k_p) of 0.00. Opaque and colored materials, particularly those of unknown composition, are generally assigned extinction coefficients ranging from 0.10 to 1.00. The <710-µm C-109 residual solids were (visually) a light tan to moderate reddish-brown in color. Microscopic examination also suggested that the surfaces of most of the solid particles were moderately to highly textured (surface texture can contribute to k_p). In the absence of other sample specific input, an intermediate value of k_p = 0.10 was assigned.

Based on the preceding discussion, the RRI value used in the PSD calculations for the <710-µm C-109 residual solids was

$$\text{RRI} = (n_p/n_m) - k_p i = (1.575/1.333) - 0.10i = 1.18 - 0.10i. \tag{C-4}$$

If the client has input suggesting that an alternate RRI value would be more appropriate or desirable, PSDs can be recalculated using revised value(s) without reanalysis of physical specimens.

3.4 DEVIATIONS FROM TEST PLAN

No deviations from the PSD measurement requirements specified in test plan LAB-PLN-11-00006 occurred.

[14] Crystalline gibbsite actually has three values of n_p: a = 1.569, b = 1.569, and g = 1.5865. The average value of n_p = 1.575 was used in the RRI calculation. Small changes in values of n_p (and RRI) have little effect on calculated PSDs.

4.0 PARTICLE SIZE DISTRIBUTION RESULTS

The results of the light-scattering-based PSD measurements on the <710-µm C-109 heel solids dissolution test residues are presented in this section. As previously noted, these measurements were made on three specimens taken from sample vial S12R000199. The data files for corresponding PSD runs on the primary, duplicate, and triplicate specimens were subsequently combined numerically to produce the 'average' data presented in this report.

The scattered-light patterns measured by the LA-910 analyzer are directly related to the volumes of small particles and the cross-sectional areas of larger particles. The default analyzer output is a PSD with frequencies of occurrence weighted according to the volumes of the scattering particles. The analyzer software allows these primary distributions to be recalculated as area-, length-, or number-based distributions. These converted distributions are obtained by re-weighting the original, partially processed, volume-based data using the appropriate power of the particle diameters (d) and then renormalizing the resulting distributions. For example, number-based PSDs are obtained by applying weighting factors proportional to $1/d^3$ to the original volume-based PSD data. All numerical PSD results presented in this report are derived from the volume-based PSDs. The histograms and %-undersize curves derived from the recalculated number-based PSDs are presented in this attachment for comparison.

All the PSD data presented for the <710-µm C-109 residual solids are based on 'standard-form' distributions. These distributions are generated when the analyzer calculation algorithms proceed through 30 iterations as opposed to the 150 iterations used to calculate 'sharp-form' distributions. Standard-form distributions are broader with poor resolution of distinct features. However, unless specific knowledge of the PSD of a sample is available, a large number of calculation iterations can cause the results of the deconvolution/inversion function to diverge rather than converge to a reasonable result and create distinct features in the reported PSD when only small discontinuities in the light-scattering patterns were actually measured.

4.1 TRANSCRIPTS OF RAW DATA FILES

The default data sets generated by the Horiba® LA-910 analyzer consist of 80 logarithmically spaced particle-diameter bins that are populated with occurrence frequency data. Transcripts of these "raw" data files for the volume-based distributions for the <710-µm C-109 residual solids are provided on the following three pages. Each data sheet presents the averaged results for one of the three PSD runs on the primary, duplicate, and triplicate analysis specimens. The Diameter vs. Frequency % and Undersize % data tables can be provided as Excel spreadsheets if requested. Data files for the recalculated number-based PSDs can also be provided on request.

The span values associated with the reported PSD median diameters are a measure of the breadth of the PSDs. The span is calculated as

(90%-undersize diameter – 10%-undersize diameter) / median diameter

SAMPLE MATERIAL:	241-C-109 Heel Solids: <710 µm Large-scale Dissolution Test Residue
SAMPLE #:	S12R000199 — Average of Run 1 on three specimens
MEASUREMENT ID:	05/02/2012 — (Runs 058, 061, and 064)
FILENAME:	S12R199R1Avg

INSTRUMENT:	Horiba LA-910
SUSPENSION LIQUID	Water
DISPERSANT:	None
DISPERSION METHOD:	Stirring and pumping in analyzer during load-in and measurement
FLOW REGIME:	Flow Cell Measurement

RRI:	1.18 – 0.10i
DISTRIBUTION BASE:	Volume
DISTRIBUTION FORM:	Standard

STIR SET: 3 PUMP SET: 5 SONIC: OFF DELAY: 0 s SONIC DURING MEAS: NO

MEASURE (Laser/Lamp Cycles): 36/36 Σ WORK TIME: 2.4 min Σ SONIC: 0 min

MEAN (µm): 40.6 MEDIAN (µm): 34.9 MODE (µm): 42.0 S.P. Area (cm^2/cm^3): 6044

σ (µm): 30.5 SPAN: 1.942

Diameter (µm)	Frequency (%)	Undersize (%)	Diameter (µm)	Frequency (%)	Undersize (%)	Diameter (µm)	Frequency (%)	Undersize (%)
0.020	0.00	0.00	0.766	0.33	1.75	29.91	6.40	41.81
0.023	0.00	0.00	0.877	0.46	2.21	34.25	7.14	48.94
0.026	0.00	0.00	1.005	0.58	2.79	39.23	7.71	56.65
0.030	0.00	0.00	1.151	0.55	3.34	44.94	7.96	64.61
0.034	0.00	0.00	1.318	0.47	3.81	51.47	7.76	72.37
0.039	0.00	0.00	1.510	0.28	4.09	58.95	7.08	79.45
0.044	0.00	0.00	1.729	0.21	4.30	67.52	6.00	85.45
0.051	0.00	0.00	1.981	0.20	4.50	77.34	4.72	90.16
0.058	0.00	0.00	2.269	0.23	4.73	88.58	3.44	93.61
0.067	0.00	0.00	2.599	0.20	4.93	101.5	2.36	95.97
0.076	0.00	0.00	2.976	0.21	5.15	116.2	1.55	97.52
0.087	0.00	0.00	3.409	0.23	5.37	133.1	0.98	98.50
0.100	0.00	0.00	3.905	0.25	5.62	152.5	0.60	99.10
0.115	0.00	0.00	4.472	0.30	5.92	174.6	0.38	99.47
0.131	0.00	0.00	5.122	0.39	6.30	200.0	0.25	99.72
0.150	0.00	0.00	5.867	0.51	6.81	229.1	0.15	99.87
0.172	0.00	0.00	6.720	0.68	7.49	262.4	0.08	99.96
0.197	0.00	0.00	7.697	0.90	8.38	300.5	0.05	100.00
0.226	0.00	0.00	8.816	1.16	9.54	344.2	0.00	100.00
0.259	0.00	0.00	10.10	1.46	11.00	394.2	0.00	100.00
0.296	0.00	0.00	11.56	1.80	12.80	451.6	0.00	100.00
0.339	0.13	0.13	13.25	2.21	15.01	517.2	0.00	100.00
0.389	0.20	0.33	15.17	2.71	17.72	592.4	0.00	100.00
0.445	0.26	0.59	17.38	3.30	21.02	678.5	0.00	100.00
0.510	0.28	0.87	19.90	4.00	25.02	777.1	0.00	100.00
0.584	0.27	1.14	22.80	4.79	29.81	890.1	0.00	100.00
0.669	0.28	1.42	26.11	5.60	35.41	1019.5	0.00	100.00

SAMPLE MATERIAL:	241-C-109 Heel Solids: <710 µm Large-scale Dissolution Test Residue
SAMPLE #:	S12R000199 — Average of Run 2 on three specimens
MEASUREMENT ID:	05/02/2012 — (Runs 059, 062, and 065)
FILENAME:	S12R199R2Avg

INSTRUMENT:	Horiba LA-910
SUSPENSION LIQUID	Water
DISPERSANT:	None
DISPERSION METHOD:	Stirring and pumping for 2.0 minutes before measurement
FLOW REGIME:	Flow Cell Measurement

RRI:	1.18 – 0.10i
DISTRIBUTION BASE:	Volume
DISTRIBUTION FORM:	Standard

STIR SET: 3 PUMP SET: 5 SONIC: OFF DELAY: 0 s SONIC DURING MEAS: NO

MEASURE (Laser/Lamp Cycles): 36/36 Σ WORK TIME: 5.6 min Σ SONIC: 0 min

MEAN (µm): 41.1 MEDIAN (µm): 34.7 MODE (µm): 42.0 S.P. Area (cm^2/cm^3): 6104

σ (µm): 31.9 SPAN: 2.017

Diameter (µm)	Frequency (%)	Undersize (%)	Diameter (µm)	Frequency (%)	Undersize (%)	Diameter (µm)	Frequency (%)	Undersize (%)
0.020	0.00	0.00	0.766	0.34	1.75	29.91	6.37	42.31
0.023	0.00	0.00	0.877	0.47	2.23	34.25	7.05	49.35
0.026	0.00	0.00	1.005	0.60	2.82	39.23	7.55	56.90
0.030	0.00	0.00	1.151	0.57	3.39	44.94	7.74	64.64
0.034	0.00	0.00	1.318	0.48	3.87	51.47	7.51	72.15
0.039	0.00	0.00	1.510	0.29	4.16	58.95	6.85	79.00
0.044	0.00	0.00	1.729	0.21	4.37	67.52	5.84	84.84
0.051	0.00	0.00	1.981	0.21	4.58	77.34	4.65	89.49
0.058	0.00	0.00	2.269	0.24	4.82	88.58	3.47	92.96
0.067	0.00	0.00	2.599	0.21	5.03	101.5	2.46	95.42
0.076	0.00	0.00	2.976	0.22	5.25	116.2	1.67	97.09
0.087	0.00	0.00	3.409	0.24	5.49	133.1	1.11	98.20
0.100	0.00	0.00	3.905	0.26	5.74	152.5	0.70	98.89
0.115	0.00	0.00	4.472	0.31	6.05	174.6	0.45	99.34
0.131	0.00	0.00	5.122	0.40	6.45	200.0	0.27	99.62
0.150	0.00	0.00	5.867	0.52	6.97	229.1	0.19	99.81
0.172	0.00	0.00	6.720	0.69	7.66	262.4	0.14	99.95
0.197	0.00	0.00	7.697	0.92	8.58	300.5	0.05	100.00
0.226	0.00	0.00	8.816	1.18	9.76	344.2	0.00	100.00
0.259	0.00	0.00	10.10	1.49	11.25	394.2	0.00	100.00
0.296	0.00	0.00	11.56	1.84	13.08	451.6	0.00	100.00
0.339	0.13	0.13	13.25	2.25	15.33	517.2	0.00	100.00
0.389	0.20	0.33	15.17	2.75	18.08	592.4	0.00	100.00
0.445	0.26	0.59	17.38	3.36	21.44	678.5	0.00	100.00
0.510	0.28	0.87	19.90	4.05	25.49	777.1	0.00	100.00
0.584	0.27	1.14	22.80	4.83	30.32	890.1	0.00	100.00
0.669	0.28	1.42	26.11	5.62	35.94	1019.5	0.00	100.00

SAMPLE MATERIAL:	241-C-109 Heel Solids: <710 µm Large-scale Dissolution Test Residue
SAMPLE #:	S12R000199 — Average of Run 3 on three specimens
MEASUREMENT ID:	05/02/2012 — (Runs 060, 063, and 066)
FILENAME:	S12R199R3Avg

INSTRUMENT:	Horiba LA-910
SUSPENSION LIQUID	Water
DISPERSANT:	None
DISPERSION METHOD:	Stirring, pumping and sonication for 2.0 minutes before measurement
FLOW REGIME:	Flow Cell Measurement

