Boildown Study on Supernatant Liquid Retrieved from AP-107 in May 2010

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Date Published
January 2013

Prepared for the U.S. Department of Energy
Office of River Protection

Contract No. DE-AC27-08RV14800

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EXECUTIVE SUMMARY

A boildown study was completed on a composite prepared from supernatant liquid grab samples retrieved from tank 241-AP-107 in May of 2010. The composite was a clear, yellow liquid containing no visible solids at hot cell ambient temperatures (25-27 °C). The density of the test composite was 1.216 g/mL at 26.8 °C.

The boiling temperature curves generated at three reduced pressures—40-, 60-, and 80 Torr—displayed steadily increasing boiling temperatures with increasing volume reduction with no significant discontinuities. Only minimal foaming was observed after the volume reduction proceeded beyond 50 %WVR (percent waste volume reduction).

The bulk densities ($D_{\text{Bulk}}^{18\text{ }^{\circ}\text{C}}$) and quantities of settled and centrifuged solids present were measured on samples of the boildown concentrates that were kept at 18 °C for 7-8 days. Estimated values of the bulk densities of the concentrates at 60-Torr boiling temperatures ($D_{\text{Bulk}}^{60\text{ Torr}}$) were also calculated. The densities and solids content of concentrates at four levels of volume reduction were:

%WVR	27.7	46.0	54.0	57.5
$D_{\text{Bulk}}^{18\text{ }^{\circ}\text{C}}$ (measured)	1.294	1.387	1.447	1.484
$D_{\text{Bulk}}^{60\text{ Torr}}$ (estimated)	1.277	1.366	1.425	1.464
Vol% Settled Solids (18 °C)	4.5	10.4	15.5	76.2
Vol% Centrifuged Solids (18 °C)	2.0	5.0	6.9	36.6

Solids were observed in all boildown concentrates at process temperatures, at hot cell ambient temperatures (25-27 °C), and at 18 °C. The quantity of solids found in the cooled concentrates increased slowly through 50.2 %WVR. The quantity of solids found in concentrates after 54.0 %WVR was noticeably greater. Beyond 54.0 %WVR, the quantity of solids found in cooled concentrates increased dramatically.

Analysis of boildown test samples indicated that sodium oxalate and sodium carbonate solids form in cooled concentrates after volume reduction of 8.4 %WVR or less.

After 50 %WVR, the solids formed in cooled concentrates contained:

>90% of the oxalate and fluoride present in the initial tank 241-AP-107 feed composite as sodium oxalate, sodium fluoride phosphate, and sodium fluoride sulfate.

≈59% of the phosphate and ≈24% of the sulfate in the initial composite as sodium fluoride phosphate and sodium fluoride sulfate.

≈7% each of the nitrate, nitrite, and carbonate in the starting composite as sodium nitrate, sodium nitrite, and sodium carbonate salts.

The major contributors to the large increase in the quantity of solids found in concentrates after 54 %WVR were sodium nitrate and sodium carbonate.

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LIST OF TERMS**Abbreviations and Acronyms**

222-S	[WRPS] 222-S Laboratory
ATL	Advanced Technologies and Laboratories International, Inc.
AP-107	tank 241-AP-107
C-Liq	centrifuged liquid
C-Sld(s)	centrifuged solid(s)
CON #	step or sample number in boildown volume reduction
CR	cumulative concentration ratio
CR °C	cumulative concentration ratio at indicated temperature
CR _{Model} °C	cumulative concentration ratio at indicated temperature calculated using additive volumes model
CR _{Step}	concentration ratio for a discrete step in a boildown test sequence
D	density
D _{Bulk} °C	bulk density (i.e. total density of a sample containing liquid and solids)
D _{Bulk} °C	bulk density at indicated temperature
D _{Bulk} 60 Torr	estimated bulk density at boiling temperature at 60 Torr
D _{Liq} °C	liquid density at indicated temperature
D _{Model} °C	density at indicated temperature calculated using additive volumes model
DIL #	step or sample number in dilution of end-point slurry
IC	ion chromatography
ICP-AES	inductively coupled plasma-atomic emission spectrometry
Liq	liquid(s)
MDL	minimum detection limit
MDW	minimum detectable weight
	National Institute of Standards and Technology
%-Rec	percent recovery
%WVR	percent waste volume reduction
%WVR °C	percent waste volume reduction at indicated temperature
%WVR _{Model} °C	percent waste volume reduction at indicated temperature calculated using additive volumes model
P _{Read}	pressure as read from transducer display
P _{Corrected} or P _{Corr}	corrected system pressure
P/T	combination measurement of boiling temperature at specified pressure
PLM	polarized light microscopy
Pt-RTD	platinum resistance temperature detector
RPD	relative percent difference
SEM/EDX	scanning electron microscopy with energy dispersive X-ray spectroscopy
Slds	solids
S-Sld(s)	settled solid(s)
SPC	solid phase characterization
SpG	specific gravity
T	temperature
T _{Read}	temperature as read from thermocouple display
T _{Corrected} or T _{Corr}	corrected system temperature

TIC	total inorganic carbon
TOC	total organic carbon
Δ TOC	reported TOC minus the sum of the carbon from reported concentrations of organic acid anions
Total C	total carbon
TSAP	Tank Sampling Analysis Plan
Vol	volume
Vol _{Corrected}	corrected volume of sample in centrifuge cone
Vol _{Read}	volume read from centrifuge cone volume scale
vol%	percent by volume
f_{Liq}	volume fraction of liquid in a sample
f_{Slds}	volume fraction of solids in a sample
Wt	weight
wt%	percent by weight
XRD	[powder] X-ray diffraction (or diffractometry)

Units

°C	degree(s) Celcius
cm	centimeter(s)
g	acceleration due to gravity (used to define relative centrifugal force)
g	grams
g/L	grams per liter
g/mL	grams per milliliter
kV	kilovolts
hr(s)	hour(s)
L	liter(s)
μm	micrometer
μg/mL	micrograms per milliliter
mL	milliliter
(ΔmL/mL)/°C	change in volume (in milliliters) per milliliter of solution per change in degrees Celcius (units of thermal expansion coefficient)
mm	millimeter
Torr	unit of pressure equivalent to 1 mm Hg

1.0 INTRODUCTION

This report presents the results of a boildown study performed at the 222-S Laboratory (222-S) on supernatant liquid retrieved from tank 241-AP-107 (AP-107) in May of 2010. This study examined the behavior of the retrieved supernatant liquid during volume reduction under reduced pressures as requested in the tank sampling and analysis plan (TSAP) prepared to support operations at the 242-A Evaporator (RPP-PLAN-40837, *Tank 241-AP-107 Grab Sampling and Analysis Plan in Support of Evaporator Campaign for Fiscal Year 2010*). The boildown study was performed in hot cells 11A-5 and 11A-6 at 222-S. The study was conducted in accordance with the customer-approved test plan (LAB-PLN-10-00007, *Test Plan for Boildown Study on Supernate Retrieved from 241-AP-107 in May 2010*). Test operations altering the volume of the AP-107 liquid waste sample were performed as described in Laboratory Technology Procedure ATS-LT-519-183, "222-S Laboratory Determination of Properties of Radiological Solutions as a Function of Volume Reduction at Reduced Pressure."

The boildown study was a vacuum distillation of a composite of the AP-107 supernatant liquid grab samples at client-approved pressures of 40, 60, and 80 Torr (mm Hg). It was designed (1) to determine the relationship between solids formation and percent waste volume reduction (%WVR), (2) to determine the relationship between concentrate density and %WVR, (3) to determine the type and quantity of solids that may form in the AP-107 concentrates after cooling to ambient temperatures in storage tanks, and (4) to define the relationship between boiling temperature and concentration of the pot liquor/slurry. To obtain this information, the boildown study progressed through four stages:

Composite preparation	A composite of the AP-107 supernatant liquid waste was prepared from grab samples retrieved in May of 2010.
Boildown concentration	Volume reduction of the AP-107 composite was performed at reduced pressures. Samples of pot liquor/slurry were withdrawn at sampling points defined by calculated densities of the concentrate.
End-point slurry dilution	The end-point slurry from the concentration sequence was rediluted, in increments, reconstituting the concentrates from early stages of the volume reduction.
Sample processing	Samples collected during the concentration and dilution sequences were processed and all physicochemical analyses completed.

2.0 PRELIMINARY OPERATIONS

2.1 PREPARATION OF TEST COMPOSITE

A composite of the supernatant liquid grab samples retrieved from AP-107 in May 2010 was prepared on September 22, 2011 as shown in Table 2-1. The composite was specified volumetrically and prepared gravimetrically. Density values reported for each grab sample by 222-S (RPP-RPT-47358, *Final Report for Tank 241-AP-107 Liquid Grab Samples in Support of the Evaporator Program*) were used to convert target volume additions to target weights. No appreciable solids were observed in any of the grab samples used to prepare the composite nor were any physical changes observed as the composite was prepared. The final composite was a yellow liquid that was very slightly cloudy but contained no visible solids. No changes were observed during the 11 days at hot cell ambient temperatures that preceded the initiation of the boildown concentration sequence on October 3, 2011.

Table 2-1. Preparation of Composite for Boildown Study.

Sample Number		Location	SpG ^a	Added to Composite		
Grab Sample	OmniLIMS			g	mL ^b	Vol%
7AP-10-01	S10T002464	Surface	1.211	—	—	—
7AP-10-02A	S10T002472	≈1/6 of distance surface to solids	1.209	121.145	100.203	33.27
7AP-10-02B	S10T002478			80.294	66.414	
7AP-10-03A	S10T002473	≈1/2 of distance surface to solids	1.213	188.442	155.352	33.34
7AP-10-03B	S10T002479			14.090	11.616	
7AP-10-04A	S10T002474	≈5/6 of distance surface to solids	1.208	89.987	74.493	33.38
7AP-10-04B	S10T002480			111.960	92.682	
Composite	S11T011047	—	—	605.918	500.760	99.99

OmniLIMS – 222-S Laboratory Information Management System

Vol% – percent by volume

SpG – specific gravity

^a Values of specific gravity (SpG) measured and reported by the 222-S Laboratory.

^b Calculated volumes using weights added and reported SpG values.

The density of the test composite was measured in the 11A-6 hot cell as it was transferred to the glass pot in the boildown apparatus. The density measurement data is presented in Table 2-2. Three 100-mL Class A volumetric flasks were filled with the composite liquid and weighed. The temperature of the composite liquid was 26.8 °C. The average density of the AP-107 composite was 1.216 ± 0.001 g/mL.

Table 2-2. Density of Composite Measured in Hot Cell.

	Flask A	Flask B	Flask C
Reagent H₂O (not deaerated)			
Nominal flask volume (mL)	100 ± 0.08		
Wt reagent H ₂ O to fill flask (g)	99.501	99.465	99.514
Temperature of H ₂ O (°C)	27.1		
Reference H ₂ O density (g/mL)	0.996485		
Corrected flask volume (mL)	99.852	99.816	99.865
Composite			
Wt composite to fill flask (g)	121.529	121.329	121.371
Calculated composite density (g/mL)	1.2171	1.2155	1.2154
Temperature of composite (°C)	26.8		
Average composite density (g/mL)	1.216 ± 0.001 g/mL		

On September 28, 2011 a sample of the AP-107 composite was collected for physicochemical analyses. Approximately 12 mL of the sample (sample CON 0B) was transferred to a 15-mL centrifuge cone; the remaining liquid (sample CON 0A) was transferred to a sample vial and assigned sample number S11T011048. The cone was suspended in a water bath maintained at 18 °C for 26 days. No physical changes (specifically, no solids formation) were observed. The liquid in the cone was then transferred to a second sample vial and assigned sample number S11T011049. Both vials were loaded out of the 11A hot cells and submitted to 222-S for physicochemical analysis at the conclusion of the boildown study.

Specific gravity (SpG) was one of the analyses requested for the composite sample. The value reported by 222-S is actually a density. The average of duplicate 222-S measurements was 1.243 ± 0.018 g/mL. The temperature of the composite when the density was measured was not reported.

2.2 BOILDOWN APPARATUS

2.2.1 Temperature Sensor

A K-type thermocouple was used to measure the temperature of the test liquid/slurry in the boildown apparatus. The thermocouple signal was converted to a temperature readout (T_{Read}) using a Fisher¹ Model M107568 thermocouple reader. The T_{Read} values were changed to corrected temperatures ($T_{\text{Corrected}}$) using a calibration equation comparing the T_{Read} with ‘true’ temperatures measured with a calibrated platinum resistance temperature detector (Pt-RTD) (ebro Electronic,² Model TFX410-1/TPX200, Serial Number 15061117/15059510). Comparative temperature readings were made as a charge of initially cold reagent water in the

¹ Thermo Fisher Scientific Inc., Waltham, Massachusetts.

² ebro Electronic GmbH & Co. KG, Ingolstadt.

boildown pot was slowly heated to boiling and then allowed to cool while being stirred gently. The resulting temperature calibration equation was:

$$T_{\text{Corrected}}\text{ }^{\circ}\text{C} = (1.69 \times 10^{-6})(T_{\text{Read}})^3 - (5.59 \times 10^{-4})(T_{\text{Read}})^2 + (1.03)(T_{\text{Read}}) - 0.197 \quad (2-1)$$

2.2.2 Pressure Sensor/Controller

The pressure within the boildown apparatus was read from a Cole-Parmer[®] Instrument Company,³ Model 68801-23 pressure reader/controller. A calibration equation was generated to change pressure readings (P_{Read}) from the reader/controller to corrected pressures ($P_{\text{Corrected}}$). First, sets of $T_{\text{Corrected}}$ $^{\circ}\text{C}$ and P_{Read} values were recorded as reagent water was boiled at pressures ranging from 28 to 119 Torr. The P_{Read} values were then compared with reference values for the vapor pressure of water at $T_{\text{Corrected}}$. The resulting pressure calibration equation was:

$$P_{\text{Corrected}}\text{ Torr} = (0.9823)(P_{\text{Read}}) + 1.720 \quad (2-2)$$

2.2.3 Boildown Pot

The glass boildown pot used in this study has a maximum working volume of 500 mL and a minimum working volume of ≈ 75 mL. The main portion of the pot is a flat-bottomed cylindrical vessel with an inner diameter of ≈ 7.9 cm and a height of ≈ 9.5 cm. Three vertical, fin-like baffles protruding ≈ 6 mm into the interior of the pot facilitate mixing of the pot contents when stirred by a magnetic stirbar on the bottom of the pot. The upper portion of the pot is tapered (over ≈ 2.5 cm) to a cylindrical throat with an inner diameter of 5 cm. The top surface of the upper throat contains a groove for the O-ring joint by which the pot is connected to the rest of the boildown apparatus. Heating of the pot contents is accomplished by bringing a laboratory hotplate/magnetic stirrer into contact with the bottom of the pot.

2.2.4 Centrifuge Cones

All but one of the AP-107 boildown samples submitted for analysis were initially collected in 15-mL, plastic centrifuge cones. The volume scales on the cones were used to estimate the quantities of solids present in the samples and to calculate the bulk densities (D_{Bulk}) of the test samples. These cones are graduated in 0.5-mL increments from 1 to 14.5 mL and in 0.1-mL increments from 0 to 1 mL. The accuracy of the volume scales was checked by measuring the weight of reagent water required to fill the cones to the 10.0-, 11.5-, and 13.0-mL marks. The reference densities of water at the measurement temperatures were then used to calculate the actual volumes of water added. Triplicate measurements were performed for each cone (additional measurements were completed for Cones 1 and 8). A linear function was derived for each cone relating the volume read from the cone gradations (Vol_{Read}) to the ‘actual’ volume ($\text{Vol}_{\text{Corrected}}$) contained. The gravimetric data, reference densities, calculations, and derived correction functions for each cone/boildown sample are presented in Attachment A.

³ Cole-Parmer[®] is a registered trademark of Cole-Parmer Instrument Company, Vernon Hills, Illinois.

3.0 TEST OPERATIONS AND RESULTS

3.1 HOT CELL TEST OPERATIONS

The basic operations performed and observations made during the boildown concentration, dilution, and sample processing stages are summarized in this section. Transcripts of the hot cell data logs that were used to control the boildown operations and perform the first data processing calculations are presented in Attachment B. Photographs of the samples withdrawn from the pot during the boildown are presented in Attachment C.

3.1.1 Boildown Progress Parameters

Three parameters are used to monitor the progress of the boildown operations and in post-boildown data processing: concentration ratio (CR), %WVR, and D_{Bulk} . The CR and %WVR are defined as

$$CR = Vol_{Start}/Vol_{End} \quad (3-1)$$

$$\%WVR = [(Vol_{Start} - Vol_{End})/Vol_{Start}] \times 100 \quad (3-2)$$

The CR is the factor to which nonvolatile, infinitely soluble species become concentrated over their initial concentrations as the solution volume is reduced ($CR > 1$) or increased ($0 < CR < 1$).

Accurate measurement of the concentrate volume during a boildown run is very difficult. Even if this were not so, when multiple samples are withdrawn from the pot the concentrate volume during a boildown is no longer the appropriate value of Vol_{End} for calculation of CR or %WVR.

In practice, a boildown test is planned and controlled using an additive volumes model based on the following assumptions: (1) all solutes are nonvolatile (i.e., only water is removed from the test system), (2) the quantities of all additions to or removals from the system are accurately measured, (3) the volumes of all system components are measured at a common temperature, and (4) the volumes of system components are additive (i.e., $Vol_{Start} = Vol_{End} \pm Vol_{H_2O}$). Using this model, boildown progress is calculated stepwise. For each Step N

$$Vol_{Start \text{ Step } N} = Vol_{End \text{ Step } N-1} - Vol_{Sample \text{ N}}, \text{ and} \quad (3-3)$$

$$CR_{Step \text{ N}} = Vol_{Start \text{ Step } N} / (Vol_{Start \text{ Step } N} \pm Vol_{H_2O}) \quad (3-4)$$

The cumulative CR based on the additive volumes model, CR_{Model} , is

$$CR_{Model} = CR_{Step \text{ 1}} \times CR_{Step \text{ 2}} \times CR_{Step \text{ 3}} \times \dots \times CR_{Step \text{ N}} \quad (3-5)$$

and

$$\%WVR_{Model} = [(CR_{Model} - 1) / CR_{Model}] \times 100 \quad (3-6)$$

In the additive volumes model, the bulk density of the concentrate, D_{Model} , is a linear function of CR :

$$D_{Model} = [(D_{AP-107 \text{ Composite}} - D_{H_2O}) \times CR_{Model}] + D_{H_2O} \quad (3-7)$$

The density of the AP-107 test composite was measured in the 11A-6 hot cell (Table 2-2) and found to be 1.216 g/mL at 26.8 °C. This value was used to convert the starting weight of the test composite to a volume. The weights of water removed/added during the boildown were converted to volumes by dividing by the reference density of water at 26.8 °C. The weights of samples removed from the pot were converted to volumes by dividing by the $D_{\text{Model}}^{26.8\text{ }^{\circ}\text{C}}$ values calculated in the spreadsheet log. The resulting values of $CR_{\text{Model}}^{26.8\text{ }^{\circ}\text{C}}$ and $\%WVR_{\text{Model}}^{26.8\text{ }^{\circ}\text{C}}$ are shown in the boildown log in Table B-1.

3.1.2 Boildown Test Log

The following is a chronological log of the AP-107 boildown study, annotated with key observations and descriptions. Where pressures are cited only as integral values, they refer to the nominal target reflux pressures of 40, 60, or 80 Torr, not actual P_{Read} or $P_{\text{Corrected}}$ values.

The boildown concentration and dilution steps were performed in the 222-S 11A-6 hot cell; sample processing was performed in the adjacent 11A-5 hot cell. Typical ambient temperatures in the hot cells were $\approx 27\text{ }^{\circ}\text{C}$.

All test samples (with the exception of Composite Sample 0A) were collected in disposable plastic 15-mL centrifuge cones (Section 2.2.4) and processed in these cones through the final separation into centrifuged liquid (C-Liq) and centrifuged solids (C-Slds) fractions.

Boildown Composite Preparation—September 22, 2011

13:00 The AP-107 boildown test composite was prepared using 6 of the 7 grab samples received at the 222-S Laboratory (Section 2.1). No appreciable solids were observed in any of the grab samples, and the final composite was a yellow liquid that had a very slight cloudiness but contained no visible solids.

A sample was withdrawn from the composite for analysis. Sample CON 0A was transferred to a labeled sample vial and set aside. Sample CON 0B was transferred to a 15-mL centrifuge cone and set aside.

Boildown Volume Reduction Sequence—October 3, 2011

08:30 A 578.170-g portion of AP-107 composite was transferred to the boildown pot. The density of the composite was measured during the transfer (Section 2.1) and determined to be 1.216 g/mL at 26.8 °C. The calculated initial volume of the composite test sample was 475.473 mL.

09:25 Boiling pressure/temperature (P/T) measurements on the unconcentrated composite were completed at 40, 60, and 80 Torr. Boiling was gentle and well behaved; no foaming was observed. The test composite was a clear yellow liquid with no solids.

10:00 Concentration at 40 Torr was begun (CON 1a). On completion at 10:54, 45.3 mL of water was removed: $9.54\%WVR_{\text{Model}}^{26.8\text{ }^{\circ}\text{C}}$. At 11:13, P/T measurements at 40, 60, and 80 Torr were completed. Boiling was gentle and well behaved with no foaming. Composite was a clear, medium yellow liquid with no visible solids.

- 11:17 Concentration at 40 Torr was begun (CON 1b). On completion at 12:00, 46.2 mL of water was removed: $19.26 \%WVR_{Model}^{26.8^{\circ}C}$. At 12:18, P/T measurements at 40, 60, and 80 Torr were completed. Boiling was gentle and well behaved with no foaming. Composite was a clear, medium yellow liquid with no visible solids.
- 12:22 Concentration at 40 Torr was begun (CON-1c). On completion at 13:02, 44.0 mL of water was removed: $28.52 \%WVR_{Model}^{26.8^{\circ}C}$. At 13:21, P/T measurements at 40, 60, and 80 Torr were completed. Boiling was gentle and well behaved with no foaming. Composite was a clear, medium-yellow liquid with no visible solids (Figure 3-1a).
- 13:40 Sample CON 1 was drawn from the pot and transferred to centrifuge Cone 2. A photograph of the sample was taken after the pot was reconnected to the still head.
- 13:50 Boildown was halted overnight. The heat to the boildown pot was turned off and the pot was vented to hot cell ambient pressure. Magnetic stirrer was left on.

Boildown Volume Reduction Sequence—October 4, 2011

- 06:30 Overnight at hot cell ambient pressure and temperature (25.6 °C), the test composite became cloudy and was completely opaque.
- 07:30 Composite reflux established at 40 Torr (38.0 °C). Boiling was well behaved with no foaming and no visible change in solids loading.
- 07:45 Composite reflux established at 60 Torr (46.0 °C). Boiling was well behaved with no foaming and no visible change in solids loading.
- 07:55 Composite reflux established at 80 Torr (52.0 °C). Boiling was well behaved with no foaming and no visible change in solids loading.
- 07:58 Concentration at 80 Torr was begun (CON 2). On completion at 08:38, 42.7 mL of water was removed: $37.82 \%WVR_{Model}^{26.8^{\circ}C}$. At 08:48, P/T measurements at 40, 60, and 80 Torr were completed. Boiling was gentle and well behaved with no foaming and no visible change in appearance of the solids (Figure 3-1b).
- 09:10 Sample CON 2 was drawn from the pot and transferred to centrifuge Cone 3. A photograph of the sample was taken after the pot was reconnected to the still head.
- 09:30 Concentration at 40 Torr was begun (CON 3). On completion at 09:50, 21.5 mL of water was removed: $42.73 \%WVR_{Model}^{26.8^{\circ}C}$. At 10:07, P/T measurements at 40, 60, and 80 Torr were completed. Boiling was gentle and well behaved with no appreciable foaming, and no visible change in appearance of the solids.
- 10:30 Sample CON 3 was drawn from the pot and transferred to centrifuge Cone 4. A photograph of the sample was taken after the pot was reconnected to the still head.
- 10:45 Concentration at 80 Torr was begun (CON 4). On completion at 11:12, 18.8 mL of water was removed: $47.22 \%WVR_{Model}^{26.8^{\circ}C}$. At 11:28, P/T measurements at 40, 60, and 80 Torr were completed. Boiling was gentle and well behaved with no foaming and no appreciable change in appearance of the solids.

Figure 3-1. Appearance of the Composite During Boildown Test.

- 11:36 Sample CON 4 was drawn from the pot and transferred to centrifuge Cone 5. A photograph of the sample was taken after the pot was reconnected to the still head.
- 11:50 Concentration at 40 Torr was begun (CON 5). On completion at 12:12, 16.7 mL of water was removed: 51.45 %WVR_{Model}^{26.8 °C}. At 12:23, P/T measurements at 40, 60, and 80 Torr were completed. Boiling was well behaved with minor foaming (a single layer of ≈1 cm bubbles) and no appreciable change in appearance of the solids.
- 12:35 Sample CON 5 was drawn from the pot and transferred to centrifuge Cone 6. A photograph of the sample was taken after the pot was reconnected to the still head.
- 13:00 Concentration at 80 Torr was begun (CON 6). On completion at 13:35, 15.1 mL of water was removed: 55.53 %WVR_{Model}^{26.8 °C}. At 13:44, P/T measurements at 40, 60, and 80 Torr were completed. Boiling was bumpy with moderate foaming (a single layer of ≈1 cm bubbles). The loading of fine, white solids in the slurry was noticeably greater.
- 14:02 Sample CON 6 was drawn from the pot and transferred to centrifuge Cone 7. A photograph of the sample was taken after the pot was reconnected to the still head.
- 14:18 Concentration at 40 Torr was begun (CON 7). On completion at 14:36, 11.6 mL of water was removed: 58.92 %WVR_{Model}^{26.8 °C}. At 14:47, P/T measurements at 40, 60, and 80 Torr were completed. This was the end-point of the boildown concentration. The test composite was a viscous slurry of fine, white solids in a yellow liquid (Figure 3-1c).
- 15:00 Sample CON 7 was drawn from the pot and transferred to centrifuge Cone 8. A photograph of the sample was taken after the pot was reconnected to the still head.

End-point Slurry Dilution Sequence—October 4, 2011

- 15:34 Dilution Step DIL 1 was completed by adding 120.5 mL of water back to the pot: 20.31 %WVR_{Model}^{26.8 °C}. Reflux of the test slurry at 60 Torr was established, and a P/T measurement was completed at 16:00. Boiling was well behaved with no foaming. The concentration of solids was visibly reduced but the test slurry remained opaque (high contrast markings on the opposite wall of the pot were not visible).
- 16:12 Sample DIL 1 was drawn from the pot and transferred to centrifuge Cone 9. A photograph of the sample was taken after the pot was reconnected to the still head.
- 16:40 Dilution Step DIL 2 was completed by adding 16.3 mL of water back to the pot: 14.81 %WVR_{Model}^{26.8 °C}. Reflux of the test slurry at 60 Torr was established, and a P/T measurement was completed at 16:59. Boiling was well behaved with no foaming. The concentration of solids was visibly reduced but the test slurry remained opaque.
- 17:07 Sample DIL 2 was drawn from the pot and transferred to centrifuge Cone 10. A photograph of the sample was taken after the pot was reconnected to the still head.
- 17:37 Dilution Step DIL 3 was completed by adding 18.2 mL of water back to the pot: 8.33 %WVR_{Model}^{26.8 °C}. Reflux of the test slurry at 60 Torr was established and a P/T measurement was completed at 17:54. Boiling was well behaved with no foaming. The test slurry was no longer completely opaque but was still very cloudy (Figure 3-1d).
- 18:05 Sample DIL 3 was drawn from the pot and transferred to centrifuge Cone 11. A photograph of the sample was taken after the pot was reconnected to the still head.

18:12 Heat to the boiling pot was turned off and the pot was vented to ambient hot cell pressure. Stirring of the pot contents was continued overnight.

Boildown Completion—October 5, 2011

07:00 Appearance of the test slurry in the boildown pot did not change significantly after cooling to ambient temperature overnight. The weight of the remaining slurry was 293.97 g. The final slurry was transferred to a jar for temporary storage.

3.2 HOT CELL SAMPLE PROCESSING

3.2.1 Initial Sample Processing

October 5, 2011: By mid-morning, composite Sample CON 0B had sat at hot cell composite temperatures for 168 hrs, Sample CON 1 for 45 hrs, and Samples CON 2-7 and DIL 1-3 for 18-26 hrs. All 11 cones were weighed and photographed (Attachment C). The total sample volume and the estimated volume of any settled solids (S-Slds) were recorded for each sample.

All 11 sample cones and the jar containing the final slurry recovered from the boildown pot were transferred to hot cell 11A-5 and suspended in a water bath cooled to 18 °C.

October 11, 2011: Starting at 12:40, each sample cone was removed from the water bath, photographed, and then returned to the 18 °C water bath.

October 12, 2011: Starting at 07:00, each boildown sample was removed from the 18 °C water bath and photographed. The total sample volume, S-Slds volume, and visual appearance of the S-Slds were recorded for each sample. The samples were then returned to the water bath.

The appearance and settling behavior observed in boildown Samples CON 1-10 are summarized in Table 3-1. Descriptions of the samples shortly after removal from the pot, after at least 18 hrs at hot cell ambient temperature, and after ≈ 7 days at 18 °C are included. Photographs of the sample cones at approximately the same times are presented in Attachment C.

3.2.2 Final Sample Processing

October 12, 2011: Starting at 13:45, the boildown samples were removed from the 18 °C water bath, in pairs, and centrifuged for 5-6 minutes at $\approx 1000 \times g$. The samples were then returned to the water bath.

No solids were observed in Sample CON 0B, the subsample of the initial composite liquid. The sample liquid was transferred to a labeled sample vial and set aside for laboratory analysis.

