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Results of Physicochemical Characterization and Caustic Dissolution Tests on Tank 241-C-108 Heel Solids

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EXECUTIVE SUMMARY

On April 27, 2007, single-shell tank 241-C-108 (C-108) was retrieved to the limit of technology using modified sluicing. Approximately 6,800 gal (910 ft^3) of waste, primarily heel solids, remained in the tank after completion of the retrieval operations. In mid-summer of 2009, eight samples of the heel solids were retrieved from the tank using the Off-Riser Sampling System. This test program was developed to characterize the heel solids and to provide data needed to evaluate retrieval technologies that could be used to remove more of the residual solid material from C-108.

A 488.28-g composite of C-108 heel solids was prepared for this study. Only the first six samples of heel solids retrieved from C-108 were used to prepare the composite. Testing on the final two samples was limited to solid phase characterization.

Visual inspection of the heel solids during preparation of the test composite revealed the presence of a significant quantity of fine to medium pebble-sized solids and a few very coarse pebble-sized pieces. The larger solids included pieces that were very darkly colored, medium-to-dark green, white to off-white, and nearly transparent. The space between the larger solids was filled with coarse to very fine sand-sized solids that were, in general, lightly colored. A very small volume of free liquid was recovered from one of the six tank samples (Sample 4). The remaining samples were slightly to very slightly damp.

Solid phase characterization of a subsample of the test composite identified $Na_7F(PO_4)_2 \cdot 19H_2O$ (natrophosphate) and $Al(OH)_3$ (gibbsite) as major phases. Some large crystals of natrophosphate were observed. Two minor phases were identified: a Ni-Al-(U)-phosphate and large flakes of steel scale. Trace phases included an Fe-Al-Pb-phosphate and $NaSrPO_4 \cdot 9H_2O$ (nastrophite). Solid phase characterization of Heel Samples 7 and 8 identified natrophosphate, gibbsite, and a sodium-bicarbonate as major phases (all about equally distributed). The minor and trace phases were the same as in the composite. This shows that the treatment of the composite prior to the characterization removed most of the water-soluble bicarbonate but only minor amounts of the natrophosphate.

A bulk density (D_{Bulk}) measurement was made on the entire 488.28 g of test composite. The measured D_{Bulk} of the C-108 heel composite was 1.884 g/cm^3. The amount of interstitial liquid that was in the test composite is unknown; however, no free liquid was present. At the completion of the D_{Bulk} measurement, the test composite had been through a *de facto* water wash of 0.45 parts water to 1 part composite (w/w) and the calculated weight of the remaining heel solids was 404.78 g. The calculated dry density of the remaining solids was 1.933 g/cm^3.

After completion of the density measurements, the test composite was separated into >1/4-in. and <1/4-in. size fractions by wet sieving. The remaining solids that measured >1/4 in. were 18.6 wt% of the initial composite; the remaining solids that were <1/4 in. were 63.1 wt% of the initial composite. At this point, the *de facto* water wash of the test composite was 0.51 parts water to 1 part composite. Composite weight loss due to the water wash (including removal of interstitial liquid and solids dissolution) was 18.3 wt%.

A second wet sieve separation was performed on a subsample of the <1/4-in. heel composite solids. The recovered solids that were <1/4 in. and >600 µm in size were ≈19.9 wt% of the initial composite weight. The *de facto* water wash of the composite solids was approximately 7.2 parts water to 1 part composite. The weight of the <1/4-in. composite solids that may have dissolved during this operation is unknown.

The particle size distribution (PSD) within the <600-µm heel solids recovered after the second sieve separation was measured using a light-scattering-based technique. The calculated PSD within the <600-µm composite solids that survived the measurement is:

Particle diameters (µm)	Volume %
<38	7.6
38 to 75	4.9
75 to 150	19.6
150 to 300	42.8
300 to 600	25.1

By the time the PSD measurements were initiated, the *de facto* water wash of the initial composite was in excess of 160 parts water to 1 part composite. The portion of the fine-grained composite solids that remained and how their original PSD may have been altered is unknown.

Settling tests were performed on four additional subsamples of the <1/4-in. fraction of the heel composite. The tests were performed on aqueous slurries containing 1.36 wt% solids and 6.87 wt% solids. In all four settling behavior tests, 35-50% of the solids settled from suspension very rapidly (a few seconds to a few minutes). The slurries then exhibited the settling behavior of relatively dilute slurries of small and/or low density solids with no, or very weak, interparticle interactions. The supernatant liquid in each test clarified completely in 24-40 hours. The settling rate tests were designed to estimate the portions of the C-108 heel solids that would settle from aqueous slurries of two concentrations at rates of 1.27 cm/s (0.5 in./s) and 0.25 cm/s (0.1 in./s). When the >1/4-in. solids previously removed from the test samples are included, solids representing the following percentages of the initial composite weight settled from suspension in water at rates greater than or equal to the test rates.

	≤1.36 wt% solids	≤6.87 wt% solids
1.23 cm/s	41 wt%	48 wt%
0.253 cm/s	43 wt%	50 wt%

By the conclusion of the settling tests, the *de facto* water wash of the initial composite was in excess of 68 parts water to 1 part composite for the 1.36 wt% slurries and in excess of 12.5 parts water to 1 part composite for the 6.87 wt% slurries. Again, the quantity of the original composite solids that was dissolved and how, as a result, the measured test behavior may differ from that of the original heel solids is unknown.

Caustic dissolution tests were performed on overall 301 g (170 mL) of heel material under ambient hot cell temperatures, on average 29 °C. Of these, 217 g were <1/4 in. in diameter, and the rest were treated separately based on their main constituents (green – Ni-bearing phosphates, clear – natrophosphate, black – steel scale, light brown – gibbsite). The theoretical amount of caustic needed to dissolve the 40 wt% gibbsite in the overall sample was 242 mL (3:1 molar ratio caustic to gibbsite). During the actual tests, e.g., due to small sample sizes of parts of the coarse fraction, 310 mL of caustic was added. In order to remove the water-soluble material after the caustic addition, 3124 mL of water was added in 6 steps. The water-soluble material contained about equal amounts of sodium aluminate and natrophosphate. The residual solids were 17 mL of very fine-grained material. The Ni-Al-P-phase released the phosphate during dissolution leaving a Ni-Al-phase. The only phase of concern in the dissolution residue is nastrophite. The chemical data show a 16-fold increase in strontium in the dissolution residue over the heel composite; the activity of this phase increased from 0.2 to 5.4 mCi/g.

Adjusting the amounts used in the caustic dissolution test to the size of heel in C-108 tranlates to:

6,800 gal of heel in C-108 => Addition of 9,520 gal 19.4 M caustic => Leached by 89,162 gal water => 680 gal of residual (10 vol.%).

The reverse approach of adding the appropriate amount of water to dissolve the natrophosphate first and then adding the caustic for the gibbsite dissolution was not tested in this study. However, based on these results, starting with a water leach should remove most of the natrophosphate, making the remaining gibbsite more accessible to caustic added. Additionally, the amount of caustic could be reduced to 40 wt% of the amount calculated above, since only the gibbsite is successfully dissolved by the caustic addition.

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Abbreviations and Acronyms

222-S	WRPS 222-S Laboratory
AFA	automated feature analysis
APD	WRPS Analytical Process Development group at 222-S Laboratory
ATL	Advanced Technologies and Laboratories International, Inc. (222-S Laboratory analytical contractor)
BDL	bulk density liquid
BSE	backscattered electron
C-108	single-shell waste tank 241-C-108
CSL	coarse (1/4-inch) sieving liquid
d	particle diameter
d##	particle diameter at a specified (##) %-undersize cut point
D	density
D_{Bulk}	bulk density
D_{BDL}	density of bulk density liquid
D_{CSL}	density of coarse-sieving liquid
$D_{Dry\ Slds}$	dry solids density
D_{Liq}	density of liquid
DSC	differential scanning calorimetry
EDS	energy dispersive spectrometry
ISL	interstitial liquid
PLM	polarized light microscopy
PSD	particle size distribution
RRI	relative refractive index
R-Slds	recovered solids (i.e., fast settling solids recovered in settling rate tests)
SAP	sampling and analysis plan
SEM-AFA	scanning electron microscopy-automated feature analysis
SEM/EDX	scanning electron microscopy/energy dispersive X-ray spectrometry
SPC	solid phase characterization
S-Slds	settled solids
SpG	specific gravity
vol	volume
wt	weight
f_{H_2O}	weight fraction of water
XRD	X-ray diffraction or diffractometry

Units

cm	centimeter
cm/hr	centimeters per hour
cm/min	centimeters per minute
cm/s	centimeter per second
°C	degrees Celsius
ft^3	cubic feet
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
in.	inch(es)
µCi/g	microcuries per gram
µCi/mL	microcuries per milliliter
µg/g	micrograms per gram
µg/mL	micrograms per milliliter
µg	microgram
µm	micrometer
mR/hr	milliroentgen per hour
mL	milliliter
mm	millimeter
min	minute(s)
M	molar
nm	nanometer
vol%	volume percent
wt%	weight percent

1.0 INTRODUCTION

On April 27, 2007, single-shell tank 241-C-108 (C-108) was retrieved to the limit of technology using modified sluicing (CH2M-0603302.4, "Contract Number DE-AC27-99RL14047 – Completion of Performance Based Incentive 3, Revision 2, Fee Bearing Milestone PBI-3.2.a.05, C-108 Completion of Retrieval Operations – Request for Incremental Fee Approval"). An estimated 6,800 gallons (910 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-108 was issued in July 2009 (RPP-PLAN-40585, *Sampling and Analysis Plan for Waste Solids in Tank 241-C-108*). The 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-108. At a minimum, the test program was required to produce the following information:

- The bulk density of the C-108 residual waste
- The density of the C-108 heel solids
- The mineralogical properties (i.e., the solid phase composition) of the heel solids
- The particle size distribution in the heel solids
- The mode and strength of grain adhesion in solid aggregates
- The settling rate and behavior of the heel solids in water and 3 M caustic
- The amount of heel solids dissolved (or amount of gibbsite converted to water-soluble sodium aluminate) in 19 M caustic at ≈25 °C using a 3:1 molar ratio of hydroxide (OH) to aluminum (Al)
- The composition of any solids remaining at the end of caustic dissolution test(s)

A laboratory test plan presenting a strategy to acquire the required information was issued in May 2009 (WRPS-0900639, "Test Plan for Characterization and Retrieval Testing of Tank 241-C-108 Heel Solids"). The test plan was prepared by members of the Analytical Process Development group (APD) at the 222-S Laboratory (222-S) and was reviewed and approved by the client(s). An addendum to the test plan, issued in September 2009 with the same level of review and concurrence (WRPS-0901437, "Addendum to Test Plan for Characterization of Tank 241-C-108 Heel Solids"), addressed revised sample handling and composite preparation instructions in the SAP (RPP-PLAN-40585). Specific details of the planned execution of the test plan were further developed in 222-S test procedures issued in June 2009 (WRPS-0900764, "Test Procedures for Characterization and Retrieval Testing of 241-C-108 Heel Solids").

An informal preliminary report, presenting results from the caustic dissolution tests on the C-108 heel solids, was issued in November 2009 (e-mail from H. J. Huber to W. B. Barton and D. M. Nguyen, "Preliminary Report on the Solid Phase Characterization and Caustic Dissolution Test Results of Heel Material from Tank 241-C-108," [Huber, H. J., 2009-11-05]). This is the final report of all the characterization and test measurements on the C-108 heel solids.

A workflow diagram depicting the work completed is presented in Figure 1-1.

Figure 1-1. Workflow Diagram (2 Sheets)

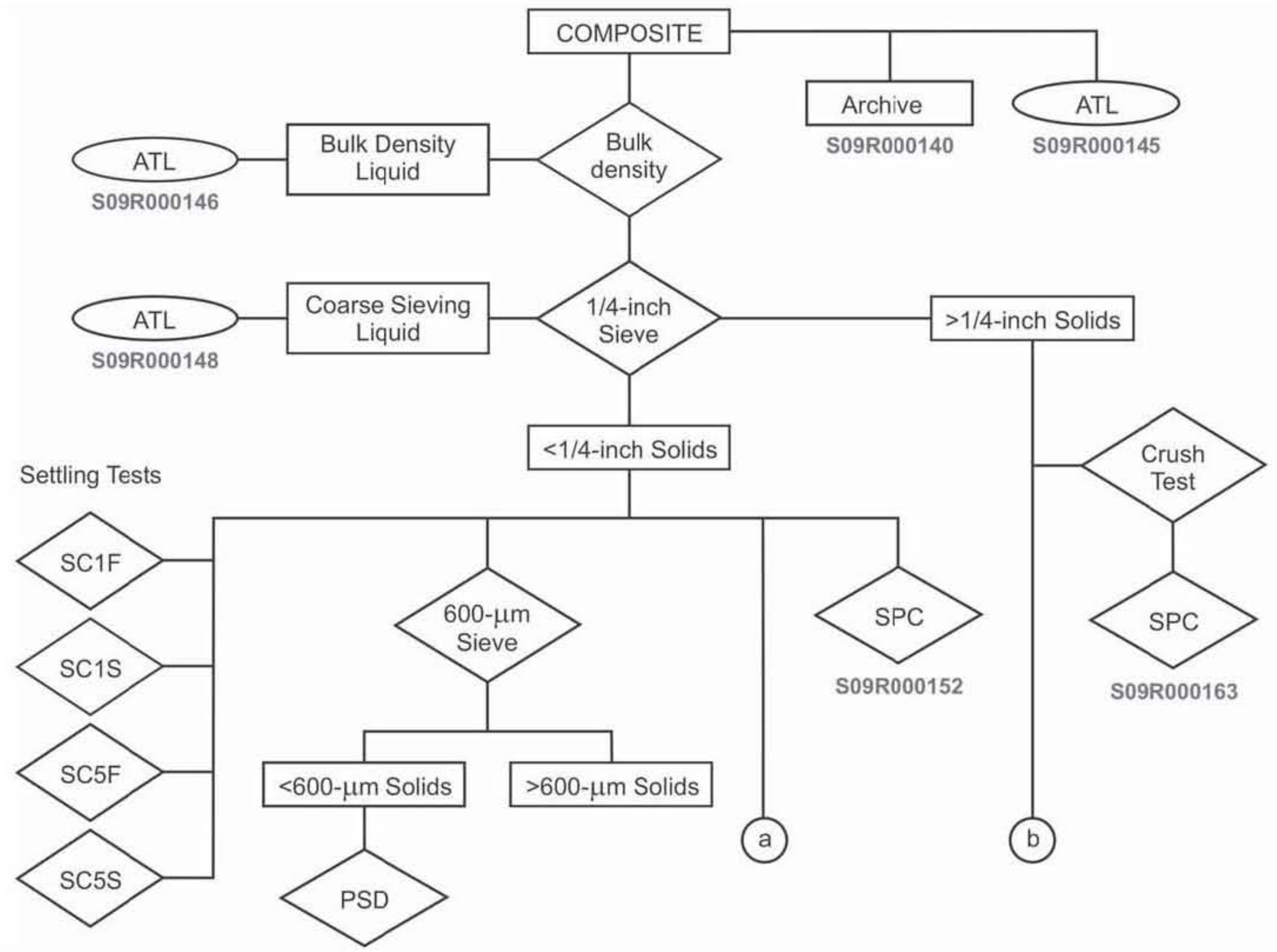
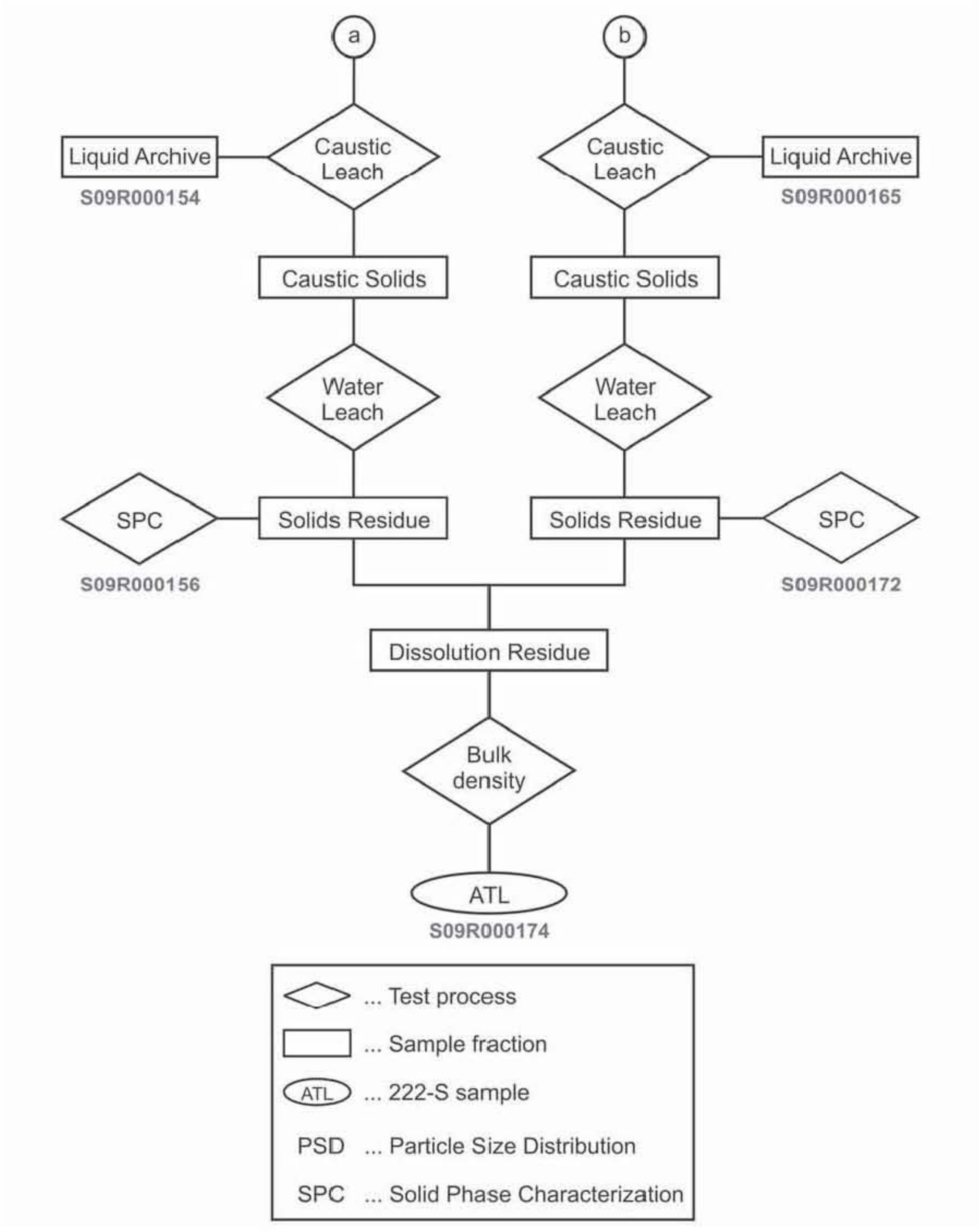


Figure 1-1. Workflow Diagram (2 sheets)



Questions concerning solid phase characterization, crush testing, and caustic dissolution testing should be directed to H. J. Huber (373-1828). Questions concerning all other measurements and test results reported herein should be directed to W. S. Callaway (373-4995).

2.0 TEST SAMPLES

2.1 TANK SAMPLES

Most of the residual waste solids in C-108 were in two large piles on the east and west sides of the tank. The solids in these two piles appeared to be "white to light gray in color and [to] vary in size from fine particles to relatively large chunks" (RPP-PLAN-40585). Three samples of heel solids were retrieved from each of these piles. Samples 1, 2, and 3 were taken from the pile on the west side of the tank; Samples 4, 5, and 6 were taken from the pile on the east side. Because the bulk of the heel solids were not located below C-108 access risers, the Off-Riser Sampling System was used to collect the samples.

A smaller quantity of residual heel material was present as a thin, hard layer of solids in the middle of C-108. Extreme difficulty was encountered in retrieving samples of these heel solids. Only two of the planned three samples were taken. Sample 7 consisted of a small amount of solids from the south edge of the central layer and some solids from the southern edge of the pile of solids on the east side of the tank. Sample 8 contained a small amount of solids that was scraped from a location near the middle of the central hard layer. The sample scoop on the sampler was damaged during the collection of Sample 8 and retrieval of samples of heel solids from C-108 was declared complete.

Over-the-top photographs were taken of the contents of C-108 Samples 1-7 during preparation of composites on July 21, 2009. These photographs are included in the report documenting component closure analytical results prepared by the 222-S analytical contractor, Advanced Technologies and Laboratories International, Inc. (ATL) (RPP-RPT-43234, *Final Report for Tank 241-C-108 Solid Samples Taken After Modified Sluicing*).

2.2 TEST COMPOSITE

The majority of the measurements and analyses performed during completion of this test program were based on the characterization of a composite of the C-108 heel samples. As required by the SAP (RPP-PLAN-40585) and described in RPP-RPT-43234, this composite was prepared in two steps. First, three composites were prepared from the six samples of heel solids taken from the large piles on the east and west sides of C-108. The composition of these three primary composites and qualitative notes on the appearance of the tank samples are presented in Table 2-1.

After the three primary composites were prepared, the contents of each composite jar were mixed by stirring with a large spatula. With the exception of Sample 6 (see Table 2-1), an effort was made not to break up any large pieces of solids during subsampling and mixing of the primary composites.

Table 2-1. Composition of Primary C-108 Heel Sample Composites

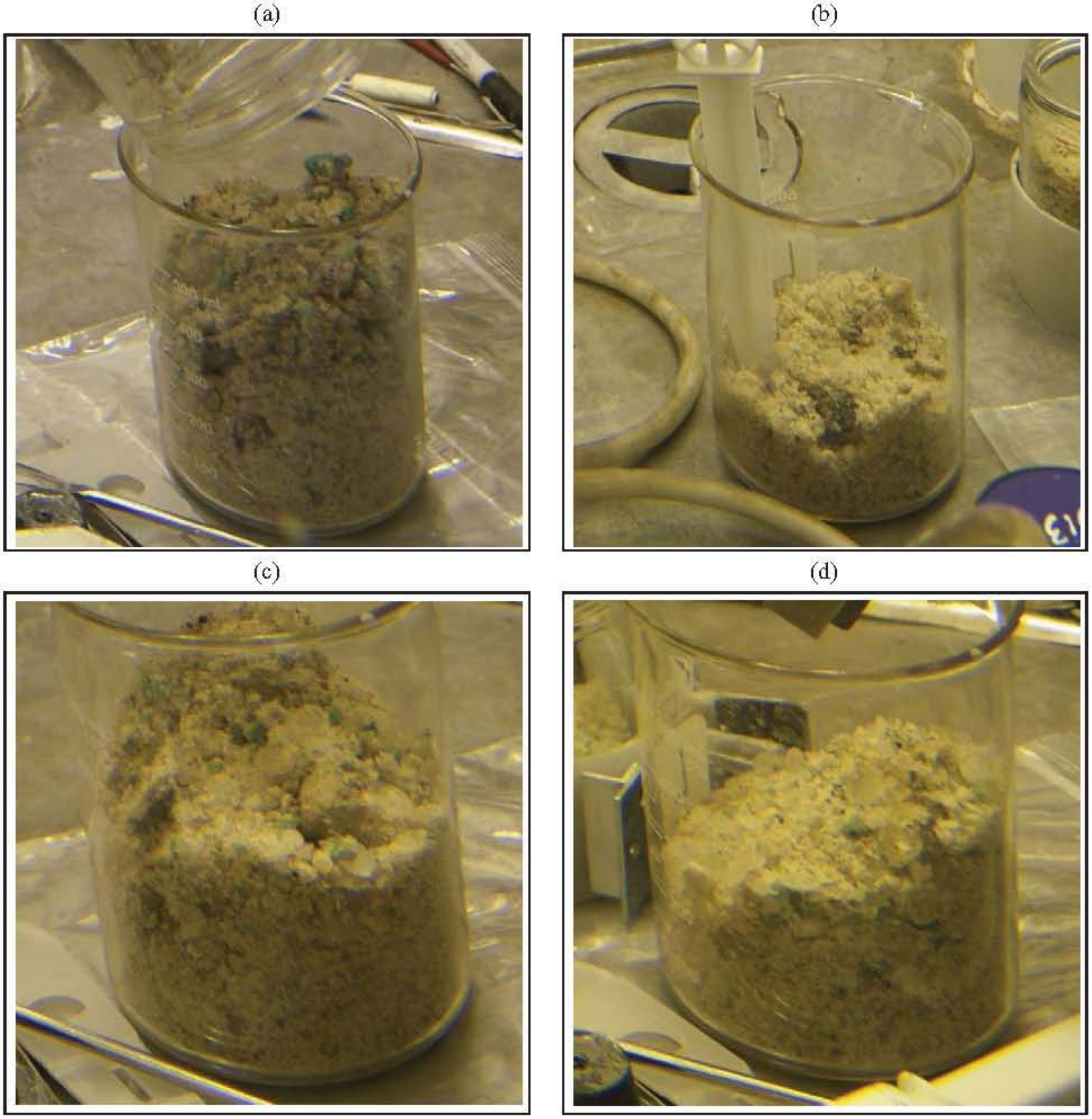
Composite	Sample C108-09-	Added (g)	Description
A	1	≈200	No free liquid was present; the solids appeared to be slightly damp. The solids were generally light-colored but numerous dark grains were visible. Several coarse pebble-sized pieces were visible. One large piece of dark colored solids and one large piece of light tan-gray solids were added to the composite.
	4	≈200	A small volume of yellow liquid was present. The free liquid was decanted off before sampling for the composite. The remaining solids were very moist. The solids appeared to be mostly light-colored solids ranging in size from coarse sand to medium pebbles. Several fine pebble to medium pebble-sized pieces of green-colored solids were also visible.
B	2	≈175	No free liquid was present; the sample solids appeared to be slightly damp. The composition of the solids was similar to that of Sample 4 with the addition of numerous fine pebble to medium pebble-sized pieces of nearly clear solids.
	5	≈175	No free liquid was present; the solids appeared to be slightly damp. Very few pieces of the green colored solids were observed. Numerous fine pebble to medium pebble-sized pieces of light-colored and clear solids were observed. The balance of the solids was a mixture of medium sand to very coarse sand - sized solids.
C	3	≈150	No free liquid was present; the solids appeared to be slightly damp. The appearance of the solids was very similar to that of Sample 5, though the overall grain size may have been slightly smaller.
	6	≈150	No free liquid was observed; the solids appeared to be very slightly damp. On the surface, the composition of the solids was very similar to that of Sample 3. However, one-third to one-half of the sample volume was a single cobble-sized piece of solids. The large piece appeared to be a strongly cemented aggregate of clear and light-colored solids with a dark-colored crust covering one side. The large aggregate was broken up (with difficulty) with a pestle and then the sample was stirred with a spatula before withdrawing the composite subsample.

The composite for the retrieval test program was prepared by mixing approximately equal portions of the three primary composites. The following weights of material were removed from the primary composite jars during the evening of July 21, 2009 and were transferred to two temporary holding jars:

Primary Composite	Wt to Test Composite
A	201.2 g
B	200.8 g
C	200.5 g

At the beginning of the work shift on July 22, the two holding jars were transferred to hot cell 11A-6. The total weight of the C-108 heel solids at this point was 602.730 g. The composite solids were then transferred to a 600-mL beaker (0.376 g was left in the transfer jars). As before, the composite solids were then thoroughly mixed by stirring with a large spatula while attempting not to break up any of the larger solid pieces. Selected photographs of the test composite at this stage of preparation are shown in Figure 2-1.

Figure 2-1. Preparation of C-108 Heel Solids Composite for Retrieval Testing



After the test composite solids were thoroughly mixed, 59.57 g were withdrawn and transferred to Jar 20133. This subsample of the C-108 heel solids was set aside as archive sample S09R000140.

An additional 49.69 g of the test composite was then transferred to a ceramic mortar and pestle. The heel solids were crushed and ground until a visually uniform consistency was obtained. A 10.317 g subsample of the crushed solids (Sample S09R000143) was immediately transferred to a sample vial and set aside for the measurement of wt% H_2O. The remaining 37.421 g of crushed heel solids were transferred to sample jar HC-0 and set aside for chemical analysis.

The test composite solids remaining in the 600-mL beaker were then transferred to a 400-mL wide-mouth sample jar. A total of 488.282 g of solids were transferred to the composite jar. A total of 1.39 g were left in the beaker.

A mass balance for the preparation and initial subsampling of the test composite of the C-108 heel solids follows.

	Wt of initial composite additions	602.730 g	
–	Wt of sample left in primary transfer jars	0.376 g	
–	Wt of archive sample	59.57 g	
–	Wt of sample transferred to mortar and pestle	49.69 g	
–	Wt of sample left in 600-mL beaker	1.39 g	
	Calculated final wt of composite sample	491.704 g	
–	Preparation and handling losses	5.188 g	(0.86 wt%)
	Actual final wt of composite sample	488.282 g	

There were ≈3.42 g of undefined losses of C-108 heel solids during the preparation and initial subsampling of the test composite. Combined with the 1.77 g that were left in the preparation containers, a total of 5.19 g (0.86 wt%) of the initial heel solids were lost.

2.3 HEEL SAMPLES 7 AND 8

As was previously noted, the final two samples of heel solids retrieved from C-108, Samples C108-09-7 and C108-09-8, were taken from the thin, hard layer of solids in the middle of the tank. Due to the extreme difficulties encountered in retrieving samples from the central layer, this test program was initiated with the preparation of a test composite that did not include the final two heel samples (as described in Section 2.2). The only measurement requested for the final two heel solids samples was characterization of the solid phase composition (see Section 3.2.2).

No free liquid was present in Samples 7 and 8. Visual observations suggested that the texture and composition of the heel solids in the central layer of residual solids in C-108 were, in general, similar to those observed in the other six samples.

3.0 ANALYSIS OF C-108 HEEL SOLIDS

3.1 PHYSICOCHEMICAL ANALYSES

3.1.1 Analysis Results

Four samples collected during the C-108 heel solids test program were submitted to the 222-S Laboratory for physicochemical analysis:

Test Composite	S09R000145	Sample of initial test composite solids (Section 2.2)
Bulk Density Liquid	S09R000146	Sample of liquid added to test composite to complete bulk density measurement (Sections 4.1.2 and 4.1.3)
Coarse Sieving Liquid	S09R000148	Sample of liquid used to facilitate separation of composite solids into <1/4-in. and >1/4-in. fractions (Section 4.3)
Dissolution Residue (DTR-6)	S09R000174	Sample of solids remaining after completion of the caustic dissolution test series (Section 7.0)

Transcripts of the data summary reports tabulating all analytical data for these four samples are presented in Attachment A. Details regarding sample preparation(s) and analytical methods/procedures used are included in Attachment A.

The results of the physicochemical analyses of the four samples are summarized in Table 3-1. All target analytes for which the results of all analyses 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 sample of the test composite for physicochemical analysis was originally collected in jar HC-0 on July 22, 2009 (Section 2.2). Prior to submitting the sample to the 222-S Laboratory in early October 2009, visual inspection revealed the presence of solid grains of sufficient size to make representative subsampling difficult. Approximately 15 g of the composite heel solids were transferred to a small mortar and pestle, where they were ground until no particles >1 mm in size were visible. A 10.096-g subsample of the homogenized composite was then transferred to sample vial S09R000145 and submitted to the laboratory for analysis. The remaining re-ground solids were returned to the portion of the July 22 sample remaining in jar HC-0 and set aside.

An additional subsample of the C-108 heel solids composite (S09R000143) was collected on July 22, 2009 and used to measure wt% H_2O. The subsample was immediately transferred to an oven set at ≈90 °C where it remained for 5 days. The 10.317-g subsample lost 3.612 g (35.01 wt%) upon drying. This measurement result is included in Table 3-1.

Table 3-1. Summary Results of Physicochemical Analyses of Test Samples

Analyte	Sample			
	Test Composite (S09R000145)	**Bulk Density Liquid (S09R000146)**	**Coarse Sieving Liquid (S09R000148)**	**Dissolution Residue (S09R000174)**
pH	11.7	—	—	9.92
Specific Gravity	—	1.138	1.126	—
Wt% water	35.0	—	—	69.3
	µg/g	**µg/mL**	**µg/mL**	**µg/g**
Total organic carbon	630	482	566	<366
Total inorganic carbon	5,790	6,870	6,930	1,490
Fluoride	15,544	2,225	2,066	20.78
Chloride	<78	109	109	7.43
Formate	<44.1	67.4	52.5	19.9
Acetate	<355	140	134	20.7
Oxalate	1,206	1,290	1,600	<59.0
Nitrite	8,220	11,700	11,800	99.0
Nitrate	7,495	13,600	13,700	345
Phosphate	158,000	25,100	23,600	3,620
Sulfate	1,465	1,380	1,500	21.0
Sodium	152,000	47,400	47,200	12,650
Aluminum	106,500	104	112	20,300
Silicon	788	<12.0	<12.0	10,545
Phosphorus	48,250	8,530	8,000	2,480
Sulfur	<2,550	498	506	<2,630
Calcium	<1,280	<20.0	<20.0	2,445
Chromium	<128	19.6	19.8	<132
Manganese	<76.5	<1.20	<1.20	1,179
Iron	3,075	44.1	45.2	75,300
Cobalt	<255	<4.00	<4.00	233
Nickel	3,530	<8.00	<8.00	46,900
Copper	<239	<2.00	<2.00	178
Zinc	<245	<2.00	<2.00	154
Strontium	580	8.75	10.8	9,450
Barium	<76.5	<1.20	<1.20	130
Lead	<1,280	<20.0	23.4	3,590
Thorium	<1,280	25.7	23.3	<1,320
Uranium	<2,550	1,090	1,120	9,045
Uranium-233	<0.0444	—	—	0.263
Uranium-234	0.0476	—	—	0.471
Uranium-235	6.14	—	—	57.6
Uranium-236	0.0728	—	—	0.779
Uranium-238	946	—	—	8,760
	µCi/g	—	—	**µCi/g**
Strontium-89/90	174	—	—	5,410
Technetium-99	0.00455	—	—	0.00260
Cesium-137	19.0	—	—	2.57
Plutonium-239/240	0.0105	—	—	0.124
Americium-241	0.0125	—	—	0.186

The wt% H_2O in the submitted samples of the test composite and the dissolution residue was also measured in the 222-S Laboratory. These measurements were based on thermogravimetric analyses performed on October 7, 2009. The average wt% H_2O found in ≈7.5-mg portions of the test composite was 28.2 wt%. The average wt% H_2O found in ≈20-mg portions of the dissolution residue was 69.3 wt%.

3.1.2 Free Liquid in C-108 Test Composite

The 35.0 wt% H_2O value reported for the test composite is somewhat misleading. No free liquid was visible in the test composite (Figure 2-1). Clearly, 170.9 g of free water (35.0 wt% of 488.3 g) was not present. As will be seen in Section 3.2 of this report, a major component of the C-108 heel solids is the fluoride-phosphate double salt $Na_7F(PO_4)_2 \cdot 19H_2O$ (natrophosphate). If the reported 15,544 µg/g fluoride were all in solid natrophosphate, the test composite would have contained 284.5 g of the double salt, 136.7 g of which would be waters of hydration. Another hydrated species believed to be present is $Na_3H(CO_3)_2 \cdot 2H_2O$ (trona). If the reported 5,790 µg/g of total inorganic carbon is converted first to carbonate (CO_3) and then to trona, up to 4.24 g of the water in the composite could have been bound as hydration water in 26.60 g of the carbonate salt. This would leave ≈30 g of H_2O (6.1 wt%) as a possible component of interstitial liquid (ISL) in the test composite, which is a value in much better agreement with visual observation. Small amounts of natrophosphate and trona were probably in solution in the composite ISL and, therefore, slightly more than 30 g of H_2O was probably in the ISL. However, the portion of free water in the ISL was much closer to 6.1 wt% than 35.0 wt%. (The water balance is revisited in Section 4.2.1.)

3.2 SOLID PHASE CHARACTERIZATION

Solid phase characterization (SPC) was performed on subsamples of water-washed >1/4-in. and <1/4-in. heel solids. SPC included examination of the heel solids using polarized light microscopy (PLM) (ATS-LT-519-107, “222-S Laboratory Polarized Light Microscopy”), scanning electron microscopy with energy dispersive X-ray spectrometry (SEM/EDS) (ATS-LT-161-100, “222-S Laboratory Sample Preparation and Operating Procedure for Scanning Electron Microscopes”), and powder X-ray diffraction (XRD) (ATS-LT-507-101, “222-S Laboratory X-ray Diffractometry”).

3.2.1 Test Composite

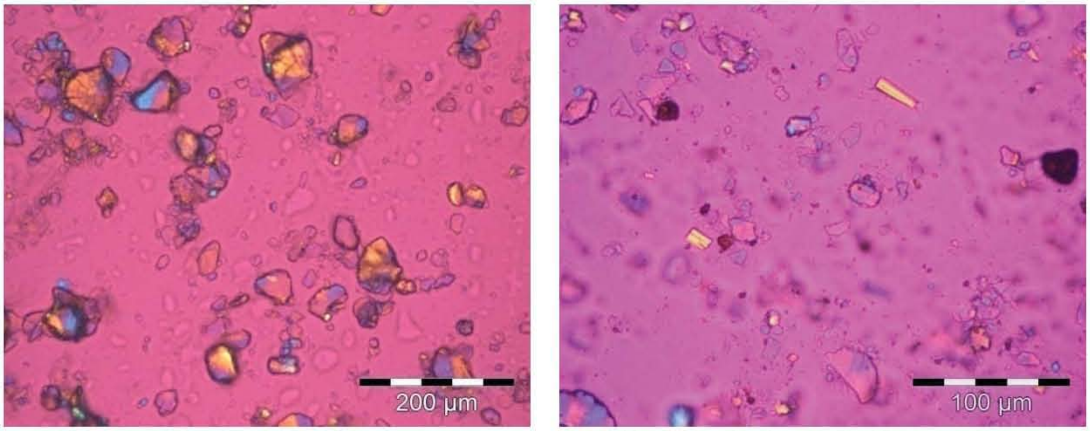
Coarse sieving (Section 4.3) created two sample fractions: one (S09R000147) containing fine-grained (<1/4-in.) solids and one (S09R000162) containing coarse-grained (>1/4-in.) solids. A subsample of the fine-grained fraction (S09R000152) was used directly for SPC. The coarse-grained particles were subdivided based on optical appearance into a clear white-, a green-, a black-, and a tan-colored fraction. Then a piece from each fraction was chosen for the crush test

(Section 4.5). After crushing, the solids were identified as sample S09R000163 and were used for SPC.

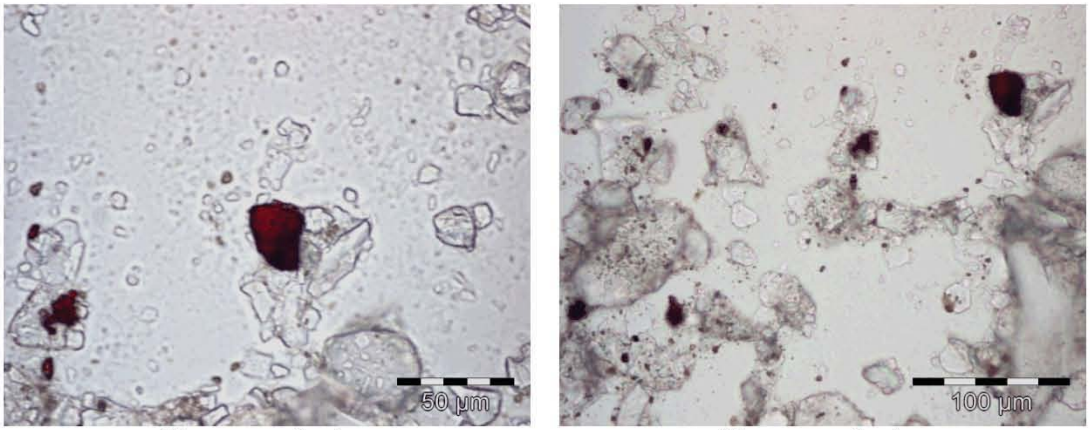
3.2.1.1. Polarized Light Microscopy. PLM of the fine-grained sample S09R000152 showed the presence of three components: Gibbsite, Natrophosphate, and an amber phase. Gibbsite was found to be slightly less abundant than Natrophosphate (~40% gibbsite, ~60% Natrophosphate), with minor amounts of the amber phase.

Figure 3-1 shows the variation in composition within the slide. Approximately equal amounts of gibbsite (multi-colored) and Natrophosphate (clear) can be seen on the top left image. On the top right image, a very small amount of tiny gibbsite crystals is visible but there is nearly the same amount of Natrophosphate as in the left picture. Three amber particles and two Natrophosphate crystals (yellow laths on top of each other) are also visible in the right image. The bottom row shows images with uncrossed polars revealing the amber and black particles. The green Ni-Al-phosphate phase could not be identified.

Figure 3-1. Polarized Light Microscopy Images of the Fine-Grained Fraction



20x, crossed polars w. Red I compensator   40x, crossed polars w. Red I compensator



60x, uncrossed polars   40x, uncrossed polars

3.2.1.2. X-ray Diffraction

In the fine-grained sample, two major phases were identified: Sodium Fluoride Phosphate [$Na_7F(PO_4)_2 \cdot 19H_2O$] and Gibbsite [$Al(OH)_3$] in an approximately 1:1 ratio (Figure 3-2).

From the coarse-grained fraction (S09R000163), three subsamples were prepared. The first subsample (Figure 3-3) was prepared from the white- and the tan-colored particles. Nastrophosphate $Na_7F(PO_4)_2(H_2O)_{19}$ and Gibbsite $Al(OH)_3$ were the phases identified.

Figure 3-2. X-ray Diffraction Spectrum of the Fine-Grained Fraction

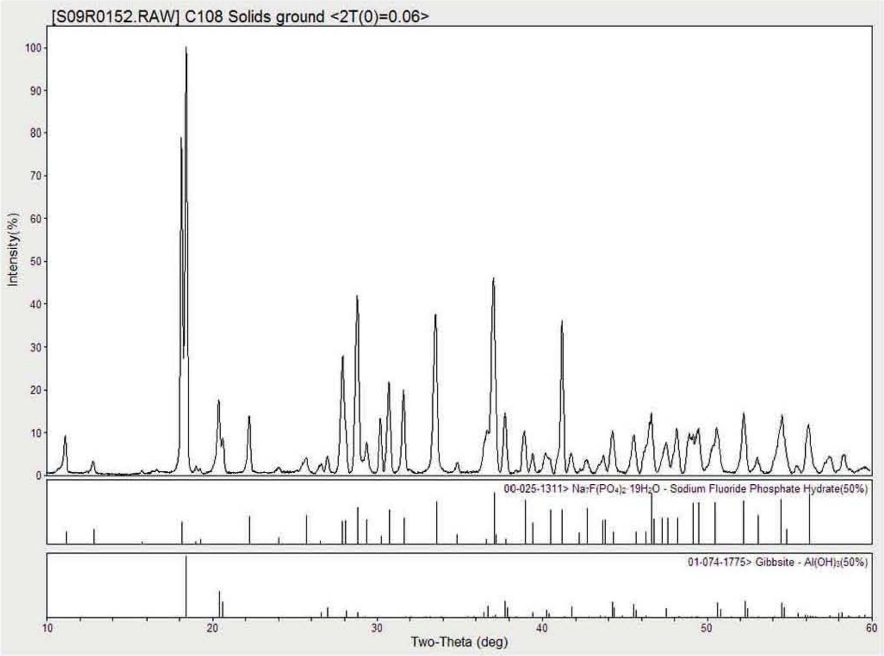
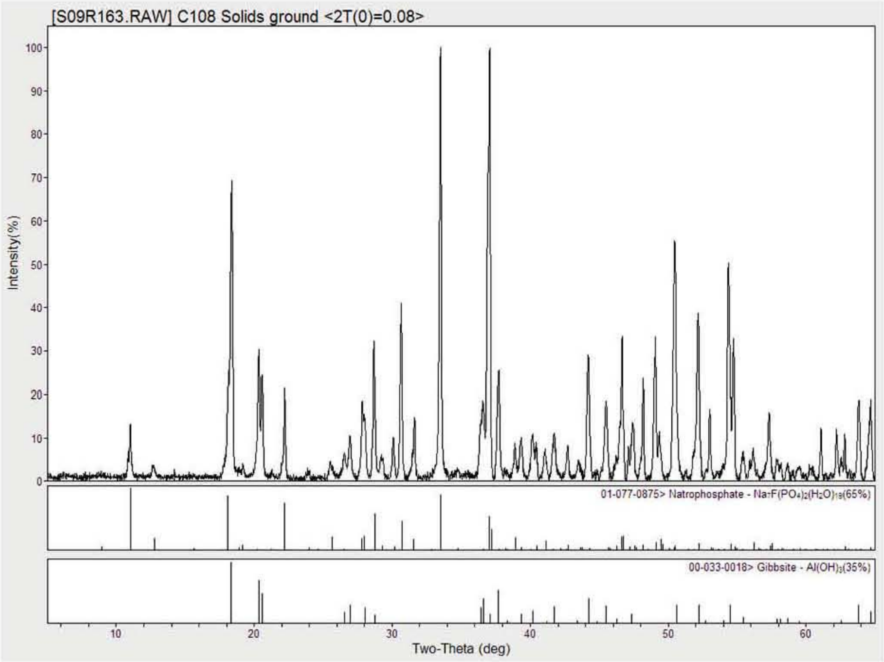
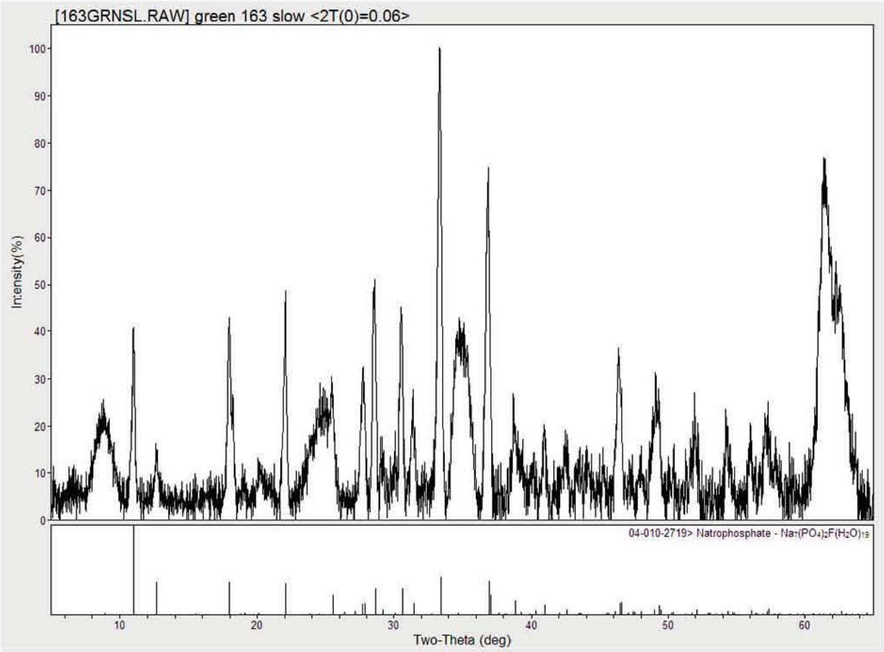


Figure 3-3. X-ray Diffraction Spectrum of the White- and Tan-Colored Particles in the Coarse-Grained Fraction



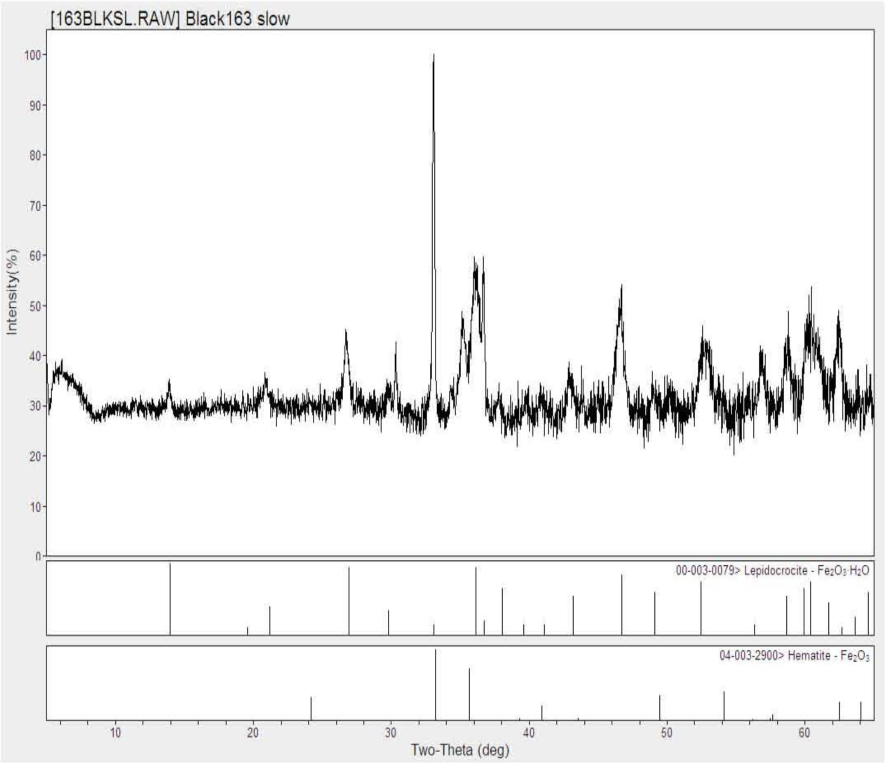
The second subsample was prepared to identify the green-colored phase. The spectrum in Figure 3-4 shows sharp Natrophosphate peaks caused by the white 'crud' (original comment by technician) during preparation of the XRD sample. The small crystallite size of the unknown substance causes the broadness of the other peaks. To date, no matching pattern has been found.

Figure 3-4. X-ray Diffraction Spectrum of the Green Particles in the Coarse-Grained Fraction



A piece of the black scale was prepared as the third subsample. The spectrum in Figure 3-5 shows peaks of lepidocrocite and hematite (both phases rust scales) with a high background caused by non-crystalline Fe-bearing material.

Figure 3-5. X-ray Diffraction Spectrum of the Black Particles in the Coarse-Grained Fraction



3.2.1.3. Scanning Electron Microscopy. SEM-EDS was performed on two different sample preparations of the fine-grained material. One of the preparations was analyzed manually to search for specific phases as well as with several runs of the automated feature analysis (AFA) mode [1] to identify average sample compositions. The other preparation contained five large rounded particles that were not used in the first preparation. These particles turned out to be relatively large (~2 mm) Natrophosphate crystal. Figure 3-6 shows the backscattered electron images (BSE) of one of these large crystals. Natrophosphate was not found in a smaller variety since it most likely dissolved in the water during SEM preparation. Therefore, the results of the automated runs presented in Table D-2 on run 37 and run 38 lack one of the major components of the original sample, ~50 vol% Natrophosphate.

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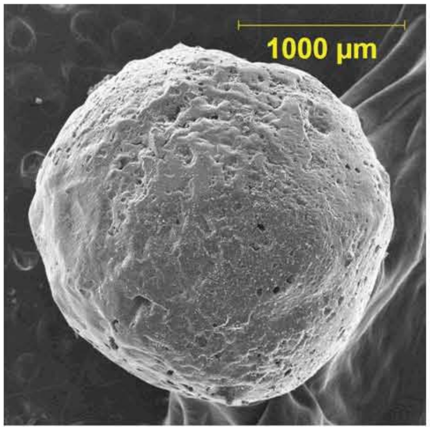
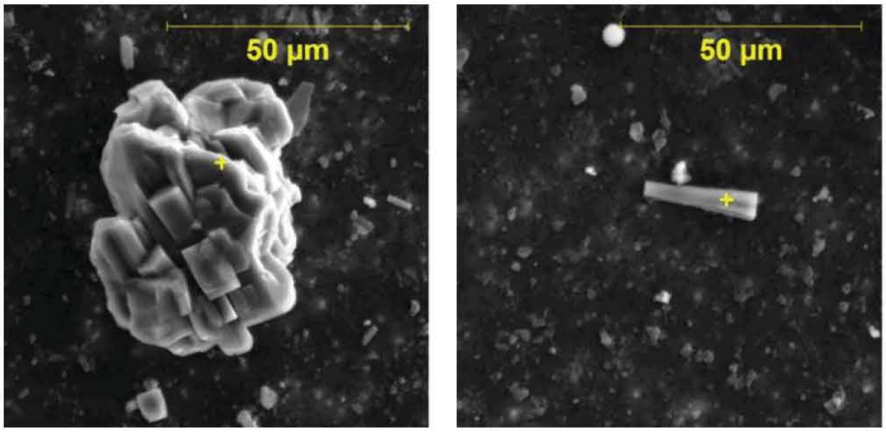
[1] AFA is a registered trademark of ASPEX Corporation, Delmont, Pennsylvania.

Figure 3-6. Backscattered Electron Image of Large Natrophosphate Crystal from the Fine-Grained Fraction



Gibbsite is present in two morphologies and makes up about 32 vol%[2]. Most of the particles are agglomerates as shown in the left BSE image of Figure 3-7. Some appear as tapering laths, which might be cleavage fragments of the larger particles. The bright white ball at the top of the right image is a piece of cancrinite (Na-Al-silicate).

Figure 3-7. Backscattered Electron Images of Two Morphologies of Gibbsite from the Fine-grained Fraction



The ***green particles***, which could not be characterized with XRD, turned out to be Ni-(U)-Al-P-bearing. The coloration is most likely due to the presence of Nickel in these samples. As shown by the SEM results in Figures 3-8 and 3-9, this species seems to be poorly crystalline with crystallite sizes of less than 10 µm. The broad peaks in the XRD spectrum of Figure 3-4 also reflect the poor crystallinity.

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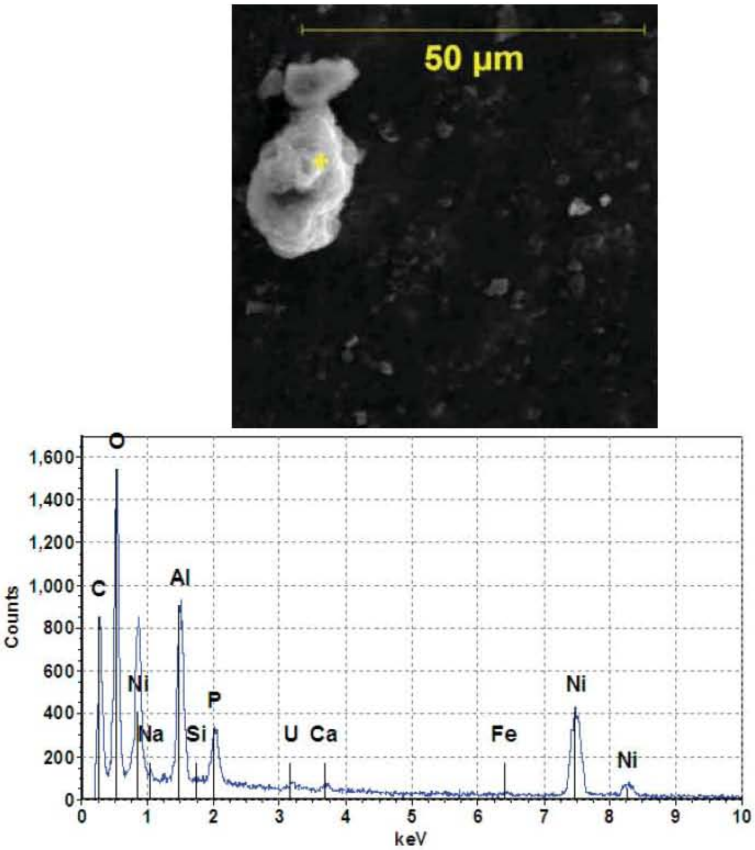
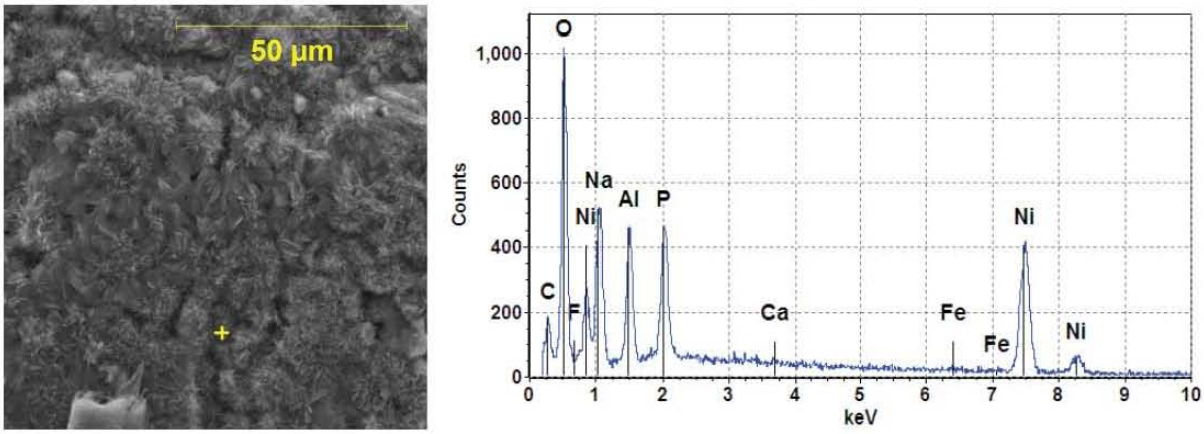
[2] Based on the automated SEM runs but taking 50 vol% of Natrophosphate into account.