RRI:	1.18 – 0.10i
DISTRIBUTION BASE:	Volume
DISTRIBUTION FORM:	Standard

STIR SET: 3 PUMP SET: 5 SONIC: ON DELAY: 6 s SONIC DURING MEAS: NO

MEASURE (Laser/Lamp Cycles): 36/36 Σ WORK TIME: 9.0 min Σ SONIC: 2.0 min

MEAN (µm): 35.8 MEDIAN (µm): 28.8 MODE (µm): 36.5 S.P. Area (cm^2/cm^3): 8202

σ (µm): 34.3 SPAN: 2.085

Diameter (µm)	Frequency (%)	Undersize (%)	Diameter (µm)	Frequency (%)	Undersize (%)	Diameter (µm)	Frequency (%)	Undersize (%)
0.020	0.00	0.00	0.766	0.43	2.64	29.91	7.40	51.98
0.023	0.00	0.00	0.877	0.60	3.24	34.25	7.83	59.81
0.026	0.00	0.00	1.005	0.74	3.98	39.23	7.86	67.67
0.030	0.00	0.00	1.151	0.71	4.69	44.94	7.37	75.04
0.034	0.00	0.00	1.318	0.62	5.30	51.47	6.41	81.45
0.039	0.00	0.00	1.510	0.40	5.70	58.95	5.16	86.61
0.044	0.00	0.00	1.729	0.30	6.01	67.52	3.86	90.47
0.051	0.00	0.00	1.981	0.31	6.31	77.34	2.72	93.19
0.058	0.00	0.00	2.269	0.34	6.65	88.58	1.85	95.04
0.067	0.00	0.00	2.599	0.31	6.96	101.5	1.26	96.29
0.076	0.00	0.00	2.976	0.32	7.27	116.2	0.89	97.18
0.087	0.00	0.00	3.409	0.33	7.60	133.1	0.66	97.84
0.100	0.00	0.00	3.905	0.36	7.96	152.5	0.50	98.34
0.115	0.00	0.00	4.472	0.41	8.37	174.6	0.43	98.76
0.131	0.00	0.00	5.122	0.52	8.90	200.0	0.35	99.11
0.150	0.00	0.00	5.867	0.67	9.56	229.1	0.31	99.42
0.172	0.00	0.00	6.720	0.87	10.44	262.4	0.28	99.71
0.197	0.00	0.00	7.697	1.14	11.58	300.5	0.16	99.86
0.226	0.00	0.00	8.816	1.45	13.03	344.2	0.09	99.95
0.259	0.00	0.00	10.10	1.81	14.83	394.2	0.05	100.00
0.296	0.15	0.15	11.56	2.22	17.05	451.6	0.00	100.00
0.339	0.23	0.38	13.25	2.72	19.77	517.2	0.00	100.00
0.389	0.33	0.70	15.17	3.33	23.10	592.4	0.00	100.00
0.445	0.39	1.09	17.38	4.06	27.16	678.5	0.00	100.00
0.510	0.39	1.48	19.90	4.91	32.07	777.1	0.00	100.00
0.584	0.37	1.84	22.80	5.83	37.89	890.1	0.00	100.00
0.669	0.36	2.21	26.11	6.69	44.58	1019.5	0.00	100.00

4.2 HISTOGRAMS AND PERCENT-UNDERSIZE CURVES

Volume-based histograms and %-undersize curves depicting the results of the averaged PSD run data on the three specimens of sample S12R000199 are presented in Figure C-1.

Recalculated number-based histograms and %-undersize curves for the averaged PSD run data are presented in Figure C-2 for comparison. The number-based histograms and curves emphasize the degree to which volume-based data can be influenced by the presence of relatively small populations of larger particles.

The header for each PSD run displays the stirrer speed setting, the circulation pump speed setting, whether the specimen slurry was sonicated for 2 min, and the average duration of the run (in minutes). For Run 1, the average duration includes the approximate time required to introduce the PSD specimen into the analyzer sample tank.

4.3 CUMULATIVE %-UNDERSIZE DATA

The particle diameters associated with five pre-selected points on the cumulative %-undersize curves describing the volume-based PSDs for the <710-µm C-109 residual solids are presented in Table C-2. The particle diameters associated with 10, 25, 50, 75, and 90 %-undersize cut points are presented in the table. (These values are commonly reported as d10, d25, d50, d75, and d90 particle diameters, respectively.) The 50 %-undersize values are, by definition, the median particle diameters of the PSDs.

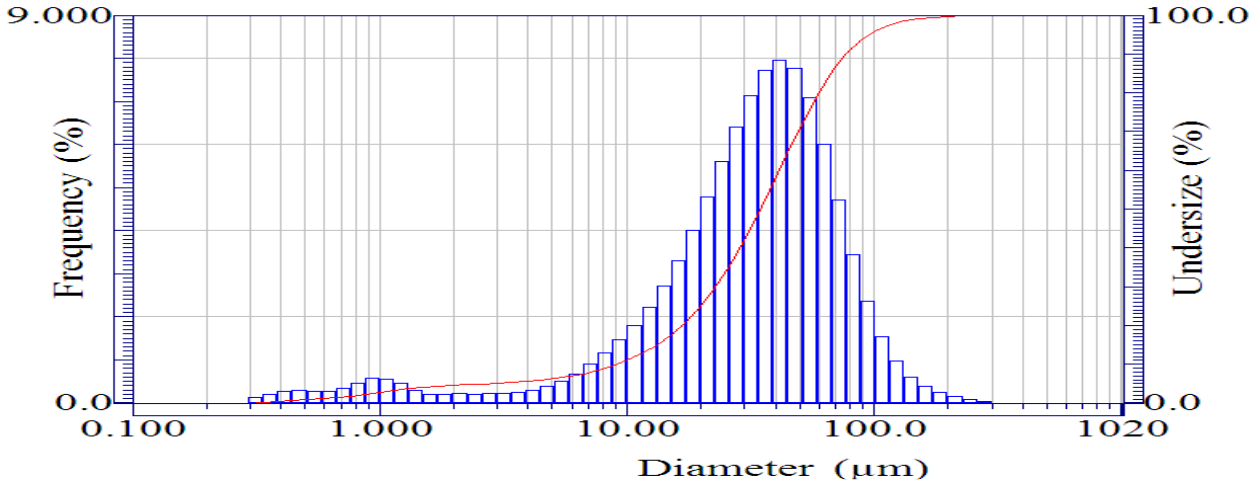
Table C-2. Diameter versus %-Undersize for <710-µm Residual Solids.

Specimen	PSD Run	Diameters of Particles (µm) at Volume-based %-Undersize Cut Points				
		10%	25%	50%	75%	90%
Primary	1	9.1	19.7	35	54	77
	2	8.9	19.2	34	54	78
	3	6.2	15.9	28	43	62
Duplicate	1	9.1	19.6	34	52	73
	2	8.7	19.0	33	51	71
	3	5.8	15.5	27	41	56
Triplicate	1	9.4	20	36	57	83
	2	9.5	20	37	59	89
	3	6.9	17.3	31	53	96

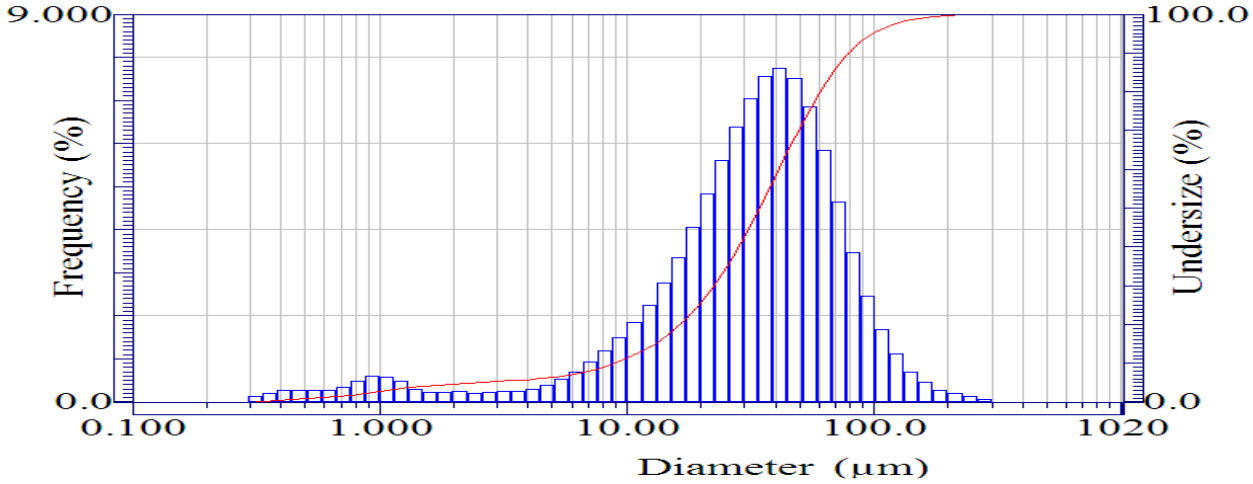
The cumulative %-undersize data for the averaged PSD run files is presented in the main body of this report.

Figure C-1. Averaged, Volume-based Particle Size Distribution Histograms and %-Undersize Curves for <710-µm Residual Solids.

Run 1 — Stir Speed: S1-3 Pump Speed: P3-5 Sonication: None Duration: 2.4 min



Run 2 — Stir Speed: S3 Pump Speed: P5 Sonication: None Duration: 3.2 min



Run 3 — Stir Speed: S3 Pump Speed: P5 Sonication: 2.0 min Duration: 3.4 min

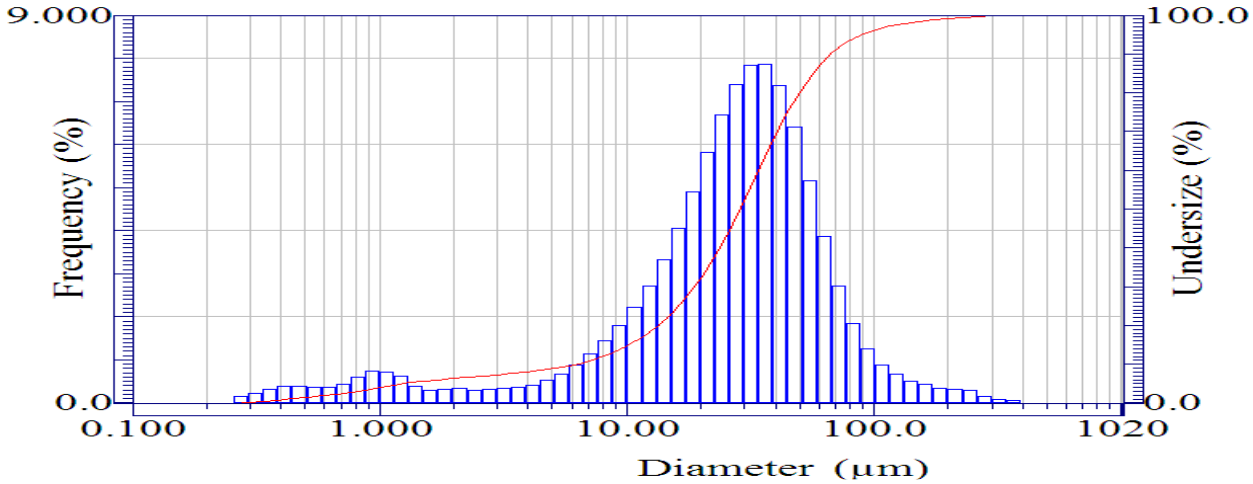
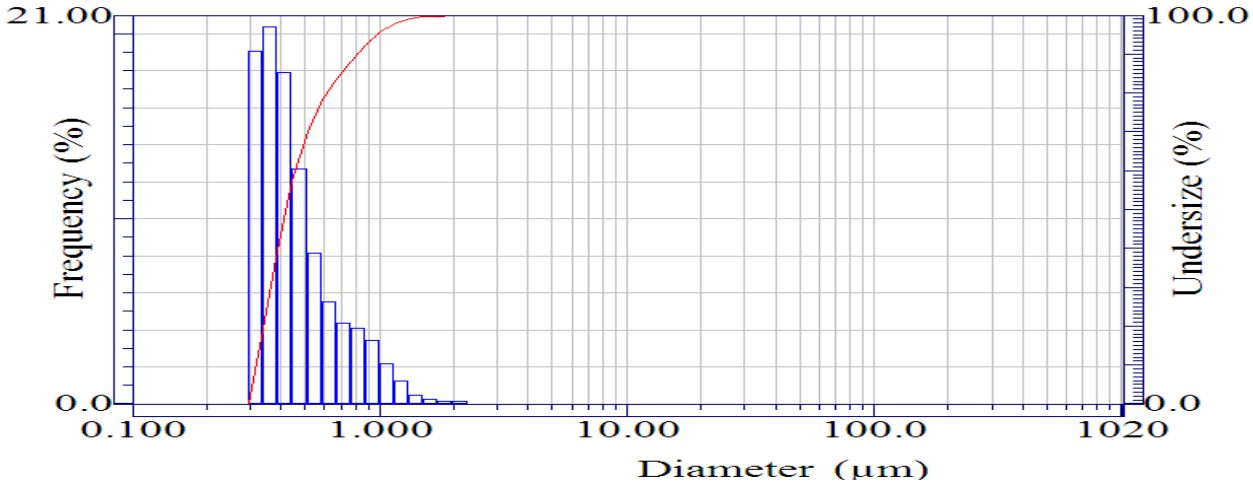
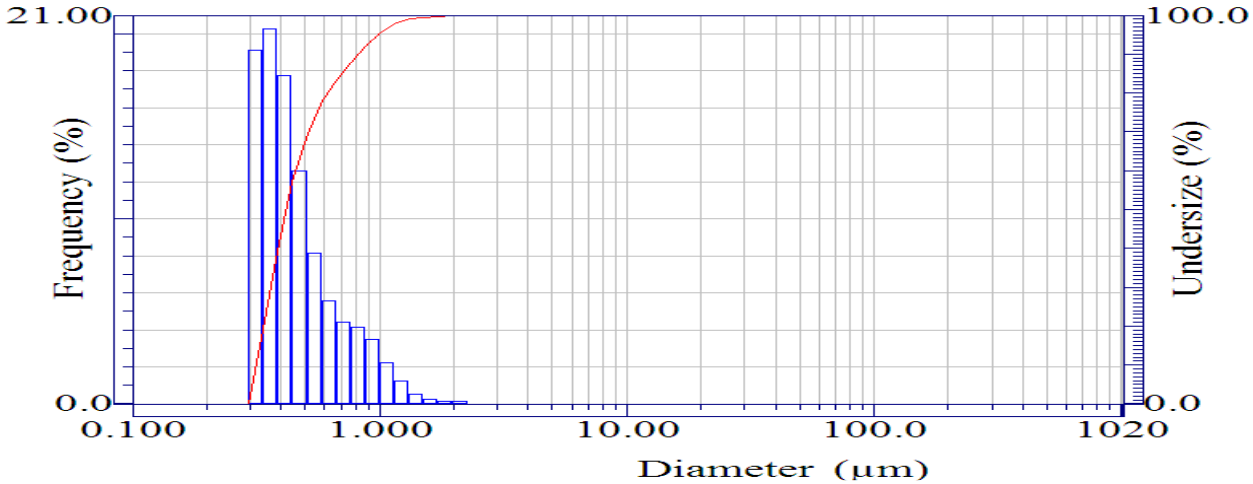


Figure C-2. Averaged, Number-based Particle Size Distribution Histograms and %-Undersize Curves for <710-µm Residual Solids.