Table 3-1. Appearance of Settled Solids in Boildown Samples. (2 sheets)

Sample	Appearance of Settled Solids
CON 1	<p><1 hr after sampling: The pot sample was a clear, yellow liquid with no solids visible.</p> <p>45 hrs at 26.2-25.1 °C: Very fine-grained, white/colorless solids formed as the sample cooled to hot cell ambient temperature. After 45 hrs, the supernatant was clear and S-Slds filled the ≈ 0.4 mL bottom of the cone.</p> <p>7 days at 18 °C: ≈ 0.6 mL of fine grained white/colorless S-Slds filled the bottom of the cone. Particles in the S-Slds were too small to observe any morphology.</p>
CON 2	<p><1 hr after sampling: The pot sample contained white/colorless solids in very small flocs dispersed throughout the sample volume. The solids settled fairly quickly.</p> <p>26 hrs at 25.9-25.1 °C: The supernatant and cone walls were clear. Very fine-grained, white/colorless solids filled the bottom ≈ 1.5 mL of the cone. Particles in the S-Slds were too small to observe any morphology.</p> <p>7 days at 18 °C: No significant change in the appearance of the S-Slds.</p>
CON 3	<p><1 hr after sampling: The pot sample was similar to Sample CON 2 in appearance and settling rate.</p> <p>25 hrs at 26.0-25.2 °C: The supernatant and cone walls were clear. Very fine-grained, white/colorless S-Slds filled the bottom ≈ 1.6 mL of the cone.</p> <p>7 days at 18 °C: No significant change in the appearance of the S-Slds. Particles in the S-Slds were too small to observe any morphology.</p>
CON 4	<p><1 hr after sampling: The pot sample was similar to Samples CON 2 and CON 3 in appearance and settling rate.</p> <p>24 hrs at 25.9-25.2 °C: The supernatant and cone walls were clear. Very fine-grained, white/colorless solids filled the bottom ≈ 1.4 mL of the cone.</p> <p>7 days at 18 °C: No significant change in the appearance of the S-Slds. Particles in the S-Slds were too small to observe any morphology.</p>
CON 5	<p><1 hr after sampling: The pot sample was very similar to Samples CON 2-4 in appearance. The solids settled very quickly.</p> <p>23 hrs at 25.9-25.2 °C: The supernatant and cone walls were clear. Very fine-grained, white/colorless solids filled the bottom ≈ 1.7 mL of the cone.</p> <p>7 days at 18 °C: No significant change in the appearance of the S-Slds. Particles in the S-Slds were too small to observe any morphology.</p>
CON 6	<p><1 hr after sampling: The pot sample was similar to Samples CON 2-5 in appearance and settling rate.</p> <p>21 hrs at 26.2-25.2 °C: The supernatant and cone walls were clear. Very fine-grained, white/colorless solids filled the bottom ≈ 2.0 mL of the cone.</p> <p>7 days at 18 °C: No significant change in the appearance of the S-Slds. Particles in the S-Slds were too small to observe any morphology.</p>
CON 7	<p><1 hr after sampling: This was the end-point slurry sample. The slurry had a high concentration of very fine-grained, white/colorless, crystalline solids in a yellow liquid.</p> <p>21 hrs at 26.0-25.2 °C: Settling of the solids was severely hindered, and after 21 hrs the S-Slds still occupied $\approx 89\%$ of the sample volume.</p> <p>7 days at 18 °C: The S-Slds compacted slowly to a mass occupying $\approx 76\%$ of the sample volume. There was no significant change in appearance of the S-Slds. Particles in the S-Slds were too small to observe any morphology.</p>

Table 3-1. Appearance of Settled Solids in Boildown Samples. (2 sheets)

Sample	Appearance of Settled Solids
DIL 1	<p><1 hr after sampling: The pot sample contained white/colorless solids in a mixture of fine-grained particulates and small flocs. The particulates settled out within a few minutes leaving a very disperse suspension of the small flocs in suspension.</p> <p>19 hrs at 26.2-25.2 °C: The supernatant and cone walls were clear. Fine-grained white/colorless solids filled ≈ 0.4 mL of the bottom of the cone.</p> <p>7 days at 18 °C: There was no significant change in the appearance of the S-Slds. Particles in the S-Slds were too small to observe any morphology.</p>
DIL 2	<p><1 hr after sampling: The pot sample was similar to Sample DIL 1 in appearance and settling rate.</p> <p>19 hrs at 26.1-25.2 °C: The supernatant and cone walls were clear. Fine-grained, white/colorless S-Slds filled the bottom ≈ 0.3 mL of the cone.</p> <p>7 days at 18 °C: There was no significant change in the appearance of the S-Slds. Particles in the S-Slds were too small to observe any morphology.</p>
DIL 3	<p><1 hr after sampling: The pot sample was similar to Samples DIL 1 and DIL 2 in appearance and settling rate.</p> <p>18 hrs at 26.0-25.3 °C: The supernatant and cone walls were clear. Fine-grained, white/colorless S-Slds filled the bottom ≈ 0.2 mL of the cone.</p> <p>7 days at 18 °C: No significant change in the appearance of the S-Slds. Particles in the S-Slds were too small to observe any morphology.</p>

October 13, 2011: Starting at 06:50, boildown Samples CON 1-7 and DIL 1-3 were removed from the 18 °C water bath, in pairs, centrifuged for 5-6 minutes at $\approx 1000 \times g$, and then returned to the bath. After all 10 samples had been through the second centrifuge cycle, they were photographed.

The following processing sequence was then followed for each sample. A sample cone was removed from the 18 °C water bath and centrifuged for a third period of 5-6 minutes at $\approx 1000 \times g$. The apparent volumes of the total sample and of the C-Slds were recorded. The C-Liq was then decanted into a labeled sample vial. The wet C-Slds were then centrifuged for a final period of 5-6 minutes and any additional C-Liq was decanted to the sample vial. The final C-Liq and C-Slds weights were measured and recorded. The sample vials containing the C-Liq samples were set aside. The centrifuge cones and damp C-Slds were returned to the 18 °C water bath.

October 17, 2011: Starting at 08:15, samples of the C-Slds from the boildown samples were collected for solid phase characterization (SPC).

Due to the small quantities of C-Slds present, the solids recovered from Samples CON 1-CON 3 were combined. A small quantity of C-Liq from Sample CON 3 was used to facilitate recovery and transfer of the C-Slds to a sample vial.

Due to the small quantities of C-Slds present, the solids recovered from Samples CON 4 and CON 5 were also combined. A small quantity of C-Liq from Sample CON 5 was used to facilitate recovery and transfer of the C-Slds to a sample vial.

All the C-Slds from Sample CON 6 were recovered using a small volume of Sample CON 6 C-Liq and transferred to a sample vial.

A portion (≈ 4.5 g) of the C-Slds from the end-point slurry, Sample CON 7, was transferred to a labeled sample vial.

The C-Slds from Sample DIL 3 were transferred to a sample vial. Most of the S-Slds that had settled to the bottom of the jar containing the final slurry set aside at the conclusion of the boildown test were also recovered and combined with the DIL 3 C-Slds.

The final identifications of the AP-107 boildown study samples are tabulated in Table 3-2.

Table 3-2. Identification of Boildown Samples.

Test Sample	222-S Sample Number S11T0-	
	Liquid	Solids
Test Composite		
CON 0	-11048	—
	-11049	
Volume Reduction		
CON 1	-11062	-14140
CON 2	-11063	
CON 3	-11064	
CON 4	-11065	-14141
CON 5	-11066	
CON 6	-11067	-14280
CON 7	-11070	-11074
Dilution of End-point Slurry		
DIL 1	-11071	—
DIL 2	-11072	—
DIL 3	-11073	-11075

October 18, 2011: At 11:00, the 12 samples of boildown liquids (including composite Sample CON 0A that was transferred directly to a sample vial) were loaded out of the 11A hot cells and submitted to 222-S for analysis. The 5 samples of boildown sample solids were loaded out and submitted for SPC measurements.

3.2.3 Densities of Boildown Sample Liquids

The densities of the boildown C-Liq samples were measured before the samples were loaded out of the hot cells and submitted to 222-S for analysis. The measurements were performed as follows: The temperature of the C-Liq sample was measured (the temperatures of the samples ranged from 25.3-25.5 °C). Using a positive-displacement pipette, a 1-mL portion of liquid was transferred to a sample vial and weighed. A second 1-mL sample portion was then added to the vial and its weight was determined. The average C-Liq density was calculated. The 2-mL portion of C-Liq was returned to the parent sample using a disposable pipette.

The density of demineralized water was measured several times before and after the C-Liq sample measurements. All the measured densities were within 1% of the reference values for pure water.

3.3 FINAL VOLUME AND WEIGHT DATA

The results of gravimetric and volumetric measurements on the boildown samples are summarized in Table 3-3.

The total, S-Slds and C-Slds volumes at hot cell ambient temperature (25.1-25.3 °C) and at 18 °C are based on examination of sample photographs. Apparent volumes (Vol_{Read}) were measured as accurately as possible from enlargements of sample photographs. The Vol_{Read} values were then converted to $Vol_{Corrected}$ total volumes using the cone calibration functions in Attachment A. The total volumes of the samples at 18 °C are believed to be the most accurately measured values.

The vol% S-Slds and vol% C-Slds in each sample are the apparent volumes of the solids divided by the corrected total volumes at 18 °C or hot cell ambient temperature with the results multiplied by 100.

The D_{Bulk} values are the weights of the bulk samples (i.e., combined liquid and solids) divided by the total sample volumes measured at the indicated temperature.

The D_{Liq} values in Table 3-3 tabulate the results of measurements of the densities of the C-Liq portions of the boildown samples. Each $D_{Liq}^{18\text{ }^{\circ}\text{C}}$ value is the weight of C-Liq decanted from a sample divided by the estimated difference between the total and C-Slds volumes while still in the 15-mL centrifuge cone at 18 °C.

Table 3-3. Gravimetric and Volumetric Data for Boildown Samples.

Test Sample	CON 0B	CON 1	CON 2	CON 3	CON 4	CON 5	CON 6	CON 7	DIL 1	DIL 2	DIL 3
%WVR ^{18 °C a}	0.00	27.74	36.88	41.58	46.05	50.15	54.04	57.47	19.90	14.66	8.37
CR ^{18 °C a}	1.000	1.384	1.584	1.712	1.854	2.006	2.176	2.352	1.248	1.172	1.091
Total Wt (g)	14.674	15.820	17.340	15.949	16.600	17.600	17.759	19.869	16.323	16.409	15.864
Wt Wet 18 °C C-Slds (g)	0.000	0.301	0.603	0.730	0.956	1.096	1.745	7.699	0.192	0.147	0.090
Wt 18 °C C-Liq (g)	14.674	15.519	16.737	15.219	15.644	16.504	16.014	12.170	16.131	16.262	15.774
Total Vol ^{Ambient} (mL)	11.98	12.26	13.01	11.79	11.99	12.42	12.26	13.41	12.85	13.12	12.80
Vol ^{Ambient} S-Slds (mL)	0.00	0.35	1.50	1.60	1.40	1.70	2.00	11.75	0.40	0.30	0.20
Total Vol ^{18 °C} (mL)	12.02	12.22	13.00	11.74	11.97	12.43	12.27	13.39	12.85	13.06	12.78
Vol ^{18 °C} S-Slds (mL)	0.00	0.55	1.40	1.45	1.25	1.50	1.90	10.20	0.40	0.35	0.20
Vol ^{18 °C} C-Slds (mL)	0.00	0.25	0.35	0.45	0.60	0.70	0.85	4.90	0.15	0.10	0.07
Vol ^{18 °C} C-Liq (mL)	12.02	11.97	12.64	11.30	11.37	11.73	11.42	8.49	12.70	12.96	12.71
Vol% S-Slds at Ambient T	0.0	2.9	11.5	13.6	11.7	13.7	16.3	87.60	3.1	2.3	1.6
Vol% S-Slds at 18 °C	0.0	4.5	10.8	12.3	10.4	12.1	15.5	76.20	3.1	2.7	1.6
Vol% C-Slds at 18 °C	0.0	2.0	2.7	3.8	5.0	5.6	6.9	36.6	1.2	0.8	0.5
Wt% Wet 18 °C C-Slds	0.00	1.90	3.48	4.58	5.76	6.227	9.826	38.749	1.18	0.90	0.57
D _{Bulk} ^{18 °C} (g/mL)	1.220	1.294	1.334	1.358	1.387	1.416	1.447	1.484	1.270	1.257	1.241
D _{Liq} ^{18 °C} (g/mL)	1.220	1.296	1.324	1.347	1.376	1.407	1.402	1.434	1.270	1.255	1.241
D _{Liq} ^{25.3 °C} (g/mL)	1.226	1.302	1.339	1.356	1.396	1.407	1.422	1.422	1.264	1.248	1.236
D _{Liq} ^{222-S} (g/mL)	1.243	1.319	1.386	1.313	1.332	1.317	1.381	1.342	1.292	1.299	1.259

T = temperature

^a Actual test values of boildown progress parameters as calculated in Section 5.1.2.

The $D_{\text{Liq}}^{25.3\text{ }^{\circ}\text{C}}$ values are the results of the density measurements performed using a 1-mL positive displacement pipette before the C-Liq samples were loaded out of the 222-S hot cells (Section 3.2.3).

The $D_{\text{Liq}}^{222\text{-S}}$ values are the densities of the C-Liq samples measured by 222-S Laboratory personnel. (The $D_{\text{Liq}}^{222\text{-S}}$ values were reported as specific gravities or SpG; the measurement temperature(s) are not known).

The three sets of liquid density data are presented graphically in Figure 3-2. In this figure and all other plots in this report, values for the initial test composite (CON 0) and the volume reduction sequence samples (CON 1 to CON 7) are displayed with solid data markers; values for the dilution sequence samples (DIL 1 to DIL 3) are displayed with hollow data markers of the same shape and border color.

The plots of the $D_{\text{Liq}}^{18\text{ }^{\circ}\text{C}}$ and $D_{\text{Liq}}^{25.3\text{ }^{\circ}\text{C}}$ values both display similar trends. The measured densities at 25.3 °C are greater than those at 18 °C. Since this relationship should be reversed, this indicates that there is some bias in one or both of these data sets. The scatter in the $D_{\text{Liq}}^{222\text{-S}}$ values is problematic.

Figure 3-2. Density of Sample Liquids versus Boildown Progress.

4.0 LABORATORY ANALYSIS OF BOILDOWN SAMPLES

The liquid samples retrieved during the AP-107 boildown or created during the subsequent sample processing were submitted to 222-S for chemical analysis. These analyses were performed by the 222-S analytical contractor, Advanced Technologies and Laboratories International, Inc. (ATL). A transcript of the analysis results is presented in Attachment D.

The analysis results are summarized in Table 4-1. Analysis results for all priority analytes are presented. Other (opportunistic) analytes for which none of the analysis results exceeded the reported detection limit have been omitted from the table.

A few anomalies are observed in the analytical data in Table 4-1.

The sum of the total organic carbon (TOC) and total inorganic carbon (TIC) analysis results is equal to the total carbon (Total C) result for the composite sample (Sample CON 0). However, TOC + TIC for the end-point liquid (Sample CON 7) is only $\approx 60\%$ of the Total C result; TOC + TIC for the final dilution liquid (Sample DIL 3) is $\approx 85\%$ of the Total C value. The cause(s) for this discrepancy is unknown.

The concentrations of four organic acid anions—formate, acetate, glycolate, and oxalate—in the sample liquids were analyzed by ion chromatography (IC). The concentrations of organic carbon represented by the reported concentrations of the anions were calculated and subtracted from the reported TOC values measured by acid sparging/persulfate oxidation/coulometry. The differences are tabulated as ΔTOC . For Samples CON 0, CON 1-7, and DIL 1-2, the reported TOC is 43-71% greater than the sum of the carbon from the organic acid anions. For Sample DIL 3, the sum of the carbon from the acid anions is 36% greater than the reported TOC. The cause(s) for this discrepancy is unknown.

The concentrations of phosphate (PO_4^{3-}) in the liquid samples measured with IC can be compared with concentrations of P measured with inductively coupled plasma-atomic emission spectrometry (ICP-AES) and then recalculated as PO_4 . The difference between the recalculated ICP-AES and IC values ranges from -11% to +24%. This degree of variation between the two data sets is probably due, in large part, to combined analytical errors. The trends of concentration versus boildown progress displayed by both the IC and ICP-AES data sets for phosphorus species are similar.

The concentrations of sulfate (SO_4^{2-}) in the liquid samples measured with IC can be compared with concentrations of S measured with ICP-AES and then recalculated as SO_4 . The calculated ICP-AES SO_4 values are 2-25% greater than the IC sulfate concentrations for 10 of the 11 samples; the calculated SO_4 concentration is 7% smaller than the IC sulfate value for Sample DIL 1. This degree of variation between the two data sets is also probably mostly due to combined analytical errors. The trends of concentration versus boildown progress displayed by both the IC and ICP-AES data sets for sulfur species are similar.

Table 4-1. Results of Analyses of Boildown Sample Liquids. (2 sheets)

(All analysis results in units of µg/mL)

Test Sample %WVR ^{18 °C}	CON 0	CON 1	CON 2	CON 3	CON 4	CON 5	CON 6	CON 7	DIL 1	DIL 2	DIL 3
	0.00	27.74	36.88	41.58	46.05	50.15	54.04	57.47	19.90	14.66	8.37
Total C	7,170	—	—	—	—	—	—	12,300	—	—	7,540
TOC	1,380	982	1,040	1,110	1,150	1,340	1,640	946	991	925	407
ΔTOC ^a	745	517	584	668	698	886	1,160	483	486	400	0
TIC	5,820	7,350	8,500	6,680	10,300	11,200	12,400	6,390	6,370	6,240	5,980
as CO ₃ ^b	29,077	36,721	42,467	33,374	51,460	55,957	61,952	31,925	31,825	31,176	29,877
OH ⁻	10,000	14,000	16,000	18,000	19,000	23,000	20,000	25,000	13,000	12,000	11,000
Formate	644	889	994	1,080	1,150	1,220	1,340	1,400	810	762	700
Acetate	194	289	296	351	373	393	446	462	265	235	221
Glycolate	172	253	273	312	331	354	397	417	228	205	194
Oxalate	1,610	699	577	406	365	300	251	128	931	1,070	1,240
Nitrite	30,300	41,500	47,200	50,000	53,200	57,200	63,100	64,000	38,000	35,600	33,100
Nitrate	118,000	162,000	185,000	197,000	211,000	224,000	216,000	196,000	148,000	141,000	129,000
Fluoride	469	533	488	337	376	73	31	25	585	555	513
Phosphate	2,900	2,910	2,180	2,120	1,250	2,430	3,070	2,070	3,630	3,450	3,190
Sulfate	9,080	12,500	14,000	14,200	15,700	14,300	15,200	13,600	11,300	10,700	9,890
Chloride	1,400	1,920	2,180	2,310	2,450	2,610	2,880	3,020	1,750	1,650	1,520
Bromide	< 58	< 58	< 58	< 58	< 58	< 58	< 58	< 58	< 58	< 58	< 58

Table 4-1. Results of Analyses of Boildown Sample Liquids. (2 sheets)

(All analysis results in units of µg/mL)

Test Sample	CON 0	CON 1	CON 2	CON 3	CON 4	CON 5	CON 6	CON 7	DIL 1	DIL 2	DIL 3
%WVR ^{18 °C}	0.00	27.74	36.88	41.58	46.05	50.15	54.04	57.47	19.90	14.66	8.37
Na	107,000	144,000	167,000	182,000	187,000	206,000	220,000	216,000	121,000	130,000	140,000
Al	4,420	6,290	7,250	8,100	8,370	9,140	10,200	11,600	4,990	5,350	5,760
Si	108	355	182	263	183	233	141	158	142	211	198
P	929	939	817	676	472	927	1,140	885	1,050	1,170	1,290
(as PO ₄) ^c	2,848	2,879	2,505	2,073	1,447	2,842	3,495	2,714	3,219	3,587	3,955
S	3,100	4,370	5,210	5,430	5,790	5,500	5,830	5,670	3,520	3,760	4,000
(as SO ₄) ^c	9,287	13,092	15,609	16,268	17,346	16,478	17,466	16,987	10,546	11,265	11,984
K	1,350	2,170	2,060	2,850	2,850	2,810	3,490	4,230	1,850	1,690	2,320
Ca	< 125	< 125	< 125	< 125	< 125	< 125	< 125	< 125	< 125	< 125	< 125
Cr	571	833	959	1,080	1,110	1,210	1,320	1,530	651	700	758
Co	< 25	< 25	< 25	< 25	< 25	< 25	< 25	< 25	< 25	< 25	26
Mo	< 50	62	56	64	54	71	66	87	< 50	< 50	< 50

^a ΔTOC µg/mL = µg/mL TOC – [(µg/mL Formate × 0.2668) + (µg/mL Acetate × 0.4068) + (µg/mL Glycolate × 0.3201) + (µg/mL Oxalate × 0.2729)]^b µg/mL CO₃ = µg/mL TIC × 4.9961^c P and S were analyzed by ICP-AES. Analysis results were converted to phosphate and sulfate as follows: PO₄ µg/mL = µg/mL P × 3.0662 and SO₄ µg/mL = µg/mL S × 2.9959.

5.0 DATA ANALYSIS

5.1 DENSITY

In this report, bulk density or D_{Bulk} , is the density of a total sample, i.e., the density of the combined liquid and solid fractions of a sample. Liquid densities or D_{Liq} are the measured densities of the C-Liq portion of the samples processed in 15-mL centrifuge cones. The density of the initial composite is reported as a D_{Bulk} when measured in triplicate in 100-mL volumetric flasks and as a D_{Liq} when measured on the subsample in a 15-mL cone.

5.1.1 Bulk Density of Boildown Samples

The $D_{\text{Bulk}}^{26.8\text{ }^{\circ}\text{C}}$ for the initial composite is believed to be the most accurately measured density in this study. The $D_{\text{Bulk}}^{18\text{ }^{\circ}\text{C}}$ values, tabulated in Table 3-3, are based on volume measurements initiated while the boildown samples, at 18 °C, were in the 15-mL centrifuge cones in which they were collected. These volume (and $D_{\text{Bulk}}^{18\text{ }^{\circ}\text{C}}$) measurements were made as carefully as possible but are probably slightly less accurate than those for the initial composite.

In Table B-2 (Attachment B), the additive volumes model calculations are reset to a base temperature of 18 °C. The volume of the initial composite sample was reduced to account for cooling. Previous measurements (ARCHO 1974, “Densities of Caustic Waste Solutions as a Function of Temperature”) showed that the thermal expansion coefficients of two synthetic tank waste concentrates were 4.409×10^{-4} and 4.838×10^{-4} ($\Delta\text{mL/mL}/^{\circ}\text{C}$) over a range of 22-56.5 °C. Another set of measurements (WRPS-0900372, “Results of Lifetime Testing of Anton Paar DMA 35N Portable Density Meter”) showed that thermal expansion coefficients of a double-shell tank waste simulant were 3.757×10^{-4} ($\Delta\text{mL/mL}/^{\circ}\text{C}$) from 18-30 °C and 6.843×10^{-4} ($\Delta\text{mL/mL}/^{\circ}\text{C}$) from 30-42 °C. An average value of 4.335×10^{-4} ($\Delta\text{mL/mL}/^{\circ}\text{C}$) was used to adjust the initial composite volume. The corresponding $D_{\text{Bulk}}^{18\text{ }^{\circ}\text{C}}$ was 1.221 g/mL.

The calculated $D_{\text{Model}}^{18\text{ }^{\circ}\text{C}}$ and measured $D_{\text{Bulk}}^{18\text{ }^{\circ}\text{C}}$ values are plotted against $CR_{\text{Model}}^{18\text{ }^{\circ}\text{C}}$ in Figure 5-1. The $D_{\text{Liq}}^{25.3\text{ }^{\circ}\text{C}}$ values have also been replotted for comparison. In this figure and all other plots in this report, values for the initial test composite (CON 0) and the volume reduction sequence samples (CON 1 to CON 7) are displayed with solid data markers; values for the dilution sequence samples (DIL 1 to DIL 3) are displayed with hollow data markers of the same shape and border color.

The plot of measured $D_{\text{Bulk}}^{18\text{ }^{\circ}\text{C}}$ vs. $CR_{\text{Model}}^{18\text{ }^{\circ}\text{C}}$ in Figure 5-1 clearly deviates from the linear projection of the additive volumes model. The concentrate volumes indicated by the $D_{\text{Bulk}}^{18\text{ }^{\circ}\text{C}}$ values are larger than those predicted by the additive volumes model. Small downward curvature in plots of density vs. CR is common for concentrated solutions of chemical species found in tank wastes. It is also quite likely that as the quantity of solid precipitates in the concentrates increases, the additive volumes assumption of the model becomes an increasingly poor one. The pronounced decrease in the slope of the $D_{\text{Liq}}^{25.3\text{ }^{\circ}\text{C}}$ plot beginning at Sample CON 5 suggests that this is a factor.

Figure 5-1. Comparison of Model and Measured Bulk Densities.

5.1.2 Test Values of CR and %WVR

The measured $D_{Bulk}^{18^{\circ}C}$ values can be used to calculate the virtual volumes of $18^{\circ}C$ pot concentrates at each step of the boildown test. These volumes can then be used to calculate boildown progress parameters, $CR^{18^{\circ}C}$ and $\%WVR^{18^{\circ}C}$, that are not based on the additive volumes assumption of the model parameters. These calculations are shown in Table 5-1. The calculation chain proceeds down a column of the table for each boildown step.

1. The weight and volume of concentrate at the beginning of the step are entered. These values are the weight and volume of concentrate at the end of the previous step.
2. The weight of the concentrate is adjusted by the weight of water removed/added.
3. The new concentrate weight is converted to a new volume by dividing by $D_{Bulk}^{18^{\circ}C}$.
4. The $CR^{18^{\circ}C}$ is calculated using Equations 3-1 and 3-5.
5. The $\%WVR^{18^{\circ}C}$ is calculated using Equation 3-6.
6. The final concentrate weight is calculated by subtracting the weight of sample removed.
7. The final step concentrate weight is converted to a final step volume by dividing by $D_{Bulk}^{18^{\circ}C}$.

Table 5-1. Calculation of Test Values of CR and %WVR.

	CON 0	CON 1	CON 2	CON 3	CON 4	CON 5	CON 6	CON 7
Wt Concentrate (g)	578.170	578.170	427.233	367.355	330.019	294.702	260.446	227.625
Vol Concentrate (mL)	473.659	473.659	330.044	275.303	243.038	212.485	183.910	157.281
ΔH_2O (g)	0.000	135.117	42.538	21.387	18.717	16.656	15.062	11.606
Wt Concentrate (g)	578.170	443.053	384.695	345.968	311.302	278.046	245.384	216.019
$D_{Bulk}^{18^\circ C}$ (g/mL)	1.2206	1.2945	1.3344	1.3579	1.3869	1.4162	1.4472	1.4843
Vol Concentrate (mL)	473.657	342.266	288.297	254.784	224.454	196.338	169.552	145.538
$CR^{18^\circ C}$	1.0000	1.3839	1.5843	1.7119	1.8536	2.0061	2.1759	2.3515
%WVR $^{18^\circ C}$	0.000	27.740	36.880	41.585	46.052	50.151	54.043	57.474
Wt Sample Rmvd (g)	0.000	15.820	17.340	15.949	16.600	17.600	17.759	19.869
Wt Concentrate (g)	578.170	427.233	367.355	330.019	294.702	260.446	227.625	196.150
Vol Concentrate (mL)	473.659	330.044	275.303	243.038	212.485	183.910	157.281	132.151

	DIL 1	DIL 2	DIL 3
Wt Concentrate (g)	196.150	299.867	299.658
Vol Concentrate (mL)	132.151	236.067	238.453
ΔH_2O (g)	-120.040	-16.200	-18.130
Wt Concentrate (g)	316.190	316.067	317.788
$D_{Bulk}^{18^\circ C}$ (g/mL)	1.2703	1.2567	1.2413
Vol Concentrate (mL)	248.918	251.510	256.014
$CR^{18^\circ C}$	1.2484	1.1718	1.0914
%WVR $^{18^\circ C}$	19.899	14.659	8.374
Wt Sample Rmvd (g)	16.323	16.409	15.864
Wt Concentrate (g)	299.867	299.658	301.924
Vol Concentrate (mL)	236.067	238.453	243.235

The values of $CR^{18^\circ C}$ and %WVR $^{18^\circ C}$ calculated in Table 5-1 are the buildown progress parameters used throughout the remainder of this report. The $D_{Bulk}^{18^\circ C}$ values are replotted against $CR_{Model}^{18^\circ C}$ and $CR^{18^\circ C}$ in Figure 5-2 for comparison.

Figure 5-2. Bulk Densities at 18 °C versus Concentration Ratio.

5.1.3 Volumes and Densities at Process Temperatures

Estimates of D_{Bulk} of the concentrated slurries at boildown process temperatures (boiling temperatures) are of interest as an indicator of real-time densities that may be observed during a 242-A Evaporator campaign. To estimate these in-process D_{Bulk} values, the volumes of the boildown samples at 18 °C were adjusted to estimated volumes at the boiling temperatures of the pot concentrates at 60 Torr.

The temperature adjustment process is shown in Table 5-2. The total and C-Slds volumes at 18 °C are the base measurements. The fraction of the measured C-Slds volumes that are true solids must be assumed and is assigned a value of 0.5 in the table. The volumes of the solid and liquid portions of the samples at 18 °C are then calculated. A thermal expansion coefficient of $4.335 \times 10^{-4} (\Delta\text{mL/mL})/^{\circ}\text{C}$ was used to adjust the volumes of the liquids from 18-30 °C. A coefficient of $5.364 \times 10^{-4} (\Delta\text{mL/mL})/^{\circ}\text{C}$ was used to adjust the volumes from 30 °C to boiling temperatures at 60 Torr. The sources of the thermal expansion factors were noted in Section 5.1.1.

The adjusted total sample volumes were then calculated by adding the volumes of the 18 °C solids and the adjusted liquid volumes. Temperature adjusted densities, $D_{\text{Bulk}}^{60 \text{ Torr}}$, are simply the sample weights divided by the adjusted total volumes.

Table 5-2. Adjusted Sample Volumes and Densities at Process Temperatures.[Adjusted densities of samples at boiling temperatures at 60 Torr (T_3) in bottom-right column.]

