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Tank 41H Post-Dissolution Saltcake Core and Supernate Sample Analysis

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Summary

This report provides analyses of the samples from Tank 41H taken after the June 2003 dissolution campaign. The characterization also includes supernate samples pulled from the surface (347 inch) and 250 inch levels of the salt supernate below the C1 and C3 risers in September 2003 (HTF-E-03-124, 125, 129, 130, and 132).

- The three saltcake samples (HTF-E-03-145, 146, and 147) were filled to nearly their capacity with saltcake and free liquid, with an average saltcake bulk density of 2.06 g/cm^3 .
- The undrained saltcake from the bottom of the middle sample (HTF-E-03-146) had a water content of 4.1 wt %, a ^{137}Cs activity of 0.14 Ci per gallon of saltcake, and an alpha content of $1.4\text{E}+4 \text{ pCi/g}$.
- The undrained saltcake from the bottom of the bottom sample (HTF-E-03-147) had a water content of 8.5 wt %, a ^{137}Cs activity of 0.17 Ci per gallon of saltcake, and an alpha content of $3.0\text{E}+4 \text{ pCi/g}$.
- Interstitial liquid drained from the middle sample (HTF-E-03-146) had a density of 1.43 g/cm^3 , a soluble solids content of 45.4 wt %, a ^{137}Cs activity of 0.78 Ci per gallon of interstitial liquid, and an alpha content of $1.1\text{E}+4 \text{ pCi/mL}$.
- An analysis of material from the top of the top sample (HTF-E-03-145) is provided in support of Nuclear Criticality Safety Evaluations. The as-received saltcake and the residual insoluble solids had a uranium-235 enrichment of approximately 12.2%. Characterization focused on providing information on fissile radionuclides, potential neutron poisons, and other potential diluents.
- Supernate samples pulled from the surface and from the 250 in. levels of the C1 and C3 risers yielded information on the vertical and lateral tank supernate homogeneity. The ^{137}Cs content of these Tank 41H samples averaged 0.56 Ci/gal for the C3 riser and 0.65 Ci/gal for the C1 riser. The ^{238}Pu content of the four unfiltered samples ranged from $1.41\text{E}+4 \text{ pCi/mL}$ to $2.21\text{E}+4 \text{ pCi/mL}$.
- Scanning electron microscopy of saltcake from sample HTF-E-03-146 revealed several features consistent with mineral dissolution: sub-rounded appearance of grains, lack of coatings by secondary precipitates, and crevices and indentations in some grains.

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List of Abbreviations

AA	Atomic Absorption spectroscopy
ADS	Analytical Development Section
DI	de-ionized
Dip	Dip sample of supernatant liquid from the tank's liquid surface
EDS	X-ray Emission Dispersive Spectroscopy
FL	Free Liquid supernatant to a saltcake sample upon receipt
IC	Ion Chromatography
ICP-ES	Inductively-Coupled Plasma – Emission Spectroscopy
ICP-MS	Inductively-Coupled Plasma – Mass Spectroscopy
I.D.	inner diameter
IL	Interstitial Liquid drained from a saltcake sample
LCS	Low-Curie Salt
LWD	Liquid Waste Disposition
NCSE	Nuclear Criticality Safety Evaluation
O.D.	outer diameter
SEM	Scanning Electron Microscopy
SL	Supernatant Liquid from a tank supernate sample
SRS	Savannah River Site
SRTC	Savannah River Technology Center
TIC	Total Inorganic Carbon
TOC	Total Organic Carbon
VDS	Variable Depth Sample of supernatant liquid from a specific tank elevation
WAC	Waste Acceptance Criteria (for Saltstone)
WCS	Waste Characterization System
XRD	X-Ray Diffraction spectroscopy

Introduction

In support of Low-Curie Salt (LCS) process validation, Liquid Waste Disposition (LWD) has undertaken a program of tank characterization, including salt sampling.^{1,2} This program includes characterization of key radioactive components in the unfiltered and filtered supernate samples, including ¹³⁷Cs and ²³⁸Pu. Additional chemical analyses are performed to provide information on salt elemental, ionic, and radiological composition to aid in assessment of the suitability of processing drained and dissolved material in the Saltstone facility. Additional information will aid in refining the Waste Characterization System (WCS). This series of Tank 41H saltcake samples corresponds to a three-foot-deep region of saltcake at the bottom of a canyon below the B3 riser as it existed after the first stage of Tank 41H dissolution.

Background

Beginning in the September 2002, a portion of the interstitial liquid was drained from Tank 41H. At that point (prior to dissolution), a three-foot series of drained-saltcake core samples was pulled from the surface of the salt below the B3 riser.³ In June 2003, 244 kgal of flush water was added to the surface of the Tank 41H saltcake, dissolving approximately 130 kgal of salt while resaturating the saltcake pores.⁴ Shortly after the first stage of Tank 41H salt dissolution was completed, liquid variable depth samples (HTF-E-03-91 and 92) were pulled from near the 250" level of the B3 riser and analyzed for comparison with the Saltstone Waste Acceptance Criteria (WAC).⁵ As a follow up to this effort, LWD sampled the supernate from various locations in Tank 41H to determine radial and vertical uniformity and changes from the sampling two months prior. The analysis of these supernate samples (HTF-E-03-124, 125, 129, 130, and 132) is included in this report. Several previous saltcake samples were taken from the surface and subsurface of Tank 41H to support nuclear criticality safety evaluations.^{6,7,8}

After the dissolution campaign, another series of three 1-foot core samples was pulled from the surface of the saltcake beneath the B3 riser. The surface of the saltcake in the canyon below this water-addition riser was estimated (after dissolution) to be 232 inches tank elevation and is beneath 115 inches of liquid.⁴ This set of samples is thought to also encompass the lower of two hard layers (at approximately 200 inches tank elevation) that were noted during well-screen mining. This report includes the results from the analysis of these Tank 41 H saltcake core samples that were taken after the June 2003 dissolution campaign (HTF-E-03-145 – 147). The top-most sample was primarily analyzed in a manner to support the Nuclear Criticality Safety Evaluation (NCSE) and the other two samples underwent the bulk undrained saltcake and drained interstitial liquid analyses in a manner consistent with other recent saltcake samples.⁹

Samples

Saltcake Core Samples (HTF-E-03-145 – 147)

On December 9, 2003, three salt core samples were collected from Riser B3 of Tank 41H. These samples were contained in 1.00 in. I.D., 13.7 in. long hardened stainless steel tubes with capped ends. The top 2 inches of these samples tubes contained a thicker portion of threaded tubing, which contained no sample material because it was attached to the mast during sampling. The top of the top sample roughly corresponds to the surface of the saltcake below the B3 riser at a tank elevation of 232 inches.

The samples were delivered to the Savannah River Technology Center (SRTC), placed into the Shielded Cells (B Block, Cell 8), and weighed on December 10th. Table 1 contains a description of

the three Tank 41H samples documented in this report. Note that the sample volume is approximate because the depth to the salt in the top of the samples was determined by inserting a pipette and assuming that the surface was flat. Likewise, Figure 1 displays the approximate contents of the Tank 41H samples and indicates the regions within the samples that were used for characterization.

Figure 2 shows, from left to right, the bottoms of samples HTF-E-03-145, 146, and 147. Figure 3 shows the salt and free liquid removed from the top of HTF-E-03-145 for nuclear criticality safety characterization. When the top of the sample was opened, 30.4 g of free liquid was decanted from the top by pipetting and pouring. The free liquid (FL) removed from the top of HTF-E-03-145 was cloudy and light brown in color and appeared to contain some salt crystals in addition to fine solids. Although not measured, the density of the free liquid can be surmised from similar Tank 41H samples to be between 1.40 and 1.43 g/cm³.

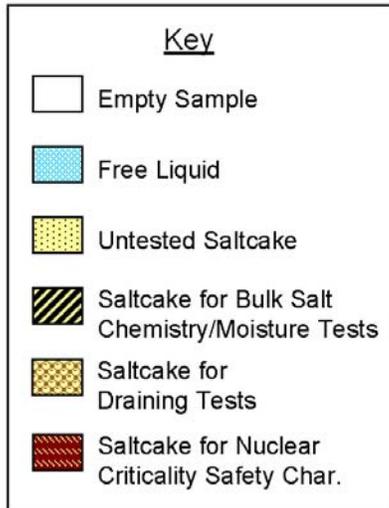
After removal of the free liquid, the remainder of the sample tube (124.6 cm³) contained 258.6 g of saltcake, for an average as-received saltcake density of 2.08 g/cm³. Approximately 75 g of salt was removed from the top of sample HTF-E-03-145, which corresponds to roughly the top three inches of saltcake. This 75 g of material was mixed well and used for the analysis contained in this report. This saltcake was very wet and was easily scooped from the sample tube. It was light brown in color, similar to the free liquid that was located above it in the top of the sample tube. The sample had a water content of 9.9 ± 1.8 wt %, which corresponds to an interstitial liquid content of roughly 26 vol. %.

The free liquid (FL) removed from the top of HTF-E-03-145 was cloudy and light brown in color and apparently contained some salt crystals in addition to fine solids. The top three inches of saltcake from HTF-E-03-145 was removed for analysis. Saltcake at the top of the sample was very wet and was easily scooped from the sample tube. This wet salt was light brown in color, similar to the free liquid that was co-located in the top of the sample tube. There was no noticeable change in salt characteristics throughout the top of sample HTF-E-03-145, although the appearance was different from the bottom of the same sample tube. Material from the bottom of HTF-E-03-145 was lighter in color and appeared more rigid and less wet than the material from the top.

Salt from the bottom of HTF-E-03-146 appeared dry and was white in color. It was hard packed and very difficult to remove by scooping from the tube. It was similar to hardness, perhaps harder, than the previous drained Tank 41H core samples.³ This observed hardness, coupled with the field sampling observations, lead to the belief that this material was the hard layer in the tank and was of analytical interest. The wetness apparent in the picture of HTF-E-03-146 in Figure 2 was liquid that lightly coated only the bottom surface of the sample. A small amount of FL was decanted from the top of the sample. No observation was made of the salt from the top this sample due to the extra threaded length of the tube.

Salt from the bottom of HTF-E-03-147 was very white and appeared more moist than HTF-E-03-146. The saltcake was relatively easy to remove from the bottom of tube HTF-E-03-147 and it looked like the typical sodium nitrate salt sample. A small amount of FL was decanted from the top of the sample. No observation was made of the salt from the top this sample due to the extra threaded length of the tube.

Tank 41H
“36 inch” Saltcake
Post-Dissolution Core Sample



Surface at B3 riser ~232 inches.
All interface levels are approximate.

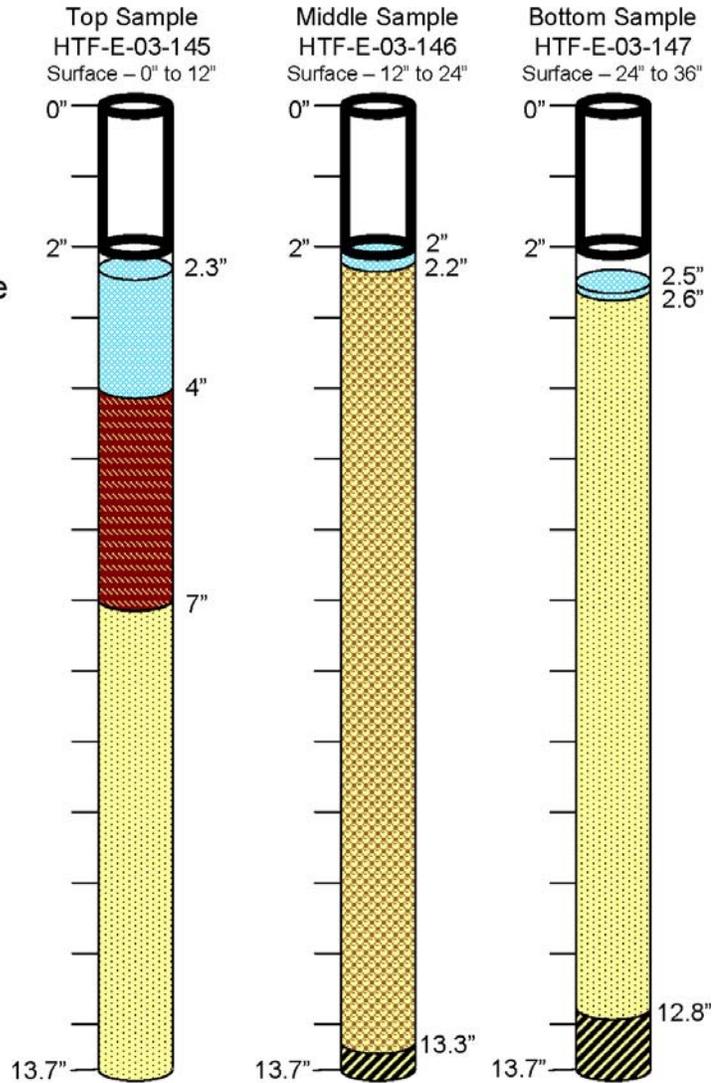


Figure 1: Material contained in Tank 41H core samples

Table 1: Tank 41H Post-Dissolution Saltcake Sample Description

Relative Location	Tank Farm Name	Approximate Tank Elevation (in)	Free Liquid Mass (g)	Salt Mass (g)	Salt Volume (cm ³)	Salt Density (g/cm ³)
top	HTF-E-03-145	232 – 220	30.4	258.6	124.6	2.08
middle	HTF-E-03-146	220 – 208	4.3	307.2	148.6	2.07
bottom	HTF-E-03-147	208 – 196	1.9	294.3	144.1	2.04
total		232 – 196	36.6	860.2	417.3	2.06



Figure 2: Tank 41H saltcake from the bottom of samples HTF-E-03-145, 146, and 147.



Figure 3: 75 grams of salt (left) and 30 grams of supernatant fluid (right) collected from the top of HTF-E-03-145 and utilized in the nuclear criticality safety characterization.

Supernatant Samples (HTF-E-03-124, 125, 129, 130, and 132)

On September 12 and 18, 2003, SRTC received supernatant samples from Tank 41H C3 and C1 risers, respectively. The Tank 41H C3-riser samples consisted of a surface dip sample (HTF-E-03-124) and a 250" variable depth sample (VDS) (HTF-E-03-125). The Tank 41H C1-riser samples consisted of a surface dip sample (HTF-E-03-129) and two 250" VDSs (HTF-E-03-130 and 132). The surface of the supernatant was at approximately 347" tank elevation.

Figure 4 contains pictures of the unfiltered supernatant samples. All samples contained yellow liquid that was slightly clouded by a small amount of brown suspended solids. The C3 riser surface sample also had a small amount of dark floating solids that were on the order of 0.1 to 1 mm in diameter. The C1 riser VDS had one large floating agglomerated solid particle approximately 1 cm in diameter,

which broke apart upon filtration and could not be analyzed. Because HTF-E-03-130 contained less than 10 mL of material, the two C1 riser VDSs were combined prior to analysis and the resulting composite is referred to as HTF-E-03-130/2. Table 2 contains information on the sample contents.



Figure 4: As received (unfiltered) Tank 41H supernate samples from (left to right) the surface at the C3 riser, the 250" level at the C3 riser, the surface at the C1 riser, and the 250" level at the C1 riser

Table 2: Tank 41H Post-Dissolution Supernate Sample Description

Tank Farm ID Number	Riser	Type	Tank Elevation	SRTC Code	Volume (mL)
HTF-E-03-124	C3	Dip	347 in.	A	80
HTF-E-03-125	C3	VDS	250 in.	B	65
HTF-E-03-129	C1	Dip	347 in.	C	55
HTF-E-03-130 HTF-E-03-132	C1	VDS	250 in.	D	85

Experimental

The saltcake material was analyzed using two separate approaches, one geared toward understanding salt chemical and radiological characteristics and another specifically concerned with the nuclear criticality safety of saltcake dissolution. The first approach, used on samples HTF-E-03-146 and 147, involved analysis similar to that performed on the other recent core samples, such as those from Tank 10H.⁹ Portions of each sample were completely dissolved for bulk saltcake analysis. The remaining portion of HTF-E-146 was drained of a portion of its interstitial liquid (IL), and the IL was analyzed for chemical and radiological components. The second analysis, to support the NCSE, involved dissolving away most of the salt from the top of HTF-E-03-145, followed by digestion of the residual insoluble solids and analysis of fissile radionuclides and potential neutron poisons and mass diluents. Additionally, several supernate surface and variable depth samples were analyzed to provide information on tank liquid variability after the June 2003 dissolution.

As part of these analyses, all three saltcake samples were weighed, uncapped, and visually inspected upon receipt in the shielded cells. Free liquid from the top of the samples were decanted and the distance to the top of the saltcake in the samples was noted. The largest uncertainty in determining the volume of the saltcake in the sample tubes is determining the distance to the top of the salt in the sample tube since the tubes contained an empty 2-inch threaded portion that could not be removed.

Bulk Saltcake Analysis (HTF-E-03-146 and 147)

Material from the bottom of samples HTF-E-03-146 and HTF-E-03-147 was removed for undrained bulk saltcake chemical and radiological characterization and water content measurements. Undrained bulk salt subsamples were prepared for analysis either by aqua regia (acid) dissolution or by DI water dissolution. The aqua regia dissolutions were performed by dissolving approximately 2 grams of the material into 100 mL of liquid. The aqua regia dissolutions were submitted to radiological chemistry for ^{137}Cs (gamma scan) and Pu isotopics, inductively coupled plasma – emission spectroscopy (ICP-ES) for elemental analysis, inductively coupled plasma – mass spectroscopy (ICP-MS) for certain actinide and fission product quantification, and atomic absorption (AA) spectroscopy for the measurement of As, Se, Hg, and K. The DI water dissolutions were performed using an approximately 2 grams of salt and 40 g of water. No significant residual solids were visible. The water dilutions/dissolutions were submitted for ion chromatography (IC) for anion characterization, wet chemistry/titration for total base and free hydroxide analysis, and total inorganic and organic carbon (TIC/TOC) analysis. An additional portion of the water-dissolved sample was submitted for ^{14}C analysis. Similar analyses were performed on the filtered interstitial liquid (IL). Portions of the undrained bulk saltcake were analyzed by X-ray diffraction (XRD) spectroscopy for crystalline solid identification and by scanning electron microscopy with energy dispersive spectroscopy (SEM/EDS) for salt grain size, morphology, and localized concentration information. Note that this sample was carbon coated (in contrast to solids from SL samples that were gold-palladium coated) and analyzed on the contained SEM Leo model 440.