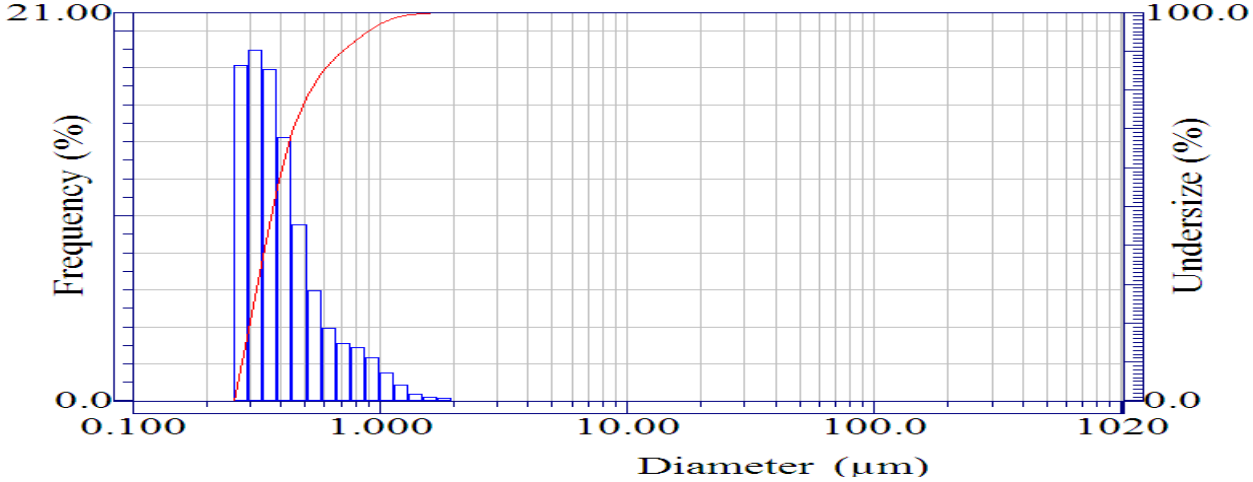
Run 1 — Stir Speed: S1-3 Pump Speed: P3-5 Sonication: None Duration: 2.4 min



Run 2 — Stir Speed: S3 Pump Speed: P5 Sonication: None Duration: 3.2 min



Run 3 — Stir Speed: S3 Pump Speed: P5 Sonication: 2.0 min Duration: 3.4 min



5.0 DISCUSSION OF MEASUREMENT RESULTS

Discussion of the results of these PSD measurements in relation to the characterization of the C-109 residual solids is included in the main body of this report. Discussion pertinent to evaluating the limitations on the accuracy or applicability of the light-scattering-based measurements is presented in this attachment.

5.1 PARTICLE SIZE DISTRIBUTION MEASUREMENT CAVEATS

Light-scattering-based PSD measurements are based on several assumptions:

a. All sample particles are spherical.
b. All particles with diameters less than ≈25 μm are compositionally and/or optically identical.
c. The optical properties of both the particles and the suspending medium are well known when samples contain particles with diameters less than ≈25 μm.
d. There is no interaction between light scattered from different particles (i.e., no multiple scattering phenomena).

Deviations from these assumptions will introduce some degree of error in the PSD measurements due to the inability of the deconvolution and inversion algorithms to account for the deviations.

It should also be reemphasized that the Horiba® LA-910 is an ensemble type, light-scattering-based PSD analyzer, not a sensing-zone or image-analysis type of instrument. Direct observation and/or measurement of individual particles are not made. The calculated PSDs are based, in part, on assumptions regarding the shapes and statistical properties of distributions that may not apply to the samples being measured.

5.1.1 Range

The results reported for the PSD analyses apply only to particles with diameters within the 0.02-1020 μm measuring range of the analyzer. The calculated PSDs are normalized so that the sum of the occurrence frequencies of particles within this range is always 100%. This should not be taken to represent that particles with diameters <0.02 μm or >1020 μm were determined, by measurement, to be absent from the samples.

Since the analyzed sample solids had passed through a 710-μm sieve, the absence of particulates >710 μm in diameter was as expected. Visual observation and tactile evaluation during mixing and transfer of PSD specimens to the analyzer also supported this expectation.

The presence of particles with diameters <0.02 μm cannot be ruled out by these PSD measurements.

5.1.2 Nonspherical Particles

All light-scattering-based PSD data are presented in terms of an equivalent spherical diameter—the diameter of a spherical particle having the same light-scattering function as that assigned to the sample particle. By design, a nonlaminar, quasi-turbulent flow regime is maintained in the LA-910 flow cell. Under "ideal" conditions, the equivalent spherical diameter reported for any nonspherical particle would be derived from a combination of all cross-sectional diameters that a rapidly and randomly rotating particle could present to the probe light beams. The degree to which measurements on any significantly nonspherical component of a real, nonuniform sample actually achieves this ideal cannot be determined without undertaking an extensive study including both light-scattering-based and direct measurements of particle dimensions. This type of developmental program was not performed as part of these PSD measurements.

The degree to which PSD measurements on nonspherical components in the <710-µm C-109 residual solids have yielded meaningful, averaged spherical diameters or the effect their presence might have on the overall measured distributions is not known. The degree to which (even given ideal performance of the analyzer and its software package) averaged particle dimensions address actual project requirements should also be carefully considered.

5.1.3 Dispersion of Sample Particles

Laser diffraction instruments cannot distinguish between scattering by single particles and scattering by clusters of primary particles forming an agglomerate or aggregate. Usually the measured particle size for agglomerates or aggregates is related to the cluster size, but sometimes the size of the primary particles is reflected in the PSD as well[15] (ISO 13320-1). Furthermore, no technique not based on direct observation of sample solids can distinguish between agglomerates that may exist in a sample in its native state and agglomerates that may form as a result of the measurement process (e.g., by introduction of the sample solids into a different liquid medium with different electrostatic properties). As a result, ensuring a good degree of dispersion prior to sample analysis is generally considered to be an important step to ensure reliable and reproducible size analysis (NIST SP 960-1, *NIST Recommended Practice Guide: Particle Size Characterization*).

In the current PSD measurements, Run 1 on each specimen was made under conditions where dispersive forces and the time of their application were purposefully minimized. In Run 2 the intensity of the dispersive forces was not changed but the duration of their action on the sample solids was extended to 2 min. In Run 3, low-energy sonication of the specimen suspensions, a standard dispersive technique in PSD analysis, was employed.

The results of the Run 1 and Run 2 measurements were very similar indicating that additional dissolution of sample solids, precipitation of new solids, or dispersal of weakly bound

[15] In some cases the light-scattering pattern produced by agglomerates or aggregates can be interpreted as multiple scattering resulting from localized high concentrations of discrete particles. In extreme cases, the calculated diameters may not only be unrelated to the cluster size but also may underestimate the diameters of the primary particles.

agglomerates was occurring slowly, if at all, under the low-shear conditions applied. The Run 3 distributions shifted slightly towards smaller particle diameters. It is suspected that this shift was the result of the break-up of friable solid aggregates that were present in the particulates in the 10-100 μm size range.

5.1.4 Refractive Indexes

In numerous cases, the results of a particle size analysis are only as good as the optical model chosen to interpret and convert the measured pattern of scattered light into a PSD (NIST SP 960-1). In particular, input of accurate refractive indexes of the sample solids and suspension liquid to the algorithms can be of critical importance.

When particle diameters are much larger than the wavelength of the light probe(s), scattering is effectively described as Fraunhofer diffraction and is independent of the optical properties of the sample material. To describe the scattering of light by smaller particles (down to diameters somewhat smaller than the light wavelength), use of Mie Scattering Theory is required. Application of Mie Theory requires that the complex refractive indexes of both the (assumed optically isotropic and spherical) particulate phase (N_p) and the suspension liquid (N_m) be known. This requirement is of increasing importance as the (a) particle diameters approach or become smaller than the wavelength(s) of the light scattered, (b) particles become increasingly transparent to the light probe(s), (c) particulates significantly absorb at the wavelength(s) of the light probe(s), and/or (d) refractive indexes of the liquid and solid phases approach one another.

The minimum particle size at which the Fraunhofer approximation holds varies depending on the actual solid-liquid system being measured. As a general rule (ISO 13320-1), the accuracy of the optical model data is not a significant concern for particles with diameters >50 μm and has only minor impact for particles with diameters as small as 18 μm to 25 μm for the 450-nm and 632-nm light sources employed in the LA-910 analyzer. The input of accurate optical data is of increasing importance as the diameters of sample particles become smaller than 25 μm and is critical when particle diameters are less than 1 μm-2 μm.

A small change in the assigned RRI may cause a significant change in calculated PSDs. Also, the effect of the RRI on PSDs calculated for samples containing particles of diverse composition and morphology is, generally, quite complicated. Unfortunately, it is sometimes difficult, even for a single well-defined phase, to obtain an accurate value for the real index of refraction (n_p). It is often very difficult to obtain an accurate value for the imaginary component (k_pi) of the complex refractive index: absorption is often strongly dependent on wavelength, and the extinction coefficient can also be affected by surface structure of the particles (e.g., surface roughness) and intraparticle density heterogeneity. Indeed, it is common practice to determine appropriate values for the imaginary part (and often the real part also) of the refractive index using trial-and-error procedures of size determination using a microscopy-based technique, a light-scattering-based instrument, and samples of the solids to be measured.

For the <710-μm C-109 residual solids, the light-scattering-based PSD measurements indicated that ≈75% of the particulate volume (and presumably mass) resides in particles with diameters

<50 μm and 30-35% resides in particles with diameters <25 μm. This is generally supported by the observed settling behavior of the solids. Visual observation suggested that the residual solids were generally opaque and lightly colored though some darkly colored phases were also present.

The <710-μm C-109 heel solids are believed to have been composed predominantly of gibbsite and were assigned an average value of the real index of refraction for that phase, $n_p = 1.575$. Small amounts of other solid phases with unknown values of n_p were also present. The impact of the assigned value of n_p on the measurement of the PSD for these phases is unknown. The assignment of $k_p i = 0.10i$ for the particulate solids was based on the visual appearance of the PSD sample solids, but is also unsupported by experiment or theory.

The real index of refraction of pure water is $n_m = 1.333$. If the assigned values of n_p and n_m are both reasonably accurate, they are sufficiently different to allow light-scattering-based measurements to be completed successfully.

If the RRI of 1.18 – 0.10i applies, with moderate tolerance, to the majority of the <710-μm C-109 residual solids in the aqueous specimen slurries, the calculated PSDs should reasonably characterize those present in the sample. If large and small diameter particulates are comprised of different solid phases with significantly different indexes of refraction, the calculated PSDs may differ significantly from those actually present in the sample.

5.1.5 Specimen Size

Volume-based PSD measurements can be very sensitive to the presence of small numbers of large-diameter particles that can nevertheless represent a very large fraction of the sample particulate volume. For example, in a population of 10^6 particles, 1 particle with a diameter 100 times larger than the diameter of the remaining 999,999 particles would have a number-based occurrence frequency of 0.0001%. However, in a volume-based distribution the single large particle would represent 50% of the particulate volume. Clearly, in samples containing a broad range of particle sizes, obtaining an analytical specimen in which the relative proportions of large and small particles are accurately represented is of importance. This requirement can, however, create a dilemma.