				Vol _{Solids} / Vol _{C-Slds}		
				0.500		
Sample	Wt _{Total} (g)	T ₁ (°C)	Vol _{Total} at T ₁ (mL)	Vol _{C-Slds} at T ₁ (mL)	Vol _{Solids} at T ₁ (mL)	Vol _{Liquid} at T ₁ (mL)
CON-0	14.674	18.0	12.025	0.00	0.000	12.025
CON-1	15.820	18.0	12.221	0.25	0.125	12.096
CON-2	17.340	18.0	12.995	0.35	0.175	12.820
CON-3	15.949	18.0	11.745	0.45	0.225	11.520
CON-4	16.600	18.0	11.969	0.60	0.300	11.669
CON-5	17.600	18.0	12.428	0.70	0.350	12.078
CON-6	17.759	18.0	12.271	0.85	0.425	11.846
CON-7	19.869	18.0	13.386	4.90	2.450	10.936
DIL-1	16.323	18.0	12.850	0.15	0.075	12.775
DIL-2	16.409	18.0	13.058	0.10	0.050	13.008
DIL-3	15.864	18.0	12.780	0.07	0.035	12.745

T = temperature

Sample	(ΔmL/mL)/°C					
	0.00043349		0.00053635			
	T ₂ (°C)	Vol _{Liquid} at T ₂ (mL)	T ₃ (°C)	Vol _{Liquid} at T ₃ (mL)	Vol _{Total} at T ₃ (mL)	D _{Bulk} at T ₃ (g/mL)
CON-0	30.0	12.088	44.87	12.184	12.184	1.204
CON-1	30.0	12.159	46.26	12.265	12.390	1.277
CON-2	30.0	12.887	47.45	13.007	13.182	1.315
CON-3	30.0	11.580	47.84	11.691	11.916	1.338
CON-4	30.0	11.730	49.08	11.850	12.150	1.366
CON-5	30.0	12.141	49.92	12.270	12.620	1.395
CON-6	30.0	11.907	50.81	12.040	12.465	1.425
CON-7	30.0	10.993	51.50	11.120	13.570	1.464
DIL-1	30.0	12.842	45.86	12.951	13.026	1.253
DIL-2	30.0	13.075	45.66	13.185	13.235	1.240
DIL-3	30.0	12.812	45.26	12.916	12.951	1.225

In the above calculations the 18 °C C-Slds are assumed to contain a constant 50% interstitial liquid, and the volumes of solids present at 18 °C and at process temperatures are assumed to be equal. Both these assumptions are almost certainly incorrect. The impact of the assumed solids content of the samples at elevated temperature on the adjusted volumes increases as the solids volume increases and is not large until large volumes of solids are present. Trial calculations showed that the temperature adjusted volume of the sample with the largest volume of solids,

Sample CON 7, decreased by 0.062 mL when the assumed solids fraction of the C-Slds was increased from 0.125 to 0.875. The corresponding increase in $D_{\text{Bulk}}^{60 \text{ Torr}}$ was from 1.461 to 1.468 g/mL

The measured $D_{\text{Bulk}}^{18^\circ\text{C}}$ and estimated $D_{\text{Bulk}}^{60 \text{ Torr}}$ values are plotted against $\% \text{WVR}^{18^\circ\text{C}}$ in Figure 5-3. The estimated $D_{\text{Bulk}}^{60 \text{ Torr}}$ values are based on significant assumptions (true solids content at process temperatures) and indirect supporting data (liquid thermal expansion coefficients). The temperature-adjusted values do indicate, however, that real-time measurements of the densities of concentrated boil-down slurries will return values that are smaller than those based on measurements at lower temperatures. For Samples CON 5 and CON 6, collected before the quantity of solids present began to increase precipitously, the estimated $D_{\text{Bulk}}^{60 \text{ Torr}}$ are 0.022 g/mL smaller than the measured $D_{\text{Bulk}}^{18^\circ\text{C}}$ values over a wide range of assumed solids content.

Figure 5-3. Bulk Densities at Process Temperatures versus $\% \text{WVR}^{18^\circ\text{C}}$.

$$\begin{aligned} \text{Fit Lines: } D_{\text{Bulk}}^{18^\circ\text{C}} &= (1.1721 \times 10^{-6})(\% \text{WVR})^3 - (3.7166 \times 10^{-5})(\% \text{WVR})^2 + (2.8222 \times 10^{-3})(\% \text{WVR}) + 1.2206 \\ D_{\text{Bulk}}^{60 \text{ Torr}} &= (1.3324 \times 10^{-6})(\% \text{WVR})^3 - (5.2423 \times 10^{-5})(\% \text{WVR})^2 + (3.0858 \times 10^{-3})(\% \text{WVR}) + 1.2044 \end{aligned}$$

5.2 BOILDOWN TEMPERATURE CURVES

The boiling temperatures, (T_{Corr}) of the AP-107 liquid/slurries at the three nominal test pressures of 40, 60, and 80 Torr are plotted against boil-down progress in Figure 5-4. The boiling

temperatures are taken from Table B-2 in Attachment B. Boiling temperatures are plotted against $\%WVR^{18\text{ }^{\circ}\text{C}}$ in Figure 5-4(a) and against estimated $D_{\text{Bulk}}^{60\text{ Torr}}$ in Figure 5-4(b).

The temperature curves shown in the figures were constrained to pass through the reference boiling temperatures and densities of pure water at the nominal test pressures. The resulting curves display steadily increasing boiling temperatures as the volume reduction progressed without any significant discontinuities. Within the estimated measurement precision, boiling temperatures measured during the dilution sequence at 60 Torr were those that would have been predicted based on the concentration sequence measurements.

Figure 5-4. Boiling Temperature versus Boildown Progress.

5.3 QUANTITY OF SOLIDS IN COOL CONCENTRATES

5.3.1 Boildown Volume Reduction Samples

The quantities of solids found in the 7 samples that were withdrawn from the boildown pot during the volume reduction sequence (CON 1-7) and then cooled to 18 °C for 8 days are graphically displayed in Figure 5-5. The quantity of solids data was tabulated in Table 3-3. Three quantities are displayed against $D_{\text{Bulk}}^{60 \text{ Torr}}$ in Figure 5-5(a): the vol% S-Slds found after a maximum of 45 hrs⁴ at hot cell ambient temperature (≈ 25.2 °C), the vol% S-Slds found after the samples had been in the 18 °C water bath for 7 days, and the vol% C-Slds found after the samples had been in the water bath for 8 days and were then centrifuged. Photographs of the S-Slds and C-Slds in the samples are presented in Attachment C.

Solids were present in all of the boildown samples at 18 °C, at hot cell ambient temperature, and at process temperatures. The vol% of solids in the cooled CON 2 concentrate was slightly greater than in Sample CON 1. The vol% of solids in the 18 °C concentrates from Steps CON 2-5 changed little. The vol% of solids in the CON 6 concentrate at 18 °C increased slightly. A large increase in the volumes of solids in the CON 7 concentrate signaled the generation of the end-point slurry.

Figure 5-5(a) and the photographs in Attachment C indicate that most of the solids in the boildown samples were present after cooling to ambient hot cell temperatures. The volumes of additional solids that formed while the samples were in the 18 °C water bath were smaller than the decreases in the volumes of the S-Slds layers due to compaction. Sample photographs and observations during the boildown also suggest that a significant portion of the solids found in the 18 °C concentrate samples were already present in the boildown pot at process temperatures.

Comparison of the vol% S-Slds and vol% C-Slds in the samples after cooling to 18 °C for 8 days is informative. The samples settled to produce a clear supernatant liquid within a few hours after removal from the boildown pot. Figure 5-5(a) indicates that the S-Slds layers then compacted slowly. The vol% C-Slds data suggests that the initial volume of S-Slds found in concentrated evaporator feeds might be expected to decrease by as much as 50% as the S-Slds compact over time.

In Figure 5-5(b) the vol% C-Slds and wt% C-Slds data are compared. Both of these quantities can be difficult to measure accurately when small quantities of solids are present. Nevertheless, the two data sets display very similar trends in increase of solids with boildown progress.

⁴ Sample CON 1 sat at hot cell ambient temperature for 45 hrs. The remaining 6 samples sat at hot cell ambient temperature for 26-18 hrs.

Figure 5-5. Quantity of Solids in Cooled Concentrates.

Part of the apparent increase in the amounts of solids present in the concentrates as the volume reduction progresses is due solely to the reduction in the volume of the test slurry. The predicted quantity of solids that would be found in a sample if solids formation ceased after the preceding boildown step is:

$$\text{Predicted \%Slds} = (\% \text{Slds at the end of the previous step}) (CR_{\text{Step}}) \quad (5-1)$$

The predicted and measured quantities of solids present in the boildown volume reduction samples are displayed in Figure 5-6 for the vol% and wt% C-Slds data (18 °C). This format emphasizes that the quantities of new solids in the CON2-CON 6 concentrates were relatively small and relatively consistent. By contrast, the end-point slurry (CON 7) resulted from the formation of a large quantity of new solids.

Figure 5-6. Quantities of New Solids in Concentrates.

5.3.2 End-point Slurry Dilution Samples

The quantities of solids found in the three dilution sequence samples, DIL 1-3, are displayed in Figure 5-7. Data for the vol% C-Slds and wt% C-Slds found in the samples after cooling to 18 °C for 8 days are shown.

Initially, solids were not formed in the CON 1 concentrate at process temperatures. Solids did form after the concentrate cooled to ambient temperature overnight; however, the solids did not redissolve when the CON 1 slurry was heated back to process temperatures. The dilution sample data confirms that the test slurry was saturated with slightly soluble species well before concentration to 27.74 %WVR. Indeed, Sample DIL 3 shows that given sufficient time and/or nucleation sites, solids will precipitate from cooled concentrates after 8.37 %WVR (or less).

Figure 5-7. Quantity of Solids in Dilution Samples at 18 °C.

5.3.3 Calculated Weights of Solids in Boildown Samples

A check of the volume and density measurements/calculations performed in this study can be made by calculating the weights of dry solids predicted to be present after the concentrates from each volume reduction step cooled to 18 °C. After conversion to wt% dry solids values, these calculated weights can be compared to the measured wt% wet C-Slds values.

The calculation series is shown in Table 5-3. Proceeding down a column for each boildown step, the following calculations are performed (rows where the calculations are performed are highlighted).

1. The final volume of the concentrate at 18 °C ($\text{Vol}_{\text{Step}}^{18\text{ °C}}$) is calculated:

$$\text{Vol}_{\text{Initial}}^{18\text{ °C}} / \text{CR}^{18\text{ °C}} \quad (5-2)$$

2. The final weight of the step concentrate, Wt_{Step} , is calculated:

$$(\text{Vol}_{\text{Step}}^{18\text{ °C}}) (\text{D}_{\text{Bulk}}^{18\text{ °C}}) \quad (5-3)$$

3. The estimated volume of liquid in the concentrate at 18 °C, $\text{Vol}_{\text{Liq}}^{18\text{ °C}}$, is calculated:

$$(\text{Vol}_{\text{Step}}^{18\text{ °C}}) (f_{\text{Liq}}) \quad (5-4)$$

4. The weight of the liquid in the concentrate, Wt Liquid, is calculated:

$$(\text{Vol}_{\text{Liq}}^{18\text{ }^{\circ}\text{C}}) (D_{\text{Liq}}^{18\text{ }^{\circ}\text{C}}) \quad (5-5)$$

5. The weight of dry solids in the concentrate, Wt Dry Solids, is calculated:

$$\text{Wt}_{\text{Step}} - \text{Wt Liquid} \quad (5-6)$$

6. The wt% of dry solids in the concentrate is calculated:

$$(\text{Wt Dry Solids} / \text{Wt}_{\text{Step}}) (100) \quad (5-7)$$

Table 5-3. Estimated Dry Weights of Solids in Concentrates at 18 °C.

Step	CON 0	CON 1	CON 2	CON 3	CON 4	CON 5	CON 6	CON 7
%WVR ^{18 °C}	0.00	27.76	36.90	41.60	46.07	50.17	554.06	57.49
Vol _{Initial} ^{18 °C} mL	473.66	—	—	—	—	—	—	—
CR ^{18 °C}	1.000	1.384	1.584	1.712	1.854	2.006	2.176	2.352
Vol _{Step} ^{18 °C} mL	473.7	342.3	299.0	276.7	255.5	236.1	217.7	201.4
D _{Bulk} ^{18 °C} g/mL	1.221	1.294	1.334	1.358	1.387	1.416	1.447	1.484
Wt _{Step} g	578.2	443.0	398.9	375.7	354.4	334.4	315.0	299.0
f _{Liq}	1.000	0.990	0.986	0.981	0.975	0.972	0.965	0.817
Vol _{Liq} ^{18 °C} mL	473.7	338.8	295.0	271.4	249.1	229.5	210.1	164.6
D _{Liq} ^{18 °C} g/mL	1.220	1.296	1.324	1.347	1.376	1.407	1.402	1.434
Wt Liquid g	578.0	439.2	390.4	365.7	342.8	322.9	294.7	236.0
Wt Dry Solids g	0.2	3.9	8.5	10.0	11.6	11.5	20.4	63.0
Wt% Dry Solids	0.0	0.9	2.1	2.7	3.3	3.4	6.5	21.1
Wt% Wet 18 °C C-Slds	0.0	1.9	3.5	4.6	5.8	6.2	9.8	38.8

The liquid fraction, f_{Liq} , in each step concentrate was estimated by assuming that the C-Slds from each concentrate contained 50 vol% solids and 50 vol% interstitial liquid. This was the same assumption used to calculate the total volume of liquid in the boildown samples at 18 °C (Section 5.1.3).

The calculated wt% dry solids in the pot concentrates and the wt% wet 18 °C C-Slds measured in the boildown samples (from Table 3-3) are compared graphically in Figure 5-8. The two values display a very similar trend in solids weight versus boildown progress. The calculated wt% dry solids values are 46 to 66 % of the wt% wet C-Slds values; within the range of commonly observed differences between wet and dry C-Slds. The relative agreement of the calculated and measured solids weights suggests that the volumes and densities measured/calculated in this study are internally consistent and reasonably accurate.

Figure 5-8. Calculated and Measured Weights of Solids in Concentrates.

5.4 COMPOSITION OF BOILDOWN SOLIDS

5.4.1 Quantities of Species Remaining in Solution

Concentrations of chemical species in the C-Liq fractions of the AP-107 test composite and the 10 boildown test samples were tabulated in Table 4-1. It is informative to reformat this data to show how the estimated quantities of species remaining in solution after the concentrates were cooled to 18 °C changed with boildown progress.

The total weight of a species present in a pot concentrate is:

$$Wt_{\text{Species}} = Wt_{\text{In solution}} + Wt_{\text{In precipitated solids}} \quad (5-8)$$

From Eq. 3-1, $CR_{\text{Total}} = \text{Vol}_{\text{Composite}} / \text{Vol}_{\text{Pot Concentrate}}$. The total weight of a species present in a pot concentrate is, therefore:

$$Wt_{\text{Species}} = [(g/L)_{\text{C-Liq}} \times (0.4737 \text{ L} / CR^{18^\circ\text{C}}) \times f_{\text{Liq}}] + [(g/L)_{\text{C-Slds}} \times (0.4737 \text{ L} / CR^{18^\circ\text{C}}) \times f_{\text{Slds}}] \quad (5-9)$$

where f_{Liq} and f_{Slds} are the volume fractions of liquid and solids in the concentrate.

If a species is not removed from the liquid phase by precipitation (or volatilization) during a boildown, the total weight of the species in a pot concentrate reduces to

$$Wt_{\text{Species}} = Wt_{\text{In Solution}} = (g/L)_{\text{C-Liq}} \times (0.4737 \text{ L} / CR^{18^\circ\text{C}}) \times f_{\text{Liq}} \quad (5-10)$$

These values are displayed for the boildown samples in Figure 5-9(a)-(r). The ‘Constant Wt’ line in each plot shows the weight of the species that was present in the 473.7-mL charge of

AP-107 composite added to the pot at the beginning of the boil-down test. As previously noted, values for the test composite (CON 0) and the volume reduction sequence samples (CON 1 to CON 7) are displayed with solid data markers; values for the dilution sequence samples (DIL 1 to DIL 3) are displayed with hollow data markers of the same shape. Plots for the species are presented in decreasing order of their estimated contribution (by weight) to solids present in the 54.04 %WVR^{18 °C} (CON 6) concentrate. Plots for priority analytes bromide and Ca are not included because no analytical results exceeded reported detection limits for any of the boil-down liquid samples. Plots for priority analytes P and S displayed essentially the same trends as those for phosphate and sulfate and have also been omitted.

If a species is not removed from the liquid phase by precipitation (or volatilization) during a boil-down, the calculated weights for all the samples should plot on or near the Constant Wt line. If a species is removed from solution by precipitation, species weights calculated using Equation 5-10 should fall below the Constant Wt line for some or all of the boil-down samples.

Use of a reasonably accurate value of f_{Liq} is of importance (particularly for highly concentrated species and/or when large quantities of solids are present). However, the C-Slds from the boil-down samples were not analyzed and values for f_{Liq} and f_{Slds} were not directly measured. Values for f_{Liq} were estimated using the same assumed composition of sample C-Slds—50 vol% solids and 50 vol% interstitial liquid—used in previous calculations (Sections 5.1.3 and 5.3.3).

The errors associated with the displayed weights of species in solution combine analytical errors, errors in determination of %WVR^{18 °C} ($CR^{18 °C}$) values, and errors in estimation of f_{Liq} . The displayed values are, however, of sufficient accuracy to distinguish between changes due to removal/addition of water from/to the boil-down pot and changes due to solids formation.

Reported minimum detection limit (MDL) values associated with each species have been converted to minimum detectable weight (MDW) values for each sample in the same manner used to calculate the weights in solution. The MDW values are displayed as small horizontal bars below each data point for the boil-down samples.

Complementary to the weights of species in solution are the estimated weights of species in the solids in the 18 °C concentrates. These estimates can be calculated by subtracting the weights of a species remaining in solution from the total weight of the species in the initial composite (i.e., the Constant Wt value). The estimated weights of chemical species that would be found in precipitated solids if the pot concentrates from Steps CON 1, CON 4, CON 6, and CON 7 were cooled to 18 °C are displayed in Figure 5-10. (Small negative calculated weights are displayed with red data markers.)

Figure 5-9. Weights of Species Remaining in Solution at 18 °C. (6 sheets)

Figure 5-9. Weights of Species Remaining in Solution at 18 °C. (6 sheets)

Figure 5-9. Weights of Species Remaining in Solution at 18 °C. (6 sheets)

Figure 5-9. Weights of Species Remaining in Solution at 18 °C. (6 sheets)

Figure 5-9. Weights of Species Remaining in Solution at 18 °C. (6 sheets)

Figure 5-9. Weights of Species Remaining in Solution at 18 °C. (6 sheets)

Figure 5-10. Estimated Weights of Species in 18 °C Solids.

The chemical analysis data indicate that chloride, formate, Si, Al, K, Cr, acetate, and glycolate species do not contribute significantly to the solids formed in the boildown concentrates. The weights of Si, acetate, and glycolate in the starting composite were <1% of the estimated weight of the solids formed in the CON 6 (54.0 %WVR^{18°C}) concentrate. The estimated weights of chloride, formate, Al, K, Cr, and Δ TOC removed from solution were <1% of the estimated weight of the CON 6 solids.

Though not definitive, the chemical analysis data suggests that some or all of the TOC in the starting composite not associated with analyzed organic acid anions (Δ TOC) may have been volatilized and removed from the composite as a gas early in the volume reduction sequence.

5.4.2 Solid Phase Characterization Results

The 5 samples of C-Slds recovered from the AP-107 boildown samples (as identified in Table 3-2) were submitted to 222-S for SPC to identify solid phases present. Three methods were used: polarized light microscopy (PLM), powder X-ray diffraction (XRD), and scanning electron microscopy with energy-dispersive X-ray spectrometry (SEM/EDX). The SPC analyses were performed by members of the 222-S Special Analytical Services and Process Chemistry groups. Analysis results for the three SPC methods, including photographs and spectra, are presented in Attachment E.

The results of the SPC analyses are summarized in Table 5-4. The quantity descriptors are those used by the analysts in the method-specific reports.

Each of the SPC methods is limited to examination of very small quantities of solids. Homogenization and subsampling of radiologically “hot” samples to collect representative specimens of the required weight/volume is difficult at best. Nevertheless, the tabulated results in Table 5-4 are based on analysis of a single specimen for each method. Also, specimens for XRD and SEM/EDX analyses are dried during preparation, and solids dissolved in the liquids collected with the samples may precipitate and complicate interpretation of analysis results. For these reasons, the SPC analyses may identify solid phases that were not actually present in the boildown solids and/or may not identify solid phases that were present. The SPC results should certainly be taken to be qualitative in nature.

The solid phase composition of the first four samples, i.e., the C-Slds from the samples preceding and including the end-point slurry, represent the accumulating inventory of solids in the boildown pot during the volume reduction sequence. The final sample [Table 5-4(e)] shows the solid phases remaining after dilution of the the end-point slurry solids was complete.

Table 5-4. Solid Phases Identified in C-Slds Samples. (2 sheets)(a) S11T014140: Composite of C-Slds from Samples CON 1, CON 2, and CON 3 (41.6 %WVR^{18°C})

Solid Phase	Formula	Observations of Characterization Technique		
		XRD	SEM/EDX	PLM
Natroxalate (sodium oxalate)	Na ₂ C ₂ O ₄	Major	Major	Present
Natrophosphate (sodium fluoride phosphate hydrate)	Na ₇ F(PO ₄) ₂ ·19 H ₂ O	Major	Major	Present
Kogarkoite (sodium fluoride sulfate)	Na ₃ FSO ₄	Minor	Moderate	—
Thermonatrite (sodium carbonate monohydrate)	Na ₂ CO ₃ ·H ₂ O	Minor	Minor	—
Sodium nitrate	NaNO ₃	Major ^a	—	—
Sodium nitrite	NaNO ₂	Minor ^a	—	—

^a Identified as a probable specimen preparation artifact where solids dissolved in the liquid collected with the sample precipitated upon drying.(b) S11T014141: Composite of C-Slds from Samples CON 4 and CON 5 (50.2 %WVR^{18°C})

Solid Phase	Formula	Observations of Characterization Technique		
		XRD	SEM/EDX	PLM
Natroxalate	Na ₂ C ₂ O ₄	Trace	Moderate	Present
Natrophosphate	Na ₇ F(PO ₄) ₂ ·19H ₂ O	Trace	Major	Present
Kogarkoite	Na ₃ FSO ₄	—	Moderate	—
Thermonatrite	Na ₂ CO ₃ ·H ₂ O	Minor	Minor	—
Sodium nitrate	NaNO ₃	Major ^a	—	—
Sodium nitrite	NaNO ₂	Minor ^a	—	—

^a Identified as a probable specimen preparation artifact where solids dissolved in the liquid collected with the sample precipitated upon drying.(c) S11T014280: C-Slds from Sample CON 6 (54.0 %WVR^{18°C})

Solid Phase	Formula	Observations of Characterization Technique		
		XRD	SEM/EDX	PLM
Natroxalate	Na ₂ C ₂ O ₄	—	—	Present
Natrophosphate	Na ₇ F(PO ₄) ₂ ·19 H ₂ O	Minor	Major	Present
Kogarkoite	Na ₃ FSO ₄	—	Moderate	—
Thermonatrite	Na ₂ CO ₃ ·H ₂ O	Minor	—	—
Sodium nitrate	NaNO ₃	Major ^a	Minor	Present
Sodium nitrite	NaNO ₂	Trace ^a	—	—
Sodium phosphate	Na ₃ PO ₄ ·12 H ₂ O	—	—	Present
Sodium sulfate	Na ₂ SO ₄ ^b	—	Present	—

^a Identified as a probable specimen preparation artifact where solids dissolved in the liquid collected with the sample precipitated upon drying.^b A sodium sulfate solid phase with this specific composition was not identified.

Table 5-4. Solid Phases Identified in C-Slds Samples. (2 sheets)(d) S11T011074: C-Slds from end-point slurry (Sample CON 7 at 57.5 %WVR^{18°C})

Solid Phase	Formula	Observations of Characterization Technique		
		XRD	SEM/EDX	PLM
Natroxalate	Na ₂ C ₂ O ₄	—	—	Mostly
Natrophosphate	Na ₇ F(PO ₄) ₂ ·19 H ₂ O	Trace	Major	Some
Kogarkoite	Na ₃ FSO ₄	—	Moderate	—
Thermonatrite	Na ₂ CO ₃ ·H ₂ O	Minor	Moderate	Some
Sodium nitrate	NaNO ₃	Major ^a	Major	—
Sodium nitrite	NaNO ₂ ^a	Minor ^a	—	—
Sodium phosphate	Na ₃ PO ₄ ·12 H ₂ O	—	—	—
Sodium sulfate	Na ₂ SO ₄ ^b	—	—	—
Unidentified solids	—	—	—	Minor ^c

^a Identified as a probable specimen preparation artifact where solids dissolved in the liquid collected with the sample precipitated upon drying.^b A sodium sulfate solid phase with this specific composition was not identified.^c Light-brown, fine-grained material.(e) S11T011075: Solids remaining after dilution of end-point slurry to 8.4 %WVR^{18°C} (Sample DIL 3)

Solid Phase	Formula	Observations of Characterization Technique		
		XRD	SEM/EDX	PLM
Natroxalate	Na ₂ C ₂ O ₄	Major	Major	Present
Natrophosphate	Na ₇ F(PO ₄) ₂ ·19 H ₂ O	Minor	—	—
Kogarkoite	Na ₃ FSO ₄	—	—	—
Thermonatrite	Na ₂ CO ₃ ·H ₂ O	Trace	Trace	—
Sodium nitrate	NaNO ₃	Major ^a	—	—
Sodium nitrite	NaNO ₂	Minor ^a	—	—
Sodium phosphate	Na ₃ PO ₄ ·12 H ₂ O	—	—	—
Sodium sulfate	Na ₂ SO ₄ ^b	—	—	—
Natrite (sodium carbonate)	Na ₂ CO ₃	Minor	Trace	—

^a Identified as a probable specimen preparation artifact where solids dissolved in the liquid collected with the sample precipitated upon drying.^b A sodium sulfate solid phase with this specific composition was not identified.

5.4.3 Solid Phases Present in Concentrate Solids

In Figure 5-11, the chemical analysis data of Section 5.4.1 and the SPC results of Section 5.4.2 are combined. The chemical analysis and SPC results are somewhat inconsistent in their predictions of the composition of the solids formed in the boil-down concentrates, and the data displayed in Figure 5-11 should be treated as semiquantitative in nature.

The estimated weights of the chemical species in the solid fractions of the 18 °C concentrates (displayed in Figure 5-10) were first converted to weights of solid phases identified in SPC analysis or assigned as proxies. (For example, Na_2SO_4 was used as proxy phase for Na-sulfate solids even though this specific phase was not identified in samples of the boil-down solids.) The weights of the solid phases were then adjusted so the sums of the phase weights for each boil-down step were equal to the estimated dry solids weights calculated in Section 5.3.3.

The adjusted phase weights were then converted to solid phase volumes by dividing by the reference densities of the solid phases. Finally, the solid phase volumes were converted to vol% values by dividing by the sum of the volumes of the solid phases estimated to be present in the CON 6 (54.04 %WVR^{18 °C}) concentrate.

In general, the AP-107 boil-down testing indicates that given sufficient time/nucleation sites, sodium oxalate will form in cooled ≤ 8 -%WVR concentrates. The chemical analysis data also suggest that a small amount of carbonate solids precipitate from the cooled <15 -%WVR concentrates.

In cooled <46 -%WVR concentrates most of the oxalate in the feed composite precipitates as sodium oxalate while the relative quantity of the sodium carbonate formed early in the volume reduction is unchanged. At this level of volume reduction $\approx 80\%$ of the phosphate, $\approx 20\%$ of the sulfate, and $\approx 75\%$ of the fluoride in the initial composite precipitates from a cooled concentrate as the sodium fluoride double salts natrophosphate $[\text{Na}_7\text{F}(\text{PO}_4)_2 \cdot 19 \text{H}_2\text{O}]$ and kogarkoite (Na_3FSO_4).

As the volume reduction progresses beyond 46 %WVR, the phosphate in solution actually begins to increase. Phosphate is the only species that displays an increase in solution concentration after significant volume reduction. It is proposed that beyond ≈ 46 %WVR, natrophosphate begins to redissolve in the concentrates due to reduced solution concentrations of sodium, fluoride, and water. Most of the released fluoride is recaptured as additional kogarkoite.

At 54 %WVR nearly all the oxalate and fluoride in the initial feed composite precipitates from a cooled concentrate as sodium oxalate, natrophosphate, and kogarkoite. There is insufficient fluoride in the initial feed composite to form only the sodium fluoride double salts with the available phosphate and sulfate. Portions of the phosphate and sulfate remaining in solution after the fluoride has been exhausted precipitate as sodium phosphate and sodium sulfate salts.

Figure 5-11. Estimated Vol% of Solid Phases in Concentrate Solids.

As the volume reduction exceeds 54 %WVR, significant quantities of sodium nitrate and sodium carbonate precipitate from a cooled concentrate and become the major components of the solid precipitates.

Nitrate, nitrite, and TIC (as CO_3) were, by far, the most concentrated anionic species in the initial feed composite, and relatively small analytical and/or computational errors could result in significant errors in estimation of their relative contributions to solids formed in cooled boildown concentrates. While the contributions of the solid phases containing these species may be somewhat overestimated (particularly in solids from cooled concentrates produced early in the boildown volume reduction), the analytical data clearly indicate that precipitation of sodium nitrate, sodium carbonate, and sodium nitrite is the major cause of the dramatic increase in the quantity of solids in the concentrate after volume reduction of the AP-107 feed composite progressed beyond 54 %WVR.