The saltcake bulk density of the as-received material was calculated using the weight of sample obtained and an estimate of the filled volume of the tube. Density of the interstitial liquid was measured as the weight of material required to fill 2 mL Class A volumetric flasks. The water content of the saltcake and the drained interstitial liquid were estimated gravimetrically: small portions (~1g) of material were dried at 115 °C (± 5 °C) to drive off water until a constant weight had been achieved.

Draining and Interstitial Liquid Analysis (HTF-E-03-146)

Interstitial liquid (IL) was drained from sample HTF-E-03-146 using 5 inches of mercury vacuum for 19 days and 12 inches of mercury for 14 days respectively using the apparatus shown in Figure 5. IL drained from HTF-E-03-146 during the initial draining period was characterized.

The IL was prepared for analysis by filtering through a 0.45-micron filter and diluting with 2M nitric acid or DI water. Acid dilutions of the filtered IL were submitted for the following analyses: radiological chemistry (^{137}Cs and Pu isotopics), ICP-ES, ICP-MS, and AA. Water dilutions of the filtered IL were submitted for the following analyses: IC, wet chemistry titration, and TIC/TOC.

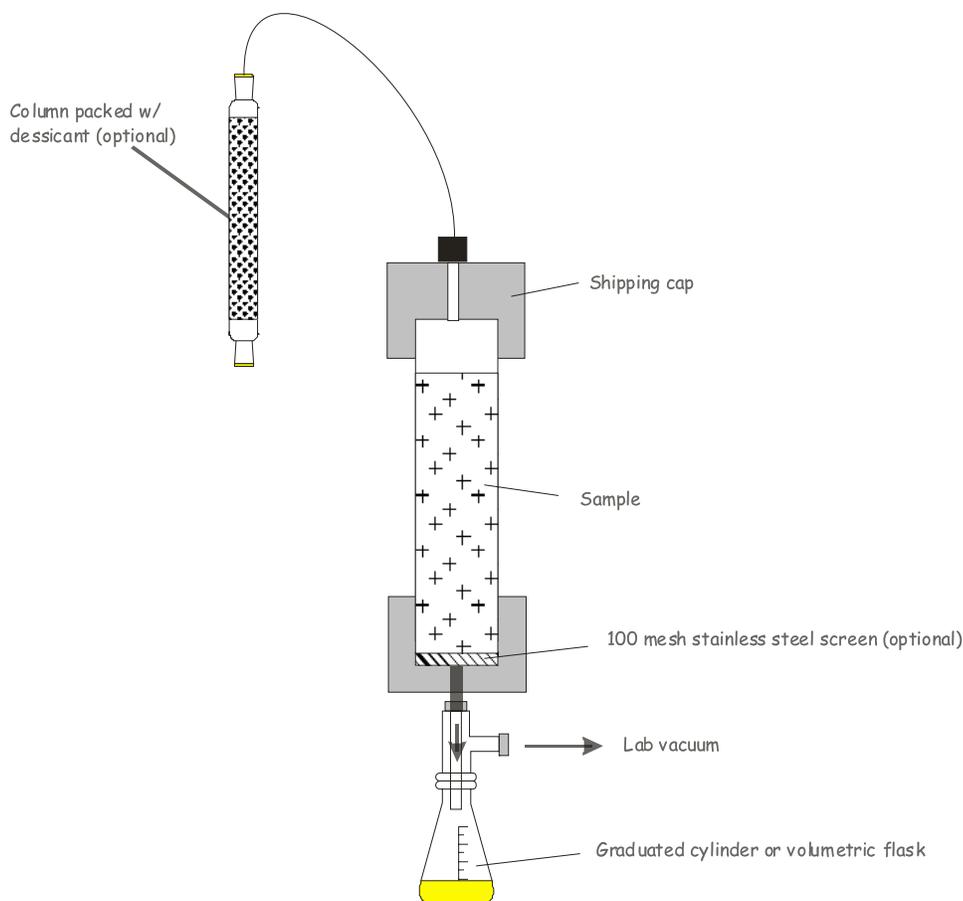


Figure 5: Apparatus used for interstitial liquid drainage of sample HTF-E-03-146.

Supernate Sample Analysis (HTF-E-03-124, 125, 129, 130, and 132)

Upon entry into the SRTC shielded cells, the samples were poured into glass beakers to allow for visual observation. Small (2 mL) portions of the as-received samples were retained for unfiltered analysis. The samples were filtered through 0.45-micron filters and portions of the filtered samples were analyzed. The solids on the filters were washed with water and dried for determination of sample solids content and particle-size distribution. An additional filtration was performed on the C1 riser VDS using 0.1-micron filters and a portion of the filtrate was analyzed.

Unfiltered and filtered supernate samples were prepared for analysis either by pressurized HNO₃/HF (acid) dissolution or by DI water dilution. The dissolutions/dilutions were performed by dissolving/diluting approximately 2 grams of the sample with 100 mL of liquid. Pressurized HNO₃/HF dissolutions of the filtered and unfiltered samples were submitted to radiological chemistry for ¹³⁷Cs (gamma scan), Pu isotopics, ²⁴¹Am, and ⁹⁰Sr, inductively coupled plasma – emission spectroscopy (ICP-ES) for elemental analysis, inductively coupled plasma – mass spectroscopy (ICP-MS) for certain actinide and fission product quantification, and atomic absorption (AA) spectroscopy for the measurement of As, Se, Hg, and K. The water dilutions of the filtered samples were submitted for ion chromatography (IC) for anion characterization, wet chemistry/titration for total base and free hydroxide, and total inorganic and organic carbon (TIC/TOC) analysis. Total inorganic carbon is reported as mass of CO₃²⁻ and total organic carbon is reported as mass of carbon. An additional portion of the water-diluted sample was submitted for ¹⁴C analysis.

Sample densities were measured in 2 mL Class A volumetric flasks. The total solids content, which is approximately equivalent to the soluble solids content for these samples, was estimated gravimetrically: ~1 g portions of sample were dried at 115 °C (± 5 °C) to drive off water until a constant weight had been achieved. The insoluble solids content was estimated by filtration of the sample followed by washing and drying of the filtered solids.

Viscosity measurements of the filtered composite HTF-E-03-130/2 were performed in a cylindrical viscometer described elsewhere.¹⁰

Portions of the solids filtered from HTF-E-03-124 and HTF-E-03-130/2 were analyzed by XRD and SEM/EDS.

NCSE-Related Analysis of Saltcake and Residual Insoluble Solids (HTF-E-03-145)

The saltcake material (75 g) from the top ~3 inches of sample HTF-E-03-145 was homogenized and characterized for uranium and plutonium isotopes and potential neutron poisons. Portions of this 3-inch segment of as-received saltcake material were digested by two methods and characterized in duplicate. The remainder of the top ~3 inches of the saltcake, along with the additional solid-laden fluid decanted from the top of the sample, was washed three times with deionized (DI) water, resulting in residual insoluble solids that were dried, digested by two methods, and characterized in duplicate or triplicate. The following are the details of this general procedure.

Two aliquots of the homogenized top 3-inch segment of as-received sample were pulled for aqua regia dissolution and two were pulled for sodium peroxide fusion digestion. Masses of 1 to 1.5 grams of original as-received sample material were dissolved into 100 mL of liquid. The dissolved samples were removed from the shielded cells and transferred to the Analytical Development Section (ADS) for analysis for actinides and neutron poisons. Analyses conducted include inductively coupled plasma-emission spectrometry (ICP-ES) for various elemental species including neutron poisons, inductively coupled plasma-mass spectrometry (ICP-MS) for various actinide isotopes, and PuTTA (Pu-238/241) analysis for ²³⁸Pu and ²⁴¹Pu. Gamma scan for ¹³⁷Cs was also conducted.

The insoluble solids that would be the ultimate residue from saltcake dissolution were prepared from the as-received saltcake and solids-laden free liquid by the following methodology: To avoid potential over-washing of the saltcake with quantities of water that are much larger than would be used during in-tank dissolution, LWD has requested that a specific amount of water be used during laboratory preparation of residual insoluble solids.¹¹ The dissolution protocol involved one wash at a 2:1 mass ratio of water to as-received sample, followed by two additional washes each of 0.5:1 mass ratio of water to as-received sample. Four polypropylene centrifuge tubes were filled with a total of 58.8 g of the as-received sample, 22.5 g of the free liquid, and 118.4 g of DI water. Dissolution proceeded at ambient temperature over a period of ≥ 3 hours with occasional agitation of the mixture, after which the mixtures were centrifuged for ≥ 30 minutes to settle the solids and the supernatant liquid was decanted. This initial dissolution was followed up by two additional dissolution steps, each of which involved the addition of 30.1 grams of DI water followed by processing in a manner similar to the initial dissolution. The resulting total amount of insoluble solids, when dried, was adequate for dissolution and analysis.

The residual insoluble solids were transferred into a glass beaker and dried to a constant weight at 115 ± 5 °C. Three aliquots of the dried solids were dissolved using aqua regia dissolution and two aliquots of the solids were dissolved using sodium peroxide fusion digestion. Masses of 0.1 to 0.17 g of dried insoluble solids were dissolved into 100 mL of liquid in each of the dissolutions. No observable solids remained in the fluids prepared by the aqua regia and peroxide fusion dissolution methods. The dissolutions of the dried insoluble solids were submitted for the same analyses as the as-received sample dissolutions. Additionally, small portions of the dry residual insoluble solids were

analyzed by XRD for crystalline solid identification and by SEM/EDS for particle size, morphology, and localized concentration information.

The liquid fractions from the three dissolution stages were diluted with DI water and analyzed in duplicate by ICP-ES, ICP-MS, IC, wet chem. titration, and TIC/TOC.

Results

Density, Moisture, and Percent Liquid

Table 3 contains measurements of the density and water content for the undrained post-dissolution saltcake samples, the drained interstitial liquid (IL), the four post-dissolution supernatant liquid (SL) samples from the C1 and C3 risers, and the previous post-dissolution SL sample from the B3 riser.⁵ Liquid content of the undrained saltcake, also contained in Table 3, is calculated using the following 2 equations:

$$\text{Liquid in Saltcake (wt \%)} = \frac{\text{Water in Saltcake (wt \%)}}{\text{Water in Liquid (wt \%)}} \times 100$$

$$\text{Liquid in Saltcake (vol \%)} = \text{Liquid in Saltcake (wt \%)} \times \frac{\text{Density of Saltcake (g/mL)}}{\text{Density of Liquid (g/mL)}} \times 100$$

Table 3: Tank 41H Post-Dissolution Samples: Density and Water Content

Sample Name	Sample Type	Density (g/mL)		Water (wt%)		Liquid (wt%)	Liquid (vol%)
		Average	St Dev	Average	St Dev		
HTF-E-03-145	undrained saltcake	2.08	--	9.9%	1.8%	17.8%	25.9%
HTF-E-03-146	undrained saltcake	2.07	--	4.1%	0.8%	7.4%	10.7%
HTF-E-03-146	IL (Filtered)	1.427	0.002	55.6%	0.5%	100.0%	100.0%
HTF-E-03-147	undrained saltcake	2.04	--	8.5%	0.5%	15.3%	21.9%
HTF-E-03-124	SL (unfiltered)	1.403	0.009	54.4%	0.3%	> 99.6%	> 99.6%
HTF-E-03-125	SL (unfiltered)	1.397	0.005	55.0%	0.2%	> 99.6%	> 99.6%
HTF-E-03-129	SL (unfiltered)	1.401	0.008	55.3%	1.1%	> 99.6%	> 99.6%
HTF-E-03-130/2	SL (unfiltered)	1.415	0.005	54.4%	0.4%	> 99.6%	> 99.6%
HTF-E-03-91/2	SL (unfiltered)	1.401	0.017	53.8%	0.02%	99.7%	99.7%

The viscosity of the filtered Tank 41H SL sample HTF-E-03-130/2 was measured at three temperatures: 25 °C, 35 °C, and 50 °C. Steady state measurements were recorded and the results are shown in Figure 6. The data was fitted to an exponential equation and the equation is also shown in Figure 6. The fitted equations can only be used in the temperature range (25°C to 50°C) in which they were fitted. As expected, this Tank 41H SL sample was less viscous than a recent Tank 38H SL sample at equivalent temperatures.¹⁰ Typically, dissolved saltcake solutions have lower hydroxide concentrations, and thus lower viscosities, than do concentrated supernate solutions. The HTF-E-03-130 SL sample measured viscosity of 7.1 cP at 25 °C compares favorably with the previous B3-riser SL sample (HTF-E-03-91) viscosity of 6.4 cP at 25 °C.⁵

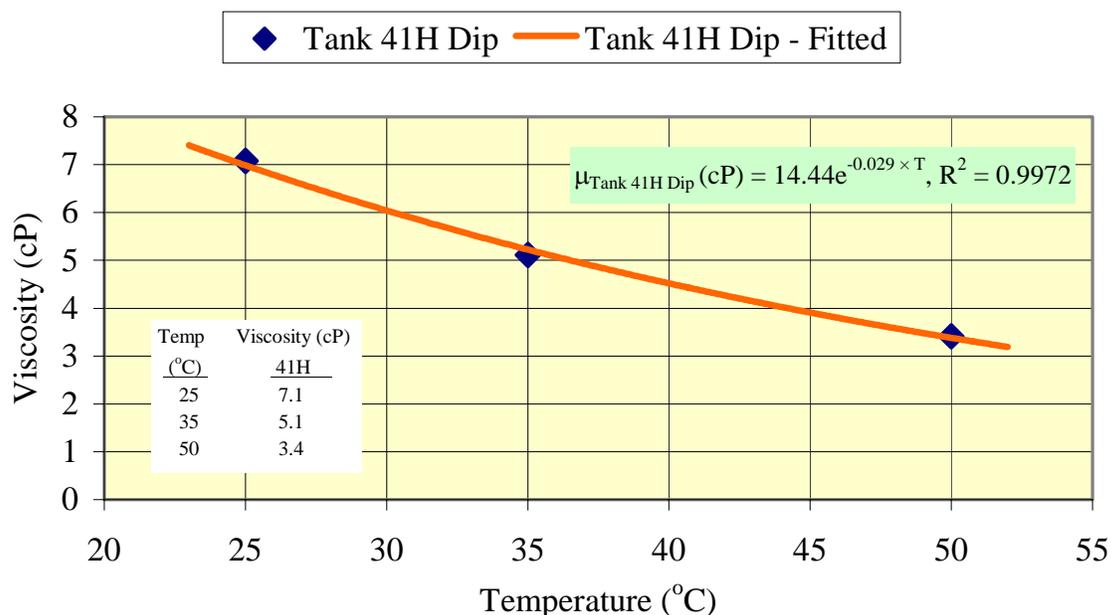


Figure 6: Viscosity of Tank 41H variable depth supernate sample HTF-E-03-130/2

Bulk Saltcake

Table 6, in the appendix, contains the water content results for saltcake removed from the bottom of HTF-E-03-146 and HTF-E-03-147. These results show moisture content variations within the sample, consistent with observations. The volume percent interstitial liquid (IL) in the salt samples are estimated by taking into account the undrained saltcake density and the density and moisture content of the interstitial liquid drained from HTF-E-03-146.

Table 7 through Table 11 in the appendix contain the characterization information for the undrained bulk saltcake. Results are reported as the average of analyses of triplicate sample dissolutions. Values reported as their detection limits are preceded by "<" and values that contain an average of detectable concentrations and detection limits are preceded by "<=".

Salt from the bottoms of HTF-E-03-146 and 147 were primarily sodium nitrate salt and were consistent with the previous Tank 41H saltcake samples. The ¹³⁷Cs activity of salt from the bottoms of HTF-E-03-146 and HTF-E-03-147 were 1.81E+7 pCi/g (0.14 Ci/gal) and 2.25E+7 pCi/g (0.17 Ci/gal), respectively. Both portions of salt were fully (>99%) and quickly (<1 minute) soluble with a 20:1 water:salt dissolution. While this is typical for a mostly sodium nitrate salt, it is contrary to the in-tank well-mining operation expectation of a hard and potentially insoluble layer sampled in the lower part of HTF-E-03-146. This result indicates that, although material at the bottom of HTF-E-03-146 was hard, it does not correspond to a region of low solubility.

Interstitial Liquid

Table 12 through Table 15 contain the characterization information for the drained and filtered interstitial liquid (IL) from HTF-E-03-146. As seen in Figure 8, approximately 28 grams of IL was drained from 298 g of HTF-E-03-146 over a period of 28 days. The density of the interstitial liquid was 1.427 g/cm³ with a standard deviation of 0.002 g/cm³ determined from two measurements. The soluble solids content of the interstitial liquid was 45.4 wt % with a standard deviation of 0.5 wt %

determined from three measurements. The interstitial liquid was 8.4 M $[\text{Na}^+]$ and had a ^{137}Cs content of $2.05\text{E}+8$ pCi/mL (0.78 Ci/gal).

The uranium content of the drained IL is inconsistent with the uranium content of the undrained bulk saltcake samples. Based on the sample water content and the ICP-MS data, uranium was observed to be partitioned >100% into the interstitial liquid. This inconsistency is likely due to an analytical error and warrants further investigation.

Dip Samples

Table 19 through Table 23 contain the initial data from the analysis of the as-received (unfiltered) and the filtered Tank 41H supernate samples (HTF-E-03-124, 125, 129, 130, and 132). The average and standard deviation of duplicate analyses of a single diluted sample are provided. Values reported as their detection limits are preceded by "<" and values that contain an average of detectable concentrations and detection limits are preceded by "<!=".

Overall, these four samples from different areas of Tank 41H demonstrate that the sampled regions of the Tank 41H supernate are essentially homogeneous, with no striking differences in activities and concentrations.

The average ^{137}Cs activity was $1.47\text{E}+8$ pCi/mL (0.56 Ci/gal) for the C3 riser samples and $1.72\text{E}+8$ pCi/mL (0.65 Ci/gal) for the C1 riser samples. The ^{137}Cs concentration in these four locations are higher than the initial post-dissolution analysis performed on samples pulled in July 2003 from near the 250" level of the B3 riser, which was $9.97\text{E}+7$ pCi/mL (0.38 Ci/gal). As expected, filtration did not significantly reduce the ^{137}Cs content of the samples.