Particle concentration in specimen suspensions (by volume) should be above a minimum level required to produce an acceptable signal-to-noise ratio. In the LA-910 analyzer this concentration corresponds to a reduction in %-transmission to about 95% (reduction in transmission of the probe light beam(s) through the suspension by 5% relative to the particle-free suspension liquid). To avoid multiple scattering, the solids concentration should be below a level that corresponds to about 65%-transmission for particles larger than 20 μm and about 85%-transmission for smaller particles (ISO 13320-1). In general, the multiple scattering that results when the specimen suspensions are significantly overconcentrated redistributes scattered light intensity to larger scattering angles resulting in calculation of minimum particle diameters that are smaller than the true values.

The dilemma arises when analyzing samples containing a broad range of particle sizes including some with relatively large diameters. Statistics indicate that a specimen that will accurately and reproducibly represent the proportion of both the large and small particles in the parent sample must be of a minimum weight or volume (*Particle Size Measurement: Volume 1 − Powder Sampling and Particle Size Measurement* [Allen 1997]). However, if specimens of this size were analyzed, the target %-transmission limits for the PSD suspensions would often be exceeded; indeed, the target values for the small diameter particles would be exceeded very often. Smaller specimen sizes are used in order to limit the effects of multiple scattering by the smaller particles even though the accuracy in representation of the relative amounts of larger particles present is potentially sacrificed.

It was relatively easy to achieve the desired levels of light obscuration when loading the <710-µm C-109 residual solids into the PSD analyzer. Accordingly, PSD artifacts caused by multiple scattering should be insignificant.

5.2 REPRESENTATIVENESS

Particle size analysis is carried out on samples extracted from the bulk, which, irrespective of the precautions taken, never represent the bulk exactly. The degree to which the samples withdrawn from tank C-109 represented the bulk of the heel solids present is unknown. The heel solids samples were then subjected to a multitude of test operations to produce the residual solids analyzed herein. Finally, two additional sampling steps were required to transfer specimens of the <710-µm C-109 residual solids into the PSD analyzer. Clearly, the degree to which the PSD results can or should be taken to represent solids that might remain in C-109 after actual retrieval operations in the field must be carefully considered.

The measured PSDs indicated that ≈90% of the solids volume in the <710-µm C-109 residual solids was in particles <80 µm in diameter and that essentially no particles with diameters in the range of 300-710 µm were present. However, the settling rate test results (Section 7.3) suggested that a small portion of particulates in the 300-710 µm size range may actually have been present. Direct measurements on the 18.8-g settling test sample would be expected to describe the quantities of large and small particulates in the residual solids more accurately than indirect measurements on the <200-mg PSD specimens. Also, for radioactive samples containing very small quantities of relatively large particles in a mass of much smaller particulates, physical constraints on withdrawing very small PSD subsamples could easily lead to underrepresentation of the larger particulates.

6.0 QUALITY CONTROL

6.1 REPEAT MEASUREMENTS

As previously noted, PSD measurements were performed on triplicate portions (specimens) withdrawn from sample S12R000199 of the <710-µm solids remaining after the large-scale dissolution tests on the C-109 heel solids. The distributions calculated for each of the three PSD runs on the triplicate specimens were quite similar qualitatively and quantitatively. The closeness of this agreement suggests that the protocols followed in the preparation and transfer of specimens to the PSD analyzer and in the execution of the measurements themselves resulted in PSD analyses with an acceptable level of precision (repeatability).

6.2 STANDARD MEASUREMENTS

Measurements of the PSDs of certified particle size standards were performed before sample measurements began and after they were completed. The standards measured were selected from a set of samples of monodisperse, polystyrene microspheres acquired from Thermo Scientific.[16] The 222-S Standards Laboratory acquired the standards and maintains files of the original standard certificates. These standards have mean diameter(s) certified with linear dimensions transferred from the National Institute of Standards and Technology. The results of the standard measurements are tabulated in Table C-3. The calculated mean particle diameters are within ±10% of the certified values for all five standard measurements.

Table C-3. Results of Particle Size Distribution Measurements on Certified Standards.

Standard	Mean Diameter		PSD Run File
	Certified	Measured	
Thermo Scientific #4010A (Lot 37781) Polystyrene microspheres	1.034 ± 0.021 µm	1.024 ± 0.075 µm (–1.0 %-Error)	201205011343057 PS-1um-050112a
Thermo Scientific #4314A (Lot 39966) Polystyrene microspheres	138 ± 2.2 µm	141 ± 14 µm (+2.2 %-Error)	201205030846076 PS-140um-050312a

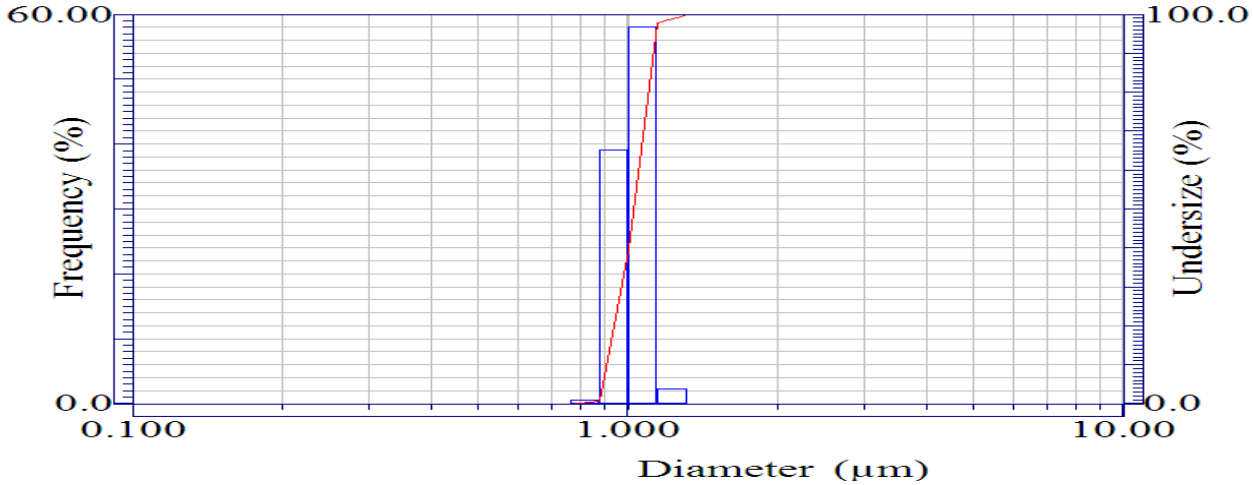
The measured PSDs for the standards are presented in Figure C-3. The sharp-form distributions presented are appropriate for these uniformly sized (i.e., monodisperse) standards.

[16] Thermo Fisher Scientific Inc., Waltham, Massachusetts.

Figure C-3. Volume-based Histograms and %-Undersize Curves for Particle Size Distribution Standards.

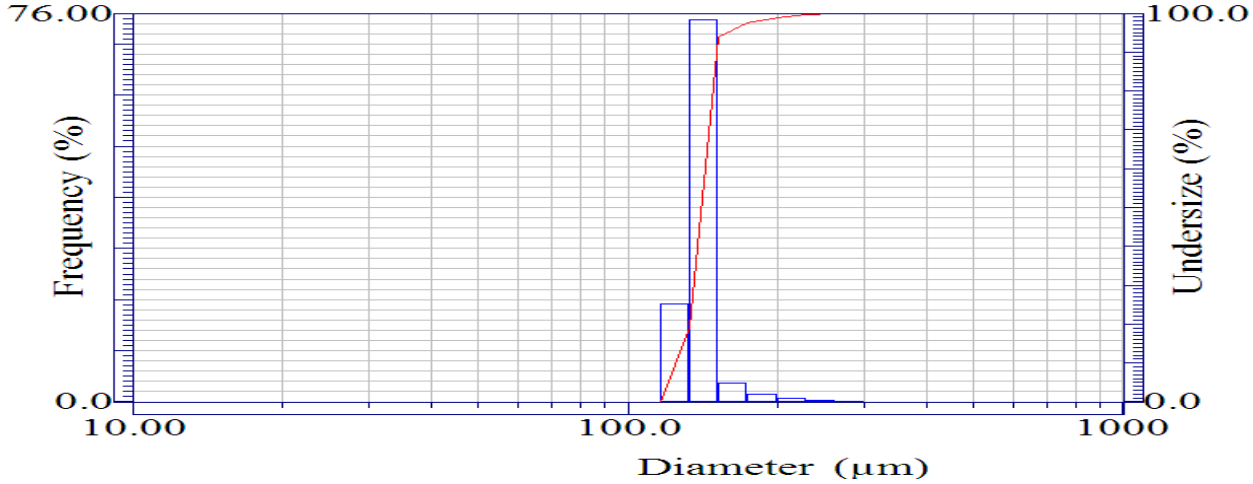
(a) Thermo Scientific 4010A (Lot 37781)
Polystyrene spheres
Distribution form: Sharp

Run Date: May 1, 2012—0343
Certified Mean Diameter: 1.034 ± 0.021 µm
Calculated Mean Diameter: 1.02 ± 0.07 µm



(b) Thermo Scientific 4314A (Lot 39966)
Polystyrene spheres
Distribution form: Sharp

Run Date: May 3, 2012—0846
Certified Mean Diameter: 138 ± 2.2 µm
Calculated Mean Diameter: 141 ± 14 µm



7.0 REFERENCES

Allen, T., 1997, *Particle Size Measurement: Volume 1 – Powder Sampling and Particle Size Measurement*, 5th Edition, Powder Technology Series, Chapman and Hall, New York, New York.

ISO-13320-1, 1999, *Particle size analysis – Laser diffraction methods – Part 1: General principles*, 1st Edition, International Organization for Standardization, Geneva, Switzerland.

LAB-PLN-11-00006, 2011, *Test Plan for Tank 241-C-109 Heel Solids Characterization and Dissolution Testing*, Rev. 0, Washington River Protection Solutions LLC, Richland, Washington.

NIST SP 960-1, 2001, *NIST Recommended Practice Guide: Particle Size Characterization*, National Institute of Standards and Technology, U.S. Department of Commerce, Gaithersburg, Maryland.

ATTACHMENT D

SOLID PHASE CHARACTERIZATION OF RESIDUAL SOLIDS

ATTACHMENT D
SOLID PHASE CHARACTERIZATION OF RESIDUAL SOLIDS

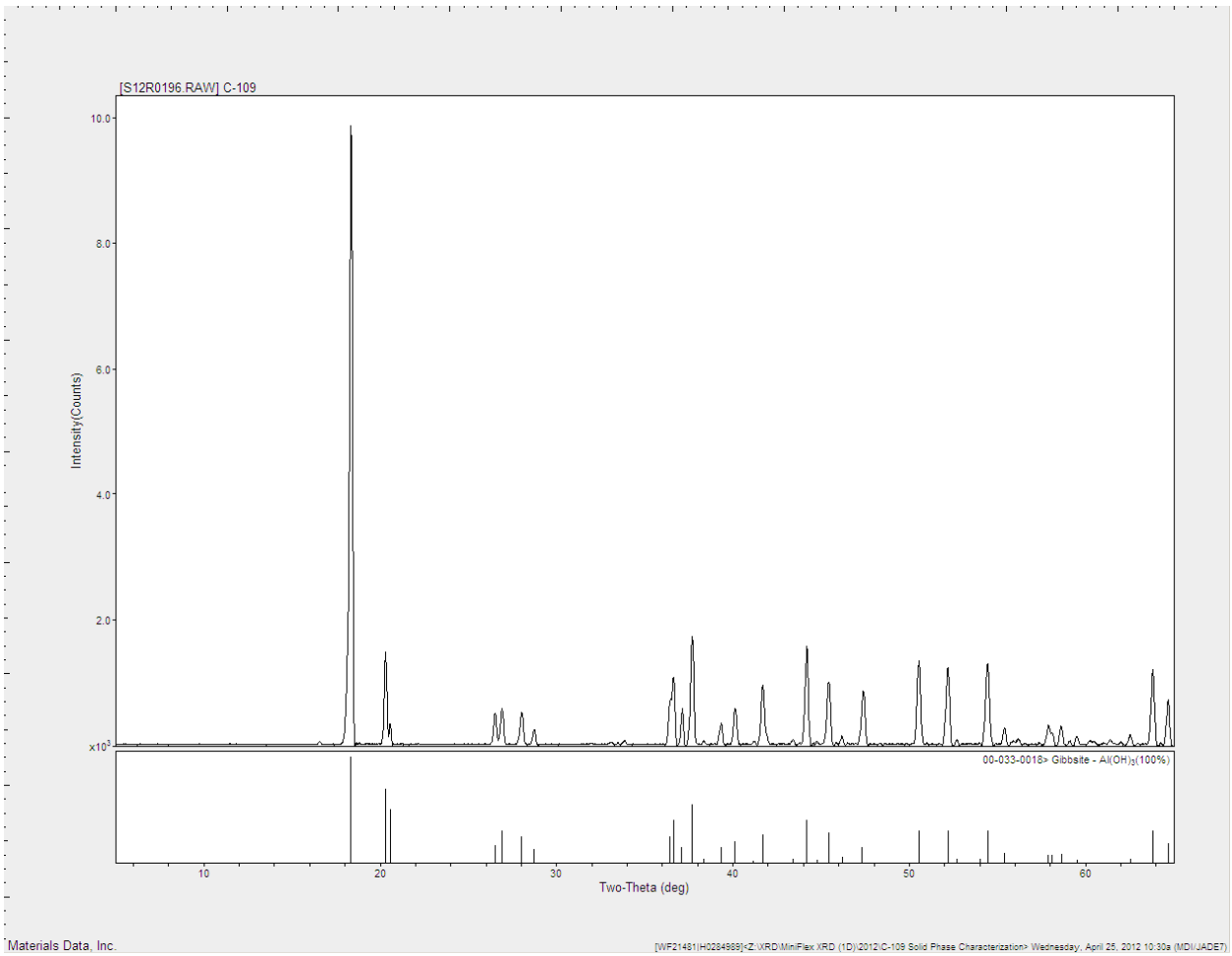
Powder X-ray diffraction was performed in accordance with ATS-LT-507-101, "222-S Laboratory X-Ray Diffractometry (XRD)." Scanning electron microscopy/energy dispersive X-ray spectrometry (SEM/EDX) was performed in accordance with ATS-LT-161-100, "222-S Laboratory Sample Preparation and Operating Procedure for Scanning Electron Microscope."