6.0 QUALITY ASSURANCE

6.1 HOT CELLS OPERATIONS

As discussed in Section 2.2, temperature measurements recorded during the hot cells boildown operations were corrected to true readings provided by a certified Pt-RTD. The resistance thermometer was calibrated using equipment which can be traced back to established national standards, e.g. the National Institute of Standards and Technology (NIST). The Certificate of Calibration for this reference thermometer was issued May 10, 2011, and was valid for 1 year. A copy of the calibration certificate is included in the project file.

Pressure measurements in the hot cells operations were indirectly calibrated through the verified temperature readings. The measured pressures over pure water boiling at measured temperatures were compared with tabulated vapor pressures of pure water at the same temperatures.

The in-tolerance status of each analytical balance used during the boildown study 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.

6.2 LABORATORY ANALYSES

Quality control measurements required to ensure the quality of the analyses of the boildown samples performed at 222-S 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 are included in the transcript of the analytical results presented in Attachment D.

6.3 DEVIATIONS FROM TEST PLAN

The AP-107 boildown study was executed, in general, as described in the client-approved laboratory test plan (LAB-PLN-10-00007). The test plan incorporated some minor deviations from the guidance for completion of the boildown study provided in the TSAP (RPP-PLAN-40837); these changes were executed with the concurrence of the 242-A Evaporator Technical Point of Contact.

7.0 REFERENCES

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ATTACHMENT A

VOLUME CALIBRATION OF CENTRIFUGE CONES

ATTACHMENT A **VOLUME CALIBRATIONS OF CENTRIFUGE CONES**

The volumes read (Vol_{Read}) from graduations on the 15-mL centrifuge cones used to collect the boildown samples were adjusted to corrected values ($\text{Vol}_{\text{Corrected}}$). The gravimetric data collected and calculations performed in deriving the correction functions are presented in Table A-1.

Table A-1. Centrifuge Cone Volume Calibrations. (6 sheets)

Cone ID	Cal Date	H ₂ O		Cone Mark = Volume Mark on Cone (mL)			
		T (°C)	D (g/mL)	Wt H ₂ O Added = Wt H ₂ O Added to Cone (g)			
				Vol H ₂ O Added = Wt H ₂ O (g) / D H ₂ O (g/mL)			
AP-107 BLDN Cone 1	07/25/11	22.5	0.99766	Cone Mark	10.0	11.5*	13.0
				Wt H ₂ O Added (g)	9.9940	11.3624*	12.8902
				Vol H ₂ O (mL)	10.0175	11.3891*	12.9205
	07/25/11	22.5	0.99766	Cone Mark	10.0	11.5	13.0
				Wt H ₂ O Added (g)	9.9774	11.4073	12.8693
				Vol H ₂ O (mL)	10.0008	11.4341	12.8995
	07/25/11	22.5	0.99766	Cone Mark	10.0	11.5	13.0
				Wt H ₂ O Added (g)	9.9661	11.4437	12.9021
				Vol H ₂ O (mL)	9.9895	11.4706	12.9324
	07/27/11	22.9	0.99756	Cone Mark	10.0	11.5	13.0
				Wt H ₂ O Added (g)	9.9574	11.4301	12.9225
				Vol H ₂ O (mL)	9.9817	11.4580	12.9541
	07/27/11	22.9	0.99756	Cone Mark	10.0	11.5	13.0
				Wt H ₂ O Added (g)	9.9351	11.4089	12.9062
				Vol H ₂ O (mL)	9.9594	11.4368	12.9377
	07/27/11	22.9	0.99756	Cone Mark	10.0	11.5	13.0
				Wt H ₂ O Added (g)	9.9844	11.4330	12.9027
				Vol H ₂ O (mL)	10.0088	11.4609	12.9342
Cone 1 — Sample CON 0B				Vol _{Corrected} = (0.9789 × Vol _{Read}) + 0.2009			

D = density

T = temperature

* Outlier: not used in formulation of the calibration equation.

Table A-1. Centrifuge Cone Volume Calibrations. (6 sheets)

Cone ID	Cal Date	H ₂ O		Cone Mark = Volume Mark on Cone (mL)			
				Wt H ₂ O Added = Wt H ₂ O Added to Cone (g)			
		T (°C)	D (g/mL)	Vol H ₂ O Added = Wt H ₂ O (g) / D H ₂ O (g/mL)			
AP-107 BLDN Cone 2	07/25/11	22.5	0.99766	Cone Mark	10.0	11.5	13.0
				Wt H ₂ O Added (g)	10.0414	11.4710	12.9498
				Vol H ₂ O (mL)	10.0650	11.4979	12.9802
	07/25/11	22.5	0.99766	Cone Mark	10.0	11.5	13.0
				Wt H ₂ O Added (g)	10.0206	11.4863	12.9615
				Vol H ₂ O (mL)	10.0441	11.5133	12.9919
	07/25/11	22.5	0.99766	Cone Mark	10.0	11.5	13.0
				Wt H ₂ O Added (g)	10.0291	11.4762	12.9468
				Vol H ₂ O (mL)	10.0526	11.5031	12.9772
Cone 2 — Sample CON 1				Vol _{Corrected} = (0.9764 × Vol _{Read}) + 0.2857			

Cone ID	Cal Date	H ₂ O		Cone Mark = Volume Mark on Cone (mL)			
				Wt H ₂ O Added = Wt H ₂ O Added to Cone (g)			
		T (°C)	D (g/mL)	Vol H ₂ O Added = Wt H ₂ O (g) / D H ₂ O (g/mL)			
AP-107 BLDN Cone 3	07/25/11	22.2	0.99773	Cone Mark	10.0	11.5	13.0
				Wt H ₂ O Added (g)	9.9056	11.3682	12.8385
				Vol H ₂ O (mL)	9.9282	11.3941	12.8678
	07/25/11	22.3	0.99770	Cone Mark	10.0	11.5	13.0
				Wt H ₂ O Added (g)	9.8817	11.4072	12.8355
				Vol H ₂ O (mL)	9.9045	11.4335	12.8651
	07/25/11	22.4	0.99768	Cone Mark	10.0	11.5	13.0
				Wt H ₂ O Added (g)	9.9485	11.3702	12.8204
				Vol H ₂ O (mL)	9.9716	11.3967	12.8502
Cone 3 — Sample CON 2				Vol _{Corrected} = (0.9754 × Vol _{Read}) + 0.1840			

Table A-1. Centrifuge Cone Volume Calibrations. (6 sheets)

Cone ID	Cal Date	H ₂ O		Cone Mark = Volume Mark on Cone (mL)			
				Wt H ₂ O Added = Wt H ₂ O Added to Cone (g)			
		T (°C)	D (g/mL)	Vol H ₂ O Added = Wt H ₂ O (g) / D H ₂ O (g/mL)			
AP-107 BLDN Cone 4	07/25/11	22.5	0.99766	Cone Mark	10.0	11.5	13.0
				Wt H ₂ O Added (g)	9.9743	11.3731	12.8469
				Vol H ₂ O (mL)	9.9977	11.3998	12.8771
	07/25/11	22.5	0.99766	Cone Mark	10.0	11.5	13.0
				Wt H ₂ O Added (g)	9.9443	11.3821	12.8540
				Vol H ₂ O (mL)	9.9677	11.4088	12.8842
	07/25/11	22.4	0.99768	Cone Mark	10.0	11.5	13.0
				Wt H ₂ O Added (g)	9.9455	11.3803	12.8814
				Vol H ₂ O (mL)	9.9686	11.4068	12.9114
Cone 4 — Sample CON 3				Vol _{Corrected} = (0.9710 × Vol _{Read}) + 0.2587			

Cone ID	Cal Date	H ₂ O		Cone Mark = Volume Mark on Cone (mL)			
				Wt H ₂ O Added = Wt H ₂ O Added to Cone (g)			
		T (°C)	D (g/mL)	Vol H ₂ O Added = Wt H ₂ O (g) / D H ₂ O (g/mL)			
AP-107 BLDN Cone 5	07/25/11	22.5	0.99766	Cone Mark	10.0	11.5	13.0
				Wt H ₂ O Added (g)	9.9526	11.4132	12.8517
				Vol H ₂ O (mL)	9.9760	11.4400	12.8819
	07/25/11	22.5	0.99766	Cone Mark	10.0	11.5	13.0
				Wt H ₂ O Added (g)	9.9346	11.4323	12.8827
				Vol H ₂ O (mL)	9.9579	11.4592	12.9130
	07/25/11	22.5	0.99766	Cone Mark	10.0	11.5	13.0
				Wt H ₂ O Added (g)	9.9396	11.4126	12.8840
				Vol H ₂ O (mL)	9.9629	11.4394	12.9143
Cone 5 — Sample CON 4				Vol _{Corrected} = (0.9792 × Vol _{Read}) + 0.1779			

Table A-1. Centrifuge Cone Volume Calibrations. (6 sheets)

Cone ID	Cal Date	H ₂ O		Cone Mark = Volume Mark on Cone (mL)			
		T (°C)	D (g/mL)	Wt H ₂ O Added = Wt H ₂ O Added to Cone (g)			
				Vol H ₂ O Added = Wt H ₂ O (g) / D H ₂ O (g/mL)			
AP-107 BLDN Cone 6	07/25/11	22.3	0.99770	Cone Mark	10.0	11.5	13.0
				Wt H ₂ O Added (g)	9.9429	11.3936	12.8602
				Vol H ₂ O (mL)	9.9658	11.4198	12.8898
	07/25/11	22.3	0.99770	Cone Mark	10.0	11.5	13.0
				Wt H ₂ O Added (g)	9.9982	11.4400	12.8992
				Vol H ₂ O (mL)	10.0212	11.4663	12.9289
	07/25/11	22.4	0.99768	Cone Mark	10.0	11.5	13.0
				Wt H ₂ O Added (g)	9.9628	11.4156	12.8535
				Vol H ₂ O (mL)	9.9860	11.4422	12.8834
Cone 6 — Sample CON 5				Vol _{Corrected} = (0.9699 × Vol _{Read}) + 0.2909			

Cone ID	Cal Date	H ₂ O		Cone Mark = Volume Mark on Cone (mL)			
		T (°C)	D (g/mL)	Wt H ₂ O Added = Wt H ₂ O Added to Cone (g)			
				Vol H ₂ O Added = Wt H ₂ O (g) / D H ₂ O (g/mL)			
AP-107 BLDN Cone 7	07/25/11	22.6	0.99763	Cone Mark	10.0	11.5	13.0
				Wt H ₂ O Added (g)	9.9323	11.3952	12.8581
				Vol H ₂ O (mL)	9.9559	11.4222	12.8886
	07/25/11	22.6	0.99763	Cone Mark	10.0	11.5	13.0
				Wt H ₂ O Added (g)	9.9572	11.3749	12.8559
				Vol H ₂ O (mL)	9.9808	11.4019	12.8864
	07/25/11	22.6	0.99763	Cone Mark	10.0	11.5	13.0
				Wt H ₂ O Added (g)	9.9149	11.4045	12.8549
				Vol H ₂ O (mL)	9.9384	11.4316	12.8854
Cone 7 — Sample CON 6				Vol _{Corrected} = (0.9761 × Vol _{Read}) + 0.1963			

Table A-1. Centrifuge Cone Volume Calibrations. (6 sheets)

Cone ID	Cal Date	H ₂ O		Cone Mark = Volume Mark on Cone (mL)			
		T (°C)	D (g/mL)	Wt H ₂ O Added = Wt H ₂ O Added to Cone (g)			
				Vol H ₂ O Added = Wt H ₂ O (g) / D H ₂ O (g/mL)			
AP-107 BLDN Cone 8	07/25/11	22.5	0.99766	Cone Mark	10.0	11.5	13.0
				Wt H ₂ O Added (g)	10.0309	11.4779	12.9459
				Vol H ₂ O (mL)	10.0545	11.5049	12.9763
	07/25/11	22.5	0.99766	Cone Mark	10.0	11.5	13.0
				Wt H ₂ O Added (g)	10.0791	11.4883	12.9631
				Vol H ₂ O (mL)	10.1028	11.5153	12.9936
	07/25/11	22.6	0.99763	Cone Mark	10.0	11.5	13.0
				Wt H ₂ O Added (g)	10.0323	11.5509	12.9661
				Vol H ₂ O (mL)	10.0561	11.5783	12.9969
	07/27/11	22.9	0.99756	Cone Mark	10.0	11.5	13.0
				Wt H ₂ O Added (g)	10.0317	11.5397	12.9927
				Vol H ₂ O (mL)	10.0562	11.5679	13.0245
	07/27/11	22.9	0.99756	Cone Mark	10.0	11.5	13.0
				Wt H ₂ O Added (g)	10.0576	11.5299	12.9661
				Vol H ₂ O (mL)	10.0822	11.5581	12.9978
	07/27/11	22.9	0.99756	Cone Mark	10.0	11.5	13.0
				Wt H ₂ O Added (g)	10.0616	11.4803	13.0049
				Vol H ₂ O (mL)	10.0862	11.5084	13.0367
Cone 8 — Sample CON 7				Vol _{Corrected} = (0.9771 × Vol _{Read}) + 0.3019			

Cone ID	Cal Date	H ₂ O T (°C) D (g/mL)		Cone Mark = Volume Mark on Cone (mL)			
				Wt H ₂ O Added = Wt H ₂ O Added to Cone (g)			
		Vol H ₂ O Added = Wt H ₂ O (g) / D H ₂ O (g/mL)					
AP-107 BLDN Cone 9	07/25/11	22.5	0.99766	Cone Mark	10.0	11.5	13.0
				Wt H ₂ O Added (g)	9.9209	11.3912	12.8626
				Vol H ₂ O (mL)	9.9442	11.4180	12.8928
	07/25/11	22.4	0.99768	Cone Mark	10.0	11.5	13.0
				Wt H ₂ O Added (g)	9.9363	11.3755	12.8726
				Vol H ₂ O (mL)	9.9594	11.4020	12.9025
	07/25/11	22.5	0.99766	Cone Mark	10.0	11.5	13.0
				Wt H ₂ O Added (g)	9.9521	11.4144	12.8538
				Vol H ₂ O (mL)	9.9754	11.4412	12.8840
Cone 9 — Sample DIL 1				Vol _{Corrected} = (0.9778 × Vol _{Read}) + 0.1796			

Table A-1. Centrifuge Cone Volume Calibrations. (6 sheets)

Cone ID	Cal Date	H ₂ O		Cone Mark = Volume Mark on Cone (mL)			
				Wt H ₂ O Added = Wt H ₂ O Added to Cone (g)			
		T (°C)	D (g/mL)	Vol H ₂ O Added = Wt H ₂ O (g) / D H ₂ O (g/mL)			
AP-107 BLDN Cone 10	07/25/11	22.3	0.99770	Cone Mark	10.0	11.5	13.0
				Wt H ₂ O Added (g)	9.9715	11.4610	12.9076
				Vol H ₂ O (mL)	9.9945	11.4874	12.9373
	07/25/11	22.4	0.99768	Cone Mark	10.0	11.5	13.0
				Wt H ₂ O Added (g)	10.0295	11.4442	12.9045
				Vol H ₂ O (mL)	10.0528	11.4708	12.9345
	07/25/11	22.5	0.99766	Cone Mark	10.0	11.5	13.0
				Wt H ₂ O Added (g)	10.0144	11.4744	12.9323
				Vol H ₂ O (mL)	10.0379	11.5014	12.9627
Cone 10 — Sample DIL 2				Vol _{Corrected} = (0.9721 × Vol _{Read}) + 0.3069			

Cone ID	Cal Date	H ₂ O		Cone Mark = Volume Mark on Cone (mL)			
				Wt H ₂ O Added = Wt H ₂ O Added to Cone (g)			
		T (°C)	D (g/mL)	Vol H ₂ O Added = Wt H ₂ O (g) / D H ₂ O (g/mL)			
AP-107 BLDN Cone 11	07/25/11	22.6	0.99763	Cone Mark	10.0	11.5	13.0
				Wt H ₂ O Added (g)	9.9381	11.3823	12.8376
				Vol H ₂ O (mL)	9.9617	11.4093	12.8681
	07/25/11	22.6	0.99763	Cone Mark	10.0	11.5	13.0
				Wt H ₂ O Added (g)	9.9010	11.3961	12.8335
				Vol H ₂ O (mL)	9.9245	11.4232	12.8640
	07/25/11	22.6	0.99763	Cone Mark	10.0	11.5	13.0
				Wt H ₂ O Added (g)	9.9402	11.4269	12.8353
				Vol H ₂ O (mL)	9.9638	11.4540	12.8658
Cone 11 — Sample DIL 3				Vol _{Corrected} = (0.9719 × Vol _{Read}) + 0.2379			

ATTACHMENT B

BOILDOWN HOT CELL DATA LOGS

ATTACHMENT B

BOILDOWN HOT CELL DATA LOGS

The hot cell data log reproduced in Table B-1 was used to track the progress of the boildown operations and to calculate volumes of water ($\Delta \text{H}_2\text{O}$) that needed to be distilled from or added to the test sample to reach target concentrations or densities. Quantities of water removed from or added to the boildown pot were weighed and then converted to volumes by dividing by the density of pure water at 26.8 °C. The volumes of samples removed from the pot were calculated by dividing the weight of the samples by the model density ($D_{\text{Model}}^{26.8\text{ °C}}$) calculated by the log spreadsheet.

A registered and verified Excel spreadsheet (RPP-38843, *Spreadsheet Description Document for Boildown Test Data Spreadsheet*; SVF-1543 Rev. 0, “CH2M HILL Spreadsheet Verification and Release Form”) was used to generate the hot cell data log. Within the log, the chronology of entries proceeds from left to right across a row, and then down to the following row.

The primary hot cell data log was subsequently modified by changing the base reference temperature for the model calculations from 26.8 °C to 18 °C. This revised data log is presented in Table B-2.

The column headings/entries in the data logs are defined as follows:

Sample Rmvd (mL)	Volume of sample withdrawn from pot at beginning of Step
	= Manual data entry
Pot Vol Initial (mL)	Volume of pot contents after removal of a sample following the conclusion of a preceding concentration/dilution step
	= (Pot Vol Final) _{previous} – (Sample Vol Corrected) _{current}
$\Delta \text{H}_2\text{O}$ (mL)	Volume of water removed from or added to pot contents during a concentration/dilution step
	= Manual data entry with additions entered as negative values
Pot Vol Final (mL)	Final volume of pot contents for a step
	= (Pot Vol Initial) _{current} – ($\Delta \text{H}_2\text{O}$) _{current}
CR Step	(Incremental) concentration ratio for a step
	= (Pot Vol Initial) _{current} ÷ (Pot Vol Final) _{current}
CR Total	(Cumulative) concentration ratio applicable to a boildown since the last baseline reset
	= (CR Total) _{previous} × (CR Step) _{current}

%WVR	% waste volume reduction applicable to a boildown since the last baseline reset
	$= \{[(CR \text{ Total})_{\text{current}} - 1.0] \div (CR \text{ Total})_{\text{current}}\} \times 100$
D_{Model} (g/mL)	Calculated density of pot contents, based on simple additive additions model, at the end of a boildown step
	$= [(D_{\text{Initial Composite}} - D_{\text{H}_2\text{O}}) \times CR \text{ Total}] + D_{\text{H}_2\text{O}}$
P_{Read} (Torr)	Pressure in the boildown apparatus read from pressure reader/controller
	= Manual data entry
P_{Corr} (Torr)	Corrected pressure in boildown apparatus
	$= 0.9823 (P_{\text{Read}}) + 1.720$
T_{Read} (°C)	Temperature of pot contents from readout of K-type thermocouple/reader
	= Manual data entry
T_{Corr} (°C)	Corrected temperature of pot contents
	$= 1.69 \times 10^{-6} (T_{\text{Read}})^3 - 5.59 \times 10^{-4} (T_{\text{Read}})^2 + 1.03(T_{\text{Read}}) - 0.197$

The abbreviations used in the row entries in the ‘Action’ column are defined as follows:

CON #	Boildown volume reduction Step # and Sample #
DIL #	Boildown dilution Step # and Sample #
P/T Step # [P_{nominal}]	Measurement of boiling temperature (T) and system pressure (P) during specified boildown/ dilution step (#) at nominal test pressure of 40, 60, or 80 Torr

Table B-1. Tank 241-AP-107 Boildown Hot Cell Data Log. (3 sheets)

Vol Initial Composite (mL) = 578.170 g / 1.21599 g/mL

$\Delta \text{H}_2\text{O (mL)} = \text{Wt H}_2\text{O} / D_{\text{H}_2\text{O}}$ at 26.8 °C

Sample Rmvd (mL) = Wt Sample / $D_{\text{Model}}^{26.8 \text{ }^\circ\text{C}}$

2011		Action	Sample Rmvd (mL)	Pot Vol Initial (mL)	$\Delta \text{H}_2\text{O}$ (mL)	Pot Vol Final (mL)	CR Step	CR Total	%WVR	$D_{\text{H}_2\text{O}}$	D_{Model}	P_{Read} (Torr)	P_{Corr} (Torr)	T_{Read} (°C)	T_{Corr} (°C)
Date mm/dd	Time hh:mm										(g/mL)				
10/03	08:30	Initial Composite	0.0	475.473	0.0	475.473	1.0000	1.0000	0.000		1.2160				
	09:25	P/T 0 [40]	0.0	475.473	0.0	475.473	1.0000	1.0000	0.000		1.2160	40	41	37.6	37.9
	09:38	P/T 0 [60]	0.0	475.473	0.0	475.473	1.0000	1.0000	0.000		1.2160	60	61	44.6	44.9
	09:53	P/T 0 [80]	0.0	475.473	0.0	475.473	1.0000	1.0000	0.000		1.2160	79	79	50.4	50.6
	10:00	CON 1a [40]	0.0	475.473	45.342	430.131	1.1054	1.1054	9.536		1.2391				
	10:54	P/T 1a [40]	0.0	430.131	0.0	430.131	1.0000	1.1054	9.536		1.2391	39	40	37.3	37.6
	11:06	P/T 1a [60]	0.0	430.131	0.0	430.131	1.0000	1.1054	9.536		1.2391	60	61	45.3	45.6
	11:13	P/T 1a [80]	0.0	430.131	0.0	430.131	1.0000	1.1054	9.536		1.2391	79	79	50.7	50.9
	11:17	CON 1b [40]	0.0	430.131	46.228	383.903	1.1204	1.2385	19.259		1.2683				
	12:01	P/T 1b [40]	0.0	383.903	0.0	383.903	1.0000	1.2385	19.259		1.2683	39	40	38.0	38.3
	12:11	P/T 1b [60]	0.0	383.903	0.0	383.903	1.0000	1.2385	19.259		1.2683	58	59	45.2	45.5
	12:18	P/T 1b [80]	0.0	383.903	0.0	383.903	1.0000	1.2385	19.259		1.2683	79	79	51.3	51.5
	12:22	CON 1c [40]	0.0	383.903	44.013	339.890	1.1295	1.3989	28.515		1.3035				
	13:03	P/T 1c [40]	0.0	339.890	0.0	339.890	1.0000	1.3989	28.515		1.3035	39	40	38.5	38.8
	13:15	P/T 1c [60]	0.0	339.890	0.0	339.890	1.0000	1.3989	28.515		1.3035	59	60	46.0	46.3
	13:21	P/T 1c [80]	0.0	339.890	0.0	339.890	1.0000	1.3989	28.515		1.3035	79	79	51.8	52.0
	13:50	SAMPLE CON 1	12.136	327.753	0.0	327.753	1.0000	1.3989	28.515		1.3035				

Table B-1. Tank 241-AP-107 Boildown Hot Cell Data Log. (3 sheets)

. D _{H2O}										0.996568				
2011		Action	Sample Rmvd (mL)	Pot Vol Initial (mL)	Δ H ₂ O (mL)	Pot Vol Final (mL)	CR Step	CR Total	%WVR	D _{Model}	P _{Read}	P _{Corr}	T _{Read}	T _{Corr}
Date mm/dd	Time hh:mm									(g/mL)	(Torr)	(Torr)	(°C)	(°C)
10/04	07:30	P/T 1d [40]	0.0	327.753	0.0	327.753	1.0000	1.3989	28.515	1.3035	38	39	37.7	38.0
	07:45	P/T 1d [60]	0.0	327.753	0.0	327.753	1.0000	1.3989	28.515	1.3035	58	59	45.7	46.0
	07:55	P/T 1d [80]	0.0	327.753	0.0	327.753	1.0000	1.3989	28.515	1.3035	79	79	51.8	52.0
	07:58	CON 2 [80]	0.0	327.753	42.684	285.069	1.1497	1.6084	37.825	1.3495				
	08:40	P/T 2 [80]	0.0	285.069	0.0	285.069	1.0000	1.6084	37.825	1.3495	79	79	52.8	53.0
	08:44	P/T 2 [60]	0.0	285.069	0.0	285.069	1.0000	1.6084	37.825	1.3495	60	61	47.2	47.4
	08:48	P/T 2 [40]	0.0	285.069	0.0	285.069	1.0000	1.6084	37.825	1.3495	39	40	39.3	39.6
	09:15	SAMPLE CON 2	12.849	272.220	0.0	272.220	1.0000	1.6084	37.825	1.3495				
	09:30	CON 3 [40]	0.0	272.220	21.461	250.759	1.0856	1.7460	42.727	1.3797				
	09:54	P/T 3 [40]	0.0	250.759	0.0	250.759	1.0000	1.7460	42.727	1.3797	39	40	40.0	40.3
	10:02	P/T 3 [60]	0.0	250.759	0.0	250.759	1.0000	1.7460	42.727	1.3797	59	60	47.6	47.8
	10:07	P/T 3 [80]	0.0	250.759	0.0	250.759	1.0000	1.7460	42.727	1.3797	79	79	53.4	53.6
	10:30	SAMPLE CON 3	11.560	239.199	0.0	239.199	1.0000	1.7460	42.727	1.3797				
	10:45	CON 4 [80]	0.0	239.199	18.781	220.418	1.0852	1.8948	47.224	1.4123				
	11:16	P/T 4 [80]	0.0	220.418	0.0	220.418	1.0000	1.8948	47.224	1.4123	79	79	54.4	54.6
	11:20	P/T 4 [60]	0.0	220.418	0.0	220.418	1.0000	1.8948	47.224	1.4123	60	61	48.9	49.1
	11:28	P/T 4 [40]	0.0	220.418	0.0	220.418	1.0000	1.8948	47.224	1.4123	40	41	41.2	41.5
	11:36	SAMPLE CON 4	11.754	208.664	0.0	208.664	1.0000	1.8948	47.224	1.4123				
	11:50	CON 5 [40]	0.0	208.664	16.713	191.951	1.0871	2.0598	51.451	1.4485				
	12:15	P/T 5 [40]	0.0	191.951	0.0	191.951	1.0000	2.0598	51.451	1.4485	40	41	42.3	42.6
	12:20	P/T 5 [60]	0.0	191.951	0.0	191.951	1.0000	2.0598	51.451	1.4485	60	61	49.7	49.9
	12:23	P/T 5 [80]	0.0	191.951	0.0	191.951	1.0000	2.0598	51.451	1.4485	79	79	55.2	55.3
	12:35	SAMPLE CON 5	12.150	179.801	0.0	179.801	1.0000	2.0598	51.451	1.4485				

Table B-1. Tank 241-AP-107 Boildown Hot Cell Data Log. (3 sheets)

2011		Action	Sample Rmvd (mL)	Pot Vol Initial (mL)	Δ H ₂ O (mL)	Pot Vol Final (mL)	CR Step	CR Total	%WVR	ρ_{H_2O}	D _{Model} (g/mL)	P _{Read} (Torr)	P _{Corr} (Torr)	T _{Read} (°C)	T _{Corr} (°C)
Date mm/dd	Time hh:mm									0.996568					
10/04	13:00	CON 6 [80]	0.0	179.801	15.114	164.687	1.0918	2.2488	55.532	1.4900					
	13:36	P/T 6 [80]	0.0	164.687	0.0	164.687	1.0000	2.2488	55.532	1.4900	79	79	56.7	56.8	
	13:40	P/T 6 [60]	0.0	164.687	0.0	164.687	1.0000	2.2488	55.532	1.4900	59	60	50.6	50.8	
	13:44	P/T 6 [40]	0.0	164.687	0.0	164.687	1.0000	2.2488	55.532	1.4900	39	40	42.7	43.0	
	14:02	SAMPLE CON 6	11.919	152.768	0.0	152.768	1.0000	2.2488	55.532	1.4900					
	14:18	CON 7 [40]	0.0	152.768	11.646	141.122	1.0825	2.4344	58.922	1.5307					
	14:37	P/T 7 [40]	0.0	141.122	0.0	141.122	1.0000	2.4344	58.922	1.5307	39	40	43.1	43.4	
	14:41	P/T 7 [60]	0.0	141.122	0.0	141.122	1.0000	2.4344	58.922	1.5307	60	61	51.3	51.5	
	14:47	P/T 7 [80]	0.0	141.122	0.0	141.122	1.0000	2.4344	58.922	1.5307	80	80	57.0	57.1	
	15:04	SAMPLE CON 7	12.980	128.142	0.0	128.142	1.0000	2.4344	58.922	1.5307					
	15:34	DIL 1	0.0	128.142	-120.453	248.595	0.5155	1.2548	20.308	1.2719					
	16:00	P/T 8 [60]	0.0	248.595	0.0	248.595	1.0000	1.2548	20.308	1.2719	59	60	45.6	45.9	
	16:12	SAMPLE DIL 1	12.834	235.762	0.0	235.762	1.0000	1.2548	20.308	1.2719					
	16:40	DIL 2	0.0	235.762	-16.256	252.018	0.9355	1.1739	14.814	1.2541					
	16:59	P/T 9 [60]	0.0	252.018	0.0	252.018	1.0000	1.1739	14.814	1.2541	59	60	45.4	45.7	
	17:07	SAMPLE DIL 2	13.084	238.934	0.0	238.934	1.0000	1.1739	14.814	1.2541					
	17:37	DIL 3	0.0	238.934	-18.192	257.126	0.9292	1.0908	8.328	1.2359					
	17:54	P/T 10 [60]	0.0	257.126	0.0	257.126	1.0000	1.0908	8.328	1.2359	59	60	45.0	45.3	
	18:05	SAMPLE DIL 3	12.836	244.290	0.0	244.290	1.0000	1.0908	8.328	1.2359					