Figure 3-8. Backscattered Electron Image and Energy Dispersive Spectrometry Spectrum of Green Particulate from the Fine-Grained Fraction



During the examination of a larger particle of the green material, the surface turned out to be made of an assemblage of small crystallites as shown in Figure 3-9.

Figure 3-9. Backscattered Electron Image and Energy Dispersive Spectrometry Spectrum of a ~2 mm Green Particulate

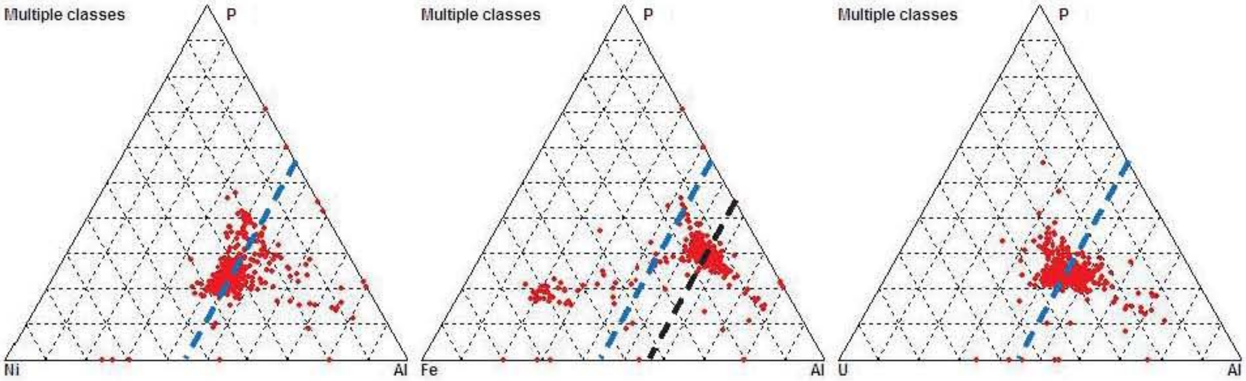


A stoichiometric mineral composition of this phase could not be derived. However, based on the results of the automated SEM runs some key features were identifiable. For example, the ratios of the peak intensities for Ni, Al, Fe, U, P and other elements led to a rough estimate of the composition. Ternary plots of these intensities can help significantly to identify qualitative similarities between elemental contents. However, these observations should only be considered in terms of ratios of individual elements to each other.

For illustration of the derivation process, three examples of ternary plots showing the Ni-variations within the particles of run 37 are displayed in Figure 3-10. The graphs plot only data from three categories in the AFA run[3]: the Ni-Al-U-P rich, Ni-Al-P rich, and a group called "miscellaneous." The remaining categories were considered as not associated with the "green phase."

As an example, the left ternary diagram will be explained in detail. The graph takes the peak intensities of Al, Ni, and P, normalized to 100% Al+Ni+P. The apex of the triangle represents 100% P, the left corner 100% Al, and the right corner 100% Ni. The central diagram plots the intensities of Fe, P, and Al, and the diagram on the right side compares the intensities of U, P, and Al. Using the average Al/P ratio as a handle (indicated by the blue line), the other elements can be set in perspective.

Figure 3-10. Ternary Energy Dispersive Spectrometry Peak Intensity Diagrams to Identify the Green Phase



The observations can be summarized as:

(1) Uranium substitutes for Nickel. The Al/P ratio for the majority of U-bearing particles is about equal to the ones with high Ni contents. Na (not shown) substitutes equally for Ni.
(2) Iron does not substitute for Nickel (central diagram). The average Al/P for the main cluster is different for the Fe- and the Ni-bearing phase (middle diagram, blue versus black line). Therefore, these are two separate phases.
(3) The intensity ratios of Ni/Al, Al/P, etc. implement the following:
    a. Average molar amounts[4] in the Ni-bearing phase: Al:P ~ 2, Na:Ni ~ 2, Al:Ni ~ 2.7.
    b. Average molar amounts[4] in the Fe-bearing phase: Fe:P ~ 0.3, Al:P ~ 2.

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[3] See Table D-2 for a description of the individual categories from the AFA run.

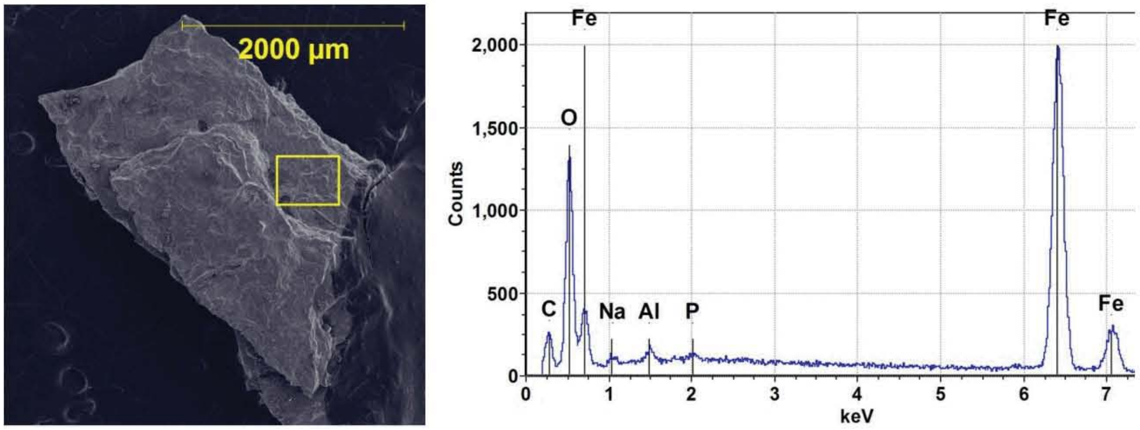
[4] Average molar amounts are calculated as the average peak intensity ratios (~ ratios of wt%) divided by the molecular weights.

Combining this information, a possible composition is in the range of $Na_{2x}(Ni,U)_xAl_{2y}(PO_4)_yZ^-_{6y}$ with x ~3, y ~4, Z = (OH^-, F^- etc.). Another interpretation of the ratios might be that the material contains ~$(Ni,U)_3Al_2(PO_4)_4$ plus 6 $NaAl(OH)_4$. The XRD pattern does not indicate the presence of sodium aluminate; however, this phase might be hidden in the elevated background due to its poor crystallinity.

In any case, this phase (Ni-Al-U-bearing phosphate) has not been identified in any other tank, and contains uranium in a non-standard phase for tank farm inventory. Likewise, the identification of the Fe-bearing phase is not finished. This phase most likely also contains Pb, as in the example presented below (see ‘minor phases’).

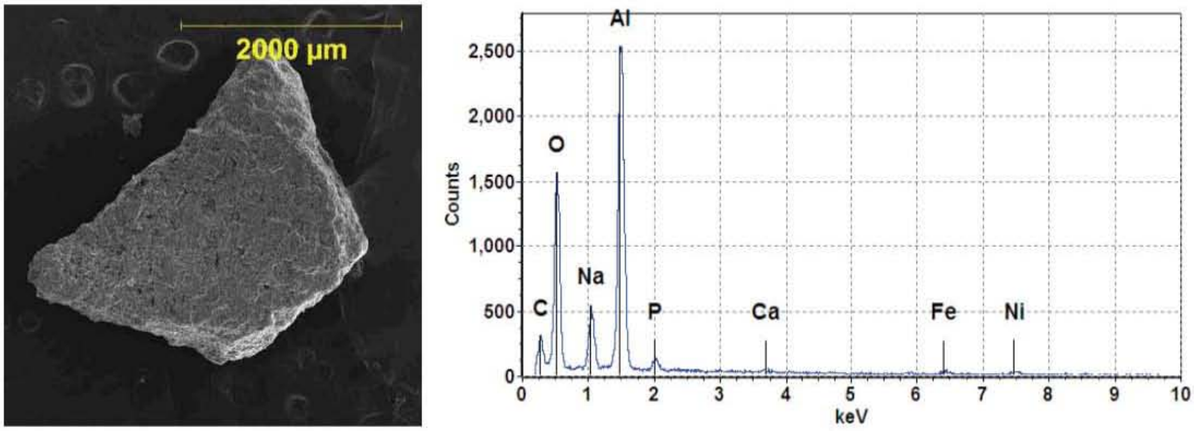
The ***black material*** turned out to be made of two entirely different phases. The majority of the particles appear like carbon steel scale and were magnetic containing mostly Fe, with traces of Na, Al, and P. These flakes were identified by XRD as being made of Fe-hydroxides.

Figure 3-11. Backscattered Electron Image and Energy Dispersive Spectrometry Spectrum of the Magnetized Black Scales from the Fine-Grained Fraction



A different kind of very dark material in the fine-grained fraction turned out to be Al-rich (see Figure 3-12). Due to the large Na-peak, it is potentially a mix of dawsonite and gibbsite. The EDS-spectrum in Figure 3-12 was taken over the entire surface of the particle so as not to miss any trace phases. In Figure 4-1, this dark material can be seen on the surface of the large chunks of gibbsite.

Figure 3-12. Backscattered Electron Image and Energy Dispersive Spectrometry Spectrum of the Second Variety of Fine-Grained Dark Material



Minor phases found in the fine-grained pre-dissolution material include an Fe-Pb-rich phase (see Figure 3-13) and a Sr-phosphate-rich phase (Figure 3-14). The Sr-phosphate turns out to be one of the main phases in the post-dissolution sample (see XRD spectrum in Figure 8-14). This phase was missed in the automated run (see Table D-2), since the L-line of Sr is between the Si and P lines, and the K-line is too weak to be detected.

Figure 3-13. Backscattered Electron Image and Energy Dispersive Spectrometry Spectrum of Fe-Al-Pb-P-Phase

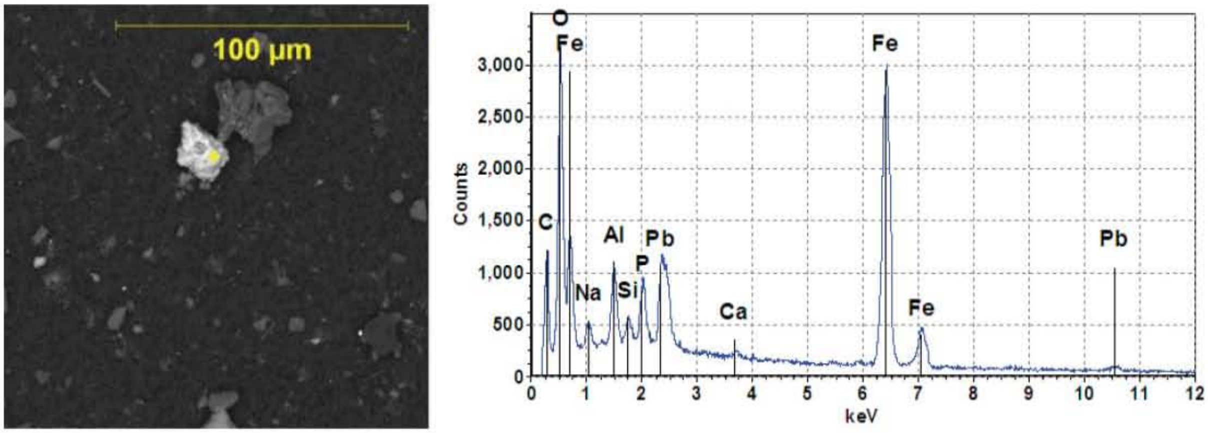
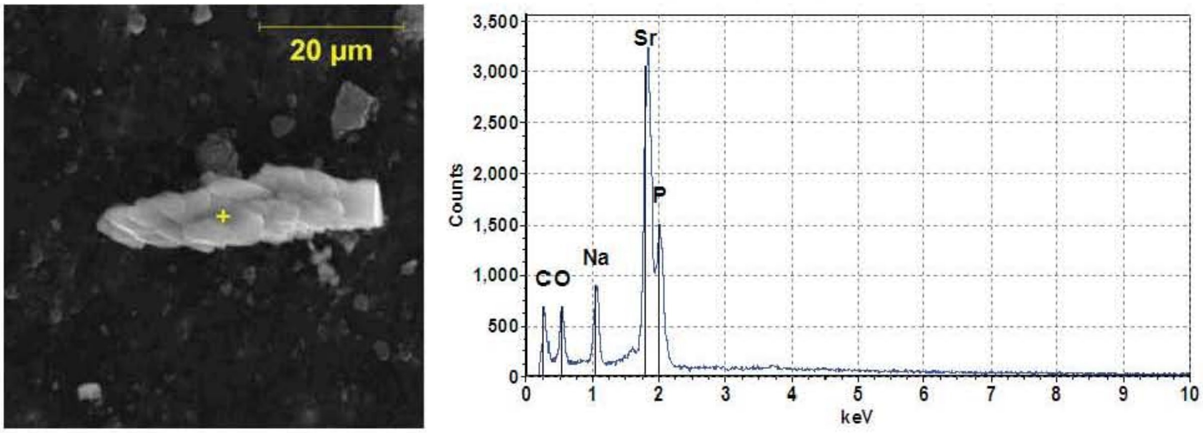


Figure 3-14. Backscattered Electron Image and Energy Dispersive Spectrometry Spectrum of Na-Sr-P-Phase (nastrophite)



3.2.2 Heel Samples 7 and 8

Subsamples taken from heel samples C108-09-07 and C108-09-08 were identified as laboratory samples S09R000308 and S09R000309, respectively. The only analysis requested for these samples was solid phase characterization. Since all phases identified are equivalent to the ones described above, the images and spectra are added in Attachment B. Figure B-1 contains the PLM results and Figures B-2 and B-3 show the XRD spectra; SEM results are displayed in Figures B-4 and B-5.

3.2.2.1. Polarized Light Microscopy. PLM was performed on both samples with very similar results. The main phases are gibbsite and sodium fluoride phosphate with some carbonate and amber particles interspersed.

3.2.2.2. X-ray Diffraction. Slight differences were observed for the two samples:

Mineral	Formula	S09R000308 [rel.%]	S09R000309 [rel.%]
Gibbsite	Aluminum Hydroxide $Al(OH)_3$	35	20
Natrophosphate	Sodium Fluoride Phosphate $Na_7F(PO_4)_2 \cdot 19H_2O$	30	55
Trona	Sodium Bicarbonate Hydrate $Na_3H(CO_3)_2 \cdot 2H_2O$	30	25
Sodium Nitrite[*]	$NaNO_2$	5	-

[*] The sodium nitrite is most likely precipitated from solution in the supernatant liquid and was not part of the original solids.

3.2.2.3. Scanning Electron Microscopy. Both samples were prepared by smearing a portion of sample on a plastic Petri dish, chopping the dried sample with a razor, and lifting the particulate onto a SEM stub coated with an adhesive tab.

Sample S09R000308 contained gibbsite, natrophosphate, and a sodium-rich phase identified by XRD as trona as the primary phases. Minor phases included nastrophite [Na(Sr,Ba)(PO_4)•9(H_2O)], a Na-U rich phase (sodium diuranate, Clarkeite or Cejkaite), an Fe-Pb-bearing phase and a Ni-Al-P bearing phase. Examples of the morphology of each phase and the chemistry of these crystals are shown in Figure B-4.

Sample S09R000309 contained the same phases as sample S09R000308. Examples of the morphology of each phase and the chemistry of these crystals are shown in Figure B-5.

4.0 PHYSICAL CHARACTERIZATION OF TEST COMPOSITE

4.1 BULK DENSITY

4.1.1 Apparatus

The bulk density (D_{Bulk}) of the C-108 heel solids composite was measured by differential pycnometry. The test sample was transferred to a wide-mouthed, 400-mL sample jar. A special lid had been fabricated for this jar 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 478.68 ± 0.17 mL. The volume calibration data is included in Section 8.2.3.

4.1.2 Bulk Density Measurement

The D_{Bulk} measurement was made on all of the damp, C-108 heel composite solids that remained after the archive and two analytical subsamples were removed (Section 2.2). No free liquid was present in the heel solids composite.

The C-108 heel solids composite sample was transferred (as completely as possible) to the 400-mL pycnometer jar at 15:00 on July 22, 2009. The pycnometer lid was loosely attached and the pycnometer containing the composite test sample was weighed. An initial ≈100-mL portion of reagent water was then added to the jar. The composite and added water were gently, but thoroughly mixed by stirring with a spatula. A regular (solid) lid was then attached to the pycnometer jar and the sample was set aside to settle.

The D_{Bulk} measurement resumed at 09:30 on July 23. The supernatant liquid (the bulk density liquid or BDL) was completely clear. While removing the regular lid from the pycnometer jar, a small quantity of the BDL was spilled. The weight of the liquid spilled was determined to be 3.464 g. A second ≈40-mL portion of reagent water was added to the pycnometer jar and the contents gently stirred. A third ≈40-mL portion of reagent water was added nearly completely filling the working volume of the jar. Stirring was limited to that required to mix the added water with the BDL in the jar. The pycnometer lid was attached to the jar and torqued. The pycnometer was then filled to the calibrated volume mark with reagent water and weighed. This operation was completed by 10:30. The temperature of the BDL in the pycnometer jar was 27.5 °C. The reference density of pure water at 27.5 °C is 0.996374 g/cm^3. Using the reference density, the weight of the water added to the pycnometer was converted to a volume. The volume of the test sample was calculated as the difference between the pycnometer jar volume and the volume of the added water. Using this volume, D_{Bulk} of the damp C-108 heel composite (replacing the spilled BDL with reagent water) was calculated as 1.879 g/cm^3 (at 27.5 °C). The gravimetric and volumetric data recorded during the measurement and the sequence of calculations leading to D_{Bulk} follows.

Temperature hot cell (°C)	27.5					
D_{H2O} at hot cell temperature (g/mL)	0.996374					
Vol pycnometer (mL)	478.680					
Wt pycnometer (g)	317.532					
Wt (pycnometer + sample) (g)	805.814					
Wt sample (g)	488.282					
Wt 1st H_2O addition (g)	101.828					
Wt spilled liquid (g)	3.464					
Wt 2nd H_2O addition (g)	36.412					
Wt 3rd H_2O addition (g)	39.971					
Wt final H_2O addition (g)	43.275					
Wt (pycnometer + sample + H_2O) (g)	1023.836					
Assumed density of spilled liquid (g/mL)		1.120	1.220	1.320	1.420	1.520
Vol spilled liquid (mL)		3.093	2.839	2.624	2.439	2.279
Wt replacement water (g)		3.082	2.829	2.615	2.431	2.271
Adjusted wt (pycnometer + sample + H_2O) (g)		1024.218	1024.471	1024.685	1024.869	1025.029
Wt added H_2O (g)		218.404	218.657	218.871	219.055	219.215
Vol of added H_2O (mL)		219.199	219.453	219.668	219.853	220.013
Vol sample (mL)		259.481	259.227	259.012	258.827	258.667
D_{Bulk} of composite at 27.5 °C (g/cm^3)		1.8818	1.8836	1.8852	1.8865	1.8877

The net effect of the small spill after the first addition of water was to replace a volume of the intermediate BDL with an equal volume of pure water. It is assumed that the density of the added water was less than that of the spilled BDL. Without applying a correction, the final weight of the filled pycnometer [i.e., Wt (pycnometer + sample + H_2O)] would be less than it would have been had the spill not occurred. As a result, the calculated volume of the sample would be too large and the calculated D_{Bulk} too small. Without knowing the quantity and density of the ISL in the original composite or the density of the spilled BDL, it is difficult to quantify the bias.

The D_{Bulk} of the C-108 heel composite has been calculated using five densities for the spilled liquid. The density of the final BDL was measured as 1.120 g/mL and this is taken to be a lower limit for the density of the spilled liquid. If the ≈120 mL of water added after the spill only diluted the liquid remaining in the pycnometer, the implied density of the spilled liquid increases to 1.220 g/mL. As shown, further incremental increases in the assumed density of the spilled liquid lead to small increases in the calculated bulk density values. For this study, it will be assumed that the density of the spilled liquid was between 1.120 and 1.320 g/mL. The reported best estimate of D_{Bulk} of the C-108 heel solids composite sample is 1.884 g/cm^3.

4.1.3 Bulk Density Liquid

After the final weight of the filled pycnometer was recorded, ≈100 mL of BDL was removed with a pipette so the pycnometer lid could be removed. The liquid and entrained solids were transferred to a 400-mL wide-mouthed jar that was used as a receiver during the forthcoming coarse-sieving operation (Section 4.3). The composite solids and liquid remaining in the pycnometer jar were mixed for a final time by gentle stirring before the regular lid was attached to the jar at the end of the work shift on July 23, 2009.

At the start of the work shift on July 27, the supernatant liquids in both the pycnometer and sieve receiver jars were completely clear. All the liquid that could be retrieved without solids was removed from both jars with a pipette and transferred to a common sample bottle. A total of 170.10 g of solids-free BDL was recovered. The very fine solids (≈4.2 g) that had settled to the bottom of the sieve receiver were retained in that jar.

Two subsamples of the BDL were retrieved: one (S09R000146) for submission to the laboratory for physicochemical analysis (Table 3-1) and one for measurement of wt% dissolved solids. The wt% dissolved solids sample was dried to constant weight at 90 °C. The final 0.710 g of dry residue represented 13.26 wt% of the original 5.356-g sample; the entire weight loss was assigned to water making the BDL 86.74 wt% H_2O.

The density of the BDL was measured by filling and weighing a 25-mL, Class A volumetric flask (volume 25 ± 0.03 mL at 20 °C). The measured density of the BDL was 1.120 g/mL at 26.9 °C.

4.2 DRY SOLIDS DENSITY

Usually, the average density of the dry solids contained in the C-108 heel composite ($D_{\text{Dry Slds}}$) would be estimated by backing out the weight and volume of the ISL in the composite from the bulk density calculations. The most direct calculation sequence is based on an assumption that all water in the composite is associated with the ISL and requires input of two measured properties of the ISL, density and weight fraction of water (f_{H2O}). However, the majority of the water in the C-108 heel composite is not associated with ISL. Furthermore, because no free liquid was present in the C-108 heel test composite, no proxy sample for the ISL could be collected for analysis. Also, estimation of $D_{\text{Dry Slds}}$ was further complicated by the fact that a major portion of the C-108 heel solids are water soluble.

An alternate approach to $D_{\text{Dry Slds}}$ for the C-108 heel solids in essence replaces the ISL with the BDL. The 'wet' composite solids are redefined as the combination of the composite sample and the BDL that was present after the final filling of the pycnometer during the D_{Bulk} measurement. The $D_{\text{Dry Slds}}$ value returned applies to solids that did not dissolve as a result of the addition of water to the composite during the D_{Bulk} measurement.

4.2.1 Weights of Key Components

In Table 4-1, the concentration data presented in Table 3-1 has been converted to weights of key components in the test composite, in the BDL, and in the coarse sieving liquid (CSL).

Table 4-1. Weights of Components in Heel Composite and Test Liquids

Analyte	C-108 Heel Composite	Bulk Density Liquid	BDL Samples and Losses	Coarse Sieving Liquid
Total wt (g)	488.282	301.524	24.987	315.400
Density (g/mL)	—	1.120	1.120	1.118
Total wt% water	35.01	86.74	86.74	86.83
Water and Solids	**g**	**g**	**g**	**g**
Total Water	170.948	261.554	21.675	273.875
Water Added	0	218.022	18.067	232.971
Water in/from natrophosphate	136.746	10.796	0.895	10.495
Water from carbonate	4.240	2.775	0.230	2.931
Unassigned Water	29.961	29.961	2.483	27.478
Dissolved Solids	?	39.970	3.312	41.526
Highly Soluble	**g**	**g**	**g**	**g**
Chloride	<0.038	0.029	0.002	0.031
Formate	<0.022	0.018	0.002	0.015
Acetate	<0.173	0.038	0.003	0.038
Nitrite	4.014	3.151	0.261	3.327
Nitrate	3.660	3.662	0.303	3.863
Moderately Soluble	**g**	**g**	**g**	**g**
Total inorganic carbon (as CO_3)	14.125	9.243	0.766	9.763
Fluoride	7.590	0.599	0.050	0.582
Oxalate	0.589	0.347	0.029	0.451
Phosphate	77.149	6.759	0.560	6.655
Sulfate	0.715	0.372	0.031	0.423
Sodium	74.219	12.764	1.058	13.310
Uranium	0.465	0.294	0.024	0.316
Insoluble	**g**	**g**	**g**	**g**
Aluminum	52.002	0.028	0.002	0.032
Silicon	0.385	<0.003	<0.0003	<0.003
Calcium	<0.625	<0.005	<0.0004	<0.006
Manganese	<0.037	<0.0003	<0.00003	<0.000
Iron	1.501	0.012	0.001	0.013
Nickel	1.724	<0.002	<0.0002	<0.002
Strontium	0.283	0.002	0.0002	0.003
Lead	<0.625	<0.005	<0.0004	0.007

The 'Insoluble' components in the table were present in the test composite and/or were concentrated into the final dissolution test residue but were not present at significant levels in the BDL or CSL. The 'Highly Soluble' components were probably dissolved in the composite ISL or were nearly completely dissolved as the BDL was created during the D_{Bulk} measurement.

The 'Moderately Soluble' components are assumed to have been incorporated (at least in part) in the composite solids and then partially dissolved during the water additions of the D_{Bulk} measurement. Two key components of these solids are natrophosphate [$Na_7F(PO_4)_2 \cdot 19H_2O$] and trona [$Na_3H(CO_3)_2 \cdot 2H_2O$]. These were the only fluoride and carbonate containing species identified in the C-108 heel solids by solid phase characterization.

The tabulated weights of the chemical species in the test composite were calculated by multiplying the reported µg/g analytical data (Table 3-1) by the known sample weight (488.282 g). Similarly, the total weight of water in the composite was calculated by multiplying the sample weight by the measured wt% H_2O (Section 3.1.1). The entire 7.59 g of fluoride calculated to be present in the composite is assumed to be associated with natrophosphate; the entire 14.12 g of carbonate is assumed to be associated with trona. Multiplication of the fluoride weight by a gravimetric factor of 18.02 indicates that 136.7 g of the total water in the composite was associated with natrophosphate (in the solid state and/or in the composite ISL). Multiplication of the carbonate weight by a gravimetric factor of 0.3002 indicates that 4.2 g of the total water in the composite was associated with trona (in the solid state and/or in the composite ISL). The remaining 30.0 g of unassigned water is assumed to have been part of the composite ISL and to have been completely incorporated in the BDL and CSL.

The remaining columns in Table 4-1 characterize test liquids. The total weights of the BDL and CSL were not directly measured or measureable. An initial estimate of the total weight of water present was first calculated by making appropriate adjustments to the aqueous subcomponents; initially, the 'Water in/from natrophosphate' and 'Water in/from carbonate' were set to zero. An initial total weight for the test liquid was then calculated by dividing the initial total water weight by the measured f_{H2O} for the test liquid (Sections 4.1.3 and 4.3.2.2). The initial total weight, the measured liquid density, and the µg/mL analytical data were then used to calculate initial weights of the chemical species present in the test liquid. This calculation sequence was then repeated several times. In each iteration, the fluoride and carbonate weights calculated in the previous step were first used to calculate revised 'Water in/from natrophosphate' and 'Water in/from carbonate' values. The revised total water weight then led to a revised total weight and then to revised species weights. The iterative calculations were terminated when the calculated fluoride and carbonate weights became constant (i.e., no longer increased.)

The 'BDL Samples and Losses' column describes the quantity of BDL removed as samples at the conclusion of the D_{Bulk} measurement (18.921 g) and estimated to have been lost during the coarse-sieving operations (6.066 g). The composition of the test liquid removed from the test system was assumed to be identical to the BDL at the conclusion of the D_{Bulk} measurements.

4.2.2 Calculated Dry Solids Density

The amounts of natrophosphate and trona in solution in the composite ISL plus any that dissolved during the D_{Bulk} measurement transferred 0.599 g of fluoride and 9.24 g of carbonate to the final BDL. Converting the fluoride to natrophosphate water and carbonate to trona water indicates that 13.6 g of the water in the BDL was associated with the hydrated salts. Combining the 13.6 g of water from the dissolved salts, the 30.0 g of unassigned (ISL) water carried over from the composite, and the 218.022 g of water added during the D_{Bulk} measurement indicates that the BDL contained a total of 261.6 g of water. The measured f_{H2O} in the BDL was 0.8674. The total weight of the BDL in the filled pycnometer can thus be calculated as:

$$\begin{aligned} Wt_{BDL} &= (Wt\ H_2O)_{BDL} / (f_{H2O})_{BDL} \\ &= 261.554\ g / 0.8674_4 \\ &= 301.524\ g. \end{aligned} \tag{4-1}$$

The weight of the dry C-108 heel solids remaining at the end of the bulk density measurement can be calculated as:

$$\begin{aligned} Wt_{Dry\ C\text{-}108\ Solids} &= Wt_{Sample + BDL} - Wt_{BDL} \\ &= 706.304\ g - 301.524\ g \\ &= 404.780\ g. \end{aligned} \tag{4-2}$$

The total volume occupied by the BDL in the filled pycnometer (assuming all volumes are additive) is:

$$\begin{aligned} Vol_{BDL} &= Wt_{BDL} / D_{BDL} \\ &= 301.524\ g / 1.119_7\ g{\cdot}mL^{-1} \\ &= 269.290\ mL. \end{aligned} \tag{4-3}$$

The D_{BDL} used in Equation 4-3 was the result of a direct measurement on a subsample of the BDL performed in the 11A hot cells (Section 4.1.3).

The total volume occupied by the dry, undissolved C-108 heel solids is:

$$\begin{aligned} Vol_{Dry\ C\text{-}108\ Solids} &= Vol_{Pycnometer} - Vol_{BDL} \\ &= 478.68\ mL - 269.290\ mL \\ &=209.390\ cm^3. \end{aligned} \tag{4-4}$$

The calculated density of the dry, undissolved C-108 heel solids at the conclusion of the bulk density measurement is then simply:

$$\begin{aligned} D_{Dry\ C\text{-}108\ Solids} &= Wt_{Dry\ C\text{-}108\ Solids} / Vol_{Dry\ C\text{-}108\ Solids} \\ &= 404.780\ g / 209.390\ mL \\ &= 1.933_1\ g/cm^3. \end{aligned} \tag{4-5}$$

This value is the average density of the dry solids in the C-108 heel composite that were not dissolved at the conclusion of the D_{Bulk} measurement. As would be expected, the dry solids density is somewhat greater than the 1.884 g/mL bulk density of the damp composite. The relative closeness of the two values is also not surprising given (a) the small quantity of ISL present in the composite and (b) the presence of sufficient fine-grained material so that the test composite contained little void space.

It has been shown that the C-108 heel solids were a mixture of several solid phases. The two dominant phases were natrophosphate with an average density of 1.72 g/cm^3 and gibbsite [$Al(OH)_3$] with a density of 2.43 g/cm^3. Smaller quantities of trona with a density of 2.13 g/cm^3 and lepidocrocite (FeOOH) with a density of 3.97 g/cm^3 are also believed to have been present in the composite solids. The chemical analysis data (Tables 3-1 and 4-1) suggest that, at the conclusion of the D_{Bulk} measurement, these phases may have been present in the remaining heel solids in a ratio, by weight, of—

111 parts natrophosphate : 63 parts gibbsite : 4 parts trona : 1 part lepidocrocite.

The calculated nominal density of such a mixture would be 1.93 g/cm^3. While the relatively close agreement between this value and the measured dry solids density is probably fortuitous, it does suggest that the numerous assumptions and approximations required in the dry density calculations were reasonably correct or at least internally self-correcting.

The data in Table 4-1 also indicate that the 301.5 g of final BDL contained 39.97 g of dissolved solids. If the 13.57 g of water associated with natrophosphate and trona is added to this value, then the table values suggest that up to 53.54 g of the solids in the initial composite may have dissolved during the creation of the BDL. However, combining this value, the dry solids weight calculated for the composite in Equation 4-2, and the weight of unassigned water in the composite returns a total composite weight of 488.3 g. This value is too great as it would require the composite ISL to have been essentially pure water. If the composite ISL contained 29.96 g of water and 9.99 g of dissolved solids, then dissolution of 43.6 g of the composite solids would have been sufficient to create the BDL. The 75 wt% H_2O content implied would seem to be a reasonable upper limit for the water content of the composite ISL.

4.3 COARSE SIEVING

At the conclusion of the bulk density measurement, the pycnometer jar contained all the remaining C-108 heel solids and the ≈133 g (≈119 mL) of BDL that could not be removed without disturbing the solids. The next test operation performed was a separation of the C-108 heel solids into >1/4-in. and <1/4-in. size fractions by sieving.

4.3.1 Apparatus

A 3-in. diameter, stainless steel, 1/4-in. mesh test sieve was procured for the coarse sieving.

The inner diameter of the neck of a 400-mL wide-mouth jar (the sieve receiver jar) was widened slightly so that the bottom of the 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 sieving liquid on the solids to facilitate the sieve separation.

4.3.2 Coarse Sieving Operation

4.3.2.1. Solids Separation. At the conclusion of the bulk density measurements, the sieve receiver jar contained ≈4.2 g of very fine C-108 solids that had settled from the BDL. These solids were retained in the sieve jar. The 1/4-in. test sieve was placed in the mouth of the receiver jar. The small wash bottle was filled with BDL.

Portions of the composite solids sufficient to cover about half of the wire mesh were then transferred from the pycnometer jar to the sieve mesh. The solids were gently manipulated on the sieve mesh with a spatula while periodically directing a stream of BDL on them from the wash bottle. After all the <1/4-in. solids had been washed into the receiver jar, each portion of >1/4-in. solids was transferred to a 250-mL jar. This process was repeated until, to the extent achievable, all solids had been removed from the pycnometer jar.

Due to the high surface tension of the relatively dilute BDL and ergonomic restrictions imposed by performing the coarse sieving with hot cell manipulators, a small portion of <1/4-in. solids were included in the >1/4-in. fraction. Also, a small amount (≈5 mL) of the sieving BDL was lost during the sieve separation due to misdirection of rinsing streams from the wash bottle.

After the composite solids had been washed through the 1/4-in. sieve, all the remaining, unused BDL was transferred to a CSL bottle. A 33.02 g portion of water was used to rinse the pycnometer jar, wash bottle, and test sieve. The rinse water was captured in the receiver jar with the <1/4-in. solids and sieve liquid. These operations were completed near the end of the work shift on July 27, 2009. The contents of the receiver jar were mixed by gently stirring with a spatula. Then both the receiver and the >1/4-in. solids jars were capped and set aside.

Subsequent weighing determined that 0.40 g of air-dry C-108 solids had not been recovered from the pycnometer jar. Approximately 0.67 g of air-dry residue was retained on the test sieve.

4.3.2.2. Coarse Sieving Liquid. At the start of the work shift on July 28, 2009, all the free liquid that had drained to the bottom of the >1/4-in. solids jar (≈6.1 g) was removed with a pipette and added to the CSL bottle.

The supernatant liquid in the receiver jar was completely clear. All the clear supernate that could be removed with a pipette without disturbing the settled solids was drawn off and added to the CSL bottle. The remaining sample of <1/4-in. solids was then stirred with a spatula and discovered to be more fluid than desired to facilitate representative subsampling.

Additional liquid was removed from the <1/4-in. solids by transferring ≈30 mL of liquid and fine solids from near the bottom of the receiver jar to a 50-mL centrifuge cone with a pipette.

Centrifuging the cone contents for 10 minutes clarified the CSL. The clear liquid was added to the CSL bottle. This process was repeated again and then the centrifuged solids were transferred back to the receiver jar using the minimum volume of CSL required. Approximately 30-40 mL of additional CSL was removed from the <1/4-in. solids in this manner.

The final contents of the CSL bottle were thoroughly mixed. The density of the CSL was then measured by filling and weighing a 25-mL, Class A volumetric flask (volume 25 ± 0.03 mL at 20 °C). The measured density of the CSL was 1.118_5 g/mL at 27.6°C.

Two subsamples of the CSL were retrieved: one (S09R000148) for submission to the laboratory for physicochemical analysis (Tables 3-1 and 4-2) and one for measurement of wt% dissolved solids. The wt% dissolved solids sample was dried to constant weight at 90 °C. The final 1.556 g of dry residue represented 13.17 wt% of the original 11.818-g sample (i.e., the CSL was 86.83 wt% H_2O).

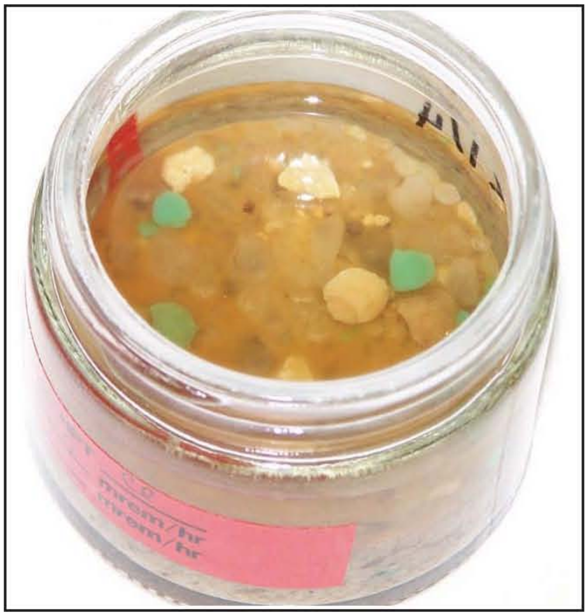
4.3.3 Coarse Sieving Results

4.3.3.1. Primary Coarse Sieving Results. The total weight of the >1/4-in. C-108 heel solids separated from the test composite was 90.940 g. A small portion, 1-2 mL, of water was added back to the >1/4-in. solids jar. The jar and solids, identified as sample S09R000162, were then transferred to APD personnel for crush (Section 4.5) and caustic dissolution (Section 7) testing. A photograph of the >1/4-in. C-108 heel solids is presented in Figure 4-1(b).

The weight of dry, <1/4-in., C-108 heel solids remaining in the receiver jar at the end of the coarse sieving operation can be estimated using the following gravimetric bookkeeping:

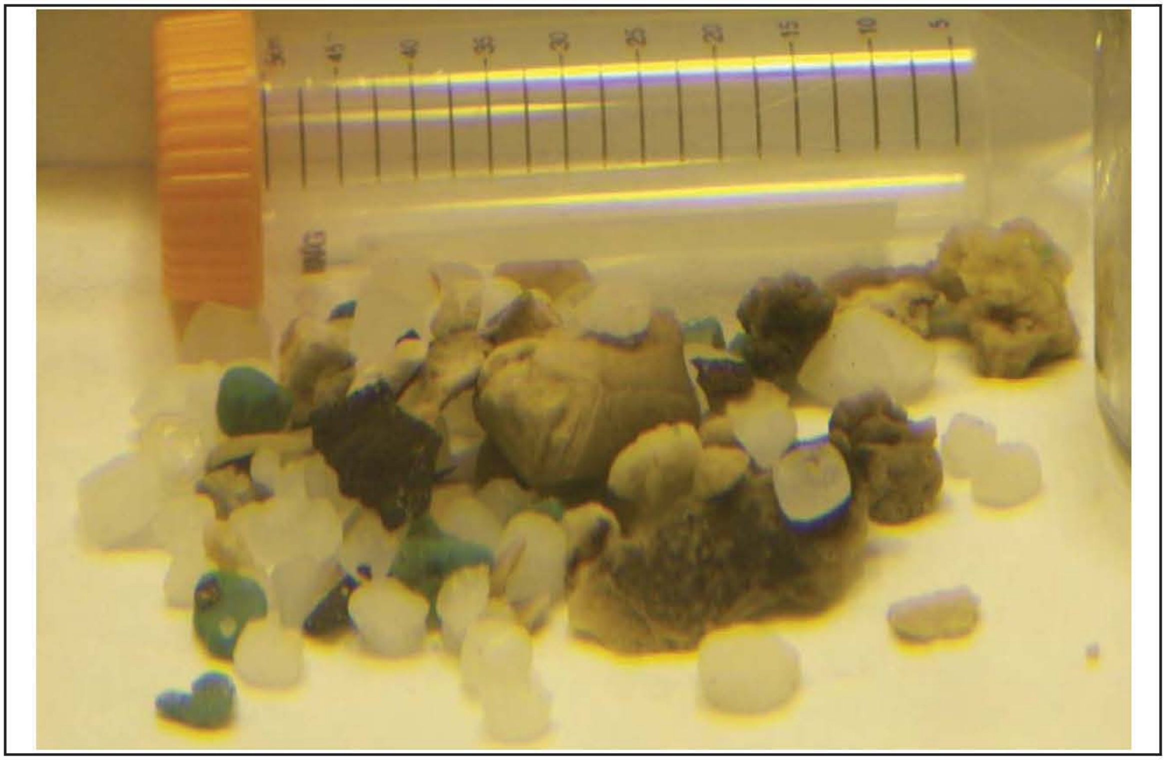
	Total Sample (g)	Total Solids (g)	Total Liquid (g)	Dissolved Solids (g)	H_2O from Solids (g)	Other H_2O (g)
After D_{Bulk} measurement	706.304	404.780	301.524	39.970	13.571	247.983
BDL samples removed	—		-18.921	-2.508	-0.852	-15.561
Before coarse sieving	687.383	404.780	282.603	37.462	12.719	232.422
<1/4-in. solids lost	—	-1.070	—	—	—	—
Estimated losses of BDL	—	—	-6.066	-0.804	-0.273	-4.989
Rinse water added	—	—	+33.016	0	0	+33.016
>1/4-in. solids recovered	—	-90.940	—	—	—	—
Interim <1/4-in. solids and diluted BDL	622.323	312.770	309.553	36.658	12.446	260.449
<1/4-in. solids and CSL	622.323	306.923	315.400	41.526	13.426	260.449

Figure 4-1. C-108 Heel Composite Solids After Coarse Sieving



(a) Left. Sample S09R000151. Subsample of <1/4-in. solids taken for particle size analysis (the diameter of the mouth of the jar is 5 cm)

(b) Below. Sample S09R000162. Total mass of air-dry >1/4-in. solids separated from heel composite (the divisions on the volume scale on the centrifuge cone are 5 mm apart)



The external appearance of the wet <1/4-in. solids (after the subsampling described in Section 4.4) is shown in Figure 4-1(a).

Of the total 398.93 g of dry C-108 heel solids accounted for after the coarse sieving operation, 22.80 wt% (90.94 g) were >1/4-in. in size and 77.20 wt% (307.99 g) were <1/4-in. in size.

4.3.3.2. Overall Results After Coarse Sieving. Subtracting the 398.93 g of C-108 heel solids remaining after the coarse sieving operation from the 404.87 g remaining after the D_{Bulk} measurement shows that an additional 5.85 g of heel solids were dissolved during the coarse sieving. While the total weight of solids dissolved during the D_{Bulk} measurement is unknown, it is believed to have been <44 g (Section 4.2.2). Given that the interim CSL was only an ≈12% dilution of the BDL, the much smaller composite weight loss is not surprising.

Overall, 251.0 g of water were added to the test sample through the D_{Bulk} and coarse sieving operations. This represents a 0.51 part water to 1 part composite (w/w) water wash of the starting C-108 heel test composite. It is estimated that <49.8 g of the initial composite solids were dissolved as a result of the *de facto* water wash.

In general, the partitioning of the initial C-108 heel solids composite into liquid and solid fractions through this point in the test program can be summarized as follows:

Initial wt of the test composite	488.28 g	100.0 wt%
Wt of >1/4-in. solids after coarse sieving	90.94 g	18.6 wt%
Calculated wt of <1/4-in. solids after coarse sieving	307.99 g	63.1 wt%
Calculated wt lost from initial composite	89.35 g	18.3 wt%

The estimated 89.35 g of the initial composite lost through the end of the coarse sieving includes the ISL originally present in the composite and composite solids dissolved during the density measurements and sieve separation.

4.4 SUBSAMPLING

After the final CSL removal on July 28, the contents of the sieve receiver jar weighed 381.731 g comprised of 306.923 g (80.40 wt%) of <1/4-in. C-108 heel solids and 74.808 g (19.60 wt%) of interstitial CSL. The water content of the CSL was measured as 86.83 wt% H_2O (Section 4.3.2.2). Thus, the interstitial CSL in the wet <1/4-in. solids contributed 64.959 g (17.02 wt%) of water to the wet solids.

A 15.910-g subsample (S09R000149) of the wet, <1/4-in. solids was transferred to a sample vial for determination of wt% H_2O. The subsample was immediately transferred to an oven and dried to constant weight at 90 °C. The subsample lost 5.797 g or 36.44 wt% upon drying. This scales to a loss of 139.088 g of water from the entire wet <1/4-in. sample. The additional 74.129 g of water above that from the CSL is assumed to be hydration water of undissolved natrophosphate and trona. This solids-associated water represents 19.42 wt% of the wet solids and 24.15 wt% of the dry solids.

On July 30, the 365.821 g of wet, <1/4-in. heel solids remaining in the receiver jar were subsampled for the additional characterization and dissolution tests. The sample identifications and the weight of the wet <1/4-in. solids distributed to each are summarized in Table 4-2.

The weight of dry <1/4-in. solids in each sample (0.8040 × Wt wet solids) is also shown in Table 4-2. These samples were taken before the large quantity of highly hydrated salts in the test composite was identified. The 36.44 wt% H_2O measured in the wet solids was used to determine sample weights that would contain target weights of dry solids. Since the water content of the CSL in the wet solids was actually only 17.0 wt%, the weights of the dry <1/4-in. solids transferred to the samples was 38.5 % larger than intended.

Finally, the weight of extra water added to samples S09R000151 and -157 through -160 is shown in the final column of Table 4-2. The target tests for these samples required extensive direct contact in analytical fume hoods. The extra water was added to reduce the radiological dose of the samples. Again, the water additions were planned and made before it was known that the <1/4-in. solids would still contain very significant quantities of water soluble salts.

Table 4-2. Subsamples of Wet <1/4-in. Heel Solids

Laboratory Sample Number	Test	Wet <1/4-inch Heel Solids (g)	Dry <1/4-inch Heel Solids (g)	Extra H_2O Added (g)
S09R000149	Wt% H_2O	15.910	—	—
S09R000151	Particle size analysis	46.659	37.515	13.4
S09R000152	Solid phase characterization	3.842	3.089	0
S09R000157	Settling Test: 1 wt%-Slow	9.267	7.451	5.7
S09R000158	Settling Test: 1 wt%-Fast	9.182	7.383	5.5
S09R000159	Settling Test: 5 wt%-Slow	47.790	38.425	12.2
S09R000160	Settling Test: 5 wt%-Fast	48.317	38.848	13.5
S09R000150	Caustic Dissolution	225.022	180.920	0

The total weight of wet <1/4-in. solids transferred to the subsamples was 165.057 g. Sample losses during the transfers were 1.429 g. The weight of wet solids remaining in the receiver jar was 198.795 g. To maximize the quantity of solids available for dissolution testing, the heel composite sample and the sample of the wet <1/4-in. solids that had been dried at 90 °C to determine wt% H_2O were reconstituted and added to the contents of the receiver jar. The wt% H_2O samples were reconstituted by adding weights of water equal to the measured weight losses on drying. The final total weight of the wet <1/4-in. solids was 225.022 g. The wet solids, identified as sample S09R000150, contained 180.92 g of dry <1/4-in. solids. The entire sample was transferred to APD personnel for caustic dissolution testing.

4.5 CRUSH TESTING

4.5.1 Apparatus

Four representative pieces of the visually identified main mineralogical components (black, clear, green, light-brown) were chosen for the crush testing. The test was designed as a purely qualitative identification of the hardness and was performed in hot cell 11A-5. The tools of choice were mortar and pestle.

4.5.2 Crush Testing Results

All four specimens could be easily crushed with the mortar and pestle. The natrophosphate was the hardest particle and the green phase (Ni-Al-phosphate) was the softest material. The black non-crystalline iron phase was hard, but crushed easier than the natrophosphate crystals. The tan-colored gibbsite crystals were somewhere between the black and the green phase. One of the key observations was that the green material does not contain a host crystal with a Ni-rich coating, but is on a macroscopical scale entirely made of one phase.

The crush test results can be summarized as follows:

Specimen	Crush description
Tan (Gibbsite)	Felt like dry chalk
White (Natrophosphate)	Felt like hard sugar candy
Green (Ni-Al-phosphate)	Felt like moist chalk
Black (amorphous Fe-hydroxide)	Felt crunchy, then smeared

5.0 PARTICLE SIZE ANALYSIS

The particle size distribution (PSD) in the <1/4-in. C-108 heel solids was measured in two steps. A sample of the wet <1/4-in. heel solids was first separated into >600-µm and <600-µm fractions by wet sieving. The >600-µm solids were dried and weighed. The PSD in the <600-µm fraction of heel solids was then measured by a light-scattering-based technique.

5.1 PARTICLE SIZE SAMPLE

As described in Section 4.4, a 46.659-g portion (sample S09R000151) of the wet <1/4-in. heel solids was retrieved from the coarse-sieving receiver jar for particle size measurements. A 13.4-g portion of water was added to the sample jar immediately after retrieving the sample to reduce the radiological dose of the sample and to ensure that it would not dry out prior to the particle size measurements. The added water was not stirred into the wet solids. An over-the-top photograph of the particle size sample is provided in Figure 4-1(a). The composition of the particle size sample immediately after it was collected on July 30, 2009 was:

Dry <1/4-in. heel solids	37.515 g
Coarse sieving liquid	9.144 g
Added water	13.4 g

The 37.515 g of dry <1/4-in. solids in the particle size sample represented 12.2 wt% of the total mass of <1/4-in. solids estimated to be present after the coarse sieving operation.

5.2 FINE SIEVING

5.2.1 600-µm Sieving Operations

The solids in the particle size test sample were first separated into >600-µm and <600-µm size fractions by wet sieving. A 3-in. diameter, all stainless steel, U.S. Standard No. 30 sieve was used. The square apertures in this sieve are 600 ± 25 µm across with not more than 5% of the openings falling between 660-695 µm in dimension (ASTM E11, *Standard Specification for Wire Cloth and Sieves for Testing Purposes.*) A sieve holder was fabricated that allowed vacuum to be used to facilitate passage of liquid and <600-µm solids through the sieve mesh and to draw air over solids retained on the wire mesh to speed drying. The same holder channeled the liquid and solids passing through the sieve mesh directly into a 400-mL receiver jar.

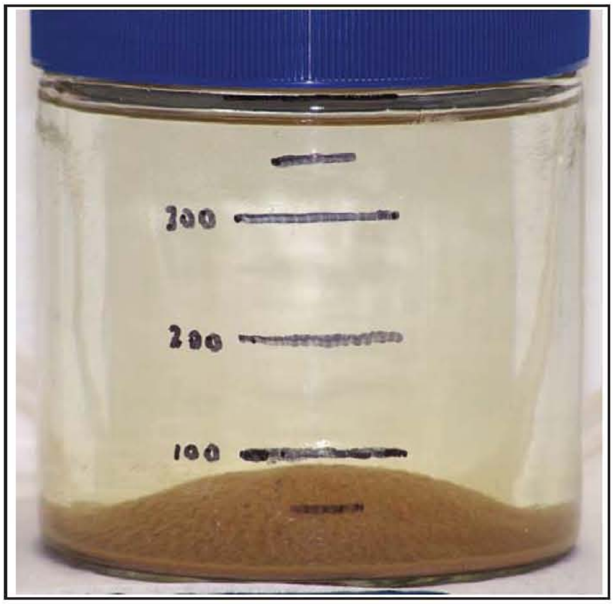
The sieve separation was performed on August 5, 2009. Particle size sample S09R000151, including all sample liquid, was transferred quantitatively onto the sieve mesh. Vacuum was then applied to the sieve holder apparatus. A low-pressure stream of water from a small wash bottle was directed on the test sample accompanied by very gentle manipulation of the solids with a small spatula. The separation took nearly 45 minutes due to relatively high solids loading on the sieve mesh but was visually effective. Rinsing and gentle stirring of the retained solids was continued until the water passing through the sieve mesh was clear. At this point, the receiver jar contained ≈400 mL of <600-µm solids, sample liquid, and water. Air was drawn through the >600-µm solids retained on the sieve mesh for ≈15 minutes, removing all visible free liquid from the solids and sieve surfaces. A photograph of the >600-µm heel solids at this point of the sieve separation is shown in Figure 5-1(a).

Figure 5-1. Particle Size Sample After 600-µm Sieve Separation

(a) Left. <1/4-in. and >600 µm heel solids from Sample S09R000151

(b) Bottom left. <600-µm solids before removal of sieving liquid

(c) Bottom right. <600-µm solids after removal of sieving liquid



The sieve was carefully removed from the sieve holder. An Al-foil cover was crimped onto the top of the sieve and then the sieve was inverted. The >600-µm solids now rested on the foil cover, were contained by the walls of the test sieve, and were covered by the sieve mesh. The package was transferred to a laboratory oven set at 89 °C at 13:30 on August 5. The receiver jar was capped and set aside to allow the suspended fines to settle.

The sieve+solids package was weighed at 09:30 and 13:00 on August 6. The weight of the >600-µm solids was 8.966 g at both weighings. An additional Al-foil cover was crimped over the bottom of the sieve (covering the wire mesh) and the entire package was placed in a slip-lid metal can. The entire sieve+solids+covers package was assigned sample number S09R000306 and placed in interim storage.

After settling overnight, the supernatant liquid in the fines receiver jar was totally clear on the morning of August 6 [Figure 5-1(b)]. Most of the sieve liquid was pipetted from the receiver jar and disposed of as waste. The <600-µm solids and remaining liquid [Figure 5-1(c)] were transferred to a 30-mL glass vial and set aside to resettle. Sample number S09R000151 was reassigned to the contents of this sample vial.

On August 19, the clear supernatant liquid was pipetted from the vial containing the <600-µm solids. The radiological dose of the remaining sample was too high to work further. After the contents of the vial were mixed thoroughly by stirring with a spatula, a subsample was removed using a large-bore disposable pipette and transferred to sample vial S09R000307. The inner walls of both vials were rinsed down with reagent water after the transfer. Sample S09R000151, containing the remaining <600-µm solids, was returned to interim storage. Sample S09R000307 was set aside to settle prior to light-scattering based PSD analysis.