Large-scale Dissolution Testing: >710-µm Residual Solids (S12R000196)

XRD

Sample S12R000196 contained whitish-tan, dry, fine solids. A portion of the sample was ground slightly, placed in the well of a quartz zero-background slide, compressed with the plane of a glass slide, and then fixed with collodion binder. Once dried, the sample appeared to be homogeneous, tan, fine-grained solids. The major solid phase identified in sample S12R000196 was gibbsite [$(Al(OH)_3$].

Figure D-1. Powder X-ray Diffraction Pattern for >710-µm Residual Solids.

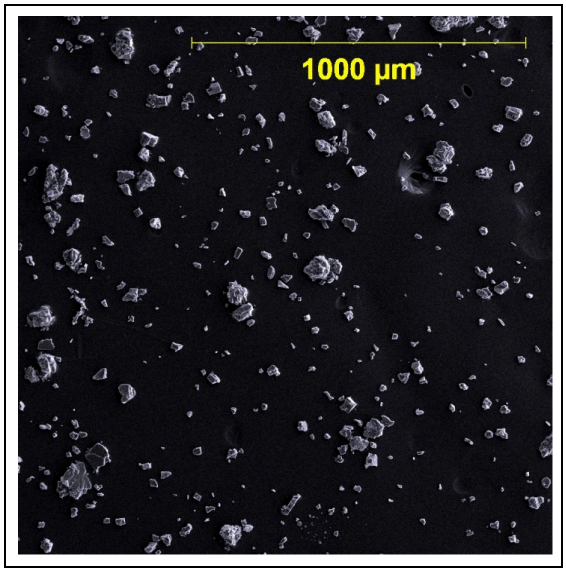


SEM/EDX

NOTE: As described in Section 4.5.4, the >710-µm residual solids from the large-scale dissolution tests were ground in a mortar and pestle prior to sampling for SPC. Size data from SEM images does not apply to the solids remaining at the conclusion of the dissolution tests. Any size-related interpretative comments were provided by I&C personnel who performed the microscopic examination of this sample.

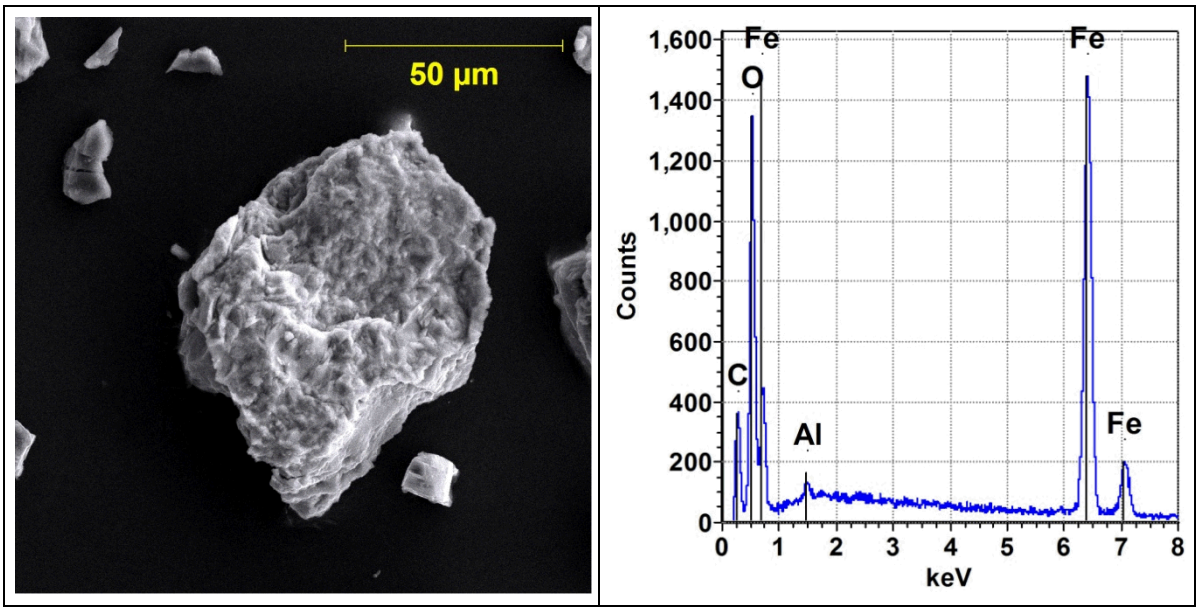
The >710-µm residual solids (Figure D-2) were gibbsite with a maximum size of 100 µm.

Figure D-2. Wide-field View of Specimen of >710-µm Residual Solids.



The 'High-Z' component in the >710-µm residual solids (i.e., solids containing elements with relatively large atomic numbers that appear in high contrast in backscattered electron imaging) is shown in Figure D-3. The High-Z component was dominated by a Fe-rich particle type (sometimes associated with Pb). Possible solid phases represented by these particulates include hematite (Fe_2O_3), ferrihydrite ($Fe_2O_3 \cdot 0.5\ H_2O$), and goethite/lepidocrocite/akaganeite (all FeOOH). Minor amounts of U-rich (without Na) and calcium phosphate particulates were also observed.

Figure D-3. High-Z Material in >710-µm Residual Solids.

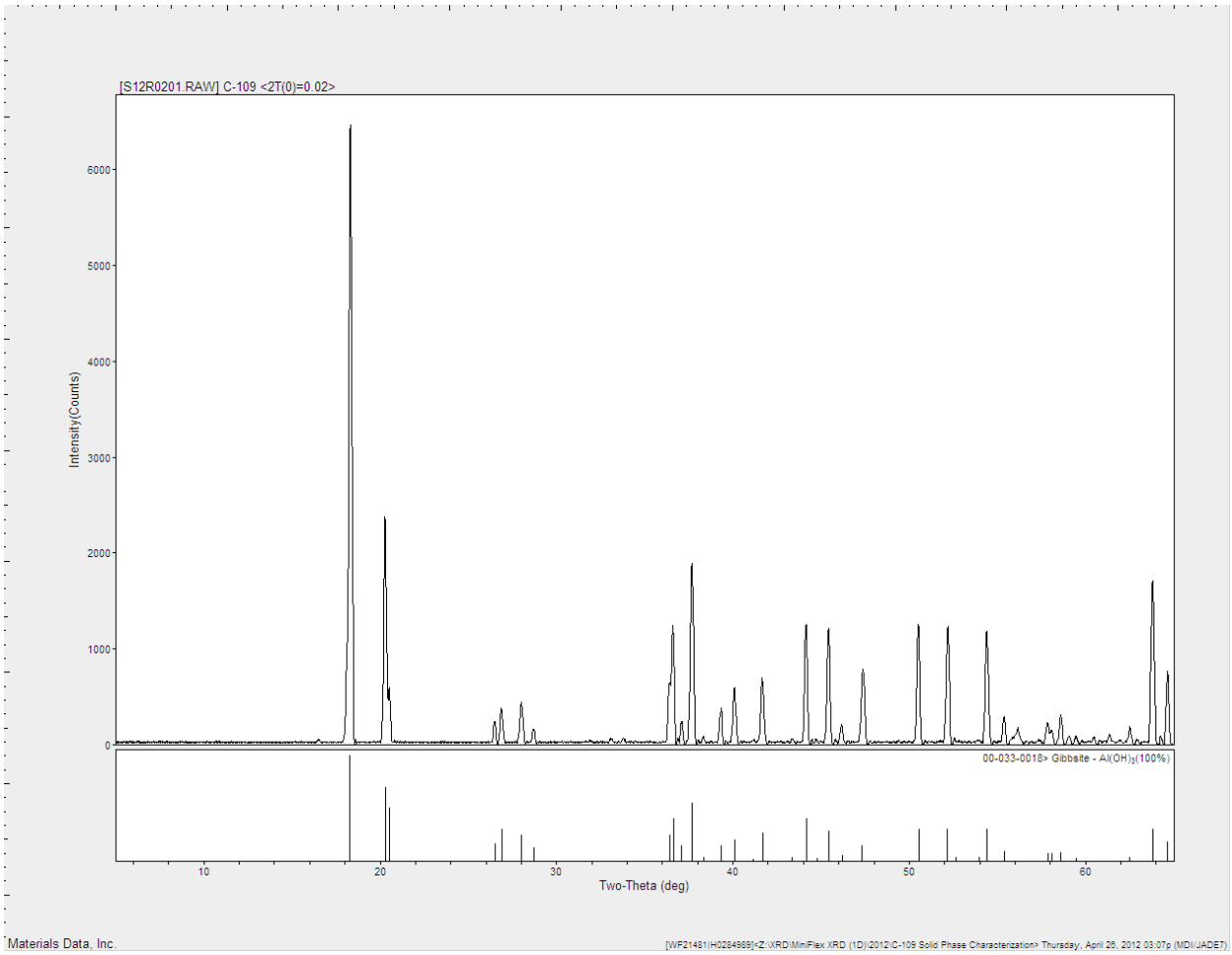


Large-scale Dissolution Testing: <710-µm Residual Solids (S12R000201)

XRD

Sample S12R000201 contained tan, dry, fine solids. A portion of the sample was ground slightly, placed in the well of a quartz zero-background slide, compressed with the plane of a glass slide, and then fixed with collodion binder. Once dried, the sample appeared to be light-brown, fine-grained solids. The major solid phase identified in sample S12R000201 was gibbsite [($Al(OH)_3$].

Figure D-4. Powder X-ray Diffraction Pattern for <710-µm Residual Solids.