Table B-2. Revised Tank 241-AP-107 Boildown Hot Cell Data Log. (3 sheets)

$$\text{Initial Composite Vol}^{18^\circ\text{C}} (\text{mL}) = \text{Vol}^{26.8^\circ\text{C}} + [(\text{Vol}^{26.8^\circ\text{C}}) (0.00043349 \Delta\text{V/mL}) (-8.8^\circ\text{C})]$$

$$\text{Initial Composite D}_{\text{Model}}^{18^\circ\text{C}} (\text{g/mL}) = 578.170 \text{ g} / \text{Vol}^{18^\circ\text{C}}$$

$$\Delta \text{H}_2\text{O} (\text{mL}) = \text{Wt H}_2\text{O} / \text{D}_{\text{H}_2\text{O}}^{18^\circ\text{C}}$$

$$\text{Sample Rmvd} (\text{mL}) = \text{Vol}_{\text{Corrected}} \text{ based on Vol}_{\text{Read}} \text{ measured from sample photograph}$$

D _{H2O}										0.998595					
2011		Action	Sample Rmvd (mL)	Pot Vol Initial (mL)	Δ H ₂ O (mL)	Pot Vol Final (mL)	CR Step	CR Total	%WVR	D _{Model}	P _{Read}	P _{Corr}	T _{Read}	T _{Corr}	
Date mm/dd	Time hh:mm									(g/mL)	(Torr)	(Torr)	(°C)	(°C)	
10/03	08:30	Initial Composite	0.0	473.659	0.0	473.659	1.0000	1.0000	0.000	1.2206					
	09:25	P/T 0 [40]	0.0	473.659	0.0	473.659	1.0000	1.0000	0.000	1.2206	40	41.0	37.6	37.86	
	09:38	P/T 0 [60]	0.0	473.659	0.0	473.659	1.0000	1.0000	0.000	1.2206	60	60.7	44.6	44.87	
	09:53	P/T 0 [80]	0.0	473.659	0.0	473.659	1.0000	1.0000	0.000	1.2206	79	79.3	50.4	50.56	
	10:00	CON 1a [40]	0.0	473.659	45.250	428.409	1.1056	1.1056	9.553	1.2441					
	10:54	P/T 1a [40]	0.0	428.409	0.0	428.409	1.0000	1.1056	9.553	1.2441	39	40.0	37.3	37.61	
	11:06	P/T 1a [60]	0.0	428.409	0.0	428.409	1.0000	1.1056	9.553	1.2441	60	60.7	45.3	45.56	
	11:13	P/T 1a [80]	0.0	428.409	0.0	428.409	1.0000	1.1056	9.553	1.2441	79	79.3	50.7	50.91	
	11:17	CON 1b [40]	0.0	428.409	46.134	382.276	1.1207	1.2391	19.293	1.2737					
	12:01	P/T 1b [40]	0.0	382.276	0.0	382.276	1.0000	1.2391	19.293	1.2737	39	40.0	38.0	38.30	
	12:11	P/T 1b [60]	0.0	382.276	0.0	382.276	1.0000	1.2391	19.293	1.2737	58	58.7	45.2	45.46	
	12:18	P/T 1b [80]	0.0	382.276	0.0	382.276	1.0000	1.2391	19.293	1.2737	79	79.3	51.3	51.50	
	12:22	CON 1c [40]	0.0	382.276	43.924	338.352	1.1298	1.3999	28.566	1.3094					
	13:03	P/T 1c [40]	0.0	338.352	0.0	338.352	1.0000	1.3999	28.566	1.3094	39	40.0	38.5	38.75	
	13:15	P/T 1c [60]	0.0	338.352	0.0	338.352	1.0000	1.3999	28.566	1.3094	59	59.7	46.0	46.26	
	13:21	P/T 1c [80]	0.0	338.352	0.0	338.352	1.0000	1.3999	28.566	1.3094	79	79.3	51.8	52.00	
	13:50	SAMPLE CON 1	12.221	326.131	0.0	326.131	1.0000	1.3999	28.566	1.3094					

Table B-2. Revised Tank 241-AP-107 Boildown Hot Cell Data Log. (3 sheets)

D _{H2O}										0.998595					
2011		Action	Sample Rmvd (mL)	Pot Vol Initial (mL)	Δ H ₂ O (mL)	Pot Vol Final (mL)	CR Step	CR Total	%WVR	D _{Model}	P _{Read}	P _{Corr}	T _{Read}	T _{Corr}	
Date mm/dd	Time hh:mm									(g/mL)	(Torr)	(Torr)	(°C)	(°C)	
10/04	07:30	P/T 1d [40]	0.0	326.131	0.0	326.131	1.0000	1.3999	28.566	1.3094	38	39.0	37.7	38.01	
	07:45	P/T 1d [60]	0.0	326.131	0.0	326.131	1.0000	1.3999	28.566	1.3094	58	58.7	45.7	45.96	
	07:55	P/T 1d [80]	0.0	326.131	0.0	326.131	1.0000	1.3999	28.566	1.3094	79	79.3	51.8	52.00	
	07:58	CON 2 [80]	0.0	326.131	42.598	283.533	1.1502	1.6102	37.897	1.3561					
	08:40	P/T 2 [80]	0.0	283.533	0.0	283.533	1.0000	1.6102	37.897	1.3561	79	79.3	52.8	52.98	
	08:44	P/T 2 [60]	0.0	283.533	0.0	283.533	1.0000	1.6102	37.897	1.3561	60	60.7	47.2	47.45	
	08:48	P/T 2 [40]	0.0	283.533	0.0	283.533	1.0000	1.6102	37.897	1.3561	39	40.0	39.3	39.60	
	09:15	SAMPLE CON 2	12.995	270.538	0.0	270.538	1.0000	1.6102	37.897	1.3561					
	09:30	CON 3 [40]	0.0	270.538	21.417	249.121	1.0860	1.7487	42.813	1.3869					
	09:54	P/T 3 [40]	0.0	249.121	0.0	249.121	1.0000	1.7487	42.813	1.3869	39	40.0	40.0	40.30	
	10:02	P/T 3 [60]	0.0	249.121	0.0	249.121	1.0000	1.7487	42.813	1.3869	59	59.7	47.6	47.84	
	10:07	P/T 3 [80]	0.0	249.121	0.0	249.121	1.0000	1.7487	42.813	1.3869	79	79.3	53.4	53.57	
	10:30	SAMPLE CON 3	11.745	237.376	0.0	237.376	1.0000	1.7487	42.813	1.3869					
	10:45	CON 4 [80]	0.0	237.376	18.743	218.633	1.0857	1.8986	47.329	1.4202					
	11:16	P/T 4 [80]	0.0	218.633	0.0	218.633	1.0000	1.8986	47.329	1.4202	79	79.3	54.4	54.56	
	11:20	P/T 4 [60]	0.0	218.633	0.0	218.633	1.0000	1.8986	47.329	1.4202	60	60.7	48.9	49.08	
	11:28	P/T 4 [40]	0.0	218.633	0.0	218.633	1.0000	1.8986	47.329	1.4202	40	41.0	41.2	41.49	
	11:36	SAMPLE CON 4	11.969	206.664	0.0	206.664	1.0000	1.8986	47.329	1.4202					
	11:50	CON 5 [40]	0.0	206.664	16.679	189.984	1.0878	2.0652	51.580	1.4572					
	12:15	P/T 5 [40]	0.0	189.984	0.0	189.984	1.0000	2.0652	51.580	1.4572	40	41.0	42.3	42.58	
	12:20	P/T 5 [60]	0.0	189.984	0.0	189.984	1.0000	2.0652	51.580	1.4572	60	60.7	49.7	49.92	
	12:23	P/T 5 [80]	0.0	189.984	0.0	189.984	1.0000	2.0652	51.580	1.4572	79	79.3	55.2	55.35	
	12:35	SAMPLE CON 5	12.428	177.556	0.0	177.556	1.0000	2.0652	51.580	1.4572					

Table B-2. Revised Tank 241-AP-107 Boildown Hot Cell Data Log. (3 sheets)

2011		Action	Sample Rmvd (mL)	Pot Vol Initial (mL)	Δ H ₂ O (mL)	Pot Vol Final (mL)	CR Step	CR Total	%WVR	D _{H2O}	D _{Model} (g/mL)	P _{Read} (Torr)	P _{Corr} (Torr)	T _{Read} (°C)	T _{Corr} (°C)
Date mm/dd	Time hh:mm									0.998595					
10/04	13:00	CON 6 [80]	0.0	177.556	15.083	162.473	1.0928	2.2570	55.693	1.4998					
	13:36	P/T 6 [80]	0.0	162.473	0.0	162.473	1.0000	2.2570	55.693	1.4998	79	79.3	56.7	56.83	
	13:40	P/T 6 [60]	0.0	162.473	0.0	162.473	1.0000	2.2570	55.693	1.4998	59	59.7	50.6	50.81	
	13:44	P/T 6 [40]	0.0	162.473	0.0	162.473	1.0000	2.2570	55.693	1.4998	39	40.0	42.7	42.98	
	14:02	SAMPLE CON 6	12.271	150.202	0.0	150.202	1.0000	2.2570	55.693	1.4998					
	14:18	CON 7 [40]	0.0	150.202	11.622	138.580	1.0839	2.4463	59.121	1.5418					
	14:37	P/T 7 [40]	0.0	138.580	0.0	138.580	1.0000	2.4463	59.121	1.5418	39	40.0	43.1	43.38	
	14:41	P/T 7 [60]	0.0	138.580	0.0	138.580	1.0000	2.4463	59.121	1.5418	60	60.7	51.3	51.50	
	14:47	P/T 7 [80]	0.0	138.580	0.0	138.580	1.0000	2.4463	59.121	1.5418	80	80.3	57.0	57.12	
	15:04	SAMPLE CON 7	13.386	125.194	0.0	125.194	1.0000	2.4463	59.121	1.5418					
	15:34	DIL 1	0.0	125.194	-120.209	245.403	0.5102	1.2480	19.870	1.2757					
	16:00	P/T 8 [60]	0.0	245.403	0.0	245.403	1.0000	1.2480	19.870	1.2757	59	59.7	45.6	45.86	
	16:12	SAMPLE DIL 1	12.850	232.553	0.0	232.553	1.0000	1.2480	19.870	1.2757					
	16:40	DIL 2	0.0	232.553	-16.223	248.776	0.9348	1.1666	14.280	1.2576					
	16:59	P/T 9 [60]	0.0	248.776	0.0	248.776	1.0000	1.1666	14.280	1.2576	59	59.7	45.4	45.66	
	17:07	SAMPLE DIL 2	13.057	235.719	0.0	235.719	1.0000	1.1666	14.280	1.2576					
	17:37	DIL 3	0.0	235.719	-18.156	253.874	0.9285	1.0832	7.678	1.2391					
	17:54	P/T 10 [60]	0.0	253.874	0.0	253.874	1.0000	1.0832	7.678	1.2391	59	59.7	45.0	45.26	
	18:05	SAMPLE DIL 3	12.780	241.094	0.0	241.094	1.0000	1.0832	7.678	1.2391					

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LAB-RPT-12-00008, R0

REFERENCES

- SVF-1543, 2008, "CH2M HILL Spreadsheet Verification and Release Form," Rev. 0, CH2M HILL Hanford Group, Inc., Richland, Washington.
- RPP-38843, 2008, *Spreadsheet Description Document for Boildown Test Data Spreadsheet*, Rev. 0, CH2M HILL Hanford Group, Inc., Richland, Washington.

ATTACHMENT C

PHOTOGRAPHS OF BOILDOWN SAMPLES

Figure C-1. Sample CON 1 Collected after Concentration to 27.74 %WVR^{18 °C}. (enlarged ≈25%)

(a) <1 hr out of pot	(b) After 45 hrs at ≈25.7 °C	(c) After 6.8 days at 18 °C	(d) Centrifuged after 8.8 days at 18 °C

Figure C-2. Sample CON 2 Collected after Concentration to 36.88 %WVR^{18 °C}. (enlarged ≈25%)

(a) <1 hr out of pot	(b) After 26 hrs at ≈25.5 °C	(c) After 6.8 days at 18 °C	(d) Centrifuged after 8.8 days at 18 °C

Figure C-3. Sample CON 3 Collected after Concentration to 41.58 %WVR^{18 °C}. (enlarged ≈25%)

(a) <1 hr out of pot	(b) After 25 hrs at ≈25.6 °C	(c) After 6.8 days at 18 °C	(d) Centrifuged after 8.8 days at 18 °C

Figure C-4. Sample CON 4 Collected after Concentration to 46.05 %WVR^{18 °C}. (enlarged ≈25%)

(a) <1 hr out of pot	(b) After 24 hrs at ≈25.6 °C	(c) After 6.8 days at 18 °C	(d) Centrifuged after 8.8 days at 18 °C

Figure C-5. Sample CON 5 Collected after Concentration to 50.15 %WVR^{18 °C}. (enlarged ≈25%)

Figure C-6. Sample CON 6 Collected after Concentration to 54.04 %WVR^{18 °C}. (enlarged ≈25%)

(a) <1 hr out of pot	(b) After 21 hrs at ≈25.7 °C	(c) After 6.8 days at 18 °C	(d) Centrifuged after 8.8 days at 18 °C

Figure C-7. Sample CON 7 Collected after Concentration to 57.47 %WVR^{18 °C}. (enlarged ≈25%)

(a) <1 hr out of pot	(b) After 21 hrs at ≈25.6 °C	(c) After 6.8 days at 18 °C	(d) Centrifuged after 8.8 days at 18 °C

Figure C-8. Sample DIL 1 Collected after Dilution of End-point Slurry to 19.90 %WVR^{18 °C}. (enlarged ≈25%)

(a) <1 hr out of pot	(b) After 19 hrs at ≈25.7 °C	(c) After 6.8 days at 18 °C	(d) Centrifuged after 8.8 days at 18 °C

Figure C-9. Sample DIL 2 Collected after Dilution of End-point Slurry to 14.66 %WVR^{18 °C}. (enlarged ≈25%)

(a) <1 hr out of pot	(b) After 19 hrs at ≈25.7 °C	(c) After 6.8 days at 18 °C	(d) Centrifuged after 8.8 days at 18 °C

Figure C-10. Sample DIL 3 Collected after Dilution of End-point Slurry to 8.37 %WVR^{18 °C}. (enlarged ≈25%)

(a) <1 hr out of pot	(b) After 18 hrs at ≈25.6 °C	(c) After 6.8 days at 18 °C	(d) Centrifuged after 8.8 days at 18 °C

ATTACHMENT D

LABORATORY ANALYSIS RESULTS

ATTACHMENT D

LABORATORY ANALYSIS RESULTS

Transcripts of the data summary reports for the physicochemical analyses of the AP-107 boildown sample liquids are presented in this attachment. All analyses were performed at the 222-S Laboratory by ATL personnel. The following analyses were performed:

Component (Method)	222-S Procedure Specific Analytes
Specific gravity (SpG) (gravimetric)	LA-510-112, Rev/Mod I-0
Total inorganic carbon / total organic carbon (acid-sparging/persulfate-oxidation coulometry)	LA-342-100, Rev/Mod J-0
Anions (ion chromatography)	LA-533-115, Rev/Mod J-0 Br ⁻ , Cl ⁻ , F ⁻ , NO ₂ ⁻ , NO ₃ ⁻ , PO ₄ ³⁻ , and SO ₄ ²⁻ , and Small organic anions: formate, acetate, oxalate, and glycolate
Elemental components (inductively coupled plasma–atomic emission)	LA-505-161, Rev. J-0-A Priority analytes: Na, Al, Si, P, S, K, Ca, and Cr. All others reported as opportunistic analytes.
Hydroxide (potentiometric titration)	LA-211-102, Rev/Mod I-1
Total carbon (combustion-coulometry)	LA-344-105, Rev/Mod J-0

The following abbreviations are in the header for each data table:

Std %-Rec	Standard percent recovery
RPD	Relative recent difference
Spk %-Rec	Spike percent recovery
Det Limit	Minimum detection limit
Qual Flags	Quality assurance data qualifier codes

The quality assurance data ‘flags’ in the right-most column of the data summary tables are defined as follows:

J	Estimated
U	Less than detection limit

Table D-1. Tank 241-AP-107 Boildown Composite (Sample CON 0). (2 sheets)

Sample(s): S11T011048 and S11T011049 (Test Samples CON 0A and CON 0B)

Sample Group: 20111225

Sample #	Analyte	Unit	Std %-Rec	Blank	Result	Duplicate	Average	RPD	Spk %-Rec	Det Limit	Qual Flags
S11T011048	Fluoride	ug/mL	103	<1.61E-03	469	469	469	0.0575	104	1.61	
S11T011048	Glycolate	ug/mL	101	<9.37E-03	175	169	172	3.45	102	9.37	
S11T011048	Acetate	ug/mL	101	<6.04E-03	204	185	194	9.48	101	6.04	
S11T011048	Formate	ug/mL	101	<4.67E-03	651	637	644	2.26	101	4.67	
S11T011048	Chloride	ug/mL	101	0.0131	1.41E+03	1.39E+03	1.40E+03	1.36	99.8	9.98	
S11T011048	Nitrite	ug/mL	100	<0.0192	3.04E+04	3.02E+04	3.03E+04	0.456	101	192	
S11T011048	Sulfate	ug/mL	102	<0.0187	9.09E+03	9.08E+03	9.08E+03	0.0609	99.5	18.7	
S11T011048	Oxalate	ug/mL	102	<0.0231	1.62E+03	1.61E+03	1.61E+03	0.547	96.7	23.1	
S11T011048	Bromide	ug/mL	101	<0.0580	<58.0	<58.0	n/a	n/a	96.3	58.0	U
S11T011048	Nitrate	ug/mL	100	0.0709	1.19E+05	1.18E+05	1.18E+05	0.679	100	208	
S11T011048	Phosphate	ug/mL	101	<0.0167	2.91E+03	2.90E+03	2.90E+03	0.238	101	16.7	
S11T011048	Total organic carbon	ug/mL	97.2	<20.0	1.70E+03	1.06E+03	1.38E+03	46.4	85.8	400	J
S11T011048	Total inorganic carbon	ug/mL	97.9	<7.00	5.82E+03	5.81E+03	5.82E+03	0.172	96.6	140	
S11T011048	Total carbon	ug/mL	97.2	<5.00	7.22E+03	7.12E+03	7.17E+03	1.39	99.9	55.0	
S11T011049	Silver	ug/mL	98.6	n/a	<12.5	n/a	n/a	n/a	n/a	12.5	
S11T011049	Aluminum	ug/mL	101	<0.0300	4.38E+03	4.46E+03	4.42E+03	1.70	99.4	75.0	
S11T011049	Arsenic	ug/mL	100	n/a	<125	n/a	n/a	n/a	n/a	125	
S11T011049	Boron	ug/mL	99.3	n/a	<75.0	n/a	n/a	n/a	n/a	75.0	
S11T011049	Barium	ug/mL	101	n/a	<7.50	n/a	n/a	n/a	n/a	7.50	
S11T011049	Beryllium	ug/mL	98.9	n/a	<2.50	n/a	n/a	n/a	n/a	2.50	
S11T011049	Bismuth	ug/mL	100	n/a	<250	n/a	n/a	n/a	n/a	250	
S11T011049	Calcium	ug/mL	101	<0.0500	<125	<125	n/a	n/a	95.3	125	U
S11T011049	Cadmium	ug/mL	102	n/a	<12.5	n/a	n/a	n/a	n/a	12.5	
S11T011049	Cerium	ug/mL	101	n/a	<75.0	n/a	n/a	n/a	n/a	75.0	
S11T011049	Cobalt	ug/mL	101	n/a	<25.0	n/a	n/a	n/a	n/a	25.0	
S11T011049	Chromium	ug/mL	101	<5.00E-03	553	588	571	6.15	95.9	12.5	
S11T011049	Copper	ug/mL	102	n/a	<12.5	n/a	n/a	n/a	n/a	12.5	
S11T011049	Europium	ug/mL	95.4	n/a	<12.5	n/a	n/a	n/a	n/a	12.5	

Table D-1. Tank 241-AP-107 Boildown Composite (Sample CON 0). (2 sheets)

Sampl(s): S11T011048 and S11T011049 (Test Samples CON 0A and CON 0B)

Sample Group: 20111225

Sample #	Analyte	Unit	Std %-Rec	Blank	Result	Duplicate	Average	RPD	Spk %-Rec	Det Limit	Qual Flags
S11T011049	Iron	ug/mL	101	n/a	<12.5	n/a	n/a	n/a	n/a	12.5	
S11T011049	Potassium	ug/mL	92.3	<0.500	<1.25E+03	1.45E+03	1.35E+03	14.6	89.4	1.25E+03	U
S11T011049	Lanthanum	ug/mL	99.5	n/a	<7.50	n/a	n/a	n/a	n/a	7.50	
S11T011049	Lithium	ug/mL	109	n/a	<7.50	n/a	n/a	n/a	n/a	7.50	
S11T011049	Magnesium	ug/mL	102	n/a	<125	n/a	n/a	n/a	n/a	125	
S11T011049	Manganese	ug/mL	100	n/a	<7.50	n/a	n/a	n/a	n/a	7.50	
S11T011049	Molybdenum	ug/mL	98.8	n/a	<50.0	n/a	n/a	n/a	n/a	50.0	
S11T011049	Sodium	ug/mL	106	<0.100	1.07E+05	1.07E+05	1.07E+05	0.684	88.9	250	
S11T011049	Neodymium	ug/mL	100	n/a	<25.0	n/a	n/a	n/a	n/a	25.0	
S11T011049	Nickel	ug/mL	101	n/a	<50.0	n/a	n/a	n/a	n/a	50.0	
S11T011049	Phosphorus	ug/mL	102	<0.0500	973	884	929	9.68	96.3	125	J
S11T011049	Lead	ug/mL	104	n/a	<125	n/a	n/a	n/a	n/a	125	
S11T011049	Sulfur	ug/mL	98.4	<0.100	3.08E+03	3.13E+03	3.10E+03	1.38	98.5	250	
S11T011049	Antimony	ug/mL	94.6	n/a	<125	n/a	n/a	n/a	n/a	125	
S11T011049	Selenium	ug/mL	104	n/a	<250	n/a	n/a	n/a	n/a	250	
S11T011049	Silicon	ug/mL	94.7	<0.0300	92.3	125	108	29.7	93.3	75.0	J
S11T011049	Samarium	ug/mL	103	n/a	<125	n/a	n/a	n/a	n/a	125	
S11T011049	Strontium	ug/mL	101	n/a	<7.50	n/a	n/a	n/a	n/a	7.50	
S11T011049	Thorium	ug/mL	97.5	n/a	<125	n/a	n/a	n/a	n/a	125	
S11T011049	Titanium	ug/mL	97.3	n/a	<12.5	n/a	n/a	n/a	n/a	12.5	
S11T011049	Thallium	ug/mL	96.2	n/a	<250	n/a	n/a	n/a	n/a	250	
S11T011049	Uranium	ug/mL	101	n/a	<250	n/a	n/a	n/a	n/a	250	
S11T011049	Vanadium	ug/mL	101	n/a	<12.5	n/a	n/a	n/a	n/a	12.5	
S11T011049	Yttrium	ug/mL	94.7	n/a	<5.00	n/a	n/a	n/a	n/a	5.00	
S11T011049	Zinc	ug/mL	99.9	n/a	<12.5	n/a	n/a	n/a	n/a	12.5	
S11T011049	Zirconium	ug/mL	98.9	n/a	<12.5	n/a	n/a	n/a	n/a	12.5	
S11T011049	Hydroxide	ug/mL	105	<41.7	1.0E+04	1.03E+04	1.0E+04	2.09	103	550	
S11T011049	Specific gravity	unitless	100.5	n/a	1.225	1.261	1.243	2.896	n/a	1.00E-03	

Table D-2. Centrifuged Liquid from Sample CON 1. (2 sheets)

Sample(s): S11T011062 (Test Sample CON 1)

Sample Group: 20111225

Sample #	Analyte	Unit	Std %-Rec	Blank	Result	Duplicate	Average	RPD	Spk %-Rec	Det Limit	Qual Flags
S11T011062	Fluoride	ug/mL	103	<1.61E-03	533	n/a	n/a	n/a	n/a	1.61	
S11T011062	Glycolate	ug/mL	101	<9.37E-03	253	n/a	n/a	n/a	n/a	9.37	
S11T011062	Acetate	ug/mL	101	<6.04E-03	289	n/a	n/a	n/a	n/a	6.04	
S11T011062	Formate	ug/mL	101	<4.67E-03	889	n/a	n/a	n/a	n/a	4.67	
S11T011062	Chloride	ug/mL	101	0.0131	1.92E+03	n/a	n/a	n/a	n/a	9.98	
S11T011062	Nitrite	ug/mL	100	<0.0192	4.15E+04	n/a	n/a	n/a	n/a	96.0	
S11T011062	Sulfate	ug/mL	102	<0.0187	1.25E+04	n/a	n/a	n/a	n/a	18.7	
S11T011062	Oxalate	ug/mL	102	<0.0231	699	n/a	n/a	n/a	n/a	23.1	
S11T011062	Bromide	ug/mL	101	<0.0580	<58.0	n/a	n/a	n/a	n/a	58.0	U
S11T011062	Nitrate	ug/mL	101	0.0680	1.62E+05	n/a	n/a	n/a	n/a	208	
S11T011062	Phosphate	ug/mL	101	<0.0167	2.91E+03	n/a	n/a	n/a	n/a	16.7	
S11T011062	Silver	ug/mL	98.6	n/a	<12.5	n/a	n/a	n/a	n/a	12.5	
S11T011062	Aluminum	ug/mL	101	<0.0300	6.29E+03	n/a	n/a	n/a	n/a	75.0	
S11T011062	Arsenic	ug/mL	100	n/a	<125	n/a	n/a	n/a	n/a	125	
S11T011062	Boron	ug/mL	99.3	n/a	<75.0	n/a	n/a	n/a	n/a	75.0	
S11T011062	Barium	ug/mL	101	n/a	<7.50	n/a	n/a	n/a	n/a	7.50	
S11T011062	Beryllium	ug/mL	98.9	n/a	<2.50	n/a	n/a	n/a	n/a	2.50	
S11T011062	Bismuth	ug/mL	100	n/a	<250	n/a	n/a	n/a	n/a	250	
S11T011062	Calcium	ug/mL	101	<0.0500	<125	n/a	n/a	n/a	n/a	125	U
S11T011062	Cadmium	ug/mL	102	n/a	<12.5	n/a	n/a	n/a	n/a	12.5	
S11T011062	Cerium	ug/mL	101	n/a	<75.0	n/a	n/a	n/a	n/a	75.0	
S11T011062	Cobalt	ug/mL	101	n/a	<25.0	n/a	n/a	n/a	n/a	25.0	
S11T011062	Chromium	ug/mL	101	<5.00E-03	833	n/a	n/a	n/a	n/a	12.5	
S11T011062	Copper	ug/mL	102	n/a	<12.5	n/a	n/a	n/a	n/a	12.5	
S11T011062	Europium	ug/mL	95.4	n/a	<12.5	n/a	n/a	n/a	n/a	12.5	
S11T011062	Iron	ug/mL	101	n/a	<12.5	n/a	n/a	n/a	n/a	12.5	
S11T011062	Potassium	ug/mL	92.3	<0.500	2.17E+03	n/a	n/a	n/a	n/a	1.25E+03	J
S11T011062	Lanthanum	ug/mL	99.5	n/a	<7.50	n/a	n/a	n/a	n/a	7.50	

Table D-2. Centrifuged Liquid from Sample CON 1. (2 sheets)

Sample Number(s): S11T011062 (Test Sample CON 1)

Sample Group: 20111225

Sample #	Analyte	Unit	Std %-Rec	Blank	Result	Duplicate	Average	RPD	Spk %-Rec	Det Limit	Qual Flags
S11T011062	Lithium	ug/mL	109	n/a	<7.50	n/a	n/a	n/a	n/a	7.50	
S11T011062	Magnesium	ug/mL	102	n/a	<125	n/a	n/a	n/a	n/a	125	
S11T011062	Manganese	ug/mL	100	n/a	<7.50	n/a	n/a	n/a	n/a	7.50	
S11T011062	Molybdenum	ug/mL	98.8	n/a	61.6	n/a	n/a	n/a	n/a	50.0	
S11T011062	Sodium	ug/mL	106	<0.100	1.44E+05	n/a	n/a	n/a	n/a	250	
S11T011062	Neodymium	ug/mL	100	n/a	<25.0	n/a	n/a	n/a	n/a	25.0	
S11T011062	Nickel	ug/mL	101	n/a	<50.0	n/a	n/a	n/a	n/a	50.0	
S11T011062	Phosphorus	ug/mL	102	<0.0500	939	n/a	n/a	n/a	n/a	125	J
S11T011062	Lead	ug/mL	104	n/a	<125	n/a	n/a	n/a	n/a	125	
S11T011062	Sulfur	ug/mL	98.4	<0.100	4.37E+03	n/a	n/a	n/a	n/a	250	
S11T011062	Antimony	ug/mL	94.6	n/a	<125	n/a	n/a	n/a	n/a	125	
S11T011062	Selenium	ug/mL	104	n/a	<250	n/a	n/a	n/a	n/a	250	
S11T011062	Silicon	ug/mL	94.7	<0.0300	355	n/a	n/a	n/a	n/a	75.0	J
S11T011062	Samarium	ug/mL	103	n/a	<125	n/a	n/a	n/a	n/a	125	
S11T011062	Strontium	ug/mL	101	n/a	<7.50	n/a	n/a	n/a	n/a	7.50	
S11T011062	Thorium	ug/mL	97.5	n/a	<125	n/a	n/a	n/a	n/a	125	
S11T011062	Titanium	ug/mL	97.3	n/a	<12.5	n/a	n/a	n/a	n/a	12.5	
S11T011062	Thallium	ug/mL	96.2	n/a	<250	n/a	n/a	n/a	n/a	250	
S11T011062	Uranium	ug/mL	101	n/a	<250	n/a	n/a	n/a	n/a	250	
S11T011062	Vanadium	ug/mL	101	n/a	<12.5	n/a	n/a	n/a	n/a	12.5	
S11T011062	Yttrium	ug/mL	94.7	n/a	<5.00	n/a	n/a	n/a	n/a	5.00	
S11T011062	Zinc	ug/mL	99.9	n/a	<12.5	n/a	n/a	n/a	n/a	12.5	
S11T011062	Zirconium	ug/mL	98.9	n/a	<12.5	n/a	n/a	n/a	n/a	12.5	
S11T011062	Hydroxide	ug/mL	105	<41.7	1.4E+04	n/a	n/a	n/a	n/a	550	
S11T011062	Specific gravity	unitless	100.5	n/a	1.319	n/a	n/a	n/a	n/a	1.00E-03	
S11T011062	Total organic carbon	ug/mL	97.9	<20.0	982	n/a	n/a	n/a	n/a	100	J
S11T011062	Total inorganic carbon	ug/mL	80.0	<7.00	7.35E+03	n/a	n/a	n/a	n/a	35.0	