5.2.2 Results of 600-µm Sieve Separation

As was the case for the density measurements, interpretation of the results of the 600-µm sieve separation are complicated by the presence of significant amounts of highly hydrated, water soluble species in the heel solids.

The oven-dry weight of the <1/4-in. and >600-µm solids, 8.966 g, is too small because it includes both evaporation of free water and dehydration of the solid salts. Examination of Figure 5-1(a) indicates the deviation could be significant. The clear to translucent solids visible in significant quantity in the photograph are natrophosphate which contains 48 wt% of easily removed hydration water.

In Section 4.4, it was shown that the dry <1/4-in. solids extant after the coarse sieving contained ≈24.1 wt% H_2O in the form of solid hydration water. If this proportion also holds for the >600-µm portion of these solids, then the air-dry <1/4-in. and >600-µm solids would have weighed 11.821 g. This would represent 31.51 wt% of the dry solids initially in the particle size sample.

However, recall that 13.4 g of extra water was added to the particle size sample after it was collected and that more than 300 mL of water was used to wash the <600-µm solids through the test sieve. It is likely that somewhat less than 37.515 g of dry <1/4-in. solids remained in the particle size sample before the 600-µm sieving was started and that a portion of the >600-µm solids were dissolved on the wire mesh during the sieve separation. No rational correction for either potential sample loss presents itself. However, it is reasonable to assume that the relatively large <1/4-in. and >600-µm solids would be significantly less affected by dissolution losses than the finer grained solids in the <1/4-in. solids.

The results of the 600-µm sieve separation of the <1/4-in. heel solids are, therefore, reported as:

	PSD Sample	Total <1/4-inch solids		Starting Composite	
Total wet	—	—	—	488.282 g	100.0 wt%
Total dry <1/4-in. solids	37.515 g	307.933 g	100.0 wt%	—	—
<1/4-in. and >600-µm solids	11.821 g	97.049 g	31.5 wt%	97.049 g	19.9 wt%
<600-µm solids	<25.694 g	<210.994 g	<68.5 wt%	<210.994 g	<43.2 wt%

5.3 LIGHT-SCATTERING MEASUREMENTS

The PSD within the <600-µm fraction of C-108 heel solids from the 600-µm sieve separation 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.

5.3.1 <600-µm Solids Particle Size Distribution Sample

The sample of <600-µm solids, S09R000307 (Section 5.2.1) sat undisturbed from August 19 to August 26, 2009. The completely clear supernatant liquid was then carefully removed with a pipette. The remaining solids were mixed by stirring with a spatula.

The actual specimens measured were very dilute slurries of small portions of the <600-µm solids in ≈210 mL of reagent water. Triplicate measurements were completed. Between each measurement, the contents of the parent sample (S09R000307) were remixed by stirring with a spatula.

5.3.2 Light Scattering Particle Size Distribution Measurements

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 the 210 mL of water circulating in the PSD analyzer. The minimum circulation pump and stirrer speeds consistent with reliable PSD measurements were utilized. The results of Run 1 represent specimen 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 were kept constant. The specimen slurry was circulated through the analyzer for 2 min prior to initiation of the second light-scattering measurement. The results of

Run 2 represent specimen 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 for 2 min prior to initiation of the third (and final) light-scattering measurement. The results of Run 3 represent specimen PSDs after exposure to moderate shear conditions.

The PSD measurements are described in detail in Attachment C where the specific measurement conditions in each of the three PSD runs are summarized in Table C-1.

The light-scattering-based PSD algorithms require indirect input of a complex refractive index for the sample solids. An estimate of the volume-weighted average refractive index of the solids possibly present in the C-108 heel solids after the 1/4-in. sieve separation is $N_p = 1.49 - 0.2i$. If all the natrophosphate and trona in the <600-µm solids were dissolved, the estimated average index would increase to 1.58 – 0.2i. An intermediate value of 1.54 – 0.2i was assigned to the solids in the PSD specimen slurries.

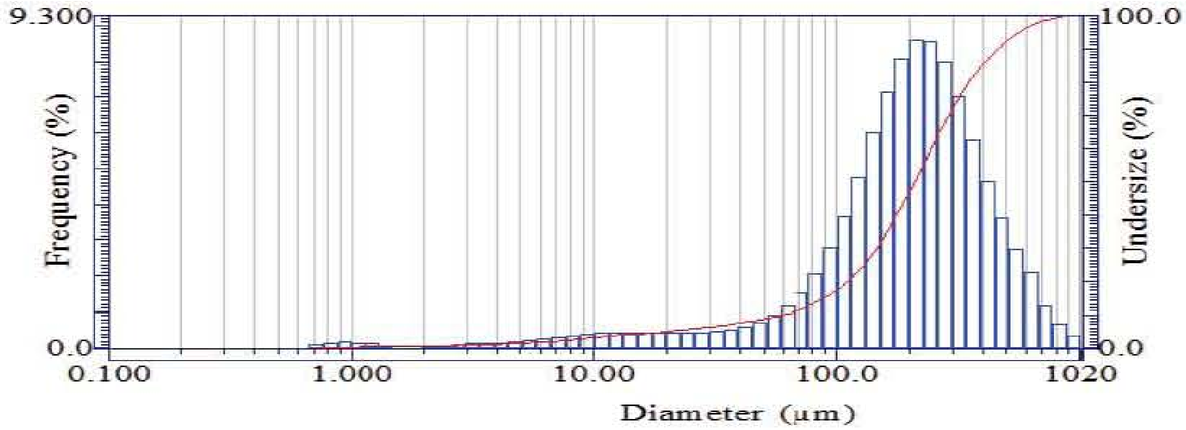
5.3.3 Histograms and Percent-Undersize Curves

The histograms and cumulative %-undersize curves displaying the results of the PSD measurements on the <600-µm C-108 heel solids are presented in Figures 5-2 and 5-3. The data files for corresponding PSD runs on the triplicate specimens were combined into 'average' data files. The PSD histograms, %-undersize curves, and numerical data derived from these average data files are presented in Sections 5.3.3 and 5.3.4. (Complete measurement results for each of the triplicate specimens are included in Attachment C.)

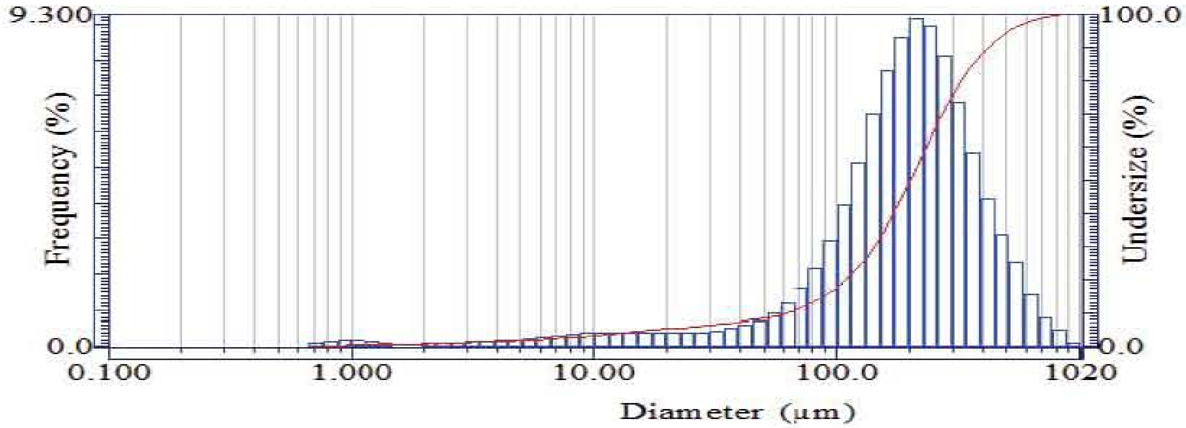
The light-scattering patterns measured by the PSD analyzer are directly related to the volumes of small particles and the cross-sectional area of larger particles. The default output of the analyzer is a PSD with frequencies of occurrence weighted according to the volumes of the scattering particles. These volume-based histograms and cumulative %-undersize curves are presented in Figure 5-2. 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 time required to introduce the PSD specimen into the analyzer sample tank.

Figure 5-2. Volume-based Particle Size Distribution Histograms and %-Undersize Curves for <600-µm Solids

Run 1 — Stir Speed: S2-3 Pump Speed: P4-5 Sonication: None Duration: 2.8 min



Run 2 — Stir Speed: S3 Pump Speed: P5 Sonication: None Duration: 3.4 min



Run 3 — Stir Speed: S3 Pump Speed: P5 Sonication: 2.0 min Duration: 3.6 min

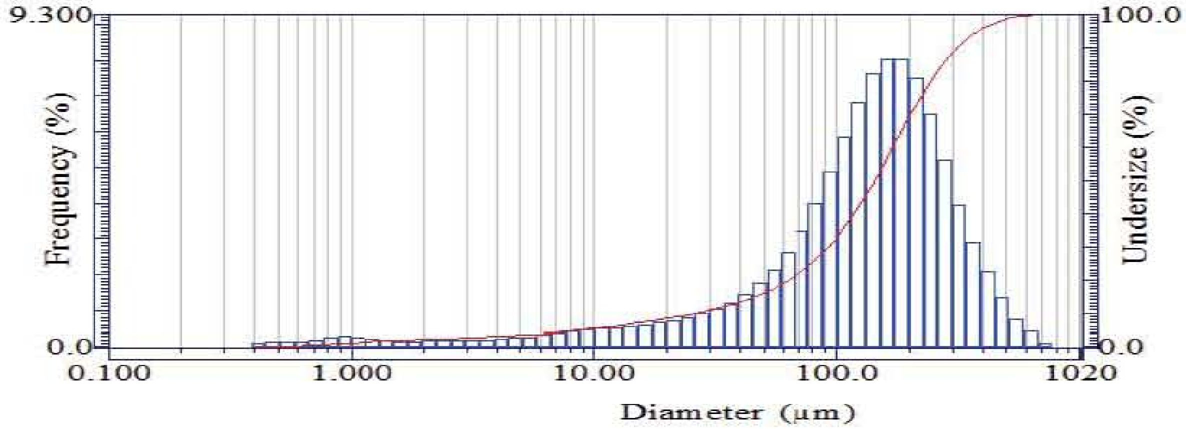
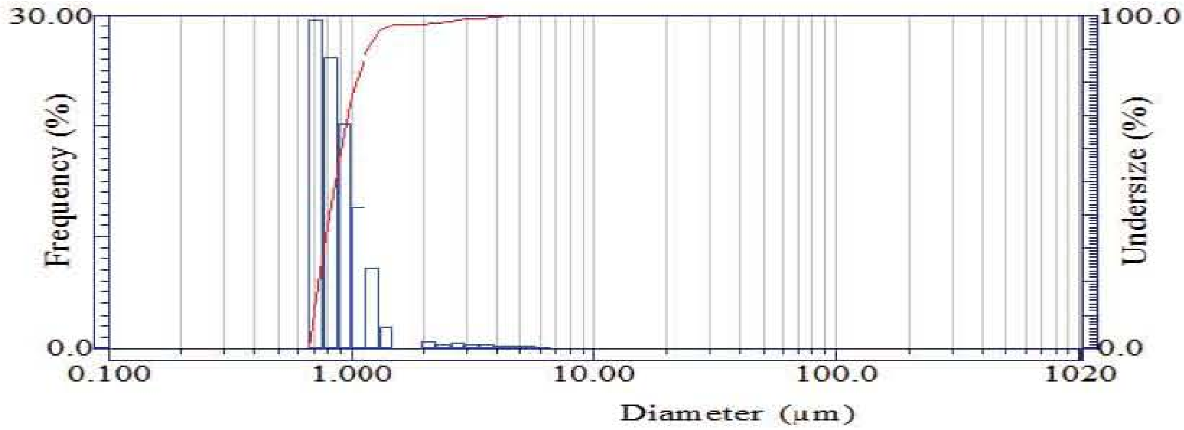
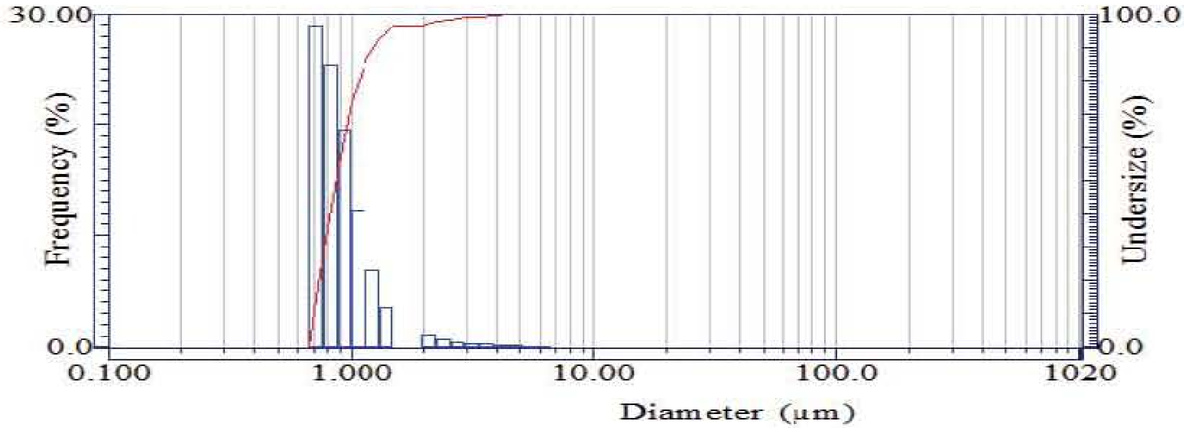


Figure 5-3. Number-based Particle Size Distribution Histograms and %-Undersize Curves for <600-μm Solids

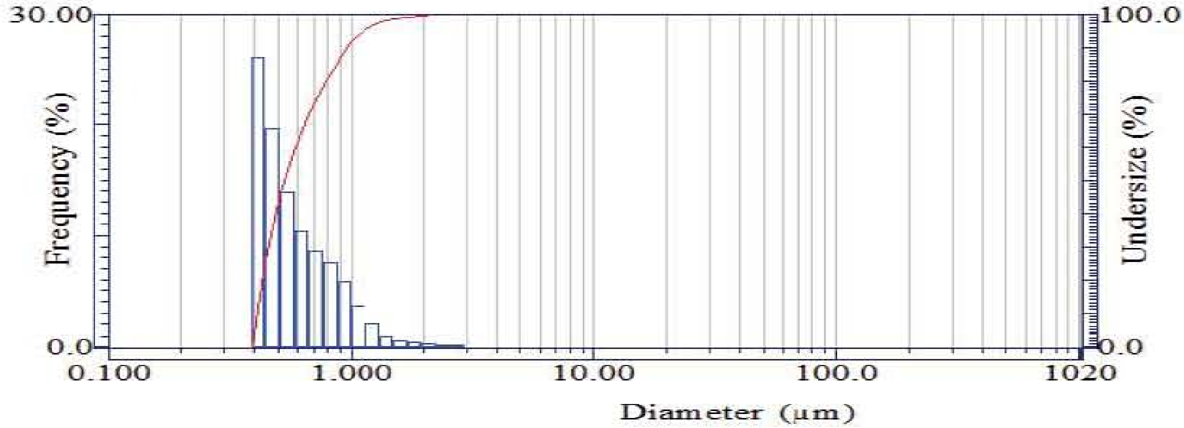
Run 1 — Stir Speed: S2-3 Pump Speed: P4-5 Sonication: None Duration: 2.8 min



Run 2 — Stir Speed: S3 Pump Speed: P5 Sonication: None Duration: 3.4 min



Run 3 — Stir Speed: S3 Pump Speed: P5 Sonication: 2.0 min Duration: 3.6 min



The analyzer software allows the primary volume-based 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 and then renormalizing the resulting distributions. The histograms and %-undersize curves for the recalculated number-based PSDs are presented in Figure 5-3 for comparison. The number-based data are displayed against the same particle diameter axis used for the volume-based data to facilitate comparison.

All the PSD data presented for the <600-µm C-108 heel solids are based on standard-form distributions (as opposed to the "sharp-form" alternative in the analyzer algorithms). Standard-form distributions are generally characterized by broad, poorly resolved features but are appropriate for the calculation of PSDs of samples with unknown composition and morphology and/or a wide range of particle diameters.

5.3.4 Cumulative Percent-Undersize Data

The particle diameters associated with five pre-selected points on the cumulative %-undersize curves describing the volume-based PSDs for the <600-µm C-108 heel solids are presented in Table 5-1. 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 5-1. Diameter versus %-Undersize for <600-µm C-108 Heel Solids

	Diameters of Particles (µm) at Volume-based %-Undersize Cut Points				
PSD Run	**10%**	**25%**	**50%**	**75%**	**90%**
1	62	131	211	319	462
2	62	128	202	298	425
3	27	80	144	222	317

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 distribution data for the <600-µm C-108 heel solids are presented in this format in Table 5-2.

Table 5-2. %-Undersize versus Diameter for <600-µm C-108 Heel Solids

	Vol% of Solids from Particles with Diameters Smaller than Cut Points				
PSD Run	**38 µm**	**75 µm**	**150 µm**	**300 µm**	**600 µm**
1	7.3	12.0	30.8	71.8	95.9
2	7.2	11.9	32.2	75.4	97.3
3	12.7	23.4	52.3	88.4	99.5

5.3.5 Particle Size Distribution Results for <600-µm Solids

The calculated PSDs for Run 1 and Run 2 are very similar. The upper 50% of the volume-based distributions show a small shift to smaller particle diameters but the general relationship between the large and small particle populations is essentially unchanged. The transmission of the analyzer light probes through the specimen slurries also suggests that the overall volumetric loading of particulates in the slurries and the relative proportions of large to small particles was essentially unchanged. A possible physical change that would produce these results would combine (a) the breakup/dissolution of very large, loosely bound agglomerates into smaller particles with (b) dissolution of smaller particulate solids at approximately the same rate.

After the sonication in Run 3, the calculated PSDs show a significant shift to smaller particle diameters. The histograms of Figures 5-2 and 5-3 and light transmission data both suggest that there is a slight increase in the number of light-scattering particles in the Run 3 slurries and that the relative proportion of small particles to large particles has increased. Again, a possible physical alteration that would produce these results would combine (a) the breakup/dissolution of moderately bound aggregates or relatively friable monolithic particles with (b) dissolution of smaller particles at a rate that is slightly less than their rate of formation.

The number-based histograms and %-undersize curves for the Run 1 PSDs in Figure 5-3 indicate that, on the basis of sheer numbers, the <600-µm C-108 solids are dominated by particles with diameters of ≈1 µm. These results were recalculated using cubic weighting factors and should be interpreted very cautiously. However, the indication that a large proportion, by number, of the <600-µm C-108 heel solids are relatively small in diameter is not inconsistent with other test observations.

5.4 COMBINED PARTICLE SIZE DISTRIBUTION

At the conclusion of the coarse sieving in the hot cells (Section 4.3.3.2), it was concluded that 18.6 wt% of the C-108 heel solids composite sample consisted of pieces of solids measuring >1/4 in.

After completion of the 600-µm sieve separation (Section 5.2.2), it was concluded that 19.9 wt% of the initial composite sample consisted of solids <1/4 in and >600 µm in size.

It would be desirable if the volume-based PSD data for the <600-µm solids in Section 5.3.4 could be taken to be equivalent to weight-based data. These results could then be combined with the results of the larger size fraction measurements to produce an overall weight-based particle size distribution for the C-108 heel solids composite. This would be possible if (a) the total weight of solids in the particle size sample remained constant during the 600-µm sieving and PSD measurements, (b) all the particles in the <600-µm C-108 heel solids had approximately the same density, (c) the density of the <600-µm solids was constant during the light-scattering based PSD measurements, and (d) the final density of the <600-µm solids was essentially the same as that of the solids in the initial test composite and those remaining after the coarse-sieving operation.

None of these conditions hold for the <600-µm C-108 solids. The parent particle size analysis subsample, S09R000151 (Section 5.1), is known to have contained solid phases with significantly different densities.

By the time the PSD sample (S09R000307) was collected, the particle size sample (S09R000151) had been subjected to a *de facto* water wash of approximately 9 parts water to 1 part sample. As the PSD slurries were prepared in the analyzer, the <600-µm solids (S09R000307) underwent an additional water wash of ≈200 parts water to 1 part sample solids. Which or how much of the particle size sample solids may have dissolved during the 600-µm sieve separation is unknown. For that matter, the amount of additional solids that may have dissolved as the dilute specimen slurries were prepared for the Run 1 PDSs is also unknown.

For the reasons above, the PSD results for the <600-µm solids are reported only as volume-based descriptors of the <43.2 wt% of the starting test composite consisting of solids <600 µm in size. The Run 1 results in Table 5-2 were first normalized to total 100% and then converted from cumulative %-undersize to particle diameter bin format. The results indicate that the <600-µm solids that persisted through the initial light-scattering-based PSD measurement had the following size composition:

Particle diameters (µm)	Vol%
<38	7.6
38 to 75	4.9
75 to 150	19.6
150 to 300	42.8
300 to 600	25.1
<600 µm	100.0

6.0 SETTLING TESTS

A set of two sequential settling tests was performed on four subsamples of the wet <1/4-in. C-108 heel solids (Section 4.4).

The first test in each set was a settling behavior test. Slurries of heel solids in water were prepared and then allowed to settle for 30 min. The qualitative appearance of the test slurries was observed and documented in a series of photographs.

The second test in each set was a settling rate test. These tests were performed on the same slurries of <1/4-in. C-108 solids in water that were observed in the settling behavior tests. The purpose of the rate tests was to characterize the weights of the C-108 heel solids that settled from well-mixed aqueous slurries containing 1 wt% and 5 wt% solids at rates exceeding 0.25 cm/s and 1.27 cm/s.

The settling tests were identified with four digit test codes: SC1F, SC1S, SC5F, and SC5S. The first two digits, SC, identify a **S**ettling test on a subsample of the <1/4-in. **C**omposite solids. The third digit identifies a test with nominal solids loading of **1** wt% or **5** wt%. The final digit identifies a **S**low target settling rate of 0.25 cm/s or a **F**ast target rate of 1.27 cm/s.

A similar series of settling tests was planned for the solid residues remaining after the caustic dissolution tests (Section 7.0). However, insufficient solids remained at the conclusion of the caustic dissolution tests to support desired physicochemical analysis and to perform any settling tests. Consequently, the settling tests on the dissolution residues were not performed.

6.1 SETTLING TEST PROCEDURE

6.1.1 Test Apparatus

A glass settling column was fabricated for 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. A settling distance scale, extending 43 cm down from the 0.0 mark, was inscribed on the outside of the column. The volume of the 44.5-cm working depth of the column was 544.1 mL (Section 8.2.3).

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 sample capture flask. The suction wand inserted into the settling column during aspiration allowed the time-to-depth of liquid removal to be controlled adequately.

6.1.2 Test Procedure

Each settling behavior/rate test set was performed as follows:

Settling Behavior Test

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 attached.
4. The column was rotated end-over-end several times until the sample solids appeared to be 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. Photographs of the settling column were taken at timed intervals over a period of 30 min.

Settling Rate Test

7. The 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 88.5% of the test slurry was removed by vacuum aspiration at such a rate that the 39.4-cm mark near the bottom of the column was reached at the time dictated by the test target settling rate:

   After 31 sec for a target rate of 1.27 cm/s (0.5 in./s)
   After 155 sec for a target rate of 0.254 cm/s (0.1 in./s)

   Care was taken that the liquid removal rate did not exceed the target solids settling rate at any time during the aspiration. The actual aspiration depth and time were recorded.
10. The liquid and suspended solids aspirated from the column were transferred to a labeled sample bottle and set aside.
11. The bottom screw cap was removed and the bottom of the column was oriented within a labeled, pre-weighed 250-mL sample jar.
12. The bottom plug was carefully dislodged from the column and captured in the sample jar along with ≈62 mL of sample solids (Recovered Solids or R-Slds) and test liquid. Recovery of the solids was facilitated by rinsing the inner walls of the column with water from a small squeeze bottle.
13. The plug was retrieved from the sample jar and any recovered solids were rinsed back into the jar with water. (The final volume of R-Slds + water was 170 to 190 mL.)

14. The sample jar containing the R-Slds was capped and set aside.
15. After settling for 5-6 days, the clear supernatant liquid in the sample jar was carefully pipetted off until the dose rate at the hood face approached 10 mR/hr. (The liquid removed was disposed of as waste.)
16. A photograph of the fast settling R-Slds was taken and then the jar was recapped and set aside.
17. After settling for an additional 2 days, all the remaining supernatant liquid was pipetted from the jar.
18. The sample jar containing the wet R-Slds was transferred to a laboratory oven and dried to constant weight at 38-42 °C.

The four sample bottles containing aspirated-water+fine-solids and the four sample jars containing dried R-Slds from the settling tests will be retained until released for disposal by the client.

6.2 SETTLING TEST SAMPLES

The four settling test samples were withdrawn from the receiver jar containing the wet <1/4-in. C-108 heel solids on July 30, 2009 (Section 4.4). These samples were taken at the same time and in the same manner as the PSD sample discussed in Section 5.1. As was the case for the PSD sample, extra water was added to each sample jar to preclude drying and to reduce radiological dose. The added water was not stirred into the sample solids. The identifications of the settling test samples and the quantity of <1/4-in. heel solids believed to have been present immediately after the samples were collected are summarized in Table 6-1.

Table 6-1. Identification and Quantity of Settling Test Samples

Settling Test	**SC1F**	**SC1S**	**SC5F**	**SC5S**
Sample S09R000-	**-158**	**-157**	**-160**	**-159**
<1/4-in. solids in sample	7.383 g	7.451 g	38.848 g	38.425 g
% of <1/4-in. solids in composite	2.397 wt%	2.419 wt%	12.613 wt%	12.476 wt%
CSL in sample	1.799 g	1.816 g	9.469 g	9.365 g
Added water in sample	5.5 g	5.7 g	13.5 g	12.2 g
Initial solids concentration in settling test slurry[a]	1.351 wt% 0.702 vol%	1.363 wt% 0.709 vol%	6.906 wt% 3.694 vol%	6.833 wt% 3.653 vol%

[a] Calculated concentrations assuming: $D_{Dry\ Slds}$ = 1.933 g/mL, D_{CSL} = 1.118 g/mL, D_{H2O} = 0.9975 g/mL, and Vol_{Total} = 544.1 mL.

The final row in Table 6-1 presents the calculated concentrations of solids present in the 544.1 mL test slurries of <1/4-in. solids in reagent water. These concentrations are based on the estimated compositions of the settling test samples following their collection on July 30. The settling tests were carried out on September 15-16, 2009. It is quite possible that some of the

<1/4-in. solids in the samples may have dissolved over the intervening 16-17 days. Since the samples sat undisturbed over this time period, it is not believed that large amounts of additional solids would have dissolved.

The nominal solids concentrations specified for the settling tests were 1 wt% and 5 wt% solids. The calculated concentrations in Table 6-1 exceed these values by ≈36%. As was previously noted, the settling test samples were collected before the large proportion of highly hydrated salts in the heel solids was known. The weights of the wet <1/4-in. solids samples collected were intended to include the required weights of dry solids; however, the sample weights were based on the measured wt%-H_2O in the wet solids. The wt%-H_2O values, which were based on oven drying at 90 °C, were too large and, as a result, the settling test samples were as well.

6.3 SETTLING BEHAVIOR TEST RESULTS

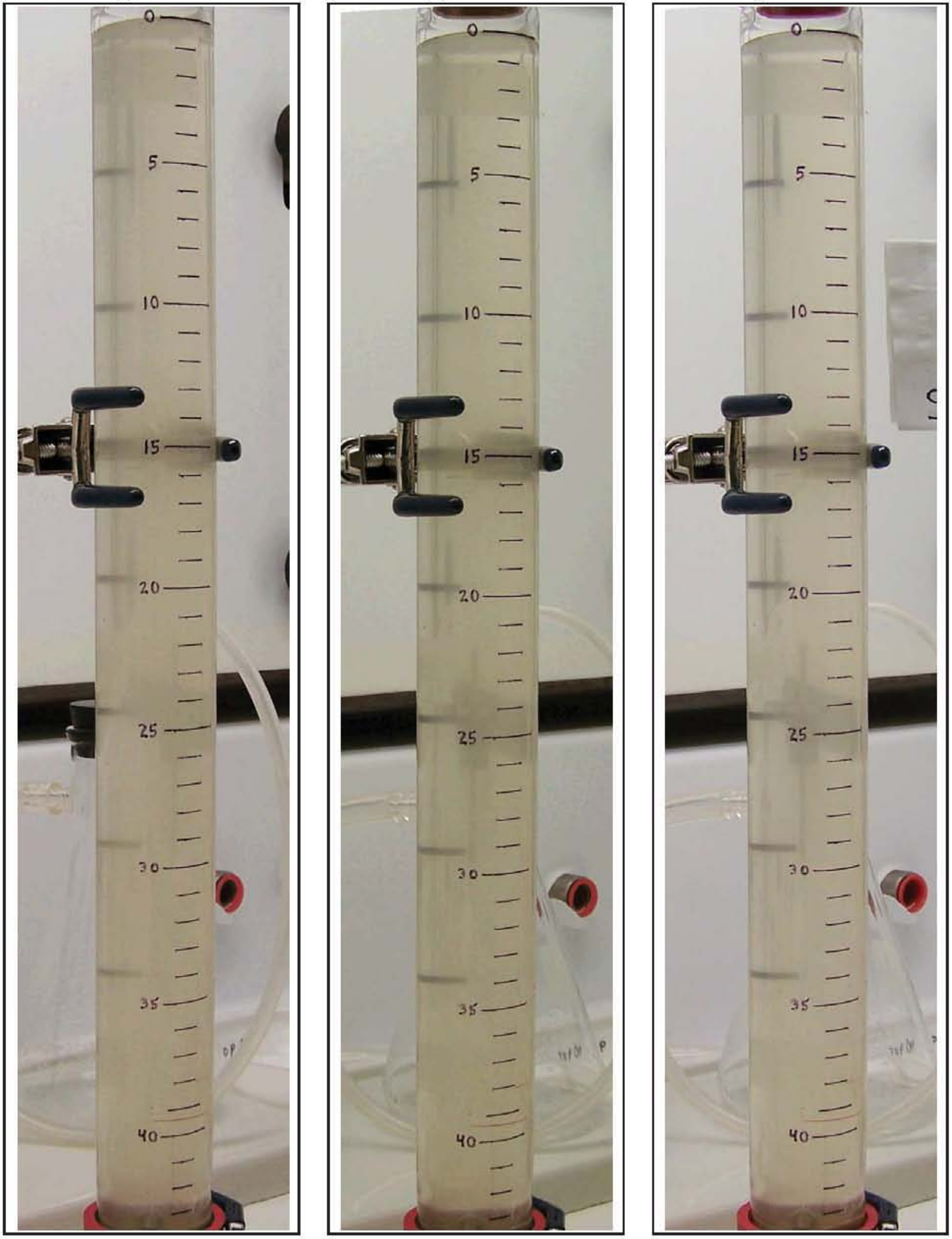
6.3.1 Settling Column Observations

The settling behavior was observed for all four settling test slurries. The slurries for two of the tests, SC1F and SC1S, contained ≈1.36 wt% <1/4-in. heel solids (≈0.71 vol%). The slurries for the other two tests, SC5F and SC5S, contained ≈6.87 wt% heel solids (≈3.67 vol%).

The liquid medium in all four tests was a dilute aqueous solution. This solution was formed by dilution of the liquid present in the settling test samples by the ≈500 mL of reagent water added at the beginning of each settling test set. The dissolution of additional sample solids during the settling tests is also possible but was not directly observed. No testing or analysis of the settling test liquids was performed. The average temperature in the hood where the tests were performed was 21.1 °C. The density of pure water at this temperature is 0.9980 g/mL; the viscosity (η) is 0.009779 g/cm·s. Slightly greater values of D_{Liq} = 1.018 g/mL and η = 0.01027 g/cm·s were assigned to the liquid medium in the settling tests.

A series of photographs of the settling column were taken at 3-min. intervals over a period of 30 min. during all four settling behavior tests. The observed behavior in the test slurries with approximately equal solids loading was indistinguishable. In Figure 6-1, a set of photographs taken during test SC1S are presented; Figure 6-2 presents a set taken during test SC5S. In both figures, the photographs portray the appearance of the settling column after settling times of 30 sec, 15 min, and 30 min.

Figure 6-1. Settling Behavior of ≤1.36 wt% of Heel Solids in Water



(a) 30 sec

(b) 15.0 min

(c) 30.0 min

Figure 6-2. Settling Behavior of ≤6.83 wt% of <1/4-inch Heel Solids in Water

(a) 30 seconds (b) 15 minutes (c) 30 minutes

In all the tests, the bulk of the coarse-grained solids settled very rapidly accumulating below the 39.4-cm slurry depth before the initial photographs were taken after 30 sec. Very little change in the settling slurries was observed for the remainder of the tests. Thick black marks were applied to the side of the settling column opposite the settling distance scale at 5-cm intervals. In Figure 6-1, the appearance of these marks through the column after 30 min. suggests that the overall turbidity of the more dilute slurry has decreased and that the turbidity increases uniformly with depth. The same observations applied to the more concentrated slurry in Figure 6-2 though, to the unaided eye, these slurries remained nearly opaque after 30 min. No distinct interface between the settling solids and a clear supernatant liquid formed (or appeared to be forming) in any of the tests and there was no visible evidence of any flocculation.

6.3.2 Observation of Removed Slurries

A settling rate test followed each settling behavior test with only a remixing of the test slurry intervening. After the appropriate settling times, the upper 482 mL of test slurry was removed from the column in each test and transferred to a 500-mL bottle. After all required rinses, the volume of test slurry removed in each test was very near 500 mL. The depth of the slurries in the bottles was ≈13 cm. After the settling rate tests were completed, these slurries of fine, slow-settling heel solids in dilute aqueous solution were remixed and their settling behavior observed.

After 1 hr., a layer of settled solids was visible on the bottom of each bottle. After 4 hr., the layer of settled solids in each jar had very nearly reached its maximum thickness. Over the following 20 hr., the turbidity of the liquid in each bottle decreased very slowly; in each bottle, turbidity increased fairly uniformly with depth. After 24 hr., only slight to moderate haziness remained in each bottle and the top 1-3 cm of supernatant liquid was completely clear. After 40 hr.,, the supernatant liquid in each bottle was a completely clear liquid with a very pale yellow tint. The settled solids were off-white to light-brown in color and loosely consolidated. The settled solids appeared to be very fine-grained with no discrete particles visible.

6.3.3 Summary of Settling Behavior

In general, after the initial very rapid settling of the larger solid particles, the residual test slurries exhibited the settling behavior of relatively dilute slurries of small-sized and/or low-density solids with no, or only relatively weak, interparticle interactions. The distinct interface between settling solids and clear supernatant liquid characteristic of hindered settling[5] was not observed in any test and there was no visible evidence of flocculation.

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[5] The terms ‘hindered settling’ or ‘mass subsidence’ generally describe the settling of an initially homogeneous and relatively dense 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.

The terminal settling velocity ($V_{Settling}$) of a spherical particle in a homogeneous fluid of infinite extent can be calculated as

$$V_{Settling} = \{[4 \cdot g \cdot (D_{Sld} - D_{Liq}) \cdot d] / (3 \cdot C_D \cdot D_{Liq})\}^{1/2} \qquad (6\text{-}1)$$

where

$$C_D \equiv \text{Coefficient of drag} = (24/N_R) + (3/N_R^{1/2}) + 0.34 \qquad (6\text{-}2)$$

and

$$N_R \equiv \text{Reynolds number} = (\varphi \cdot V_{Settling} \cdot d \cdot D_{Liq}) / \eta \qquad (6\text{-}3)$$

with

$g \equiv$ acceleration due to gravity = 980.7 cm/s^2
$D_{Sld} \equiv$ density of solid phase
$D_{Liq} \equiv$ density of liquid phase = 1.018 g/cm^3
$\varphi \equiv$ shape factor for solid particles = 0.85
$d \equiv$ particle diameter (cm)
$\eta \equiv$ viscosity of liquid phase = 0.0103 g/cm·s

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

Solving Equations 6-1 through 6-3 over a range of D_{Sld} values encompassing those of the solids believed to be present in the C-108 heel solids produces estimates of the settling rates of the heel solids. The D_{Liq} and η values for the liquid medium are assigned values that are 2% and 5% greater, respectively, than those for pure water at the test temperature. The particle shape factor $\varphi = 1.0$ for a spherical particle; a value of 0.85 is used to account for irregularities in the heel solid particles. The results of these calculations are summarized in Table 6-2.

Table 6-2. Terminal Settling Velocities of Solids of Varying Densities

	D_{Sld} (g/cm^3)			
d (µm)	**1.716** [a]	**1.933** [a]	**2.430** [a]	**3.970** [a]
2.3	0.62 cm/hr	0.82 cm/hr	1.26 cm/hr	2.64 cm/hr
9.4	9.94	13.0	20.1	41.9
37.5	2.62 cm/min	3.42 cm/min	5.26 cm/min	10.9 cm/min
75	10.2	13.3	20.3	41.3
150	0.63 cm/s	0.81 cm/s	1.22 cm/s	2.38 cm/s
300	2.08	2.62	3.77	6.80
600	5.51	6.72	9.14	15.1
1200	11.4	13.5	17.6	27.4
2400	19.8	23.0	29.4	44.1
4800	31.1	36.0	45.4	66.9
6350	36.8	42.5	53.4	78.4

[a] The density of natrophosphate = 1.716 g/cm^3; the calculated density of the dry heel solids = 1.933 g/cm^3; the density of gibbsite = 2.43 g/cm^3; and, the density of lepidocrocite = 3.97 g/cm^3

Examining the estimated settling velocities in Table 6-2, the observed settling behavior of the test slurries could be interpreted as indicating that the <1/4-in. C-108 heel solids are dominated by particles exceeding 75 µm in size but also include a minor portion of low-to-medium density solids with diameters <2 µm.

6.4 SETTLING RATE TESTS

The settling rate tests were intended to determine the quantity of <1/4-in. C-108 heel solids that settled from aqueous slurries in specified times. Slurries containing two different concentrations of solids, ≈1.36 wt% and ≈6.87 wt%, were prepared for the tests. The working depth of the settling column prepared for the tests was 44.5 cm. The target settling times were the times required for particulates starting at the surface of the aqueous slurries and settling at rates of 1.27 cm/s (0.5 in./s) or 0.254 cm/s (0.1 in./s) to reach a depth of 39.4 cm. In a perfectly executed test, the settling time would have been either 31.0 s or 155.1 s. The target solids were captured in the bottom 5.1 cm of the column, recovered, dried, and weighed.

6.4.1 Settling Rate Test Results

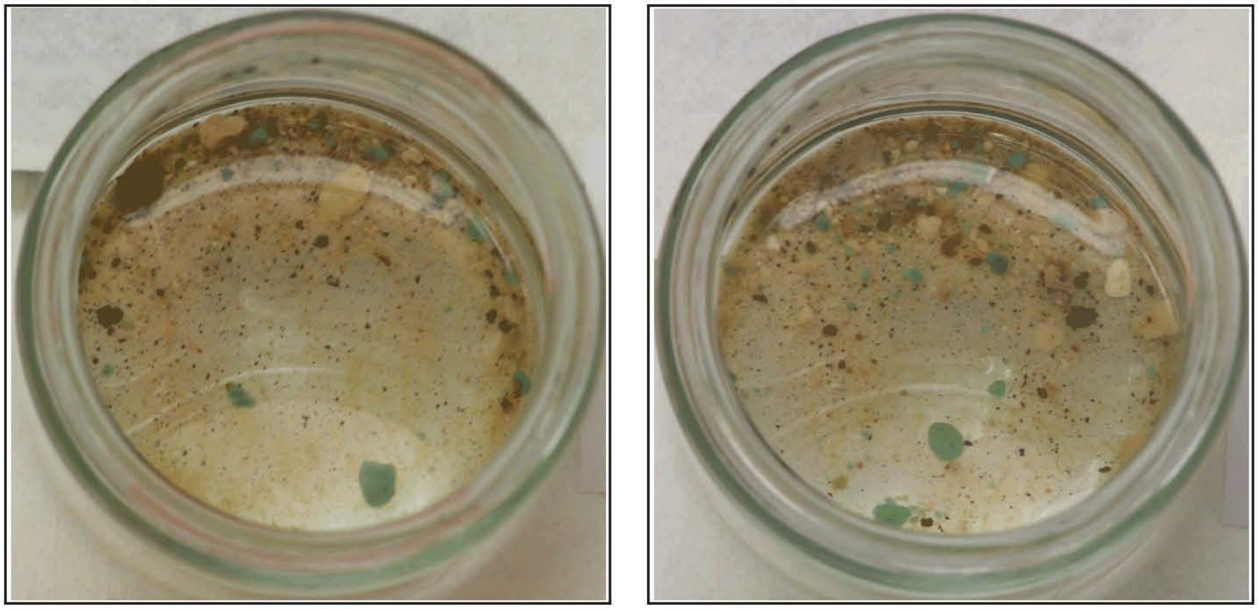
The physical appearance of the solids captured in the bottom 5.1 cm of the settling column in each settling rate test is shown in Figure 6-3. (These solids are referred to as Recovered Solids or R-Slds.) The samples had settled for 5-6 days and a portion of the supernatant liquid removed prior to taking these photographs. For comparison, the initial appearance of the settling test samples was identical to that of the particle size sample (S09R000151) shown in Figure 4-1 and would have contained portions of both the >600-µm and <600-µm solids shown in Figure 5-1.

Figure 6-3. Recovered Solids from Settling Rate Tests

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

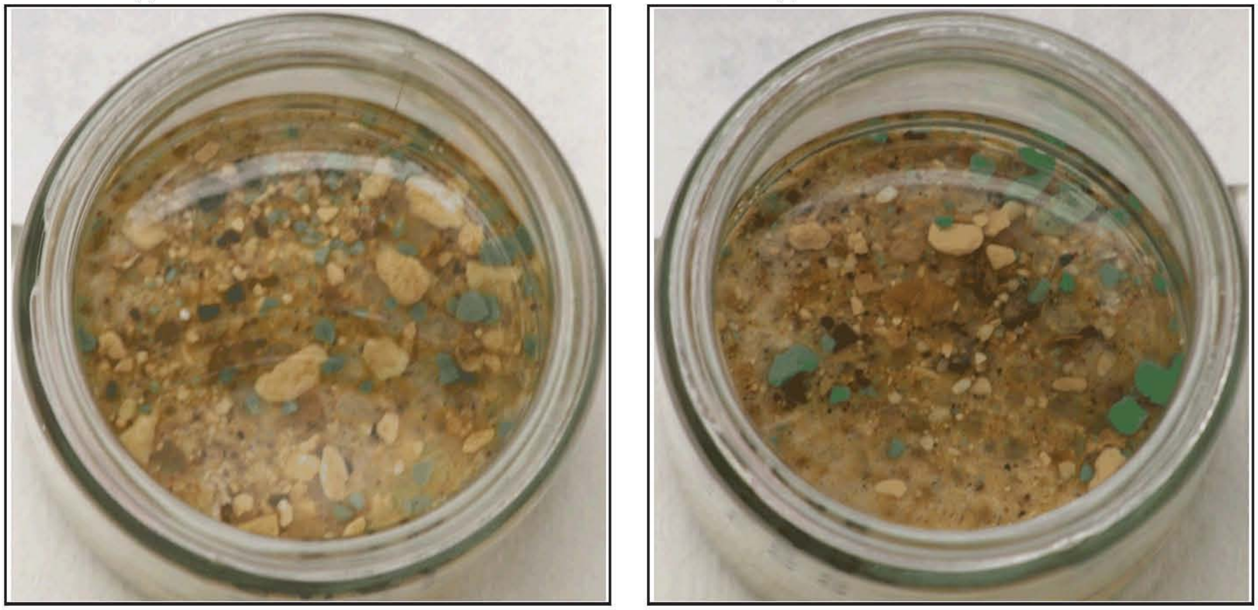
(a) SC1F: 1.35 wt% at 1.24 cm/s

(b) SC1S: 1.36 wt% at 0.25 cm/s



(c) SC5F: 6.91 wt% at 1.22 cm/s

(d) SC5S: 6.83 wt% at 0.26 cm/s



Quantitative observations recorded during the settling rate tests and the calculated primary test results are presented in Table 6-3.

Table 6-3. Settling Rate Test Results

	Settling Test			
	SC1F	SC1S	SC5F	SC5S
Maximum wt of solids in test sample	7.383 g	7.451 g	38.848 g	38.425 g
Maximum wt% of solids in test slurry	1.35 wt%	1.36 wt%	6.91 wt%	6.83 wt%
Depth of slurry removed	39.7 cm	39.6 cm	39.6 cm	39.8 cm
Elapsed settling time	32.1 s	158.4 s	32.4 s	156.0 s
Test settling rate	1.236 cm/s	0.250 cm/s	1.224 cm/s	0.255 cm/s
Wt of Recovered Solids	3.100 g	3.330 g	20.472 g	21.480 g
R-Slds as wt% of solids in test sample	41.99 wt%	44.69 wt%	52.70 wt%	55.90 wt%
Maximum wt of co-recovered Fines	0.466 g	0.473 g	2.086 g	2.064 g
Wt% R-Slds adjusted for Fines	35.68 wt%	38.34 wt%	47.33 wt%	50.53 wt%

The initial wt% R-Slds values in the seventh row of Table 6-3 are simply:

$$(\text{g R-Slds} / \text{g test sample solids}) \times 100. \qquad (6\text{-}4)$$

For the reasons discussed below, these values may be biased low.

6.4.1.1. Measurement Bias. The settling test samples were collected on July 30, 2009 and a small quantity of water was added to each sample at that time (as was discussed for the particle size sample, S09R000151, in Section 5.0). It is possible that some of the <1/4-in. heel solids originally present in the samples dissolved before the settling tests were performed on September 15-16. It is also possible that some of the solids were dissolved as the samples were introduced to the settling column and mixed with more than 500 mL of water while preparing the test slurries. Therefore, the solid weights reproduced in the first row of Table 6-3 may be too large.

An additional portion of reagent water, ≈120 mL, was used to rinse the R-Slds from the settling column into the collection jars. The R-Slds were immersed in this diluted settling liquid for 7-8 days. It is possible that some of the R-Slds were dissolved during this period and, therefore, that the R-Slds weights reported in the sixth row of Table 6-3 are too small.

Also, the weights of the R-Slds in the sixth row of Table 6-3 are oven-dried weights. As was previously discussed, the possible presence of significant amounts of highly hydrated salts in the solids could result in these weights being too small. This potential problem had been identified by the time the settling test samples were being processed. Oven temperatures ranged from 22-42 °C while the R-Slds were being dried. Weight loss was carefully monitored and oven time limited to the minimum required (9-12 hours). The reported R-Slds weights may still be too small; however, the drying process described should have minimized the potential loss of waters of hydration from the recovered heel solids.

6.4.1.2. Fines Adjusted Results. The R-Slds that were recovered, dried, and weighed in each settling rate test contained all solids that settled below the aspiration depth in the allotted settling time. The R-Slds also contained some of the sample solids that settled at slower rates because of their small size and/or low density (i.e., Fines) but were located below the surface of the slurry at the beginning of the test. The R-Slds also included all Fines that were already present in the bottom 4.7 to 4.9 cm of the settling column at the beginning of each test and were not displaced above the aspiration depth by the accumulation of the fast settling solids.

A rough estimate of the weight of Fines in the volume of test slurry below the slurry aspiration depth at the beginning of each settling test can be calculated. It is assumed that the concentration of Fines was relatively constant throughout the settling column over the relatively short time scales of the tests.

First, the projected weight of the R-Slds at the beginning of each settling test was determined by plotting [(wt R-Slds/wt test sample Slds)/(cm of slurry containing the R-Slds)] vs. settling time. The y-intercept was then converted back to a test specific R-Slds$_{T-0}$ value by multiplying by the R-Slds slurry depth and the weight of the total solids. The estimated weight of Fines in each centimeter of slurry at the beginning of a test was then calculated as

$$(\text{g test sample solids} - \text{g R-Slds}_{T-0}) \,/\, (44.5 \text{ cm}). \qquad (6\text{-}5)$$

The weight of Fines initially present in the R-Slds volume was then

$$(\text{g Fines/cm})_{T-0} \times (\text{cm of slurry containing the R-Slds}). \qquad (6\text{-}6)$$

These weights of co-recovered Fines are shown in the eighth row of Table 6-3. Since the weights of total solids actually present in the test samples may have been less that those shown in the first row of the table, these values are identified as maximum weights of co-recovered Fines.

The wt% R-Slds adjusted for the co-recovered Fines, presented in the ninth row of Table 6-3, are then

$$[(\text{g R-Slds} - \text{g co-recovered Fines}) \,/\, (\text{g test sample solids})] \times 100. \qquad (6\text{-}7)$$

As discussed in 6.4.1.1, the weights of solids in the settling test samples may have been smaller and the weights of R-Slds larger than the values in the first and sixth rows of Table 6-3. If so, the estimated weights of the co-recovered Fines from Equations 6-5 and 6-6 would be too large. The overestimation could be exacerbated for the ≈6.87 wt% slurries if some of the Fines were displaced out of the R-Slds volume by the relatively large mass/volume of fast settling solids (Figure 6-2 as compared to Figure 6-1). Of course, to the extent that the correction for co-recovered Fines is too large, the adjusted wt% R-Slds values would be too small.

These adjusted wt% R-Slds values still include the weights of Fines that settled below the slurry aspiration depth during the tests. However, given the varying particle sizes and densities present in the <1/4-in. heel solids, a defensible correction (even compared to the rough approximation of Equations 6-5 and 6-6) does not present itself.

The wt% values in the final row of Table 6-3 might be interpreted as the relative portions of <1/4-in. C-108 heel solids that would be predicted to settle to the bottom of a vessel in fixed times after being completely suspended in a dilute aqueous solution. The fixed settling times would be defined by the ratios of the depths of the slurries of suspended solids to targeted settling rates.

6.4.1.3. Comparison to Particle Size Results. Equations 6-1 through 6-3 can be evaluated to predict the minimum diameters of particles of varying densities that should have been recovered in the 0.254 cm/s and 1.27 cm/s rates used in the tests.

Settling Rate	D_{Sld} = 1.716 g/cm^3	D_{Sld} = 1.933 g/cm^3	D_{Sld} = 2.430 g/cm^3	D_{Sld} = 3.97 g/cm^3
0.25 cm/s	92 µm	81 µm	65 µm	45 µm
1.27 cm/s	222 µm	193 µm	154 µm	105 µm

For example, the settling equations indicate that if the heel solids had a density equal to that of natrophosphate, 1.72 g/cm^3, all particles with diameters >92 µm should have been captured in the R-Slds in the two –S tests targeting settling rates of 0.254 cm/s. All particles with diameters >222 µm should have been captured in the two –F tests targeting settling rates of 1.27 cm/s. (Of course, this assumes that the liquid density, the liquid viscosity, and the particle shape factor used in the calculations were reasonably close to the actual values.)

The particle size and settling test samples were collected at the same time and, presumably, originally contained similar relative proportions of solids of similar size distribution. The 600-µm sieve separation of the particle size sample indicated 31.5 wt% of the estimated solids present were <1/4-in. but >600 µm in size (Section 5.2.2). The light-scattering-based PSD measurement results (Section 5.3.4) suggest that capture of all solids with diameters >92 µm in the R-Slds would have required capture of ≈85% of the remaining solids. Capture of all solids with diameters >222 µm would have required capture of ≈45% of the remaining solids. If this is correct, the wt% R-Slds values in Table 6-3 would seem to be too small by significant margins. The apparent discrepancy grows larger as the assumed particle density increases.

A possible explanation of this apparent discrepancy would hold if the sample solids partially dissolved before or during the settling tests, particularly the <600-µm solids. The weights of >600-µm solids captured in the R-Slds might still be close to 31.5 wt% of the estimated total weights of solids originally present in the test samples. However, at the conclusion of the settling tests, the weights of the <600-µm solids might be significantly less than 68.5 wt% of the estimated original total solids weights. If so, smaller weights of the <600-µm solids might be required to include all particles with diameters greater than set cut points. If a slight decrease in the weights of the >600-µm solids also occurred, capture of weights of the <600-µm solids equivalent to 10-25 wt% of the estimated original total sample weights in the R-Slds would be sufficient to capture all solids larger than the calculated cut diameters.

Also, the relative quantity of water added to the <600-µm solids during the PSD measurements was much greater than that added during the settling tests. The measured PSDs in the <600-µm solids may have been shifted to larger particle diameters than those present in the settling samples. This would also have the effect of requiring addition of smaller weights of <600-µm solids in the settling test samples to the >600-µm solids present to capture smaller particle diameters.

6.4.1.4. Comparison of Low and High Concentration Test Slurries. There was no evidence of the retardation of settling rates due to high solids loading (or strong interparticle attractive forces) in the settling tests. A large portion of the settling test samples were composed of >600-µm solids which settled very rapidly (a few seconds). The concentration of the remaining solids in the test slurries was not great enough for hindered settling to be observed over the time frame of the tests. The absence of hindered settling is also supported by the observation that the relative increase in the wt% R-Slds collected in the 0.25 cm/s (–S) tests over that collected in the 1.27 cm/s (–F) tests was approximately the same for the ≤1.36 wt% and ≤6.87 wt% slurries.

The settling rate test results in Table 6-3 show that the wt% R-Slds (relative to the estimated original solids weights) was significantly greater for the two test slurries containing ≤6.87 wt% solids than for the two containing ≤1.36 wt% solids. There are two possible causes for this observation.

First, the samples used to prepare the test slurries containing ≤6.87 wt% solids were significantly larger than those used to prepare those containing ≤1.36 wt% solids. It is quite possible that the >600-µm solids were a larger (and more representative) weight fraction of the larger test samples resulting in larger wt% R-Slds values relative to those prepared with the smaller samples.

Second, it is quite possible that a relatively greater amount of sample solids was dissolved in the two low concentration slurries than in the two higher concentration slurries. The relative quantity of water added to the settling test samples was quite different. Preparation of the ≤1.36 wt% test slurries required addition of ≈58 parts water to 1 part sample; preparation of the ≤6.87 wt% slurries required ≈11 parts water to 1 part solids. By the time that drying began, ≈71 parts water to 1 part solids had been added to the R-Slds from the ≤1.36 wt% slurries; ≈13 parts water to 1 part solids had been added to the R-Slds from the ≤6.87 wt% slurries. The visual appearance of the test slurries in Figures 6-1 and 6-2 and of the R-Slds in Figure 6-3 lends some support to this hypothesis.

6.4.2 Summary Settling Test Results

The overall results of the settling rate tests are summarized in Table 6-4. In the table, the settling test results on the <1/4-in. heel solids samples have been normalized to the total weight of the original test composite. This was done by multiplying the

wt R-Slds – wt co-recovered Fines

from each test by the ratio of

(wt <1/4-in. solids in the test composite) / (wt <1/4-in. solids in the settling test sample).

The weight of <1/4-in. solids in the test composite was 307.993 g (Section 4.3.3).

The scaled-up Fines-adjusted R-Slds weights were then added to the weight of >1/4-in. solids determined to be present in the test composite, 90.940 g (Section 4.3.3). The sum of the solids weights were then converted to a wt% of the initial total weight of the test composite. For example, in settling test SC5F, the 90.94 g of >1/4-in. solids were combined with the normalized weight of 145.767 g of Fines-adjusted R-Slds. The resulting 236.71 g of solids is 48.5 wt% of the initial test composite weight. Therefore, settling test SC5F is taken to indicate that ≥48.5 wt% of C-108 heel solids would settle out of a slurry containing up to 10.9 wt% of the heel material in water in a time defined by a settling rate of 1.22 cm/s.