SEM/EDX

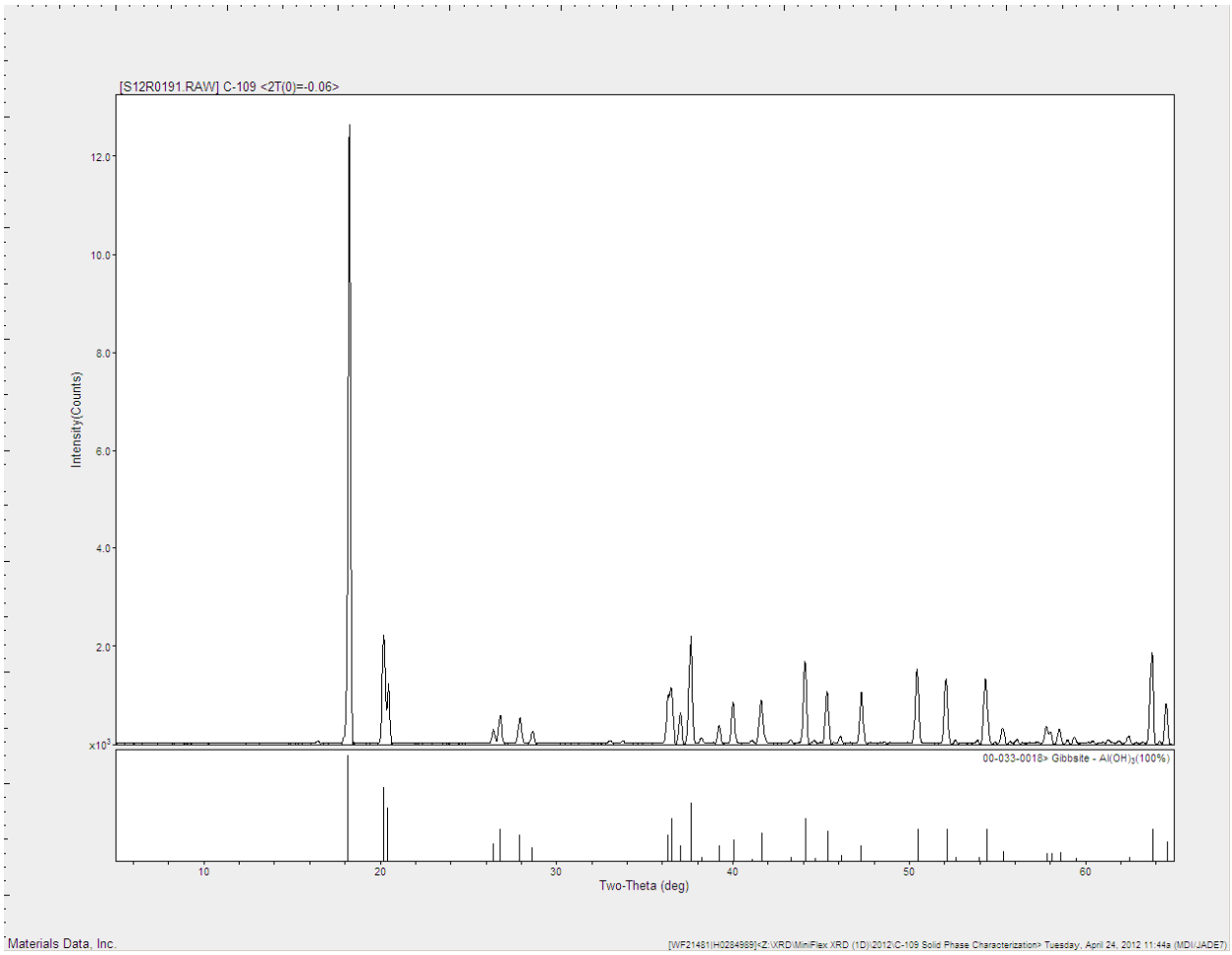
No results of SEM/EDX examination of this sample provided.

Aggregate Dissolution Testing: Residual Solids (S12R000191)

XRD

Sample S12R000191 appeared to contain whitish-tan, dry, fine solids. A portion of the sample was ground slightly, placed in the well of a quartz zero-background slide, compressed with the plane of a glass slide, and then fixed with collodion binder. Once dried, the sample appeared to be homogeneous, tan, fine-grained solids. The major solid phase identified in sample S12R000191 was gibbsite [($Al(OH)_3$].

Figure D-5. Powder X-ray Diffraction Pattern for Aggregate Residual Solids.

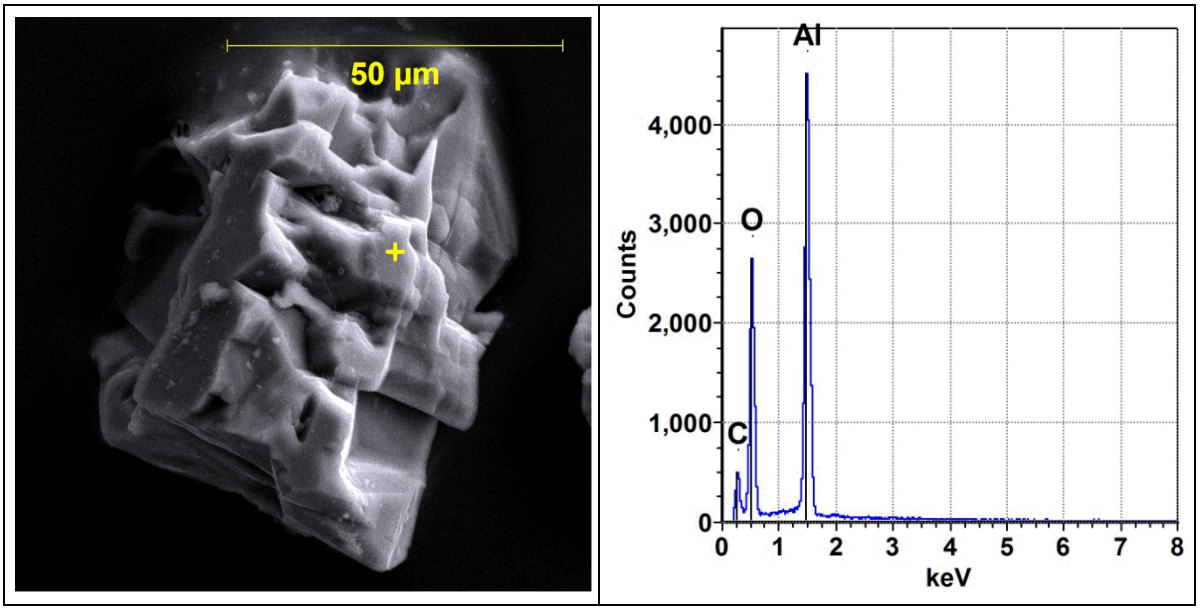


SEM/EDX

NOTE: As described in Section 5.5.3, the residual solids from the aggregate dissolution tests were ground in a mortar and pestle prior to sampling for SPC. Size (and probably texture) data from SEM images does not apply to the solids remaining at the conclusion of the dissolution tests.

Most of the particulate solids in the aggregate testing residual solids were gibbsite. (Minor amounts of other Low-Z phases (e.g. silicates) dispersed in the gibbsite would be difficult to detect.) Some of the gibbsite particles appeared to be well etched (Figure D-6).

Figure D-6. Gibbsite Particle in Aggregate Residual Solids.



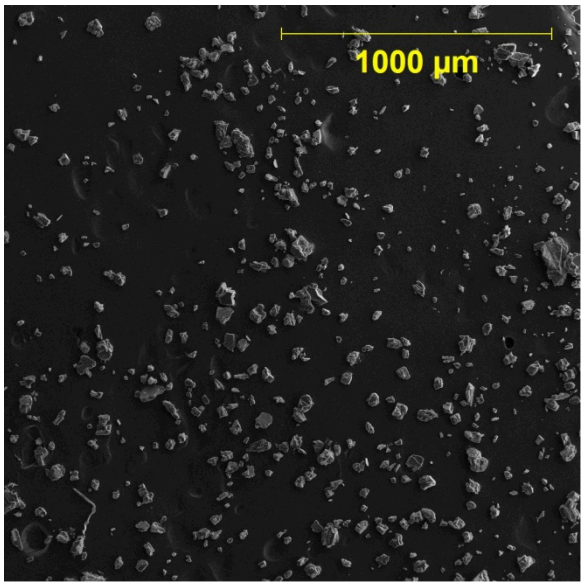
High-Z particulates represented a minor component of the aggregate residual solids. When the particle field in Figure D-7(a) was imaged in back-scattered electron mode, only a single High-Z particle appeared in high contrast in the center of Figure D-7(b).

A survey of the High-Z particulates identified a variety of solids including some U-rich (without Na) solids and calcium phosphate solids. (Both these phases were also identified in the >710-µm large-scale residual solids.) Much of the High-Z material in the aggregate test residues was a fine-grained Al/Fe/Ni mix (Figure D-8). The Al detected in these solids may be from gibbsite near or coated by Fe/Ni-rich material.

Both Fe-rich and Fe/Pb-rich particulates were also observed (as in the >710-µm large-scale residual solids) but in minor amounts relative to the Al/Fe/Ni material.

Figure D-7. High-Z Component of Aggregate Residual Solids.

(a) Secondary electron imaging mode



(b) Back-scattered electron imaging mode

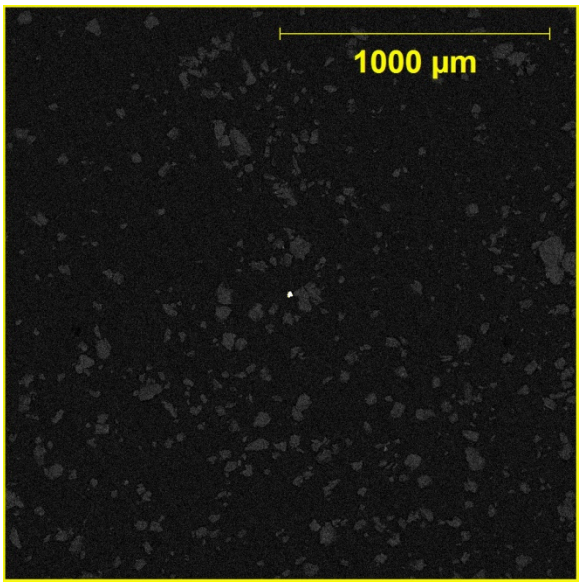
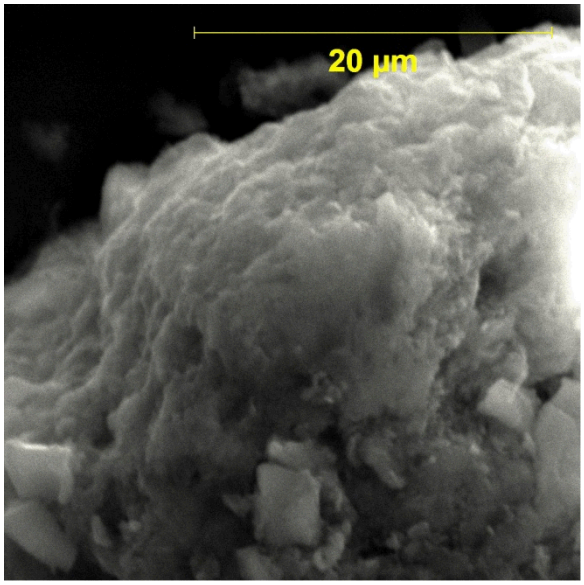
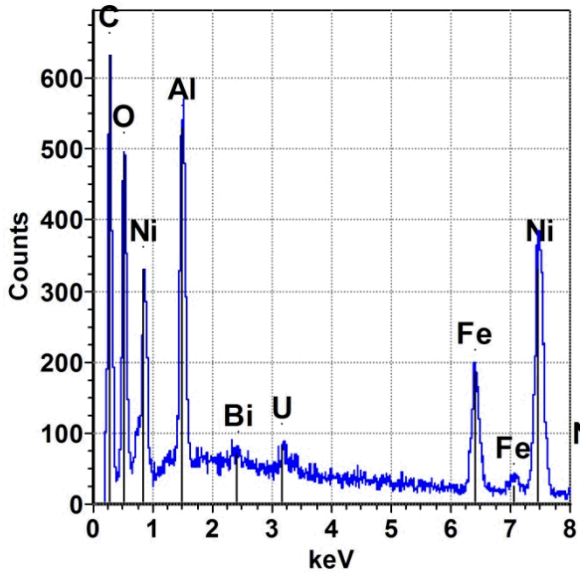


Figure D-8. Al/Fe/Ni Component of Aggregate Residual Solids.





ATTACHMENT E

SETTLING RATE CALCULATIONS

ATTACHMENT E — SETTLING RATE CALCULATIONS

E.1 SETTLING VELOCITIES

The settling velocity of a particle settling under laminar (non-turbulent) flow through a fluid is given by Stokes' Law:

$$d = \sqrt{\frac{18\,\eta\,V_{Stokes}}{(D_{Sld}-D_{Liq})\,g}} \tag{E-1}$$

where

d	=	particle diameter (cm)
η	=	viscosity of the fluid (g/cm·s)
V_{Stokes}	=	Stokes settling velocity (cm/s)
D_{Sld}	=	density of the solid particle (g/cm^3)
D_{Liq}	=	density of the fluid (g/cm^3)
g	=	acceleration due to gravity (980.665 cm/s^2)

The equivalent viscosity of a suspension of particles in a fluid can be estimated using an equation deduced by Einstein:

$$\eta_{Suspension} = \eta(1 + kc) \tag{E-2}$$

where η is the viscosity of the fluid, c is the volume fraction of the solids, and k is a constant which equals 2.5 for rigid, inertialess, spherical particles. This equation holds for suspensions with $c \leq 0.01$.

The assumptions made in the derivation of Stokes' law are:

- the particle is spherical, smooth, and rigid and there is no slip between it and the fluid;
- the particle moves as it would in a fluid of infinite extent;
- the particle reaches terminal settling velocity quickly;
- the settling velocity is slow enough that inertia effects are negligible;
- the fluid is homogeneous compared with the size of the particle.

Stokes' law is not applicable when the gravitational settling velocity is not much greater than Brownian motion of the particles. This is generally the case for particle diameters less than ≈1 µm when D_{Sld} is ≈2.5 g/cm^3 or less.

Stokes' law also fails at settling velocities so great that flow of the fluid around the particles becomes turbulent (non-laminar). In general, the error in settling velocities predicted by Stokes' law become unacceptably large when the Reynolds number $N_R \geq 0.25$.