Table D-3. Centrifuged Liquid from Sample CON 2. (2 sheets)

Sample Number(s): S11T011063 (Test Sample CON 2)

Sample Group: 20111225

Sample #	Analyte	Unit	Std %-Rec	Blank	Result	Duplicate	Average	RPD	Spk %-Rec	Det Limit	Qual Flags
S11T011063	Fluoride	ug/mL	103	<1.61E-03	488	n/a	n/a	n/a	n/a	1.61	
S11T011063	Glycolate	ug/mL	101	<9.37E-03	273	n/a	n/a	n/a	n/a	9.37	
S11T011063	Acetate	ug/mL	101	<6.04E-03	296	n/a	n/a	n/a	n/a	6.04	
S11T011063	Formate	ug/mL	101	<4.67E-03	994	n/a	n/a	n/a	n/a	4.67	
S11T011063	Chloride	ug/mL	101	0.0131	2.18E+03	n/a	n/a	n/a	n/a	9.98	
S11T011063	Nitrite	ug/mL	101	<0.0192	4.72E+04	n/a	n/a	n/a	n/a	96.0	
S11T011063	Sulfate	ug/mL	102	<0.0187	1.40E+04	n/a	n/a	n/a	n/a	18.7	
S11T011063	Oxalate	ug/mL	102	<0.0231	577	n/a	n/a	n/a	n/a	23.1	
S11T011063	Bromide	ug/mL	101	<0.0580	<58.0	n/a	n/a	n/a	n/a	58.0	U
S11T011063	Nitrate	ug/mL	100	0.0709	1.85E+05	n/a	n/a	n/a	n/a	208	
S11T011063	Phosphate	ug/mL	101	<0.0167	2.18E+03	n/a	n/a	n/a	n/a	16.7	
S11T011063	Silver	ug/mL	98.6	n/a	<12.5	n/a	n/a	n/a	n/a	12.5	
S11T011063	Aluminum	ug/mL	101	<0.0300	7.25E+03	n/a	n/a	n/a	n/a	75.0	
S11T011063	Arsenic	ug/mL	100	n/a	<125	n/a	n/a	n/a	n/a	125	
S11T011063	Boron	ug/mL	99.3	n/a	<75.0	n/a	n/a	n/a	n/a	75.0	
S11T011063	Barium	ug/mL	101	n/a	<7.50	n/a	n/a	n/a	n/a	7.50	
S11T011063	Beryllium	ug/mL	98.9	n/a	<2.50	n/a	n/a	n/a	n/a	2.50	
S11T011063	Bismuth	ug/mL	100	n/a	<250	n/a	n/a	n/a	n/a	250	
S11T011063	Calcium	ug/mL	101	<0.0500	<125	n/a	n/a	n/a	n/a	125	U
S11T011063	Cadmium	ug/mL	102	n/a	<12.5	n/a	n/a	n/a	n/a	12.5	
S11T011063	Cerium	ug/mL	101	n/a	<75.0	n/a	n/a	n/a	n/a	75.0	
S11T011063	Cobalt	ug/mL	101	n/a	<25.0	n/a	n/a	n/a	n/a	25.0	
S11T011063	Chromium	ug/mL	101	<5.00E-03	959	n/a	n/a	n/a	n/a	12.5	
S11T011063	Copper	ug/mL	102	n/a	<12.5	n/a	n/a	n/a	n/a	12.5	
S11T011063	Europium	ug/mL	95.4	n/a	<12.5	n/a	n/a	n/a	n/a	12.5	
S11T011063	Iron	ug/mL	101	n/a	<12.5	n/a	n/a	n/a	n/a	12.5	
S11T011063	Potassium	ug/mL	92.3	<0.500	2.06E+03	n/a	n/a	n/a	n/a	1.25E+03	J
S11T011063	Lanthanum	ug/mL	99.5	n/a	<7.50	n/a	n/a	n/a	n/a	7.50	

Table D-3. Centrifuged Liquid from Sample CON 2. (2 sheets)

Sample Number(s): S11T011063 (Test Sample CON 2)

Sample Group: 20111225

Sample #	Analyte	Unit	Std %-Rec	Blank	Result	Duplicate	Average	RPD	Spk %-Rec	Det Limit	Qual Flags
S11T011063	Lithium	ug/mL	109	n/a	<7.50	n/a	n/a	n/a	n/a	7.50	
S11T011063	Magnesium	ug/mL	102	n/a	<125	n/a	n/a	n/a	n/a	125	
S11T011063	Manganese	ug/mL	100	n/a	<7.50	n/a	n/a	n/a	n/a	7.50	
S11T011063	Molybdenum	ug/mL	98.8	n/a	55.8	n/a	n/a	n/a	n/a	50.0	
S11T011063	Sodium	ug/mL	106	<0.100	1.67E+05	n/a	n/a	n/a	n/a	250	
S11T011063	Neodymium	ug/mL	100	n/a	<25.0	n/a	n/a	n/a	n/a	25.0	
S11T011063	Nickel	ug/mL	101	n/a	<50.0	n/a	n/a	n/a	n/a	50.0	
S11T011063	Phosphorus	ug/mL	102	<0.0500	817	n/a	n/a	n/a	n/a	125	J
S11T011063	Lead	ug/mL	104	n/a	<125	n/a	n/a	n/a	n/a	125	
S11T011063	Sulfur	ug/mL	98.4	<0.100	5.21E+03	n/a	n/a	n/a	n/a	250	
S11T011063	Antimony	ug/mL	94.6	n/a	<125	n/a	n/a	n/a	n/a	125	
S11T011063	Selenium	ug/mL	104	n/a	<250	n/a	n/a	n/a	n/a	250	
S11T011063	Silicon	ug/mL	94.7	<0.0300	182	n/a	n/a	n/a	n/a	75.0	J
S11T011063	Samarium	ug/mL	103	n/a	<125	n/a	n/a	n/a	n/a	125	
S11T011063	Strontium	ug/mL	101	n/a	<7.50	n/a	n/a	n/a	n/a	7.50	
S11T011063	Thorium	ug/mL	97.5	n/a	<125	n/a	n/a	n/a	n/a	125	
S11T011063	Titanium	ug/mL	97.3	n/a	<12.5	n/a	n/a	n/a	n/a	12.5	
S11T011063	Thallium	ug/mL	96.2	n/a	<250	n/a	n/a	n/a	n/a	250	
S11T011063	Uranium	ug/mL	101	n/a	<250	n/a	n/a	n/a	n/a	250	
S11T011063	Vanadium	ug/mL	101	n/a	<12.5	n/a	n/a	n/a	n/a	12.5	
S11T011063	Yttrium	ug/mL	94.7	n/a	<5.00	n/a	n/a	n/a	n/a	5.00	
S11T011063	Zinc	ug/mL	99.9	n/a	<12.5	n/a	n/a	n/a	n/a	12.5	
S11T011063	Zirconium	ug/mL	98.9	n/a	<12.5	n/a	n/a	n/a	n/a	12.5	
S11T011063	Hydroxide	ug/mL	105	<41.7	1.6E+04	n/a	n/a	n/a	n/a	550	
S11T011063	Specific gravity	unitless	100.5	n/a	1.386	n/a	n/a	n/a	n/a	1.00E-03	
S11T011063	Total organic carbon	ug/mL	97.9	<20.0	1.04E+03	1.05E+03	1.04E+03	0.957	101	200	J
S11T011063	Total inorganic carbon	ug/mL	80.0	<7.00	8.49E+03	8.51E+03	8.50E+03	0.235	111	7.00	

Table D-4. Centrifuged Liquid from Sample CON 3. (2 sheets)

Sample Number(s): S11T011064 (Test Sample CON 3)

Sample Group: 20111225

Sample #	Analyte	Unit	Std %-Rec	Blank	Result	Duplicate	Average	RPD	Spk %-Rec	Det Limit	Qual Flags
S11T011064	Fluoride	ug/mL	103	<1.61E-03	337	n/a	n/a	n/a	n/a	1.61	
S11T011064	Glycolate	ug/mL	101	<9.37E-03	312	n/a	n/a	n/a	n/a	9.37	
S11T011064	Acetate	ug/mL	101	<6.04E-03	351	n/a	n/a	n/a	n/a	6.04	
S11T011064	Formate	ug/mL	101	<4.67E-03	1.08E+03	n/a	n/a	n/a	n/a	4.67	
S11T011064	Chloride	ug/mL	101	0.0131	2.31E+03	n/a	n/a	n/a	n/a	9.98	
S11T011064	Nitrite	ug/mL	101	<0.0192	5.00E+04	n/a	n/a	n/a	n/a	96.0	
S11T011064	Sulfate	ug/mL	102	<0.0187	1.42E+04	n/a	n/a	n/a	n/a	18.7	
S11T011064	Oxalate	ug/mL	102	<0.0231	406	n/a	n/a	n/a	n/a	23.1	
S11T011064	Bromide	ug/mL	101	<0.0580	<58.0	n/a	n/a	n/a	n/a	58.0	U
S11T011064	Nitrate	ug/mL	100	0.0709	1.97E+05	n/a	n/a	n/a	n/a	208	
S11T011064	Phosphate	ug/mL	101	<0.0167	2.12E+03	n/a	n/a	n/a	n/a	16.7	
S11T011064	Silver	ug/mL	98.6	n/a	<12.5	n/a	n/a	n/a	n/a	12.5	
S11T011064	Aluminum	ug/mL	101	<0.0300	8.10E+03	n/a	n/a	n/a	n/a	75.0	
S11T011064	Arsenic	ug/mL	100	n/a	<125	n/a	n/a	n/a	n/a	125	
S11T011064	Boron	ug/mL	99.3	n/a	<75.0	n/a	n/a	n/a	n/a	75.0	
S11T011064	Barium	ug/mL	101	n/a	<7.50	n/a	n/a	n/a	n/a	7.50	
S11T011064	Beryllium	ug/mL	98.9	n/a	<2.50	n/a	n/a	n/a	n/a	2.50	
S11T011064	Bismuth	ug/mL	100	n/a	<250	n/a	n/a	n/a	n/a	250	
S11T011064	Calcium	ug/mL	101	<0.0500	<125	n/a	n/a	n/a	n/a	125	U
S11T011064	Cadmium	ug/mL	102	n/a	<12.5	n/a	n/a	n/a	n/a	12.5	
S11T011064	Cerium	ug/mL	101	n/a	<75.0	n/a	n/a	n/a	n/a	75.0	
S11T011064	Cobalt	ug/mL	101	n/a	<25.0	n/a	n/a	n/a	n/a	25.0	
S11T011064	Chromium	ug/mL	101	<5.00E-03	1.08E+03	n/a	n/a	n/a	n/a	12.5	
S11T011064	Copper	ug/mL	102	n/a	<12.5	n/a	n/a	n/a	n/a	12.5	
S11T011064	Europium	ug/mL	95.4	n/a	<12.5	n/a	n/a	n/a	n/a	12.5	
S11T011064	Iron	ug/mL	101	n/a	<12.5	n/a	n/a	n/a	n/a	12.5	
S11T011064	Potassium	ug/mL	92.3	<0.500	2.85E+03	n/a	n/a	n/a	n/a	1.25E+03	J
S11T011064	Lanthanum	ug/mL	99.5	n/a	<7.50	n/a	n/a	n/a	n/a	7.50	

Table D-4. Centrifuged Liquid from Sample CON 3. (2 sheets)

Sample Number(s): S11T011064 (Test Sample CON 3)

Sample Group: 20111225

Sample #	Analyte	Unit	Std %-Rec	Blank	Result	Duplicate	Average	RPD	Spk %-Rec	Det Limit	Qual Flags
S11T011064	Lithium	ug/mL	109	n/a	<7.50	n/a	n/a	n/a	n/a	7.50	
S11T011064	Magnesium	ug/mL	102	n/a	<125	n/a	n/a	n/a	n/a	125	
S11T011064	Manganese	ug/mL	100	n/a	<7.50	n/a	n/a	n/a	n/a	7.50	
S11T011064	Molybdenum	ug/mL	98.8	n/a	64.2	n/a	n/a	n/a	n/a	50.0	
S11T011064	Sodium	ug/mL	106	<0.100	1.82E+05	n/a	n/a	n/a	n/a	250	
S11T011064	Neodymium	ug/mL	100	n/a	<25.0	n/a	n/a	n/a	n/a	25.0	
S11T011064	Nickel	ug/mL	101	n/a	<50.0	n/a	n/a	n/a	n/a	50.0	
S11T011064	Phosphorus	ug/mL	102	<0.0500	676	n/a	n/a	n/a	n/a	125	J
S11T011064	Lead	ug/mL	104	n/a	<125	n/a	n/a	n/a	n/a	125	
S11T011064	Sulfur	ug/mL	98.4	<0.100	5.43E+03	n/a	n/a	n/a	n/a	250	
S11T011064	Antimony	ug/mL	94.6	n/a	<125	n/a	n/a	n/a	n/a	125	
S11T011064	Selenium	ug/mL	104	n/a	<250	n/a	n/a	n/a	n/a	250	
S11T011064	Silicon	ug/mL	94.7	<0.0300	263	n/a	n/a	n/a	n/a	75.0	J
S11T011064	Samarium	ug/mL	103	n/a	<125	n/a	n/a	n/a	n/a	125	
S11T011064	Strontium	ug/mL	101	n/a	<7.50	n/a	n/a	n/a	n/a	7.50	
S11T011064	Thorium	ug/mL	97.5	n/a	<125	n/a	n/a	n/a	n/a	125	
S11T011064	Titanium	ug/mL	97.3	n/a	<12.5	n/a	n/a	n/a	n/a	12.5	
S11T011064	Thallium	ug/mL	96.2	n/a	<250	n/a	n/a	n/a	n/a	250	
S11T011064	Uranium	ug/mL	101	n/a	<250	n/a	n/a	n/a	n/a	250	
S11T011064	Vanadium	ug/mL	101	n/a	<12.5	n/a	n/a	n/a	n/a	12.5	
S11T011064	Yttrium	ug/mL	94.7	n/a	<5.00	n/a	n/a	n/a	n/a	5.00	
S11T011064	Zinc	ug/mL	99.9	n/a	<12.5	n/a	n/a	n/a	n/a	12.5	
S11T011064	Zirconium	ug/mL	98.9	n/a	<12.5	n/a	n/a	n/a	n/a	12.5	
S11T011064	Hydroxide	ug/mL	105	<41.7	1.8E+04	n/a	n/a	n/a	n/a	550	
S11T011064	Specific gravity	unitless	100.5	n/a	1.313	n/a	n/a	n/a	n/a	1.00E-03	
S11T011064	Total organic carbon	ug/mL	97.9	<20.0	1.11E+03	n/a	n/a	n/a	n/a	200	J
S11T011064	Total inorganic carbon	ug/mL	80.0	<7.00	6.68E+03	n/a	n/a	n/a	n/a	70.0	

Table D-5. Centrifuged Liquid from Sample CON 4. (2 sheets)

Sample Number(s): S11T011065 (Test Sample CON 4)

Sample Group: 20111225

Sample #	Analyte	Unit	Std %-Rec	Blank	Result	Duplicate	Average	RPD	Spk %-Rec	Det Limit	Qual Flags
S11T011065	Fluoride	ug/mL	103	<1.61E-03	376	n/a	n/a	n/a	n/a	1.61	
S11T011065	Glycolate	ug/mL	101	<9.37E-03	331	n/a	n/a	n/a	n/a	9.37	
S11T011065	Acetate	ug/mL	101	<6.04E-03	373	n/a	n/a	n/a	n/a	6.04	
S11T011065	Formate	ug/mL	101	<4.67E-03	1.15E+03	n/a	n/a	n/a	n/a	4.67	
S11T011065	Chloride	ug/mL	101	0.0131	2.45E+03	n/a	n/a	n/a	n/a	9.98	
S11T011065	Nitrite	ug/mL	101	<0.0192	5.32E+04	n/a	n/a	n/a	n/a	96.0	
S11T011065	Sulfate	ug/mL	102	<0.0187	1.57E+04	n/a	n/a	n/a	n/a	18.7	
S11T011065	Oxalate	ug/mL	102	<0.0231	365	n/a	n/a	n/a	n/a	23.1	
S11T011065	Bromide	ug/mL	101	<0.0580	<58.0	n/a	n/a	n/a	n/a	58.0	U
S11T011065	Nitrate	ug/mL	100	0.0709	2.11E+05	n/a	n/a	n/a	n/a	208	
S11T011065	Phosphate	ug/mL	101	<0.0167	1.25E+03	n/a	n/a	n/a	n/a	16.7	
S11T011065	Silver	ug/mL	98.6	n/a	<12.5	n/a	n/a	n/a	n/a	12.5	
S11T011065	Aluminum	ug/mL	101	<0.0300	8.37E+03	n/a	n/a	n/a	n/a	75.0	
S11T011065	Arsenic	ug/mL	100	n/a	<125	n/a	n/a	n/a	n/a	125	
S11T011065	Boron	ug/mL	99.3	n/a	<75.0	n/a	n/a	n/a	n/a	75.0	
S11T011065	Barium	ug/mL	101	n/a	<7.50	n/a	n/a	n/a	n/a	7.50	
S11T011065	Beryllium	ug/mL	98.9	n/a	<2.50	n/a	n/a	n/a	n/a	2.50	
S11T011065	Bismuth	ug/mL	100	n/a	<250	n/a	n/a	n/a	n/a	250	
S11T011065	Calcium	ug/mL	101	<0.0500	<125	n/a	n/a	n/a	n/a	125	U
S11T011065	Cadmium	ug/mL	102	n/a	<12.5	n/a	n/a	n/a	n/a	12.5	
S11T011065	Cerium	ug/mL	101	n/a	<75.0	n/a	n/a	n/a	n/a	75.0	
S11T011065	Cobalt	ug/mL	101	n/a	<25.0	n/a	n/a	n/a	n/a	25.0	
S11T011065	Chromium	ug/mL	101	<5.00E-03	1.11E+03	n/a	n/a	n/a	n/a	12.5	
S11T011065	Copper	ug/mL	102	n/a	<12.5	n/a	n/a	n/a	n/a	12.5	
S11T011065	Europium	ug/mL	95.4	n/a	<12.5	n/a	n/a	n/a	n/a	12.5	
S11T011065	Iron	ug/mL	101	n/a	<12.5	n/a	n/a	n/a	n/a	12.5	
S11T011065	Potassium	ug/mL	92.3	<0.500	2.85E+03	n/a	n/a	n/a	n/a	1.25E+03	J
S11T011065	Lanthanum	ug/mL	99.5	n/a	<7.50	n/a	n/a	n/a	n/a	7.50	

Table D-5. Centrifuged Liquid from Sample CON 4. (2 sheets)

Sample Number(s): S11T011065 (Test Sample CON 4)

Sample Group: 20111225

Sample #	Analyte	Unit	Std %-Rec	Blank	Result	Duplicate	Average	RPD	Spk %-Rec	Det Limit	Qual Flags
S11T011065	Lithium	ug/mL	109	n/a	<7.50	n/a	n/a	n/a	n/a	7.50	
S11T011065	Magnesium	ug/mL	102	n/a	<125	n/a	n/a	n/a	n/a	125	
S11T011065	Manganese	ug/mL	100	n/a	<7.50	n/a	n/a	n/a	n/a	7.50	
S11T011065	Molybdenum	ug/mL	98.8	n/a	54.1	n/a	n/a	n/a	n/a	50.0	
S11T011065	Sodium	ug/mL	106	<0.100	1.87E+05	n/a	n/a	n/a	n/a	250	
S11T011065	Neodymium	ug/mL	100	n/a	<25.0	n/a	n/a	n/a	n/a	25.0	
S11T011065	Nickel	ug/mL	101	n/a	<50.0	n/a	n/a	n/a	n/a	50.0	
S11T011065	Phosphorus	ug/mL	102	<0.0500	472	n/a	n/a	n/a	n/a	125	J
S11T011065	Lead	ug/mL	104	n/a	<125	n/a	n/a	n/a	n/a	125	
S11T011065	Sulfur	ug/mL	98.4	<0.100	5.79E+03	n/a	n/a	n/a	n/a	250	
S11T011065	Antimony	ug/mL	94.6	n/a	<125	n/a	n/a	n/a	n/a	125	
S11T011065	Selenium	ug/mL	104	n/a	<250	n/a	n/a	n/a	n/a	250	
S11T011065	Silicon	ug/mL	94.7	<0.0300	183	n/a	n/a	n/a	n/a	75.0	J
S11T011065	Samarium	ug/mL	103	n/a	<125	n/a	n/a	n/a	n/a	125	
S11T011065	Strontium	ug/mL	101	n/a	<7.50	n/a	n/a	n/a	n/a	7.50	
S11T011065	Thorium	ug/mL	97.5	n/a	<125	n/a	n/a	n/a	n/a	125	
S11T011065	Titanium	ug/mL	97.3	n/a	<12.5	n/a	n/a	n/a	n/a	12.5	
S11T011065	Thallium	ug/mL	96.2	n/a	<250	n/a	n/a	n/a	n/a	250	
S11T011065	Uranium	ug/mL	101	n/a	<250	n/a	n/a	n/a	n/a	250	
S11T011065	Vanadium	ug/mL	101	n/a	<12.5	n/a	n/a	n/a	n/a	12.5	
S11T011065	Yttrium	ug/mL	94.7	n/a	<5.00	n/a	n/a	n/a	n/a	5.00	
S11T011065	Zinc	ug/mL	99.9	n/a	<12.5	n/a	n/a	n/a	n/a	12.5	
S11T011065	Zirconium	ug/mL	98.9	n/a	<12.5	n/a	n/a	n/a	n/a	12.5	
S11T011065	Hydroxide	ug/mL	105	<41.7	1.9E+04	n/a	n/a	n/a	n/a	550	
S11T011065	Specific gravity	unitless	100.5	n/a	1.332	n/a	n/a	n/a	n/a	1.00E-03	
S11T011065	Total organic carbon	ug/mL	97.9	<20.0	1.15E+03	n/a	n/a	n/a	n/a	200	J
S11T011065	Total inorganic carbon	ug/mL	80.0	<7.00	1.03E+04	n/a	n/a	n/a	n/a	7.00	

Table D-6. Centrifuged Liquid from Sample CON 5. (2 sheets)

Sample Number(s): S11T011066 (Test Sample CON 5)

Sample Group: 20111225

Sample #	Analyte	Unit	Std %-Rec	Blank	Result	Duplicate	Average	RPD	Spk %-Rec	Det Limit	Qual Flags
S11T011066	Fluoride	ug/mL	103	<1.61E-03	73.1	n/a	n/a	n/a	n/a	1.61	
S11T011066	Glycolate	ug/mL	101	<9.37E-03	354	n/a	n/a	n/a	n/a	9.37	
S11T011066	Acetate	ug/mL	101	<6.04E-03	393	n/a	n/a	n/a	n/a	6.04	
S11T011066	Formate	ug/mL	101	<4.67E-03	1.22E+03	n/a	n/a	n/a	n/a	4.67	
S11T011066	Chloride	ug/mL	101	0.0131	2.61E+03	n/a	n/a	n/a	n/a	9.98	
S11T011066	Nitrite	ug/mL	101	<0.0192	5.72E+04	n/a	n/a	n/a	n/a	96.0	
S11T011066	Sulfate	ug/mL	102	<0.0187	1.43E+04	n/a	n/a	n/a	n/a	18.7	
S11T011066	Oxalate	ug/mL	102	<0.0231	300	n/a	n/a	n/a	n/a	23.1	J
S11T011066	Bromide	ug/mL	101	<0.0580	<58.0	n/a	n/a	n/a	n/a	58.0	U
S11T011066	Nitrate	ug/mL	100	0.0709	2.24E+05	n/a	n/a	n/a	n/a	208	
S11T011066	Phosphate	ug/mL	101	<0.0167	2.43E+03	n/a	n/a	n/a	n/a	16.7	
S11T011066	Silver	ug/mL	98.6	n/a	<12.5	n/a	n/a	n/a	n/a	12.5	
S11T011066	Aluminum	ug/mL	101	<0.0300	9.14E+03	n/a	n/a	n/a	n/a	75.0	
S11T011066	Arsenic	ug/mL	100	n/a	<125	n/a	n/a	n/a	n/a	125	
S11T011066	Boron	ug/mL	99.3	n/a	<75.0	n/a	n/a	n/a	n/a	75.0	
S11T011066	Barium	ug/mL	101	n/a	<7.50	n/a	n/a	n/a	n/a	7.50	
S11T011066	Beryllium	ug/mL	98.9	n/a	<2.50	n/a	n/a	n/a	n/a	2.50	
S11T011066	Bismuth	ug/mL	100	n/a	<250	n/a	n/a	n/a	n/a	250	
S11T011066	Calcium	ug/mL	101	<0.0500	<125	n/a	n/a	n/a	n/a	125	U
S11T011066	Cadmium	ug/mL	102	n/a	<12.5	n/a	n/a	n/a	n/a	12.5	
S11T011066	Cerium	ug/mL	101	n/a	<75.0	n/a	n/a	n/a	n/a	75.0	
S11T011066	Cobalt	ug/mL	101	n/a	<25.0	n/a	n/a	n/a	n/a	25.0	
S11T011066	Chromium	ug/mL	101	<5.00E-03	1.21E+03	n/a	n/a	n/a	n/a	12.5	
S11T011066	Copper	ug/mL	102	n/a	<12.5	n/a	n/a	n/a	n/a	12.5	
S11T011066	Europium	ug/mL	95.4	n/a	<12.5	n/a	n/a	n/a	n/a	12.5	
S11T011066	Iron	ug/mL	101	n/a	<12.5	n/a	n/a	n/a	n/a	12.5	
S11T011066	Potassium	ug/mL	92.3	<0.500	2.81E+03	n/a	n/a	n/a	n/a	1.25E+03	J
S11T011066	Lanthanum	ug/mL	99.5	n/a	<7.50	n/a	n/a	n/a	n/a	7.50	

Table D-6. Centrifuged Liquid from Sample CON 5. (2 sheets)

Sample Number(s): S11T011066 (Test Sample CON 5)

Sample Group: 20111225

Sample #	Analyte	Unit	Std %-Rec	Blank	Result	Duplicate	Average	RPD	Spk %-Rec	Det Limit	Qual Flags
S11T011066	Lithium	ug/mL	109	n/a	<7.50	n/a	n/a	n/a	n/a	7.50	
S11T011066	Magnesium	ug/mL	102	n/a	<125	n/a	n/a	n/a	n/a	125	
S11T011066	Manganese	ug/mL	100	n/a	<7.50	n/a	n/a	n/a	n/a	7.50	
S11T011066	Molybdenum	ug/mL	98.8	n/a	70.6	n/a	n/a	n/a	n/a	50.0	
S11T011066	Sodium	ug/mL	106	<0.100	2.06E+05	n/a	n/a	n/a	n/a	250	
S11T011066	Neodymium	ug/mL	100	n/a	<25.0	n/a	n/a	n/a	n/a	25.0	
S11T011066	Nickel	ug/mL	101	n/a	<50.0	n/a	n/a	n/a	n/a	50.0	
S11T011066	Phosphorus	ug/mL	102	<0.0500	927	n/a	n/a	n/a	n/a	125	J
S11T011066	Lead	ug/mL	104	n/a	<125	n/a	n/a	n/a	n/a	125	
S11T011066	Sulfur	ug/mL	98.4	<0.100	5.50E+03	n/a	n/a	n/a	n/a	250	
S11T011066	Antimony	ug/mL	94.6	n/a	<125	n/a	n/a	n/a	n/a	125	
S11T011066	Selenium	ug/mL	104	n/a	<250	n/a	n/a	n/a	n/a	250	
S11T011066	Silicon	ug/mL	94.7	<0.0300	233	n/a	n/a	n/a	n/a	75.0	J
S11T011066	Samarium	ug/mL	103	n/a	<125	n/a	n/a	n/a	n/a	125	
S11T011066	Strontium	ug/mL	101	n/a	<7.50	n/a	n/a	n/a	n/a	7.50	
S11T011066	Thorium	ug/mL	97.5	n/a	<125	n/a	n/a	n/a	n/a	125	
S11T011066	Titanium	ug/mL	97.3	n/a	<12.5	n/a	n/a	n/a	n/a	12.5	
S11T011066	Thallium	ug/mL	96.2	n/a	<250	n/a	n/a	n/a	n/a	250	
S11T011066	Uranium	ug/mL	101	n/a	<250	n/a	n/a	n/a	n/a	250	
S11T011066	Vanadium	ug/mL	101	n/a	<12.5	n/a	n/a	n/a	n/a	12.5	
S11T011066	Yttrium	ug/mL	94.7	n/a	<5.00	n/a	n/a	n/a	n/a	5.00	
S11T011066	Zinc	ug/mL	99.9	n/a	<12.5	n/a	n/a	n/a	n/a	12.5	
S11T011066	Zirconium	ug/mL	98.9	n/a	<12.5	n/a	n/a	n/a	n/a	12.5	
S11T011066	Hydroxide	ug/mL	105	<41.7	2.3E+04	n/a	n/a	n/a	n/a	550	
S11T011066	Specific gravity	unitless	100.5	n/a	1.317	n/a	n/a	n/a	n/a	1.00E-03	
S11T011066	Total organic carbon	ug/mL	96.5	<20.0	1.33E+03	1.35E+03	1.34E+03	1.49	90.8	200	J
S11T011066	Total inorganic carbon	ug/mL	98.7	<7.00	1.11E+04	1.13E+04	1.12E+04	1.79	94.6	70.0	