Table 6-4. Summary Results of Settling Rate Tests

Settling Test	**SC1F**		**SC1S**	
<1/4-in. Solids in test slurry	**≤1.35 wt%**		**≤1.36 wt%**	
Composite in slurry	**≤2.1 wt%**		**≤2.2 wt%**	
Test settling rate	**1.24 cm/s**		**0.250 cm/s**	
Test Composite	488.282 g	100 wt%	488.282 g	100 wt%
>1/4-in. Slds	90.940 g	≥41.1 wt%	90.940 g	≥42.8 wt%
<1/4-in. Slds: Fines adjusted R-Slds	≥109.881 g		≥118.096 g	

Settling Test	**SC5F**		**SC5S**	
<1/4-in. Solids in test slurry	**≤6.91 wt%**		**≤6.83 wt%**	
Composite in test	**≤10.9 wt%**		**≤10.8 wt%**	
Test settling rate	**1.22 cm/s**		**0.255 cm/s**	
Test Composite	488.282 g	100 wt%	488.282 g	100 wt%
>1/4-in. Slds	90.940 g	≥48.5 wt%	90.940 g	≥50.5 wt%
<1/4-in. Slds: Fines adjusted R-Slds	≥145.767 g		≥155.628 g	

7.0 CAUSTIC DISSOLUTION TESTING

Based on the initial characterization of the heel sample, only caustic dissolution was performed. The amount of Fe-bearing particles was considered too low for an acidic dissolution to be successful. Table D-1 provides the detailed information on mass quantities throughout the test runs.

7.1 APPARATUS

Caustic dissolution tests were performed in fifteen 50-mL conical (centrifuge) vials on a Labquake[6] Rotisserie. The rotation was set to 8 rpm.

Caustic was staged in the hot cell in 500-mL bottles as a 50% (w/w) sodium hydroxide solution.

7.2 CAUSTIC DISSOLUTION

7.2.1 Sample preparation

As discussed in Section 4.3.3, sample S09R000150 was used in the dissolution tests. This sample contained the entire 225 g of wet <1/4-in. heel solids remaining after all other sampling was completed. The general consistency of these solids can be seen in Figure 4-1 (after, of course, removing the >1/4-in. solids). Figure 4-1(a) is a photograph of one of the subsamples removed from the wet <1/4-in. solids. The fine-grained fraction was primarily used to identify weight losses during dissolution.

Sample S09R000162, containing the >1/4-in. solids, was also used in the dissolution tests (see section 4.3.3). The main purpose of the coarse-grained fraction was to identify phase specific differences during dissolution. Figure 4-1(b) is a photograph of these solids. The largest pieces are the light-brown colored gibbsite in the center and in the front (≈1.5x2.5 cm). The black scale in the center left is only ≈1 mm thick, but ≈1x2 cm large. Several of the pieces of natrophosphate had a near perfect octahedral habit, e.g., the one just to the right of center-front. For additional illustration, three of the four main phases of the coarse fraction are shown in Figure 7-1. The pieces of green solids were not stable enough to be held with a pair of tweezers.

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

Figure 7-1. Close-up Pictures of Pieces of >1/4-inch Solids

Natrophosphate

Gibbsite

Fe-scale

The samples of <1/4-in. and >1/4-in. heel solids were separated into 50-mL centrifuge cones for better handling during the dissolution and to ensure proper mixing. The fine-grained fraction was split in seven subfractions, each containing 28.6 ± 1.4 g (vials labeled F1 to F7) and one vial with 16.8 g (F8). The coarse-grained fraction was subdivided into 7 cones (C1 to C7) based on the pieces' optical appearance (see Table 7-1). All of the green material was combined in one vial (C1). All of the black flakes were combined in one vial (C3). The clear (white after drying) crystals were separated into two vials (C2 and 7; in total 34 g). Two large pieces of gibbsite were put in one vial (C4; 18 g), and the remaining material (mostly tan-colored with some dark brown material) was combined in the last two vials (C5, C6; total of 26 g). This separation allowed a follow-up on the individual behavior of the phases to caustic dissolution.

Table 7-1. Combined Masses of the Material Used in the Dissolution Tests

Size	Color	Net weight	wt%
>¼"	Green	3.98	1.3
	(Clear) White	33.69	11.2
	Black	1.98	0.7
	Light brown	44.37	14.7
<¼"	Mix	217.34	72.1
Sum		301.36	

7.2.2 Caustic dissolution

The amount of caustic to be added was determined by the amount of gibbsite present in the fine fraction. The calculation was based on the required 3:1 molar ratio of caustic per mol gibbsite as required by the SAP (RPP-PLAN-40585). Based on the SEM-AFA analyses, ~40 wt% gibbsite[7] were identified. This number was used to determine the amount of caustic added as a 50 wt% solution, as shown in table 7-2.

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[7] This number is based on the result of the automated feature analyses (~30 vol%). Since gibbsite has a higher density (2.3g/cm^3) than Natrophosphate (1.7 g/cm^3), the 30 vol% translates to 40 wt%. The mass of gibbsite in the coarse fraction was ~50 wt% (see Table 8-1).

Table 7-2. Amount of Caustic Added to Individual Vials in mL of 50wt% Solution

Fine	Added	Coarse	Calculated	Actual
F1	22	C1	3	10
F2	23	C2	11	11
F3	23	C3	1.6	10
F4	24	C4	14	14 (+ 29)
F5	23	C5	7.4	10
F6	24	C6	14	14 (+ 21)
F7	23	C7	16	16
F8	13			
Sum	175		67	85 (+ 50)

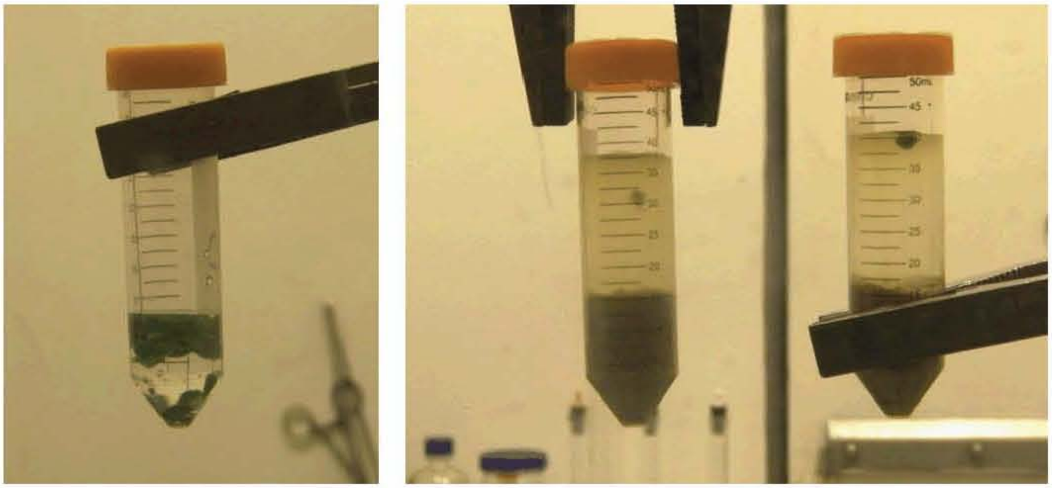
For an overall amount of 301 g (~170 mL) of heel material containing ~1.5 mol gibbsite, 241 mL of caustic addition were calculated as required. For the fine-grained fraction, the calculated amount (174 mL) and the amount actually added (175 mL) were in good alignment. However, for the coarse-grained fraction some adjustments were made:

(1) For some of the vials (C1, C3, and C5), the calculated amount was insufficient to keep the material in contact with the caustic during mixing; in these cases 10 mL were added.

(2) When applying the calculated amount of caustic to the coarse material-bearing vials (C4-C6), only 40 wt% of the material dissolved[8]. Further caustic addition resolved this issue.

The green phase showed a remarkable behavior. When adding caustic to the C1 and F-tubes, the green Ni-Al-phosphates started to float to the top of the liquid (see Figure 7-2). This implies that their specific gravity (SpG) is slightly less than ~1.5, the density of a 19.4 M caustic solution at the hot cell temperature of 29°C.

Figure 7-2. Examples of the Green Phase (Ni-Al-Phosphate) Swimming in Caustic Solution



Vial C1

Fine fractions

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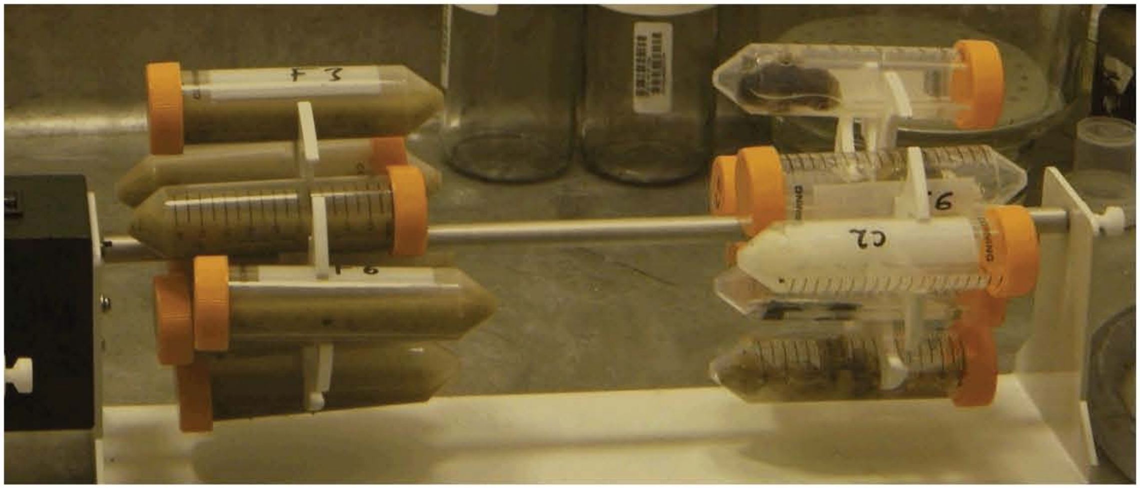
[8] This is not astonishing, since the amount added was based on the calculated 40 wt% gibbsite. Vials C4-C6, however, contained only the gibbsite chunks, more closely resembling 100 wt% gibbsite. When realizing this mistake (after the 6th water leach), additional caustic was added up to the theoretical amount able to dissolve all of the gibbsite.

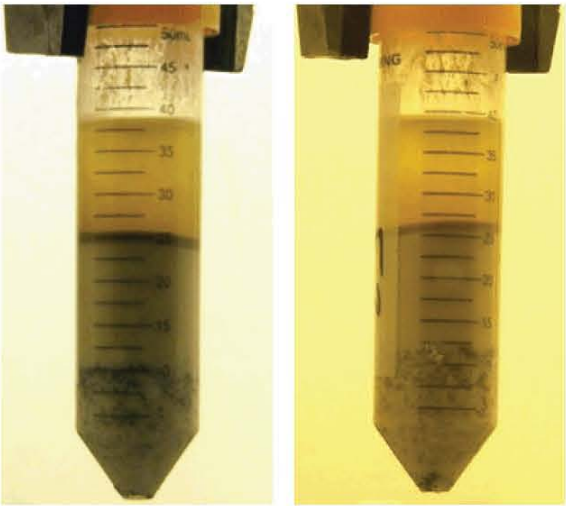
Figure 7-3. Rotisserie Shaker Loaded with Sample Vials



After 19 days of caustic dissolution in the rotisserie shaker, the rotisserie was stopped. All vials were weighed, centrifuged, decanted, and the liquid phase archived for potential future analysis. Two vials (C4 and C6) showed signs of leaking, i.e., white streaks around the vial caps. These vials were washed on the outside with D.I. water, dried, and re-weighed to determine the amount of leaking. The maximum loss of material due to leaking was < 2 wt%.

The vials were then centrifuged for 5 min, leaving all vials containing fine-grained material with the same appearance. In Figure 7-4, vials F1 and F2 are shown as an example. The lowest 10 mL contained coarse material of up to ~1 mm diameter with a brown-greyish color and dark specks interspersed. This layer seems to be more or less unaffected material (~½ of the original volume). On top of this layer were ~15 mL of fine-grained light-brown material, which is presumably made of sodium-aluminate. This off-white layer is topped by ~1 mL of very fine grained black dust. The caustic solution on top was not clear and even after centrifuging for several 10s of min, some particulate was visible, indicating colloidal size ranges.

Figure 7-4. Centrifuged Fine-Sample Vials After 19 Days of Caustic Dissolution


F1 F2

The coarse-grained samples appear different depending on their contents. In the following images of Figures 7-5 and 7-6, the seven vials are shown as they appeared after centrifuging for 5 min.

Figure 7-5. Centrifuged Coarse Sample Vials after 19 days of Caustic Dissolution

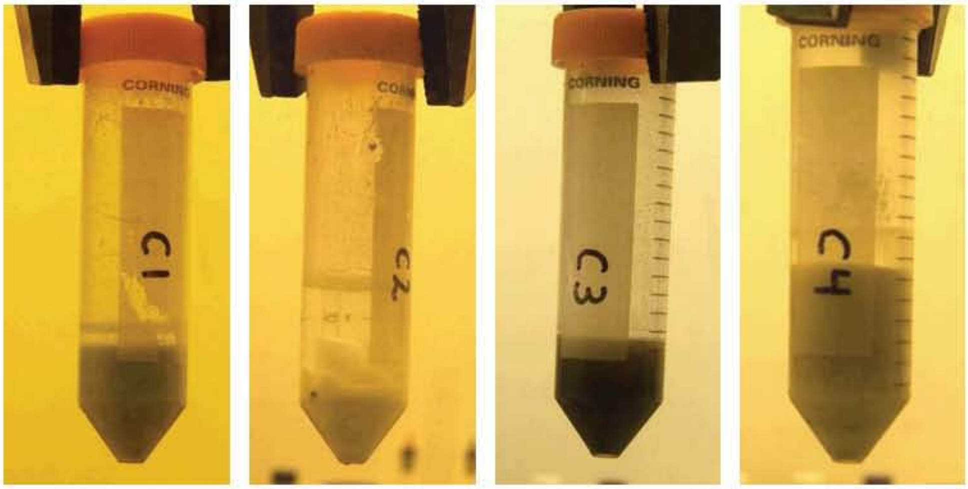
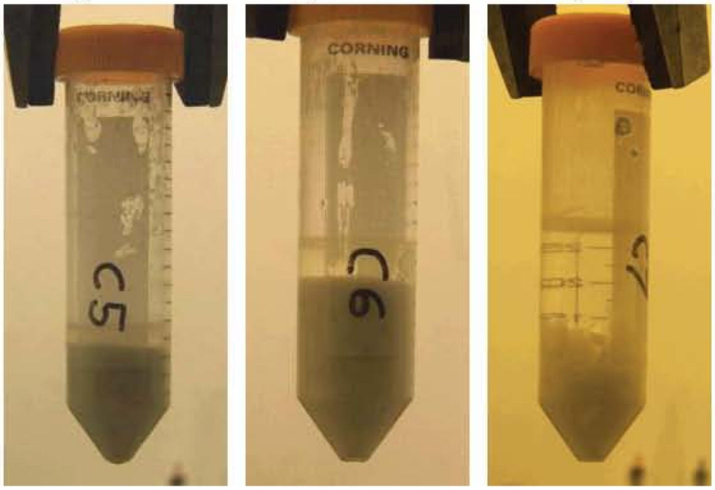

(Ni-Al-phosphate) Na-F-phosphate Fe-rich phase gibbsite

Figure 7-6. Centrifuged Coarse Sample Vials after 19 Days of Caustic Dissolution


gibbsite gibbsite Na-F-phosphate

The general appearance of the coarse-grained residuals is very fine-grained. However, the amount of caustic added per gram of material was much higher for some of these vials as mentioned earlier (see Table 7-2). This falsifies any conclusion that all of the coarse material will dissolve to such fine grained material in a pile of heel at the bottom of the tank. Most likely the coarse material would end up as the fine-grained fraction with some material up to 1 mm in diameter.

7.2.3 Mass Reduction during Water Leaches

Since both the natrophosphate and the sodium aluminate[9] are water-soluble, several water leaches were necessary to remove all water-soluble material. As shown in Figure 7-7, each step removed about 20 wt% of the original amount. Table 7-3 summarizes the amounts of water added and of material lost during these leaches. Each leaching cycle was performed with - on average - 35 mL of water per vial. The detailed amounts added can be seen in Table D-1.

Table 7-3. Volumes of Starting Material, Caustic Addition, Water Addition, and Residual Material of Heel Dissolution Testing

	Amount of heel material	Caustic added	Water needed for removal	Water added on average per cycle	Amount of residual material
Fine	217 g (125 mL)	175 mL	1639 mL	273 mL	12.5 mL
Coarse	84 g (48 mL[#])	135 mL (67 mL[$])	1484 mL	247 mL	4.5 mL[#]
Tank*	6,800 gal	9,520 gal	89,162 gal	14,851 gal	680 gal

[*] Based on the dissolution of the fine-grained fraction. Based on Letter CH2M-0603302.4, the total amount of remaining material is 6,800 gallons of waste.

[#] Calculated amount. Volume of coarse material was determined assuming wt (Fines)/vol (Fines) = wt (coarse)/vol (coarse).

[$] Theoretical need. Amount is based on 3:1 molar ratio of caustic to gibbsite.

Figure 7-7. Average Masses During Caustic Dissolution and Water Leaching

(Measured after Decanting Liquids)

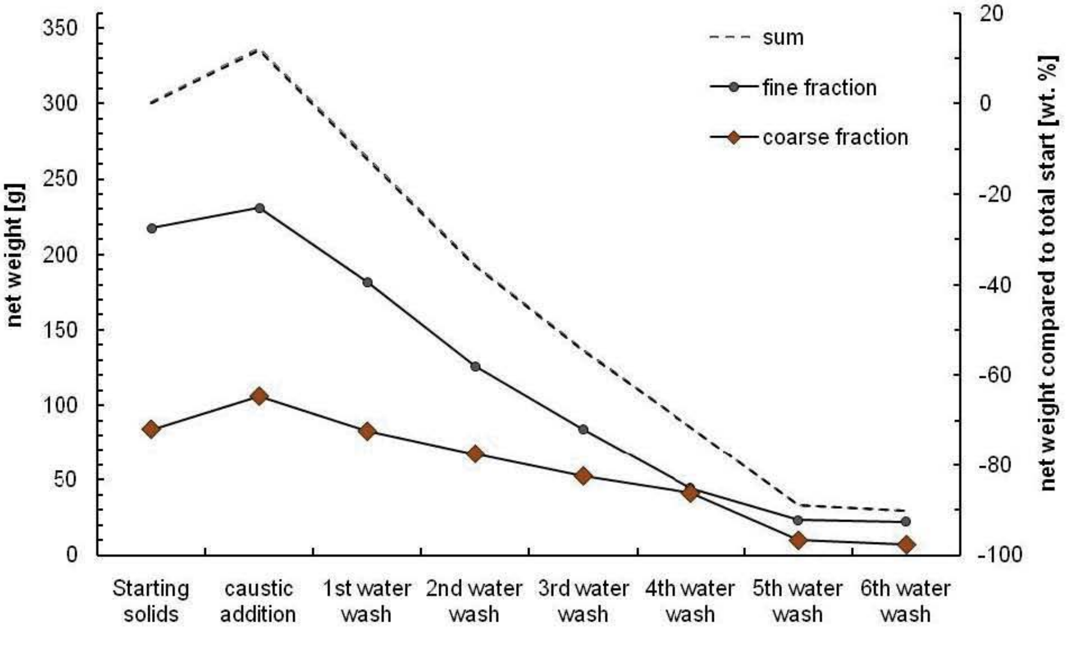


Figure 7-8 and Table 7-4 show the weight loss for the individual coarse phases. The Ni-Al-phosphate actually increased in mass during dissolution and water leaching. The Natrophosphate is reduced to 2 wt% of its original mass, and the Fe-oxide phase has 28 wt% left. The gibbsite

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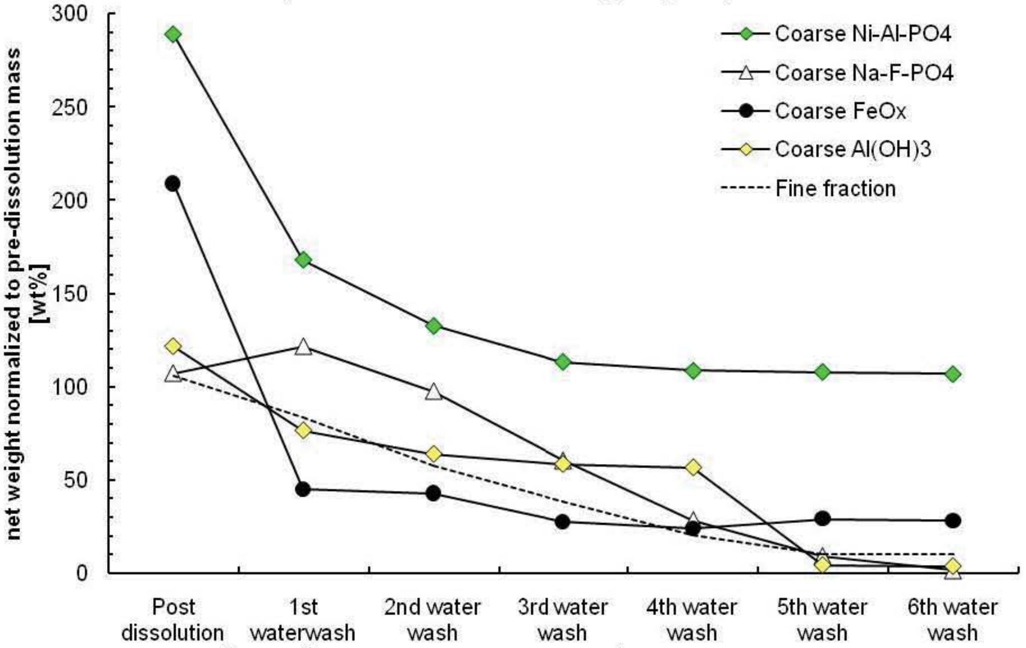
[9] Produced during caustic dissolution via the reaction: Gibbsite $[Al(OH)_3]$ + Caustic [NaOH] → Na-aluminate $[NaAl(OH)_4]$

lost 95 wt% of its original mass; the kink in the graph of Figure 7-8 after the 4th water wash is due to the 2nd caustic addition described on page 73.

Only 9.7 wt% of the starting material is left as residual. The residual material is dark, very fine grained, highly viscous, and sticky.

Figure 7-8. Masses of Coarse Phases During Caustic Dissolution and Water Leaching

(Measured after Decanting Liquids)



The data plotted for 5th and 6th wash for gibbsite are the 2nd-to-last and last wash from table D-1.

Table 7-4. Amount of Material Remaining after Each Decanting Step

Net solids [g]	Starting solids	Caustic addition	1st water wash	2nd water wash	3rd water wash	4th water wash	5th water wash	6th water wash
Fine fraction	217.3	230.9	181.7	125.8	83.9	44.2	23.2	22.1
Coarse fraction	84.0	105.8	82.6	67.5	52.5	41.2	10.1	7.2
Combined	**301.3**	**336.7**	**264.4**	**193.3**	**136.4**	**85.4**	**33.3**	**29.2**

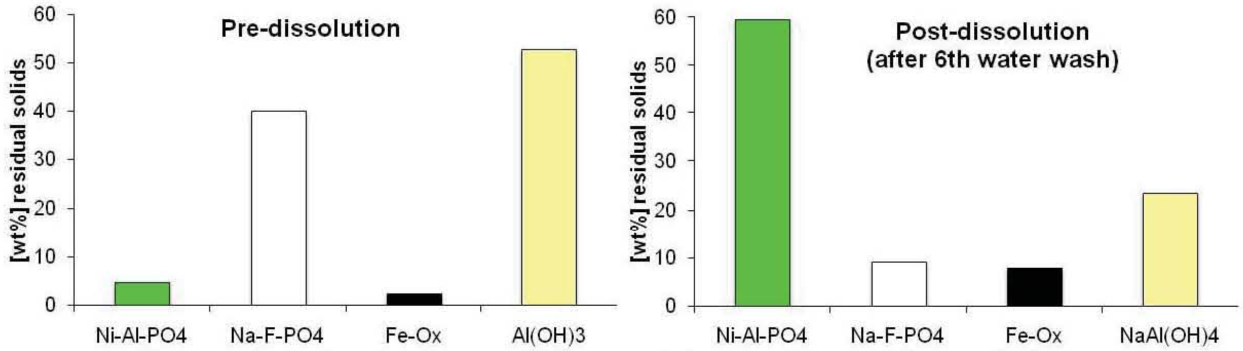
Residual material [wt.%]								
Coarse fraction	100	126	98	80	61	47	12	9
Fine fraction	100	106	84	58	39	20	11	10
Combined	**100**	**112**	**88**	**64**	**45**	**28**	**11**	**10**

Coarse fraction split [wt.% remaining]								
Coarse Ni-Al-PO$_4$	100	289	168	133	113	109	108	107
Coarse Na-F-PO$_4$	100	107	122	98	61	29	9	2
Coarse FeO$_x$	100	209	45	43	28	24	29	28
Coarse Al(OH)$_3$	100	122	77	64	59	57	5*	4*

* The data for 5th and 6th wash for gibbsite are the 2nd-to-last and last wash from table D-1.

The mass distribution of original and residual material is displayed in Figure 7-9. The graphs show the mass distribution before (left) and after (right) the dissolution and all washing steps. The Ni-Al rich material ("green"), which made only 4 wt% of the original mass is the dominant feature in the residual solids, followed by the Fe-rich phase ("black"). The fine-grained fraction was mostly made of broken pieces of the coarser material. However, some phases of the fine fraction identified by SEM-EDS were not seen in the coarse material. One of them, the Na-Sr-phosphate (Nastrophite) actually accumulated during the dissolution and water leaching process so that it became identifiable by XRD. This is also reflected in the chemical data in Table 3-1. The amount of Sr increased from 0.58 mg/g to 9.45 mg/g and the activity due to Sr-89/90 increased from 0.17 mCi/g to 5.41 mCi/g.

Figure 7-9. Mass Distribution of the Residual Coarse-Grained Phases



These graphs are based on the mass of the separated coarse phases and their residual mass.

Based on these results, starting the heel dissolution with a water leach first would remove the Na-F-phosphate (~ 40 wt%) but leave the other phases mostly untouched. Caustic dissolution of the remaining material with a subsequent water leach to remove the now water-soluble Al-phase (sodium aluminate) should result in a lower result than using the approach in this study.

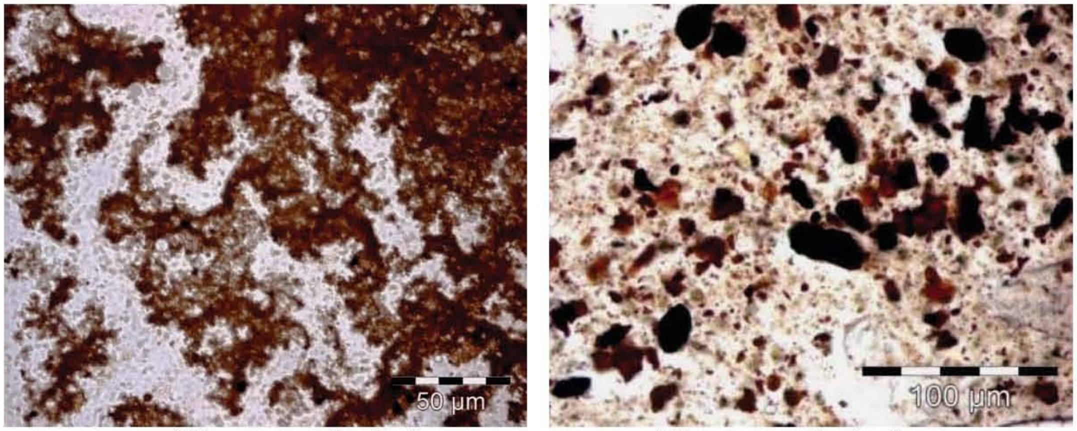
7.3 CHARACTERIZATION OF RESIDUAL MATERIAL

The residual material of the fine-grained fraction was sampled as S09R000156. The residual of the coarse-grained sample was sampled as S09R000172. This sample contained mostly the mass of vial C-1 (the former Ni/Al-phosphate), with minor amounts of gibbsite and natrophosphate residuals and some large (~1 mm) pieces of Fe-oxide. The results of PLM, SEM-EDS, and XRD are provided below.

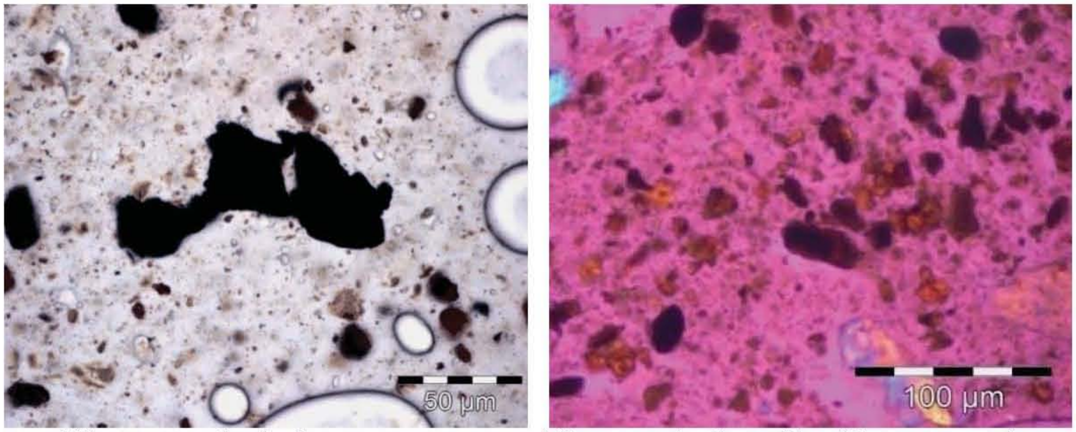
7.3.1 Polarized Light Microscopy

The fine grained residual contains ~50% very fine-grained material (< 1 μm). Few Natrophosphate shards are visible; mostly Fe-oxide and amber particles are present. In Figure 7-10, the top left image shows an overview of the fine-grained fraction. On the right side, a part of the coarser part of the slide shows mostly light- to dark-colored amber particles, very few Natrophosphate crystals (clear), and some Fe-oxide (black). Below (bottom right), the same section is photographed with crossed polars and red I compensator revealing three large anisotropic crystals (blue and orange) which is made up of submicron red-orange colored crystals. This phase might be the Ni-Al-phosphate, but it could not be clearly identified. On the bottom left, a large Fe-oxide particle is in focus with various sizes of amber particles around.

Figure 7-10. Polarized Light Microscopy Images of Sample S09R000156



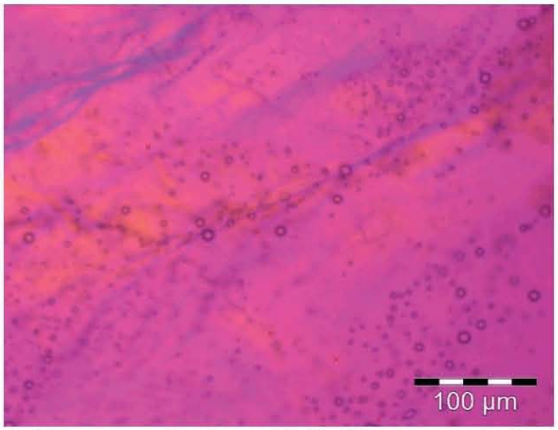
(60x, uncrossed polars) (40x, uncrossed polars)



(60x, uncrossed polars) (40x, crossed polars with red I compensator)

The PLM slide of the residual coarse-grained material (Figure 7-11) contained only submicron material showing streaks of orange and blue when using crossed polars and Red I compensator. The two-color appearance indicates the presence of a phase, however, it is too small to identify.

Figure 7-11. Polarized Light Microscopy Image of Sample S09R000172

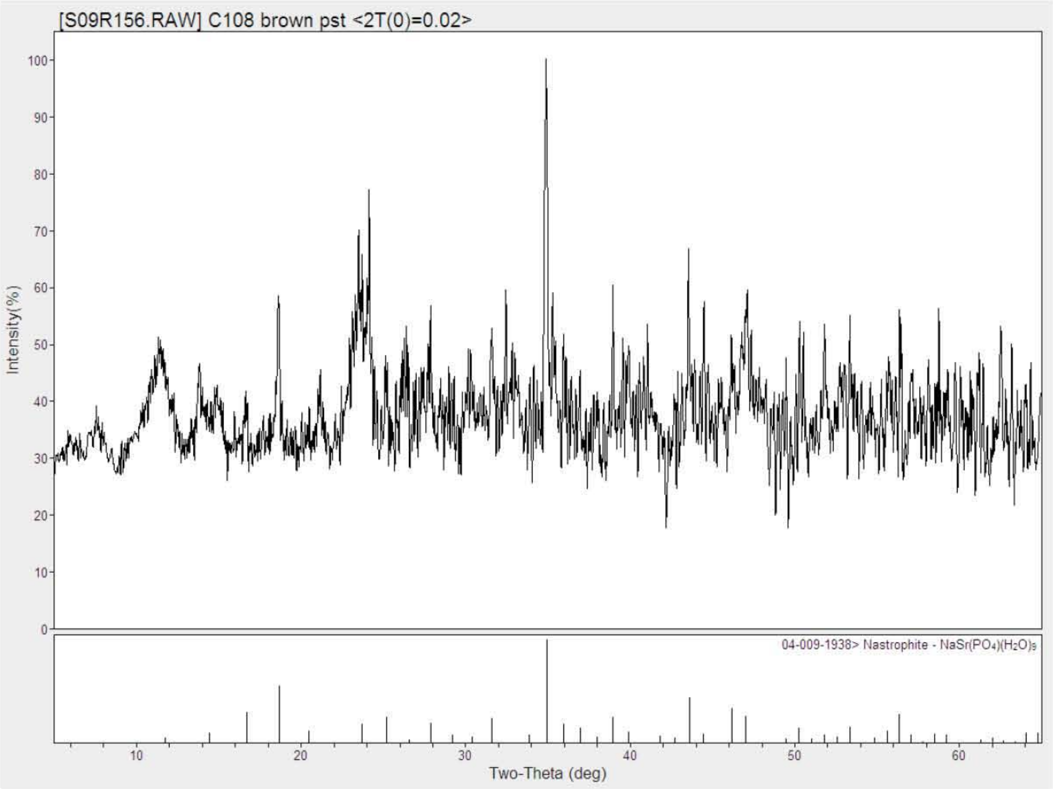


(20x, crossed polars with Red I compensator)

7.3.2 X-ray Diffraction

As shown in Figure 7-12, the fine-grained fraction (sample S09R000156) contains Nastrophite [$NaSr(PO_4)x9H_2O$] as the sole identifiable phase. The remaining broad peaks represent one or several undetermined phase(s). The elevated baseline indicates an amorphous Fe-rich phase, most likely from the residual scale.

Figure 7-12. X-ray Diffraction Spectrum of Sample S09R000156 (Fine-Grained Post-Dissolution)



The coarse-grained fraction (sample S09R000172) had insufficient material left for an XRD preparation.

7.3.3 Scanning Electron Microscopy

The fine-grained fraction was prepared on a water-washed filter; therefore, any sodium fluoride phosphate dissolved. The remaining sample consisted primarily of aluminum-rich and nickel-rich phases.

In particular, the following phases were observed (Figures 7-13 to 7-20):

Figure 7-13. Backscattered Electron Image and Energy Dispersive Spectrometry Spectrum of Gibbsite in Dissolution Residual

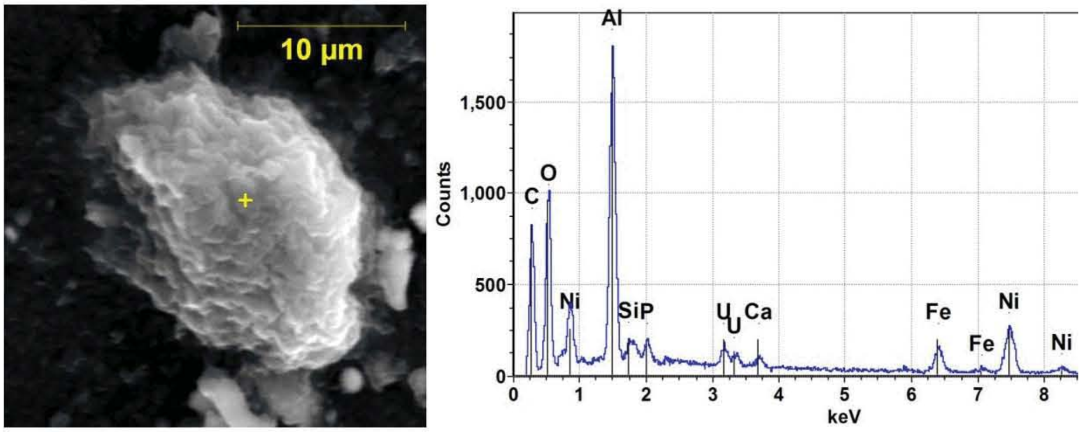


Figure 7-14. Backscattered Electron Image and Energy Dispersive Spectrometry Spectrum of Ni-Al-rich Amorphous Material in Dissolution Residual

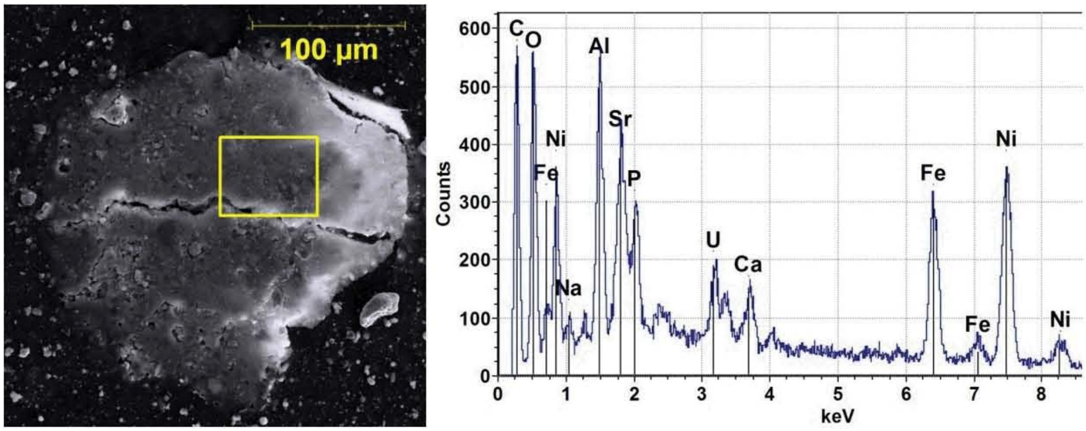


Figure 7-15. Backscattered Electron Image and Energy Dispersive Spectrometry Spectrum of Ni-Al-rich Material in Dissolution Residual

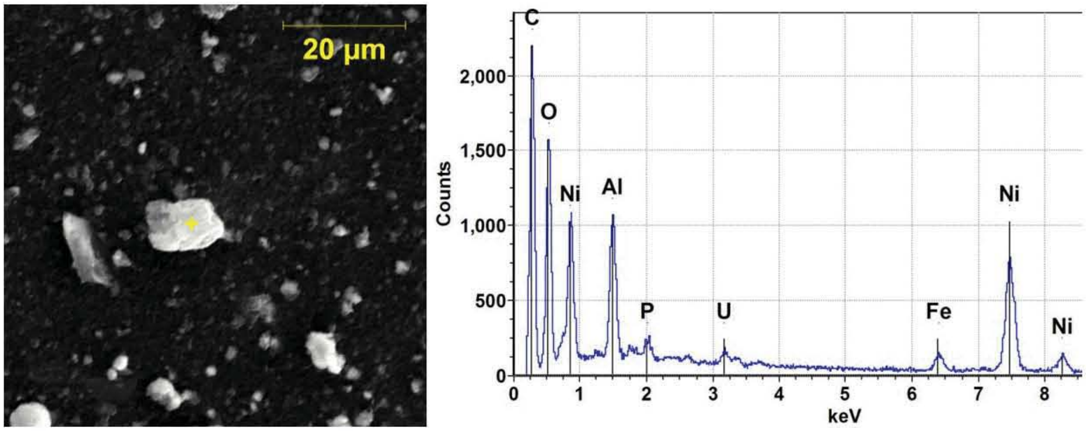
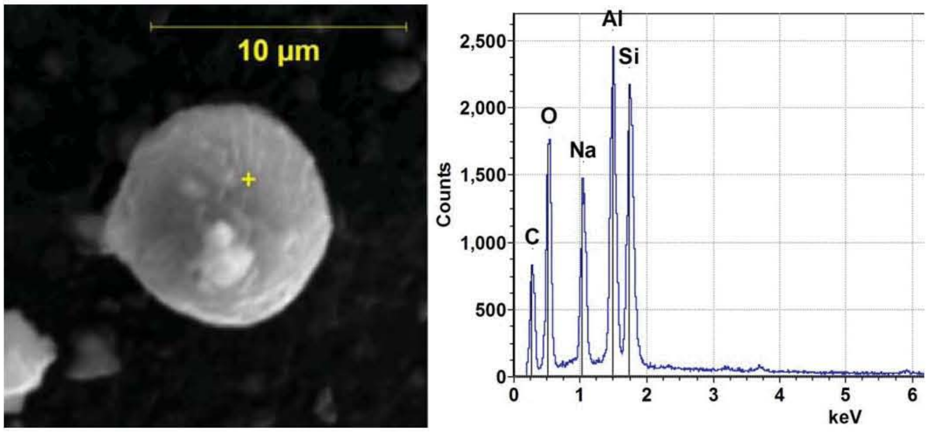


Figure 7-16. Backscattered Electron Image and Energy Dispersive Spectrometry Spectrum of Na-Aluminosilicates in Dissolution Residual



A strontium-rich phase is similar to the sodium strontium phosphate observed in other samples, but with the sodium apparently largely replaced by calcium.

Figure 7-17 Backscattered Electron Image and Energy Dispersive Spectrometry Spectrum of Sr-Phosphate in Dissolution Residual

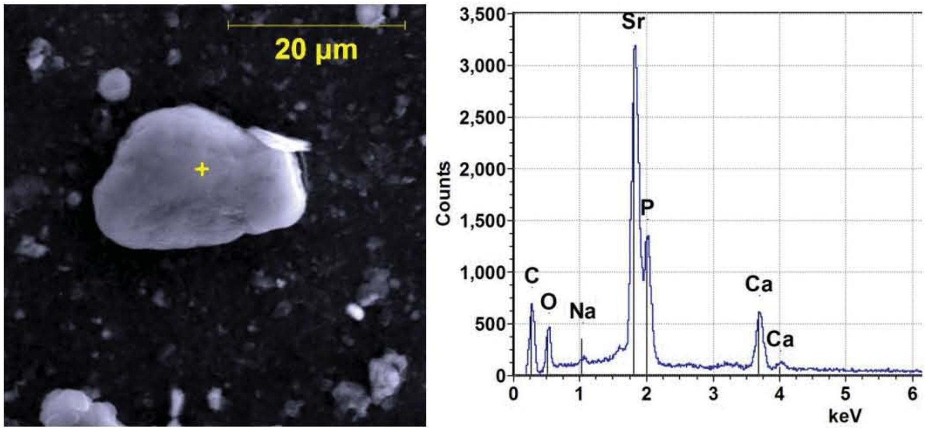


Figure 7-18. Backscattered Electron Image and Energy Dispersive Spectrometry Spectrum of Fe-rich Phase in Dissolution Residual

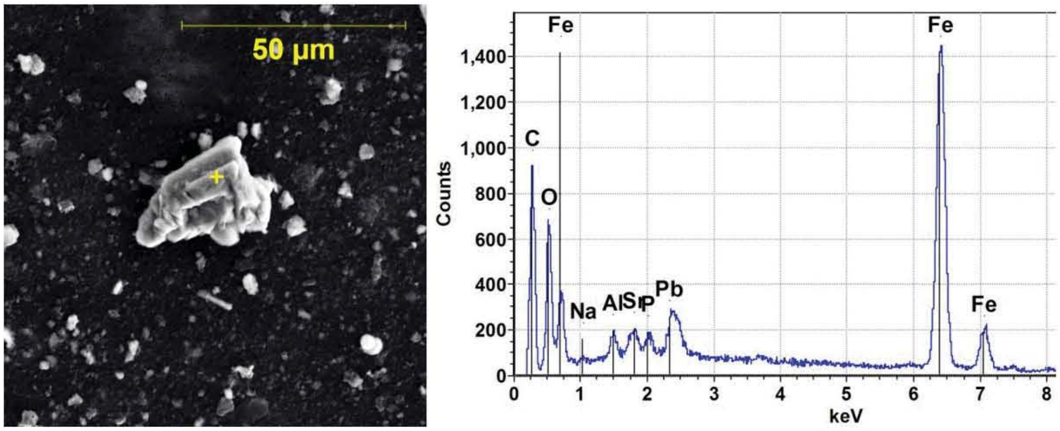


Figure 7-19. Backscattered Electron Image and Energy Dispersive Spectrometry Spectrum of U-rich Phases in Dissolution Residual

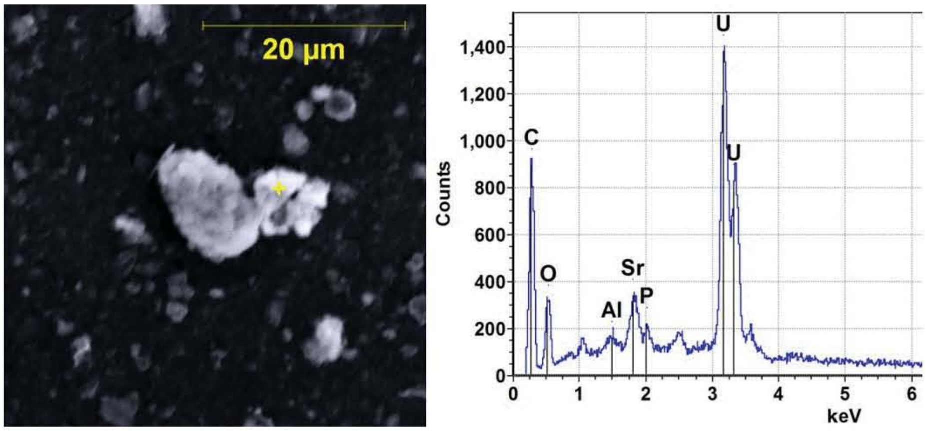
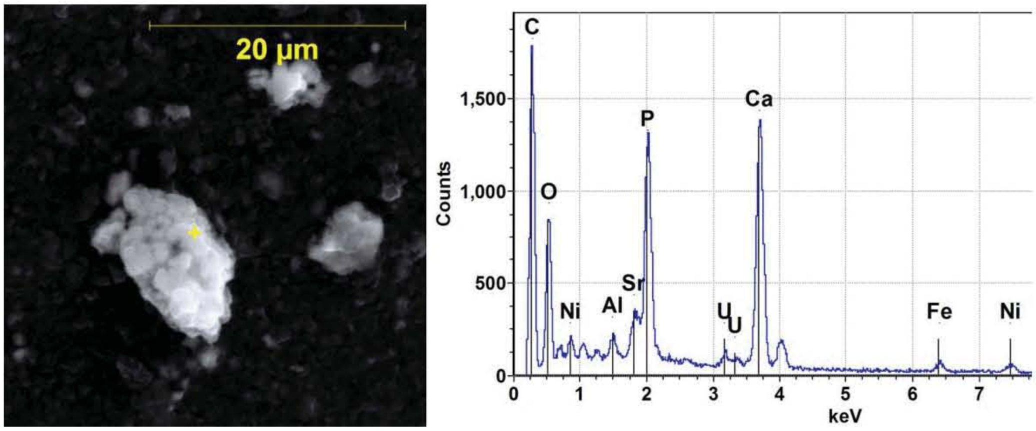


Figure 7-20. Backscattered Electron Image and Energy Dispersive Spectrometry Spectrum of Ca-Phosphate in Dissolution Residual



The coarse-grained sample mainly consisted of the Ni-Al-phosphate residue. Since this material makes up about 60 wt% of the coarse-grained residual material, a closer look was prioritized.

The nickel-aluminum bearing particulate occurs primarily as aggregates of very fine particles. The main difference of the EDS spectrum in Figure 7-21 (right) to pre-dissolution sample spectra is the huge reduction in P and U content. The same effect can be seen in the residual fine-grained sample of the Ni-Al-rich phase, e.g., in Figure 7-15. A close-up of the individual particles as shown in Figure 7-22 reveals that they are spherical and ~0.5 micron in diameter.

Figure 7-21. Backscattered Electron Image and Energy Dispersive Spectrometry Spectrum of Residual of the Ni-Al-P-rich Phase

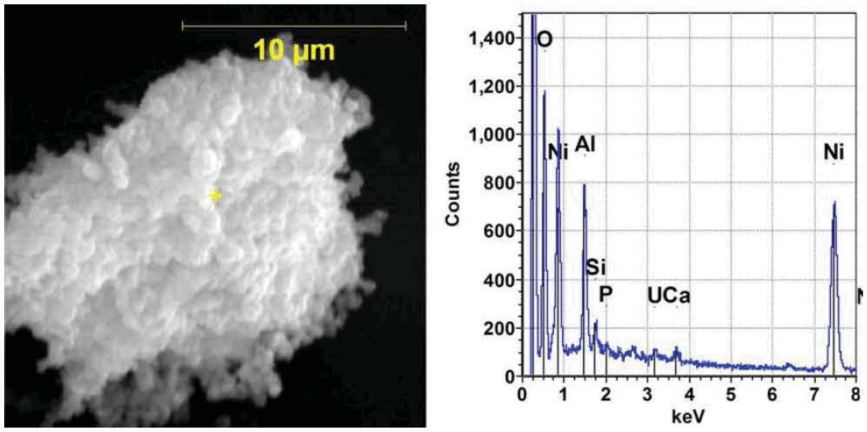
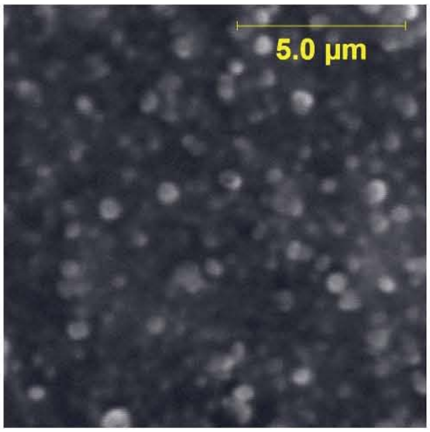


Figure 7-22. Backscattered Electron Image of a Close-Up of the Ni/Al-Phase in the Residual Sample



In addition to the Ni-Al-rich particulate, there were scattered particles of calcium phosphate and a uranium-rich phase. Some of the spectra had a small silicon peak; whether this was a separate phase, or a variation in the Ni-Al chemistry of the dominant particle type, could not be determined.

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 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 Repeat Tests

As discussed in Section 5.3 and in Attachment C, three sets of light-scattering-based PSD measurements were completed on separate portions of <600-µm C-108 solids retrieved from test sample S09R000307. The triplicate measurements produced very similar PSD data.

8.2.2 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.

8.2.3 Calibrations

The majority of the quantitative measurements during the characterization of the C-108 heel solids were gravimetric. The in-tolerance status of each analytical balance used during the test program was verified at a minimum of 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.

As discussed in Section 4.1.1, a pycnometer jar was constructed to perform the bulk density measurement on the C-108 heel solids composite. The volume of the pycnometer was measured by filling with reagent water, weighing, and converting the weight of water added to a volume using reference density values. The results of 15 sets of volume calibration measurements, performed on 8 separate days, are presented in Table 8-1.

Table 8-1. Pycnometer Jar Volume Calibration

		Pycnometer Jar			Added H_2O		Pycnometer Vol (mL)
Date	**Time**	**Wt Empty (g)**	**Wt Filled (g)**	**Wt H_2O (g)**	**T (°C)**	**Density (g/mL)**	
10-June-09	13:35	324.51	801.90	477.39	24.4	0.997197	478.73
	14:35	317.93	795.31	477.38	24.7	0.997121	478.76
11-June-09	10:45	317.92	795.20	477.28	24.95	0.997058	478.69
	11:50	317.94	794.76	476.82	25.3	0.996967	478.27
	14:00	317.87	795.19	477.32	24.8	0.997096	478.71
17-June-09	13:44	317.61	794.89	477.28	21.45	0.997894	478.29
18-June-09	13:55	317.62	795.34	477.72	21.65	0.997849	478.75
	14:45	317.62	795.28	477.66	21.4	0.997905	478.66
19-June-09	11:15	317.641	795.550	477.909	19.35	0.998336	478.706
	13:00	317.639	795.329	477.690	21.25	0.997938	478.677
22-Jun-09	10:25	317.628	795.228	477.600	22.85	0.997574	478.762
	11:10	317.628	795.230	477.602	22.9	0.997562	478.769
23-June-09	09:35	317.626	795.222	477.596	23.1	0.997515	478.786
	12:55	317.631	795.269	477.638	22.7	0.997609	478.783
30-June-09	15:15	317.611	794.090	476.479	32.0	0.995026	478.861
						Average	478.68
						Standard Deviation	0.17

The column used in the settling tests was fabricated for similar measurements in 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-108 heel characterization and retrieval testing described in this report was executed, in general, as described in the client-approved test plan (WRPS-0900639 and WRPS-0901437). However, the following deviations from the test plan did occur during completion of the test program.

a. A very large piece of solid material in Sample 6 of the C-108 heel solids had to be broken up during composite preparation of the test composite. The large solid piece occupied nearly one-half of the sample volume and a representative sample could not be taken without breaking it into a few smaller pieces. Breakage of large solids in the remaining samples was minimal but a few other pieces of solids may have been broken during withdrawal from the sample containers and mixing of the composites.

b. The measurement and calculation of the densities, both D_{Bulk} and $D_{Dry\ Slds,}$ of the composite only apply to the solids remaining after partial dissolution of heel solids resulting from addition of water to the composite during the D_{Bulk} measurement. The predominance of water soluble material in the heel solids was not determined until the bulk of the test program had been completed.

c. In the *de facto* water wash of the test composite during the bulk density and coarse sieving operations, only 0.5 parts water to 1 part composite was added instead of the 1 part water to 1 part composite requested.

d. The solids loading in the settling test slurries may have been greater than the nominal 1 wt% and 5 wt% concentrations specified in the test plan. The size of the settling test samples used was defined in terms of wt% H_2O values determined by oven drying at 90 °C. The presence of large amounts of highly hydrated salts (natrophosphate in particular) led to the misinterpretation of the results of these measurements and to use of larger samples of the <1/4-in. composite solids than required. The actual concentrations of solids in the settling test slurries may have been as high as 1.36 wt% and 6.87 wt%. However, due to additional dissolution of the composite solids that may have occurred, the actual concentrations of solids in the slurries is unknown. Once again, the composite solids were not sufficiently characterized to identify this issue until the settling tests were nearly complete.

e. During caustic dissolution, the coarse sample containing vials were filled with more caustic than originally planned. Reasons were a minimum amount necessary to cover the samples and vials that contained only gibbsite rather than the average of 40 wt% gibbsite.

f. RPP-PLAN-40585 Rev. 1 and Rev. 2, Section 8.3 specified: “Each constituent analyzed by ATL shall be identified by a CAS identification number if available.” The CAS numbers were not included in the data summary tables presented in Attachment A. A waiver of this requirement was provided by e-mail from the Sampling and Analysis Plan (RPP-PLAN-40585) author (e-mail from D. M. Nguyen to W. S. Callaway, “C-108 Heel Characterization Report,” [Nguyen, D. M., 2010-06-07]). A facsimilie of the e-mail waiver is included in the Supporting Documents folder of the IDMS review and approval workflow for this report.