The terminal velocity ($V_{Terminal}$) of a spherical particle of diameter d settling under turbulent flow through a homogeneous fluid of infinite extent can be calculated as

$$V_{Terminal} = \sqrt{\frac{4}{3}\frac{g(D_{Sld}-D_{Liq})d}{C_D D_{Liq}}} \tag{E-3}$$

where

$$C_D = \frac{24}{N_R} + \frac{3}{\sqrt{N_R}} + 0.34 \quad \text{(E-4)}$$

and

$$N_R = \frac{\varphi(V_{Settling})(d)(D_{Liq})}{\eta} \quad \text{(E-5)}$$

with

$V_{Terminal}$	=	terminal settling velocity (cm/s)
C_D	=	coefficient of drag
N_R	=	Reynolds number
φ	=	shape factor (0.85 for solid, spherical particles)

(*Water Quality* [Tchobanoglous and Schroeder, 1987]).

After an initial calculation of V_{Stokes}, Equations E-5, E-4, and E-3 are solved iteratively until the change in the final value of $V_{Terminal}$ is sufficiently small.

This series of calculations was performed for the C-109 residual solids settling tests. Where $N_R \geq 0.25$ and Stokes' Law did not hold, six iterations of the calculations described above yielded $V_{Terminal}$ values of sufficient accuracy.

In Table E-1, the settling velocities associated with particles with a range of diameters in the initial settling behavior test slurry are calculated. The results of the entire calculation series for residual solids with D_{Sld} = 2.21 g/cm^3 (measured density of the residual solids from the large-scale dissolution tests) in an aqueous slurry containing 1.03 vol% solids at 22 °C are shown. Abbreviated versions are shown for D_{Sld} = 2.42 g/cm^3 (the density of gibbsite) and D_{Sld} = 2.56 g/cm^3 (the measured density of the residual solids from the aggregate dissolution tests).

In Table E-2, the diameters of particles that would settle at selected settling velocities in each of the 12 slurries of the settling rate test are calculated. The selected settling velocities were those defined by the settling time intervals in each of the 12 decant cycles of the test.

Table E-1. Settling Velocities of Residual Solids in Settling Behavior Test Slurry. (2 sheets)

g	980.665	cm/s^2
t	22.0	°C
Solids	1.030	vol%
D_{Slds}	**2.21**	**g/cm^3**
φ	0.85	
D_{H2O}	0.99777	g/cm^3
η_{H2O}	0.009548	g/cm·s
$\eta_{Suspension}$	0.009794	g/cm·s

d µm	V_{Stokes} cm/s	N_R (1)	C_D (1)	$V_{Terminal}$ (1) cm/s	N_R (2)	C_D (2)	$V_{Terminal}$ (2) cm/s
1.0	0.000067	5.84E-07	4.11E+07	0.000062	5.38E-07	4.46E+07	0.000060
2.7	0.00049	1.15E-05	2.09E+06	0.00045	1.06E-05	2.27E+06	0.00044
12.3	0.01020	1.09E-03	2.22E+04	0.0094	1.00E-03	2.41E+04	0.0090
27.5	0.0510	1.21E-02	2.00E+03	0.0467	1.11E-02	2.19E+03	0.0447
61.4	0.254	1.35E-01	1.86E+02	0.229	1.22E-01	2.06E+02	0.218
162	1.78	2.50E+00	1.19E+01	1.48	2.07E+00	1.40E+01	1.36
710	34.0	2.09E+02	6.62E-01	13.0	8.02E+01	9.74E-01	10.8
1,400	132	1.60E+03	4.30E-01	22.7	2.76E+02	6.08E-01	19.1

d µm		N_R (3)	C_D (3)	$V_{Terminal}$ (3) cm/s	N_R (4)	C_D (4)	$V_{Terminal}$ (4) cm/s
1.0		5.17E-07	4.64E+07	0.000058	5.06E-07	4.74E+07	0.000058
2.7		1.02E-05	2.36E+06	0.00043	9.97E-06	2.41E+06	0.00042
12.3		9.59E-04	2.51E+04	0.0088	9.39E-04	2.56E+04	0.0087
27.5		1.06E-02	2.28E+03	0.0437	1.04E-02	2.33E+03	0.0433
61.4		1.16E-01	2.17E+02	0.212	1.13E-01	2.22E+02	0.210
162		1.91E+00	1.51E+01	1.31	1.84E+00	1.56E+01	1.28
710		6.62E+01	1.07E+00	10.3	6.31E+01	1.10E+00	10.1
1,400		2.32E+02	6.40E-01	18.6	2.26E+02	6.46E-01	18.6

d µm		N_R (5)	C_D (5)	$V_{Terminal}$ (5) cm/s	N_R (6)	C_D (6)	$V_{Terminal}$ (6) cm/s
1.0		5.01E-07	4.79E+07	0.000058	4.99E-07	4.81E+07	0.000057
2.7		9.87E-06	2.43E+06	0.00042	9.82E-06	2.45E+06	0.00042
12.3		9.30E-04	2.59E+04	0.0087	9.25E-04	2.60E+04	0.0087
27.5		1.03E-02	2.36E+03	0.0430	1.02E-02	2.37E+03	0.0429
61.4		1.11E-01	2.25E+02	0.208	1.11E-01	2.26E+02	0.208
162		1.81E+00	1.59E+01	1.28	1.79E+00	1.60E+01	1.270
710		6.23E+01	1.11E+00	10.1	6.21E+01	1.11E+00	10.1
1,400		2.25E+02	6.47E-01	18.5	2.25E+02	6.47E-01	18.5

Table E-1. Settling Velocities of Residual Solids in Settling Behavior Test Slurry. (2 sheets)

g	980.665	cm/s^2
t	22.0	°C
Solids	0.940	vol%
D_{Slds}	**2.42**	**g/cm^3**
φ	0.85	
D_{H2O}	0.99777	g/cm^3
η_{H2O}	0.009548	g/cm·s
$\eta_{Suspension}$	0.009772	g/cm·s

d µm	V_{Stokes} cm/s	N_R (1)	C_D (1)	$V_{Terminal}$ (1) cm/s		N_R (6)	C_D (6)	$V_{Terminal}$ (6) cm/s
1.0	0.000079	6.88E-07	3.49E+07	0.000073		5.88E-07	4.08E+07	0.000068
2.5	0.000496	1.08E-05	2.23E+06	0.00046		9.18E-06	2.61E+06	0.00042
11.3	0.01012	9.93E-04	2.43E+04	0.0093		8.45E-04	2.85E+04	0.0086
25.3	0.0508	1.11E-02	2.18E+03	0.0465		9.41E-03	2.58E+03	0.0427
56.6	0.254	1.25E-01	2.01E+02	0.229		1.02E-01	2.44E+02	0.208
149	1.76	2.28E+00	1.28E+01	1.471		1.65E+00	1.72E+01	1.270
710	40.0	2.46E+02	6.29E-01	14.5		6.93E+01	1.05E+00	11.2
1,400	155	1.89E+03	4.22E-01	24.9		2.48E+02	6.27E-01	20.4

g	980.665	cm/s^2
t	22.0	°C
Solids	0.889	vol%
D_{Slds}	**2.56**	**g/cm^3**
φ	0.85	
D_{H2O}	0.99777	g/cm^3
η_{H2O}	0.009548	g/cm·s
$\eta_{Suspension}$	0.009760	g/cm·s

d µm	V_{Stokes} cm/s	N_R (1)	C_D (1)	$V_{Terminal}$ (1) cm/s		N_R (6)	C_D (6)	$V_{Terminal}$ (6) cm/s
1.0	0.000087	7.58E-07	3.17E+07	0.000080		6.47E-07	3.71E+07	0.000074
2.4	0.000502	1.05E-05	2.29E+06	0.00046		8.95E-06	2.68E+06	0.00043
10.8	0.01017	9.55E-04	2.52E+04	0.0094		8.13E-04	2.96E+04	0.0086
24.1	0.0506	1.06E-02	2.29E+03	0.0464		8.96E-03	2.71E+03	0.0427
54.0	0.254	1.19E-01	2.10E+02	0.229		9.80E-02	2.55E+02	0.208
141	1.76	2.17E+00	1.35E+01	1.469		1.57E+00	1.80E+01	1.270
710	44.0	2.71E+02	6.11E-01	15.4		7.39E+01	1.01E+00	12.0
1,400	171	2.08E+03	4.17E-01	26.2		2.62E+02	6.17E-01	21.6

Table E-2. Settling Velocities in Settling Rate Test Slurries. (4 sheets)

Settling rate test slurries: Target $V_{Settling}$ = 0.0040 in./s (0.01016 cm/s)

Average Decants 1-3 0.009978 cm/s	D_{Slds}	2.420	g/cm^3	t	22.0	°C
	Avg Solids	0.927	vol%	D_{H2O}	0.99777	g/cm^3
	Avg $\eta_{Suspension}$	0.009769	g/cm·s	η_{H2O}	0.009548	g/cm·s
	φ	0.85		g	980.665	cm/s^2

D_{Slds} g/cm^3	d µm	V_{Stokes} cm/s	N_R (1)	C_D (1)	$V_{Terminal}$ (1) cm/s
2.420	**11.21**	0.009967	9.70E-04	2.84E+04	0.009172

Decant 1 0.01016 cm/s	D_{Slds} g/cm^3	2.210	2.420	2.560
	Solids vol%	1.030	0.940	0.889
	$\eta_{Suspension}$ g/cm·s	0.009794	0.009772	0.009760

D_{Slds} g/cm^3	d µm	V_{Stokes} cm/s	N_R (1)	C_D (1)	$V_{Terminal}$ (1) cm/s
2.210	**12.27**	0.01015	1.08E-03	2.23E+04	0.009341
2.420	**11.32**	0.01016	9.98E-04	2.41E+04	0.009349
2.560	**10.79**	0.01015	9.52E-04	2.53E+04	0.009342

Decant 2 0.01019 cm/s	D_{Slds} g/cm^3	2.210	2.420	2.560
	Solids vol%	1.012	0.924	0.874
	$\eta_{Suspension}$ g/cm·s	0.009790	0.009769	0.009757

D_{Slds} g/cm^3	d µm	V_{Stokes} cm/s	N_R (1)	C_D (1)	$V_{Terminal}$ (1) cm/s
2.210	**12.29**	0.01019	1.08E-03	2.22E+04	0.009375
2.420	**11.33**	0.01018	1.00E-03	2.41E+04	0.009369
2.560	**10.81**	0.01019	9.58E-04	2.52E+04	0.009380

Decant 3 0.009583 cm/s	D_{Slds} g/cm^3	2.210	2.420	2.560
	Solids vol%	1.004	0.917	0.866
	$\eta_{Suspension}$ g/cm·s	0.009788	0.009767	0.009755

D_{Slds} g/cm^3	d µm	V_{Stokes} cm/s	N_R (1)	C_D (1)	$V_{Terminal}$ (1) cm/s
2.210	**11.92**	0.009587	9.90E-04	2.43E+04	0.008822
2.420	**10.99**	0.009582	9.14E-04	2.63E+04	0.008817
2.560	**10.48**	0.009583	8.73E-04	2.76E+04	0.008818

Table E-2. Settling Velocities in Settling Rate Test Slurries. (4 sheets)

Settling rate test slurries: Target $V_{Settling}$ = 0.02 in./s (0.0508 cm/s)

Average Decants 4-6 0.0507 cm/s	D_{Slds}	2.420	g/cm^3	*t*	22.0	°C
	Avg Solids	0.883	vol%	D_{H2O}	0.99777	g/cm^3
	Avg $\eta_{Suspension}$	0.009759	g/cm·s	η_{H2O}	0.009548	g/cm·s
	φ	0.85		*g*	980.665	cm/s^2

D_{Slds} g/cm^3	d µm	V_{Stokes} cm/s	N_R (1)	C_D (1)	$V_{Terminal}$ (1) cm/s
2.420	**25.26**	0.05066	1.11E-02	2.19E+03	0.04640

Decant 4 0.0507 cm/s	D_{Slds} g/cm^3	2.210	2.420	2.560
	Solids vol%	0.999	0.913	0.863
	$\eta_{Suspension}$ g/cm·s	0.009787	0.009766	0.009754

D_{Slds} g/cm^3	d µm	V_{Stokes} cm/s	N_R (1)	C_D (1)	$V_{Terminal}$ (1) cm/s
2.210	**27.41**	0.05070	1.20E-02	2.02E+03	0.04642
2.420	**25.27**	0.05067	1.11E-02	2.19E+03	0.04640
2.560	**24.10**	0.05068	1.06E-02	2.29E+03	0.04642

Decant 5 0.0509 cm/s	D_{Slds} g/cm^3	2.210	2.420	2.560
	Solids vol%	0.961	0.877	0.829
	$\eta_{Suspension}$ g/cm·s	0.009777	0.009757	0.009746