Table D-7. Centrifuged Liquid from Sample CON 6. (2 sheets)

Sample Number(s): S11T011067 (Test Sample CON 6)

Sample Group: 20111225

Sample #	Analyte	Unit	Std %-Rec	Blank	Result	Duplicate	Average	RPD	Spk %-Rec	Det Limit	Qual Flags
S11T011067	Fluoride	ug/mL	103	<1.61E-03	31.2	n/a	n/a	n/a	n/a	1.61	
S11T011067	Glycolate	ug/mL	101	<9.37E-03	397	n/a	n/a	n/a	n/a	9.37	
S11T011067	Acetate	ug/mL	101	<6.04E-03	446	n/a	n/a	n/a	n/a	6.04	
S11T011067	Formate	ug/mL	101	<4.67E-03	1.34E+03	n/a	n/a	n/a	n/a	4.67	
S11T011067	Chloride	ug/mL	101	0.0131	2.88E+03	n/a	n/a	n/a	n/a	9.98	
S11T011067	Nitrite	ug/mL	101	<0.0192	6.31E+04	n/a	n/a	n/a	n/a	96.0	
S11T011067	Sulfate	ug/mL	102	<0.0187	1.52E+04	n/a	n/a	n/a	n/a	18.7	
S11T011067	Oxalate	ug/mL	102	<0.0231	251	n/a	n/a	n/a	n/a	23.1	J
S11T011067	Bromide	ug/mL	101	<0.0580	<58.0	n/a	n/a	n/a	n/a	58.0	U
S11T011067	Nitrate	ug/mL	100	0.0709	2.16E+05	n/a	n/a	n/a	n/a	208	
S11T011067	Phosphate	ug/mL	101	<0.0167	3.07E+03	n/a	n/a	n/a	n/a	16.7	
S11T011067	Silver	ug/mL	97.6	n/a	<12.5	n/a	n/a	n/a	n/a	12.5	
S11T011067	Aluminum	ug/mL	100	<0.0300	1.02E+04	n/a	n/a	n/a	n/a	75.0	
S11T011067	Arsenic	ug/mL	100	n/a	<125	n/a	n/a	n/a	n/a	125	
S11T011067	Boron	ug/mL	99.2	n/a	<75.0	n/a	n/a	n/a	n/a	75.0	
S11T011067	Barium	ug/mL	99.0	n/a	<7.50	n/a	n/a	n/a	n/a	7.50	
S11T011067	Beryllium	ug/mL	97.5	n/a	<2.50	n/a	n/a	n/a	n/a	2.50	
S11T011067	Bismuth	ug/mL	97.0	n/a	<250	n/a	n/a	n/a	n/a	250	
S11T011067	Calcium	ug/mL	110	<0.0500	<125	n/a	n/a	n/a	n/a	125	U
S11T011067	Cadmium	ug/mL	98.1	n/a	<12.5	n/a	n/a	n/a	n/a	12.5	
S11T011067	Cerium	ug/mL	99.4	n/a	<75.0	n/a	n/a	n/a	n/a	75.0	
S11T011067	Cobalt	ug/mL	98.1	n/a	<25.0	n/a	n/a	n/a	n/a	25.0	
S11T011067	Chromium	ug/mL	98.1	<5.00E-03	1.32E+03	n/a	n/a	n/a	n/a	12.5	
S11T011067	Copper	ug/mL	99.8	n/a	<12.5	n/a	n/a	n/a	n/a	12.5	
S11T011067	Europium	ug/mL	94.3	n/a	<12.5	n/a	n/a	n/a	n/a	12.5	
S11T011067	Iron	ug/mL	99.6	n/a	<12.5	n/a	n/a	n/a	n/a	12.5	
S11T011067	Potassium	ug/mL	91.2	<0.500	3.49E+03	n/a	n/a	n/a	n/a	1.25E+03	J
S11T011067	Lanthanum	ug/mL	99.2	n/a	<7.50	n/a	n/a	n/a	n/a	7.50	

Table D-7. Centrifuged Liquid from Sample CON 6. (2 sheets)

Sample Number(s): S11T011067 (Test Sample CON 6)

Sample Group: 20111225

Sample #	Analyte	Unit	Std %-Rec	Blank	Result	Duplicate	Average	RPD	Spk %-Rec	Det Limit	Qual Flags
S11T011067	Lithium	ug/mL	107	n/a	<7.50	n/a	n/a	n/a	n/a	7.50	
S11T011067	Magnesium	ug/mL	99.5	n/a	<125	n/a	n/a	n/a	n/a	125	
S11T011067	Manganese	ug/mL	96.4	n/a	<7.50	n/a	n/a	n/a	n/a	7.50	
S11T011067	Molybdenum	ug/mL	98.4	n/a	65.5	n/a	n/a	n/a	n/a	50.0	
S11T011067	Sodium	ug/mL	107	<0.100	2.20E+05	n/a	n/a	n/a	n/a	250	
S11T011067	Neodymium	ug/mL	97.4	n/a	<25.0	n/a	n/a	n/a	n/a	25.0	
S11T011067	Nickel	ug/mL	97.3	n/a	<50.0	n/a	n/a	n/a	n/a	50.0	
S11T011067	Phosphorus	ug/mL	99.1	<0.0500	1.14E+03	n/a	n/a	n/a	n/a	125	J
S11T011067	Lead	ug/mL	101	n/a	<125	n/a	n/a	n/a	n/a	125	
S11T011067	Sulfur	ug/mL	99.2	<0.100	5.83E+03	n/a	n/a	n/a	n/a	250	
S11T011067	Antimony	ug/mL	95.3	n/a	<125	n/a	n/a	n/a	n/a	125	
S11T011067	Selenium	ug/mL	101	n/a	<250	n/a	n/a	n/a	n/a	250	
S11T011067	Silicon	ug/mL	96.7	<0.0300	141	n/a	n/a	n/a	n/a	75.0	J
S11T011067	Samarium	ug/mL	102	n/a	<125	n/a	n/a	n/a	n/a	125	
S11T011067	Strontium	ug/mL	98.3	n/a	<7.50	n/a	n/a	n/a	n/a	7.50	
S11T011067	Thorium	ug/mL	97.3	n/a	<125	n/a	n/a	n/a	n/a	125	
S11T011067	Titanium	ug/mL	96.6	n/a	<12.5	n/a	n/a	n/a	n/a	12.5	
S11T011067	Thallium	ug/mL	97.3	n/a	<250	n/a	n/a	n/a	n/a	250	
S11T011067	Uranium	ug/mL	99.5	n/a	<250	n/a	n/a	n/a	n/a	250	
S11T011067	Vanadium	ug/mL	98.8	n/a	<12.5	n/a	n/a	n/a	n/a	12.5	
S11T011067	Yttrium	ug/mL	94.7	n/a	<5.00	n/a	n/a	n/a	n/a	5.00	
S11T011067	Zinc	ug/mL	96.6	n/a	<12.5	n/a	n/a	n/a	n/a	12.5	
S11T011067	Zirconium	ug/mL	98.7	n/a	<12.5	n/a	n/a	n/a	n/a	12.5	
S11T011067	Hydroxide	ug/mL	105	<41.7	2.0E+04	n/a	n/a	n/a	n/a	550	
S11T011067	Specific gravity	unitless	100.5	n/a	1.381	n/a	n/a	n/a	n/a	1.00E-03	
S11T011067	Total organic carbon	ug/mL	96.5	<20.0	1.64E+03	n/a	n/a	n/a	n/a	200	J
S11T011067	Total inorganic carbon	ug/mL	98.7	<7.00	1.24E+04	n/a	n/a	n/a	n/a	70.0	

Table D-8. Centrifuged Liquid from Sample CON 7. (2 sheets)

Sample Number(s): S11T011070 (Test Sample CON 7)

Sample Group: 20111225

Sample #	Analyte	Unit	Std %-Rec	Blank	Result	Duplicate	Average	RPD	Spk %-Rec	Det Limit	Qual Flags
S11T011070	Fluoride	ug/mL	103	<1.61E-03	25.2	n/a	n/a	n/a	n/a	1.61	J
S11T011070	Glycolate	ug/mL	101	<9.37E-03	417	n/a	n/a	n/a	n/a	9.37	
S11T011070	Acetate	ug/mL	101	<6.04E-03	462	n/a	n/a	n/a	n/a	6.04	
S11T011070	Formate	ug/mL	101	<4.67E-03	1.40E+03	n/a	n/a	n/a	n/a	4.67	
S11T011070	Chloride	ug/mL	101	0.0131	3.02E+03	n/a	n/a	n/a	n/a	9.98	
S11T011070	Nitrite	ug/mL	101	<0.0192	6.40E+04	n/a	n/a	n/a	n/a	96.0	
S11T011070	Sulfate	ug/mL	102	<0.0187	1.36E+04	n/a	n/a	n/a	n/a	18.7	
S11T011070	Oxalate	ug/mL	102	<0.0231	128	n/a	n/a	n/a	n/a	23.1	J
S11T011070	Bromide	ug/mL	101	<0.0580	<58.0	n/a	n/a	n/a	n/a	58.0	U
S11T011070	Nitrate	ug/mL	100	0.0709	1.96E+05	n/a	n/a	n/a	n/a	208	
S11T011070	Phosphate	ug/mL	101	<0.0167	2.07E+03	n/a	n/a	n/a	n/a	16.7	
S11T011070	Silver	ug/mL	97.6	n/a	<12.5	n/a	n/a	n/a	n/a	12.5	
S11T011070	Aluminum	ug/mL	100	<0.0300	1.16E+04	n/a	n/a	n/a	n/a	75.0	
S11T011070	Arsenic	ug/mL	100	n/a	<125	n/a	n/a	n/a	n/a	125	
S11T011070	Boron	ug/mL	99.2	n/a	<75.0	n/a	n/a	n/a	n/a	75.0	
S11T011070	Barium	ug/mL	99.0	n/a	<7.50	n/a	n/a	n/a	n/a	7.50	
S11T011070	Beryllium	ug/mL	97.5	n/a	<2.50	n/a	n/a	n/a	n/a	2.50	
S11T011070	Bismuth	ug/mL	97.0	n/a	<250	n/a	n/a	n/a	n/a	250	
S11T011070	Calcium	ug/mL	110	<0.0500	<125	n/a	n/a	n/a	n/a	125	U
S11T011070	Cadmium	ug/mL	98.1	n/a	<12.5	n/a	n/a	n/a	n/a	12.5	
S11T011070	Cerium	ug/mL	99.4	n/a	<75.0	n/a	n/a	n/a	n/a	75.0	
S11T011070	Cobalt	ug/mL	98.1	n/a	<25.0	n/a	n/a	n/a	n/a	25.0	
S11T011070	Chromium	ug/mL	98.1	<5.00E-03	1.53E+03	n/a	n/a	n/a	n/a	12.5	
S11T011070	Copper	ug/mL	99.8	n/a	<12.5	n/a	n/a	n/a	n/a	12.5	
S11T011070	Europium	ug/mL	94.3	n/a	<12.5	n/a	n/a	n/a	n/a	12.5	
S11T011070	Iron	ug/mL	99.6	n/a	<12.5	n/a	n/a	n/a	n/a	12.5	
S11T011070	Potassium	ug/mL	91.2	<0.500	4.23E+03	n/a	n/a	n/a	n/a	1.25E+03	J
S11T011070	Lanthanum	ug/mL	99.2	n/a	<7.50	n/a	n/a	n/a	n/a	7.50	

Table D-8. Centrifuged Liquid from Sample CON 7. (2 sheets)

Sample Number(s): S11T011070 (Test Sample CON 7)

Sample Group: 20111225

Sample #	Analyte	Unit	Std %-Rec	Blank	Result	Duplicate	Average	RPD	Spk %-Rec	Det Limit	Qual Flags
S11T011070	Lithium	ug/mL	107	n/a	<7.50	n/a	n/a	n/a	n/a	7.50	
S11T011070	Magnesium	ug/mL	99.5	n/a	<125	n/a	n/a	n/a	n/a	125	
S11T011070	Manganese	ug/mL	96.4	n/a	<7.50	n/a	n/a	n/a	n/a	7.50	
S11T011070	Molybdenum	ug/mL	98.4	n/a	86.5	n/a	n/a	n/a	n/a	50.0	
S11T011070	Sodium	ug/mL	107	<0.100	2.16E+05	n/a	n/a	n/a	n/a	250	
S11T011070	Neodymium	ug/mL	97.4	n/a	<25.0	n/a	n/a	n/a	n/a	25.0	
S11T011070	Nickel	ug/mL	97.3	n/a	<50.0	n/a	n/a	n/a	n/a	50.0	
S11T011070	Phosphorus	ug/mL	99.1	<0.0500	885	n/a	n/a	n/a	n/a	125	J
S11T011070	Lead	ug/mL	101	n/a	<125	n/a	n/a	n/a	n/a	125	
S11T011070	Sulfur	ug/mL	99.2	<0.100	5.67E+03	n/a	n/a	n/a	n/a	250	
S11T011070	Antimony	ug/mL	95.3	n/a	<125	n/a	n/a	n/a	n/a	125	
S11T011070	Selenium	ug/mL	101	n/a	<250	n/a	n/a	n/a	n/a	250	
S11T011070	Silicon	ug/mL	96.7	<0.0300	158	n/a	n/a	n/a	n/a	75.0	J
S11T011070	Samarium	ug/mL	102	n/a	<125	n/a	n/a	n/a	n/a	125	
S11T011070	Strontium	ug/mL	98.3	n/a	<7.50	n/a	n/a	n/a	n/a	7.50	
S11T011070	Thorium	ug/mL	97.3	n/a	<125	n/a	n/a	n/a	n/a	125	
S11T011070	Titanium	ug/mL	96.6	n/a	<12.5	n/a	n/a	n/a	n/a	12.5	
S11T011070	Thallium	ug/mL	97.3	n/a	<250	n/a	n/a	n/a	n/a	250	
S11T011070	Uranium	ug/mL	99.5	n/a	<250	n/a	n/a	n/a	n/a	250	
S11T011070	Vanadium	ug/mL	98.8	n/a	<12.5	n/a	n/a	n/a	n/a	12.5	
S11T011070	Yttrium	ug/mL	94.7	n/a	<5.00	n/a	n/a	n/a	n/a	5.00	
S11T011070	Zinc	ug/mL	96.6	n/a	<12.5	n/a	n/a	n/a	n/a	12.5	
S11T011070	Zirconium	ug/mL	98.7	n/a	<12.5	n/a	n/a	n/a	n/a	12.5	
S11T011070	Hydroxide	ug/mL	105	<41.7	2.5E+04	n/a	n/a	n/a	n/a	550	
S11T011070	Specific gravity	unitless	100.5	n/a	1.342	n/a	n/a	n/a	n/a	1.00E-03	
S11T011070	Total organic carbon	ug/mL	96.5	<20.0	946	n/a	n/a	n/a	n/a	200	J
S11T011070	Total inorganic carbon	ug/mL	98.7	<7.00	6.39E+03	n/a	n/a	n/a	n/a	70.0	
S11T011070	Total carbon	ug/mL	97.2	<5.00	1.23E+04	n/a	n/a	n/a	n/a	55.0	

Table D-9. Centrifuged Liquid from Sample DIL 1. (2 sheets)

Sample Number(s): S11T011071 (Test Sample DIL 1)

Sample Group: 20111225

Sample #	Analyte	Unit	Std %-Rec	Blank	Result	Duplicate	Average	RPD	Spk %-Rec	Det Limit	Qual Flags
S11T011071	Fluoride	ug/mL	103	<1.61E-03	585	n/a	n/a	n/a	n/a	1.61	
S11T011071	Glycolate	ug/mL	98.8	<9.37E-03	228	n/a	n/a	n/a	n/a	9.37	
S11T011071	Acetate	ug/mL	97.0	<6.04E-03	265	n/a	n/a	n/a	n/a	6.04	
S11T011071	Formate	ug/mL	99.7	<4.67E-03	810	n/a	n/a	n/a	n/a	4.67	
S11T011071	Chloride	ug/mL	99.3	0.0132	1.75E+03	n/a	n/a	n/a	n/a	9.98	
S11T011071	Nitrite	ug/mL	101	<0.0192	3.80E+04	n/a	n/a	n/a	n/a	96.0	
S11T011071	Sulfate	ug/mL	102	<0.0187	1.13E+04	n/a	n/a	n/a	n/a	18.7	
S11T011071	Oxalate	ug/mL	102	<0.0231	931	n/a	n/a	n/a	n/a	23.1	
S11T011071	Bromide	ug/mL	101	<0.0580	<58.0	n/a	n/a	n/a	n/a	58.0	U
S11T011071	Nitrate	ug/mL	101	0.0715	1.48E+05	n/a	n/a	n/a	n/a	208	
S11T011071	Phosphate	ug/mL	101	<0.0167	3.63E+03	n/a	n/a	n/a	n/a	16.7	
S11T011071	Silver	ug/mL	97.6	n/a	<12.5	n/a	n/a	n/a	n/a	12.5	
S11T011071	Aluminum	ug/mL	100	<0.0300	4.99E+03	n/a	n/a	n/a	n/a	75.0	
S11T011071	Arsenic	ug/mL	100	n/a	<125	n/a	n/a	n/a	n/a	125	
S11T011071	Boron	ug/mL	99.2	n/a	<75.0	n/a	n/a	n/a	n/a	75.0	
S11T011071	Barium	ug/mL	99.0	n/a	<7.50	n/a	n/a	n/a	n/a	7.50	
S11T011071	Beryllium	ug/mL	97.5	n/a	<2.50	n/a	n/a	n/a	n/a	2.50	
S11T011071	Bismuth	ug/mL	97.0	n/a	<250	n/a	n/a	n/a	n/a	250	
S11T011071	Calcium	ug/mL	110	<0.0500	<125	n/a	n/a	n/a	n/a	125	U
S11T011071	Cadmium	ug/mL	98.1	n/a	<12.5	n/a	n/a	n/a	n/a	12.5	
S11T011071	Cerium	ug/mL	99.4	n/a	<75.0	n/a	n/a	n/a	n/a	75.0	
S11T011071	Cobalt	ug/mL	98.1	n/a	<25.0	n/a	n/a	n/a	n/a	25.0	
S11T011071	Chromium	ug/mL	98.1	<5.00E-03	651	n/a	n/a	n/a	n/a	12.5	
S11T011071	Copper	ug/mL	99.8	n/a	<12.5	n/a	n/a	n/a	n/a	12.5	
S11T011071	Europium	ug/mL	94.3	n/a	<12.5	n/a	n/a	n/a	n/a	12.5	
S11T011071	Iron	ug/mL	99.6	n/a	<12.5	n/a	n/a	n/a	n/a	12.5	
S11T011071	Potassium	ug/mL	91.2	<0.500	1.85E+03	n/a	n/a	n/a	n/a	1.25E+03	J
S11T011071	Lanthanum	ug/mL	99.2	n/a	<7.50	n/a	n/a	n/a	n/a	7.50	

Table D-9. Centrifuged Liquid from Sample DIL 1. (2 sheets)

Sample Number(s): S11T011071 (Test Sample DIL 1)

Sample Group: 20111225

Sample #	Analyte	Unit	Std %-Rec	Blank	Result	Duplicate	Average	RPD	Spk %-Rec	Det Limit	Qual Flags
S11T011071	Lithium	ug/mL	107	n/a	<7.50	n/a	n/a	n/a	n/a	7.50	
S11T011071	Magnesium	ug/mL	99.5	n/a	<125	n/a	n/a	n/a	n/a	125	
S11T011071	Manganese	ug/mL	96.4	n/a	<7.50	n/a	n/a	n/a	n/a	7.50	
S11T011071	Molybdenum	ug/mL	98.4	n/a	<50.0	n/a	n/a	n/a	n/a	50.0	
S11T011071	Sodium	ug/mL	107	<0.100	1.21E+05	n/a	n/a	n/a	n/a	250	
S11T011071	Neodymium	ug/mL	97.4	n/a	<25.0	n/a	n/a	n/a	n/a	25.0	
S11T011071	Nickel	ug/mL	97.3	n/a	<50.0	n/a	n/a	n/a	n/a	50.0	
S11T011071	Phosphorus	ug/mL	99.1	<0.0500	1.05E+03	n/a	n/a	n/a	n/a	125	J
S11T011071	Lead	ug/mL	101	n/a	<125	n/a	n/a	n/a	n/a	125	
S11T011071	Sulfur	ug/mL	99.2	<0.100	3.52E+03	n/a	n/a	n/a	n/a	250	
S11T011071	Antimony	ug/mL	95.3	n/a	<125	n/a	n/a	n/a	n/a	125	
S11T011071	Selenium	ug/mL	101	n/a	<250	n/a	n/a	n/a	n/a	250	
S11T011071	Silicon	ug/mL	96.7	<0.0300	142	n/a	n/a	n/a	n/a	75.0	J
S11T011071	Samarium	ug/mL	102	n/a	<125	n/a	n/a	n/a	n/a	125	
S11T011071	Strontium	ug/mL	98.3	n/a	<7.50	n/a	n/a	n/a	n/a	7.50	
S11T011071	Thorium	ug/mL	97.3	n/a	<125	n/a	n/a	n/a	n/a	125	
S11T011071	Titanium	ug/mL	96.6	n/a	<12.5	n/a	n/a	n/a	n/a	12.5	
S11T011071	Thallium	ug/mL	97.3	n/a	<250	n/a	n/a	n/a	n/a	250	
S11T011071	Uranium	ug/mL	99.5	n/a	<250	n/a	n/a	n/a	n/a	250	
S11T011071	Vanadium	ug/mL	98.8	n/a	<12.5	n/a	n/a	n/a	n/a	12.5	
S11T011071	Yttrium	ug/mL	94.7	n/a	<5.00	n/a	n/a	n/a	n/a	5.00	
S11T011071	Zinc	ug/mL	96.6	n/a	<12.5	n/a	n/a	n/a	n/a	12.5	
S11T011071	Zirconium	ug/mL	98.7	n/a	<12.5	n/a	n/a	n/a	n/a	12.5	
S11T011071	Hydroxide	ug/mL	105	<41.7	1.3E+04	n/a	n/a	n/a	n/a	550	
S11T011071	Specific gravity	unitless	100.5	n/a	1.292	n/a	n/a	n/a	n/a	1.00E-03	
S11T011071	Total organic carbon	ug/mL	96.5	<20.0	991	n/a	n/a	n/a	n/a	200	J
S11T011071	Total inorganic carbon	ug/mL	98.7	<7.00	6.37E+03	n/a	n/a	n/a	n/a	70.0	

Table D-10. Centrifuged Liquid from Sample DIL 2. (2 sheets)

Sample Number(s): S11T011072 (Test Sample DIL 2)

Sample Group: 20111225

Sample #	Analyte	Unit	Std %-Rec	Blank	Result	Duplicate	Average	RPD	Spk %-Rec	Det Limit	Qual Flags
S11T011072	Fluoride	ug/mL	103	<1.61E-03	555	n/a	n/a	n/a	n/a	1.61	
S11T011072	Glycolate	ug/mL	98.8	<9.37E-03	205	n/a	n/a	n/a	n/a	9.37	
S11T011072	Acetate	ug/mL	97.0	<6.04E-03	235	n/a	n/a	n/a	n/a	6.04	
S11T011072	Formate	ug/mL	99.7	<4.67E-03	762	n/a	n/a	n/a	n/a	4.67	
S11T011072	Chloride	ug/mL	99.3	0.0132	1.65E+03	n/a	n/a	n/a	n/a	9.98	
S11T011072	Nitrite	ug/mL	101	<0.0192	3.56E+04	n/a	n/a	n/a	n/a	96.0	
S11T011072	Sulfate	ug/mL	102	<0.0187	1.07E+04	n/a	n/a	n/a	n/a	18.7	
S11T011072	Oxalate	ug/mL	102	<0.0231	1.07E+03	n/a	n/a	n/a	n/a	23.1	
S11T011072	Bromide	ug/mL	101	<0.0580	<58.0	n/a	n/a	n/a	n/a	58.0	U
S11T011072	Nitrate	ug/mL	101	0.0715	1.41E+05	n/a	n/a	n/a	n/a	208	
S11T011072	Phosphate	ug/mL	101	<0.0167	3.45E+03	n/a	n/a	n/a	n/a	16.7	
S11T011072	Silver	ug/mL	97.6	n/a	<12.5	n/a	n/a	n/a	n/a	12.5	
S11T011072	Aluminum	ug/mL	100	<0.0300	5.35E+03	n/a	n/a	n/a	n/a	75.0	
S11T011072	Arsenic	ug/mL	100	n/a	<125	n/a	n/a	n/a	n/a	125	
S11T011072	Boron	ug/mL	99.2	n/a	<75.0	n/a	n/a	n/a	n/a	75.0	
S11T011072	Barium	ug/mL	99.0	n/a	<7.50	n/a	n/a	n/a	n/a	7.50	
S11T011072	Beryllium	ug/mL	97.5	n/a	<2.50	n/a	n/a	n/a	n/a	2.50	
S11T011072	Bismuth	ug/mL	97.0	n/a	<250	n/a	n/a	n/a	n/a	250	
S11T011072	Calcium	ug/mL	110	<0.0500	<125	n/a	n/a	n/a	n/a	125	U
S11T011072	Cadmium	ug/mL	98.1	n/a	<12.5	n/a	n/a	n/a	n/a	12.5	
S11T011072	Cerium	ug/mL	99.4	n/a	<75.0	n/a	n/a	n/a	n/a	75.0	
S11T011072	Cobalt	ug/mL	98.1	n/a	<25.0	n/a	n/a	n/a	n/a	25.0	
S11T011072	Chromium	ug/mL	98.1	<5.00E-03	700	n/a	n/a	n/a	n/a	12.5	
S11T011072	Copper	ug/mL	99.8	n/a	<12.5	n/a	n/a	n/a	n/a	12.5	
S11T011072	Europium	ug/mL	94.3	n/a	<12.5	n/a	n/a	n/a	n/a	12.5	
S11T011072	Iron	ug/mL	99.6	n/a	<12.5	n/a	n/a	n/a	n/a	12.5	
S11T011072	Potassium	ug/mL	91.2	<0.500	1.69E+03	n/a	n/a	n/a	n/a	1.25E+03	J
S11T011072	Lanthanum	ug/mL	99.2	n/a	<7.50	n/a	n/a	n/a	n/a	7.50	

Table D-10. Centrifuged Liquid from Sample DIL 2. (2 sheets)

Sample Number(s): S11T011072 (Test Sample DIL 2)

Sample Group: 20111225

Sample #	Analyte	Unit	Std %-Rec	Blank	Result	Duplicate	Average	RPD	Spk %-Rec	Det Limit	Qual Flags
S11T011072	Lithium	ug/mL	107	n/a	<7.50	n/a	n/a	n/a	n/a	7.50	
S11T011072	Magnesium	ug/mL	99.5	n/a	<125	n/a	n/a	n/a	n/a	125	
S11T011072	Manganese	ug/mL	96.4	n/a	<7.50	n/a	n/a	n/a	n/a	7.50	
S11T011072	Molybdenum	ug/mL	98.4	n/a	<50.0	n/a	n/a	n/a	n/a	50.0	
S11T011072	Sodium	ug/mL	107	<0.100	1.30E+05	n/a	n/a	n/a	n/a	250	
S11T011072	Neodymium	ug/mL	97.4	n/a	<25.0	n/a	n/a	n/a	n/a	25.0	
S11T011072	Nickel	ug/mL	97.3	n/a	<50.0	n/a	n/a	n/a	n/a	50.0	
S11T011072	Phosphorus	ug/mL	99.1	<0.0500	1.17E+03	n/a	n/a	n/a	n/a	125	J
S11T011072	Lead	ug/mL	101	n/a	<125	n/a	n/a	n/a	n/a	125	
S11T011072	Sulfur	ug/mL	99.2	<0.100	3.76E+03	n/a	n/a	n/a	n/a	250	
S11T011072	Antimony	ug/mL	95.3	n/a	<125	n/a	n/a	n/a	n/a	125	
S11T011072	Selenium	ug/mL	101	n/a	<250	n/a	n/a	n/a	n/a	250	
S11T011072	Silicon	ug/mL	96.7	<0.0300	211	n/a	n/a	n/a	n/a	75.0	J
S11T011072	Samarium	ug/mL	102	n/a	<125	n/a	n/a	n/a	n/a	125	
S11T011072	Strontium	ug/mL	98.3	n/a	<7.50	n/a	n/a	n/a	n/a	7.50	
S11T011072	Thorium	ug/mL	97.3	n/a	<125	n/a	n/a	n/a	n/a	125	
S11T011072	Titanium	ug/mL	96.6	n/a	<12.5	n/a	n/a	n/a	n/a	12.5	
S11T011072	Thallium	ug/mL	97.3	n/a	<250	n/a	n/a	n/a	n/a	250	
S11T011072	Uranium	ug/mL	99.5	n/a	<250	n/a	n/a	n/a	n/a	250	
S11T011072	Vanadium	ug/mL	98.8	n/a	<12.5	n/a	n/a	n/a	n/a	12.5	
S11T011072	Yttrium	ug/mL	94.7	n/a	<5.00	n/a	n/a	n/a	n/a	5.00	
S11T011072	Zinc	ug/mL	96.6	n/a	<12.5	n/a	n/a	n/a	n/a	12.5	
S11T011072	Zirconium	ug/mL	98.7	n/a	<12.5	n/a	n/a	n/a	n/a	12.5	
S11T011072	Hydroxide	ug/mL	105	<41.7	1.2E+04	n/a	n/a	n/a	n/a	550	
S11T011072	Specific gravity	unitless	100.5	n/a	1.299	n/a	n/a	n/a	n/a	1.00E-03	
S11T011072	Total organic carbon	ug/mL	96.5	<20.0	925	n/a	n/a	n/a	n/a	200	J
S11T011072	Total inorganic carbon	ug/mL	98.7	<7.00	6.24E+03	n/a	n/a	n/a	n/a	7.00	