The ^{238}Pu concentration in the four unfiltered samples ranged from $1.41\text{E}+4$ pCi/mL to $2.21\text{E}+4$ pCi/mL, with an average of $1.72\text{E}+4$ pCi/mL. These current samples contain less ^{238}Pu than the previous unfiltered Tank 41H post-dissolution supernate sample (HTF-E-03-91 and 92), which contained $6.05\text{E}+5$ pCi/mL of ^{238}Pu . The ^{238}Pu content of the current samples is more consistent with the concentration in the filtered HTF-E-03-91 and 92 samples, which was $2.96\text{E}+4$ to $9.2\text{E}+4$ pCi/mL.⁵ Filtration of the current samples caused only a slight reduction of ^{238}Pu activity in most cases. Note that the $^{239/240}\text{Pu}$ was too small in relation to the ^{238}Pu to be determined reliably. Additionally, $^{239/240}\text{Pu}$ was within a factor of two of the detection limit.

NCSE

The material used in the preparation of insoluble solids was 58.8 g of saltcake and 22.5 g of free liquid. The first dissolution stage used 118.4 g of DI water and resulted in a 7.7 g heel of wet solids after decanting. The second and third dissolution stages each combined the heel from the previous stage with 30.1 g of DI water and each resulted in a 1.7 g heel of wet solids after decanting. There was no substantial change in the mass or appearance of the residual insoluble solids during the third dissolution stage. After drying, the residual insoluble solid material was 0.88 g of gray powder, as seen in Figure 7. The dried insoluble solids correspond to 1.5 wt % of this portion of the original as-received 3-inch segment of sample. Note that a portion of these solids were likely contributed by the free liquid suspension.

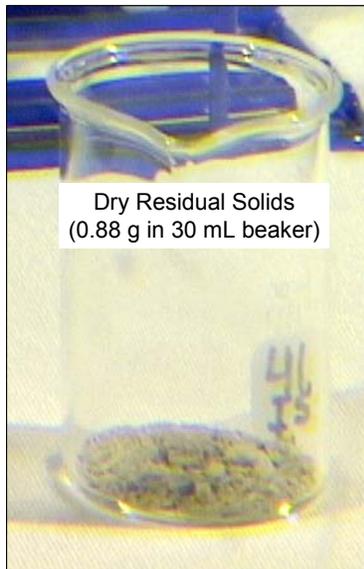


Figure 7: Dried insoluble solids resulting from the dissolution of Tank 41H material.

Table 4 and Table 5 contain the analytical results for the wet as-received saltcake and the dry insoluble solids, respectively. Values reported as their detection limits are preceded by “<” and values that contain an average of detectable concentrations and detection limits are preceded by “</=”. Results are reported as the average of analyses of duplicate dissolutions by aqua regia or peroxide fusion in most cases, with a triplicate dissolution used for the aqua regia of the insoluble solids. The peroxide fusion digestion method introduces significant Na, Zr, and Ag to the sample, and thus no peroxide fusion results are included for these analytes. Additionally, several other analytes appeared at significant quantities in the peroxide fusion blank, and those results are also not included. Due to their relatively low levels compared to Pu-238, the Pu-239/240 results for the aqua regia replicates were widely scattered and are not reported. An overall “Best/Combined” average is calculated as either the average of results from both dissolution methods or, where one dissolution method did not provide reliable results, the results from the better of the two dissolution methods. The “Best/Combined” values are reported as the averages and standard deviations of two to five replicates.

The total uranium is calculated by summing the ICP-MS isotopes that can be primarily attributed to uranium when they are present above their detection limits. Typically, this includes masses 233, 234, 235, 236, and 238. The mass of Pu-238, calculated from radiochemistry analysis, is subtracted from mass 238. The uranium-235 enrichment is calculated by dividing mass 235 by the total uranium. This data is supplied as input to NCSE calculations for salt dissolution.¹²

Table 18 contains the analysis of the dissolved salt solutions decanted during each of the three dissolution stages. These results can be used to calculate how much sodium and other materials remained in the heels of the intermediate dissolution steps.

Table 4: Analytical results for as-received Tank 41H saltcake (Top 3" of HTF-E-03-145)

Analyte	Units	Aqua Regia		Peroxide Fusion		Combined/Best	
		Average	St. Dev.	Average	St. Dev.	Average	St.Dev.
Metals (ICP-ES)							
Ag	wt %	< 3.08E-04	--	--	--	< 3.08E-04	--
Al	wt %	7.49E-01	1.48E-01	7.86E-01	2.09E-01	7.67E-01	1.50E-01
B	wt %	< 5.09E-03	--	< 5.58E-03	--	< 5.33E-03	--
Ba	wt %	5.84E-04	1.39E-04	--	--	5.84E-04	1.39E-04
Ca	wt %	2.51E-03	3.73E-04	--	--	2.51E-03	3.73E-04
Cd	wt %	< 9.25E-04	--	< 1.01E-03	--	< 9.69E-04	--
Ce	wt %	9.79E-03	1.08E-03	--	--	9.79E-03	1.08E-03
Cr	wt %	9.91E-03	1.43E-03	1.19E-02	2.9E-03	1.09E-02	2.2E-03
Cu	wt %	< 7.71E-04	--	--	--	< 7.71E-04	--
Fe	wt %	1.25E-02	4.5E-03	2.48E-02	6.4E-03	1.87E-02	8.4E-03
Gd	wt %	1.10E-03	2.7E-04	--	--	1.10E-03	2.7E-04
K	wt %	1.93E-01	1.63E-01	--	--	1.93E-01	1.63E-01
La	wt %	1.20E-03	3E-05	--	--	1.20E-03	3E-05
Li	wt %	2.20E-03	2.6E-04	--	--	2.20E-03	2.6E-04
Mg	wt %	2.27E-04	2.6E-05	--	--	2.27E-04	2.6E-05
Mn	wt %	1.48E-03	2E-05	2.18E-03	5.7E-04	1.83E-03	5.2E-04
Mo	wt %	< 5.16E-03	--	<=6.12E-03	2.14E-03	<=5.64E-03	1.55E-03
Na	wt %	2.04E+01	1.2E+00	--	--	2.04E+01	1.2E+00
Ni	wt %	< 3.47E-03	--	< 3.80E-03	--	< 3.63E-03	--
P	wt %	4.22E-02	3.3E-03	7.18E-02	2.19E-02	5.70E-02	2.13E-02
Pb	wt %	< 2.20E-02	--	< 2.42E-02	--	2.31E-02	5.1E-03
S	wt %	2.49E-01	4.1E-02	--	--	2.49E-01	4.1E-02
Sb	wt %	< 5.47E-03	--	--	--	< 5.47E-03	--
Si	wt %	<=1.98E-03	4.2E-04	--	--	1.98E-03	4.2E-04
Sn	wt %	< 8.79E-03	--	--	--	< 8.79E-03	--
Sr	wt %	1.35E-03	3.0E-04	--	--	1.35E-03	3.0E-04
Ti	wt %	< 1.00E-03	--	--	--	< 1.00E-03	--
V	wt %	1.13E-03	6E-05	--	--	1.13E-03	6E-05
Zn	wt %	< 1.54E-04	--	--	--	< 1.54E-04	--
Zr	wt %	< 1.08E-03	--	--	--	< 1.08E-03	--
Radio-Chemistry							
¹³⁷ Cs	pCi/g	4.86E+07	1.36E+07	4.16E+07	1.06E+07	4.51E+07	1.07E+07
²³⁸ Pu	pCi/g	3.60E+05	1.8E+04	7.64E+05	2.13E+05	5.62E+05	2.64E+05
^{239/240} Pu	pCi/g	--	--	< 3.56E+03	--	< 3.56E+03	--
²⁴¹ Pu	wt %	< 5.12E-08	--	2.11E-07	1.26E-07	<=1.31E-07	1.24E-07
ICP-MS							
Mass 59 (Co)	wt %	< 6.4E-06	--	--	--	< 6.4E-06	--
Mass 99 (Tc,Ru)	wt %	1.42E-04	3.8E-05	1.50E-04	4.2E-05	1.46E-04	3.3E-05
Mass 101 (Ru)	wt %	1.2E-05	3E-06	1.6E-05	3E-07	1.4E-05	3E-06
Mass 133 (Cs)	wt %	1.21E-04	3.0E-05	--	--	1.21E-04	3.0E-05
Mass 135 (Cs,Ba)	wt %	1.8E-05	4E-06	--	--	1.8E-05	4E-06
Mass 137 (Cs,Ba)	wt %	1.29E-04	1.7E-05	--	--	1.29E-04	1.7E-05
Mass 138 (Ba)	wt %	< 1.8E-05	--	--	--	< 3.4E-05	--
Mass 230 – 231	wt %	< 2.2E-06	--	< 4.7E-06	--	< 3.4E-06	--
Mass 232 (Th,U)	wt %	< 2.2E-06	--	5.3E-06	2.0E-06	<=3.7E-06	--
Mass 233 (U)	wt %	3.3E-06	4E-07	5.7E-06	2.6E-06	4.49E-06	2.01E-06
Mass 234 (U)	wt %	2.30E-05	1.4E-06	3.38E-05	6.1E-06	2.84E-05	7.2E-06
Mass 235 (U)	wt %	6.18E-05	2.1E-06	9.02E-05	2.36E-05	7.60E-05	2.14E-05
Mass 236 (U)	wt %	2.76E-05	1.4E-06	3.84E-05	1.02E-05	3.30E-05	8.6E-06
Mass 237 (Np)	wt %	1.63E-05	5E-07	2.08E-05	7.7E-06	1.86E-05	5.2E-06
Mass 238 (U,Pu)	wt %	3.94E-04	1.6E-05	5.94E-04	1.91E-04	4.94E-04	1.60E-04
Mass 239 (Pu)	wt %	<=2.3E-06	4E-07	5.4E-06	2.2E-06	<=3.9E-06	2.2E-06
Mass 240 – 245	wt %	< 2.2E-06	--	< 4.7E-06	--	< 3.4E-06	--
Uranium Summary							
Total U	wt %	5.07E-04	2.1E-05	7.55E-04	2.35E-04	6.31E-04	1.97E-04
²³⁵ U Enrichment	%	12.2%	0.1%	12.0%	0.6%	12.1%	0.4%

Table 5: Analytical results for the dried residual insoluble solids resulting from the dissolution of Tank 41H saltcake (Top 3" of HTF-E-03-145)

Analyte	Units	Aqua Regia		Peroxide Fusion		Combined/Best	
		Average	St. Dev.	Average	St. Dev.	Average	St.Dev.
Metals (ICP-ES)							
Ag	wt %	< 3.12E-03	--	--	--	< 3.12E-03	--
Al	wt %	2.77E+01	5E-01	2.69E+01	1.4E+00	2.74E+01	9E-01
B	wt %	< 5.15E-02	--	< 5.88E-02	--	< 5.44E-02	--
Ba	wt %	1.59E-02	1.7E-03	--	--	1.59E-02	1.7E-03
Ca	wt %	2.24E-01	3E-03	--	--	2.24E-01	3E-03
Cd	wt %	< 9.37E-03	--	< 1.07E-02	--	< 9.89E-03	--
Ce	wt %	7.23E-02	1.93E-02	--	--	7.23E-02	1.93E-02
Cr	wt %	7.11E-01	3.0E-02	6.66E-01	5E-03	6.93E-01	3.2E-02
Cu	wt %	8.98E-03	1.54E-03	--	--	8.98E-03	1.54E-03
Fe	wt %	2.88E+00	9.8E-01	2.22E+00	8.7E-01	2.62E+00	9.0E-01
Gd	wt %	9.63E-03	2.83E-03	--	--	9.63E-03	2.83E-03
K	wt %	< 6.93E-01	--	--	--	< 6.93E-01	--
La	wt %	8.55E-03	2.12E-03	--	--	8.55E-03	2.12E-03
Li	wt %	1.22E-02	5.4E-03	--	--	1.22E-02	5.4E-03
Mg	wt %	1.70E-02	6E-04	--	--	1.70E-02	6E-04
Mn	wt %	2.02E-01	1.5E-02	1.86E-01	1.1E-02	1.95E-01	1.5E-02
Mo	wt %	< 5.26E-02	--	7.50E-02	1.34E-02	<=6.16E-02	1.58E-02
Na	wt %	9.72E-01	1.31E-01	--	--	9.72E-01	1.31E-01
Ni	wt %	9.02E-02	1.92E-02	7.11E-02	1.51E-02	8.25E-02	1.87E-02
P	wt %	< 3.27E-01	--	< 3.73E-01	--	< 3.46E-01	--
Pb	wt %	< 2.23E-01	--	< 2.55E-01	--	< 2.36E-01	--
S	wt %	< 9.92E-02	--	--	--	< 9.92E-02	--
Sb	wt %	1.09E-01	1.1E-02	--	--	1.09E-01	1.1E-02
Si	wt %	2.18E-01	3.3E-02	--	--	2.18E-01	3.3E-02
Sn	wt %	< 8.90E-02	--	--	--	< 8.90E-02	--
Sr	wt %	5.39E-02	6E-04	--	--	5.39E-02	6E-04
Ti	wt %	< 1.02E-02	--	--	--	< 1.02E-02	--
V	wt %	1.91E-02	4.6E-03	--	--	1.91E-02	4.6E-03
Zn	wt %	1.03E-02	5E-04	--	--	1.03E-02	5E-04
Zr	wt %	< 1.09E-02	--	--	--	< 1.09E-02	--
Radio-Chemistry							
¹³⁷ Cs	pCi/g	9.28E+07	1.34E+07	1.12E+08	2E+06	1.01E+08	1.4E+07
²³⁸ Pu	pCi/g	4.75E+07	1.17E+07	5.97E+07	3.5E+06	5.24E+07	1.08E+07
^{239/240} Pu	pCi/g	--	--	9.71E+04	1.50E+04	9.71E+04	1.5E+04
²⁴¹ Pu	wt %	< 2.22E-06	--	2.66E-06	5.5E-07	<=2.39E-06	1.46E-06
ICP-MS							
Mass 59 (Co)	wt %	1.63E-03	4.0E-04	--	--	1.63E-03	4.0E-04
Mass 99 (Tc,Ru)	wt %	3.52E-03	2.9E-04	3.65E-03	2.7E-04	3.57E-03	2.6E-04
Mass 101 (Ru)	wt %	4.11E-04	3.7E-05	5.18E-04	6.9E-05	4.54E-04	7.3E-05
Mass 133 (Cs)	wt %	2.33E-04	3.7E-05	--	--	2.33E-04	3.7E-05
Mass 135 (Cs,Ba)	wt %	1.21E-04	1.4E-05	--	--	1.21E-04	1.4E-05
Mass 137 (Cs,Ba)	wt %	9.98E-03	2.1E-04	--	--	9.98E-03	2.1E-04
Mass 138 (Ba)	wt %	7.83E-04	2.0E-05	--	--	7.83E-04	2.0E-05
Mass 230 – 231	wt %	< 2.2E-05	--	< 5.0E-05	--	< 3.3E-05	--
Mass 232 (Th,U)	wt %	7.1E-05	5E-06	1.74E-04	9E-06	1.12E-04	5.7E-05
Mass 233 (U)	wt %	3.95E-04	1.2E-05	4.65E-04	2.4E-05	4.23E-04	5.1E-05
Mass 234 (U)	wt %	2.32E-03	4E-05	2.67E-03	1E-04	2.46E-03	2.1E-04
Mass 235 (U)	wt %	7.07E-03	8E-05	7.62E-03	5.9E-04	7.29E-03	4.2E-04
Mass 236 (U)	wt %	2.83E-03	5E-05	3.08E-03	2.0E-04	2.93E-03	1.7E-04
Mass 237 (Np)	wt %	1.98E-03	5E-06	2.10E-03	1.3E-04	2.03E-03	1.0E-04
Mass 238 (U,Pu)	wt %	4.50E-02	6E-05	5.04E-02	3.2E-03	4.72E-02	3.7E-03
Mass 239 (Pu)	wt %	2.52E-04	2.0E-03	3.48E-04	6E-06	2.90E-04	5.4E-05
Mass 240 – 245	wt %	< 2.2E-05	--	< 5.0E-05	--	< 3.3E-05	--
Uranium Summary							
Total U	wt %	5.73E-02	2.1E-03	6.39E-02	4.1E-03	6.00E-02	4.4E-03
²³⁵ U Enrichment	%	12.3%	0.4%	11.9%	0.2%	12.2%	0.4%
Equivalent ²³⁵ U	wt %	8.04E-03	7.8E-05	8.84E-03	6E-04	8.36E-03	5E-04
Na/Equiv. ²³⁵ U	wt. frac.	121	17	--	--	121	17
Cr/Equiv. ²³⁵ U	wt. frac.	88.5	4.0	75.4	4.6	83.3	8.0
Fe/Equiv. ²³⁵ U	wt. frac.	360	126	248	81	315	116
Mn/Equiv. ²³⁵ U	wt. frac.	25.1	2.1	21.0	0.2	23.5	2.7

Comparison of Components and Streams

The interstitial liquid drained from HTF-E-03-146 was consistent with previous C1/C3-riser SL sample analysis performed after the dissolution campaign. The density, soluble solids content, sodium content, nitrite content, total base, and ^{137}Cs activity were all slightly higher in the drained IL than in the C1/C3-riser SL samples.

A full comparison of the Tank 41H post-dissolution samples will be included in a revision of this report. Additionally, a comparison will be made of the Tank 41H post-dissolution saltcake samples with the drained pre-dissolution samples (HTF-E-03-033 – 035).

Draining

Interstitial liquid (IL) was drained from 297.5 gm of sample HTF-E-03-146 using 5 inches of mercury vacuum for 19 days and 12 inches of mercury for 14 days, respectively, using the apparatus shown in Figure 5. Five inches of mercury vacuum at 20 °C is calculated equivalent to the suction produced by a residual drained interstitial liquid column of 53 inches at a density of 1.427 g/mL. The 31 days of drainage removed 31.3 g of interstitial liquid from sample HTF-E-03-146. This amount of IL exceeds the amount that would be predicted (21.77gm) based on water content analysis of the as-received sample. The as-received water content analysis was conducted on a sample collected from the bottom of sample HTF-E-03-146 which appeared dry, was hard packed, and difficult to remove. These observations, along with the disparity in IL content measurements, indicate that the material collected from HTF-E-03-146 for the as-received sample is not representative of the entire contents of the sample.