9.0 REFERENCES

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ATTACHMENT A

LABORATORY ANALYSIS DATA

ATTACHMENT A – LABORATORY ANALYSIS DATA

Transcripts of the data summary reports for the physicochemical analyses of the liquid and solid samples generated during the characterization and testing of the C-108 heel solids are presented in this attachment. All analyses were performed in the 222-S Laboratory by personnel of Advanced Technologies and Laboratories International, Inc.

The measurements and analyses required for the test composite solids and the final residue of the caustic dissolution tests were specified in Table 4-6 of the authorizing SAP (RPP-PLAN-40855). The analyses required for the test liquids from the bulk density measurement (BDL) and the coarse sieving operation (CSL) were specified in Table 2 of the laboratory test plan (WRPS-0900639). The identifications of the samples analyzed and the analytes measured are summarized in Table A-1. The analytical procedures used are identified in the notes following the table.

Table A-1. Sample Identifications and Analyses Completed (2 sheets)

Parent Sample	Sample Preparations	Analytes
Test Composite (S09R000145)	Direct	Wt% Water [a] pH Solids [b] TIC/TOC [c] ^{129}I [d]
	Water Leach [e] (S09R000185) (S09R000541 - Rerun)	IC [f]: F^-, Cl^-, Br^-, NO_2^-, NO_3^-, PO_4^{3-}, SO_4^{2-} IC [g]: formate, acetate, oxalate, glycolate
	Acid Digest [h] (S09R000188) (S09R000542 - Rerun)	Actinides [i]: ^{233}U, ^{234}U, ^{235}U, ^{236}U, ^{237}Np, ^{238}U ^{99}Tc [j]
	Fusion – Ni Crucible [k] (S09R000181)	RCRA Metals [l] Gamma Energy Analysis [m] Sr-90 [n] Pu/Am [o]: ^{238}Pu, $^{239/240}$Pu, ^{241}Am, ^{242}Cm, $^{243/244}$Cm
	Fusion – Zr Crucible [k] (S09R000658)	RCRA Metals [l]
Bulk Density Liquid (S09R000146)	Direct	SpG [p] TIC/TOC [c] IC [f]: F^-, Cl^-, Br^-, NO_2^-, NO_3^-, PO_4^{3-}, SO_4^{2-} IC [g]: formate, acetate, oxalate, glycolate RCRA Metals [l]
Coarse Sieving Liquid (S09R000148)	Direct	

Table A-1. Sample Identifications and Analyses Completed (2 sheets)

Parent Sample	Sample Preparations	Analytes
Caustic Dissolution Residue (S09R000174)	Direct	Wt% Water [a] pH Solids [b] TIC/TOC [c] ^{129}I [d]
	Water Leach [e] (S09R000184)	IC [f]: F^-, Cl^-, Br^-, NO_2^-, NO_3^-, PO_4^{3-}, SO_4^{2-} IC [g]: formate, acetate, oxalate, glycolate
	Acid Digest [h] (S09R000187)	Actinides [i]: ^{233}U, ^{234}U, ^{235}U, ^{236}U, ^{237}Np, ^{238}U ^{99}Tc [j]
	Fusion – Ni Crucible [k] (S09R000180)	RCRA Metals [l] Gamma Energy Analysis [m] Sr-90 [n] Pu/Am [o]: ^{238}Pu, $^{239/240}$Pu, ^{241}Am, ^{242}Cm, $^{243/244}$Cm
	Fusion – Zr Crucible [k] (S09R000657)	RCRA Metals [l]

[a] LA-514-115, Rev/Mod E-0	Thermal Stability and Percent Weight Loss using the TA, DSC, and TGA
[b] LA-212-105, Rev/Mod G-0	pH Determination on Solid Matrix Samples
[c] LA-342-100, Rev/Mod J-0	Determination of Carbon by Hot Persulfate Oxidation and Coulometric Detection
[d] LA-378-104, Rev/Mod F-0	Iodine-129 in Tank Farm Solids
[e] LA-504-101, Rev/Mod L-0	Water Leach Sample Preparation
[f] LA-533-107, Rev/Mod H-0	Ion Chromatographic Analysis of Anions on DIONEX Model DX-500
[g] LA-533-115, Rev/Mod J-0	Ion Chromatographic Analysis of Anions and Cations on DIONEX Model 500
[h] LA-505-163, Rev/Mod F-0	Acid Digestion of Sediments, Sludges, or Soil Samples for Spectroscopic Analysis of Metals
[i] LA-506-102, Rev/Mod E-0	Determination of Trace Elements and Radionuclides Using the PQ EXCELL Inductively Couples Plasma-Mass Spectrometer
[j] LA-438-101, Rev/Mod J-0	Determination of Technetium-99 by Solvent Extraction and Liquid Scintillation Counting
[k] LA-549-141, Rev/Mod L-0	Fusion by Alkali Metal Hydroxide
[l] LA-505-161, Rev/Mod J-0-A	Inductively Coupled Plasma (ICP) Emission Spectrometric Method for the Thermo Jarrell Ash Type 61E
[m] LA-548-121, Rev/Mod I-0	Preparation of Sample Mounts for Gamma Energy Analysis
[m] LA-508-165, Rev/Mod D-0	Gamma Energy Analysis – The Genie 2K System
[n] LA-220-101, Rev/Mod I-0	High Level Strontium-90 in Aqueous Samples
[o] LA-953-104, Rev/Mod H-1	Determination of Plutonium and Americium by Extraction with TRU Resin
[p] LA-510-112, Rev/Mod I-0	Determination of Specific Gravity for Free Liquid Samples

The following abbreviations and codes are used throughout the 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
	Det Limit	Minimum detection limit
	Qual Flags	Codes for qualifiers for reported results
Analyte Suffixes	[Analyte]-C	Carrier %-recovery
	[Analyte]T	Tracer %-recovery
Sample Preparation Codes	[Blank]	Direct analysis of sample
	W	Water leach
	A	Acid digest
	F	KOH/KNO_3 fusion in Ni crucible
	Z	KOH/KNO_3 fusion in Zr crucible
Qualifier Codes	B	Found in blank
	c	RPD outside range
	e	Serial dilution outside range
	J	Estimated value
	U	Less than detection limit
	Y	Result of closing continuing calibration blank for TOC in S09R000145 was >MDL and <EQL. CCB result was also >5% of the sample TOC result.

Table A-2. Physicochemical Analysis Results for Test Composite Sample (4 sheets)

Sample Group: 20090517

Data Summary Report

Sample #	A	Analyte	Unit	Std %-Rec	Blank	Result	Duplicate	Average	RPD	Spk %-Rec	Det Limit	Count Error	Qual Flags
S09R000145		pH	unitless	n/a	n/a	11.7	11.7	11.7	0.427	n/a	0.0100	n/a	
S09R000145		Percent water	%	99.8	n/a	27.9	28.5	28.2	2.23	n/a	0.0100	n/a	
S09R000145		Total organic carbon	ug/g	100	<20.0	687	572	630	18.3	94.0	148	n/a	J, Y
S09R000145		Total inorganic carbon	ug/g	97.2	<7.00	5.91E+03	5.67E+03	5.79E+03	4.15	110	51.9	n/a	
S09R000658	Z	Silver	ug/g	97.5	<52.2	49.9	<50.4	n/a	n/a	n/a	48.3	n/a	J
S09R000658	Z	Aluminum	ug/g	103	<313	1.12E+05	1.12E+05	1.12E+05	0.583	n/a	290	n/a	
S09R000658	Z	Arsenic	ug/g	102	<522	<483	<504	n/a	n/a	n/a	483	n/a	U
S09R000658	Z	Boron	ug/g	100	<313	<290	<302	n/a	n/a	n/a	290	n/a	U
S09R000658	Z	Barium	ug/g	103	<31.3	<29.0	<30.2	n/a	n/a	n/a	29.0	n/a	U
S09R000658	Z	Beryllium	ug/g	98.9	<10.4	<9.67	<10.1	n/a	n/a	n/a	9.67	n/a	U
S09R000658	Z	Bismuth	ug/g	103	<1.04E+03	<9.67E+02	1.15E+03	n/a	n/a	n/a	967	n/a	U
S09R000658	Z	Calcium	ug/g	104	<522	<483	<504	n/a	n/a	n/a	483	n/a	U
S09R000658	Z	Cadmium	ug/g	101	<52.2	<48.3	<50.4	n/a	n/a	n/a	48.3	n/a	U
S09R000658	Z	Cerium	ug/g	99.3	<313	<290	<302	n/a	n/a	n/a	290	n/a	U
S09R000658	Z	Cobalt	ug/g	99.8	<104	<96.7	211	n/a	n/a	n/a	96.7	n/a	U
S09R000658	Z	Chromium	ug/g	101	<52.2	<48.3	<50.4	n/a	n/a	n/a	48.3	n/a	U
S09R000658	Z	Copper	ug/g	99.0	<52.2	<48.3	<50.4	n/a	n/a	n/a	48.3	n/a	U
S09R000658	Z	Europium	ug/g	96.3	<52.2	<48.3	<50.4	n/a	n/a	n/a	48.3	n/a	U
S09R000658	Z	Iron	ug/g	100	<52.2	3.04E+03	3.10E+03	3.07E+03	2.15	n/a	48.3	n/a	
S09R000658	Z	Lanthanum	ug/g	99.5	<31.3	29.1	<30.2	n/a	n/a	n/a	29.0	n/a	J
S09R000658	Z	Lithium	ug/g	108	<31.3	<29.0	<30.2	n/a	n/a	n/a	29.0	n/a	U
S09R000658	Z	Magnesium	ug/g	100	<522	<483	<504	n/a	n/a	n/a	483	n/a	U
S09R000658	Z	Manganese	ug/g	99.9	<31.3	50.7	41.8	46.2	19.2	n/a	29.0	n/a	J
S09R000658	Z	Molybdenum	ug/g	102	<209	<193	<202	n/a	n/a	n/a	193	n/a	U
S09R000658	Z	Sodium	ug/g	110	2.02E+03	1.49E+05	1.49E+05	1.49E+05	0.106	n/a	967	n/a	
S09R000658	Z	Neodymium	ug/g	98.1	<104	<96.7	<101	n/a	n/a	n/a	96.7	n/a	U
S09R000658	Z	Nickel	ug/g	98.6	<209	3.49E+03	3.57E+03	3.53E+03	2.24	n/a	193	n/a	
S09R000658	Z	Phosphorus	ug/g	101	<522	4.93E+04	4.88E+04	4.90E+04	0.999	n/a	483	n/a	

Table A-2. Physicochemical Analysis Results for Test Composite Sample (4 sheets)

Sample Group: 20090517 Data Summary Report

Sample #	A	Analyte	Unit	Std %-Rec	Blank	Result	Duplicate	Average	RPD	Spk %-Rec	Det Limit	Count Error	Qual Flags
S09R000658	Z	Lead	ug/g	103	<522	665	784	724	16.5	n/a	483	n/a	J
S09R000658	Z	Sulfur	ug/g	101	<1.04E+03	<9.67E+02	<1.01E+03	n/a	n/a	n/a	967	n/a	U
S09R000658	Z	Antimony	ug/g	98.3	<522	824	<504	n/a	n/a	n/a	483	n/a	J
S09R000658	Z	Selenium	ug/g	101	<1.04E+03	<9.67E+02	<1.01E+03	n/a	n/a	n/a	967	n/a	U
S09R000658	Z	Silicon	ug/g	92.0	<313	669	567	618	16.6	n/a	290	n/a	J
S09R000658	Z	Samarium	ug/g	100	<522	<483	<504	n/a	n/a	n/a	483	n/a	U
S09R000658	Z	Strontium	ug/g	101	<31.3	472	780	626	49.1	n/a	29.0	n/a	c
S09R000658	Z	Thorium	ug/g	102	<522	556	<504	n/a	n/a	n/a	483	n/a	J
S09R000658	Z	Titanium	ug/g	103	<52.2	<48.3	<50.4	n/a	n/a	n/a	48.3	n/a	U
S09R000658	Z	Thallium	ug/g	101	<1.04E+03	<9.67E+02	<1.01E+03	n/a	n/a	n/a	967	n/a	U
S09R000658	Z	Uranium	ug/g	97.4	<1.04E+03	<9.67E+02	1.01E+03	n/a	n/a	n/a	967	n/a	U
S09R000658	Z	Vanadium	ug/g	101	<52.2	<48.3	<50.4	n/a	n/a	n/a	48.3	n/a	U
S09R000658	Z	Yttrium	ug/g	97.8	<20.9	<19.3	<20.2	n/a	n/a	n/a	19.3	n/a	U
S09R000658	Z	Zinc	ug/g	98.2	<52.2	71.3	<50.4	n/a	n/a	n/a	48.3	n/a	J
S09R000188	A	Uranium-233	ug/g	n/a	<1.00E-04	<0.0177	n/a	n/a	n/a	n/a	0.0177	n/a	U
S09R000188	A	Uranium-234	ug/g	n/a	<5.00E-05	0.0506	n/a	n/a	n/a	n/a	8.84E-03	n/a	J
S09R000188	A	Uranium-235	ug/g	119	<1.10E-04	6.51	n/a	n/a	n/a	n/a	0.0194	n/a	
S09R000188	A	Uranium-236	ug/g	n/a	<4.00E-05	0.0712	n/a	n/a	n/a	n/a	7.07E-03	n/a	
S09R000188	A	Neptunium-237	ug/g	110	<5.30E-04	<0.0937	n/a	n/a	n/a	n/a	0.0937	n/a	U
S09R000188	A	Uranium-238	ug/g	114	<5.50E-03	1.00E+03	n/a	n/a	n/a	n/a	0.972	n/a	
S09R000542	A	Uranium-233	ug/g	n/a	<1.00E-04	0.0444	<0.0204	n/a	n/a	n/a	0.0208	n/a	J
S09R000542	A	Uranium-234	ug/g	n/a	<5.00E-05	0.0548	0.0345	0.0446	45.4	n/a	0.0104	n/a	J
S09R000542	A	Uranium-235	ug/g	111	<1.10E-04	6.33	5.19	5.76	19.7	86.5	0.0228	n/a	
S09R000542	A	Uranium-236	ug/g	n/a	<4.00E-05	0.0817	0.0672	0.0745	19.4	n/a	8.31E-03	n/a	J
S09R000542	A	Neptunium-237	ug/g	113	<5.30E-04	<0.110	<0.108	n/a	n/a	115	0.110	n/a	U
S09R000542	A	Uranium-238	ug/g	102	<5.50E-03	984	797	891	21.0	102	1.14	n/a	
S09R000181	F	Silver	ug/g	92.6	<124	<128	<122	n/a	n/a	n/a	128	n/a	U
S09R000181	F	Aluminum	ug/g	98.8	<744	1.04E+05	9.76E+04	1.01E+05	5.98	n/a	765	n/a	
S09R000181	F	Arsenic	ug/g	95.7	<1.24E+03	<1.28E+03	<1.22E+03	n/a	n/a	n/a	1.28E+03	n/a	U

Table A-2. Physicochemical Analysis Results for Test Composite Sample (4 sheets)

Sample Group: 20090517

Data Summary Report

Sample #	A	Analyte	Unit	Std %-Rec	Blank	Result	Duplicate	Average	RPD	Spk %-Rec	Det Limit	Count Error	Qual Flags
S09R000181	F	Boron	ug/g	96.7	<744	<765	<731	n/a	n/a	n/a	765	n/a	U
S09R000181	F	Barium	ug/g	97.0	<74.4	<76.5	<73.1	n/a	n/a	n/a	76.5	n/a	U
S09R000181	F	Beryllium	ug/g	93.9	<24.8	<25.5	<24.4	n/a	n/a	n/a	25.5	n/a	U
S09R000181	F	Bismuth	ug/g	96.2	<2.48E+03	<2.55E+03	<2.44E+03	n/a	n/a	n/a	2.55E+03	n/a	U
S09R000181	F	Calcium	ug/g	92.8	<1.24E+03	<1.28E+03	<1.22E+03	n/a	n/a	n/a	1.28E+03	n/a	U
S09R000181	F	Cadmium	ug/g	94.8	<124	<128	<122	n/a	n/a	n/a	128	n/a	U
S09R000181	F	Cerium	ug/g	101	<744	<765	<731	n/a	n/a	n/a	765	n/a	U
S09R000181	F	Cobalt	ug/g	94.5	<248	<255	250	252	2.06	n/a	255	n/a	U
S09R000181	F	Chromium	ug/g	95.9	<124	<128	<122	n/a	n/a	n/a	128	n/a	U
S09R000181	F	Copper	ug/g	98.2	128	239	<122	n/a	n/a	n/a	128	n/a	B, J
S09R000181	F	Europium	ug/g	97.3	<124	<128	<122	n/a	n/a	n/a	128	n/a	U
S09R000181	F	Iron	ug/g	95.2	266	3.14E+03	3.02E+03	3.08E+03	3.66	n/a	128	n/a	B
S09R000181	F	Lanthanum	ug/g	96.1	<74.4	<76.5	<73.1	n/a	n/a	n/a	76.5	n/a	U
S09R000181	F	Lithium	ug/g	107	<74.4	<76.5	<73.1	n/a	n/a	n/a	76.5	n/a	U
S09R000181	F	Magnesium	ug/g	98.9	<1.24E+03	<1.28E+03	<1.22E+03	n/a	n/a	n/a	1.28E+03	n/a	U
S09R000181	F	Manganese	ug/g	93.5	<74.4	<76.5	<73.1	n/a	n/a	n/a	76.5	n/a	U
S09R000181	F	Molybdenum	ug/g	93.4	<496	<510	<487	n/a	n/a	n/a	510	n/a	U
S09R000181	F	Sodium	ug/g	106	<2.48E+03	1.54E+05	1.56E+05	1.55E+05	1.49	n/a	2.55E+03	n/a	
S09R000181	F	Neodymium	ug/g	101	<248	<255	<244	n/a	n/a	n/a	255	n/a	U
S09R000181	F	Phosphorus	ug/g	96.7	<1.24E+03	4.71E+04	4.79E+04	4.75E+04	1.68	n/a	1.28E+03	n/a	
S09R000181	F	Lead	ug/g	96.9	<1.24E+03	<1.28E+03	<1.22E+03	n/a	n/a	n/a	1.28E+03	n/a	U
S09R000181	F	Sulfur	ug/g	98.8	<2.48E+03	<2.55E+03	<2.44E+03	n/a	n/a	n/a	2.55E+03	n/a	U
S09R000181	F	Antimony	ug/g	93.3	<1.24E+03	<1.28E+03	<1.22E+03	n/a	n/a	n/a	1.28E+03	n/a	U
S09R000181	F	Selenium	ug/g	98.4	<2.48E+03	<2.55E+03	<2.44E+03	n/a	n/a	n/a	2.55E+03	n/a	U
S09R000181	F	Silicon	ug/g	91.4	<744	9.02E+02	1.01E+03	9.58E+02	11.8	n/a	765	n/a	J
S09R000181	F	Samarium	ug/g	102	<1.24E+03	<1.28E+03	<1.22E+03	n/a	n/a	n/a	1.28E+03	n/a	U
S09R000181	F	Strontium	ug/g	97.1	<74.4	568	503	535	12.2	n/a	76.5	n/a	J
S09R000181	F	Thorium	ug/g	98.0	<1.24E+03	<1.28E+03	<1.22E+03	n/a	n/a	n/a	1.28E+03	n/a	U
S09R000181	F	Titanium	ug/g	94.5	<124	<128	<122	n/a	n/a	n/a	128	n/a	U

Table A-2. Physicochemical Analysis Results for Test Composite Sample (4 sheets)

Sample Group: 20090517

Data Summary Report

Sample #	A	Analyte	Unit	Std %-Rec	Blank	Result	Duplicate	Average	RPD	Spk %-Rec	Det Limit	Count Error	Qual Flags
S09R000181	F	Thallium	ug/g	97.8	<2.48E+03	<2.55E+03	<2.44E+03	n/a	n/a	n/a	2.55E+03	n/a	U
S09R000181	F	Uranium	ug/g	100	<2.48E+03	<2.55E+03	<2.44E+03	n/a	n/a	n/a	2.55E+03	n/a	U
S09R000181	F	Vanadium	ug/g	96.3	<124	<128	<122	n/a	n/a	n/a	128	n/a	U
S09R000181	F	Yttrium	ug/g	98.4	<49.6	<51.0	<48.7	n/a	n/a	n/a	51.0	n/a	U
S09R000181	F	Zinc	ug/g	95.4	<124	245	<122	n/a	n/a	n/a	128	n/a	J
S09R000181	F	Zirconium	ug/g	93.6	<124	<128	<122	n/a	n/a	n/a	128	n/a	U
S09R000185	W	Chloride	ug/g	99.1	<3.10E-03	<29.3	n/a	n/a	n/a	n/a	29.3	n/a	U
S09R000185	W	Nitrite	ug/g	96.6	0.0580	7.93E+03	n/a	n/a	n/a	n/a	378	n/a	
S09R000185	W	Bromide	ug/g	98.7	<0.0237	<224	n/a	n/a	n/a	n/a	224	n/a	U
S09R000185	W	Nitrate	ug/g	99.3	0.0222	7.50E+03	n/a	n/a	n/a	n/a	153	n/a	
S09R000185	W	Phosphate	ug/g	101	<0.0381	1.57E+05	n/a	n/a	n/a	n/a	360	n/a	
S09R000185	W	Sulfate	ug/g	101	<0.0219	1.71E+03	n/a	n/a	n/a	n/a	207	n/a	J
S09R000185	W	Oxalate	ug/g	99.2	<0.105	1.57E+03	n/a	n/a	n/a	n/a	992	n/a	J
S09R000185	W	Fluoride	ug/g	98.0	<1.61E-03	1.54E+04	n/a	n/a	n/a	n/a	15.2	n/a	
S09R000185	W	Glycolate	ug/g	101	<9.37E-03	<88.5	n/a	n/a	n/a	n/a	88.5	n/a	U
S09R000185	W	Acetate	ug/g	104	0.0597	<57.1	n/a	n/a	n/a	n/a	57.1	n/a	U
S09R000185	W	Formate	ug/g	103	0.0590	<44.1	n/a	n/a	n/a	n/a	44.1	n/a	U
S09R000541	W	Fluoride	ug/g	95.8	<0.0821	1.58E+04	1.57E+04	1.57E+04	0.439	110	15.5	n/a	
S09R000541	W	Glycolate	ug/g	97.6	<0.103	<19.5	<19.7	n/a	n/a	96.5	19.5	n/a	U
S09R000541	W	Acetate	ug/g	99.9	<0.0664	392	318	355	20.7	100	12.6	n/a	J
S09R000541	W	Formate	ug/g	98.9	<0.0514	36.1	41.2	38.7	13.2	102	9.72	n/a	J
S09R000541	W	Chloride	ug/g	96.1	<0.110	74.7	82.2	78.4	9.49	98.3	20.8	n/a	
S09R000541	W	Nitrite	ug/g	99.6	<0.211	8.38E+03	8.64E+03	8.51E+03	3.01	99.3	40.0	n/a	
S09R000541	W	Sulfate	ug/g	99.1	<0.206	1.18E+03	1.25E+03	1.22E+03	5.55	101	38.9	n/a	
S09R000541	W	Oxalate	ug/g	98.2	<0.254	880	801	841	9.50	99.4	48.1	n/a	
S09R000541	W	Bromide	ug/g	96.2	<0.638	<121	<122	n/a	n/a	94.5	121	n/a	U
S09R000541	W	Nitrate	ug/g	97.4	<0.229	7.38E+03	7.60E+03	7.49E+03	2.99	97.4	43.3	n/a	
S09R000541	W	Phosphate	ug/g	97.3	<0.852	1.59E+05	1.59E+05	1.59E+05	0.277	111	161	n/a	

Table A-3. Radiological Analysis Results for Test Composite Sample (4 sheets)

Sample Group: 20090517 Data Summary Report

Sample #	A	Analyte	Unit	Std %-Rec	Blank	Result	Duplicate	Average	RPD	Spk %-Rec	Det Limit	Count Error	Qual Flags
S09R000145		Iodine-129	uCi/g	111	<8.00E-06	<1.00E-05	<8.93E-06	n/a	n/a	n/a	1.00E-05	n/a	U
S09R000145		Iodine-C	%	n/a	80.1	66.6	78.8	n/a	n/a	n/a	n/a	n/a	
S09R000188	A	Technetium-99	uCi/g	102	<4.90E-03	<4.03E-03	n/a	n/a	n/a	n/a	4.03E-03	n/a	U
S09R000188	A	Technetium-99T	%	n/a	56.8	59.2	n/a	n/a	n/a	n/a	n/a	n/a	
S09R000542	A	Technetium-99	uCi/g	104	<4.80E-04	5.88E-03	3.21E-03	4.55E-03	58.8	99.2	5.57E-04	16.2	
S09R000542	A	Technetium-99T	%	n/a	61.1	58.3	58.1	n/a	n/a	n/a	n/a	n/a	
S09R000181	F	Curium-243/244	uCi/g	n/a	<5.62E-03	<7.36E-04	<5.04E-04	n/a	n/a	n/a	7.36E-04	n/a	U
S09R000181	F	Americium-241	uCi/g	104	<0.0141	1.56E-02	9.42E-03	1.25E-02	49.6	n/a	1.84E-03	3.87	c
S09R000181	F	Americium-243T	%	n/a	95.414	81.605	90.929	n/a	n/a	n/a	n/a	n/a	
S09R000181	F	Curium-242	uCi/g	n/a	<5.62E-03	<7.36E-04	<5.04E-04	n/a	n/a	n/a	7.36E-04	n/a	U
S09R000181	F	Beryllium-7	uCi/g	n/a	<1.04	<0.660	<0.624	n/a	n/a	n/a	0.660	n/a	U
S09R000181	F	Sodium-22	uCi/g	n/a	<0.160	<0.0536	<0.0511	n/a	n/a	n/a	0.0536	n/a	U
S09R000181	F	Sodium-24	uCi/g	n/a	<0.148	<0.0521	<0.0465	n/a	n/a	n/a	0.0521	n/a	U
S09R000181	F	Aluminum-28	uCi/g	n/a	<5.35	<0.809	<0.743	n/a	n/a	n/a	0.809	n/a	U
S09R000181	F	Chlorine-38	uCi/g	n/a	<0.980	<0.264	<0.244	n/a	n/a	n/a	0.264	n/a	U
S09R000181	F	Potassium-40	uCi/g	n/a	<3.65	<0.529	<0.500	n/a	n/a	n/a	0.529	n/a	U
S09R000181	F	Argon-41	uCi/g	n/a	<0.230	<0.0828	<0.0761	n/a	n/a	n/a	0.0828	n/a	U
S09R000181	F	Scandium-46	uCi/g	n/a	<0.193	<0.0599	<0.0576	n/a	n/a	n/a	0.0599	n/a	U
S09R000181	F	Chromium-51	uCi/g	n/a	<0.968	<0.457	<0.433	n/a	n/a	n/a	0.457	n/a	U
S09R000181	F	Manganese-54	uCi/g	n/a	<0.139	<0.0569	<0.0537	n/a	n/a	n/a	0.0569	n/a	U
S09R000181	F	Cobalt-56	uCi/g	n/a	<0.145	<0.0574	<0.0546	n/a	n/a	n/a	0.0574	n/a	U
S09R000181	F	Manganese-56	uCi/g	n/a	<0.200	<0.0796	<0.0755	n/a	n/a	n/a	0.0796	n/a	U
S09R000181	F	Cobalt-57	uCi/g	n/a	<0.0647	<0.0315	<0.0302	n/a	n/a	n/a	0.0315	n/a	U
S09R000181	F	Cobalt-58	uCi/g	n/a	<0.140	<0.0576	<0.0534	n/a	n/a	n/a	0.0576	n/a	U
S09R000181	F	Iron-59	uCi/g	n/a	<0.302	<0.107	<0.0992	n/a	n/a	n/a	0.107	n/a	U
S09R000181	F	Cobalt-60	uCi/g	102	<0.153	<0.0510	<0.0475	n/a	n/a	n/a	0.0510	n/a	U
S09R000181	F	Copper-64	uCi/g	n/a	<31.6	<11.3	<10.8	n/a	n/a	n/a	11.3	n/a	U
S09R000181	F	Zinc-65	uCi/g	n/a	<0.368	<0.121	<0.114	n/a	n/a	n/a	0.121	n/a	U

Table A-3. Radiological Analysis Results for Test Composite Sample (4 sheets)

Sample Group: 20090517 Data Summary Report

Sample #	A	Analyte	Unit	Std %-Rec	Blank	Result	Duplicate	Average	RPD	Spk %-Rec	Det Limit	Count Error	Qual Flags
S09R000181	F	Copper-66	uCi/g	n/a	<31.3	<12.8	<11.9	n/a	n/a	n/a	12.8	n/a	U
S09R000181	F	Selenium-75	uCi/g	n/a	<0.146	<0.0684	<0.0654	n/a	n/a	n/a	0.0684	n/a	U
S09R000181	F	Selenium-79	uCi/g	n/a	<14.8	<7.40	<7.19	n/a	n/a	n/a	7.40	n/a	U
S09R000181	F	Krypton-85	uCi/g	n/a	<38.0	<14.4	<13.7	n/a	n/a	n/a	14.4	n/a	U
S09R000181	F	Strontium-85	uCi/g	n/a	<0.165	<0.0628	<0.0594	n/a	n/a	n/a	0.0628	n/a	U
S09R000181	F	Yttrium-88	uCi/g	n/a	<0.128	<0.0147	<0.0156	n/a	n/a	n/a	0.0147	n/a	U
S09R000181	F	Yttrium-91	uCi/g	n/a	<59.0	<19.6	<18.2	n/a	n/a	n/a	19.6	n/a	U
S09R000181	F	Niobium-94	uCi/g	n/a	<0.138	<0.0572	<0.0528	n/a	n/a	n/a	0.0572	n/a	U
S09R000181	F	Zirconium/Niobium-95	uCi/g	n/a	<0.293	<0.113	<0.104	n/a	n/a	n/a	0.113	n/a	U
S09R000181	F	Ruthenium-103	uCi/g	n/a	<0.131	<0.0678	<0.0648	n/a	n/a	n/a	0.0678	n/a	U
S09R000181	F	Ruthenium/Rhodium-106	uCi/g	n/a	<2.38	<1.11	<1.06	n/a	n/a	n/a	1.11	n/a	U
S09R000181	F	Silver-108	uCi/g	n/a	<0.151	<0.0608	<0.0567	n/a	n/a	n/a	0.0608	n/a	U
S09R000181	F	Cadmium-109	uCi/g	n/a	<1.46	<0.740	<0.707	n/a	n/a	n/a	0.740	n/a	U
S09R000181	F	Silver-110	uCi/g	n/a	<0.174	<0.286	<0.271	n/a	n/a	n/a	0.286	n/a	U
S09R000181	F	Tin-113	uCi/g	n/a	<0.158	<0.0828	<0.0773	n/a	n/a	n/a	0.0828	n/a	U
S09R000181	F	Tellurium-123	uCi/g	n/a	<0.0804	<0.0362	<0.0344	n/a	n/a	n/a	0.0362	n/a	U
S09R000181	F	Antimony-124	uCi/g	n/a	<0.128	<0.0557	<0.0526	n/a	n/a	n/a	0.0557	n/a	U
S09R000181	F	Antimony-125	uCi/g	n/a	<0.383	<0.215	<0.202	n/a	n/a	n/a	0.215	n/a	U
S09R000181	F	Tellurium-125	uCi/g	n/a	<0.132	<0.106	<0.102	n/a	n/a	n/a	0.106	n/a	U
S09R000181	F	Antimony-126	uCi/g	n/a	<0.129	<0.0517	<0.0484	n/a	n/a	n/a	0.0517	n/a	U
S09R000181	F	Tin-126	uCi/g	n/a	<0.119	<0.0599	<0.0576	n/a	n/a	n/a	0.0599	n/a	U
S09R000181	F	Iodine-129	uCi/g	n/a	<0.829	<0.678	<0.654	n/a	n/a	n/a	0.678	n/a	U
S09R000181	F	Iodine-131	uCi/g	n/a	<0.117	<0.0613	<0.0585	n/a	n/a	n/a	0.0613	n/a	U
S09R000181	F	Xenon-131	uCi/g	n/a	<3.51	<1.57	<1.50	n/a	n/a	n/a	1.57	n/a	U
S09R000181	F	Barium-133	uCi/g	n/a	<0.186	<0.0815	<0.0778	n/a	n/a	n/a	0.0815	n/a	U
S09R000181	F	Cesium-134	uCi/g	n/a	<0.159	<0.0569	<0.0532	n/a	n/a	n/a	0.0569	n/a	U
S09R000181	F	Cesium-136	uCi/g	n/a	<0.135	<0.0579	<0.0525	n/a	n/a	n/a	0.0579	n/a	U
S09R000181	F	Cesium-137	uCi/g	106	<0.191	19.6	18.4	19.0	6.07	n/a	0.0610	4.28	
S09R000181	F	Cesium-138	uCi/g	n/a	<0.617	<0.197	<0.175	n/a	n/a	n/a	0.197	n/a	U

Table A-3. Radiological Analysis Results for Test Composite Sample (4 sheets)

Sample Group: 20090517

Data Summary Report

Sample #	A	Analyte	Unit	Std %-Rec	Blank	Result	Duplicate	Average	RPD	Spk %-Rec	Det Limit	Count Error	Qual Flags
S09R000181	F	Cerium-139	uCi/g	n/a	<0.0877	<0.0381	<0.0365	n/a	n/a	n/a	0.0381	n/a	U
S09R000181	F	Barium-140	uCi/g	n/a	<0.495	<0.234	<0.223	n/a	n/a	n/a	0.234	n/a	U
S09R000181	F	Lanthanum-140	uCi/g	n/a	<0.158	<0.0358	<0.0314	n/a	n/a	n/a	0.0358	n/a	U
S09R000181	F	Cerium-141	uCi/g	n/a	<0.120	<0.0578	<0.0550	n/a	n/a	n/a	0.0578	n/a	U
S09R000181	F	Cerium-144	uCi/g	n/a	<0.520	<0.248	<0.237	n/a	n/a	n/a	0.248	n/a	U
S09R000181	F	Cerium/Praseodymium-144	uCi/g	n/a	<1.04	<0.496	<0.473	n/a	n/a	n/a	0.496	n/a	U
S09R000181	F	Europium-152	uCi/g	n/a	<0.744	<0.223	<0.206	n/a	n/a	n/a	0.223	n/a	U
S09R000181	F	Europium-154	uCi/g	n/a	<0.463	<0.156	<0.149	n/a	n/a	n/a	0.156	n/a	U
S09R000181	F	Europium-155	uCi/g	n/a	<0.244	<0.123	<0.116	n/a	n/a	n/a	0.123	n/a	U
S09R000181	F	Hafnium-181	uCi/g	n/a	<0.141	<0.0809	<0.0767	n/a	n/a	n/a	0.0809	n/a	U
S09R000181	F	Tantalum-182	uCi/g	n/a	<0.554	<0.172	<0.165	n/a	n/a	n/a	0.172	n/a	U
S09R000181	F	Gold-198	uCi/g	n/a	<0.112	<0.0605	<0.0570	n/a	n/a	n/a	0.0605	n/a	U
S09R000181	F	Mercury-203	uCi/g	n/a	<0.121	<0.0544	<0.0519	n/a	n/a	n/a	0.0544	n/a	U
S09R000181	F	Bismuth-207	uCi/g	n/a	<0.181	<0.0772	<0.0719	n/a	n/a	n/a	0.0772	n/a	U
S09R000181	F	Thallium-208	uCi/g	n/a	<0.164	<0.0653	<0.0612	n/a	n/a	n/a	0.0653	n/a	U
S09R000181	F	Lead-210	uCi/g	n/a	<1.38	<0.958	<0.921	n/a	n/a	n/a	0.958	n/a	U
S09R000181	F	Bismuth-212	uCi/g	n/a	<1.16	<0.461	<0.439	n/a	n/a	n/a	0.461	n/a	U
S09R000181	F	Lead-212	uCi/g	n/a	<0.217	<0.0896	<0.0844	n/a	n/a	n/a	0.0896	n/a	U
S09R000181	F	Bismuth-214	uCi/g	n/a	<0.341	<0.121	<0.113	n/a	n/a	n/a	0.121	n/a	U
S09R000181	F	Lead-214	uCi/g	n/a	<0.304	<0.131	<0.125	n/a	n/a	n/a	0.131	n/a	U
S09R000181	F	Radium-224	uCi/g	n/a	<2.46	<1.02	<0.969	n/a	n/a	n/a	1.02	n/a	U
S09R000181	F	Radium-226	uCi/g	n/a	<2.30	<1.06	<1.01	n/a	n/a	n/a	1.06	n/a	U
S09R000181	F	Actinium-228	uCi/g	n/a	<0.573	<0.208	<0.198	n/a	n/a	n/a	0.208	n/a	U
S09R000181	F	Thorium-228	uCi/g	n/a	<4.16	<2.18	<2.09	n/a	n/a	n/a	2.18	n/a	U
S09R000181	F	Thorium-229	uCi/g	n/a	<0.451	<0.236	<0.228	n/a	n/a	n/a	0.236	n/a	U
S09R000181	F	Uranium-232	uCi/g	n/a	<24.0	<15.6	<15.0	n/a	n/a	n/a	15.6	n/a	U
S09R000181	F	Protactinium-233	uCi/g	n/a	<0.250	<0.115	<0.109	n/a	n/a	n/a	0.115	n/a	U
S09R000181	F	Uranium/Thorium-233	uCi/g	n/a	<82.9	<36.5	<34.7	n/a	n/a	n/a	36.5	n/a	U
S09R000181	F	Protactinium-234	uCi/g	n/a	<22.7	<8.77	<8.26	n/a	n/a	n/a	8.77	n/a	U

Table A-3. Radiological Analysis Results for Test Composite Sample (4 sheets)

Sample Group: 20090517 | Data Summary Report

Sample #	A	Analyte	Unit	Std %-Rec	Blank	Result	Duplicate	Average	RPD	Spk %-Rec	Det Limit	Count Error	Qual Flags
S09R000181	F	Thorium-234	uCi/g	n/a	<1.33	<0.772	<0.743	n/a	n/a	n/a	0.772	n/a	U
S09R000181	F	Uranium-235	uCi/g	n/a	<0.141	<0.0641	<0.0606	n/a	n/a	n/a	0.0641	n/a	U
S09R000181	F	Neptunium-237	uCi/g	n/a	<0.434	<0.222	<0.213	n/a	n/a	n/a	0.222	n/a	U
S09R000181	F	Uranium-237	uCi/g	n/a	<0.201	<0.105	<0.102	n/a	n/a	n/a	0.105	n/a	U
S09R000181	F	Neptunium-238	uCi/g	n/a	<0.505	<0.209	<0.195	n/a	n/a	n/a	0.209	n/a	U
S09R000181	F	Neptunium-239	uCi/g	n/a	<0.235	<0.118	<0.112	n/a	n/a	n/a	0.118	n/a	U
S09R000181	F	Plutonium-239	uCi/g	n/a	<895	<437	<421	n/a	n/a	n/a	437	n/a	U
S09R000181	F	Americium-241	uCi/g	n/a	<0.132	<0.0852	<0.0820	n/a	n/a	n/a	0.0852	n/a	U
S09R000181	F	Americium-243	uCi/g	n/a	<0.129	<0.0484	<0.0464	n/a	n/a	n/a	0.0484	n/a	U
S09R000181	F	Plutonium-239/240	uCi/g	98.0	<0.0129	1.15E-02	9.43E-03	1.05E-02	19.9	n/a	9.46E-04	3.99	
S09R000181	F	Plutonium-236T	%	n/a	67.1	89.1	94.0	n/a	n/a	n/a	n/a	n/a	
S09R000181	F	Plutonium-238	uCi/g	n/a	<0.0129	1.18E-03	<7.95E-04	n/a	n/a	n/a	9.46E-04	9.65	
S09R000181	F	Strontium-89/90	uCi/g	104	<1.37	173	175	174	1.44	n/a	0.0140	0.44	
S09R000181	F	Strontium-C	%	n/a	77.5	76.7	76.2	n/a	n/a	n/a	n/a	n/a	

Table A-4. Physicochemical Analysis Results for Bulk Density Liquid Sample (2 sheets)

Sample Group: 20090517 Data Summary Report

Sample #	A	Analyte	Unit	Std %-Rec	Blank	Result	Duplicate	Average	RPD	Spk %-Rec	Det Limit	Count Error	Qual Flags
S09R000146		Fluoride	ug/mL	99.5	<1.61E-03	2.23E+03	2.24E+03	2.23E+03	0.685	n/a	3.41	n/a	
S09R000146		Glycolate	ug/mL	100	<9.37E-03	<19.9	<19.9	n/a	n/a	n/a	19.9	n/a	U
S09R000146		Acetate	ug/mL	102	<6.04E-03	166	114	140	37.3	n/a	12.8	n/a	J
S09R000146		Formate	ug/mL	102	<4.67E-03	77.3	57.5	67.4	29.4	n/a	9.91	n/a	J
S09R000146		Chloride	ug/mL	99.5	<9.98E-03	109	108	109	0.893	n/a	21.2	n/a	J
S09R000146		Nitrite	ug/mL	101	<0.0192	1.16E+04	1.17E+04	1.17E+04	0.570	n/a	40.7	n/a	
S09R000146		Sulfate	ug/mL	101	<0.0187	1.37E+03	1.39E+03	1.38E+03	1.32	n/a	39.7	n/a	
S09R000146		Oxalate	ug/mL	101	<0.0231	1.30E+03	1.29E+03	1.29E+03	0.492	n/a	49.0	n/a	
S09R000146		Bromide	ug/mL	100	<0.0580	<123	<123	n/a	n/a	n/a	123	n/a	U
S09R000146		Nitrate	ug/mL	99.9	<0.0208	1.36E+04	1.37E+04	1.36E+04	0.860	n/a	44.1	n/a	
S09R000146		Phosphate	ug/mL	99.7	<0.0167	2.50E+04	2.52E+04	2.51E+04	1.03	n/a	35.4	n/a	
S09R000146		Silver	ug/mL	95.1	<5.00E-03	<2.00	<2.00	n/a	n/a	95.8	2.00	n/a	U
S09R000146		Aluminum	ug/mL	99.0	<0.0300	105	103	104	1.55	97.1	12.0	n/a	J
S09R000146		Arsenic	ug/mL	100	<0.0500	<20.0	<20.0	n/a	n/a	103	20.0	n/a	U
S09R000146		Boron	ug/mL	98.8	<0.0300	<12.0	<12.0	n/a	n/a	99.3	12.0	n/a	U
S09R000146		Barium	ug/mL	99.9	<3.00E-03	<1.20	<1.20	n/a	n/a	98.8	1.20	n/a	U
S09R000146		Beryllium	ug/mL	101	<1.00E-03	<0.400	<0.400	n/a	n/a	98.8	0.400	n/a	U
S09R000146		Bismuth	ug/mL	99.5	<0.100	<40.0	<40.0	n/a	n/a	98.6	40.0	n/a	U
S09R000146		Calcium	ug/mL	97.8	<0.0500	<20.0	<20.0	n/a	n/a	95.2	20.0	n/a	U
S09R000146		Cadmium	ug/mL	101	<5.00E-03	<2.00	<2.00	n/a	n/a	99.5	2.00	n/a	U
S09R000146		Cerium	ug/mL	105	<0.0300	<12.0	<12.0	n/a	n/a	97.8	12.0	n/a	U
S09R000146		Cobalt	ug/mL	102	<0.0100	<4.00	<4.00	n/a	n/a	100	4.00	n/a	U
S09R000146		Chromium	ug/mL	102	<5.00E-03	19.1	20.2	19.6	5.55	102	2.00	n/a	J
S09R000146		Copper	ug/mL	99.7	<5.00E-03	<2.00	<2.00	n/a	n/a	97.6	2.00	n/a	U
S09R000146		Europium	ug/mL	97.8	<5.00E-03	<2.00	<2.00	n/a	n/a	n/a	2.00	n/a	U
S09R000146		Iron	ug/mL	101	<5.00E-03	44.4	43.7	44.1	1.63	98.7	2.00	n/a	
S09R000146		Potassium	ug/mL	96.3	<0.500	<200	<200	n/a	n/a	90.7	200	n/a	U
S09R000146		Lanthanum	ug/mL	99.4	<3.00E-03	<1.20	<1.20	n/a	n/a	97.8	1.20	n/a	U

Table A-4. Physicochemical Analysis Results for Bulk Density Liquid Sample (2 sheets)

Sample Group: 20090517

Data Summary Report

Sample #	A	Analyte	Unit	Std %-Rec	Blank	Result	Duplicate	Average	RPD	Spk %-Rec	Det Limit	Count Error	Qual Flags
S09R000146		Lithium	ug/mL	101	<3.00E-03	<1.20	<1.20	n/a	n/a	90.8	1.20	n/a	U
S09R000146		Magnesium	ug/mL	101	<0.0500	<20.0	<20.0	n/a	n/a	94.9	20.0	n/a	U
S09R000146		Manganese	ug/mL	100	<3.00E-03	<1.20	<1.20	n/a	n/a	101	1.20	n/a	U
S09R000146		Molybdenum	ug/mL	101	<0.0200	<8.00	<8.00	n/a	n/a	98.9	8.00	n/a	U
S09R000146		Sodium	ug/mL	99.1	<0.100	4.85E+04	4.63E+04	4.74E+04	4.76	75.6	40.0	n/a	
S09R000146		Neodymium	ug/mL	98.1	<0.0100	<4.00	<4.00	n/a	n/a	94.3	4.00	n/a	U
S09R000146		Nickel	ug/mL	101	<0.0200	<8.00	<8.00	n/a	n/a	98.5	8.00	n/a	U
S09R000146		Phosphorus	ug/mL	101	<0.0500	8.56E+03	8.51E+03	8.53E+03	0.514	95.8	20.0	n/a	
S09R000146		Lead	ug/mL	105	<0.0500	<20.0	<20.0	n/a	n/a	100	20.0	n/a	U
S09R000146		Sulfur	ug/mL	101	<0.100	506	491	498	2.93	100	40.0	n/a	
S09R000146		Antimony	ug/mL	97.4	<0.0500	<20.0	<20.0	n/a	n/a	95.8	20.0	n/a	U
S09R000146		Selenium	ug/mL	103	<0.100	<40.0	<40.0	n/a	n/a	112	40.0	n/a	U
S09R000146		Silicon	ug/mL	94.6	<0.0300	<12.0	<12.0	n/a	n/a	90.8	12.0	n/a	U
S09R000146		Samarium	ug/mL	100	<0.0500	<20.0	<20.0	n/a	n/a	96.6	20.0	n/a	U
S09R000146		Strontium	ug/mL	100	<3.00E-03	8.81	8.70	8.75	1.23	98.0	1.20	n/a	J
S09R000146		Thorium	ug/mL	102	<0.0500	25.5	25.8	25.7	1.08	105	20.0	n/a	J
S09R000146		Titanium	ug/mL	98.9	<5.00E-03	<2.00	<2.00	n/a	n/a	97.7	2.00	n/a	U
S09R000146		Thallium	ug/mL	104	<0.100	<40.0	<40.0	n/a	n/a	94.6	40.0	n/a	U
S09R000146		Uranium	ug/mL	101	<0.100	1.09E+03	1.09E+03	1.09E+03	0.470	93.2	40.0	n/a	
S09R000146		Vanadium	ug/mL	103	<5.00E-03	<2.00	<2.00	n/a	n/a	101	2.00	n/a	U
S09R000146		Yttrium	ug/mL	99.8	<2.00E-03	<0.800	<0.800	n/a	n/a	n/a	0.800	n/a	U
S09R000146		Zinc	ug/mL	99.6	<5.00E-03	<2.00	<2.00	n/a	n/a	99.8	2.00	n/a	U
S09R000146		Zirconium	ug/mL	94.6	<5.00E-03	<2.00	<2.00	n/a	n/a	91.0	2.00	n/a	U
S09R000146		Specific gravity	unitless	99.59	n/a	1.138	n/a	n/a	n/a	n/a	1.000E-03	n/a	
S09R000146		Total organic carbon	ug/mL	91.6	<20.0	464	501	482	7.67	101	80.0	n/a	J
S09R000146		Total inorganic carbon	ug/mL	97.0	<7.00	6.89E+03	6.85E+03	6.87E+03	0.582	96.5	28.0	n/a	

Table A-5. Physicochemical Analysis Results for Coarse Sieving Liquid Sample (2 sheets)

Sample Group: 20090517 Data Summary Report

Sample #	A	Analyte	Unit	Std %-Rec	Blank	Result	Duplicate	Average	RPD	Spk %-Rec	Det Limit	Count Error	Qual Flags
S09R000148		Fluoride	ug/mL	99.5	<1.61E-03	2.07E+03	n/a	n/a	n/a	n/a	1.79	n/a	
S09R000148		Glycolate	ug/mL	100	<9.37E-03	<10.4	n/a	n/a	n/a	n/a	10.4	n/a	U
S09R000148		Acetate	ug/mL	102	<6.04E-03	134	n/a	n/a	n/a	n/a	6.71	n/a	J
S09R000148		Formate	ug/mL	102	<4.67E-03	52.5	n/a	n/a	n/a	n/a	5.19	n/a	J
S09R000148		Chloride	ug/mL	99.5	<9.98E-03	109	n/a	n/a	n/a	n/a	11.1	n/a	J
S09R000148		Nitrite	ug/mL	101	<0.0192	1.18E+04	n/a	n/a	n/a	n/a	21.3	n/a	
S09R000148		Sulfate	ug/mL	101	<0.0187	1.50E+03	n/a	n/a	n/a	n/a	20.8	n/a	
S09R000148		Oxalate	ug/mL	101	<0.0231	1.60E+03	n/a	n/a	n/a	n/a	25.7	n/a	
S09R000148		Bromide	ug/mL	100	<0.0580	<64.4	n/a	n/a	n/a	n/a	64.4	n/a	U
S09R000148		Nitrate	ug/mL	99.9	<0.0208	1.37E+04	n/a	n/a	n/a	n/a	23.1	n/a	
S09R000148		Phosphate	ug/mL	99.7	<0.0167	2.36E+04	n/a	n/a	n/a	n/a	18.6	n/a	
S09R000148		Silver	ug/mL	95.1	<5.00E-03	<2.00	n/a	n/a	n/a	n/a	2.00	n/a	U
S09R000148		Aluminum	ug/mL	99.0	<0.0300	112	n/a	n/a	n/a	n/a	12.0	n/a	J
S09R000148		Arsenic	ug/mL	100	<0.0500	<20.0	n/a	n/a	n/a	n/a	20.0	n/a	U
S09R000148		Boron	ug/mL	98.8	<0.0300	<12.0	n/a	n/a	n/a	n/a	12.0	n/a	U
S09R000148		Barium	ug/mL	99.9	<3.00E-03	<1.20	n/a	n/a	n/a	n/a	1.20	n/a	U
S09R000148		Beryllium	ug/mL	101	<1.00E-03	<0.400	n/a	n/a	n/a	n/a	0.400	n/a	U
S09R000148		Bismuth	ug/mL	99.5	<0.100	<40.0	n/a	n/a	n/a	n/a	40.0	n/a	U
S09R000148		Calcium	ug/mL	97.8	<0.0500	<20.0	n/a	n/a	n/a	n/a	20.0	n/a	U
S09R000148		Cadmium	ug/mL	101	<5.00E-03	<2.00	n/a	n/a	n/a	n/a	2.00	n/a	U
S09R000148		Cerium	ug/mL	105	<0.0300	<12.0	n/a	n/a	n/a	n/a	12.0	n/a	U
S09R000148		Cobalt	ug/mL	102	<0.0100	<4.00	n/a	n/a	n/a	n/a	4.00	n/a	U
S09R000148		Chromium	ug/mL	102	<5.00E-03	19.8	n/a	n/a	n/a	n/a	2.00	n/a	J
S09R000148		Copper	ug/mL	99.7	<5.00E-03	<2.00	n/a	n/a	n/a	n/a	2.00	n/a	U
S09R000148		Europium	ug/mL	97.8	<5.00E-03	<2.00	n/a	n/a	n/a	n/a	2.00	n/a	U
S09R000148		Iron	ug/mL	101	<5.00E-03	45.2	n/a	n/a	n/a	n/a	2.00	n/a	
S09R000148		Potassium	ug/mL	96.3	<0.500	<200	n/a	n/a	n/a	n/a	200	n/a	U
S09R000148		Lanthanum	ug/mL	99.4	<3.00E-03	<1.20	n/a	n/a	n/a	n/a	1.20	n/a	U

Table A-5. Physicochemical Analysis Results for Coarse Sieving Liquid Sample (2 sheets)

Sample Group: 20090517

Data Summary Report

Sample #	A	Analyte	Unit	Std %-Rec	Blank	Result	Duplicate	Average	RPD	Spk %-Rec	Det Limit	Count Error	Qual Flags
S09R000148		Lithium	ug/mL	101	<3.00E-03	<1.20	n/a	n/a	n/a	n/a	1.20	n/a	U
S09R000148		Magnesium	ug/mL	101	<0.0500	<20.0	n/a	n/a	n/a	n/a	20.0	n/a	U
S09R000148		Manganese	ug/mL	100	<3.00E-03	<1.20	n/a	n/a	n/a	n/a	1.20	n/a	U
S09R000148		Molybdenum	ug/mL	101	<0.0200	<8.00	n/a	n/a	n/a	n/a	8.00	n/a	U
S09R000148		Sodium	ug/mL	99.1	<0.100	4.72E+04	n/a	n/a	n/a	n/a	40.0	n/a	
S09R000148		Neodymium	ug/mL	98.1	<0.0100	<4.00	n/a	n/a	n/a	n/a	4.00	n/a	U
S09R000148		Nickel	ug/mL	101	<0.0200	<8.00	n/a	n/a	n/a	n/a	8.00	n/a	U
S09R000148		Phosphorus	ug/mL	101	<0.0500	8.00E+03	n/a	n/a	n/a	n/a	20.0	n/a	
S09R000148		Lead	ug/mL	105	<0.0500	23.4	n/a	n/a	n/a	n/a	20.0	n/a	J
S09R000148		Sulfur	ug/mL	101	<0.100	506	n/a	n/a	n/a	n/a	40.0	n/a	
S09R000148		Antimony	ug/mL	97.4	<0.0500	<20.0	n/a	n/a	n/a	n/a	20.0	n/a	U
S09R000148		Selenium	ug/mL	103	<0.100	<40.0	n/a	n/a	n/a	n/a	40.0	n/a	U
S09R000148		Silicon	ug/mL	94.6	<0.0300	<12.0	n/a	n/a	n/a	n/a	12.0	n/a	U
S09R000148		Samarium	ug/mL	100	<0.0500	<20.0	n/a	n/a	n/a	n/a	20.0	n/a	U
S09R000148		Strontium	ug/mL	100	<3.00E-03	10.8	n/a	n/a	n/a	n/a	1.20	n/a	J
S09R000148		Thorium	ug/mL	102	<0.0500	23.3	n/a	n/a	n/a	n/a	20.0	n/a	J
S09R000148		Titanium	ug/mL	98.9	<5.00E-03	<2.00	n/a	n/a	n/a	n/a	2.00	n/a	U
S09R000148		Thallium	ug/mL	104	<0.100	<40.0	n/a	n/a	n/a	n/a	40.0	n/a	U
S09R000148		Uranium	ug/mL	101	<0.100	1.12E+03	n/a	n/a	n/a	n/a	40.0	n/a	
S09R000148		Vanadium	ug/mL	103	<5.00E-03	<2.00	n/a	n/a	n/a	n/a	2.00	n/a	U
S09R000148		Yttrium	ug/mL	99.8	<2.00E-03	<0.800	n/a	n/a	n/a	n/a	0.800	n/a	U
S09R000148		Zinc	ug/mL	99.6	<5.00E-03	<2.00	n/a	n/a	n/a	n/a	2.00	n/a	U
S09R000148		Zirconium	ug/mL	94.6	<5.00E-03	<2.00	n/a	n/a	n/a	n/a	2.00	n/a	U
S09R000148		Specific gravity	unitless	99.59	n/a	1.126	1.127	1.126	0.08877	n/a	1.000E-03	n/a	
S09R000148		Total organic carbon	ug/mL	91.6	<20.0	566	n/a	n/a	n/a	n/a	80.0	n/a	J
S09R000148		Total inorganic carbon	ug/mL	97.0	<7.00	6.93E+03	n/a	n/a	n/a	n/a	28.0	n/a	