Table D-11. Centrifuged Liquid from Sample DIL 3. (2 sheets)

Sample Number(s): S11T011073 (Test Sample DIL 3)

Sample Group: 20111225

Sample #	Analyte	Unit	Std %-Rec	Blank	Result	Duplicate	Average	RPD	Spk %-Rec	Det Limit	Qual Flags
S11T011073	Fluoride	ug/mL	103	<1.61E-03	513	n/a	n/a	n/a	n/a	1.61	
S11T011073	Glycolate	ug/mL	98.8	<9.37E-03	194	n/a	n/a	n/a	n/a	9.37	
S11T011073	Acetate	ug/mL	97.0	<6.04E-03	221	n/a	n/a	n/a	n/a	6.04	
S11T011073	Formate	ug/mL	99.7	<4.67E-03	700	n/a	n/a	n/a	n/a	4.67	
S11T011073	Chloride	ug/mL	99.3	0.0132	1.52E+03	n/a	n/a	n/a	n/a	9.98	
S11T011073	Nitrite	ug/mL	101	<0.0192	3.31E+04	n/a	n/a	n/a	n/a	96.0	
S11T011073	Sulfate	ug/mL	102	<0.0187	9.89E+03	n/a	n/a	n/a	n/a	18.7	
S11T011073	Oxalate	ug/mL	102	<0.0231	1.24E+03	n/a	n/a	n/a	n/a	23.1	
S11T011073	Bromide	ug/mL	101	<0.0580	<58.0	n/a	n/a	n/a	n/a	58.0	U
S11T011073	Nitrate	ug/mL	101	0.0715	1.29E+05	n/a	n/a	n/a	n/a	208	
S11T011073	Phosphate	ug/mL	101	<0.0167	3.19E+03	n/a	n/a	n/a	n/a	16.7	
S11T011073	Silver	ug/mL	97.6	n/a	<12.5	n/a	n/a	n/a	n/a	12.5	
S11T011073	Aluminum	ug/mL	100	<0.0300	5.76E+03	n/a	n/a	n/a	n/a	75.0	
S11T011073	Arsenic	ug/mL	100	n/a	<125	n/a	n/a	n/a	n/a	125	
S11T011073	Boron	ug/mL	99.2	n/a	<75.0	n/a	n/a	n/a	n/a	75.0	
S11T011073	Barium	ug/mL	99.0	n/a	<7.50	n/a	n/a	n/a	n/a	7.50	
S11T011073	Beryllium	ug/mL	97.5	n/a	<2.50	n/a	n/a	n/a	n/a	2.50	
S11T011073	Bismuth	ug/mL	97.0	n/a	<250	n/a	n/a	n/a	n/a	250	
S11T011073	Calcium	ug/mL	110	<0.0500	<125	n/a	n/a	n/a	n/a	125	U
S11T011073	Cadmium	ug/mL	98.1	n/a	<12.5	n/a	n/a	n/a	n/a	12.5	
S11T011073	Cerium	ug/mL	99.4	n/a	<75.0	n/a	n/a	n/a	n/a	75.0	
S11T011073	Cobalt	ug/mL	98.1	n/a	26.2	n/a	n/a	n/a	n/a	25.0	
S11T011073	Chromium	ug/mL	98.1	<5.00E-03	758	n/a	n/a	n/a	n/a	12.5	
S11T011073	Copper	ug/mL	99.8	n/a	<12.5	n/a	n/a	n/a	n/a	12.5	
S11T011073	Europium	ug/mL	94.3	n/a	<12.5	n/a	n/a	n/a	n/a	12.5	
S11T011073	Iron	ug/mL	99.6	n/a	<12.5	n/a	n/a	n/a	n/a	12.5	
S11T011073	Potassium	ug/mL	91.2	<0.500	2.32E+03	n/a	n/a	n/a	n/a	1.25E+03	J
S11T011073	Lanthanum	ug/mL	99.2	n/a	<7.50	n/a	n/a	n/a	n/a	7.50	

Table D-11. Centrifuged Liquid from Sample DIL 3. (2 sheets)

Sample Number(s): S11T011073 (Test Sample DIL 3)

Sample Group: 20111225

Sample #	Analyte	Unit	Std %-Rec	Blank	Result	Duplicate	Average	RPD	Spk %-Rec	Det Limit	Qual Flags
S11T011073	Lithium	ug/mL	107	n/a	<7.50	n/a	n/a	n/a	n/a	7.50	
S11T011073	Magnesium	ug/mL	99.5	n/a	<125	n/a	n/a	n/a	n/a	125	
S11T011073	Manganese	ug/mL	96.4	n/a	<7.50	n/a	n/a	n/a	n/a	7.50	
S11T011073	Molybdenum	ug/mL	98.4	n/a	<50.0	n/a	n/a	n/a	n/a	50.0	
S11T011073	Sodium	ug/mL	107	<0.100	1.40E+05	n/a	n/a	n/a	n/a	250	
S11T011073	Neodymium	ug/mL	97.4	n/a	<25.0	n/a	n/a	n/a	n/a	25.0	
S11T011073	Nickel	ug/mL	97.3	n/a	<50.0	n/a	n/a	n/a	n/a	50.0	
S11T011073	Phosphorus	ug/mL	99.1	<0.0500	1.29E+03	n/a	n/a	n/a	n/a	125	
S11T011073	Lead	ug/mL	101	n/a	<125	n/a	n/a	n/a	n/a	125	
S11T011073	Sulfur	ug/mL	99.2	<0.100	4.00E+03	n/a	n/a	n/a	n/a	250	
S11T011073	Antimony	ug/mL	95.3	n/a	<125	n/a	n/a	n/a	n/a	125	
S11T011073	Selenium	ug/mL	101	n/a	<250	n/a	n/a	n/a	n/a	250	
S11T011073	Silicon	ug/mL	96.7	<0.0300	198	n/a	n/a	n/a	n/a	75.0	J
S11T011073	Samarium	ug/mL	102	n/a	<125	n/a	n/a	n/a	n/a	125	
S11T011073	Strontium	ug/mL	98.3	n/a	<7.50	n/a	n/a	n/a	n/a	7.50	
S11T011073	Thorium	ug/mL	97.3	n/a	<125	n/a	n/a	n/a	n/a	125	
S11T011073	Titanium	ug/mL	96.6	n/a	<12.5	n/a	n/a	n/a	n/a	12.5	
S11T011073	Thallium	ug/mL	97.3	n/a	<250	n/a	n/a	n/a	n/a	250	
S11T011073	Uranium	ug/mL	99.5	n/a	<250	n/a	n/a	n/a	n/a	250	
S11T011073	Vanadium	ug/mL	98.8	n/a	<12.5	n/a	n/a	n/a	n/a	12.5	
S11T011073	Yttrium	ug/mL	94.7	n/a	<5.00	n/a	n/a	n/a	n/a	5.00	
S11T011073	Zinc	ug/mL	96.6	n/a	<12.5	n/a	n/a	n/a	n/a	12.5	
S11T011073	Zirconium	ug/mL	98.7	n/a	<12.5	n/a	n/a	n/a	n/a	12.5	
S11T011073	Hydroxide	ug/mL	105	<41.7	1.1E+04	n/a	n/a	n/a	n/a	550	
S11T011073	Specific gravity	unitless	100.5	n/a	1.259	n/a	n/a	n/a	n/a	1.00E-03	
S11T011073	Total organic carbon	ug/mL	96.5	<20.0	407	n/a	n/a	n/a	n/a	200	J
S11T011073	Total inorganic carbon	ug/mL	98.7	<7.00	5.98E+03	n/a	n/a	n/a	n/a	7.00	
S11T011073	Total carbon	ug/mL	97.2	<5.00	7.54E+03	n/a	n/a	n/a	n/a	55.0	

ATTACHMENT E

SOLID PHASE CHARACTERIZATION OF BOILDOWN SOLIDS

ATTACHMENT E

SOLID PHASE CHARACTERIZATION OF BOILDOWN SOLIDS

Five samples of centrifuged solids (C-Slds) recovered from tank 241-AP-107 boildown samples were submitted (October 18, 2011) for solid phase characterization (SPC) to identify solid phases present.

Sample Number	Solids
S11T014140	Composite of C-Slds recovered from Samples CON 1 (27.7 %WVR ^{18 °C}), CON 2 (36.9 %WVR ^{18 °C}), and CON 3 (41.6 %WVR ^{18 °C})
S11T014141	Composite of C-Slds recovered from Samples CON 4 (46.1 %WVR ^{18 °C}) and CON 5 (50.2 %WVR ^{18 °C})
S11T014280	C-Slds recovered from boildown Sample CON 6 (54.0 %WVR ^{18 °C})
S11T011074	C-Slds recovered from the end-point slurry Sample CON 7 (57.5 %WVR ^{18 °C})
S11T011075	Solids remaining after final dilution Step DIL 3 (8.4 %WVR ^{18 °C})

All 5 samples were submitted in 25-mL glass vials and contained white, fine-grained solids covered with a 2-5 mm layer of clear, yellow supernatant liquid.

Three analysis methods were used: polarized light microscopy (PLM), powder X-ray diffraction (XRD), and scanning electron microscopy with energy-dispersive X-ray spectrometry (SEM/EDX). The SPC analyses were performed by members of the 222-S Special Analytical Services and Process Chemistry groups under the following procedures:

ATS-LT-519-107	222-S Laboratory Polarized Light Microscopy
ATS-LT-507-101	222-S Laboratory X-Ray Diffractometry (XRD)
ATS-LT-161-100	222-S Laboratory Sample Preparation and Operating Procedure for Scanning Electron Microscopes

Each of the SPC methods is limited to examination of very small quantities of solids. Homogenization and subsampling of radiologically “hot” samples to collect representative specimens of the required weight/volume is difficult at best. Nevertheless, a single specimen was prepared and analyzed for each method. Also, specimens for XRD and SEM/EDX analyses must be dried during preparation, and solids dissolved in the liquids collected with the samples may precipitate and complicate interpretation of analysis results. For these reasons, the results of the SPC analyses are qualitative to semiquantitative in nature.

SEM/EDX Specimen Preparation and Analysis

Due to the high solubility of the solids, specimens for SEM/EDX analysis were prepared using the supernatant liquid in each sample to help disperse the particulates. This was accomplished by placing a 37-mm diameter cellulose backing pad and a 0.45-μm pore size, Nucleopore polycarbonate filter on the fritted glass base of a filtration flask connected to a trap and house

vacuum. A filtration funnel was then placed on the filter stack and clamped into place. For each sample, approximately 2 to 3 mL of the supernatant liquid was applied to the top of the filter, with the vacuum turned off, using a disposable plastic pipette. Using the same pipette, a small portion of the sample solids were then transferred to the liquid on the filter. The vacuum apparatus was then quickly and gently shaken to help disperse the solids across the filter surface, and the vacuum pump was immediately turned on. When the fluid had drained from the filter surface, the clamp and funnel were removed, and the filter was allowed to completely dry. A section of the filter was cut out and affixed to a carbon planchet (attached to an aluminum SEM stub) using carbon paint. The resulting SEM specimens were then coated with a conductive coating of carbon by evaporative deposition.

Analysis by SEM was conducted using an Aspex®⁵ Personal Scanning Electron Microscope Model II, with a Noran light element EDX spectrometer for chemical analysis. The instrument was operated at an accelerating voltage of 20 kV, and the samples were mounted at a working distance of 15 to 20 mm. All images were acquired in secondary electron imaging mode. The EDX spectra were acquired for 30 seconds live time (unless noted) on relatively smooth surfaces (either flat or sloping toward the detector) near the center of the images.

⁵ Aspex is a registered trademark of ASPEX Instruments, Delmont, Pennsylvania.

Sample S11T014140: Composite of Solids from Boildown Samples
CON 1 (27.7 %WVR^{18 °C}) through CON 3 (41.6 %WVR^{18 °C})

XRD Results

The major solid phases identified were natroxalate ($\text{Na}_2\text{C}_2\text{O}_4$) and natrophosphate [$\text{Na}_7\text{F}(\text{PO}_4)_2 \cdot 19\text{H}_2\text{O}$]. Nitratine (NaNO_3) was identified as a major phase and sodium nitrite (NaNO_2) as a minor phase of the sample; both are speculated to have crystallized from the interstitial liquid when the sample was dried. Thermonatrite ($\text{Na}_2\text{CO}_3 \cdot \text{H}_2\text{O}$) and kogarkoite (Na_3FSO_4) were identified as minor phases of the sample.

Chemical Name	Mineral Name	Formula	Relative Amount
Sodium Oxalate	Natroxalate	$\text{Na}_2\text{C}_2\text{O}_4$	Major
Sodium Fluoride Phosphate Hydrate	Natrophosphate	$\text{Na}_7\text{F}(\text{PO}_4)_2 \cdot 19\text{H}_2\text{O}$	Major
Sodium Nitrate	Nitratine	NaNO_3	Major
Sodium Nitrite	---	NaNO_2	Minor
Sodium Carbonate Hydrate	Thermonatrite	$\text{Na}_2\text{CO}_3 \cdot \text{H}_2\text{O}$	Minor
Sodium Fluoride Sulfate	Kogarkoite	Na_3FSO_4	Minor

Figure XRD-1. Sample S11T014140.

PLM Results (S11T014140)

Natrophosphate crystals and small (<20 μm) natroxalate needles and clusters were observed.

Figure PLM-1. Sample S11T014140.

SEM/EDX Results (S11T014140)

The solid particulates were a mix of acicular (needle-like), hexagonal plate, and octahedral particles. One example of the hexagonal plate particles is shown in Figure SEM-1 including an EDX spectrum acquired from the location marked with a yellow '+'. The left SEM image shows the general area containing the hexagonal particles, and the right image is a magnified view of a twinned particle. The particle was composed of sodium, fluorine, sulfur, and oxygen. These sodium fluoride sulfate particles were identified as kogarkoite based upon the elemental composition and morphology.

Figure SEM-1. SEM Images and EDX Spectrum of Kogarkoite Particle from S11T014140.

Further examination of the acicular particulate is shown in Figure SEM-2. Here, several spots free from underlying particles were selected for EDX analysis (underlying particles can also give off X-rays complicating EDX spectra interpretation). The spectra suggest that there are at least two different phases making up the acicular particulate; one containing sodium and oxygen (possibly sodium oxalate) and another containing sodium, oxygen, and sulfur.

Figure SEM-2. SEM Image and EDX Spectra of Various Acicular Particles in S11T011040.

Figure SEM-3 shows that the acicular particles are typically $\approx 20\ \mu\text{m}$ in length and $\approx 1.5\ \mu\text{m}$ wide.

Figure SEM-3. SEM Image Showing Typical Dimensions of Acicular Particles in S11T014140.

Another location of well-dispersed acicular solids is shown in Figure SEM-4. Analyses of the particles (EDX analysis spots indicated by yellow '+' signs in the left image) show a single phase composing sodium and oxygen. A feature with these particles is highlighted in the right image. A circle was drawn around each spot where an EDX spectrum was acquired. Beam damage at these points is visible as dark spots. Previous studies of tank waste showed that this phenomenon is common with sodium carbonate, and this assignment fits well with the EDX spectra for these solids.

Figure SEM-4. SEM Images and EDX Spectra of Sodium Carbonate Particles in S11T014140.

Another common feature observed in Sample S11T014140 was the aggregation of the acicular particles. Often, this created clumps as seen in most of the region in the left image of Figure SEM-5. However, occasionally, these clumps took the form of spheres (right image). The EDX spectra from the clumps and the spheres are very similar suggesting that they are composed of the same solid phase.

Figure SEM-5. SEM Images and EDX Spectra of Clumps of Acicular Particles in S11T014140.

Octahedral particles were observed throughout Sample S11T014140. Figure SEM-6 shows several of these particles (left image) and a magnification of a particle (right image). These particles are identified as sodium fluoride phosphate (natrophosphate) based on the distinctive morphology and the EDX spectrum.

Figure SEM-6. SEM Images and EDX Spectrum of a Natrophosphate Particle in S11T014140.

Sample S11T014141: Composite of Solids from Boildown Samples
CON 4 (46.1 %WVR^{18 °C}) and CON 5 (50.2 %WVR^{18 °C})

XRD Results

Nitratine (NaNO_3) was identified as a major phase and sodium nitrite (NaNO_2) as a minor phase of the sample; they are speculated to have crystallized from the interstitial liquid when the sample was dried. Thermonatrite ($\text{Na}_2\text{CO}_3 \cdot \text{H}_2\text{O}$) was identified as a minor phase of the sample. Natroxalate ($\text{Na}_2\text{C}_2\text{O}_4$) and natrophosphate [$\text{Na}_7\text{F}(\text{PO}_4)_2 \cdot 19\text{H}_2\text{O}$] were identified as trace phases of the sample.

Chemical Name	Mineral Name	Formula	Relative Amount
Sodium Nitrate	Nitratine	NaNO_3	Major
Sodium Nitrite	---	NaNO_2	Minor
Sodium Carbonate Hydrate	Thermonatrite	$\text{Na}_2\text{CO}_3 \cdot \text{H}_2\text{O}$	Minor
Sodium Oxalate	Natroxalate	$\text{Na}_2\text{C}_2\text{O}_4$	Trace
Sodium Fluoride Phosphate Hydrate	Natrophosphate	$\text{Na}_7\text{F}(\text{PO}_4)_2 \cdot 19\text{H}_2\text{O}$	Trace

Figure XRD-2. Sample S11T014141.

PLM Results (S11T014141)

Natrophosphate crystals and natroxalate needles were observed.

Figure PLM-2. Sample S11T014141.

SEM/EDX Results (S11T014141)

The solids were a mix of acicular particles ($\approx 6\text{--}8\ \mu\text{m} \times 0.8\ \mu\text{m}$), euhedral, hexagonal particles (up to $30\ \mu\text{m}$ in size), and octahedral particles (many over $50\ \mu\text{m}$ in size). Figure SEM-7 shows a typical area of the specimen (left image). The expanded image on the right provides more detail on the acicular and euhedral particulates. Analysis of the acicular particles (EDX Spots 1 and 2) indicate the elemental composition is dominated by sodium and oxygen with traces of sulfur, potassium, and aluminum. The EDX spectra from the euhedral particles (Spots 3 and 4) indicate that these solids are sodium fluoride sulfate (kogarkoite).

Figure SEM-7. SEM Images and EDX Spectra of Solids in S11T014141.

Further analysis of the acicular particles, Figure SEM-8, confirmed that these solids were composed mainly of sodium and oxygen with trace sulfur. The chlorine and potassium in the EDX spectrum from Spot 1 could be surface residue. Based upon these results, these acicular particles were identified as sodium oxalate (natroxalate). Beam damage (dark marks) of the crystals during EDX spectrum acquisition (most prominent at Spot 1) could indicate that the particles are very thin.

Figure SEM-8. SEM Image and EDX Spectra from Acicular Solids in S11T014141.

Another image of the small euhedral particles, many displaying a hexagonal to hexagonal-rod morphology, is shown in Figure SEM-9. Elemental analysis (EDX Spot 1) confirmed the identification of these solids as sodium fluoride sulfate. The beam damage visible at Spot 1 again suggests that these particles may be very thin. The spectrum from Spot 2 is characteristic of sodium oxalate. The spectrum from Spot 3 shows a calcium-rich particle (this was the only calcium-rich particle observed in this sample).

Figure SEM-9. SEM Image and EDX Spectra of Small Euhedral Particles in S11T014141.

Larger octahedral particles were scattered throughout the SEM specimen, Figure SEM-10. Again, the distinct morphology and EDX spectrum indicates these solids are natrophosphate.

Figure SEM-10. SEM Image and EDX Spectrum of a Natrophosphate Particle in S11T014141.

Sodium chloride was observed in trace amounts in the matted particulate on the SEM specimen, Figure SEM-11.

Figure SEM-11. SEM Image and EDX Spectrum of Sodium Chloride Particles in S11T014141.

Small plate-like particles were also observed (at a lower abundance) in the matted particulate on the SEM specimen, Figure SEM-12. The EDX spectrum indicates the presence of sodium and oxygen with a trace of potassium. Based on the morphology and elemental composition, these solids were identified as sodium carbonate. A prominent beam damage spot is visible in the right image.

Figure SEM-12. SEM Images and EDX Spectrum of a Sodium Carbonate Particle in S11T014141.

Sample S11T014280: Solids from Boildown Sample CON 6 (54.0 %WVR^{18 °C})**XRD Results**

Nitratine (NaNO_3) was identified as a major phase and sodium nitrite (NaNO_2) as a trace phase in the sample; both are speculated to have crystallized from the interstitial liquid when the sample was dried. Natrophosphate [$\text{Na}_7\text{F}(\text{PO}_4)_2 \cdot 19\text{H}_2\text{O}$] and thermonatrite ($\text{Na}_2\text{CO}_3 \cdot \text{H}_2\text{O}$) were identified as minor phases in the sample.

Chemical Name	Mineral Name	Formula	Relative Amount
Sodium Nitrate	Nitratine	NaNO_3	Major
Sodium Nitrite	---	NaNO_2	Trace
Sodium Fluoride Phosphate Hydrate	Natrophosphate	$\text{Na}_7\text{F}(\text{PO}_4)_2 \cdot 19\text{H}_2\text{O}$	Minor
Sodium Carbonate Hydrate	Thermonatrite	$\text{Na}_2\text{CO}_3 \cdot \text{H}_2\text{O}$	Minor

Figure XRD-3. Sample S11T014280.

PLM Results (S11T014280)

Sodium nitrate crystals, natrophosphate crystals, sodium phosphate needles (see close-up), and natroxalate needles were identified. Sodium phosphate and natroxalate needles have the same shape but opposite orientation.

Figure PLM-3. Sample S11T014280.

SEM/EDX Results (S11T014280)

The solids in the sample were dominated by acicular particles. A mix of small subhedral and large euhedral particles were also observed. The left image in Figure SEM-13 is representative of the specimen. The right image is an expanded view of one of the large euhedral particles identified as natrophosphate.

Figure SEM-13. SEM Images and EDX Spectrum of a Natrophosphate Particle in S11T014280.

The particulates making up the nearly continuous mat of solids on the specimen were examined further in Figure SEM-14. Several acicular particles (EDX Spots 1–3) were found to contain sodium, oxygen, fluorine, and sulfur with varying amounts of aluminum. This appears to be another phase of sodium fluoride sulfate. One of the smaller subhedral particles was also analyzed (EDX Spot 5), and showed an elemental composition similar to that of the acicular particles with minor amounts of chlorine and potassium. An amorphous, cementing phase,

EDX Spot 4, was observed at several locations on the particulate mat. Elemental analysis showed it to be sodium nitrate, possibly precipitated from the sample supernatant liquid during the sample preparation.

Figure SEM-14. SEM Image and EDX Spectra of Dominant Solids in S11T014280.

Two different sodium fluoride sulfate phases posited to be present in the sample are shown in Figure SEM-15. One acicular particle and one subhedral particle were chosen for EDX analysis. The resulting spectra show them to be very similar in elemental composition.

Figure SEM-15. SEM Image and EDX Spectra of Sodium Fluoride Sulfate Phases in S11T014280.

Trace quantities of a subhedral, calcium-rich phase was also observed, Figure SEM-16.

Figure SEM-16. SEM Image and EDX Spectrum of Calcium-Rich Phase in S11T014280.

Sample S11T011074: Solids from Boildown Sample CON 7 (57.5 %WVR¹⁸°C)**XRD Results**

Nitratine (NaNO_3) was identified as a major phase and sodium nitrite (NaNO_2) as a minor phase in the sample; both are speculated to have crystallized from the interstitial liquid when the sample was dried. Thermonatrite ($\text{Na}_2\text{CO}_3 \cdot \text{H}_2\text{O}$) was identified as a minor phase and natrophosphate [$\text{Na}_7\text{F}(\text{PO}_4)_2 \cdot 19\text{H}_2\text{O}$] as a trace phase of the sample.

Chemical Name	Mineral Name	Formula	Relative Amount
Sodium Nitrate	Nitratine	NaNO_3	Major
Sodium Nitrite	---	NaNO_2	Minor
Sodium Carbonate Hydrate	Thermonatrite	$\text{Na}_2\text{CO}_3 \cdot \text{H}_2\text{O}$	Minor
Sodium Fluoride Phosphate Hydrate	Natrophosphate	$\text{Na}_7\text{F}(\text{PO}_4)_2 \cdot 19\text{H}_2\text{O}$	Trace

Figure XRD-4. Sample S11T011074.

PLM Results (S11T011074)

Observed solids were mostly natroxalate needles and clusters. Some sodium carbonate and isotropic shards (presumably natrophosphate) were also identified. A minor amount of an unidentified, light-brown, fine-grained material was also observed.

Figure PLM-4. Sample S11T011074.

SEM/EDX Results (S11T011074)

A population of relatively small acicular and flake-like crystals was observed in the specimen. Populations of larger octahedral and rhombohedral crystals were also observed.

Figure SEM-17 shows an area on the SEM specimen where the continuous mat of small acicular and flake-like crystals was disrupted exposing a section of the polycarbonate filter. This location allowed the EDX analysis of these particles without the electron beam penetrating into underlying particles. The right image is a magnification of the area in the left image outlined with the yellow box. The EDX spectrum from Spot 1 indicates that the flake-like crystals are rich in sodium and oxygen. This elemental composition and morphology identify these solids as sodium carbonate. The spectrum from Spot 2 indicates that the acicular solids, which are composed of sodium, sulfur, oxygen, and fluorine, are a sodium fluoride sulfate phase.

Figure SEM-17. SEM Images and EDX Spectra from Acicular and Flake-like Crystals in S11T011074.

Further analysis of the acicular solids, Figure SEM-18, showed variations in sulfur and fluorine content. Trace amounts of aluminum and potassium were also detected in some of the particles. This suggests that more than one solid phase may be present in the acicular particles.

Figure SEM-18. SEM Image and EDX Spectra of Additional Acicular Particles from S11T011074.

Large solid particles were scattered throughout the mat of smaller acicular and flake-like particulates. Figure SEM-19 shows one of these particles with a structure of twinned rhombohedral crystals. The EDX spectrum shows the presence of sodium, oxygen, and nitrogen. The elemental content and morphology indicate this particle is sodium nitrate.

Figure SEM-19. SEM Image and EDX Spectrum of a Twinned, Rhombohedral Sodium Nitrate Particle.

Figure SEM-20 shows an area with several of the larger particles. The rhombohedral crystal in Area 1 is sodium nitrate. The octahedral crystal in Area 2 is natrophosphate.

Figure SEM-20. SEM Images and EDX Spectra Showing the Two Types of Large Crystalline Solids in S11T011074.

Sample S11T011075: Solids from Boildown Step DIL 3 (8.4 %WVR¹⁸°C)**XRD Results**

The major solid phase in the sample was natroxalate ($\text{Na}_2\text{C}_2\text{O}_4$). Nitratine (NaNO_3) was identified as a major phase and sodium nitrite (NaNO_2) as a minor phase in the sample; both are speculated to have crystallized from the interstitial liquid when the sample was dried. Natrite (Na_2CO_3) and natrophosphate [$\text{Na}_7\text{F}(\text{PO}_4)_2 \cdot 19\text{H}_2\text{O}$] were identified as a minor phases, and thermonatrite ($\text{Na}_2\text{CO}_3 \cdot \text{H}_2\text{O}$) as a trace phase of the sample.

Chemical Name	Mineral Name	Formula	Relative Amount
Sodium Oxalate	Natroxalate	$\text{Na}_2\text{C}_2\text{O}_4$	Major
Sodium Nitrate	Nitratine	NaNO_3	Major
Sodium Nitrite	NaNO_2	---	Minor
Sodium Carbonate	Natrite	Na_2CO_3	Minor
Sodium Fluoride Phosphate Hydrate	Natrophosphate	$\text{Na}_7\text{F}(\text{PO}_4)_2 \cdot 19\text{H}_2\text{O}$	Minor
Sodium Carbonate Hydrate	Thermonatrite	$\text{Na}_2\text{CO}_3 \cdot \text{H}_2\text{O}$	Trace

Figure XRD-5. Sample S11T011075.

PLM Results (S11T011075)

Sodium oxalates (as individual needles and clusters) were observed. A lack of the isotropic shards (presumably natrophosphate) observed in Sample S11T011074 was noted.

Figure PLM-5. Sample S11T011075.

SEM/EDX Results (S11T011075)

The observed solids were mostly small ($\approx 15\ \mu\text{m}$ long), narrow, flat crystals. Figure SEM-21 shows an area SEM specimen where the particulates were dispersed well enough to acquire EDX spectra on three individual particles. A fourth EDX spectrum was acquired from a bare spot on the polycarbonate filter. All three EDX spectra from the particles showed consistent ratios of sodium, oxygen, and sulfur with trace aluminum. The bare spot showed sodium and trace aluminum. The observed morphology and EDX spectra are characteristic of sodium oxalate.

Figure SEM-21. SEM Image and EDX Spectra of Particles in S11T011075.

Figure SEM-22 is an image of the same area as Figure SEM-21 taken after the EDX spectra were acquired. The yellow circles highlight spots of beam damage. This phenomenon has been seen previously with oxalate crystals.

Figure SEM-22. Image of Particles from Figure SEM-21 Showing Beam Damage.

The acicular crystals in S11T011075 also displayed a tendency to cluster into $\approx 50\text{-}\mu\text{m}$ diameter balls. Figure SEM-23 shows several of these clusters and a magnified image displaying in more detail how the particles are packed together. The EDX spectrum, acquired from one of the clusters, detected sodium and oxygen with trace aluminum.

Figure SEM-23. SEM Images and EDX Spectrum of Particle Clusters in S11T011075.

A vast majority of the solids in Sample S11T011075 were the acicular particles; however, a few flake-like particles were seen on top and embedded in the particulate mat. One of these flake-like particles is shown in Figure SEM-24. The EDX analysis detected sodium and oxygen, and, along with the morphology, suggested these solids were sodium carbonate.

Figure SEM-24. SEM Image and EDX Spectrum of a Flake-like Particle in S11T011075.

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