During draining at 12" Hg, a leak developed in the volumetric flask seal, increasing air flow in the volumetric flask. This caused evaporation of water from the drained IL and influenced the draining data. A correction was applied to the draining at 12" Hg vacuum to account for this event.

Samples were collected from HTF-E-03-146 after drainage to determine the final drained IL content of the salt. Using the amount of IL drained from the sample and the measured post drainage water content of 0.024 g/g (i.e., g water per g saltcake), the initial IL content of the salt in the tube for the drainage test was calculated to be 44.21 g. This equates to removal of 71% of the IL during vacuum extraction (see Figure 8). The initial water content of the sample was also calculated using drainage data and was determined to be 0.083 g/g. This amount of drainage represents the maximum that would be expected for salt in the tank at a level 53 inches above the liquid in the caisson when the liquid level is no longer rising after successive pumping intervals. If the samples are assumed to have been 100% saturated when drainage began, then the sample can be considered 40% saturated after drainage which is less than the saturation that would be predicted for a sandy loam of 66% under the same conditions.

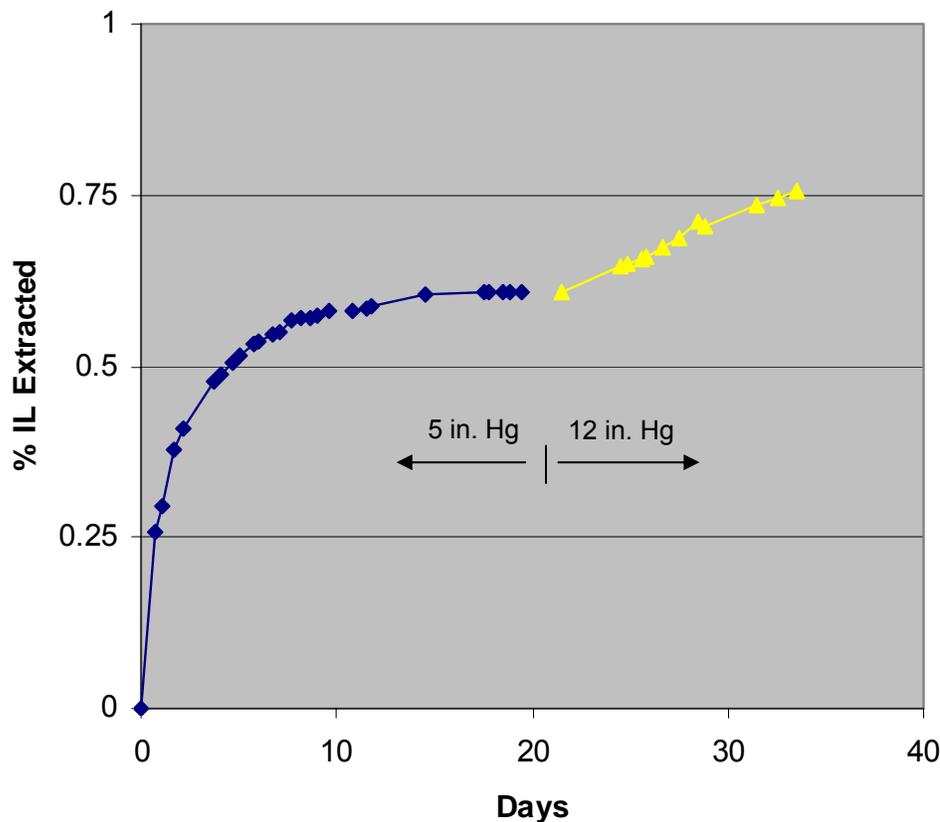


Figure 8: Vacuum draining data for HTF-E-03-146

Microscopy and Spectroscopy

Bulk Saltcake from HTF-E-03-146

The Tank 41H saltcake from the bottom of sample HTF-E-03-146, the middle sample of the three post-dissolution saltcake core samples, was investigated by SEM and XRD. The sub-rounded appearance of grains, the lack of coatings by secondary precipitates, and the crevices and indentations in some grains are among the most notable characteristics of this sample. These features are consistent with mineral dissolution and are likely related to Tank 41H dissolution event.

Grains in the HTF-E-03-146 sample appear to range in size from 10 to 200 μm (Figure 9). Some grains are dominated by single crystals (Figure 11), whereas others look as if they are a mass of smaller crystals that may be intergrown or cemented together (Figure 13 and Figure 14).

The HTF-E-03-146 sample has a similar mineralogy to the initial sample taken from this tank (pre-dissolution) and to some other tank samples. As seen in Figure 16, XRD indicated the presence of primarily sodium nitrate with minor amounts of a sodium carbonate and zeolite. XRD conducted on a sample collected before the tank dissolution suggested the presence of burkeite ($\text{Na}_6(\text{SO}_4)_2\text{CO}_3$) in addition to the sodium nitrate.³ Elemental spectra from this analysis indicate that several of the larger precipitates are dominated by sodium and nitrogen, likely reflecting the sodium nitrate mineral (Figure 10).

Many of the grains in this sample appear to have features that are consistent with mineral dissolution. Examples of these features are discussed below.

Figure 11 shows an example of some of the grains that appear to be dominated by single crystals. The elemental spectrum in Figure 10 (Spot 4) suggests that the large grain labeled "A" consists of sodium nitrate. Sodium nitrate typically has a blocky, rectangular (or rhombohedral) shape (based on observations of other SRS tank samples and Hanford simulant NaNO_3 samples). However this grain has predominately rounded corners and edges (except for one side) resulting from dissolution along the edges of the grain. The side with the flatter face and angular edges appears to have not undergone the same extent of dissolution as the rest of the grain. It may be a lower solubility phase that was coated by a higher solubility phase, or this side may have been more protected (e.g. in a restricted pore space) thereby having less contact time with the dissolution water.

The grain labeled "B" in the lower right corner of Figure 11 also has a peculiar appearance, which may reflect preferential dissolution. Small crevices or shallow indentations are evident in the secondary electron and backscattered images. These features could reflect differences in the chemistry and/or structure of the grain resulting in preferential dissolution along the small crevices. Microporosity within the original grain may also have contributed to preferential dissolution by providing more surface area and possibly access to more soluble parts of the grain.

Figure 12 shows secondary electron and backscattered images of some of the larger grains observed in saltcake samples from Tank 10H and Tank 41H (pre-dissolution) for comparison with this sample.^{3,9} Based on EDS and XRD data, these larger grains likely consist of sodium nitrate similar to the larger grains in this sample, HTF-E-03-146. However, these samples have numerous small secondary precipitates that coat the faces and edges of grains. Secondary precipitates appear to be few or absent from the larger crystals in the Tank 41H post-dissolution sample (HTF-E-03-146). In addition to the prevalent secondary precipitates, the precipitates (both large and small) in the Tank 10H and Tank 41H (pre-dissolution) tend to exhibit more angular and sharp edges compared to those in the Tank 41H post-dissolution sample.

Figure 13 and Figure 14 provide examples of some grains that consist of masses of smaller crystals. These crystals also show evidence of dissolution in their rounded edges and smooth crystal faces. Figure 14 provides an example of porosity, which may have been created or enhanced by the dissolution. As noted before, microporosity within grains can provide more surface area and access to more soluble parts of the grain.

Elemental spectra indicate that some of the smaller crystals have slightly different compositions (Figure 13 and Figure 14). In Figure 13, sodium and nitrogen were detected at Spot 7, suggesting that part of this grain consists of sodium nitrate. Spot 6 in Figure 13 and Spot 8 in Figure 14 showed the presence of sodium and sulfur with minor aluminum. The occurrence of sulfur is consistent with the XRD analysis of the Tank 41H pre-tank dissolution sample, which indicated the presence of burkeite. The crystal at Spot 9 in Figure 14 has a slightly different chemical composition and morphology. It appears to have a platy shape and consists primarily of sodium and aluminum (the small sulfur peak may be from an adjacent grain). This crystal may represent sodium aluminate or another sodium-and-aluminum-bearing phase and it is reminiscent of the thin, platy crystals observed in other SRS HLW saltcake samples (e.g. Tank 3F, Figure 15).¹³ It is unclear (particularly with Spots 6, 8 and 9) whether some of these precipitates were part of the original saltcake (i.e. pre-tank dissolution) or whether they may be precipitates formed as overgrowths or cements from the flush water. Mineral dissolution would be expected to occur while the flush water solution is undersaturated. However, if the flush water or residual flush water becomes oversaturated, or temperatures fluctuate, mineral precipitation may occur.

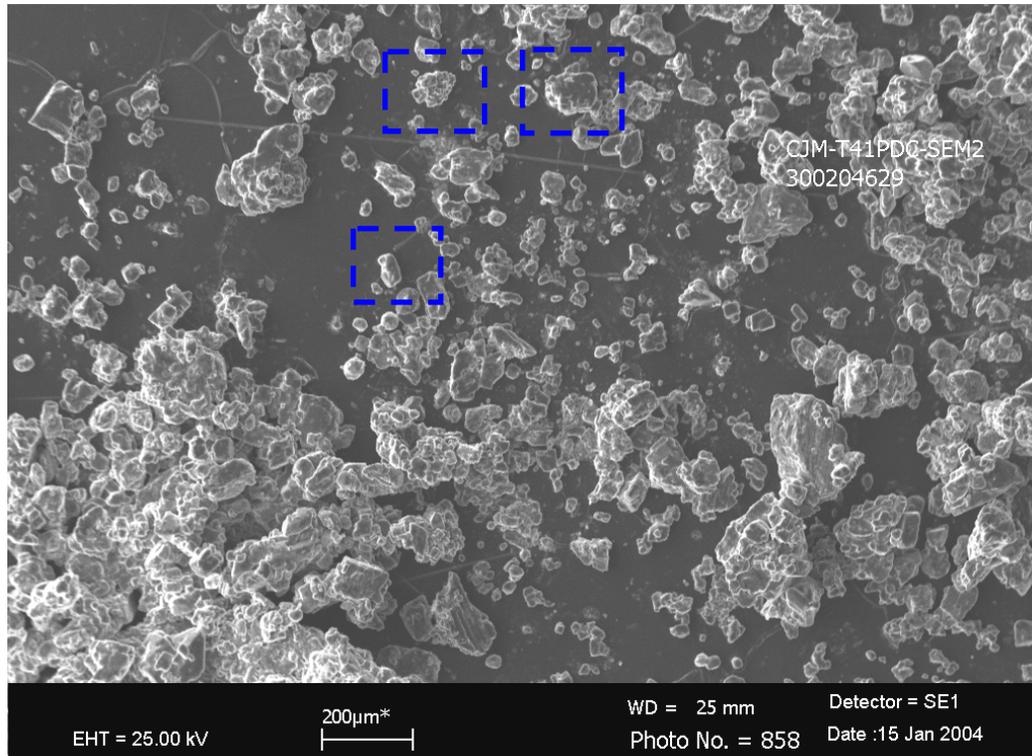


Figure 9: Scanning electron photomicrograph of HTF- E-03-146. Grains vary in size from 10 to 200 µm. Highlighted areas correspond to locations studied in more detail (Figs. 11, 13 & 14).

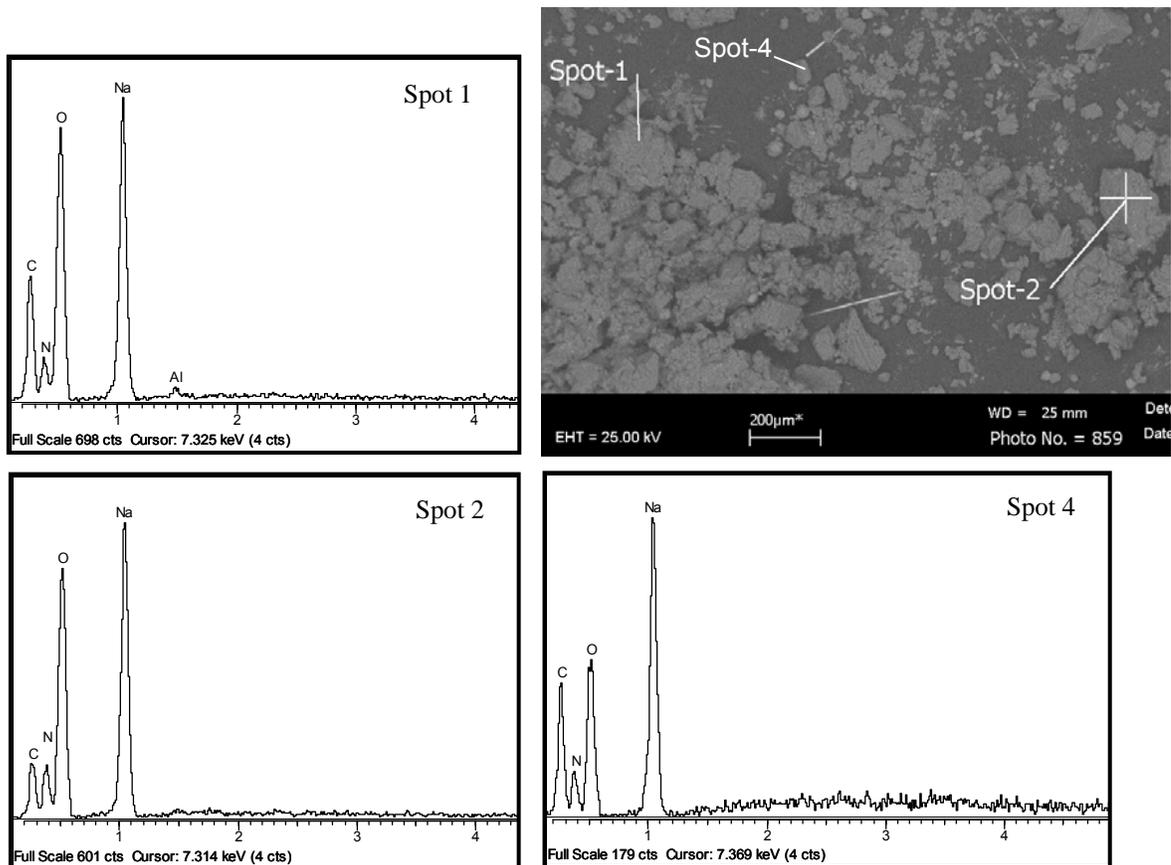


Figure 10: Elemental spectra for the larger grains; consistent with sodium nitrate mineralogy.

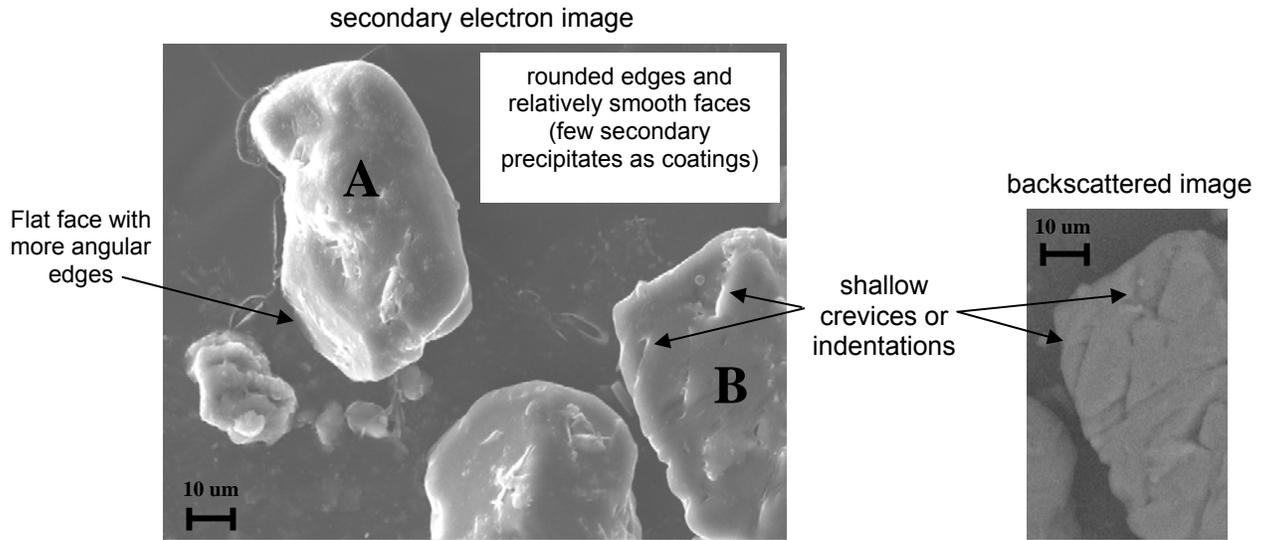


Figure 11: Example of larger grains in sample HTF-E-03-146 with possible dissolution features identified.