Table A-6. Physicochemical Analysis Results for Caustic Dissolution Residue Sample (4 sheets)

Sample Group: 20090517

Data Summary Report

Sample #	A	Analyte	Unit	Std %-Rec	Blank	Result	Duplicate	Average	RPD	Spk %-Rec	Det Limit	Count Error	Qual Flags
S09R000174		pH	unitless	n/a	n/a	9.89	9.94	9.92	0.504	n/a	0.0100	n/a	
S09R000174		Percent water	%	99.8	n/a	68.8	69.9	69.3	1.54	n/a	0.0100	n/a	
S09R000174		Total organic carbon	ug/g	95.6	<20.0	<366	332	n/a	n/a	99.0	366	n/a	U
S09R000174		Total inorganic carbon	ug/g	96.5	<7.00	1.58E+03	1.40E+03	1.49E+03	12.1	105	128	n/a	
S09R000187	A	Uranium-233	ug/g	n/a	<1.00E-04	0.273	0.252	0.263	8.13	n/a	0.188	n/a	J
S09R000187	A	Uranium-234	ug/g	n/a	<5.00E-05	0.472	0.470	0.471	0.500	n/a	0.0938	n/a	J
S09R000187	A	Uranium-235	ug/g	119	<1.10E-04	56.7	58.5	57.6	3.17	-5.07E+03	0.206	n/a	
S09R000187	A	Uranium-236	ug/g	n/a	<4.00E-05	0.839	0.720	0.779	15.3	n/a	0.0751	n/a	
S09R000187	A	Neptunium-237	ug/g	110	<5.30E-04	<0.995	<0.929	n/a	n/a	125	0.995	n/a	U
S09R000187	A	Uranium-238	ug/g	114	<5.50E-03	8.56E+03	8.95E+03	8.76E+03	4.47	-5.23E+03	10.3	n/a	
S09R000180	F	Silver	ug/g	92.6	<124	<124	<132	n/a	n/a	83.1	124	n/a	U
S09R000180	F	Aluminum	ug/g	98.8	<744	1.56E+04	1.57E+04	1.57E+04	0.444	94.1	744	n/a	e
S09R000180	F	Arsenic	ug/g	95.7	<1.24E+03	<1.24E+03	<1.32E+03	n/a	n/a	92.4	1.24E+03	n/a	U
S09R000180	F	Boron	ug/g	96.7	<744	<744	<789	n/a	n/a	90.3	744	n/a	U
S09R000180	F	Barium	ug/g	97.0	<74.4	97.9	112	105	13.5	92.7	74.4	n/a	J
S09R000180	F	Beryllium	ug/g	93.9	<24.8	<24.8	<26.3	n/a	n/a	89.1	24.8	n/a	U
S09R000180	F	Bismuth	ug/g	96.2	<2.48E+03	<2.48E+03	<2.63E+03	n/a	n/a	94.1	2.48E+03	n/a	U
S09R000180	F	Calcium	ug/g	92.8	<1.24E+03	<1.24E+03	1.53E+03	1.38E+03	20.6	91.4	1.24E+03	n/a	U
S09R000180	F	Cadmium	ug/g	94.8	<124	<124	<132	n/a	n/a	90.4	124	n/a	U
S09R000180	F	Cerium	ug/g	101	<744	<744	<789	n/a	n/a	92.4	744	n/a	U
S09R000180	F	Cobalt	ug/g	94.5	<248	<248	406	327	48.3	90.3	248	n/a	U
S09R000180	F	Chromium	ug/g	95.9	<124	<124	<132	n/a	n/a	91.3	124	n/a	U
S09R000180	F	Copper	ug/g	98.2	128	165	149	157	10.3	94.1	124	n/a	B, J
S09R000180	F	Europium	ug/g	97.3	<124	<124	<132	n/a	n/a	n/a	124	n/a	U
S09R000180	F	Iron	ug/g	95.2	266	5.14E+04	5.39E+04	5.27E+04	4.75	89.6	124	n/a	
S09R000180	F	Lanthanum	ug/g	96.1	<74.4	<74.4	<78.9	n/a	n/a	91.5	74.4	n/a	U
S09R000180	F	Lithium	ug/g	107	<74.4	<74.4	<78.9	n/a	n/a	97.1	74.4	n/a	U
S09R000180	F	Magnesium	ug/g	98.9	<1.24E+03	<1.24E+03	<1.32E+03	n/a	n/a	92.0	1.24E+03	n/a	U

Table A-6. Physicochemical Analysis Results for Caustic Dissolution Residue Sample (4 sheets)

Sample Group: 20090517 Data Summary Report

Sample #	A	Analyte	Unit	Std %-Rec	Blank	Result	Duplicate	Average	RPD	Spk %-Rec	Det Limit	Count Error	Qual Flags
S09R000180	F	Manganese	ug/g	93.5	<74.4	868	929	898	6.83	90.9	74.4	n/a	
S09R000180	F	Molybdenum	ug/g	93.4	<496	<496	<526	n/a	n/a	90.4	496	n/a	U
S09R000180	F	Sodium	ug/g	106	<2.48E+03	1.05E+04	1.13E+04	1.09E+04	8.05	98.2	2.48E+03	n/a	
S09R000180	F	Neodymium	ug/g	101	<248	<248	<263	n/a	n/a	96.2	248	n/a	U
S09R000180	F	Phosphorus	ug/g	96.7	<1.24E+03	3.69E+03	3.69E+03	3.69E+03	0.0200	93.0	1.24E+03	n/a	J
S09R000180	F	Lead	ug/g	96.9	<1.24E+03	2.14E+03	3.07E+03	2.61E+03	35.9	91.2	1.24E+03	n/a	J
S09R000180	F	Sulfur	ug/g	98.8	<2.48E+03	<2.48E+03	<2.63E+03	n/a	n/a	94.1	2.48E+03	n/a	U
S09R000180	F	Antimony	ug/g	93.3	<1.24E+03	<1.24E+03	<1.32E+03	n/a	n/a	91.5	1.24E+03	n/a	U
S09R000180	F	Selenium	ug/g	98.4	<2.48E+03	<2.48E+03	<2.63E+03	n/a	n/a	95.0	2.48E+03	n/a	U
S09R000180	F	Silicon	ug/g	91.4	<744	8.06E+03	8.92E+03	8.49E+03	10.1	88.0	744	n/a	
S09R000180	F	Samarium	ug/g	102	<1.24E+03	<1.24E+03	<1.32E+03	n/a	n/a	91.8	1.24E+03	n/a	U
S09R000180	F	Strontium	ug/g	97.1	<74.4	7.95E+03	8.24E+03	8.10E+03	3.53	92.4	74.4	n/a	
S09R000180	F	Thorium	ug/g	98.0	<1.24E+03	<1.24E+03	<1.32E+03	n/a	n/a	90.1	1.24E+03	n/a	U
S09R000180	F	Titanium	ug/g	94.5	<124	<124	<132	n/a	n/a	90.0	124	n/a	U
S09R000180	F	Thallium	ug/g	97.8	<2.48E+03	<2.48E+03	<2.63E+03	n/a	n/a	90.8	2.48E+03	n/a	U
S09R000180	F	Uranium	ug/g	100	<2.48E+03	7.96E+03	8.02E+03	7.99E+03	0.760	90.9	2.48E+03	n/a	J
S09R000180	F	Vanadium	ug/g	96.3	<124	<124	<132	n/a	n/a	91.7	124	n/a	U
S09R000180	F	Yttrium	ug/g	98.4	<49.6	<49.6	<52.6	n/a	n/a	n/a	49.6	n/a	U
S09R000180	F	Zinc	ug/g	95.4	<124	154	209	182	29.8	91.2	124	n/a	J
S09R000180	F	Zirconium	ug/g	93.6	<124	132	<132	n/a	n/a	87.2	124	n/a	J
S09R000184	W	Chloride	ug/g	99.1	<3.10E-03	6.90	7.96	7.43	14.3	101	1.74	n/a	J
S09R000184	W	Nitrite	ug/g	96.6	0.0580	97.9	100	99.0	2.13	96.9	22.5	n/a	J
S09R000184	W	Bromide	ug/g	98.7	<0.0237	<13.3	<13.3	n/a	n/a	98.5	13.3	n/a	U
S09R000184	W	Nitrate	ug/g	99.3	0.0222	328	362	345	9.95	99.6	9.11	n/a	
S09R000184	W	Phosphate	ug/g	101	<0.0381	3.54E+03	3.69E+03	3.62E+03	4.19	103	21.4	n/a	
S09R000184	W	Sulfate	ug/g	101	<0.0219	19.0	23.0	21.0	19.1	102	12.3	n/a	J
S09R000184	W	Oxalate	ug/g	99.2	<0.105	<59.0	<58.8	n/a	n/a	99.7	59.0	n/a	U
S09R000184	W	Fluoride	ug/g	98.0	<1.61E-03	20.4	21.2	20.8	3.60	104	0.302	n/a	
S09R000184	W	Glycolate	ug/g	101	<9.37E-03	<1.76	<1.75	n/a	n/a	106	1.76	n/a	U

Table A-6. Physicochemical Analysis Results for Caustic Dissolution Residue Sample (4 sheets)

Sample Group: 20090517

Data Summary Report

Sample #	A	Analyte	Unit	Std %-Rec	Blank	Result	Duplicate	Average	RPD	Spk %-Rec	Det Limit	Count Error	Qual Flags
S09R000184	W	Acetate	ug/g	104	0.0597	20.2	21.1	20.7	4.49	109	1.13	n/a	B, J
S09R000184	W	Formate	ug/g	103	0.0590	18.7	21.1	19.9	12.1	107	0.875	n/a	B, J
S09R000657	Z	Silver	ug/g	97.5	<52.2	<52.2	<50.1	n/a	n/a	98.8	52.2	n/a	U
S09R000657	Z	Aluminum	ug/g	103	<313	2.60E+04	2.38E+04	2.49E+04	8.74	109	313	n/a	
S09R000657	Z	Arsenic	ug/g	102	<522	<522	<501	n/a	n/a	104	522	n/a	U
S09R000657	Z	Boron	ug/g	100	<313	<313	<300	n/a	n/a	101	313	n/a	U
S09R000657	Z	Barium	ug/g	103	<31.3	159	154	156	3.41	101	31.3	n/a	J
S09R000657	Z	Beryllium	ug/g	98.9	<10.4	<10.4	<10.0	n/a	n/a	95.0	10.4	n/a	U
S09R000657	Z	Bismuth	ug/g	103	<1.04E+03	1.59E+03	1.37E+03	1.48E+03	14.4	108	1.04E+03	n/a	J
S09R000657	Z	Calcium	ug/g	104	<522	3.42E+03	3.61E+03	3.51E+03	5.45	106	522	n/a	J
S09R000657	Z	Cadmium	ug/g	101	<52.2	<52.2	<50.1	n/a	n/a	101	52.2	n/a	U
S09R000657	Z	Cerium	ug/g	99.3	<313	<313	<300	n/a	n/a	99.1	313	n/a	U
S09R000657	Z	Cobalt	ug/g	99.8	<104	164	114	139	35.9	103	104	n/a	J
S09R000657	Z	Chromium	ug/g	101	<52.2	<52.2	63.5	57.8	19.7	103	52.2	n/a	U
S09R000657	Z	Copper	ug/g	99.0	<52.2	125	274	199	74.8	104	52.2	n/a	J
S09R000657	Z	Europium	ug/g	96.3	<52.2	<52.2	<50.1	n/a	n/a	100	52.2	n/a	U
S09R000657	Z	Iron	ug/g	100	<52.2	9.76E+04	9.83E+04	9.79E+04	0.725	96.3	52.2	n/a	
S09R000657	Z	Lanthanum	ug/g	99.5	<31.3	<31.3	<30.0	n/a	n/a	97.0	31.3	n/a	U
S09R000657	Z	Lithium	ug/g	108	<31.3	<31.3	<30.0	n/a	n/a	105	31.3	n/a	U
S09R000657	Z	Magnesium	ug/g	100	<522	770	777	774	0.911	98.1	522	n/a	J
S09R000657	Z	Manganese	ug/g	99.9	<31.3	1.48E+03	1.45E+03	1.46E+03	2.06	103	31.3	n/a	
S09R000657	Z	Molybdenum	ug/g	102	<209	<209	<200	n/a	n/a	105	209	n/a	U
S09R000657	Z	Sodium	ug/g	110	2.02E+03	1.49E+04	1.39E+04	1.44E+04	6.79	109	1.04E+03	n/a	B
S09R000657	Z	Neodymium	ug/g	98.1	<104	<104	<100	n/a	n/a	98.4	104	n/a	U
S09R000657	Z	Nickel	ug/g	98.6	<209	4.61E+04	4.77E+04	4.69E+04	3.29	101	209	n/a	
S09R000657	Z	Phosphorus	ug/g	101	<522	1.36E+03	1.18E+03	1.27E+03	13.5	102	522	n/a	J
S09R000657	Z	Lead	ug/g	103	<522	4.46E+03	4.68E+03	4.57E+03	4.93	106	522	n/a	J
S09R000657	Z	Sulfur	ug/g	101	<1.04E+03	<1.04E+03	<1.00E+03	n/a	n/a	102	1.04E+03	n/a	U
S09R000657	Z	Antimony	ug/g	98.3	<522	<522	<501	n/a	n/a	103	522	n/a	U

Table A-6. Physicochemical Analysis Results for Caustic Dissolution Residue Sample (4 sheets)

Sample Group: 20090517

Data Summary Report

Sample #	A	Analyte	Unit	Std %-Rec	Blank	Result	Duplicate	Average	RPD	Spk %-Rec	Det Limit	Count Error	Qual Flags
S09R000657	Z	Selenium	ug/g	101	<1.04E+03	<1.04E+03	<1.00E+03	n/a	n/a	103	1.04E+03	n/a	U
S09R000657	Z	Silicon	ug/g	92.0	<313	1.36E+04	1.16E+04	1.26E+04	15.7	92.3	313	n/a	
S09R000657	Z	Samarium	ug/g	100	<522	<522	<501	n/a	n/a	95.7	522	n/a	U
S09R000657	Z	Strontium	ug/g	101	<31.3	1.04E+04	1.13E+04	1.08E+04	8.20	98.8	31.3	n/a	
S09R000657	Z	Thorium	ug/g	102	<522	686	<501	n/a	n/a	110	522	n/a	J
S09R000657	Z	Titanium	ug/g	103	<52.2	481	361	421	28.4	102	52.2	n/a	J
S09R000657	Z	Thallium	ug/g	101	<1.04E+03	<1.04E+03	<1.00E+03	n/a	n/a	100	1.04E+03	n/a	U
S09R000657	Z	Uranium	ug/g	97.4	<1.04E+03	9.67E+03	1.06E+04	1.01E+04	9.08	96.9	1.04E+03	n/a	J
S09R000657	Z	Vanadium	ug/g	101	<52.2	53.4	<50.1	n/a	n/a	101	52.2	n/a	J
S09R000657	Z	Yttrium	ug/g	97.8	<20.9	<20.9	<20.0	n/a	n/a	105	20.9	n/a	U
S09R000657	Z	Zinc	ug/g	98.2	<52.2	360	365	362	1.47	101	52.2	n/a	J

Table A-7. Radiological Analysis Results for Caustic Dissolution Residue Sample (4 sheets)

Sample Group: 20090517

Data Summary Report

	A	Analyte	Unit	Std %-Rec	Blank	Result	Duplicate	Average	RPD	Spk %-Rec	Det Limit	Count Error	Qual Flags
S09R000174		Iodine-129	uCi/g	111	<8.00E-06	<6.55E-06	<7.38E-06	n/a	n/a	n/a	6.55E-06	n/a	U
S09R000174		Iodine-C	%	n/a	80.1	80.4	78.4	n/a	n/a	n/a	n/a	n/a	
S09R000187	A	Technetium-99	uCi/g	104	<4.90E-03	2.87E-03	2.33E-03	2.60E-03	20.6	n/a	5.03E-04	24.9	
S09R000187	A	Technetium-99T	%	n/a	56.8	58.3	58.3	n/a	n/a	n/a	n/a	n/a	
S09R000180	F	Curium-243/244	uCi/g	n/a	<5.62E-03	<9.59E-03	<1.06E-02	n/a	n/a	n/a	9.59E-03	n/a	U
S09R000180	F	Americium-241	uCi/g	102	<0.0141	0.181	0.191	0.186	5.31	n/a	0.0240	3.07	
S09R000180	F	Americium-243T	%	n/a	95.414	93.847	89.860	n/a	n/a	n/a	n/a	n/a	
S09R000180	F	Curium-242	uCi/g	n/a	<5.62E-03	<9.59E-03	<1.06E-02	n/a	n/a	n/a	9.59E-03	n/a	U
S09R000180	F	Beryllium-7	uCi/g	n/a	<1.04	<6.29	<6.42	n/a	n/a	n/a	6.29	n/a	U
S09R000180	F	Sodium-22	uCi/g	n/a	<0.160	<0.853	<0.872	n/a	n/a	n/a	0.853	n/a	U
S09R000180	F	Sodium-24	uCi/g	n/a	<0.148	<0.786	<0.802	n/a	n/a	n/a	0.786	n/a	U
S09R000180	F	Aluminum-28	uCi/g	n/a	<5.35	<10.3	<10.0	n/a	n/a	n/a	10.3	n/a	U
S09R000180	F	Chlorine-38	uCi/g	n/a	<0.980	<3.62	<3.74	n/a	n/a	n/a	3.62	n/a	U
S09R000180	F	Potassium-40	uCi/g	n/a	<3.65	<7.02	<7.06	n/a	n/a	n/a	7.02	n/a	U
S09R000180	F	Argon-41	uCi/g	n/a	<0.230	<1.29	<1.33	n/a	n/a	n/a	1.29	n/a	U
S09R000180	F	Scandium-46	uCi/g	n/a	<0.193	<0.962	<0.988	n/a	n/a	n/a	0.962	n/a	U
S09R000180	F	Chromium-51	uCi/g	n/a	<0.968	<4.96	<5.10	n/a	n/a	n/a	4.96	n/a	U
S09R000180	F	Manganese-54	uCi/g	n/a	<0.139	<0.938	<0.956	n/a	n/a	n/a	0.938	n/a	U
S09R000180	F	Cobalt-56	uCi/g	n/a	<0.145	<0.925	<0.949	n/a	n/a	n/a	0.925	n/a	U
S09R000180	F	Manganese-56	uCi/g	n/a	<0.200	<1.29	<1.32	n/a	n/a	n/a	1.29	n/a	U
S09R000180	F	Cobalt-57	uCi/g	n/a	<0.0647	<0.417	<0.430	n/a	n/a	n/a	0.417	n/a	U
S09R000180	F	Cobalt-58	uCi/g	n/a	<0.140	<0.919	<0.943	n/a	n/a	n/a	0.919	n/a	U
S09R000180	F	Iron-59	uCi/g	n/a	<0.302	<1.71	<1.78	n/a	n/a	n/a	1.71	n/a	U
S09R000180	F	Cobalt-60	uCi/g	102	<0.153	<0.786	<0.808	n/a	n/a	n/a	0.786	n/a	U
S09R000180	F	Copper-64	uCi/g	n/a	<31.6	<175	<180	n/a	n/a	n/a	175	n/a	U
S09R000180	F	Zinc-65	uCi/g	n/a	<0.368	<1.89	<1.96	n/a	n/a	n/a	1.89	n/a	U
S09R000180	F	Copper-66	uCi/g	n/a	<31.3	<204	<207	n/a	n/a	n/a	204	n/a	U
S09R000180	F	Selenium-75	uCi/g	n/a	<0.146	<0.744	<0.763	n/a	n/a	n/a	0.744	n/a	U

Table A-7. Radiological Analysis Results for Caustic Dissolution Residue Sample (4 sheets)

Sample Group: 20090517

Data Summary Report

	A	Analyte	Unit	Std %-Rec	Blank	Result	Duplicate	Average	RPD	Spk %-Rec	Det Limit	Count Error	Qual Flags
S09R000180	F	Selenium-79	uCi/g	n/a	<14.8	<105	<108	n/a	n/a	n/a	105	n/a	U
S09R000180	F	Krypton-85	uCi/g	n/a	<38.0	<171	<174	n/a	n/a	n/a	171	n/a	U
S09R000180	F	Strontium-85	uCi/g	n/a	<0.165	<0.738	<0.757	n/a	n/a	n/a	0.738	n/a	U
S09R000180	F	Yttrium-88	uCi/g	n/a	<0.128	<0.189	<0.187	n/a	n/a	n/a	0.189	n/a	U
S09R000180	F	Yttrium-91	uCi/g	n/a	<59.0	<300	<312	n/a	n/a	n/a	300	n/a	U
S09R000180	F	Niobium-94	uCi/g	n/a	<0.138	<0.938	<0.956	n/a	n/a	n/a	0.938	n/a	U
S09R000180	F	Zirconium/Niobium-95	uCi/g	n/a	<0.293	<1.81	<1.86	n/a	n/a	n/a	1.81	n/a	U
S09R000180	F	Ruthenium-103	uCi/g	n/a	<0.131	<0.756	<0.776	n/a	n/a	n/a	0.756	n/a	U
S09R000180	F	Ruthenium/Rhodium-106	uCi/g	n/a	<2.38	<16.0	<16.4	n/a	n/a	n/a	16.0	n/a	U
S09R000180	F	Silver-108	uCi/g	n/a	<0.151	<0.980	<1.01	n/a	n/a	n/a	0.980	n/a	U
S09R000180	F	Cadmium-109	uCi/g	n/a	<1.46	<10.6	<10.9	n/a	n/a	n/a	10.6	n/a	U
S09R000180	F	Silver-110	uCi/g	n/a	<0.174	<0.932	<0.956	n/a	n/a	n/a	0.932	n/a	U
S09R000180	F	Tin-113	uCi/g	n/a	<0.158	<0.853	<0.866	n/a	n/a	n/a	0.853	n/a	U
S09R000180	F	Tellurium-123	uCi/g	n/a	<0.0804	<0.445	<0.455	n/a	n/a	n/a	0.445	n/a	U
S09R000180	F	Antimony-124	uCi/g	n/a	<0.128	<0.804	<0.828	n/a	n/a	n/a	0.804	n/a	U
S09R000180	F	Antimony-125	uCi/g	n/a	<0.383	<2.09	<2.14	n/a	n/a	n/a	2.09	n/a	U
S09R000180	F	Tellurium-125	uCi/g	n/a	<0.132	<1.56	<1.60	n/a	n/a	n/a	1.56	n/a	U
S09R000180	F	Antimony-126	uCi/g	n/a	<0.129	<0.853	<0.866	n/a	n/a	n/a	0.853	n/a	U
S09R000180	F	Tin-126	uCi/g	n/a	<0.119	<0.859	<0.885	n/a	n/a	n/a	0.859	n/a	U
S09R000180	F	Iodine-129	uCi/g	n/a	<0.829	<10.0	<10.3	n/a	n/a	n/a	10.0	n/a	U
S09R000180	F	Iodine-131	uCi/g	n/a	<0.117	<0.647	<0.667	n/a	n/a	n/a	0.647	n/a	U
S09R000180	F	Xenon-131	uCi/g	n/a	<3.51	<19.1	<19.6	n/a	n/a	n/a	19.1	n/a	U
S09R000180	F	Barium-133	uCi/g	n/a	<0.186	<0.865	<0.892	n/a	n/a	n/a	0.865	n/a	U
S09R000180	F	Cesium-134	uCi/g	n/a	<0.159	<0.811	<0.834	n/a	n/a	n/a	0.811	n/a	U
S09R000180	F	Cesium-136	uCi/g	n/a	<0.135	<0.938	<0.949	n/a	n/a	n/a	0.938	n/a	U
S09R000180	F	Cesium-137	uCi/g	106	<0.191	2.56	2.58	2.57	0.703	n/a	0.998	24.63	
S09R000180	F	Cesium-138	uCi/g	n/a	<0.617	<2.92	<2.98	n/a	n/a	n/a	2.92	n/a	U
S09R000180	F	Cerium-139	uCi/g	n/a	<0.0877	<0.467	<0.477	n/a	n/a	n/a	0.467	n/a	U
S09R000180	F	Barium-140	uCi/g	n/a	<0.495	<2.95	<3.05	n/a	n/a	n/a	2.95	n/a	U

Table A-7. Radiological Analysis Results for Caustic Dissolution Residue Sample (4 sheets)

Sample Group: 20090517

Data Summary Report

	A	Analyte	Unit	Std %-Rec	Blank	Result	Duplicate	Average	RPD	Spk %-Rec	Det Limit	Count Error	Qual Flags
S09R000180	F	Lanthanum-140	uCi/g	n/a	<0.158	<0.485	<0.520	n/a	n/a	n/a	0.485	n/a	U
S09R000180	F	Cerium-141	uCi/g	n/a	<0.120	<0.726	<0.738	n/a	n/a	n/a	0.726	n/a	U
S09R000180	F	Cerium-144	uCi/g	n/a	<0.520	<3.19	<3.28	n/a	n/a	n/a	3.19	n/a	U
S09R000180	F	Cerium/Praseodymium-144	uCi/g	n/a	<1.04	<6.41	<6.54	n/a	n/a	n/a	6.41	n/a	U
S09R000180	F	Europium-152	uCi/g	n/a	<0.744	<3.44	<3.41	n/a	n/a	n/a	3.44	n/a	U
S09R000180	F	Europium-154	uCi/g	n/a	<0.463	<2.47	<2.53	n/a	n/a	n/a	2.47	n/a	U
S09R000180	F	Europium-155	uCi/g	n/a	<0.244	<1.69	<1.73	n/a	n/a	n/a	1.69	n/a	U
S09R000180	F	Hafnium-181	uCi/g	n/a	<0.141	<0.817	<0.840	n/a	n/a	n/a	0.817	n/a	U
S09R000180	F	Tantalum-182	uCi/g	n/a	<0.554	<2.76	<2.84	n/a	n/a	n/a	2.76	n/a	U
S09R000180	F	Gold-198	uCi/g	n/a	<0.112	<0.604	<0.618	n/a	n/a	n/a	0.604	n/a	U
S09R000180	F	Mercury-203	uCi/g	n/a	<0.121	<0.590	<0.606	n/a	n/a	n/a	0.590	n/a	U
S09R000180	F	Bismuth-207	uCi/g	n/a	<0.181	<1.22	<1.25	n/a	n/a	n/a	1.22	n/a	U
S09R000180	F	Thallium-208	uCi/g	n/a	<0.164	<0.907	<0.937	n/a	n/a	n/a	0.907	n/a	U
S09R000180	F	Lead-210	uCi/g	n/a	<1.38	<15.0	<15.3	n/a	n/a	n/a	15.0	n/a	U
S09R000180	F	Bismuth-212	uCi/g	n/a	<1.16	<7.56	<7.70	n/a	n/a	n/a	7.56	n/a	U
S09R000180	F	Lead-212	uCi/g	n/a	<0.217	<0.956	<0.982	n/a	n/a	n/a	0.956	n/a	U
S09R000180	F	Bismuth-214	uCi/g	n/a	<0.341	<1.71	<1.78	n/a	n/a	n/a	1.71	n/a	U
S09R000180	F	Lead-214	uCi/g	n/a	<0.304	<1.40	<1.42	n/a	n/a	n/a	1.40	n/a	U
S09R000180	F	Radium-224	uCi/g	n/a	<2.46	<10.9	<11.2	n/a	n/a	n/a	10.9	n/a	U
S09R000180	F	Radium-226	uCi/g	n/a	<2.30	<11.5	<11.8	n/a	n/a	n/a	11.5	n/a	U
S09R000180	F	Actinium-228	uCi/g	n/a	<0.573	<3.37	<3.50	n/a	n/a	n/a	3.37	n/a	U
S09R000180	F	Thorium-228	uCi/g	n/a	<4.16	<31.6	<32.5	n/a	n/a	n/a	31.6	n/a	U
S09R000180	F	Thorium-229	uCi/g	n/a	<0.451	<3.31	<3.40	n/a	n/a	n/a	3.31	n/a	U
S09R000180	F	Uranium-232	uCi/g	n/a	<24.0	<241	<246	n/a	n/a	n/a	241	n/a	U
S09R000180	F	Protactinium-233	uCi/g	n/a	<0.250	<1.25	<1.28	n/a	n/a	n/a	1.25	n/a	U
S09R000180	F	Uranium/Thorium-233	uCi/g	n/a	<82.9	<391	<404	n/a	n/a	n/a	391	n/a	U
S09R000180	F	Protactinium-234	uCi/g	n/a	<22.7	<143	<146	n/a	n/a	n/a	143	n/a	U
S09R000180	F	Thorium-234	uCi/g	n/a	<1.33	<11.7	<12.1	n/a	n/a	n/a	11.7	n/a	U
S09R000180	F	Uranium-235	uCi/g	n/a	<0.141	<0.696	<0.718	n/a	n/a	n/a	0.696	n/a	U

Table A-7. Radiological Analysis Results for Caustic Dissolution Residue Sample (4 sheets)

Sample Group: 20090517

Data Summary Report

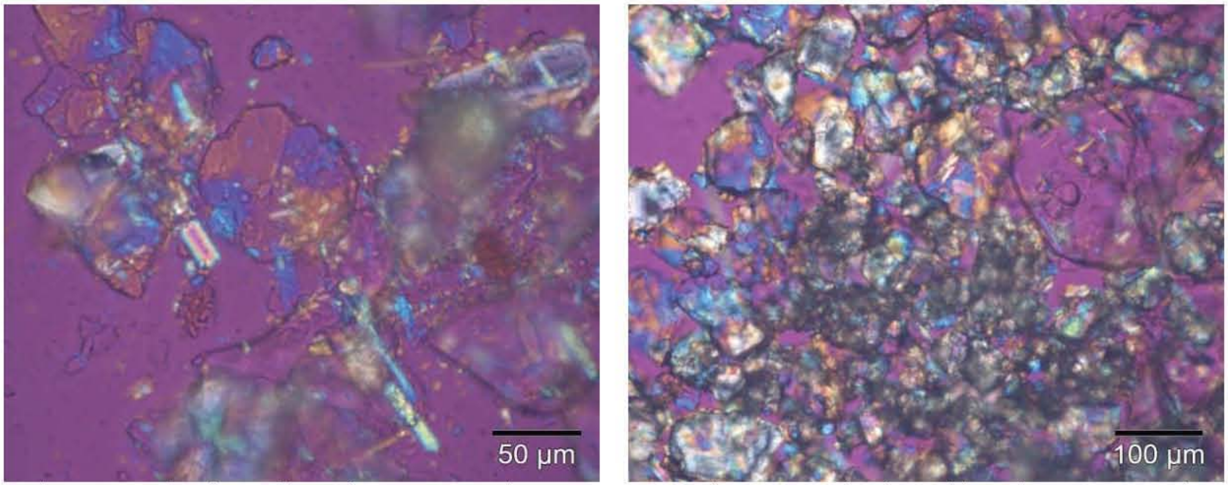
	A	Analyte	Unit	Std %-Rec	Blank	Result	Duplicate	Average	RPD	Spk %-Rec	Det Limit	Count Error	Qual Flags
S09R000180	F	Neptunium-237	uCi/g	n/a	<0.434	<3.20	<3.29	n/a	n/a	n/a	3.20	n/a	U
S09R000180	F	Uranium-237	uCi/g	n/a	<0.201	<1.47	<1.51	n/a	n/a	n/a	1.47	n/a	U
S09R000180	F	Neptunium-238	uCi/g	n/a	<0.505	<3.38	<3.52	n/a	n/a	n/a	3.38	n/a	U
S09R000180	F	Neptunium-239	uCi/g	n/a	<0.235	<1.63	<1.67	n/a	n/a	n/a	1.63	n/a	U
S09R000180	F	Plutonium-239	uCi/g	n/a	<895	<5.67E+03	<5.82E+03	n/a	n/a	n/a	5.67E+03	n/a	U
S09R000180	F	Americium-241	uCi/g	n/a	<0.132	<1.31	<1.35	n/a	n/a	n/a	1.31	n/a	U
S09R000180	F	Americium-243	uCi/g	n/a	<0.129	<0.714	<0.731	n/a	n/a	n/a	0.714	n/a	U
S09R000180	F	Plutonium-239/240	uCi/g	95.8	<0.0129	0.123	0.125	0.124	1.75	n/a	0.0165	3.77	
S09R000180	F	Plutonium-236T	%	n/a	67.1	74.5	74.4	n/a	n/a	n/a	n/a	n/a	
S09R000180	F	Plutonium-238	uCi/g	n/a	<0.0129	<0.0165	<0.0172	n/a	n/a	n/a	0.0165	n/a	U
S09R000180	F	Strontium-89/90	uCi/g	104	<1.37	5.37E+03	5.44E+03	5.41E+03	1.23	n/a	1.35	0.78	
S09R000180	F	Strontium-C	%	n/a	77.5	78.5	78.9	n/a	n/a	n/a	n/a	n/a	

ATTACHMENT B

SOLID PHASE CHARACTERIZATION OF HEEL SAMPLES 7 & 8

ATTACHMENT B – SOLID PHASE CHARACTERIZATION OF HEEL SAMPLES 7 & 8

Figure B-1. Polarized Light Microscopy Images of Heel Sample 7 (left) and Heel Sample 8 (right)



(40x, crossed polars with Red I compensator) (20x, crossed polars with Red I compensator)

Figure B-2. X-ray Diffraction Spectrum of Sample S09R000308

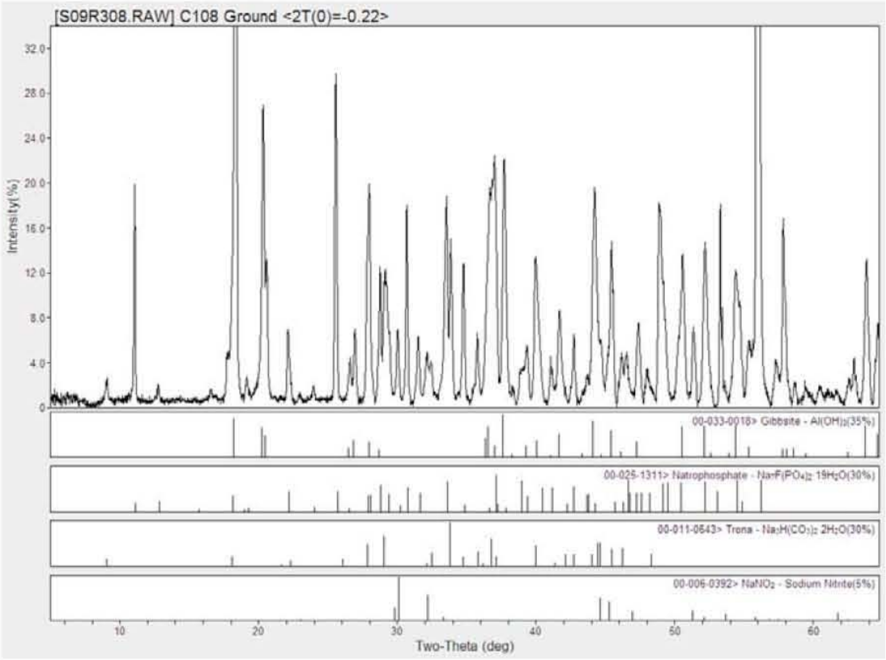


Figure B-3. X-ray Diffraction Spectrum of Sample S09R000309

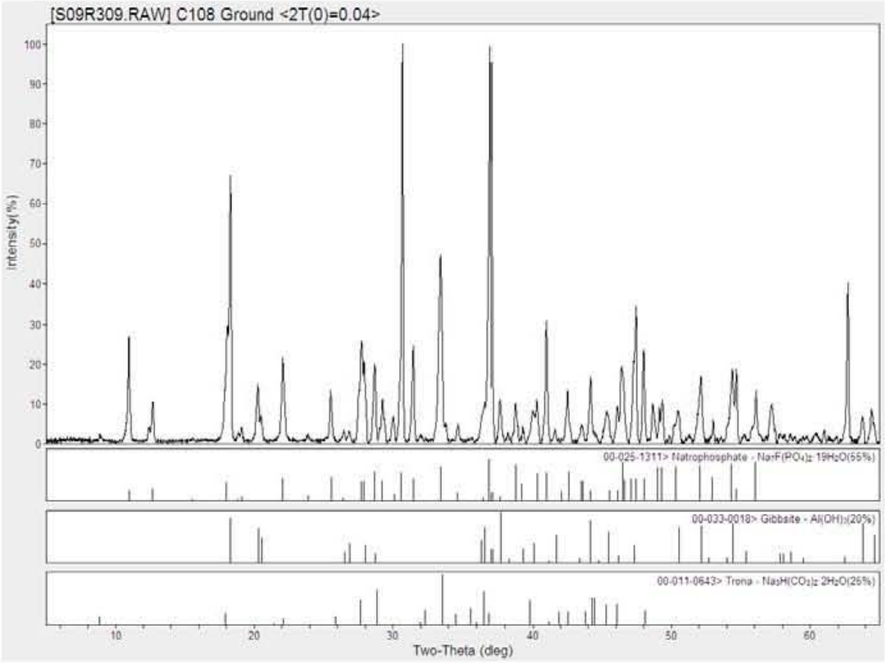
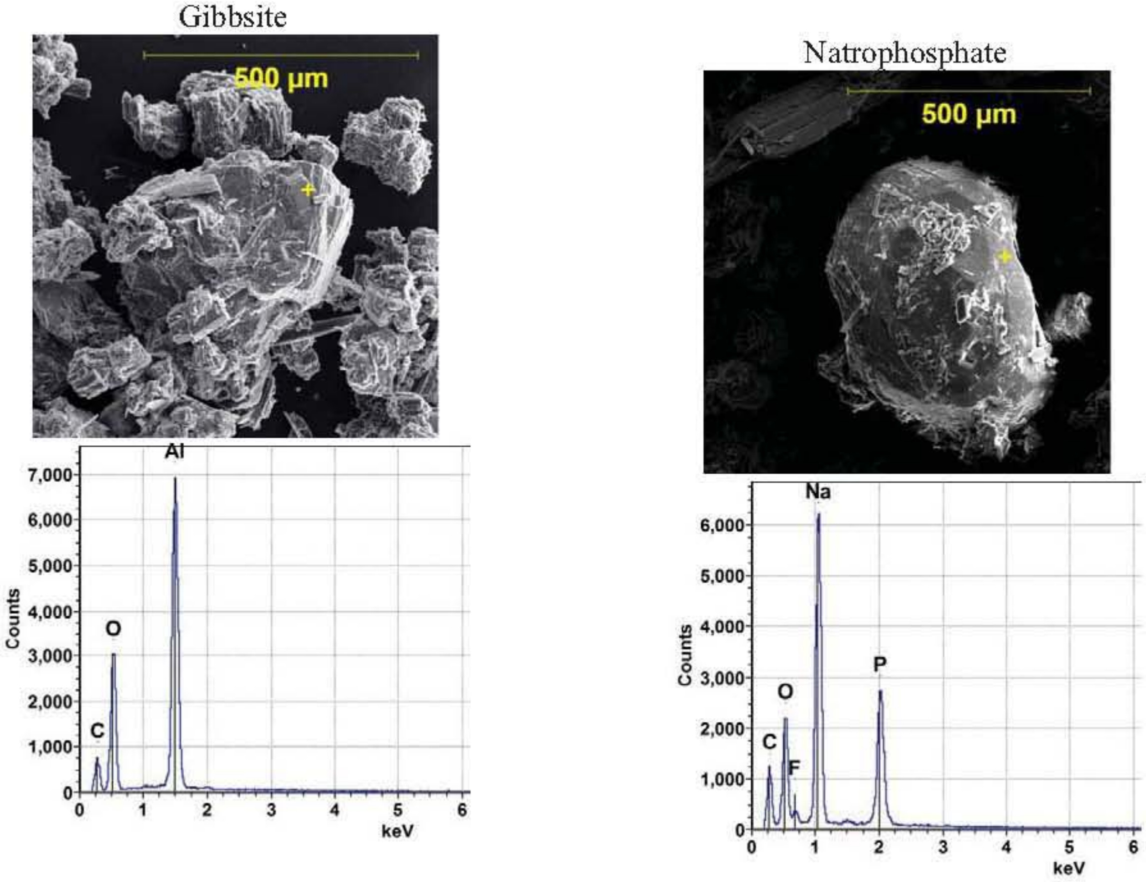
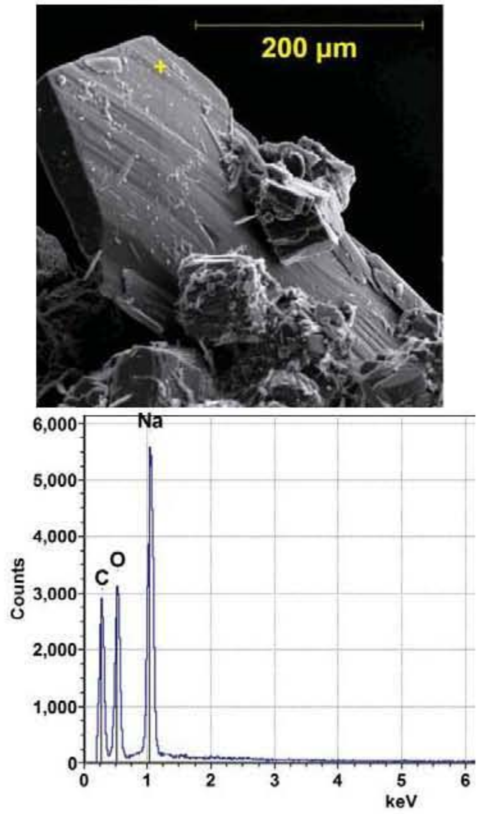


Figure B-4. Backscattered Electron Images and Energy Dispersive Spectrometry Spectra of Sample S09R000308 (Heel Sample 7)

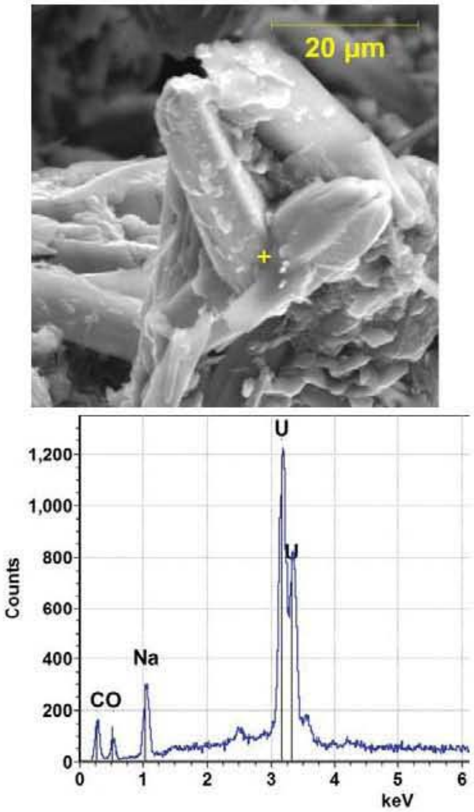

Gibbsite

Natrophosphate

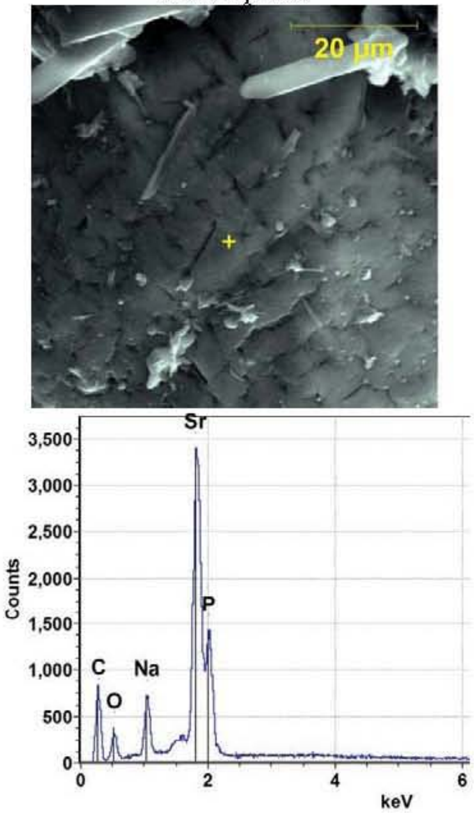
Trona



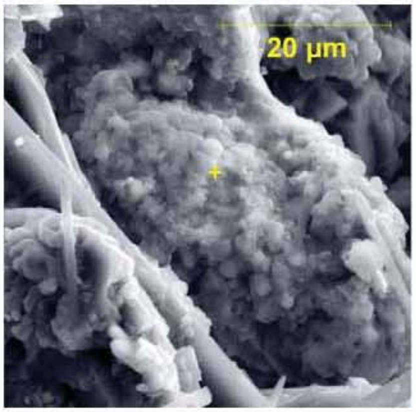
Na-Uranium-rich phase

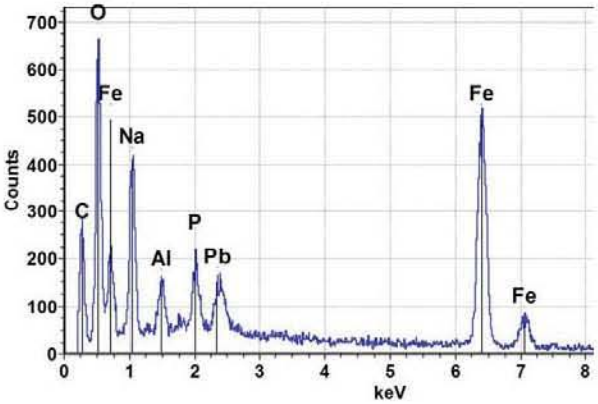


Nastrophite

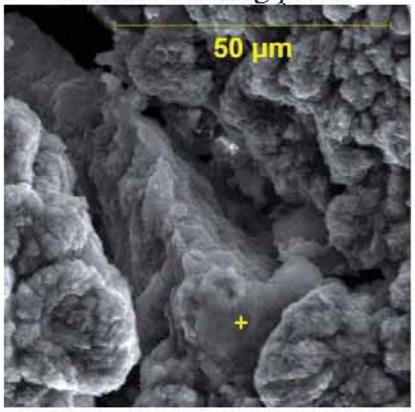


Fe-Pb-bearing phase





Ni-Al-P-bearing phase



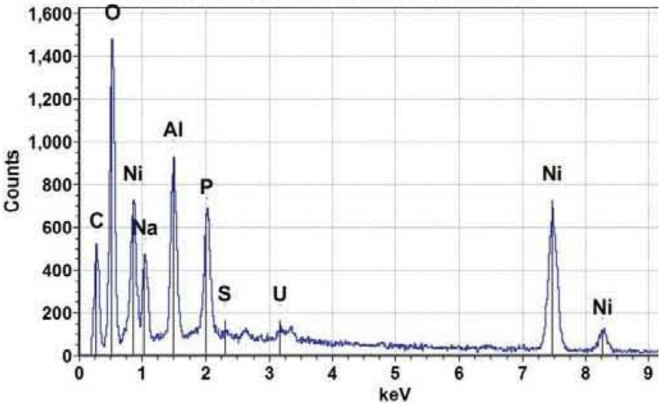
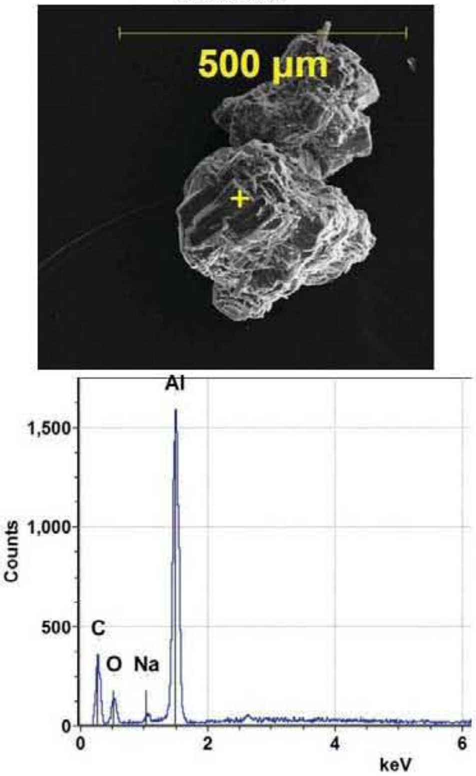
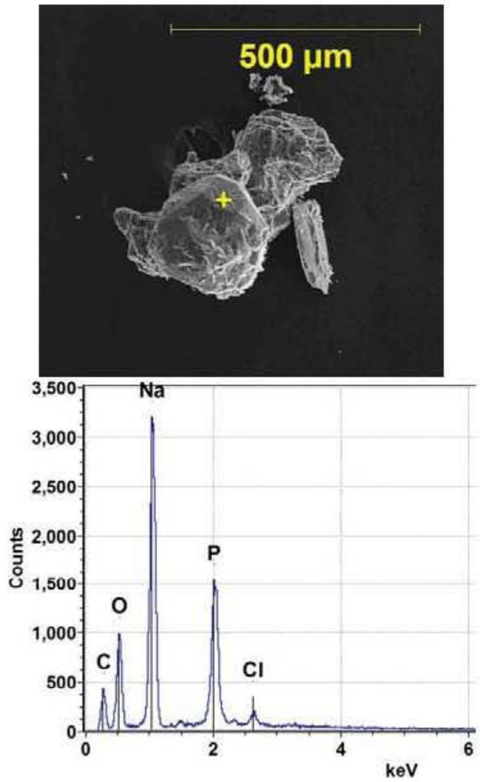


Figure B-5. Backscattered Electron Images and Energy Dispersive Spectrometry Spectra of Sample S09R000309 (Heel Sample 8)

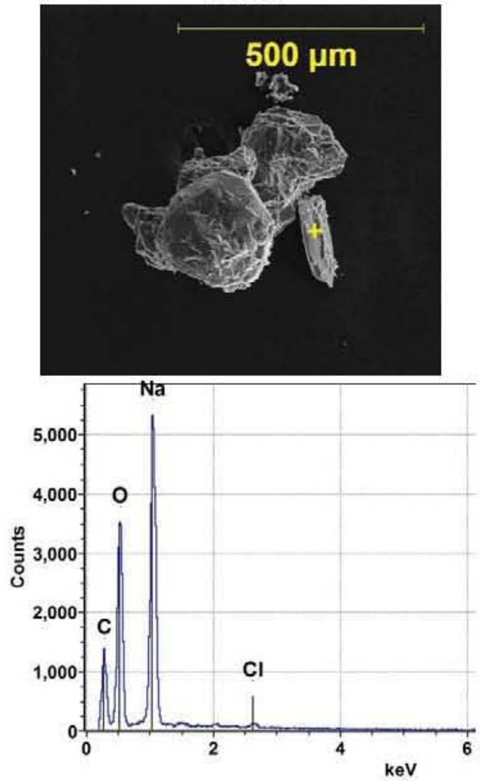
Gibbsite



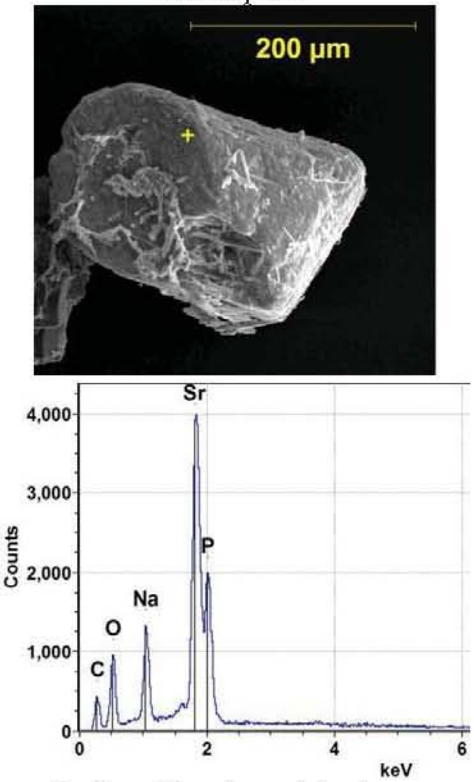
Natrophosphate



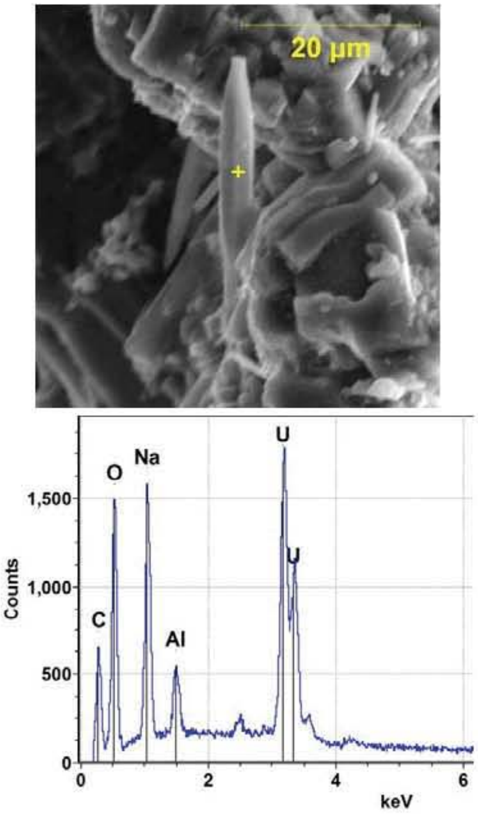
Trona



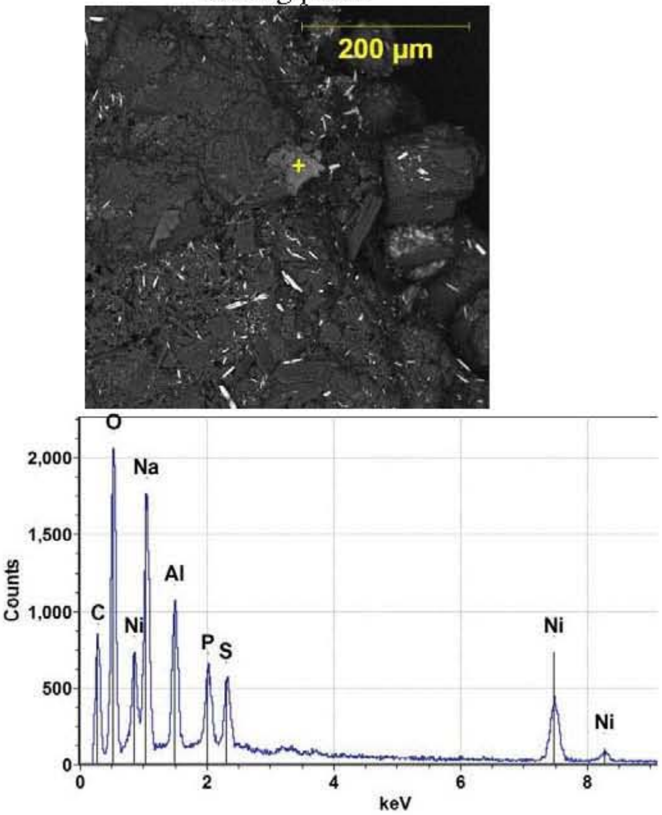
Nastrophite



Sodium-Uranium-rich phase



Nickel-Aluminum-Phosphorus-Sulfur-bearing phase



ATTACHMENT C

PARTICLE SIZE DISTRIBUTION ANALYSIS

ATTACHMENT C – PARTICLE SIZE DISTRIBUTION ANALYSIS

1.0 INTRODUCTION

Measurement of the particle size distribution (PSD) in the heel solids remaining in tank 241-C-108 (C-108) following retrieval operations was specified in a client-approved test plan (WRPS-0900639, "Test Plan for Characterization and Retrieval Testing of Tank 241-C-108 Heel Solids"). The measurement was made in three steps. First, the entire composite sample prepared for the study was separated into >1/4-in. and <1/4-in. solid fractions by wet sieving. A subsample of the <1/4-in. heel solids (sample S09R000151) was then separated into >600-µm and <600-µm solids, again by wet-sieving. The >600-µm solids were recovered, dried, and weighed. The PSD in a subsample of the <600-µm solids (sample S09R000307) was then measured using a light-scattering-based PSD measurement technique. 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 <600-µm C-108 heel solids withdrawn from subsample S09R000307. 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

PSD measurements were performed using the Horiba LA-910 Laser Scattering Particle Size Distribution Analyzer[10] 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

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

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 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-measuring loop during PSD measurements. Key components in the 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 P4 and P5 were used in these measurements. The discharge volume at the P4 setting is ≈9.4 mL/s using Tygon[11] tubing with an internal diameter of 4.8 mm; at the P5 setting the discharge volume is ≈10.6 mL/s.