D_{Slds} g/cm^3	d µm	V_{Stokes} cm/s	N_R (1)	C_D (1)	$V_{Terminal}$ (1) cm/s
2.210	**27.44**	0.05086	1.21E-02	2.01E+03	0.04657
2.420	**25.31**	0.05087	1.12E-02	2.17E+03	0.04659
2.560	**24.14**	0.05089	1.07E-02	2.27E+03	0.04662

Decant 6 0.0505 cm/s	D_{Slds} g/cm^3	2.210	2.420	2.560
	Solids vol%	0.941	0.860	0.813
	$\eta_{Suspension}$ g/cm·s	0.009773	0.009753	0.009742

D_{Slds} g/cm^3	d µm	V_{Stokes} cm/s	N_R (1)	C_D (1)	$V_{Terminal}$ (1) cm/s
2.210	**27.34**	0.05051	1.20E-02	2.03E+03	0.04625
2.420	**25.21**	0.05049	1.11E-02	2.20E+03	0.04624
2.560	**24.04**	0.05049	1.06E-02	2.30E+03	0.04625

Table E-2. Settling Velocities in Settling Rate Test Slurries. (4 sheets)

Settling rate test slurries: Target $V_{Settling}$ = 0.10 in./s (0.254 cm/s)

Average Decants 7-9 0.245 cm/s	D_{Slds}	2.420	g/cm^3	t	22.0	°C
	Avg Solids	0.572	vol%	D_{H2O}	0.99777	g/cm^3
	Avg $\eta_{Suspension}$	0.009684	g/cm·s	η_{H2O}	0.009548	g/cm·s
	φ	0.85		g	980.665	cm/s^2

D_{Slds} g/cm^3	d µm	V_{Stokes} cm/s	N_R (1)	C_D (1)	$V_{Terminal}$ (1) cm/s		N_R (6)	C_D (6)	$V_{Terminal}$ (6) cm/s
2.420	**55.33**	0.2450	1.19E-01	2.11E+02	0.2210		9.75E-02	2.56E+02	0.2007

Decant 7 0.238 cm/s	D_{Slds} g/cm^3	2.210	2.420	2.560
	Solids vol%	0.932	0.851	0.804
	$\eta_{Suspension}$ g/cm·s	0.009770	0.009751	0.009740

D_{Slds} g/cm^3	d µm	V_{Stokes} cm/s	N_R (1)	C_D (1)	$V_{Terminal}$ (1) cm/s		N_R (6)	C_D (6)	$V_{Terminal}$ (6) cm/s
2.210	**59.35**	0.2381	1.23E-01	2.05E+02	0.2147		1.01E-01	2.48E+02	0.1950
2.420	**54.74**	0.2381	1.13E-01	2.21E+02	0.2149		9.32E-02	2.68E+02	0.1953
2.560	**52.20**	0.2381	1.08E-01	2.31E+02	0.2150		8.91E-02	2.80E+02	0.1954

Decant 8 0.246 cm/s	D_{Slds} g/cm^3	2.210	2.420	2.560
	Solids vol%	0.565	0.516	0.488
	$\eta_{Suspension}$ g/cm·s	0.009683	0.009671	0.009664

D_{Slds} g/cm^3	d µm	V_{Stokes} cm/s	N_R (1)	C_D (1)	$V_{Terminal}$ (1) cm/s		N_R (6)	C_D (6)	$V_{Terminal}$ (6) cm/s
2.210	**59.99**	0.2455	1.29E-01	1.95E+02	0.2212		1.06E-01	2.36E+02	0.2008
2.420	**55.35**	0.2455	1.19E-01	2.10E+02	0.2214		9.79E-02	2.55E+02	0.2011
2.560	**52.80**	0.2455	1.14E-01	2.20E+02	0.2216		9.36E-02	2.67E+02	0.2013

Decant 9 0.251 cm/s	D_{Slds} g/cm^3	2.210	2.420	2.560
	Solids vol%	0.381	0.348	0.329
	$\eta_{Suspension}$ g/cm·s	0.009639	0.009631	0.009627

D_{Slds} g/cm^3	d µm	V_{Stokes} cm/s	N_R (1)	C_D (1)	$V_{Terminal}$ (1) cm/s		N_R (6)	C_D (6)	$V_{Terminal}$ (6) cm/s
2.210	**60.56**	0.2513	1.34E-01	1.88E+02	0.2263		1.10E-01	2.28E+02	0.2054
2.420	**55.89**	0.2513	1.24E-01	2.03E+02	0.2266		1.02E-01	2.46E+02	0.2058
2.560	**53.31**	0.2513	1.18E-01	2.12E+02	0.2266		9.70E-02	2.57E+02	0.2059

Table E-2. Settling Velocities in Settling Rate Test Slurries. (4 sheets)

Settling rate test slurries: Target $V_{Settling}$ = 0.50 in./s (1.27 cm/s)

Average Decants 10-12 1.086 cm/s	D_{Slds}	2.420	g/cm^3	t	22.0	°C
	Avg Solids	0.156	vol%	D_{H2O}	0.99777	g/cm^3
	Avg $\eta_{Suspension}$	0.009585	g/cm·s	η_{H2O}	0.009548	g/cm·s
	φ	0.85		g	980.665	cm/s^2

D_{Slds} g/cm^3	d µm	V_{Stokes} cm/s	N_R (1)	C_D (1)	$V_{Terminal}$ (1) cm/s		N_R (6)	C_D (6)	$V_{Terminal}$ (6) cm/s
2.420	**135.21**	1.478	1.77E+00	1.62E+01	1.248		1.30E+00	2.14E+01	1.0856

Decant 10 0.954 cm/s	D_{Slds} g/cm^3	2.210	2.420	2.560
	Solids vol%	0.289	0.264	0.250
	$\eta_{Suspension}$ g/cm·s	0.009617	0.009611	0.009608

D_{Slds} g/cm^3	d µm	V_{Stokes} cm/s	N_R (1)	C_D (1)	$V_{Terminal}$ (1) cm/s		N_R (6)	C_D (6)	$V_{Terminal}$ (6) cm/s
2.210	**136.87**	1.2865	1.55E+00	1.82E+01	1.0929		1.15E+00	2.39E+01	0.9536
2.420	**125.95**	1.2789	1.42E+00	1.97E+01	1.0905		1.06E+00	2.58E+01	0.9535
2.560	**119.96**	1.2748	1.35E+00	2.07E+01	1.0892		1.01E+00	2.70E+01	0.9536

Decant 11 1.062 cm/s	D_{Slds} g/cm^3	2.210	2.420	2.560
	Solids vol%	0.147	0.134	0.126
	$\eta_{Suspension}$ g/cm·s	0.009583	0.009580	0.009578

D_{Slds} g/cm^3	d µm	V_{Stokes} cm/s	N_R (1)	C_D (1)	$V_{Terminal}$ (1) cm/s		N_R (6)	C_D (6)	$V_{Terminal}$ (6) cm/s
2.210	**145.08**	1.4506	1.86E+00	1.54E+01	1.2224		1.37E+00	2.05E+01	1.0615
2.420	**133.49**	1.4413	1.70E+00	1.67E+01	1.2195		1.26E+00	2.21E+01	1.0615
2.560	**127.13**	1.4362	1.62E+00	1.75E+01	1.2180		1.20E+00	2.31E+01	1.0616

Decant 12 1.243 cm/s	D_{Slds} g/cm^3	2.210	2.420	2.560
	Solids vol%	0.075	0.069	0.065
	$\eta_{Suspension}$ g/cm·s	0.009566	0.009564	0.009563

D_{Slds} g/cm^3	d µm	V_{Stokes} cm/s	N_R (1)	C_D (1)	$V_{Terminal}$ (1) cm/s		N_R (6)	C_D (6)	$V_{Terminal}$ (6) cm/s
2.210	**158.47**	1.7338	2.44E+00	1.21E+01	1.4415		1.75E+00	1.63E+01	1.2425
2.420	**145.75**	1.7211	2.22E+00	1.31E+01	1.4378		1.61E+00	1.76E+01	1.2425
2.560	**138.77**	1.7139	2.11E+00	1.38E+01	1.4357		1.53E+00	1.84E+01	1.2425

E.2 Fractional Decantation

The settling rate tests were based on the method of classification by decantation or fractional decantation. In this method a homogeneous suspension is allowed to settle for a predetermined time. The supernatant liquid and suspended solids are then decanted and replaced by fresh dispersing liquid and the suspension re-agitated. Ideally, the process is repeated until the supernatant liquid is clear. The process may then be repeated at progressively shorter settling times so that the particulates decanted and removed become progressively coarser and/or settle at progressively faster rates (*Particle Size Measurement: Volume 1−Powder Sampling and Particle Size Measurement* [Allen, 1997]).

As described in Section 7.0, four sets of three decant cycles were performed. The relative efficiency of separation of particulates settling at different settling rates using three decant cycles can be evaluated as follows.

Consider a column containing a depth h_1 of a homogeneous suspension that is decanted to a depth h_2 after time t. All particulates that settle faster than V = h_2/t will remain in the column. However, particulates that settle slower than V = h_2/t but are located below the surface of the test suspension at t = 0 will also remain in the column.

The settling velocity of the slower settling particles can be expressed as xV = h/t where $x < 1$. Solving for x using these two equations:

$$x = \frac{h}{h_2} \tag{E-6}$$

The fraction of particulates settling at rate xV removed in the first decanting is:

$$F_D = \frac{h_2 - h}{h_1} = \frac{h_2}{h_1}(1 - x) \tag{E-7}$$

The fraction of particulates settling at rate xV remaining in the column after the first decant will be:

$$F_{R1} = 1 - F_D = 1 - \frac{h_2}{h_1}(1 - x) \tag{E-8}$$

If the suspension is made up to its original depth, redispersed, and a second decanting performed after time t, the fraction of particulates settling at rate xV in the initial sample that will be removed is:

$$(F_D)(F_{R1}) = \left[\frac{h_2}{h_1}(1 - x)\right]\left[1 - \frac{h_2}{h_1}(1 - x)\right] \tag{E-9}$$

or,

$$(F_D)(F_{R1}) = [1 - F_{R1}][F_{R1}] \tag{E-10}$$

The cumulative fraction of particulates settling at rate xV removed after two decants will be:

$$F_{D2} = F_D + [(F_D)(F_{R1})] = [(1 - F_{R1})] + [F_{R1}(1 - F_{R1})] \tag{E-11}$$

The total fraction of particulates settling at rate xV remaining in the column after two decantings will be:

$$F_{R2} = 1 - F_{D2} = F_{R1}^2 \tag{E-12}$$

In general, after n decantings, the total fraction of particulates settling at rate xV remaining in the column will be:

$$F_{Rn} = F_{R1}^n = \left[1 - \frac{h_2}{h_1}(1 - x)\right]^n \tag{E-13}$$

and the cumulative fraction of particulates settling at rate xV removed from the column will be,

$$F_{Dn} = 1 - F_{Rn} = [(1 - F_{R1})] + [F_{R1}(1 - F_{R1})] + \cdots + [F_{R1}^{n-1}(1 - F_{R1})] \tag{E-14}$$
$$= 1 - \left[1 - \frac{h_2}{h_1}(1 - x)\right]^n$$

In the apparatus used in the settling rate tests for the C-109 residual solids, h_1 = 44.5 cm and the target decant depth h_2 = 39.4 cm. Inserting these values and solving equations E-13 and E-14 for three decant cycles yields:

	After 1 Decant	After 2 Decants	After 3 Decants	
x	$\%_{Decanted}$	$\%_{Decanted}$	$\%_{Decanted}$	$\%_{Remaining}$
0	88.54	98.69	99.85	0.15
0.1	79.69	95.87	99.16	0.84
0.2	70.83	91.49	97.52	2.48
0.3	61.98	85.54	94.50	5.50
0.4	53.12	78.03	89.70	10.30
0.5	44.27	68.94	82.69	17.31
0.6	35.42	58.29	73.06	26.94
0.7	26.56	46.07	60.39	39.61
0.8	17.71	32.28	44.27	55.73
0.9	8.85	16.92	24.28	75.72
0.95	4.43	8.66	12.70	87.30
0.99	0.89	1.76	2.63	97.37

The x = 0 values show that the maximum separation achievable with three decantings is not 100%. This is because a portion of the particulates in the homogenized suspensions will always be below depth h_2 (i.e., in the bottom 5.1 cm of the column) at t = 0.

The remaining values show that as xV approaches the target separation settling rate (where x = 1) the efficiency of the separation rapidly decreases. For example, only 24.3% of particulates settling at 90% of the target rate will be removed from the column after three decantings while 75.7% will be left behind in the column. The absolute efficiency of the separation at a target settling rate V will depend on the distribution of particulates over the range of settling rates xV and cannot be determined without additional information. A nearly complete separation can be achieved with a relatively small number of decantings only when most of the particulates settle at rates xV << V.

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