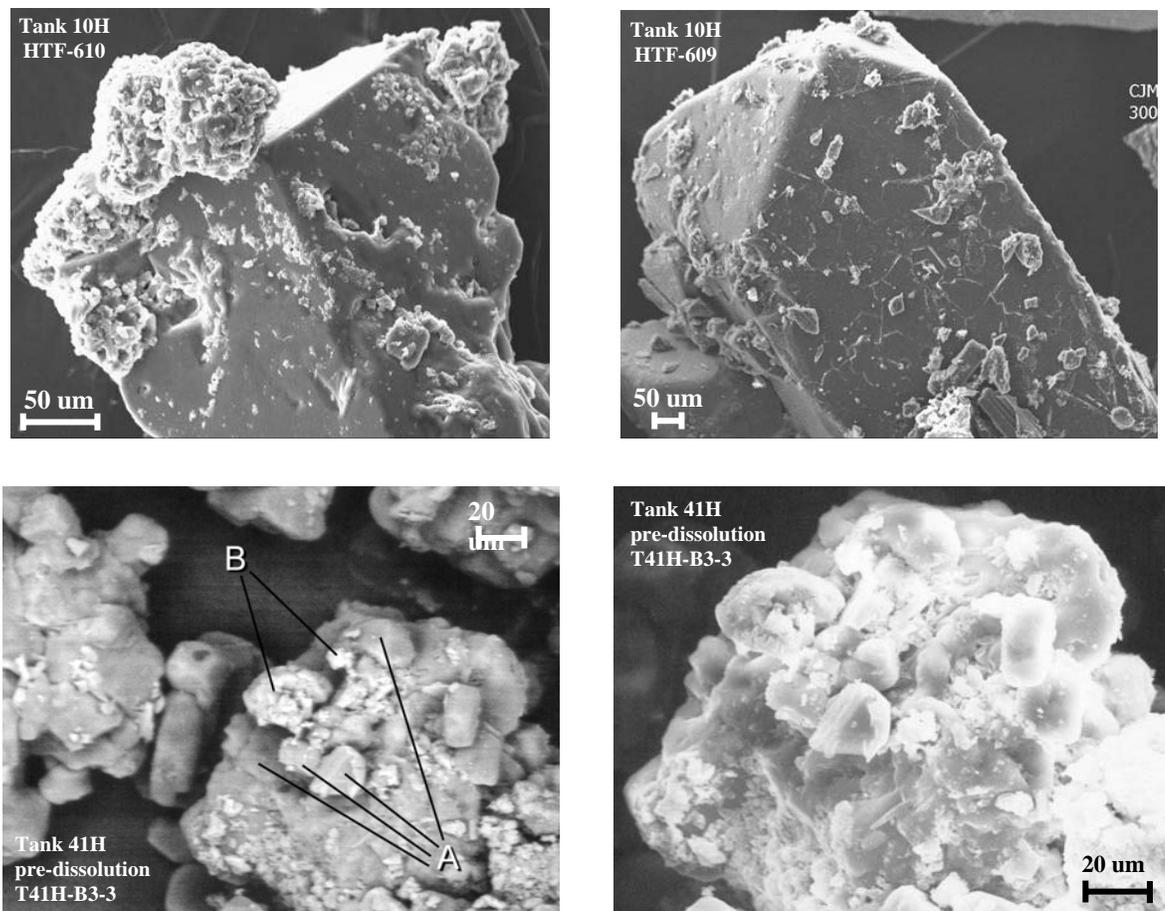


Figure 12: Larger grains in Tank10H and Tank41H (pre-dissolution) for comparison. Note angular and sharp edges of primary and secondary precipitates. Small secondary precipitates are prevalent and coat or mask the faces of larger crystals.

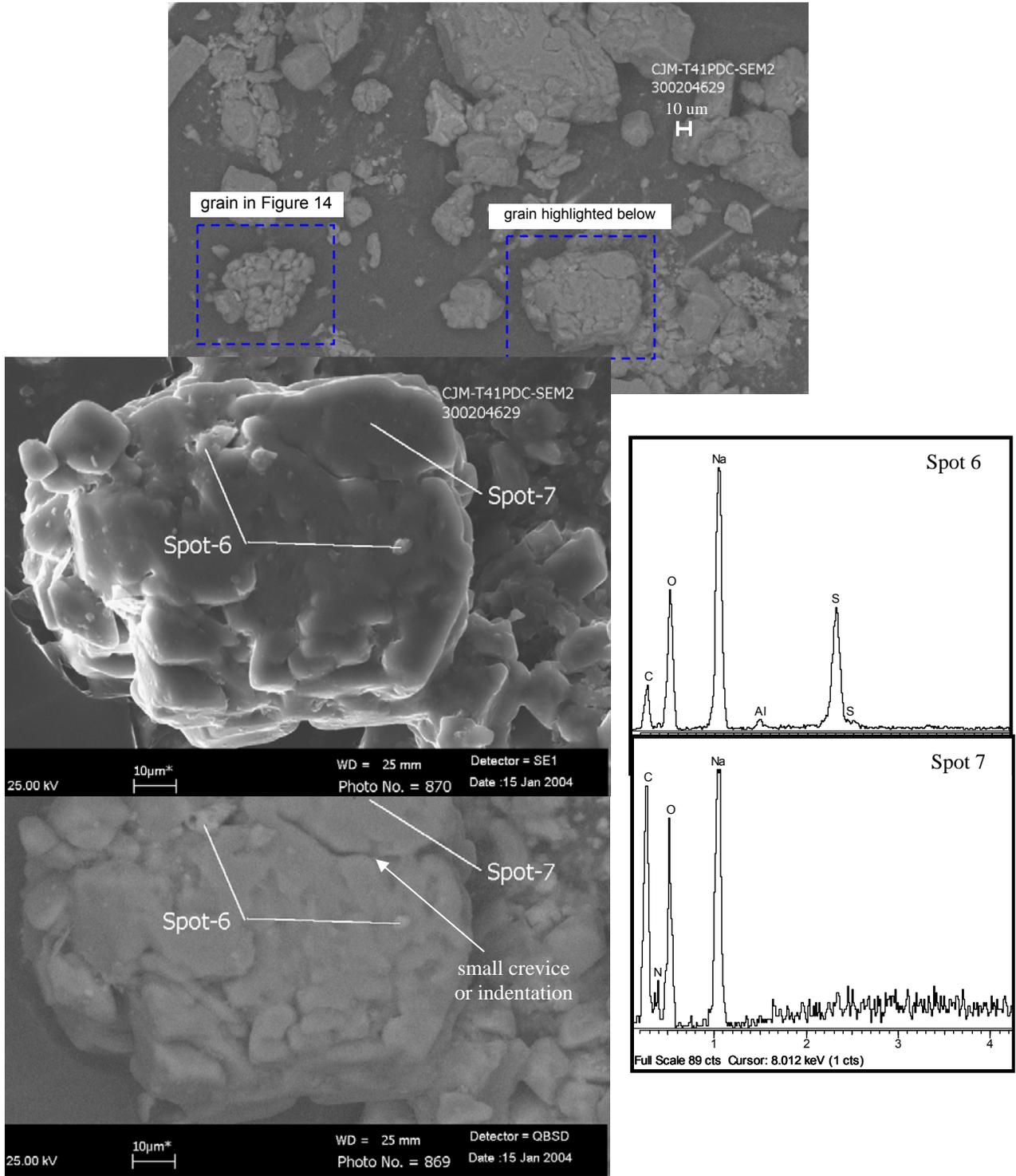


Figure 13: Example of a grain consisting of a mass of smaller crystals. Top and bottom images are backscattered; middle is secondary electron image. Crystals show evidence of dissolution in their rounded edges and smooth faces.

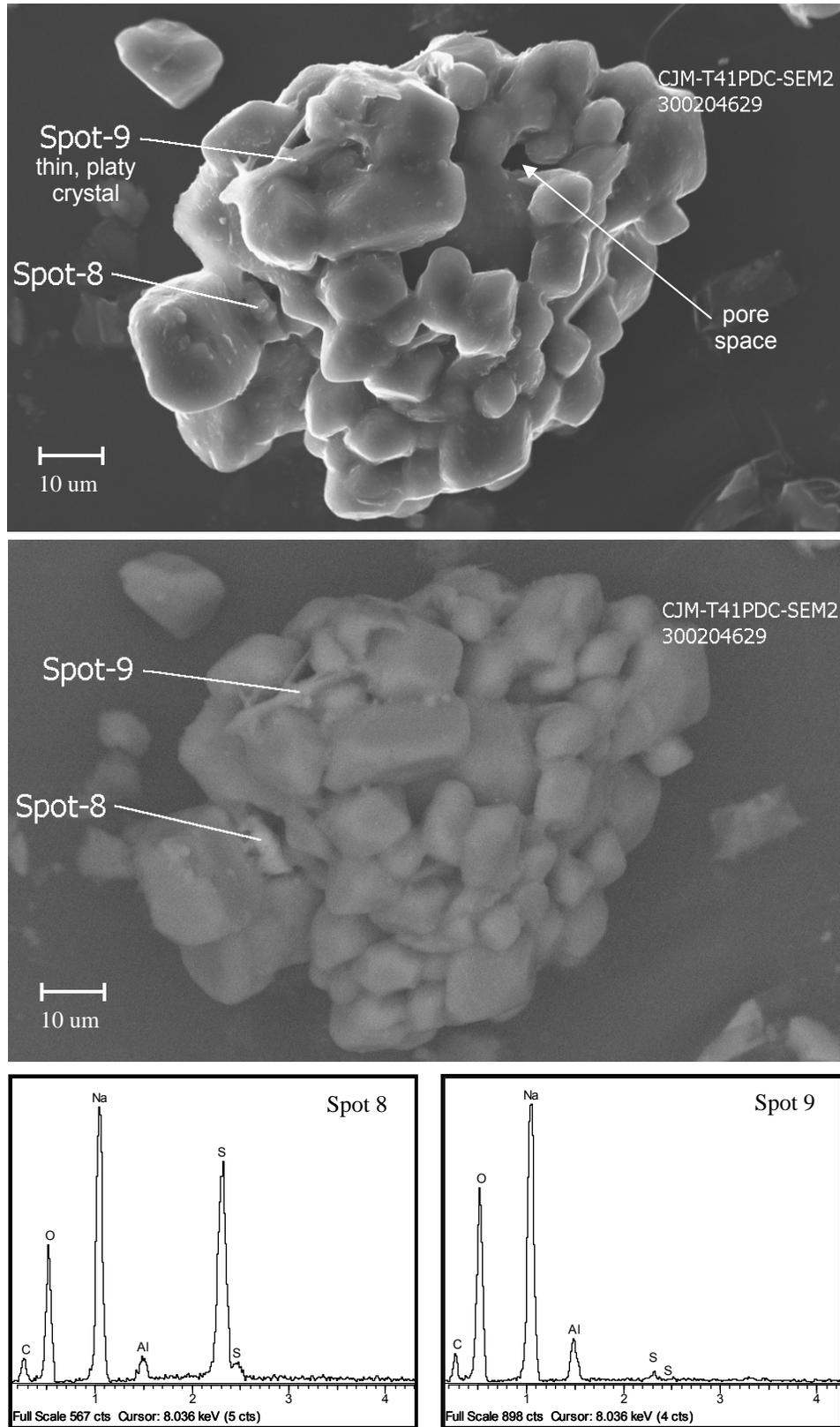


Figure 14: Example of a grain consisting of a mass of smaller crystals (secondary electron image on top; backscattered image on bottom). Crystals show evidence of dissolution in their rounded edges, smooth crystal faces and pore space. Spots 8 and 9 vary in morphology and chemical composition.

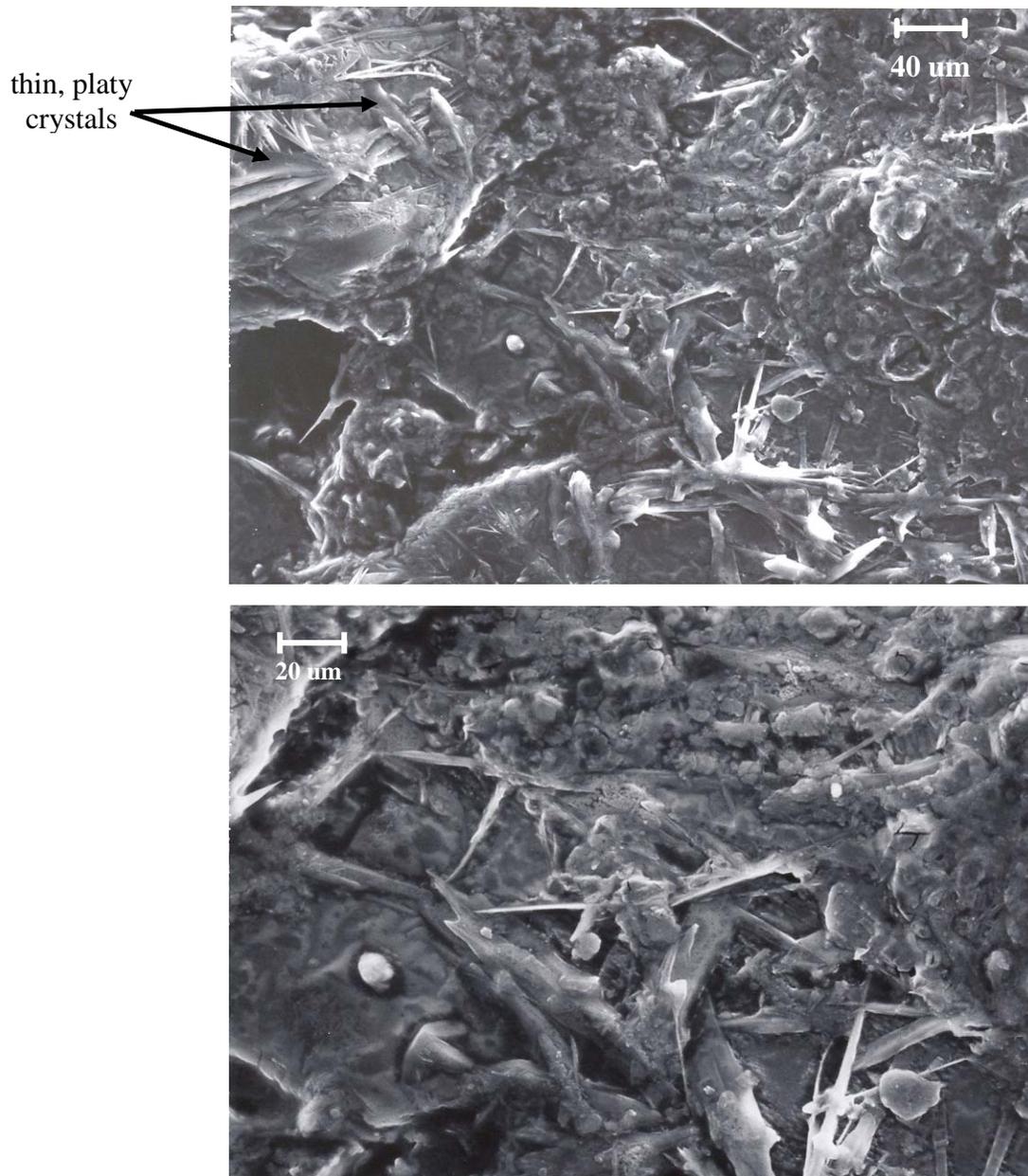


Figure 15: Examples of thin, platy crystals in Tank 3F saltcake sample (secondary electron images).

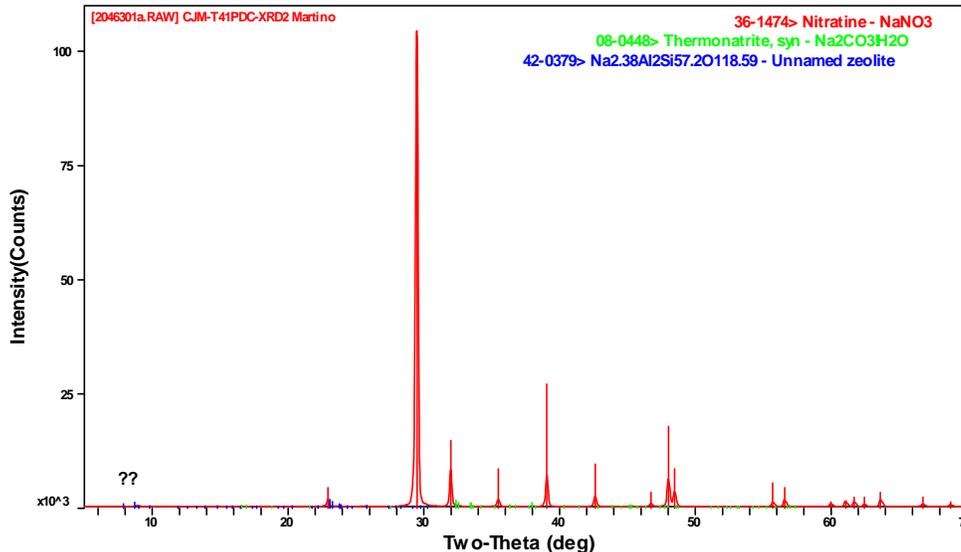


Figure 16: XRD of material from the bottom of HTF-E-03-146

Insoluble Solids in HTF-E-03-145

From the XRD analysis in Figure 17, the residual insoluble solids were composed primarily of the aluminum hydroxide polymorphs gibbsite and bayerite, which both have the chemical composition $\text{Al}(\text{OH})_3$ (also written as $\text{Al}_2\text{O}_3 \cdot 3\text{H}_2\text{O}$). This observation is consistent with the residual solids resulting from the June 2002 salt-well sample.⁸ Gibbsite appears to be more prevalent in the sample analyzed, but the gibbsite/bayerite ratio might have been influenced by the drying of the solids before analysis. If all of the aluminum from the ICP-ES results are taken as gibbsite and bayerite, the residual insoluble solids contain 80 ± 3 wt % gibbsite and bayerite. Additionally, the residual insoluble solids contained roughly 1 wt % of a sodium-aluminosilicate zeolite with an Na:Al:Si ratio of 2:2:1.68.

Figure 18 contains SEM micrographs from three representative regions of the dried residual insoluble solids from the dissolution of Tank 41H salt. The images on the left are the backscattered electron micrographs and the images on the right are the corresponding secondary electron micrographs. Figure 19 contains the EDS spectra for the spots labeled in Figure 18. In all regions that were scanned, solids containing aluminum and oxygen (with minor impurities) were predominant. This major component, which appears dark on the SEM backscatter images (Spots 2, 3, and 14), is likely aluminum hydroxide (gibbsite and bayerite). Most of the aluminum hydroxide particles appear to be in the 1 to 10 μm range, with possibly some clusters or agglomerations that are $>10 \mu\text{m}$.

Several brighter spots appear in the SEM backscatter images that correspond to heavier elements present in the predominantly aluminum hydroxide solids (Figure 18). All locations investigated, except Spot 1 and Spot 8, are predominantly aluminum and oxygen. Spot 1 corresponds to a unique particle, a $>100 \mu\text{m}$ -long shard that is apparently stainless steel (Fe, Cr, Ni). The remainder of the brighter areas were particles with the same size order as the bulk of the aluminum hydroxide particles. Spots 6, 7, and 15 correspond to areas (3 to 5 μm diameter) that, in addition to aluminum and oxygen, are seen to contain uranium and other minor components. Spots 5 and 13 also contain mercury and other metals. Spot 4 is rich in chromium and Spot 8 is rich in iron. Areas of the sample also contained Na, Si, C, and Cl.