The occlusion of the Masterflex[12] 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[13] standard PS223.

Measurement Cell. The flow cell is located immediately downstream from the circulation pump. The two optical windows of the cell are Tempax[14] 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 analyzed was ≈210 mL of a dilute slurry of the <600-µm C-108 solids in reagent water. The optimum volumetric concentration of solids in a specimen slurry 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 <600-µm solids was performed using a technique recommended in ISO-13320-1. The PSD subsample (S09R000307) had been allowed to settle, undisturbed, for six days. Prior to the PSD measurements, all the clear supernatant liquid was 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.” The consistency of the subsample of <600-µm C-108 heel 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

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[11] Tygon is a registered trademark of Norton Company, Worcester, Massachusetts.

[12] Masterflex is a registered trademark of the Cole-Parmer Instrument Company, Vernon Hills, Illinois.

[13] Whitehouse Scientific, Waverton, Chester, England.

[14] 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 <600-µm C-108 heel 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 P4 and the stirrer speed was set to S2.
5. An external timer was started and specimen transfer begun. 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. The external timer was stopped and the total load-in time recorded.
8. 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.
9. 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.
10. When Run 3 was complete, the presence of a saved data file was verified.
11. The specimen suspension was drained from the analyzer sample loop.
12. 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	1	2	3
Analyzer settings	—	—	—	—
Pump Speed setting[a]	P4	P5	P5	P5
Stirrer Speed setting[a]	S2	S3	S3	S3
Premeasurement working	—	—	—	—
Work time (seconds)	As needed	0	120	120
Sonication ON[b]	No	No	No	Yes
Premeasurement wait (seconds)	N/A	0	0	10[c]
Measurement	—	—	—	—
Measure cycles (laser/lamp)[d]	N/A	60/30	60/30	60/30
Sonication during measure	N/A	No	No	No
Total time (minutes)[e]	1.2[f]	2.5	5.9	9.5

[a] Speed settings for pump used to circulate specimen suspension through the analyzer flow loop and stirrer used to maintain homogeneity of the specimen suspension in the analyzer sample tank.

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

[c] Estimated time required to exchange the volume of suspension in the loop between the sample tank and the flow cell.

[d] A composite measurement combining 60 readouts of detectors while illuminating specimen with a He-Ne laser and 30 readouts while illuminating the sample with a blue-filtered lamp. Total measurement time was ≈75 sec.

[e] A default period of 3 sec is included for the initiation of Run 1. A period of 10 sec is included to allow for changing analyzer settings and initiating Runs 2 and 3.

[f] The average time required for the specimen load-in for the triplicate measurements was 1 min 13 sec.

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 for the load-in was recorded for each specimen as the period of time beginning with the first introduction of specimen solids to the sample tank and ending with initiation of PSD Run 1. The load-in times for each specimen were:

Primary specimen	1 minute : 11 seconds
Duplicate specimen	1 minute : 44 seconds
Triplicate specimen	44 seconds

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 = Refractive index (***p***articles) / Refractive index (liquid ***m***edium)

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

$$N = n - ki,$$

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

$$RRI = (n_p/n_m) - k_p i.$$

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 $\lambda = 589.3$ nm.

Solid phase characterization of the C-108 heel solids composite prepared for this study indicated that natrophosphate [$Na_7F(PO_4)_3 \cdot 19H_2O$] and gibbsite [$Al(OH)_3$] were the two major solid phases with smaller amounts of trona [$Na_3H(CO_3)_2 \cdot 2H_2O$] and lepidocrocite (FeOOH) probably present. The estimated proportions (by weight) of these four phases in the composite was

119 parts natrophosphate : 63 parts gibbsite : 11 parts trona : 1 part lepidocrocite.

By the conclusion of the initial <1/4-in. sieve separation, most of the trona and some of the natrophosphate had dissolved leaving an estimated solids composition of

110 parts natrophosphate : 64 parts gibbsite : 3 parts trona : 1 part lepidocrocite.

The estimated, volume-weighted, average real refractive index for this mixture is $n_p = 1.49$.

If all the trona and natrophosphate dissolved in the PSD sample, the estimated volume-weighted average of the real refractive index for the resulting mixture of gibbsite and lepidocrocite would rise to $n_p = 1.58$. An intermediate value of the real refractive index, $n_p = 1.535$, was assigned to the heel solids for the PSD calculations.

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 highly colored materials, particularly those of unknown composition, are generally assigned extinction coefficients ranging from 0.10 to 1.00. The <600-µm C-108 heel solids were (visually) a 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 $k_p = 0.20$ was assigned.

Based on the preceding discussion, the RRI value used in the PSD calculations for the <600-µm C-108 heel solids was

$$RRI = (n_p/n_m) - k_p i = (1.53/1.33) - 0.2i = 1.15 - 0.20i.$$

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

One minor deviation from the project test plan (WRPS-0900639) occurred during the completion of the particle size measurements. In PSD Run 1 for the triplicate sample portion from vial S09R000307, the pump and stirrer speeds were inadvertently set at P6 and S1 instead of the specified settings of P5 and S3. Inspection of the data files indicated that the impact of this error was not significant. However, for consistency, the data from Run 1 on the triplicate sample portion was not included when the average Run 1 values were calculated.

4.0 PARTICLE SIZE DISTRIBUTION RESULTS

The results of the light-scattering-based PSD measurements on the <600-µm C-108 heel solids are presented in this section. As previously noted, these measurements were made on three specimens taken from sample vial S09R000307. The data files for corresponding PSD runs from the primary, duplicate, and triplicate specimen measurements were subsequently combined into ‘average’ data files. The PSD histograms, %-undersize curves, and numerical data derived from each specimen are presented in this attachment. Only the data from the averaged data files are presented in Sections 5.3.3 and 5.3.4 in the main body of 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 <600-µm C-108 heel solids are based on ‘standard-form’ distributions. Standard-form 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.

All the PSD data presented are calculated using a value of 1.15 − 0.20i for the RRI.

4.1 HISTOGRAMS AND PERCENT-UNDERSIZE CURVES

The volume-based histograms and %-undersize curves depicting the results of the PSD measurements on three specimens of the <600-µm C-108 heel solids are presented in Figure C-1.

The volume-based histograms and cumulative curves of Figure C-1 are plotted using a common particle diameter (x-axis) scale, 0.1 µm-1020 µm. No particulates with diameters <0.1 µm were observed in any of the calculated PSDs. To further facilitate comparison of the graphic data, all the volume-based, PSD histogram data are also plotted against a common frequency-% (y-axis).

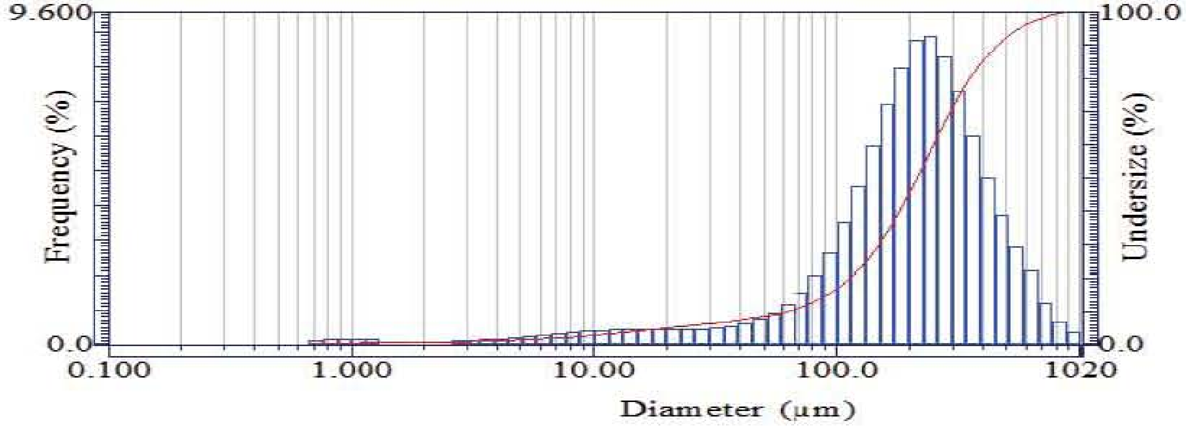
The number-based PSD histograms and %-undersize curves are presented in Figure C-2. The number-based distributions are plotted against the same particle diameter axis used for the volume-based data. Note that no particles with spherical equivalent diameters >7 µm are observed in the number-based distributions. A common frequency-% axis is used for all the number-based data.

The header for each PSD run in Figures C-1 and C-2 displays the stirrer speed setting; the circulation pump speed setting; whether the specimen slurry was sonicated and if so, for how long (in minutes); and, the duration of the run (also in minutes). For Run 1, the duration includes the specimen load-in time.

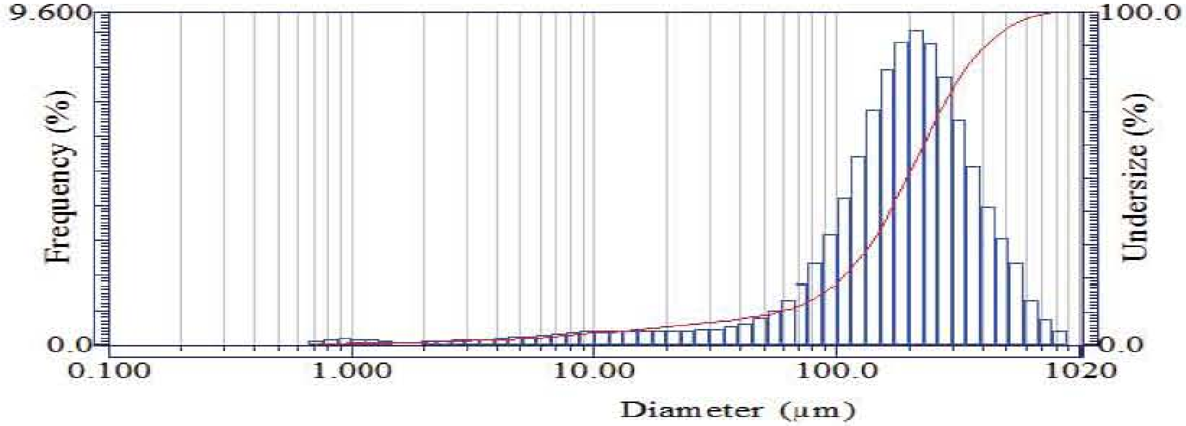
Figure C-1. Volume-Based PSD Histograms and %-Undersize Curves for <600-µm C-108 Heel Solids (3 sheets)

(a) Primary Specimen

Run 1 — Stir Speed: S2-3; Pump Speed: P4-5; Sonication: None; Duration: 2.5 min



Run 2 — Stir Speed: S3; Pump Speed: P5; Sonication: None; Duration: 3.4 min



Run 3 — Stir Speed: S3; Pump Speed: P5; Sonication: 2.0 min; Duration: 3.6 min

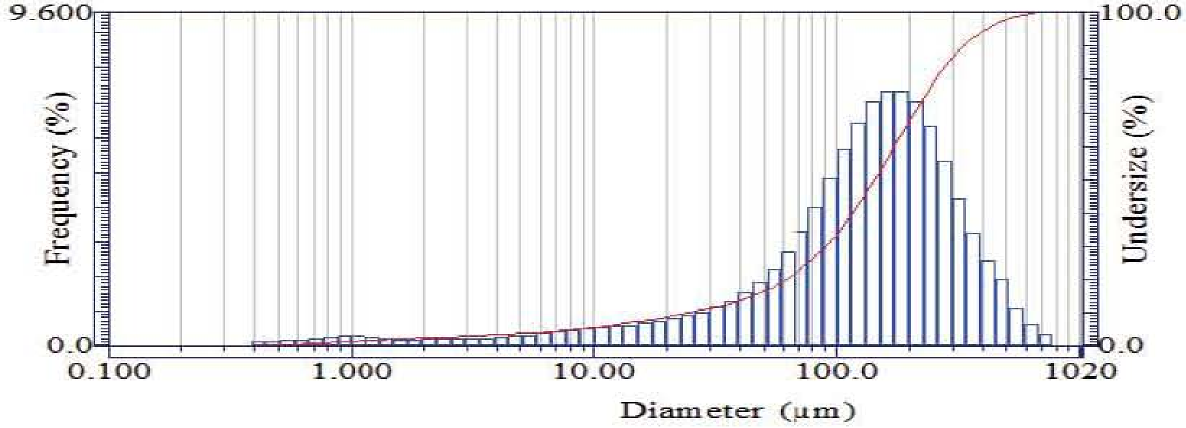
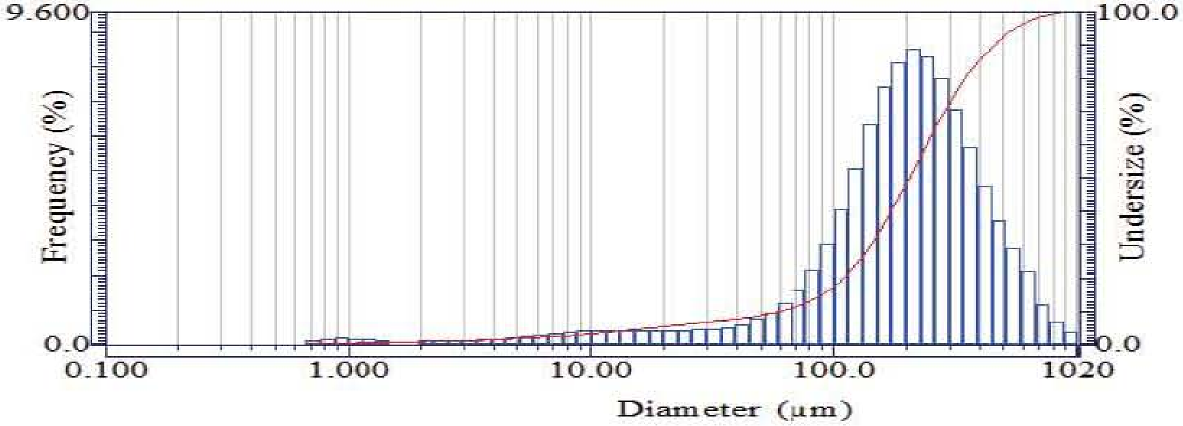


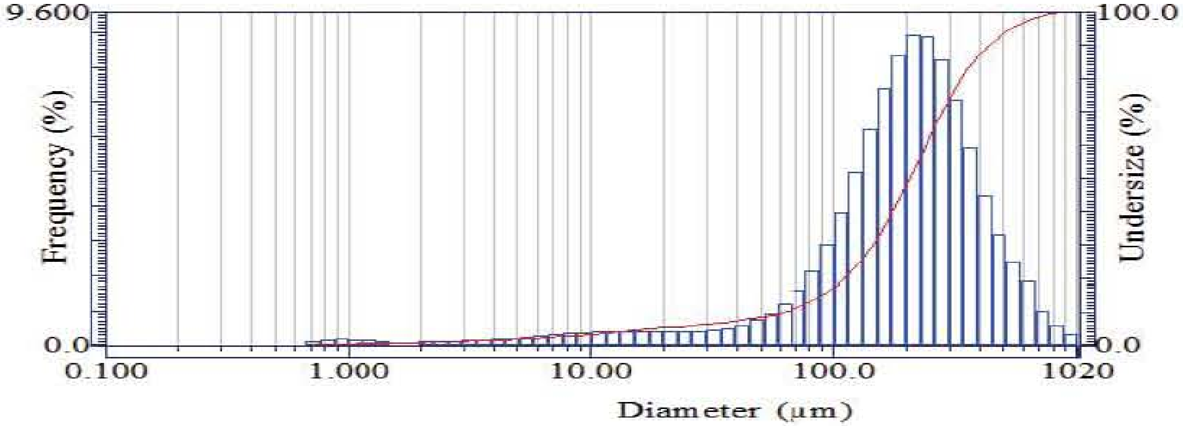
Figure C-1. Volume-Based PSD Histograms and %-Undersize Curves for <600-µm C-108 Heel Solids (3 sheets)

(b) Duplicate Specimen

Run 1 — Stir Speed: S2-3; Pump Speed: P4-5; Sonication: None; Duration: 3.0 min



Run 2 — Stir Speed: S3; Pump Speed: P5; Sonication: None; Duration: 3.4 min



Run 3 — Stir Speed: S3; Pump Speed: P5; Sonication: 2.0 min; Duration: 3.6 min

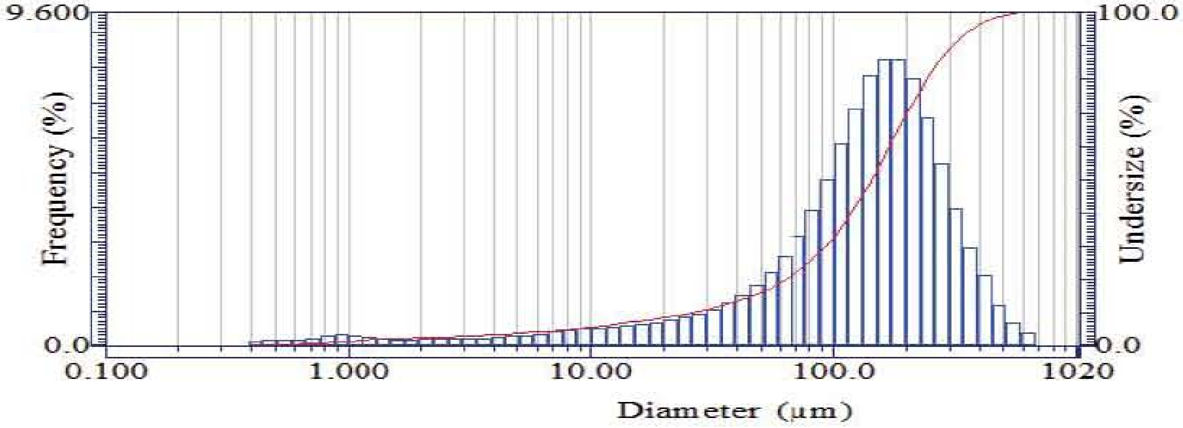
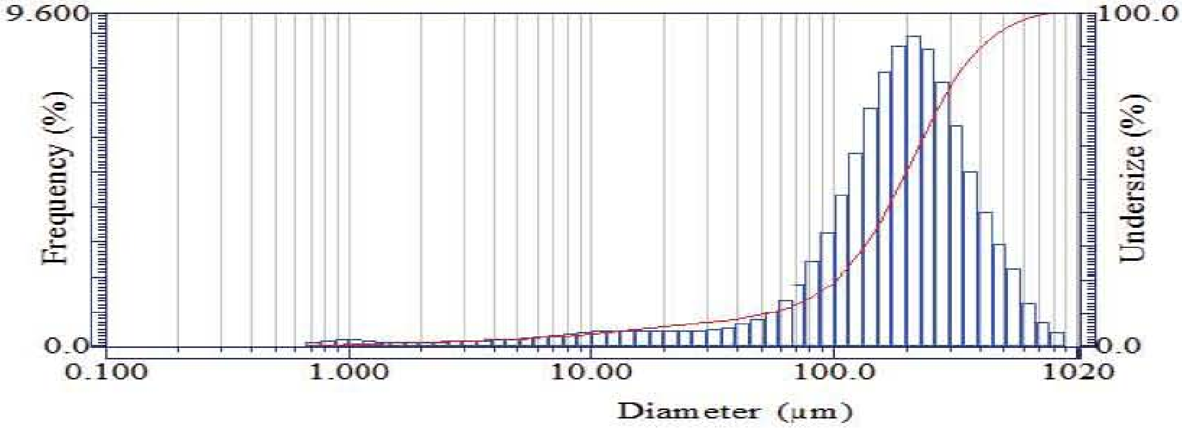


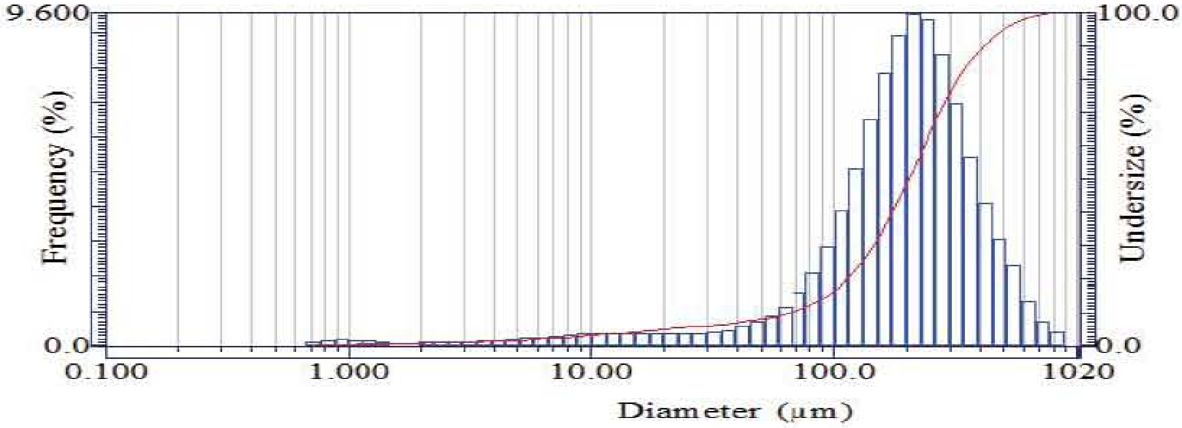
Figure C-1. Volume-Based PSD Histograms and %-Undersize Curves for <600-µm C-108 Heel Solids (3 sheets)

(c) Triplicate Specimen

Run 1 — Stir Speed: S1-2; Pump Speed: P4-6; Sonication: None; Duration: 2.0 min



Run 2 — Stir Speed: S3; Pump Speed: P5; Sonication: None; Duration: 3.4 min



Run 3 — Stir Speed: S3; Pump Speed: P5; Sonication: 2.0 min; Duration: 3.6 min

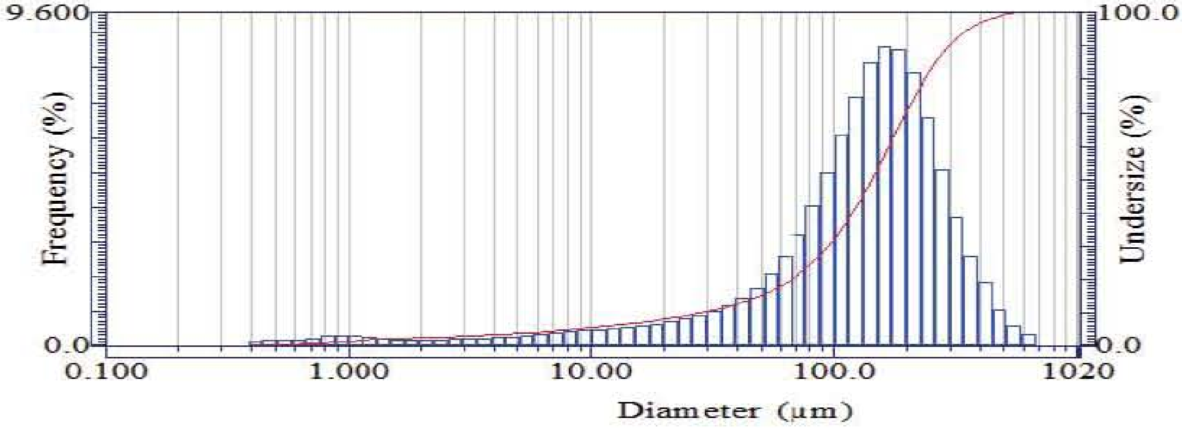
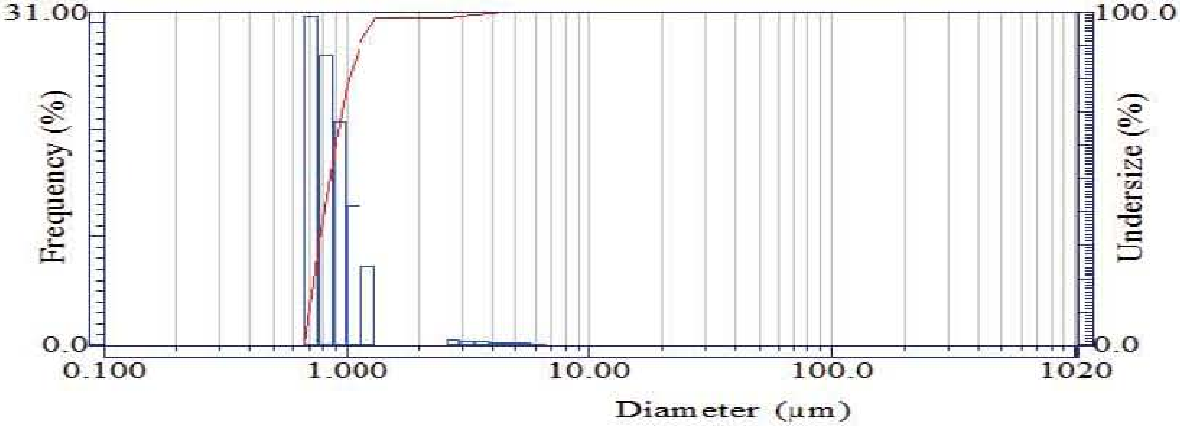


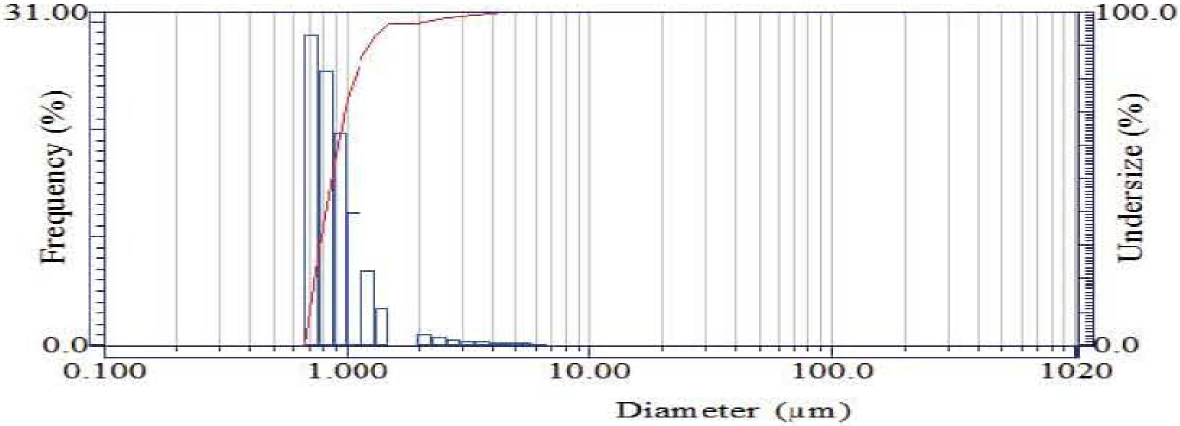
Figure C-2. Number-Based PSD Histograms and %-Undersize Curves for <600-µm C-108 Heel Solids (3 sheets)

(a) Primary specimen

Run 1 — Stir Speed: S2-3; Pump Speed: P4-5; Sonication: None; Duration: 2.5 min



Run 2 — Stir Speed: S3; Pump Speed: P5; Sonication: None; Duration: 3.4 min



Run 3 — Stir Speed: S3; Pump Speed: P5; Sonication: 2.0 min; Duration: 3.6 min

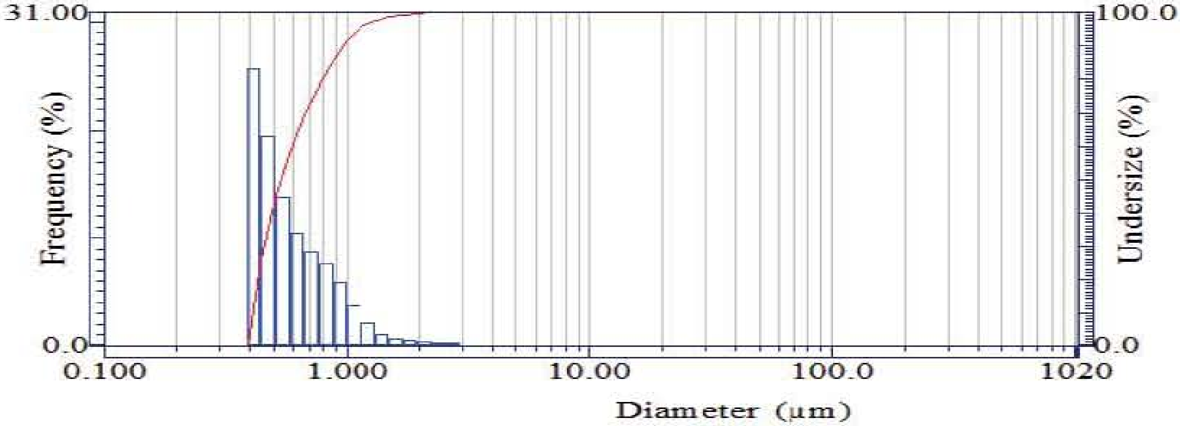
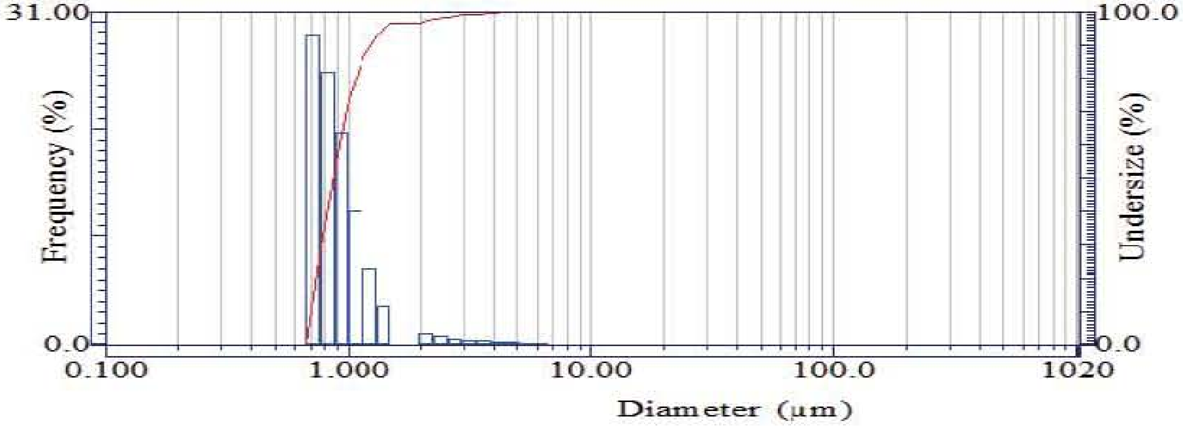


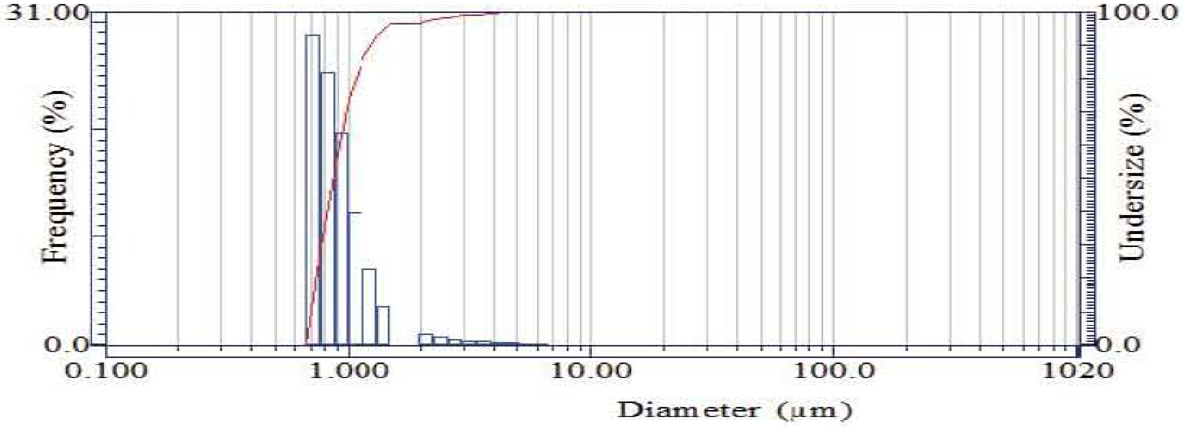
Figure C-2. Number-Based PSD Histograms and %-Undersize Curves for <600-µm C-108 Heel Solids (3 sheets)

(b) Duplicate specimen

Run 1 — Stir Speed: S2-3; Pump Speed: P4-5; Sonication: None; Duration: 3.0 min



Run 2 — Stir Speed: S3; Pump Speed: P5; Sonication: None; Duration: 3.4 min



Run 3 — Stir Speed: S3; Pump Speed: P5; Sonication: 2.0 min; Duration: 3.6 min

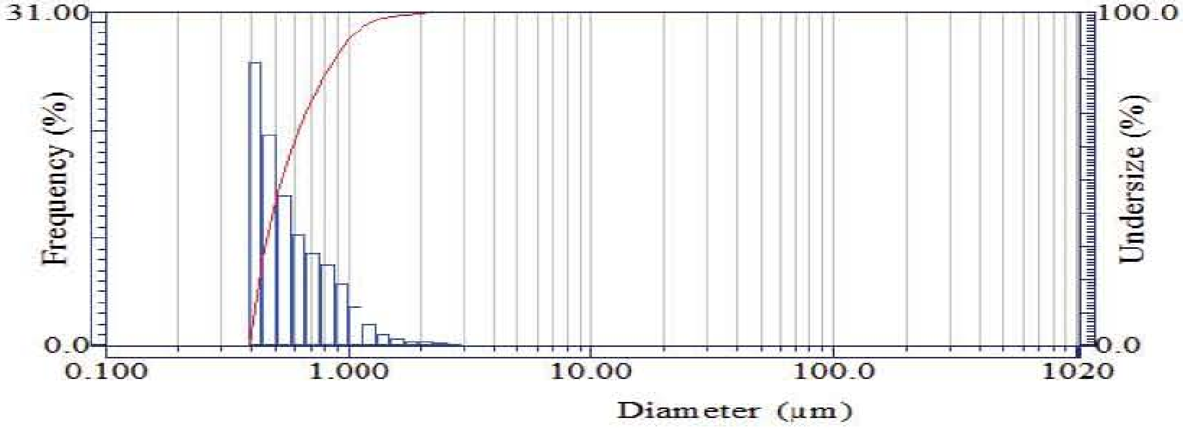
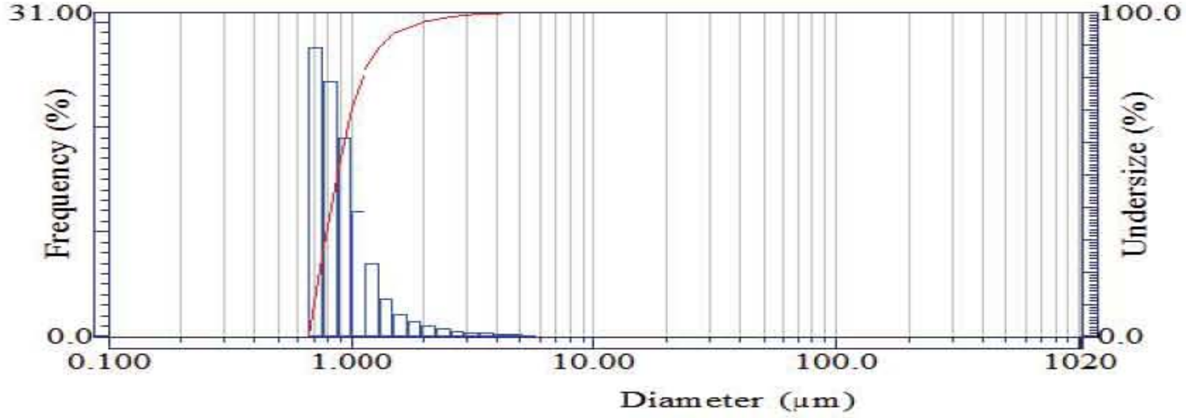


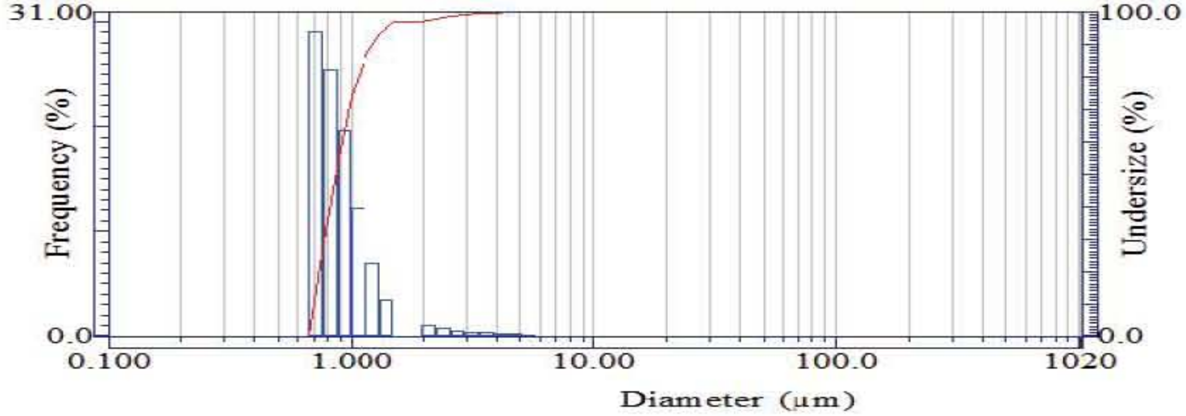
Figure C-2. Number-Based PSD Histograms and %-Undersize Curves for <600-µm C-108 Heel Solids (3 sheets)

(c) Triplicate specimen

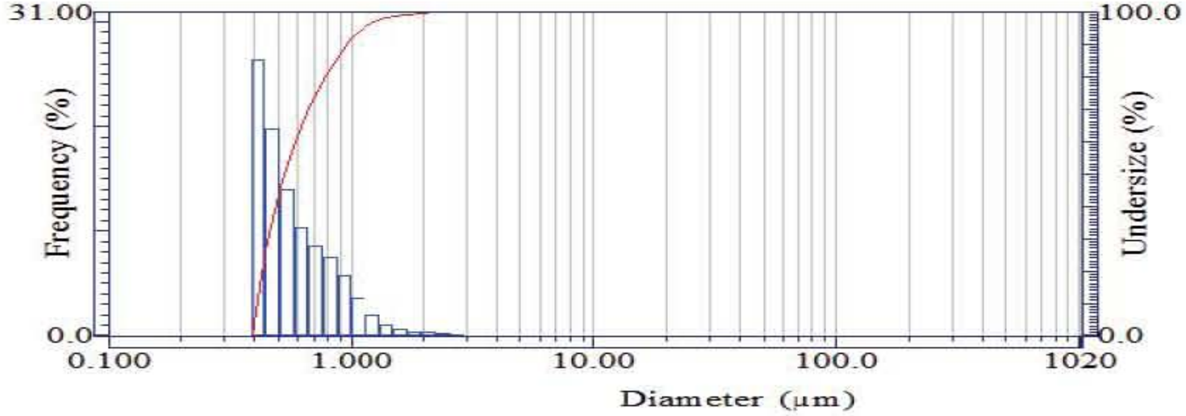
Run 1 — Stir Speed: S1-2; Pump Speed: P4-6; Sonication: None; Duration: 2.0 min



Run 2 — Stir Speed: S3; Pump Speed: P5; Sonication: None; Duration: 3.4 min



Run 3 — Stir Speed: S3; Pump Speed: P5; Sonication: 2.0 min; Duration: 3.6 min



4.2 CUMULATIVE PERCENT-UNDERSIZE DATA

The particle diameters associated with selected cut points on the volume-based cumulative %-undersize curves for the <600-µm C-108 heel solids are presented in Table C-2. The particle diameters associated with 10%, 25%, 50%, 75%, and 90% undersize cut points are tabulated. For example, in the Run 1 PSD for the primary specimen, 75% of the particulate volume was comprised of particles with spherical equivalent diameters <323 µm. The 50% undersize values are, by definition, the median particle diameters of the PSDs.

Table C-2. Volume-Based %-Undersize Data for <600-µm C-108 Heel Solids

PSD		Diameters of Particles (µm) at %-undersize Cut Points				
Run	**Specimen**	**10%**	**25%**	**50%**	**75%**	**90%**
1	Primary	62.2	134	215	323	464
	Duplicate	60.9	128	206	315	460
	Triplicate	53.9	120	192	287	411
	Average	61.5	131	211	319	462
2	Primary	58.9	123	196	291	418
	Duplicate	62.1	130	207	307	440
	Triplicate	66.8	132	204	296	418
	Average	62.3	128	202	298	425
3	Primary	25.6	77.4	144	231	339
	Duplicate	26.4	79.7	144	220	310
	Triplicate	28.8	82.2	144	216	300
	Average	26.9	79.8	144	222	317

4.3 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 <600-µm C-108 heel solids are provided on the following 12 pages. Data sheets for each of the three PSD runs on each of the three specimens of the heel solids are presented. The final three data sheets provide the data sheets for the averaged results for the primary and duplicate samples for Run 1 and for the primary, duplicate, and triplicate samples for Runs 2 and 3. 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-108 Heel Solids: <600 μm Sieve Fraction		
SAMPLE #:	S09R000307	Primary	PSD Run 1
MEASUREMENT ID:	08/26/2009	11:35	Run 742
FILENAME:	S09R307P1		

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

RRI:	1.15 – 0.20i
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): 60/30 Σ WORK TIME: 2.5 min Σ SONIC: 0 min

MEAN (μm):	245.0	MEDIAN (μm):	215.1	MODE (μm):	244.4	S.P. Area (cm^2/cm^3):	1124
σ (μm):	165.4	SPAN:	1.869				

Diameter (μm)	Frequency (%)	Undersize (%)
0.020	0.00	0.00
0.023	0.00	0.00
0.026	0.00	0.00
0.030	0.00	0.00
0.034	0.00	0.00
0.039	0.00	0.00
0.044	0.00	0.00
0.051	0.00	0.00
0.058	0.00	0.00
0.067	0.00	0.00
0.076	0.00	0.00
0.087	0.00	0.00
0.100	0.00	0.00
0.115	0.00	0.00
0.131	0.00	0.00
0.150	0.00	0.00
0.172	0.00	0.00
0.197	0.00	0.00
0.226	0.00	0.00
0.259	0.00	0.00
0.296	0.00	0.00
0.339	0.00	0.00
0.389	0.00	0.00
0.445	0.00	0.00
0.510	0.00	0.00
0.584	0.00	0.00
0.669	0.00	0.00
0.766	0.11	0.11
0.877	0.14	0.25
1.005	0.16	0.41
1.151	0.15	0.56
1.318	0.13	0.69
1.510	0.00	0.69
1.729	0.00	0.69
1.981	0.00	0.69
2.269	0.00	0.69
2.599	0.00	0.69
2.976	0.11	0.80
3.409	0.12	0.92
3.905	0.14	1.05
4.472	0.16	1.21
5.122	0.18	1.39
5.867	0.22	1.60
6.720	0.26	1.86
7.697	0.31	2.17
8.816	0.35	2.52
10.10	0.39	2.91
11.56	0.41	3.32
13.25	0.43	3.74
15.17	0.43	4.18
17.38	0.44	4.62
19.90	0.44	5.06
22.80	0.44	5.49
26.11	0.44	5.93
29.91	0.45	6.38
34.25	0.47	6.85
39.23	0.51	7.36
44.94	0.59	7.95
51.47	0.71	8.66
58.95	0.89	9.55
67.52	1.14	10.68
77.34	1.49	12.17
88.58	1.97	14.14
101.5	2.63	16.77
116.2	3.48	20.24
133.1	4.51	24.75
152.5	5.70	30.46
174.6	6.90	37.36
200.0	7.96	45.31
229.1	8.73	54.04
262.4	8.87	62.91
300.5	8.31	71.22
344.2	7.27	78.49
394.2	6.00	84.48
451.6	4.76	89.24
517.2	3.67	92.92
592.4	2.77	95.69
678.5	2.12	97.81
777.1	1.18	98.98
890.1	0.65	99.64
1019.5	0.36	100.00

SAMPLE MATERIAL:	241-C-108 Heel Solids: <600 µm Sieve Fraction		
SAMPLE #:	S09R000307	Primary	PSD Run 2
MEASUREMENT ID:	08/26/2009	11:39	Run 743
FILENAME:	S09R307P2		

INSTRUMENT:	Horiba LA-910
SUSPENSION LIQUID	Water
DISPERSANT:	None
DISPERSION METHOD:	Stirring and pumping for 2 min before measurement
FLOW REGIME:	Flow Cell Measurement

RRI:	1.15 – 0.20i
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): 60/30 Σ WORK TIME: 5.9 min Σ SONIC: 0 min

MEAN (µm):	221.7	MEDIAN (µm):	195.8	MODE (µm):	214.0	S.P. Area (cm^2/cm^3):	1309
σ (µm):	145.9	SPAN:	1.834				

Diameter (µm)	Frequency (%)	Undersize (%)
0.020	0.00	0.00
0.023	0.00	0.00
0.026	0.00	0.00
0.030	0.00	0.00
0.034	0.00	0.00
0.039	0.00	0.00
0.044	0.00	0.00
0.051	0.00	0.00
0.058	0.00	0.00
0.067	0.00	0.00
0.076	0.00	0.00
0.087	0.00	0.00
0.100	0.00	0.00
0.115	0.00	0.00
0.131	0.00	0.00
0.150	0.00	0.00
0.172	0.00	0.00
0.197	0.00	0.00
0.226	0.00	0.00
0.259	0.00	0.00
0.296	0.00	0.00
0.339	0.00	0.00
0.389	0.00	0.00
0.445	0.00	0.00
0.510	0.00	0.00
0.584	0.00	0.00
0.669	0.00	0.00
0.766	0.12	0.12
0.877	0.15	0.27
1.005	0.18	0.45
1.151	0.17	0.61
1.318	0.14	0.75
1.510	0.11	0.86
1.729	0.00	0.86
1.981	0.00	0.86
2.269	0.11	0.97
2.599	0.12	1.09
2.976	0.13	1.21
3.409	0.14	1.35
3.905	0.16	1.51
4.472	0.18	1.69
5.122	0.21	1.90
5.867	0.25	2.15
6.720	0.29	2.43
7.697	0.33	2.77
8.816	0.37	3.13
10.10	0.39	3.52
11.56	0.39	3.91
13.25	0.40	4.31
15.17	0.39	4.70
17.38	0.39	5.09
19.90	0.39	5.48
22.80	0.39	5.88
26.11	0.40	6.28
29.91	0.42	6.70
34.25	0.46	7.15
39.23	0.52	7.67
44.94	0.61	8.28
51.47	0.76	9.04
58.95	0.97	10.01
67.52	1.28	11.29
77.34	1.71	13.01
88.58	2.32	15.33
101.5	3.14	18.46
116.2	4.18	22.64
133.1	5.39	28.03
152.5	6.73	34.76
174.6	7.90	42.66
200.0	8.70	51.36
229.1	9.04	60.40
262.4	8.67	69.07
300.5	7.70	76.78
344.2	6.44	83.22
394.2	5.10	88.32
451.6	3.93	92.25
517.2	3.01	95.26
592.4	2.33	97.59
678.5	1.29	98.88
777.1	0.72	99.60
890.1	0.40	100.00
1019.5	0.00	100.00

SAMPLE MATERIAL: 241-C-108 Heel Solids: <600 μm Sieve Fraction
SAMPLE #: S09R000307 Primary PSD Run 3
MEASUREMENT ID: 08/26/2009 11:43 Run 744
FILENAME: S09R307P3

INSTRUMENT: Horiba LA-910
SUSPENSION LIQUID Water
DISPERSANT: None
DISPERSION METHOD: Stirring, pumping, and sonication for 2 min prior to measurement
FLOW REGIME: Flow Cell Measurement

RRI: 1.15 – 0.20i
DISTRIBUTION BASE: Volume
DISTRIBUTION FORM: Standard

STIR SET: 3 PUMP SET: 5 SONIC: ON DELAY: 10 s SONIC DURING MEAS: NO
MEASURE (Laser/Lamp Cycles): 60/30 Σ WORK TIME: 9.5 min Σ SONIC: 2.0 min

MEAN (μm): 169.1 MEDIAN (μm): 144.3 MODE (μm): 186.4 S.P. Area (cm^2/cm^3): 2607
σ (μm): 127.6 SPAN: 2.175

Diameter (μm)	Frequency (%)	Undersize (%)
0.020	0.00	0.00
0.023	0.00	0.00
0.026	0.00	0.00
0.030	0.00	0.00
0.034	0.00	0.00
0.039	0.00	0.00
0.044	0.00	0.00
0.051	0.00	0.00
0.058	0.00	0.00
0.067	0.00	0.00
0.076	0.00	0.00
0.087	0.00	0.00
0.100	0.00	0.00
0.115	0.00	0.00
0.131	0.00	0.00
0.150	0.00	0.00
0.172	0.00	0.00
0.197	0.00	0.00
0.226	0.00	0.00
0.259	0.00	0.00
0.296	0.00	0.00
0.339	0.00	0.00
0.389	0.00	0.00
0.445	0.11	0.11
0.510	0.12	0.23
0.584	0.13	0.37
0.669	0.15	0.52
0.766	0.19	0.70
0.877	0.25	0.95
1.005	0.29	1.24
1.151	0.27	1.51
1.318	0.23	1.73
1.510	0.17	1.91
1.729	0.15	2.06
1.981	0.16	2.22
2.269	0.17	2.39
2.599	0.17	2.55
2.976	0.18	2.73
3.409	0.19	2.92
3.905	0.21	3.13
4.472	0.23	3.35
5.122	0.26	3.61
5.867	0.29	3.90
6.720	0.34	4.24
7.697	0.39	4.63
8.816	0.44	5.07
10.10	0.48	5.54
11.56	0.51	6.05
13.25	0.54	6.59
15.17	0.58	7.18
17.38	0.63	7.81
19.90	0.69	8.50
22.80	0.77	9.27
26.11	0.85	10.12
29.91	0.96	11.08
34.25	1.09	12.17
39.23	1.28	13.44
44.94	1.51	14.96
51.47	1.82	16.78
58.95	2.21	18.99
67.52	2.70	21.69
77.34	3.29	24.97
88.58	3.99	28.97
101.5	4.80	33.77
116.2	5.65	39.41
133.1	6.42	45.83
152.5	7.03	52.86
174.6	7.32	60.18
200.0	7.34	67.52
229.1	7.04	74.56
262.4	6.34	80.89
300.5	5.32	86.21
344.2	4.23	90.44
394.2	3.25	93.68
451.6	2.46	96.15
517.2	1.89	98.04
592.4	1.05	99.09
678.5	0.58	99.68
777.1	0.33	100.00
890.1	0.00	100.00
1019.5	0.00	100.00

SAMPLE MATERIAL:	241-C-108 Heel Solids: <600 µm Sieve Fraction		
SAMPLE #:	S09R000307	Duplicate	PSD Run 1
MEASUREMENT ID:	08/26/2009	12:23	Run 745
FILENAME:	S09R307D1		

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

RRI:	1.15 – 0.20i
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): 60/30 Σ WORK TIME: 3.0 min Σ SONIC: 0 min

MEAN (µm): 239.2 MEDIAN (µm): 205.9 MODE (µm): 214.3 S.P. Area (cm^2/cm^3): 1246

σ (µm): 164.3 SPAN: 1.939

Diameter (µm)	Frequency (%)	Undersize (%)
0.020	0.00	0.00
0.023	0.00	0.00
0.026	0.00	0.00
0.030	0.00	0.00
0.034	0.00	0.00
0.039	0.00	0.00
0.044	0.00	0.00
0.051	0.00	0.00
0.058	0.00	0.00
0.067	0.00	0.00
0.076	0.00	0.00
0.087	0.00	0.00
0.100	0.00	0.00
0.115	0.00	0.00
0.131	0.00	0.00
0.150	0.00	0.00
0.172	0.00	0.00
0.197	0.00	0.00
0.226	0.00	0.00
0.259	0.00	0.00
0.296	0.00	0.00
0.339	0.00	0.00
0.389	0.00	0.00
0.445	0.00	0.00
0.510	0.00	0.00
0.584	0.00	0.00
0.669	0.00	0.00
0.766	0.11	0.11
0.877	0.15	0.25
1.005	0.17	0.42
1.151	0.16	0.58
1.318	0.14	0.72
1.510	0.10	0.82
1.729	0.00	0.82
1.981	0.00	0.82
2.269	0.10	0.92
2.599	0.10	1.03
2.976	0.11	1.14
3.409	0.12	1.26
3.905	0.14	1.40
4.472	0.16	1.56
5.122	0.19	1.75
5.867	0.22	1.97
6.720	0.26	2.24
7.697	0.31	2.55
8.816	0.35	2.90
10.10	0.38	3.28
11.56	0.40	3.68
13.25	0.41	4.09
15.17	0.42	4.51
17.38	0.42	4.92
19.90	0.41	5.33
22.80	0.41	5.75
26.11	0.41	6.16
29.91	0.42	6.58
34.25	0.45	7.03
39.23	0.50	7.53
44.94	0.58	8.12
51.47	0.71	8.83
58.95	0.90	9.73
67.52	1.17	10.90
77.34	1.56	12.46
88.58	2.11	14.57
101.5	2.87	17.44
116.2	3.87	21.30
133.1	5.03	26.34
152.5	6.32	32.66
174.6	7.40	40.06
200.0	8.14	48.20
229.1	8.48	56.68
262.4	8.27	64.95
300.5	7.64	72.59
344.2	6.75	79.34
394.2	5.64	84.99
451.6	4.53	89.51
517.2	3.55	93.06
592.4	2.72	95.78
678.5	2.08	97.85
777.1	1.15	99.00
890.1	0.64	99.64
1019.5	0.36	100.00

SAMPLE MATERIAL:	241-C-108 Heel Solids: <600 μm Sieve Fraction		
SAMPLE #:	S09R000307	Duplicate	PSD Run 2
MEASUREMENT ID:	08/26/2009	12:27	Run 746
FILENAME:	S09R307D2		

INSTRUMENT:	Horiba LA-910
SUSPENSION LIQUID	Water
DISPERSANT:	None
DISPERSION METHOD:	Stirring and pumping for 2 min before measurement
FLOW REGIME:	Flow Cell Measurement