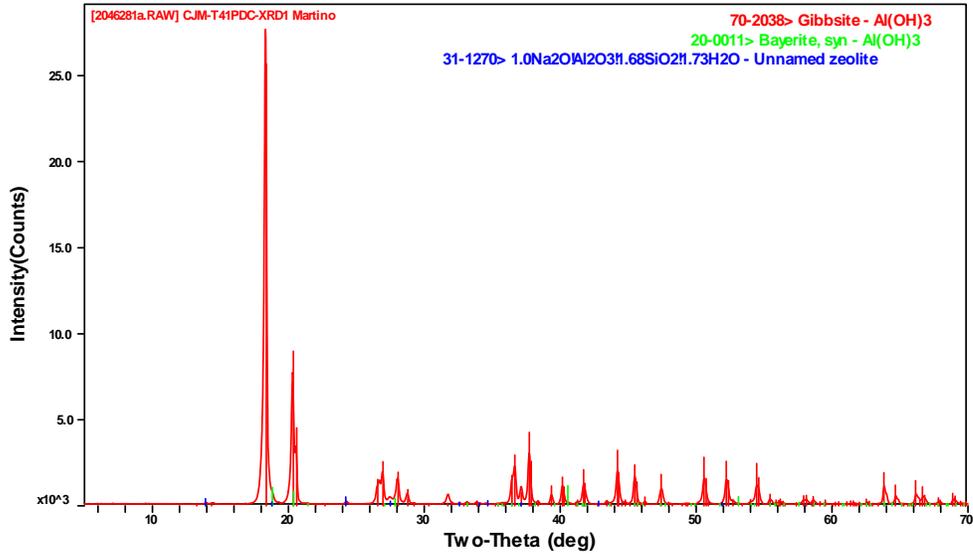


Figure 17: XRD of the residual insoluble solids from resultant from dissolution of material from the top of HTF-E-03-145

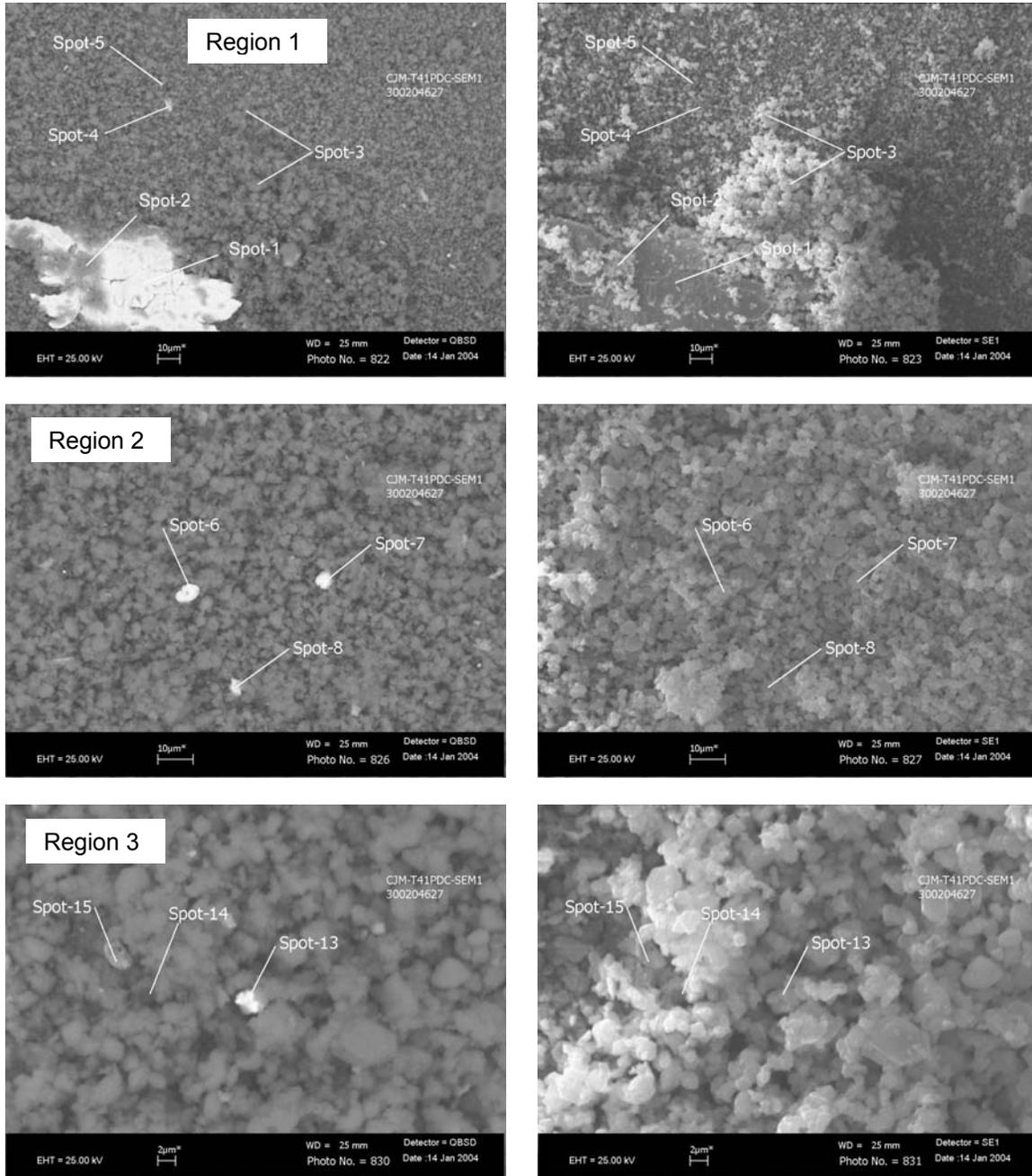


Figure 18: Scanning electron micrographs of residual insoluble solids from the dissolution of salt from the top of sample HTF-E-03-145. Micrographs of three regions are presented vertically, with corresponding backscattered electron micrographs (left) and secondary electron micrographs (right).

Insoluble Solids in Supernate Samples HTF-E-03-124 and 130/2

Figure 20 and Figure 21 contain the SEM/EDS results from investigation of insoluble solids filtered from samples HTF-E-03-124 and HTF-E-03-130/2, respectively. In Figure 21, Spot A is equivalent to Spot C. HTF-E-03-124 contained several sodium salts containing phosphorous and sulfur. Additionally, some aluminum was noted. HTF-E-03-130/2 contained primarily salts where only sodium was noted (likely sodium nitrate based on the crystal shape). Note that the EDS used for this analysis, unlike that used for the results in the previous sections, is not capable of detecting nitrogen. Additionally, particles of aluminum-containing material and sodium/sulfur-containing material were noted.

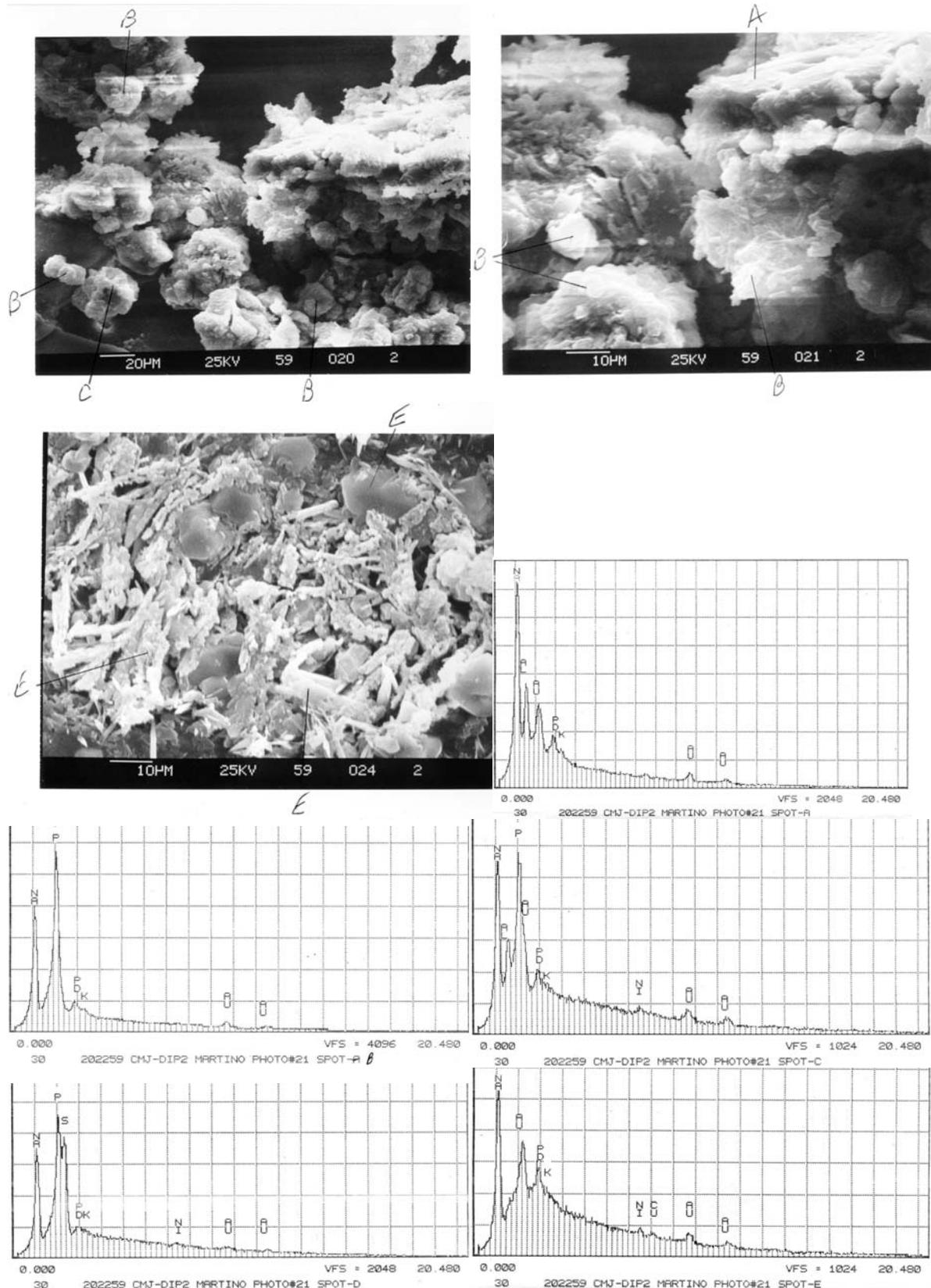


Figure 20: Insoluble solids filtered from C1 VDS SL Sample HTF-E-03-130,32

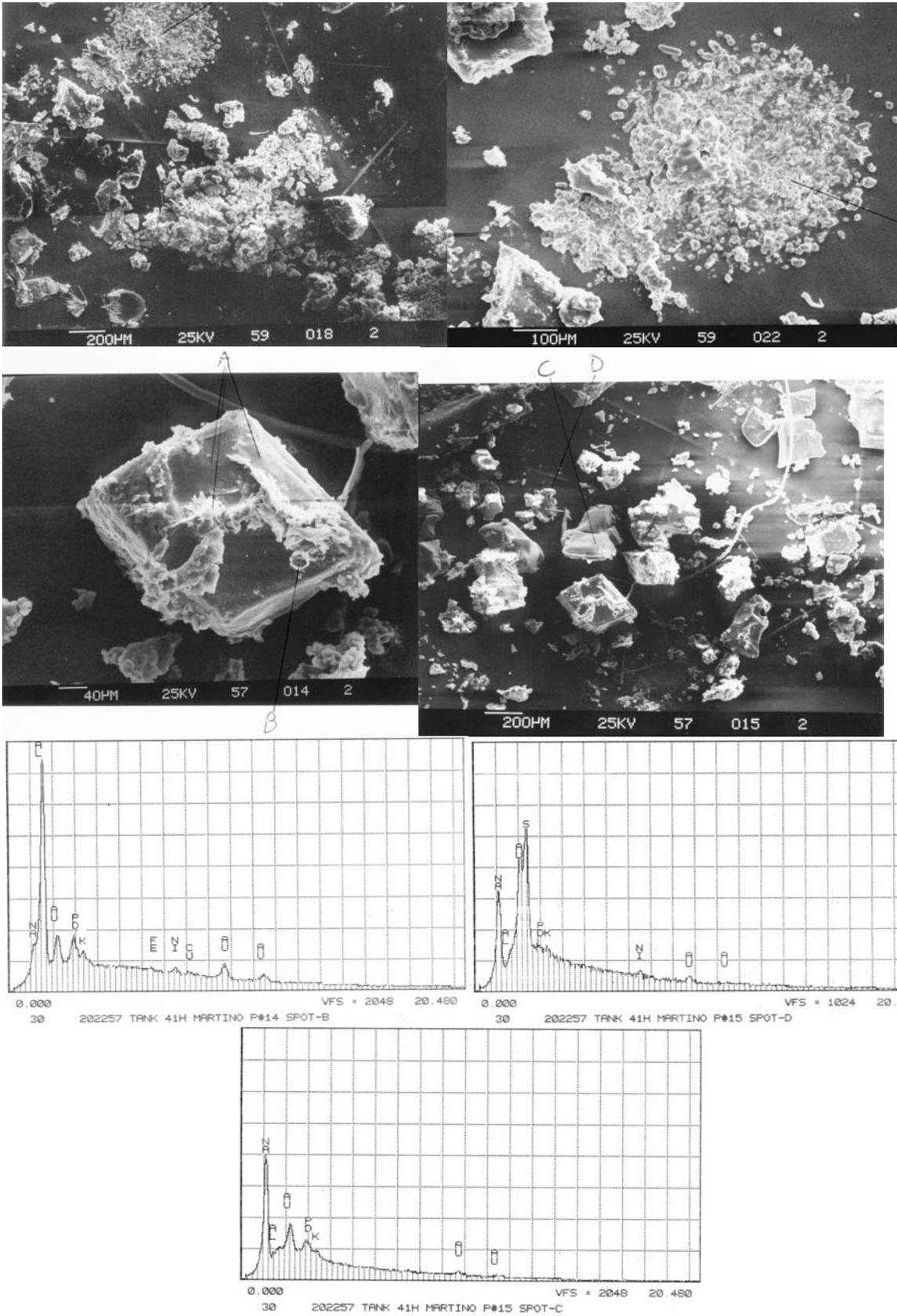


Figure 21: SEM/EDS of insoluble solids filtered from C3 surface SL Sample HTF-E-03-124

Conclusions

This report provides results of analyses of the samples from Tank 41H taken after the June 2003 dissolution campaign. The characterization also includes results from supernate samples pulled from the surface (347 inch) and 250 inch levels of the salt supernate below the C1 and C3 risers in September 2003 (HTF-E-03-124, 125, 129, 130, and 132).

The three saltcake samples (HTF-E-03-145, 146, and 147) were filled to nearly their capacity with saltcake and free liquid, with an average saltcake bulk density of 2.06 g/cm^3 . The undrained saltcake from the bottom of the middle sample (HTF-E-03-146) had a water content of 4.1 wt %, a ^{137}Cs activity of 0.14 Ci per gallon of saltcake, and an alpha content of $1.4\text{E}+4$ pCi/g. The undrained saltcake from the bottom of the bottom sample (HTF-E-03-147) had a water content of 8.5 wt %, a ^{137}Cs activity of 0.17 Ci per gallon of saltcake, and an alpha content of $3.0\text{E}+4$ pCi/g. Scanning electron microscopy of saltcake from sample HTF-E-03-146 revealed several features consistent with mineral dissolution: sub-rounded appearance of grains, lack of coatings by secondary precipitates, and crevices and indentations in some grains.

An analysis of material from the top of the top sample (HTF-E-03-145) is provided in support of Nuclear Criticality Safety Evaluations. The as-received saltcake and the residual insoluble solids had a uranium-235 enrichment of approximately 12.2%. Characterization focused on providing information on fissile radionuclides, potential neutron poisons, and other potential diluents.

Interstitial liquid drained from the middle sample (HTF-E-03-146) had a density of 1.43 g/cm^3 , a soluble solids content of 45.4 wt %, a ^{137}Cs activity of 0.78 Ci per gallon of interstitial liquid, and an alpha content of $1.1\text{E}+4$ pCi/mL. Supernate samples (HTF-E-03-124, 125, 129, 130, and 132) yielded information on the vertical and lateral tank supernate homogeneity. The ^{137}Cs content of these Tank 41H samples averaged 0.56 Ci/gal for the C3 riser and 0.65 Ci/gal for the C1 riser. The ^{238}Pu content of the four unfiltered samples ranged from $1.41\text{E}+4$ pCi/mL to $2.21\text{E}+4$ pCi/mL.

Quality Assurance

This work satisfies the requirements of the original task technical and quality assurance plans.^{14,15} Laboratory Notebooks WSRC-NB-2003-00072, WSRC-NB-2003-00199, WSRC-NB-2003-00234, and various ADS notebooks contain the experimental data.

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Appendix

Table 6: Water content (wt %) and interstitial liquid content (vol %) of the as-received (undrained) Tank 41H saltcake samples.

Tank 41H Post Dissolution Core	Water (wt %)		IL (vol %)
	average	st. dev.	
HTF-E-03-145	9.9%	1.8%	26%
HTF-E-03-146	4.1%	0.8%	11%
HTF-E-03-147	8.5%	0.5%	22%

Table 7: Undrained saltcake sample rad. chem. results (pCi/g)

Tank 41H	HTF-E-03-146		HTF-E-03-147	
(pCi/g)	Average	St. Dev.	Average	St. Dev.
¹⁴ C	< 2.25E+02	--	--	
¹³⁷ Cs	1.81E+07	1.0E+06	2.25E+07	1.8E+06
²³⁸ Pu	1.33E+04	1.8E+03	2.89E+04	2.4E+04
^{239/240} Pu ^a	9.46E+02	1.3E+02	< 1.20E+03	--

Note a: ^{239/240}Pu only present in HTF-E-03-146 near levels contained in analytical blanks.

Table 8: Undrained saltcake sample AA results (g/100g)

Tank 41H	HTF-E-03-146		HTF-E-03-147	
(g/100g)	Average	St. Dev.	Average	St. Dev.
As	< 4.8E-04	--	< 2.8E-04	--
K	1.49E-02	6E-04	1.66E-02	4E-04
Se	< 4.8E-04	--	< 2.8E-04	--
Hg	< 1.1E-03	--	< 6.2E-04	--

Table 9: Undrained saltcake sample ICP-ES results (g/100g)

Tank 41H (g/100g)	HTF-E-03-146		HTF-E-03-147	
	Average	St. Dev.	Average	St. Dev.
Ag	< 3.83E-04	--	<=2.39E-04	5.0E-05
Al	2.62E-01	1.7E-02	1.58E+00	8E-02
B	< 6.32E-03	--	< 3.73E-03	--
Ba	7.85E-04	1.62E-04	4.80E-04	9.7E-05
Ca	1.36E-03	5E-05	8.30E-04	2.5E-05
Cd	< 1.15E-03	--	< 6.78E-04	--
Ce	1.43E-02	2.9E-03	8.91E-03	2.12E-03
Cr	<=3.92E-03	6.2E-04	3.24E-03	2.4E-04
Cu	< 9.58E-04	--	< 5.65E-04	--
Fe	6.57E-03	3.92E-03	2.56E-03	5.1E-04
Gd	1.59E-03	4.3E-04	9.58E-04	2.14E-04
K	2.73E-01	3E-03	1.65E-01	2.0E-02
La	1.83E-03	3.6E-04	1.08E-03	1.7E-04
Li	3.46E-03	4.8E-04	2.21E-03	6.2E-04
Mg	<=2.02E-04	4.0E-05	<=1.18E-04	9E-06
Mn	4.36E-04	1.24E-04	<=3.02E-04	3.2E-05
Mo	< 6.42E-03	--	<=4.02E-03	2.2E-04
Na	2.60E+01	2E-01	2.53E+01	1E-01
Ni	< 4.31E-03	--	< 2.54E-03	--
P	< 4.01E-02	--	< 2.37E-02	--
Pb	< 2.74E-02	--	< 1.62E-02	--
S	2.38E-01	2E-03	2.20E-01	1.2E-02
Sb	< 6.80E-03	--	6.81E-03	2.3E-04
Si	3.26E-03	1.53E-03	6.28E-03	5.5E-04
Sn	< 1.09E-02	--	< 6.44E-03	--
Sr	1.49E-03	3.1E-04	8.96E-04	2.05E-04
Ti	< 1.25E-03	--	< 7.35E-04	--
V	<=1.12E-03	3.8E-05	1.21E-03	1.3E-04
Zn	<=2.00E-04	1.3E-05	<=1.19E-04	2.5E-05
Zr	< 1.34E-03	--	< 7.91E-04	--

Table 10: Undrained saltcake sample ICP-MS results (g/100g)

Tank 41H (g/100g)	HTF-E-03-146		HTF-E-03-147	
	Average	St. Dev.	Average	St. Dev.
Mass 59	< 8.0E-06	--	< 4.7E-06	--
Mass 88	1.24E-05	3.4E-06	1.04E-05	1.2E-05
Mass 99	4.34E-05	1E-07	5.61E-05	3.8E-06
Mass 101	< 4.2E-06	--	6.1E-06	3E-07
Mass 133	4.70E-05	3E-06	5.42E-05	7E-07
Mass 135	1.36E-05	4E-06	9.4E-06	7E-07
Mass 137	5.48E-05	5E-06	7.87E-05	6.4E-06
Mass 138	< 5.10E-05	3.8E-05	< 1.3E-05	--
Mass 230	< 2.7E-06	--	< 1.6E-06	--
Mass 231	< 2.7E-06	--	< 1.6E-06	--
Mass 232	< 2.7E-06	--	< 1.6E-06	--
Mass 233	< 2.7E-06	--	2.3E-06	3E-07
Mass 234	6.9E-06	6E-07	1.09E-05	2.1E-06
Mass 235	1.93E-05	1.4E-06	2.60E-05	5E-07
Mass 236	8.5E-06	5E-07	1.17E-05	1.6E-06
Mass 237	3.7E-06	4E-07	6.9E-06	1.1E-06
Mass 238	1.33E-04	2E-06	1.83E-04	1.5E-05
Mass 239	< 2.7E-06	--	< 1.6E-06	--
Mass 240	< 2.7E-06	--	< 1.6E-06	--
Mass 241	< 2.7E-06	--	< 1.6E-06	--
Mass 242	< 2.7E-06	--	< 1.6E-06	--
Mass 243	< 2.7E-06	--	< 1.6E-06	--
Mass 244	< 2.7E-06	--	< 1.6E-06	--

Table 11: Undrained saltcake sample IC, wet chem., and TIC/TOC results (g/100g)

Tank 41H	HTF-E-03-146		HTF-E-03-147	
(g/100g)	Average	St. Dev.	Average	St. Dev.
NO ₃ ⁻	7.37E+01	3.0E+00	6.19E+01	1.1E+00
NO ₂ ⁻	5.00E-01	3.6E-02	5.97E-01	1.7E-02
SO ₄ ²⁻	7.14E-01	5.3E-02	6.46E-01	1.7E-02
PO ₄ ³⁻	3.37E-02	2.5E-03	2.57E-02	1.3E-03
Cl ⁻	< 3.54E-03	--	< 3.81E-03	--
F ⁻	< 3.54E-03	--	< 3.81E-03	--
C ₂ O ₄ ²⁻	3.37E-02	2.5E-03	4.76E-02	0E+00
CHO ₂ ⁻	2.83E-02	0E+00	3.24E-02	0E+00
Free OH ⁻	9.58E-02	2.77E-02	4.66E-01	1.4E-02
CO ₃ ²⁻ (TIC)	9.20E-01	1.3E-02	7.31E-01	5.2E-02
TOC	4.16E-02	1.3E-03	5.73E-02	2.13E-02

Table 12: Drained interstitial liquid rad. chem. results (pCi/mL)

Tank41H	HTF-E-03-146 IL	
(pCi/mL)	Average	St. Dev.
¹³⁷ Cs	2.05E+08	6E+05
²³⁸ Pu	9.48E+03	3.2E+02
^{239/240} Pu	1.48E+03	9.5E+01

Table 13: Drained interstitial liquid IC, wet chem., and TIC results (mg/L)

Tank 41H	HTF-E-03-146 IL	
(mg/L)	Average	St. Dev.
NO ₃ ⁻	2.25E+05	4E+03
NO ₂ ⁻	2.83E+04	4.9E+03
SO ₄ ²⁻	1.41E+04	1.1E+03
PO ₄ ³⁻	1.54E+03	2.6E+02
Cl ⁻	< 1.81E+02	--
F ⁻	< 1.81E+02	--
C ₂ O ₄ ²⁻	< 9.07E+02	--
CHO ₂ ⁻	1.27E+03	2.6E+02
Tot. Base (M)	4.46E+00	2.2E-01
Free OH ⁻	2.49E+04	2.5E+03
CO ₃ ²⁻ (TIC)	3.17E+04	1.3E+03

Table 14: Drained interstitial liquid ICP-ES results (mg/L)

Tank41H (mg/L)	HTF-E-03-146 IL	
	Average	St. Dev.
Ag	< 3.58E+00	--
Al	2.66E+04	1.3E+03
B	< 5.91E+01	--
Ba	< 4.48E+00	--
Ca	< 1.07E+01	--
Cd	< 1.07E+01	--
Ce	< 4.57E+01	--
Cr	1.91E+02	1.4E+01
Cu	< 8.96E+00	--
Fe	< 7.16E+00	--
Gd	< 7.16E+00	--
K	1.01E+03	8E+01
La	< 8.06E+00	--
Li	< 2.68E+00	--
Mg	< 1.80E+00	--
Mn	2.85E+00	2.4E-01
Mo	< 6.01E+01	--
Na	1.93E+05	8E+03
Ni	< 4.02E+01	--
P	5.99E+02	8.6E+01
Pb	< 2.57E+02	--
S	5.55E+03	2.6E+02
Sb	< 6.36E+01	--
Si	3.94E+01	9.1E+00
Sn	< 1.02E+02	--
Sr	< 7.16E+00	--
Ti	< 1.16E+01	--
V	< 9.86E+00	--
Zn	< 1.80E+00	--
Zr	< 1.25E+01	--

Table 15: Drained interstitial liquid ICP-MS results (mg/L)

Tank 41H	HTF-E-03-146 IL	
(mg/L)	Average	St. Dev.
Mass 59	1.93E-02	1.4E-03
Mass 88	2.65E-02	1.35E-02
Mass 99	5.62E+00	2.2E-01
Mass 101	4.04E-01	1.1E-02
Mass 133	5.53E+00	8E-02
Mass 135	7.13E-01	2E-03
Mass 137	2.13E+00	3E-02
Mass 138	< 3.2E-02	--
Mass 230	< 5.2E-03	--
Mass 231	< 5.2E-03	--
Mass 232	8.95E-02	1.2E-01
Mass 233	3.76E-01	1.0E-02
Mass 234	2.07E+00	6E-02
Mass 235	5.24E+00	7E-02
Mass 236	2.28E+00	5E-02
Mass 237	4.13E-01	5.3E-02
Mass 238	3.64E+01	1E+00
Mass 239	< 5.2E-03	--
Mass 240	< 5.2E-03	--
Mass 241	< 5.2E-03	--
Mass 242	< 5.2E-03	--
Mass 243	< 5.2E-03	--
Mass 244	< 5.2E-03	--

Table 16: ICP-ES of NCSE dissolution fractions of HTF-E-03-145

(wt%)	Dissolution 1	Dissolution 2	Dissolution 3
Ag	< 1.86E-04	< 4.52E-05	< 1.34E-05
Al	2.88E-01	1.79E-02	7.64E-04
B	< 3.07E-03	< 7.46E-04	< 2.21E-04
Ba	< 2.32E-04	< 5.65E-05	< 1.68E-05
Ca	< 5.58E-04	< 1.36E-04	< 4.02E-05
Cd	< 5.58E-04	< 1.36E-04	< 4.02E-05
Ce	< 2.37E-03	< 5.77E-04	< 1.71E-04
Cr	2.24E-03	< 4.52E-04	< 1.34E-04
Cu	< 4.65E-04	< 1.13E-04	< 3.35E-05
Fe	< 3.72E-04	< 9.05E-05	< 2.68E-05
Gd	< 3.72E-04	< 9.05E-05	< 2.68E-05
K	< 4.37E-02	< 1.00E-02	3.85E-03
La	< 4.18E-04	< 1.02E-04	< 3.02E-05
Li	< 1.39E-04	7.69E-05	< 1.01E-05
Mg	< 9.29E-05	< 2.26E-05	< 6.70E-06
Mn	< 1.39E-04	< 3.39E-05	< 1.01E-05
Mo	< 3.11E-03	< 7.58E-04	< 2.24E-04
Na	8.02E+00	3.71E+00	1.28E-01
Ni	< 2.09E-03	< 5.09E-04	< 1.51E-04
P	2.01E-02	7.94E-03	2.07E-03
Pb	< 1.33E-02	< 3.23E-03	< 9.58E-04
S	1.16E-01	1.00E-02	< 4.26E-04
Sb	< 3.30E-03	< 8.03E-04	< 2.38E-04
Si	< 1.16E-03	< 2.83E-04	< 8.38E-05
Sn	< 5.30E-03	< 1.29E-03	< 3.82E-04
Sr	< 3.72E-04	< 9.05E-05	< 2.68E-05
Ti	< 6.04E-04	< 1.47E-04	< 4.36E-05
V	< 5.11E-04	< 1.24E-04	< 3.69E-05
Zn	< 9.29E-05	< 2.26E-05	< 6.70E-06
Zr	< 6.51E-04	< 1.58E-04	< 4.69E-05

Table 17: ICP-MS of NCSE dissolution fractions of HTF-E-03-145

(wt%)	Dissolution 1	Dissolution 2	Dissolution 3
Mass 99	6.60E-05	4.21E-06	< 3.26E-07
Mass 101	< 5.09E-06	< 1.24E-06	< 3.67E-07
Mass 133	6.65E-05	5.30E-06	< 6.09E-07
Mass 135	2.00E-05	5.10E-06	4.69E-06
Mass 137	4.68E-05	1.01E-05	8.48E-06
Mass 138	1.13E-04	4.63E-05	5.00E-05
Mass 230	< 1.99E-06	< 4.84E-07	< 1.44E-07
Mass 231	< 1.99E-06	< 4.84E-07	< 1.44E-07
Mass 232	< 1.99E-06	< 4.84E-07	< 1.44E-07
Mass 233	< 1.99E-06	< 4.84E-07	< 1.44E-07
Mass 234	< 1.99E-06	< 4.84E-07	< 1.44E-07
Mass 235	3.91E-06	< 4.84E-07	< 1.44E-07
Mass 236	< 1.99E-06	< 4.84E-07	< 1.44E-07
Mass 237	< 1.99E-06	< 4.84E-07	< 1.44E-07
Mass 238	4.09E-05	4.83E-06	5.18E-07
Mass 239	< 1.99E-06	< 4.84E-07	< 1.44E-07
Mass 240	< 1.99E-06	< 4.84E-07	< 1.44E-07
Mass 241	< 1.99E-06	< 4.84E-07	< 1.44E-07
Mass 242	< 1.99E-06	< 4.84E-07	< 1.44E-07
Mass 243	< 1.99E-06	< 4.84E-07	< 1.44E-07
Mass 244	< 1.99E-06	< 4.84E-07	< 1.44E-07

Table 18: IC, wet chem., and TIC/TOC of NCSE dissolution fractions of HTF-E-03-145

(wt%)	Dissolution 1	Dissolution 2	Dissolution 3
NO ₃ ⁻	1.73E+01	1.02E+01	3.47E-01
NO ₂ ⁻	3.37E-01	4.18E-02	1.17E-03
SO ₄ ²⁻	3.39E-01	2.77E-02	6.70E-04
PO ₄ ³⁻	5.11E-02	2.43E-02	5.19E-03
Cl ⁻	< 9.29E-03	< 2.26E-03	< 6.70E-04
F ⁻	< 9.29E-03	< 2.26E-03	6.70E-04
C ₂ O ₄ ²⁻	4.65E-02	2.09E-02	4.52E-03
CHO ₂ ⁻	< 4.65E-02	< 1.13E-02	< 3.35E-03
Free OH ⁻	7.12E-01	5.34E-02	1.14E-02
CO ₃ ²⁻ (TIC)	4.81E-01	5.98E-02	1.39E-02
TOC	9.29E-02	1.76E-02	5.48E-03

Table 19: Tank 41H supernate rad. chem. results (pCi/mL)

Tank 41H	Riser C3 Surface Sample (HTF-E-03-124)				Riser C3 250" VDS (HTF-E-03-125)				Riser C1 Surface Sample (HTF-E-03-129)				Riser C1 250" VDS (HTF-E-03-130 and 132)					
	As Received		0.45-µm Filtrate		As Received		0.45-µm Filtrate		As Received		0.45-µm Filtrate		As Received		0.45-µm Filtrate		0.1-µm Filtrate	
	Average	St. Dev.	Average	St. Dev.	Average	St. Dev.	Average	St. Dev.	Average	St. Dev.	Average	St. Dev.	Average	St. Dev.	Average	St. Dev.	Average	St. Dev.
¹⁴ C	--	--	< 1.6E+03	--	--	--	2.5E+03	--	--	--	< 1.7E+03	--	--	--	< 1.7E+03	--	--	--
¹³⁷ Cs	1.44E+08	1E+06	1.42E+08	4E+06	1.54E+08	4E+06	1.49E+08	1E+06	1.75E+08	7E+06	1.73E+08	8E+06	1.71E+08	2E+06	1.70E+08	0E+00	1.71E+08	5E+06
²³⁸ Pu	2.21E+04	1.4E+03	2.03E+04	9E+02	1.41E+04	1.6E+03	1.26E+04	1.0E+03	1.62E+04	8E+02	1.76E+04	1.2E+03	1.65E+04	3.5E+03	1.20E+04	9.9E+02	1.18E+04	1.1E+03
^{239/240} Pu ^a	2.1E+02	5E+01	3.7E+02	1.0E+02	2.2E+02	2E+01	5.8E+02	7E+01	3.3E+02	1.2E+02	3.6E+02	1.7E+02	6.8E+02	3.4E+02	4.9E+02	2.6E+02	7.3E+02	2.3E+02

Note a: Due to the relatively high ratio of ²³⁸Pu to ^{239/240}Pu, there is a high counting uncertainty (20 to 80%) in ^{239/240}Pu.