RRI:	1.15 – 0.20i
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):	60/30	Σ WORK TIME:	6.4 min	Σ SONIC:	0 min				

MEAN (μm):	235.0	MEDIAN (μm):	206.7	MODE (μm):	215.0	S.P. Area (cm^2/cm^3):	1264
σ (μm):	157.7	SPAN:	1.828				

Diameter (μm)	Frequency (%)	Undersize (%)
0.020	0.00	0.00
0.023	0.00	0.00
0.026	0.00	0.00
0.030	0.00	0.00
0.034	0.00	0.00
0.039	0.00	0.00
0.044	0.00	0.00
0.051	0.00	0.00
0.058	0.00	0.00
0.067	0.00	0.00
0.076	0.00	0.00
0.087	0.00	0.00
0.100	0.00	0.00
0.115	0.00	0.00
0.131	0.00	0.00
0.150	0.00	0.00
0.172	0.00	0.00
0.197	0.00	0.00
0.226	0.00	0.00
0.259	0.00	0.00
0.296	0.00	0.00
0.339	0.00	0.00
0.389	0.00	0.00
0.445	0.00	0.00
0.510	0.00	0.00
0.584	0.00	0.00
0.669	0.00	0.00

Diameter (μm)	Frequency (%)	Undersize (%)
0.766	0.11	0.11
0.877	0.15	0.26
1.005	0.17	0.43
1.151	0.16	0.60
1.318	0.14	0.74
1.510	0.11	0.84
1.729	0.00	0.84
1.981	0.00	0.84
2.269	0.11	0.95
2.599	0.11	1.06
2.976	0.12	1.18
3.409	0.13	1.31
3.905	0.15	1.46
4.472	0.17	1.63
5.122	0.20	1.83
5.867	0.23	2.06
6.720	0.27	2.33
7.697	0.31	2.64
8.816	0.35	2.99
10.10	0.37	3.36
11.56	0.38	3.74
13.25	0.38	4.12
15.17	0.38	4.51
17.38	0.38	4.88
19.90	0.38	5.26
22.80	0.38	5.64
26.11	0.39	6.03

Diameter (μm)	Frequency (%)	Undersize (%)
29.91	0.40	6.43
34.25	0.43	6.86
39.23	0.49	7.35
44.94	0.58	7.93
51.47	0.71	8.64
58.95	0.90	9.55
67.52	1.18	10.72
77.34	1.56	12.29
88.58	2.10	14.39
101.5	2.84	17.23
116.2	3.80	21.03
133.1	4.93	25.96
152.5	6.20	32.16
174.6	7.37	39.53
200.0	8.32	47.85
229.1	8.92	56.77
262.4	8.89	65.66
300.5	8.20	73.85
344.2	7.05	80.91
394.2	5.64	86.55
451.6	4.28	90.83
517.2	3.17	94.00
592.4	2.34	96.34
678.5	1.80	98.14
777.1	1.00	99.14
890.1	0.56	99.69
1019.5	0.31	100.00

SAMPLE MATERIAL:	241-C-108 Heel Solids: <600 µm Sieve Fraction		
SAMPLE #:	S09R000307	Duplicate	PSD Run 3
MEASUREMENT ID:	08/26/2009	12:31	Run 747
FILENAME:	S09R307D3		

INSTRUMENT:	Horiba LA-910
SUSPENSION LIQUID	Water
DISPERSANT:	None
DISPERSION METHOD:	Stirring, pumping, and sonication for 2 min prior to measurement
FLOW REGIME:	Flow Cell Measurement

RRI:	1.15 – 0.20i
DISTRIBUTION BASE:	Volume
DISTRIBUTION FORM:	Standard

STIR SET: 3 PUMP SET: 5 SONIC: ON DELAY: 10 s SONIC DURING MEAS: NO

MEASURE (Laser/Lamp Cycles): 60/30 Σ WORK TIME: 10.0 min Σ SONIC: 2.0 min

MEAN (µm): 160.9 MEDIAN (µm): 144.1 MODE (µm): 163.8 S.P. Area (cm^2/cm^3): 2646

σ (µm): 112.8 SPAN: 1.969

Diameter (µm)	Frequency (%)	Undersize (%)
0.020	0.00	0.00
0.023	0.00	0.00
0.026	0.00	0.00
0.030	0.00	0.00
0.034	0.00	0.00
0.039	0.00	0.00
0.044	0.00	0.00
0.051	0.00	0.00
0.058	0.00	0.00
0.067	0.00	0.00
0.076	0.00	0.00
0.087	0.00	0.00
0.100	0.00	0.00
0.115	0.00	0.00
0.131	0.00	0.00
0.150	0.00	0.00
0.172	0.00	0.00
0.197	0.00	0.00
0.226	0.00	0.00
0.259	0.00	0.00
0.296	0.00	0.00
0.339	0.00	0.00
0.389	0.00	0.00
0.445	0.12	0.12
0.510	0.13	0.25
0.584	0.14	0.39
0.669	0.16	0.54
0.766	0.19	0.74
0.877	0.26	0.99
1.005	0.29	1.29
1.151	0.27	1.56
1.318	0.23	1.79
1.510	0.17	1.96
1.729	0.15	2.11
1.981	0.16	2.26
2.269	0.17	2.43
2.599	0.17	2.60
2.976	0.18	2.78
3.409	0.19	2.96
3.905	0.21	3.17
4.472	0.22	3.39
5.122	0.25	3.64
5.867	0.29	3.93
6.720	0.33	4.26
7.697	0.38	4.64
8.816	0.43	5.06
10.10	0.46	5.52
11.56	0.49	6.01
13.25	0.53	6.54
15.17	0.56	7.10
17.38	0.61	7.71
19.90	0.67	8.38
22.80	0.73	9.11
26.11	0.81	9.92
29.91	0.91	10.83
34.25	1.04	11.87
39.23	1.22	13.09
44.94	1.44	14.53
51.47	1.74	16.27
58.95	2.11	18.38
67.52	2.58	20.96
77.34	3.17	24.13
88.58	3.91	28.04
101.5	4.80	32.83
116.2	5.81	38.65
133.1	6.83	45.47
152.5	7.74	53.21
174.6	8.22	61.43
200.0	8.21	69.64
229.1	7.65	77.28
262.4	6.57	83.85
300.5	5.24	89.09
344.2	3.93	93.02
394.2	2.83	95.85
451.6	2.04	97.89
517.2	1.13	99.02
592.4	0.63	99.65
678.5	0.35	100.00
777.1	0.00	100.00
890.1	0.00	100.00
1019.5	0.00	100.00

SAMPLE MATERIAL:	241-C-108 Heel Solids: <600 µm Sieve Fraction		
SAMPLE #:	S09R000307	Triplicate	PSD Run 1
MEASUREMENT ID:	08/26/2009	13:00	Run 748
FILENAME:	S09R307T1		

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

RRI:	1.15 – 0.20i
DISTRIBUTION BASE:	Volume
DISTRIBUTION FORM:	Standard

STIR SET: 1 PUMP SET: 6 SONIC: OFF DELAY: 0 s SONIC DURING MEAS: NO

MEASURE (Laser/Lamp Cycles): 60/30 Σ WORK TIME: 2.0 min Σ SONIC: 0 min

MEAN (µm):	217.7	MEDIAN (µm):	192.3	MODE (µm):	213.9	S.P. Area (cm^2/cm^3):	1411
σ (µm):	145.0	SPAN:	1.858				

Diameter (µm)	Frequency (%)	Undersize (%)	Diameter (µm)	Frequency (%)	Undersize (%)	Diameter (µm)	Frequency (%)	Undersize (%)
0.020	0.00	0.00	0.766	0.12	0.12	29.91	0.45	7.22
0.023	0.00	0.00	0.877	0.16	0.27	34.25	0.48	7.70
0.026	0.00	0.00	1.005	0.18	0.46	39.23	0.54	8.24
0.030	0.00	0.00	1.151	0.17	0.63	44.94	0.64	8.88
0.034	0.00	0.00	1.318	0.15	0.78	51.47	0.78	9.66
0.039	0.00	0.00	1.510	0.12	0.89	58.95	1.00	10.66
0.044	0.00	0.00	1.729	0.10	1.00	67.52	1.32	11.98
0.051	0.00	0.00	1.981	0.11	1.10	77.34	1.76	13.74
0.058	0.00	0.00	2.269	0.12	1.22	88.58	2.39	16.13
0.067	0.00	0.00	2.599	0.12	1.34	101.5	3.24	19.36
0.076	0.00	0.00	2.976	0.13	1.46	116.2	4.30	23.67
0.087	0.00	0.00	3.409	0.14	1.60	133.1	5.51	29.18
0.100	0.00	0.00	3.905	0.15	1.75	152.5	6.82	36.00
0.115	0.00	0.00	4.472	0.17	1.92	174.6	7.89	43.88
0.131	0.00	0.00	5.122	0.20	2.12	200.0	8.61	52.49
0.150	0.00	0.00	5.867	0.23	2.36	229.1	8.92	61.41
0.172	0.00	0.00	6.720	0.28	2.63	262.4	8.54	69.95
0.197	0.00	0.00	7.697	0.33	2.96	300.5	7.57	77.52
0.226	0.00	0.00	8.816	0.37	3.33	344.2	6.31	83.83
0.259	0.00	0.00	10.10	0.40	3.73	394.2	4.99	88.81
0.296	0.00	0.00	11.56	0.42	4.15	451.6	3.81	92.62
0.339	0.00	0.00	13.25	0.43	4.59	517.2	2.89	95.51
0.389	0.00	0.00	15.17	0.44	5.02	592.4	2.20	97.72
0.445	0.00	0.00	17.38	0.44	5.46	678.5	1.23	98.94
0.510	0.00	0.00	19.90	0.44	5.90	777.1	0.68	99.62
0.584	0.00	0.00	22.80	0.43	6.33	890.1	0.38	100.00
0.669	0.00	0.00	26.11	0.44	6.77	1019.5	0.00	100.00

SAMPLE MATERIAL:	241-C-108 Heel Solids: <600 µm Sieve Fraction		
SAMPLE #:	S09R000307	Triplicate	PSD Run 2
MEASUREMENT ID:	08/26/2009	13:04	Run 749
FILENAME:	S09R307T2		

INSTRUMENT:	Horiba LA-910
SUSPENSION LIQUID	Water
DISPERSANT:	None
DISPERSION METHOD:	Stirring and pumping for 2 min before measurement
FLOW REGIME:	Flow Cell Measurement

RRI:	1.15 – 0.20i
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): 60/30 Σ WORK TIME: 5.4 min Σ SONIC: 0 min

MEAN (µm):	227.3	MEDIAN (µm):	204.0	MODE (µm):	214.7	S.P. Area (cm^2/cm^3):	1226
σ (µm):	143.6	SPAN:	1.722				

Diameter (µm)	Frequency (%)	Undersize (%)
0.020	0.00	0.00
0.023	0.00	0.00
0.026	0.00	0.00
0.030	0.00	0.00
0.034	0.00	0.00
0.039	0.00	0.00
0.044	0.00	0.00
0.051	0.00	0.00
0.058	0.00	0.00
0.067	0.00	0.00
0.076	0.00	0.00
0.087	0.00	0.00
0.100	0.00	0.00
0.115	0.00	0.00
0.131	0.00	0.00
0.150	0.00	0.00
0.172	0.00	0.00
0.197	0.00	0.00
0.226	0.00	0.00
0.259	0.00	0.00
0.296	0.00	0.00
0.339	0.00	0.00
0.389	0.00	0.00
0.445	0.00	0.00
0.510	0.00	0.00
0.584	0.00	0.00
0.669	0.00	0.00
0.766	0.11	0.11
0.877	0.15	0.26
1.005	0.17	0.43
1.151	0.16	0.58
1.318	0.13	0.72
1.510	0.10	0.82
1.729	0.00	0.82
1.981	0.00	0.82
2.269	0.10	0.92
2.599	0.11	1.03
2.976	0.11	1.14
3.409	0.12	1.26
3.905	0.14	1.40
4.472	0.16	1.56
5.122	0.18	1.74
5.867	0.21	1.95
6.720	0.25	2.19
7.697	0.29	2.48
8.816	0.32	2.81
10.10	0.35	3.15
11.56	0.36	3.51
13.25	0.36	3.87
15.17	0.36	4.23
17.38	0.36	4.59
19.90	0.36	4.94
22.80	0.36	5.30
26.11	0.36	5.66
29.91	0.37	6.03
34.25	0.40	6.44
39.23	0.46	6.90
44.94	0.54	7.44
51.47	0.67	8.11
58.95	0.86	8.97
67.52	1.13	10.09
77.34	1.51	11.61
88.58	2.07	13.67
101.5	2.83	16.50
116.2	3.85	20.35
133.1	5.07	25.42
152.5	6.49	31.91
174.6	7.82	39.73
200.0	8.90	48.62
229.1	9.54	58.16
262.4	9.35	67.51
300.5	8.37	75.87
344.2	6.95	82.82
394.2	5.42	88.24
451.6	4.08	92.32
517.2	3.04	95.36
592.4	2.28	97.64
678.5	1.27	98.91
777.1	0.70	99.61
890.1	0.39	100.00
1019.5	0.00	100.00

SAMPLE MATERIAL:	241-C-108 Heel Solids: <600 µm Sieve Fraction		
SAMPLE #:	S009R000307	Triplicate	PSD Run 3
MEASUREMENT ID:	08/26/2009	13:07	Run 750
FILENAME:	S09R307T3		

INSTRUMENT:	Horiba LA-910
SUSPENSION LIQUID	Water
DISPERSANT:	None
DISPERSION METHOD:	Stirring, pumping, and sonication for 2 min prior to measurement
FLOW REGIME:	Flow Cell Measurement

RRI:	1.15 – 0.20i
DISTRIBUTION BASE:	Volume
DISTRIBUTION FORM:	Standard

STIR SET:	3	PUMP SET:	5	SONIC:	ON	DELAY:	10 s	SONIC DURING MEAS:	NO
MEASURE (Laser/Lamp Cycles):	60/30	Σ WORK TIME:	9.0 min	Σ SONIC:	2.0 min				

MEAN (µm):	159.2	MEDIAN (µm):	143.8	MODE (µm):	163.7	S.P. Area (cm^2/cm^3):	2605
σ (µm):	109.0	SPAN:	1.887				

Diameter (µm)	Frequency (%)	Undersize (%)
0.020	0.00	0.00
0.023	0.00	0.00
0.026	0.00	0.00
0.030	0.00	0.00
0.034	0.00	0.00
0.039	0.00	0.00
0.044	0.00	0.00
0.051	0.00	0.00
0.058	0.00	0.00
0.067	0.00	0.00
0.076	0.00	0.00
0.087	0.00	0.00
0.100	0.00	0.00
0.115	0.00	0.00
0.131	0.00	0.00
0.150	0.00	0.00
0.172	0.00	0.00
0.197	0.00	0.00
0.226	0.00	0.00
0.259	0.00	0.00
0.296	0.00	0.00
0.339	0.00	0.00
0.389	0.00	0.00
0.445	0.12	0.12
0.510	0.13	0.25
0.584	0.14	0.39
0.669	0.16	0.54
0.766	0.19	0.74
0.877	0.26	0.99
1.005	0.29	1.29
1.151	0.27	1.55
1.318	0.23	1.78
1.510	0.17	1.95
1.729	0.15	2.09
1.981	0.15	2.25
2.269	0.16	2.41
2.599	0.16	2.57
2.976	0.17	2.74
3.409	0.18	2.92
3.905	0.19	3.11
4.472	0.21	3.32
5.122	0.24	3.56
5.867	0.27	3.82
6.720	0.31	4.13
7.697	0.35	4.48
8.816	0.40	4.88
10.10	0.43	5.31
11.56	0.46	5.77
13.25	0.49	6.25
15.17	0.52	6.78
17.38	0.57	7.34
19.90	0.62	7.96
22.80	0.68	8.64
26.11	0.75	9.40
29.91	0.85	10.24
34.25	0.97	11.21
39.23	1.14	12.35
44.94	1.37	13.72
51.47	1.66	15.38
58.95	2.05	17.43
67.52	2.56	19.99
77.34	3.20	23.19
88.58	4.01	27.20
101.5	4.99	32.19
116.2	6.07	38.26
133.1	7.14	45.40
152.5	8.11	53.51
174.6	8.59	62.10
200.0	8.50	70.60
229.1	7.81	78.41
262.4	6.57	84.98
300.5	5.08	90.06
344.2	3.68	93.75
394.2	2.58	96.32
451.6	1.81	98.13
517.2	1.00	99.13
592.4	0.56	99.69
678.5	0.31	100.00
777.1	0.00	100.00
890.1	0.00	100.00
1019.5	0.00	100.00

SAMPLE MATERIAL:	241-C-108 Heel Solids: <600 μm Sieve Fraction		
SAMPLE #:	S09R000307	—	PSD Run 1 Average
MEASUREMENT ID:	08/26/2009	—	(Primary and Duplicate)
FILENAME:	S09R307Avg1		

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:	1.15 – 0.20i
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): 60/30 Σ WORK TIME: 2.8 min Σ SONIC: 0 min

MEAN (μm):	242.1	MEDIAN (μm):	210.5	MODE (μm):	214.9	S.P. Area (cm^2/cm^3):	1185
σ (μm):	164.4	SPAN:	1.904				

Diameter (μm)	Frequency (%)	Undersize (%)
0.020	0.00	0.00
0.023	0.00	0.00
0.026	0.00	0.00
0.030	0.00	0.00
0.034	0.00	0.00
0.039	0.00	0.00
0.044	0.00	0.00
0.051	0.00	0.00
0.058	0.00	0.00
0.067	0.00	0.00
0.076	0.00	0.00
0.087	0.00	0.00
0.100	0.00	0.00
0.115	0.00	0.00
0.131	0.00	0.00
0.150	0.00	0.00
0.172	0.00	0.00
0.197	0.00	0.00
0.226	0.00	0.00
0.259	0.00	0.00
0.296	0.00	0.00
0.339	0.00	0.00
0.389	0.00	0.00
0.445	0.00	0.00
0.510	0.00	0.00
0.584	0.00	0.00
0.669	0.00	0.00
0.766	0.11	0.11
0.877	0.14	0.25
1.005	0.17	0.42
1.151	0.16	0.57
1.318	0.13	0.70
1.510	0.05	0.75
1.729	0.00	0.75
1.981	0.00	0.75
2.269	0.05	0.80
2.599	0.05	0.86
2.976	0.11	0.97
3.409	0.12	1.09
3.905	0.14	1.23
4.472	0.16	1.39
5.122	0.19	1.57
5.867	0.22	1.79
6.720	0.26	2.05
7.697	0.31	2.36
8.816	0.35	2.71
10.10	0.38	3.09
11.56	0.41	3.50
13.25	0.42	3.92
15.17	0.43	4.34
17.38	0.43	4.77
19.90	0.43	5.20
22.80	0.43	5.62
26.11	0.43	6.05
29.91	0.43	6.48
34.25	0.46	6.94
39.23	0.51	7.45
44.94	0.59	8.03
51.47	0.71	8.74
58.95	0.89	9.64
67.52	1.15	10.79
77.34	1.52	12.31
88.58	2.04	14.35
101.5	2.75	17.10
116.2	3.67	20.77
133.1	4.77	25.54
152.5	6.01	31.56
174.6	7.15	38.71
200.0	8.05	46.75
229.1	8.61	55.36
262.4	8.57	63.93
300.5	7.97	71.90
344.2	7.01	78.92
394.2	5.82	84.74
451.6	4.64	89.38
517.2	3.61	92.99
592.4	2.75	95.73
678.5	2.10	97.83
777.1	1.17	98.99
890.1	0.65	99.64
1019.5	0.36	100.00

SAMPLE MATERIAL:	241-C-108 Heel Solids: <600 μm Sieve Fraction		
SAMPLE #:	S09R000307	—	PSD Run 2 Average
MEASUREMENT ID:	08/26/2009	—	(Primary, Duplicate, and Triplicate)
FILENAME:	S09R307Avg2		

INSTRUMENT:	Horiba LA-910
SUSPENSION LIQUID	Water
DISPERSANT:	None
DISPERSION METHOD:	Stirring and pumping for 2 min before measurement
FLOW REGIME:	Flow Cell Measurement

RRI:	1.15 – 0.20i
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): 60/30 Σ WORK TIME: 5.9 min Σ SONIC: 0 min

MEAN (μm):	228.0	MEDIAN (μm):	202.2	MODE (μm):	214.6	S.P. Area (cm^2/cm^3):	1266
σ (μm):	149.3	SPAN:	1.796				

Diameter (μm)	Frequency (%)	Undersize (%)	Diameter (μm)	Frequency (%)	Undersize (%)	Diameter (μm)	Frequency (%)	Undersize (%)
0.020	0.00	0.00	0.766	0.11	0.11	29.91	0.40	6.39
0.023	0.00	0.00	0.877	0.15	0.26	34.25	0.43	6.82
0.026	0.00	0.00	1.005	0.17	0.44	39.23	0.49	7.31
0.030	0.00	0.00	1.151	0.16	0.60	44.94	0.58	7.89
0.034	0.00	0.00	1.318	0.14	0.74	51.47	0.71	8.60
0.039	0.00	0.00	1.510	0.11	0.84	58.95	0.91	9.51
0.044	0.00	0.00	1.729	0.00	0.84	67.52	1.19	10.70
0.051	0.00	0.00	1.981	0.00	0.84	77.34	1.60	12.30
0.058	0.00	0.00	2.269	0.11	0.95	88.58	2.16	14.46
0.067	0.00	0.00	2.599	0.11	1.06	101.5	2.94	17.40
0.076	0.00	0.00	2.976	0.12	1.18	116.2	3.94	21.34
0.087	0.00	0.00	3.409	0.13	1.31	133.1	5.13	26.47
0.100	0.00	0.00	3.905	0.15	1.46	152.5	6.48	32.94
0.115	0.00	0.00	4.472	0.17	1.63	174.6	7.70	40.64
0.131	0.00	0.00	5.122	0.20	1.82	200.0	8.64	49.28
0.150	0.00	0.00	5.867	0.23	2.05	229.1	9.17	58.44
0.172	0.00	0.00	6.720	0.27	2.32	262.4	8.97	67.41
0.197	0.00	0.00	7.697	0.31	2.63	300.5	8.09	75.50
0.226	0.00	0.00	8.816	0.35	2.98	344.2	6.81	82.32
0.259	0.00	0.00	10.10	0.37	3.34	394.2	5.39	87.70
0.296	0.00	0.00	11.56	0.38	3.72	451.6	4.10	91.80
0.339	0.00	0.00	13.25	0.38	4.10	517.2	3.08	94.87
0.389	0.00	0.00	15.17	0.38	4.48	592.4	2.32	97.19
0.445	0.00	0.00	17.38	0.38	4.86	678.5	1.45	98.64
0.510	0.00	0.00	19.90	0.38	5.23	777.1	0.81	99.45
0.584	0.00	0.00	22.80	0.38	5.61	890.1	0.45	99.90
0.669	0.00	0.00	26.11	0.38	5.99	1019.5	0.10	100.00

SAMPLE MATERIAL:	241-C-108 Heel Solids: <600 µm Sieve Fraction
SAMPLE #:	S09R000307 — PSD Run 3 Average
MEASUREMENT ID:	08/26/2009 — (Primary, Duplicate, and Triplicate)
FILENAME:	S09R307Avg3

INSTRUMENT:	Horiba LA-910
SUSPENSION LIQUID	Water
DISPERSANT:	None
DISPERSION METHOD:	Stirring, pumping and sonication for 2 min prior to measurement
FLOW REGIME:	Flow Cell Measurement

RRI:	1.15 – 0.20i
DISTRIBUTION BASE:	Volume
DISTRIBUTION FORM:	Standard

STIR SET: 3 PUMP SET: 5 SONIC: ON DELAY: 10 s SONIC DURING MEAS: NO

MEASURE (Laser/Lamp Cycles): 60/30 Σ WORK TIME: 9.5 min Σ SONIC: 2.0 min

MEAN (µm): 163.1 MEDIAN (µm): 144.0 MODE (µm): 163.7 S.P. Area (cm^2/cm^3): 2619

σ (µm): 116.8 SPAN: 2.014

Diameter (µm)	Frequency (%)	Undersize (%)
0.020	0.00	0.00
0.023	0.00	0.00
0.026	0.00	0.00
0.030	0.00	0.00
0.034	0.00	0.00
0.039	0.00	0.00
0.044	0.00	0.00
0.051	0.00	0.00
0.058	0.00	0.00
0.067	0.00	0.00
0.076	0.00	0.00
0.087	0.00	0.00
0.100	0.00	0.00
0.115	0.00	0.00
0.131	0.00	0.00
0.150	0.00	0.00
0.172	0.00	0.00
0.197	0.00	0.00
0.226	0.00	0.00
0.259	0.00	0.00
0.296	0.00	0.00
0.339	0.00	0.00
0.389	0.00	0.00
0.445	0.11	0.11
0.510	0.13	0.24
0.584	0.14	0.38
0.669	0.15	0.53
0.766	0.19	0.73
0.877	0.25	0.98
1.005	0.29	1.27
1.151	0.27	1.54
1.318	0.23	1.77
1.510	0.17	1.94
1.729	0.15	2.09
1.981	0.16	2.24
2.269	0.17	2.41
2.599	0.17	2.57
2.976	0.17	2.75
3.409	0.18	2.93
3.905	0.20	3.13
4.472	0.22	3.35
5.122	0.25	3.60
5.867	0.28	3.88
6.720	0.33	4.21
7.697	0.37	4.58
8.816	0.42	5.00
10.10	0.46	5.46
11.56	0.49	5.94
13.25	0.52	6.46
15.17	0.56	7.02
17.38	0.60	7.62
19.90	0.66	8.28
22.80	0.73	9.01
26.11	0.81	9.81
29.91	0.90	10.72
34.25	1.03	11.75
39.23	1.21	12.96
44.94	1.44	14.40
51.47	1.74	16.14
58.95	2.13	18.27
67.52	2.61	20.88
77.34	3.22	24.10
88.58	3.97	28.07
101.5	4.86	32.93
116.2	5.84	38.77
133.1	6.80	45.57
152.5	7.63	53.19
174.6	8.04	61.24
200.0	8.02	69.25
229.1	7.50	76.75
262.4	6.49	83.24
300.5	5.21	88.45
344.2	3.95	92.40
394.2	2.89	95.29
451.6	2.10	97.39
517.2	1.34	98.73
592.4	0.75	99.48
678.5	0.42	99.89
777.1	0.11	100.00
890.1	0.00	100.00
1019.5	0.00	100.00

5.0 DISCUSSION OF MEASUREMENT RESULTS

Discussion of the results of these PSD measurements in relation to the characterization of the C-108 heel 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 600-µm sieve, the absence of any significant quantity of particulates >1020 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. Indeed, the presence of small quantities of solids with diameters >600 µm indicated in some of the calculated PSDs is probably an artifact of the peak broadening created by the deconvolution and inversion algorithms.

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 <600-µm C-108 heel 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 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 increased slightly and the duration of their action of those

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[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.

forces 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 agglomerates was occurring slowly, if at all, under the low-shear conditions applied. The Run 3 distributions shifted to smaller particle diameters. It is not known whether this shift is caused by the break-up of friable solid aggregates or dissolution of sample solids or both.

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 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 particles 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 ($k_p i$) of the 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 <600-µm C-108 heel solids, the light-scattering-based PSD measurements indicated that the majority of the particulate volume (and presumably mass) resides in particles with diameters >50 µm. This is generally supported by the observed settling behavior of the solids; however, the observed settling behavior also suggests that a significant population of particles (on a number basis) with significantly smaller diameters are also present. Also, though optical microscopy and visual observation indicated most of the <600-µm C-108 heel solids were translucent and/or very lightly colored, some were strongly colored or opaque.

The real index of refraction assigned to the <600-µm C-108 heel solids was $n_p = 1.535$. This is a volume-weighted average of the real refractive indexes of a proposed mixture of solid phases that may have been present in the PSD specimens during measurement. Even if the mixture of solids present were well known, the use of an average value for n_p for calculation of light-scattering-based PSDs is not supported by experiment or theory. The assignment of $k_p i = 0.20i$ for the particulate solids was based on the visual appearance of the PSD sample and software driven expediency, but is also unsupported by experiment or theory.

The real index of refraction of pure water is $n_m = 1.333$. While the actual liquid medium in the PSD measurement slurries was certainly no longer pure water, it is believed that the n_m for pure water is sufficiently accurate. Also, if both are reasonably accurate, the values for n_p and n_m are sufficiently different to allow light-scattering-based measurements to be completed successfully.

If the RRI of 1.15 – 0.20i applies, with moderate tolerance, to the majority of the <600-µm C-108 heel 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, a single particle 200 µm in diameter in a population of 1×10^6 particles, the balance of which are all 2 µm in diameter, would have a number-based occurrence frequency of 0.0001%. However, in a volume-based distribution the single 200-µm 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, when these size specimens are taken and analyzed, the target %-transmission limits for the PSD suspensions are often exceeded; indeed, the target values for the small diameter particles are very often exceeded. If smaller specimen sizes are used, the accuracy in representation of the relative amounts of larger particles present is potentially sacrificed in order to limit the effects of multiple scattering by the smaller.

It was relatively easy to achieve the desired levels of light obscuration when loading the <600-µm C-108 solids into the PSD analyzer. Accordingly, PSD artifacts caused by multiple scattering should be insignificant. However, given the measured PSD data presented in Figure C-1 and Table C-2, the minimum specimen size of a statistically representative sample of the <600-µm C-108 heel solids could weigh tens of grams[16]. The quantity of C-108 heel solids in the PSD specimen slurries was significantly less than this estimated minimum value. The quantitative accuracy of the PSD results is, therefore, possibly somewhat compromised.

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. For the measurements described in this report, a multitude of sampling steps and test operations separate the heel solids in C-108 from the subsample of <600-µm solids introduced into the PSD analyzer. The degree to which the later can or should be taken to represent the former must be carefully considered when evaluating or applying the reported PSD results.

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[16] For example, assume the following for the <600-µm C-108 heel solids: density = 2.0 g/cm^3; coarsest class of solids ranges from 375-500 µm; coarsest solids comprise 10.0 % by weight of the sample. To achieve a sampling error of 10%, a subsample weighing 71 g would need to be analyzed.

6.0 QUALITY CONTROL

6.1 REPEAT MEASUREMENTS

As previously noted, PSD measurements were performed on triplicate portions (specimens) withdrawn from sample S09R000307 of the <600-µm subsample of the C-108 heel solids. The distributions calculated for each of the three PSD runs on the triplicate specimens were very 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 a high 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 nanospheres and microspheres acquired from Thermo Scientific.[17] 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% relative percent difference (RPD) 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 #3495A (Lot 34485) Polystyrene nanospheres	491 ± 6.3 nm	530 ± 28.8 nm (+7.9 RPD)	200908261523752 PS-495nm-082609a
Thermo Scientific #4205A (Lot 34651) Polystyrene microspheres	5.003 ± 0.05 µm	5.390 ± 0.53 µm (+7.7 RPD)	200908271012754 PS-5um-082709a
Thermo Scientific #4250A (Lot 34618) Polystyrene microspheres	50.2 ± 0.7 µm	53.4 ± 3.7 µm (+6.4 RPD)	200908260930741 PS-50um-082609a
Thermo Scientific #4314A (Lot 35467) Polystyrene microspheres	138 ± 2.5 µm	141 ± 14.4 µm (+2.2 RPD)	200908261540753 PS-140um-082609a
Thermo Scientific #4350A (Lot 35263) Polystyrene divinlybenzene microspheres	494 ± 25.2 µm	488 ± 32.8 µm (–1.2 RPD)	200908261501751 PS-500um-082609a

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. The small

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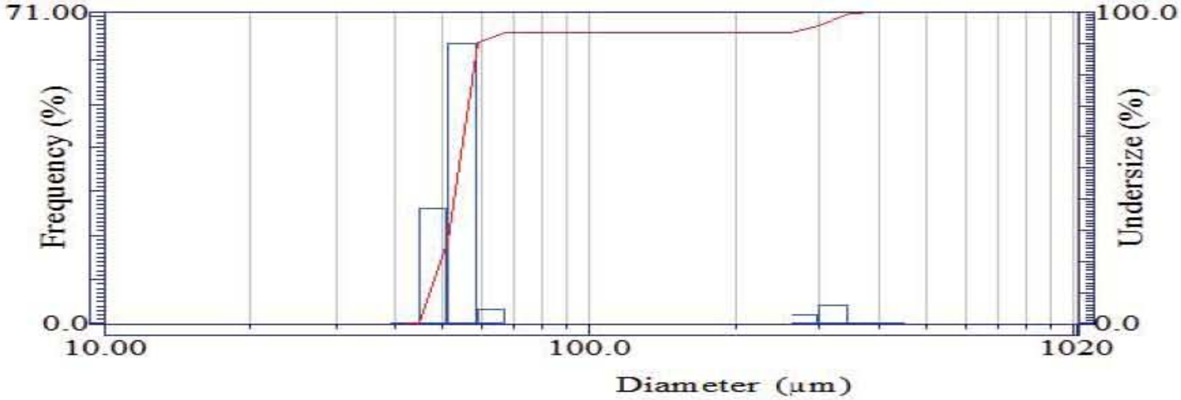
[17] Thermo Fisher Scientific Inc., Waltham, Massachusetts.

contaminant peaks observed in the histograms for the 5-µm, 50-µm, and 500-µm standard measurements were excluded from the calculations of the measured mean diameters.

Figure C-3. Volume-based Histograms and %-Undersize Curves for Particle Size Distribution Standards (2 sheets)

(a) Thermo Scientific 4250A (Lot 34618)
Polystyrene spheres
Distribution form: Sharp

Run Date: August 26, 2009—07:41
Certified Mean Diameter: 50.2 ± 0.7 µm
Calculated Mean Diameter: 53.4 ± 3.7 µm



(b) Thermo Scientific 4350A (Lot 35263)
Polystyrene divinylbenzene spheres
Distribution form: Sharp

Run Date: August 26, 2009—15:01
Certified Mean Diameter: 494 ± 25.2 µm
Calculated Mean Diameter: 488 ± 32.8 µm

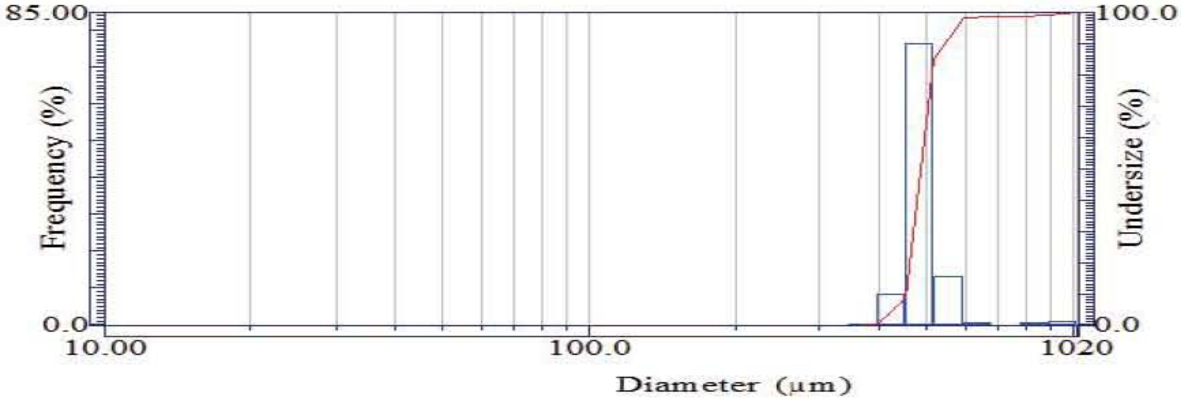
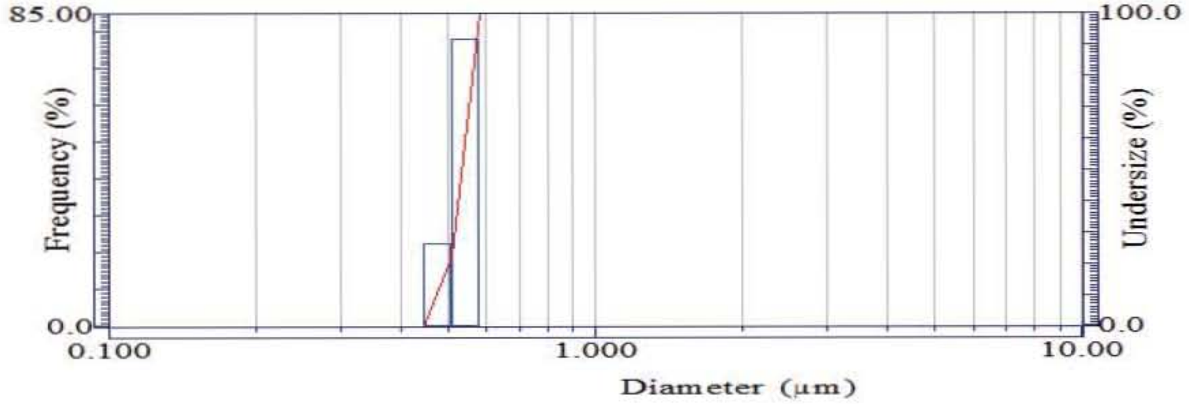


Figure C-3. Volume-based Histograms and %-Undersize Curves for Particle Size Distribution Standards (2 sheets)

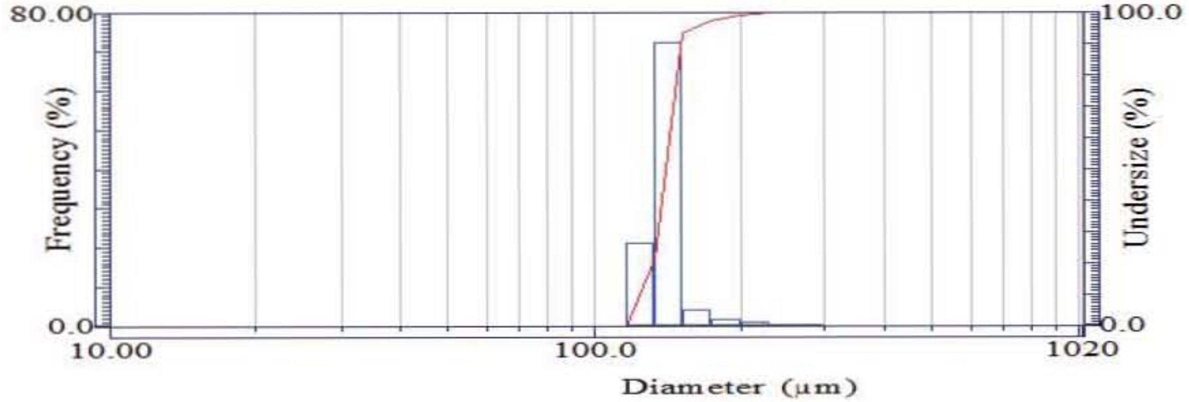
(c) Thermo Scientific 3495A (Lot 34485)
Polystyrene spheres
Distribution form: Sharp

Run Date: August 26, 2009—15:23
Certified Mean Diameter: 491 ± 6.3 nm
Calculated Mean Diameter: 530 ± 28.8 nm



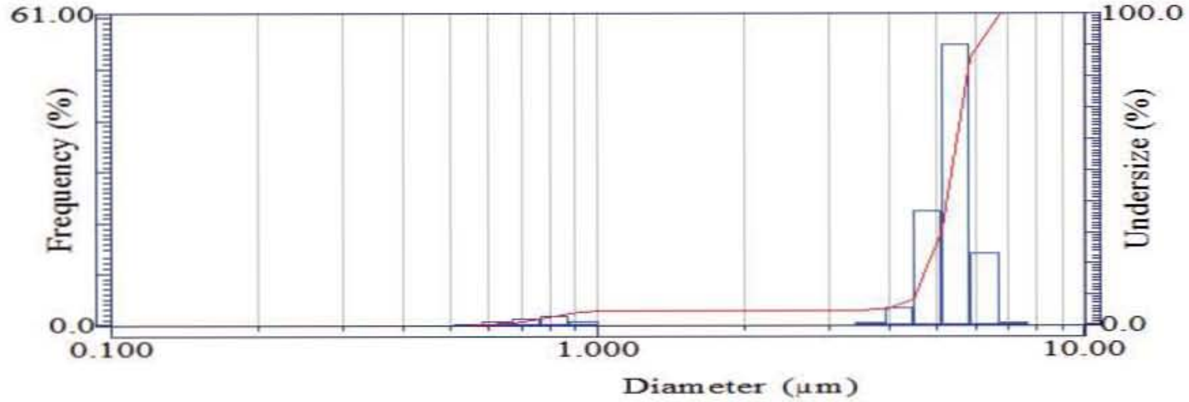
(d) Thermo Scientific 4314A (Lot 35467)
Polystyrene spheres
Distribution form: Sharp

Run Date: August 26, 2009—15:40
Certified Mean Diameter: 138 ± 2.5 µm
Calculated Mean Diameter: 141 ± 14.4 µm



(e) Thermo Scientific 4205A (Lot 34651)
Polystyrene spheres
Distribution form: Sharp

Run Date: August 27, 2009—10:12
Certified Mean Diameter: 5.003 ± 0.05 µm
Calculated Mean Diameter: 5.390 ± 0.53 µm



7.0 REFERENCES

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ATTACHMENT D

CAUSTIC DISSOLUTION RESULTS

ATTACHMENT D – CAUSTIC DISSOLUTION RESULTS

Table D-1. Mass of Vials Throughout the Dissolution Tests (2 sheets)

All data in [g]		5-Aug						10-Aug	24-Aug	26-Aug		27-Aug	
Vial number		Empty vial	Solids added			Net solids	Caustic added	Leaking check	Leaking check	Decant[18]	Water added	Decant	Water added
C1	Green	13.860	17.838			3.978	33.369*	33.373	33.548	31.307	65.769	20.551	59.648
C2	White	13.709	27.665			13.956	44.349*	44.346	44.457	28.653	64.754	30.925	63.715
C3	Black	13.704	15.68			1.976	30.24*	30.239	30.288	19.971	59.235	14.598	57.516
C4	Tan	13.935	31.79			17.855	54.714*	54.71	52.109	46.107	72.648	27.335	63.438
C5	Tan	13.654	22.854			9.200	37.525*	37.523	37.443	33.302	64.024	18.345	59.201
C6	Tan	14.282	31.592			17.310	52.493*	52.489	51.118	44.487	71.002	30.165	63.627
C7	White	13.813	33.545			19.732	57.024*	57.026	57.145	35.015	67.772	37.678	66.981
Sum coarse						84.01				141.89		82.64	
Vial number		Empty vial	Solids added	Centrif. Decant	Volume [mL]	Net solids	Caustic added	Leaking check	Leaking check	Decant[19]	Water added	Decant	Water added
F1	Mix	13.795	42.338	41.014	15	27.219	74.694	74.681	74.694	60.433	77.830	33.995	67.646
F2	Mix	13.699	43.532	42.514	16	28.815	78.325	78.393	78.460	62.396	77.800	38.339	68.575
F3	Mix	13.855	43.209	42.091	16	28.236	75.602	75.595	75.634	60.653	76.724	36.691	67.838
F4	Mix	13.643	43.897	43.047	16.5	29.404	76.709	76.703	76.729	57.192	76.071	34.566	68.361
F5	Mix	13.815	43.347	42.235	17	28.420	76.959	76.968	77.020	60.145	76.217	43.567	70.897
F6	Mix	13.837	44.575	43.498	17.5	29.661	77.168	77.163	77.270	62.822	76.945	43.799	72.204
F7	Mix	14.094	44.171	42.828	17	28.734	77.939	78.016	76.352	59.543	76.935	36.508	68.765
F8	Mix	13.940	40.084	30.791	10	16.851	50.405	50.033	49.653	38.029	67.041	24.951	63.387
Sum fines					125	217.34				350.54		181.74	
Sum all						301.35				492.42		264.38	

All data for vials plus contents in [g] except otherwise noted. Grey shaded areas are net values (actual weight minus “empty vial”).

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[18] Liquid sample archived as S09R000165.

[19] Liquid sample archived as S09R000154.

Table D-1. Mass of Vials Throughout the Dissolution Tests (2 sheets)

All data in [g]	28-Aug		31-Aug		1-Sep		2-Sep			3-Sep		8-Sep
Vial number	**Decant**	**Water added**	**Decant**	**Water added**	**Decant**	**Water added**	**Decant**	**C4 + C5**	**Water added**	**Decant**		**Net residual**
C1	19.143	58.646	18.365	59.724	18.192	57.804	18.154		54.688	18.115		4.255
C2	25.597	62.964	18.886	60.273	14.013	60.798	14.005		58.162	14.025		0.316
C3	14.553	57.165	14.250	58.444	14.178	58.669	14.282		56.787	14.263		0.559
C4	23.967	63.842	23.306	62.526	23.155	65.81	22.738	26.679	70.137*			0.949
C5	17.811	60.306	17.620	59.130	17.617	59.188	17.509		---			---
C6	28.501	65.118	27.939	62.228	27.806	65.775	27.581		58.395*			0.735
C7	34.844	66.034	29.098	64.639	23.15	62.287	16.635		57.379	14.153		0.340
Sum coarse	67.46		52.51		41.15		33.95					7.15
Vial number	**Decant**	**Water added**	**Decant**	**Water added**	**Decant**	**Water added**	**Decant**		**Water added**	**Decant**	**F1 - F8 combined**	
F1	27.717	65.439	21.552	60.568	16.558	60.747	16.279		58.826	16.399		
F2	30.514	63.784	24.461	63.765	18.523	60.281	16.536		57.638	16.630		
F3	30.115	64.316	24.729	61.718	18.697	60.860	16.307		59.193	16.365		
F4	30.260	62.389	24.113	61.910	18.433	62.724	16.238		57.92	16.128		
F5	34.203	66.203	28.650	63.478	22.910	62.267	17.085		60.63	17.111		
F6	35.163	64.445	30.023	63.033	24.782	62.501	18.603		57.785	17.587		
F7	30.429	64.988	24.597	63.263	18.603	63.890	16.639		52.367	16.579		
F8	18.079	58.301	16.414	57.826	16.386	59.422	16.205		57.165	15.971	28.341	
Sum fines	125.80		83.86		44.21		23.21			22.09	14.40	14.40
Sum all	193.26		136.37		85.37		57.16					21.56

* … caustic added. Caustic addition was necessary due to mistake on 08/05. Amount of caustic in vials containing only gibbsite was by 60 wt% too low.

	2-Sep	8-Sep				
	Caustic added	**Decant**	**Water added**	**Decant**	**Water added**	**Decant**
C4+C5	70.137	27.370	64.414	15.169	56.584	14.884
C6	58.395	26.037	62.681	15.113	56.406	15.017

Table D-2. Scanning Electron Microscopy/Energy Dispersive Microscopy Automatic Feature Analysis Results for the Pre-Dissolution Fine-Grained Sample S09R000152 (2 sheets)

Run37 - sample: S09R000152

Particle Type	**Particles**	**Particles (rel. %)**	**Area (μm^2)**	**Area (rel. %)**	**Size* (μm)**	**Na**	**Al**	**Si**	**P**	**S**	**Cl**	**Ca**	**Ti**	**Cr**	**Mn**	**Fe**	**Ni**	**U**
Gibbsite	603	46.2	22168	58.4	6.8	5.7	**64.4**	11.1	5.6	3	4.4	0.8	0	0	0	0.2	0.3	4.4
Ni-Al-P-U-rich	371	28.4	7912	20.7	5.2	10.4	**17.1**	9.7	8.8	6.7	8.3	5.7	0.0	0.1	3.1	4.4	**13.4**	**12.1**
Fe-Rich	55	4.2	1548	4.1	6.0	6.3	9.2	7.7	7.6	7.9	8.4	5	0.1	0.4	3.5	**30.7**	5	8.1
Sub total		79		83														
Miscellaneous	217	16.6	5022	13.2	5.4	10	16.9	10.9	8.7	6.7	8.3	6.4	0.7	1	3.5	5.3	10.4	10.9
Cancrinite	25	1.9	557	1.5	5.3	**19.3**	**28.4**	**27.2**	6.5	4.3	4.7	2.3	0	0	0.1	1.1	0.7	5.4
P-rich	7	0.5	266	0.7	7.0	11.4	7.5	22.1	**21**	7	6.2	6.6	0.4	0.4	1.4	5.7	2.8	7
Apatite	10	0.8	236	0.6	5.5	7.7	10	7.7	**20**	8.1	6.1	**25.2**	0	0	0	2.9	4.4	8.1
U-rich	10	0.8	164	0.4	4.6	8.8	9	8.6	7.5	6.6	8.9	8.2	0.3	0	3.1	5.8	4.5	**28.9**
Ca-rich	4	0.3	66	0.2	4.6	6.5	9	7.1	5.3	5	6.2	**46.9**	0	0	0	3.1	3.8	7.2
Ni-rich	2	0.2	25	0.1	4.0	0	2.3	0	0	0	0	3.8	3.7	5.5	7.8	11.1	**54.5**	7.7
S-rich	1	0.1	9	0.0	3.4	16.9	8.5	9.2	6	**26.3**	10.6	4.6	0	0	2.8	3.3	3.8	8
Sub total	1305	100.0	37972	100.0														

Run 38 - sample: S09R000152

Particle Type	**Particles number**	**Particles (rel. %)**	**Area (μm^2)**	**Area (rel. %)**	**Size* (μm)**	Na	Al	Si	P	S	Cl	Ca	Ti	Cr	Mn	Fe	Ni	U
Gibbsite	798	57.0	81225	64.9	11.4	5.2	**67.4**	9.9	5.7	2.7	4.2	0.4	0	0	0.1	0.1	0.1	4.2
Ni-Al-U-P-rich	310	22.1	15471	12	8.0	9.9	**17.8**	9.5	**9.2**	6.6	8.0	6.1	0.0	0.1	2.7	4.3	14.5	11.6
Fe-Rich	74	5.3	10862	8.7	13.7	5.8	8.5	7.7	7.4	7.7	8.4	5	0.1	0.2	3.3	**33.3**	4.6	8
sum		84		86														
Miscellaneous	169	12.1	14627	11.7	10.5	9.4	**19.1**	13.5	9.4	6.2	7.3	6	0.1	0.3	2.1	4.2	**13.2**	**9.2**
Cancrinite	14	1.0	737	0.6	8.2	**19.5**	**30.2**	**28.2**	5.9	4.1	4.4	1.7	0	0	0	0.8	0.3	4.9
P-rich	7	0.5	716	0.6	11.4	10.5	9.5	17.6	**20.3**	7.6	6.3	9.1	0	0.5	0.6	5.9	4.5	7.6
Apatite	13	0.9	891	0.7	9.3	6.7	8.8	7.7	**20.1**	7.8	6.8	**28.4**	0	0	0	2.3	4	7.3
U-rich	9	0.6	336	0.3	6.9	7.6	8.3	8.2	6.8	5.8	8.3	8.4	0.3	0.3	3.2	6	4.7	**32.1**
Ca-rich	3	0.2	65	0.1	5.3	5.6	6.4	6.6	5.2	5	6.1	**52**	0	0	0	3	3.5	6.7
Ni-rich	2	0.1	122	0.1	8.8	2.8	7.9	2.6	4	2.2	2.8	6.8	2	2.9	5.7	8.9	**40.2**	9.6
S-rich	1	0.1	26	0.0	5.8	7.6	8.9	10.7	13.5	**17.4**	16.9	5.3	3.8	0	0	3.3	3.4	9.3
sum	1400		125079															

*average diameter assuming circular particles.
(highlighted in bold are the elements used to identify the individual types).

Table D-2. Scanning Electron Microscopy/Energy Dispersive Microscopy Automatic Feature Analysis Results for the Pre-Dissolution Fine-Grained Sample S09R000152 (2 sheets)

Combined results sample: S09R000152

Particle Type	Particles number	Particles (rel. %)	Area (μm^2)	Area (rel. %)	Size* (μm)	Na	Al	Si	P	S	Cl	Ca	Ti	Cr	Mn	Fe	Ni	U
Gibbsite	1401	51.8	103393	63.4	9.7	5.4	**66.1**	10.4	5.7	2.8	4.3	0.6	0.0	0.0	0.1	0.1	0.2	4.3
Ni-Al-U-P-rich	681	25.2	23383	14.3	6.6	10.2	**17.4**	9.6	9.0	6.7	8.1	5.9	0.0	0.1	2.9	4.3	**13.8**	**11.8**
Fe-Rich	129	4.8	12410	7.6	11.1	6.0	8.8	7.7	7.5	7.8	8.4	5.0	0.1	0.3	3.4	**32.2**	4.8	8.0
sum		82		85														
Miscellaneous	499	18.4	18832	11.5	6.9	10.0	17.5	10.1	8.9	6.6	8.1	6.1	0.3	0.5	3.0	4.7	12.7	11.3
Cancrinite	47	1.7	2097	1.3	7.5	**9.7**	**16.6**	**9.2**	10.8	6.8	7.7	8.6	0.0	0.0	2.3	4.2	13.0	11.2
P-rich	14	0.5	981	0.6	9.4	11.0	8.5	19.9	**20.7**	7.3	6.3	7.9	0.2	0.5	1.0	5.8	3.7	7.3
Apatite	23	0.9	1127	0.7	7.9	7.1	9.3	7.7	**20.1**	7.9	6.5	**27.0**	0.0	0.0	0.0	2.6	4.2	7.6
U-rich	19	0.7	500	0.3	5.8	8.2	8.7	8.4	7.2	6.2	8.6	8.3	0.3	0.1	3.1	5.9	4.6	**30.4**
Ca-rich	7	0.3	131	0.1	4.9	6.1	7.9	6.9	5.3	5.0	6.2	**49.1**	0.0	0.0	0.0	3.1	3.7	7.0
Ni-rich	4	0.1	148	0.1	6.9	1.4	5.1	1.3	2.0	1.1	1.4	5.3	2.9	4.2	6.8	10.0	**47.4**	8.7
S-rich	2	0.1	35	0.0	4.7	12.3	8.7	10.0	9.8	**21.9**	13.8	5.0	1.9	0.0	1.4	3.3	3.6	8.7
sum	2705		163052															

*average diameter assuming circular particles.
(highlighted in bold are the elements used to identify the individual types).

Required Characteristics of Particle Types:

Gibbsite	Al>40%
Nickel-Aluminum-Uranium-Phosphor-rich	Ni>10% & Al>10% & U>10% & P>10% & Ni+Al+U+P>45%
Iron-rich	Fe>20%
Miscellaneous	--
Cancrinite	Al>20% & Na>15% & Si >20% & Na+Al+Si>60%
Phosphor-rich	P>15%
Apatite	Ca+P>35%
Uranium-rich	U>20%
Calcium-rich	Ca>40%
Nickel-rich	Ni>25%
Sulphur-rich	S>15%

Electronically Approved by:

UserName: Callaway, William (h0086007)
Title: APD Chemist
Date: Wednesday, 09 June 2010, 11:57 AM Pacific Time
Meaning: Approved by the author or delegate
==

UserName: Callaway, William (h0086007)
Title: APD Chemist
Date: Wednesday, 09 June 2010, 11:59 AM Pacific Time
Meaning: Approved by the customer or delegate
==

UserName: Jewett, James (h0070487)
Title:
Date: Thursday, 10 June 2010, 03:41 PM Pacific Time
Meaning: Approved by Quality Assurance or delegate
==

UserName: Hardy, Don (h0085161)
Title: Laboratory Facilities Manager
Date: Monday, 14 June 2010, 09:08 AM Pacific Time
Meaning: Approved by the Facility Manager or delegate
==

UserName: Seidel, Cary (h0009079)
Title: APD Manager
Date: Monday, 14 June 2010, 10:00 AM Pacific Time
Meaning: Approved by the APD Manager or delegate
==