Table 20: Tank 41H supernate ICP-MS results (mg/L)

Tank 41H	Riser C3 Surface Sample (HTF-E-03-124)				Riser C3 250" VDS (HTF-E-03-125)				Riser C1 Surface Sample (HTF-E-03-129)				Riser C1 250" VDS (HTF-E-03-130 and 132)					
	As Received		0.45-µm Filtrate		As Received		0.45-µm Filtrate		As Received		0.45-µm Filtrate		As Received		0.45-µm Filtrate		0.1-µm Filtrate	
	Average	St. Dev.	Average	St. Dev.	Average	St. Dev.	Average	St. Dev.	Average	St. Dev.	Average	St. Dev.	Average	St. Dev.	Average	St. Dev.	Average	St. Dev.
Mass 59	< 7.6E-02	8E-03	< 2.5E-01	--	< 6.8E-02	--	< 2.4E-01	--	< 6.7E-02	--	< 2.5E-01	--	< 7.5E-02	1.0E-02	< 2.5E-01	--	< 2.5E-01	--
Mass 88	< 1.5E-01	2E-02	< 3.0E-02	--	< 1.3E-01	--	< 5.1E-02	3.0E-02	< 1.3E-01	--	< 3.0E-02	--	< 1.3E-01	--	< 3.1E-02	--	< 3.0E-02	--
Mass 99	4.36E+00	1.4E-01	3.58E+00	2.7E-02	4.32E+00	1.6E-01	3.69E+00	2.45E-01	4.71E+00	3.2E-01	4.27E+00	3E-02	4.84E+00	2.6E-01	4.29E+00	3.8E-02	4.39E+00	1.7E-01
Mass 101	< 2.6E-01	3E-02	1.8E-01	7E-03	< 2.4E-01	--	1.9E-01	2E-02	< 2.8E-01	7E-02	2.5E-01	2E-02	3.1E-01	7E-02	2.1E-01	3E-02	2.5E-01	4E-02
Mass 133	3.94E+00	7.5E-04	3.73E+00	2.0E-01	4.16E+00	1.7E-01	3.93E+00	9.96E-05	4.84E+00	4.7E-01	4.52E+00	7E-03	4.83E+00	2.5E-01	4.71E+00	1.6E-01	4.58E+00	1.8E-01
Mass 135	5.7E-01	7E-03	4.3E-01	2E-02	5.8E-01	2E-02	4.5E-01	6E-03	7.3E-01	8E-02	5.8E-01	2E-02	6.6E-01	4E-02	5.9E-01	3E-02	5.7E-01	2E-02
Mass 137	1.77E+00	5.8E-02	1.43E+00	6.4E-02	1.96E+00	2.0E-01	1.52E+00	1.31E-01	2.18E+00	2.2E-01	1.78E+00	7E-03	2.26E+00	1.1E-01	1.71E+00	3.8E-02	1.83E+00	5.3E-02
Mass 138	3.2E-01	9E-02	< 4.0E-01	--	1.9E-01	7E-02	< 3.9E-01	--	9.4E-01	4.0E-01	< 4.0E-01	--	3.6E-01	1.9E-01	< 4.0E-01	--	< 4.0E-01	--
Mass 230	< 8.0E-02	--	< 1.2E-01	--	< 7.8E-02	--	< 1.2E-01	--	< 7.6E-02	--	< 1.2E-01	--	< 7.7E-02	--	< 1.2E-01	--	< 1.2E-01	--
Mass 231	< 8.0E-02	--	< 1.2E-01	--	< 7.8E-02	--	< 1.2E-01	--	< 7.6E-02	--	< 1.2E-01	--	< 7.7E-02	--	< 1.2E-01	--	< 1.2E-01	--
Mass 232	1.0E-01	1E-02	< 1.2E-01	--	< 7.8E-02	--	< 1.2E-01	--	< 7.6E-02	--	< 1.2E-01	--	< 7.7E-02	--	< 1.2E-01	--	< 1.2E-01	--
Mass 233	< 8.0E-02	--	< 1.2E-01	--	< 7.8E-02	--	< 1.2E-01	--	< 7.6E-02	--	< 1.2E-01	--	< 7.7E-02	--	< 1.2E-01	--	< 1.2E-01	--
Mass 234	< 8.0E-02	--	< 1.2E-01	--	< 7.8E-02	--	< 1.2E-01	--	< 7.6E-02	--	< 1.2E-01	--	8.0E-02	5E-06	< 1.2E-01	--	< 1.2E-01	--
Mass 235	1.7E-01	9E-03	< 1.4E-01	4E-02	1.8E-01	6E-03	1.4E-01	5E-03	1.8E-01	3E-03	1.4E-01	2E-02	1.9E-01	4E-02	1.4E-01	2E-02	1.8E-01	3E-02
Mass 236	< 8.0E-02	--	< 1.2E-01	--	< 7.9E-02	1E-03	< 1.2E-01	--	< 8.4E-02	1.1E-02	< 1.2E-01	--	< 8.3E-02	8E-03	< 1.2E-01	--	< 1.2E-01	--
Mass 237	< 8.0E-02	--	< 1.2E-01	--	< 8.7E-02	1.4E-02	< 1.2E-01	--	< 7.6E-02	--	< 1.2E-01	--	< 1.4E-01	1E-01	< 1.2E-01	--	< 1.2E-01	--
Mass 238	1.44E+00	1.6E-01	1.15E+00	4.0E-02	1.31E+00	2.0E-01	9.77E-01	1.52E-01	1.56E+00	2.9E-01	1.30E+00	1E-01	1.69E+00	1.2E-02	1.16E+00	9.8E-03	1.21E+00	5.0E-02
Mass 239	< 8.0E-02	--	< 1.2E-01	--	< 7.8E-02	--	< 1.2E-01	--	< 7.6E-02	--	< 1.2E-01	--	< 7.7E-02	--	< 1.2E-01	--	< 1.2E-01	--
Mass 240	< 8.0E-02	--	< 1.2E-01	--	< 7.8E-02	--	< 1.2E-01	--	< 7.6E-02	--	< 1.2E-01	--	< 7.7E-02	--	< 1.2E-01	--	< 1.2E-01	--
Mass 241	< 8.0E-02	--	< 1.2E-01	--	< 7.8E-02	--	< 1.2E-01	--	< 7.6E-02	--	< 1.2E-01	--	< 7.7E-02	--	< 1.2E-01	--	< 1.2E-01	--
Mass 242	< 8.0E-02	--	< 1.2E-01	--	< 7.8E-02	--	< 1.2E-01	--	< 7.6E-02	--	< 1.2E-01	--	< 7.7E-02	--	< 1.2E-01	--	< 1.2E-01	--
Mass 243	< 8.0E-02	--	< 1.2E-01	--	< 7.8E-02	--	< 1.2E-01	--	< 7.6E-02	--	< 1.2E-01	--	< 7.7E-02	--	< 1.2E-01	--	< 1.2E-01	--
Mass 244	< 8.0E-02	--	< 1.2E-01	--	< 7.8E-02	--	< 1.2E-01	--	< 7.6E-02	--	< 1.2E-01	--	< 7.7E-02	--	< 1.2E-01	--	< 1.2E-01	--

Table 21: Tank 41H supernate AA results (mg/L)

Tank 41H	Riser C3 Surface Sample (HTF-E-03-124)				Riser C3 250" VDS (HTF-E-03-125)				Riser C1 Surface Sample (HTF-E-03-129)				Riser C1 250" VDS (HTF-E-03-130 and 132)							
	As Received		0.45-µm Filtrate		As Received		0.45-µm Filtrate		As Received		0.45-µm Filtrate		As Received		0.45-µm Filtrate		0.1-µm Filtrate			
	(mg/L)	Average	St. Dev.	Average	St. Dev.	Average	St. Dev.	Average	St. Dev.	Average	St. Dev.	Average	St. Dev.	Average	St. Dev.	Average	St. Dev.	Average	St. Dev.	
As	< 3.5E+00	--	< 3.4E+00	--	< 3.4E+00	--	< 3.4E+00	--	< 3.3E+00	--	< 3.4E+00	--	< 3.3E+00	--	< 3.4E+00	--	< 3.4E+00	--	< 3.4E+00	--
K	3.9E+02	2E+01	4.2E+02	6E+00	4.2E+02	3E+00	4.2E+02	3E+00	4.7E+02	8E+00	4.7E+02	4E+00	4.6E+02	8E+00	4.8E+02	2E+01	4.9E+02	2E+01	< 3.4E+00	--
Se	< 3.5E+00	--	< 3.4E+00	--	< 3.4E+00	--	< 3.4E+00	--	< 3.3E+00	--	< 3.4E+00	--	< 3.3E+00	--	< 3.4E+00	--	< 3.4E+00	--	< 3.4E+00	--
Hg	< 7.6E+00	--	< 7.4E+00	--	< 7.4E+00	--	< 7.4E+00	--	< 7.3E+00	--	< 7.5E+00	--	< 7.4E+00	--	< 7.5E+00	--	< 7.5E+00	--	< 7.5E+00	--

Table 22: Tank 41H supernate ICP-ES results (mg/L)

Tank 41H	Riser C3 Surface Sample (HTF-E-03-124)				Riser C3 250" VDS (HTF-E-03-125)				Riser C1 Surface Sample (HTF-E-03-129)				Riser C1 250" VDS (HTF-E-03-130 and 132)							
	As Received		0.45-µm Filtrate		As Received		0.45-µm Filtrate		As Received		0.45-µm Filtrate		As Received		0.45-µm Filtrate		0.1-µm Filtrate			
	(mg/L)	Average	St. Dev.	Average	St. Dev.	Average	St. Dev.	Average	St. Dev.	Average	St. Dev.	Average	St. Dev.	Average	St. Dev.	Average	St. Dev.	Average	St. Dev.	
Ag	< 1.04E+01	--	< 1.01E+01	--	< 1.01E+01	--	< 1.01E+01	--	< 9.95E+00	--	< 1.02E+01	--	< 1.00E+01	--	< 1.03E+01	--	< 1.02E+01	--	< 1.02E+01	--
Al	9.23E+03	6.5E+02	1.18E+04	9.4E+03	1.35E+04	1.1E+03	6.50E+03	4.93E+03	1.03E+04	1.2E+03	1.82E+04	4E+02	1.27E+04	1.2E+03	1.31E+04	8.5E+03	1.92E+04	1.8E+03	< 1.10E+03	--
B	< 1.12E+03	--	< 1.10E+03	--	< 1.09E+03	--	< 1.09E+03	--	< 1.07E+03	--	< 1.10E+03	--	< 1.08E+03	--	< 1.11E+03	--	< 1.10E+03	--	< 1.10E+03	--
Ba	< 1.04E+01	--	< 1.01E+01	--	< 1.01E+01	--	< 1.01E+01	--	< 9.95E+00	--	< 1.02E+01	--	< 1.00E+01	--	< 1.03E+01	--	< 1.02E+01	--	< 1.02E+01	--
Ca	< 3.13E+02	--	< 3.06E+02	--	< 3.06E+02	--	< 3.05E+02	--	< 3.00E+02	--	< 3.07E+02	--	< 3.03E+02	--	< 3.11E+02	--	< 3.08E+02	--	< 3.08E+02	--
Cd	< 1.38E+01	--	< 1.35E+01	--	< 1.35E+01	--	< 1.34E+01	--	< 1.33E+01	--	< 1.36E+01	--	< 1.34E+01	--	< 1.37E+01	--	< 1.36E+01	--	< 1.36E+01	--
Ce	< 1.71E+02	--	< 1.68E+02	--	< 1.68E+02	--	< 1.66E+02	--	< 1.64E+02	--	< 1.68E+02	--	< 1.66E+02	--	< 1.70E+02	--	< 1.68E+02	--	< 1.68E+02	--
Cr	1.06E+02	2E+00	1.23E+02	7.4E+01	1.25E+02	3E+00	8.83E+01	3.69E+01	1.02E+02	1.1E+01	1.49E+02	1E+00	1.16E+02	9E+00	1.24E+02	4.9E+01	1.63E+02	7E+00	< 1.02E+01	--
Cu	< 2.15E+01	--	< 2.09E+01	--	< 2.10E+01	--	< 2.08E+01	--	< 2.06E+01	--	< 2.10E+01	--	< 2.07E+01	--	< 2.12E+01	--	< 2.11E+01	--	< 2.11E+01	--
Fe	< 1.52E+01	--	< 1.49E+01	--	< 1.48E+01	--	< 1.48E+01	--	<=1.57E+01	1.6E+00	< 1.50E+01	--	1.87E+01	2.3E+00	< 1.51E+01	--	<=1.67E+01	2.4E+00	< 1.84E+01	--
Gd	< 1.87E+01	--	< 1.82E+01	--	< 1.82E+01	--	< 1.82E+01	--	< 1.79E+01	--	< 1.84E+01	--	< 1.81E+01	--	< 1.85E+01	--	< 1.85E+01	--	< 1.84E+01	--
K	< 6.58E+03	--	< 6.44E+03	--	< 6.41E+03	--	< 6.40E+03	--	< 6.32E+03	--	< 6.46E+03	--	< 6.37E+03	--	< 6.54E+03	--	< 6.49E+03	--	< 6.49E+03	--
La	< 1.38E+01	--	< 1.35E+01	--	< 1.35E+01	--	< 1.34E+01	--	< 1.33E+01	--	< 1.36E+01	--	< 1.34E+01	--	< 1.37E+01	--	< 1.36E+01	--	< 1.36E+01	--
Li	< 5.88E+01	--	< 5.75E+01	--	< 5.73E+01	--	< 5.71E+01	--	< 5.63E+01	--	< 5.77E+01	--	< 5.69E+01	--	< 5.83E+01	--	< 5.79E+01	--	< 5.79E+01	--
Mg	< 4.28E+01	--	< 4.19E+01	--	< 4.18E+01	--	< 4.16E+01	--	< 4.10E+01	--	< 4.20E+01	--	< 4.15E+01	--	< 4.26E+01	--	< 4.23E+01	--	< 4.23E+01	--
Mn	< 1.52E+01	--	< 1.49E+01	--	< 1.48E+01	--	< 1.48E+01	--	< 1.46E+01	--	< 1.50E+01	--	< 1.47E+01	--	< 1.51E+01	--	< 1.50E+01	--	< 1.50E+01	--
Mo	< 1.40E+02	--	< 1.38E+02	--	< 1.37E+02	--	< 1.37E+02	--	< 1.35E+02	--	< 1.38E+02	--	< 1.36E+02	--	< 1.40E+02	--	< 1.39E+02	--	< 1.39E+02	--
Na	1.59E+05	0E+00	1.70E+05	1.8E+04	1.65E+05	2.8E+04	1.58E+05	0E+00	1.51E+05	3E+03	1.54E+05	0E+00	1.58E+05	5E+03	1.66E+05	1.8E+04	1.68E+05	1.5E+04	< 1.03E+02	--
Ni	< 5.18E+01	--	< 5.06E+01	--	< 5.06E+01	--	< 5.04E+01	--	< 4.97E+01	--	< 5.09E+01	--	< 5.01E+01	--	< 5.15E+01	--	< 5.11E+01	--	< 5.11E+01	--
P	1.11E+03	7E+01	1.08E+03	9E+01	1.06E+03	6E+01	1.03E+03	5E+01	1.06E+03	4E+01	9.72E+02	1.50E+02	1.09E+03	5E+01	9.63E+02	4.1E+01	1.06E+03	4E+01	< 1.03E+02	--
Pb	< 1.70E+02	--	< 1.67E+02	--	< 1.66E+02	--	< 1.65E+02	--	< 1.63E+02	--	< 1.67E+02	--	< 1.64E+02	--	< 1.68E+02	--	< 1.67E+02	--	< 1.67E+02	--
S	6.55E+03	9.9E+01	6.48E+03	1.2E+02	6.27E+03	6.4E+02	5.89E+03	1.3E+02	5.86E+03	1.6E+02	5.85E+03	5.3E+02	5.84E+03	2.4E+02	5.82E+03	3.1E+02	6.00E+03	2.0E+02	< 1.03E+02	--
Sb	< 1.04E+02	--	< 1.02E+02	--	< 1.02E+02	--	< 1.02E+02	--	< 1.00E+02	--	< 1.02E+02	--	< 1.01E+02	--	< 1.04E+02	--	< 1.03E+02	--	< 1.03E+02	--
Si ^a	< 8.20E+02	--	< 1.45E+03	--	< 9.97E+02	--	< 1.43E+03	--	< 5.89E+02	--	< 1.81E+03	--	< 9.02E+02	--	< 9.75E+02	--	< 2.45E+03	--	< 2.45E+03	--
Sn	< 1.70E+02	--	< 1.66E+02	--	< 1.65E+02	--	< 1.65E+02	--	< 1.63E+02	--	< 1.67E+02	--	< 1.64E+02	--	< 1.68E+02	--	< 1.67E+02	--	< 1.67E+02	--
Sr	< 6.92E+01	--	< 6.76E+01	--	< 6.73E+01	--	< 6.72E+01	--	< 6.63E+01	--	< 6.78E+01	--	< 6.68E+01	--	< 6.86E+01	--	< 6.82E+01	--	< 6.82E+01	--
Ti	< 4.15E+00	--	< 4.05E+00	--	< 4.04E+00	--	< 4.04E+00	--	< 3.98E+00	--	< 4.08E+00	--	< 4.00E+00	--	< 4.12E+00	--	< 4.09E+00	--	< 4.09E+00	--
V	< 7.60E+00	--	< 7.44E+00	--	< 7.42E+00	--	< 7.39E+00	--	< 7.30E+00	--	< 7.47E+00	--	< 7.36E+00	--	< 7.54E+00	--	< 7.50E+00	--	< 7.50E+00	--
Zn	< 3.80E+01	--	< 3.72E+01	--	< 3.70E+01	--	< 3.70E+01	--	< 3.64E+01	--	< 3.73E+01	--	< 3.68E+01	--	< 3.78E+01	--	< 3.75E+01	--	< 3.75E+01	--
Zr	< 8.29E+00	--	< 8.11E+00	--	< 8.09E+00	--	< 8.06E+00	--	< 7.96E+00	--	< 8.14E+00	--	< 8.02E+00	--	< 8.24E+00	--	< 8.09E+00	--	< 8.09E+00	--

Note a: Si analysis is reported as an upper limit due to a high bias introduced by HF etching of the quartz torch in the ICP-ES.

Table 23: Tank 41H supernate (0.45- μ m filtrate) IC anions, wet chem. titration, and TIC/TOC results (mg/L)

Tank 41H (mg/L)	Riser C3 Surface Sample (HTF-E-03-124)		Riser C3 250" VDS (HTF-E-03-125)		Riser C1 Surface Sample (HTF-E-03-129)		Riser C1 250" VDS (HTF-E-03-130 & 132)	
	Average	St. Dev.	Average	St. Dev.	Average	St. Dev.	Average	St. Dev.
NO ₃ ⁻	2.92E+05	8E+03	2.66E+05	5E+03	2.58E+05	5E+03	2.63E+05	2E+04
NO ₂ ⁻	1.53E+04	9E+02	1.45E+04	7E+02	1.74E+04	3E+02	1.78E+04	6E+02
SO ₄ ²⁻	1.87E+04	1.1E+03	1.62E+04	6E+02	1.52E+04	1E+02	1.54E+04	5E+02
PO ₄ ³⁻	3.38E+03	2.3E+02	2.71E+03	1.4E+02	2.52E+03	5E+01	2.41E+03	0E+00
Cl ⁻	< 1.29E+02	--	< 1.67E+02	--	< 2.36E+02	--	< 2.76E+02	--
F ⁻	< 1.29E+02	--	< 1.34E+02	--	< 1.35E+02	--	< 1.38E+02	--
C ₂ O ₄ ²⁻	1.93E+02	0E+00	1.67E+02	4.7E+01	1.35E+02	0E+00	1.03E+02	4.9E+01
CHO ₂ ⁻	5.48E+02	4.6E+01	5.36E+02	9.5E+01	6.06E+02	9.5E+01	7.59E+02	9.8E+01
Total Base (M)	3.05E+00	5E-02	3.06E+00	7E-02	3.44E+00	3E-02	3.37E+00	1.2E-01
Free OH ⁻	1.59E+04	1.7E+03	1.78E+04	2E+02	2.09E+04	6E+02	2.27E+04	7E+02
CO ₃ ²⁻ (TIC)	3.46E+04	1.1E+03	3.70E+04	7E+02	3.17E+04	6.5E+03	3.04E+04	9.1E+03
TOC	3.7E+03	3E+02	2.4E+03	5E+02	2.9E+03	1.4E+03	3.4E+03	1.6E+03