Tank 29H Saltcake Core and Supernate Sample Analysis

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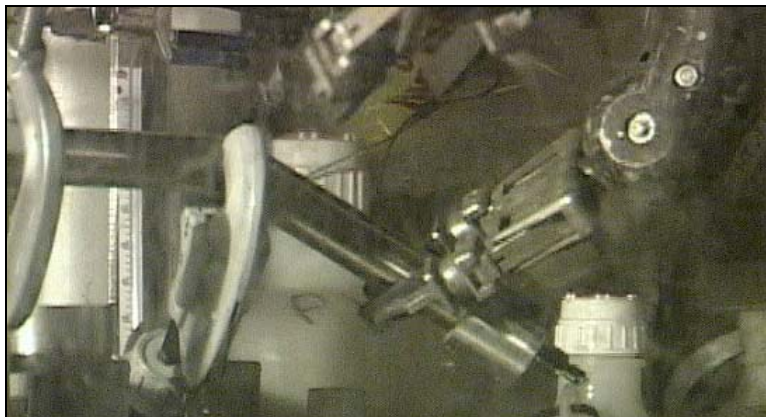
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Summary

This report provides the final results of analyses of the saltcake core sample and a separate supernate sample from Tank 29H. The portion of undrained saltcake that was analyzed contained a relatively large amount of aluminum (13 wt%), had a bulk density of 2.13 g/cm^3, a water content of 21.4 wt %, and a ^{137}Cs activity of 5.2 Ci per gallon of saltcake. The drained interstitial liquid had a ^{137}Cs activity of 9.5 Ci per gallon.

The supernate sample was markedly different from the drained interstitial liquid, with a ^{137}Cs content of 2.6 Ci/gal. The Tank 29H supernate had a reduction in ^{238}Pu from 1.32E+5 pCi/mL to 3.65E+4 pCi/mL upon filtering through a 0.45-micron filter. Approximately 25% of the U is in suspension in the supernate and can be removed by filtration.

As expected, the radionuclides Cs-137 and Tc-99 are entirely soluble and the actinides are largely (>90%) insoluble, in the interstitial liquid, within experimental error.

It is not known how representative this saltcake sample is for the entire tank. There is considerably more liquid present in this sample than in most other tank samples examined thus far. The history of the tank indicates that some of the saltcake in the top layer likely dissolved by the addition of low ionic strength solutions over the past few years. Also, the sample was collected at the bottom of a 7-foot well that was mined into the saltcake with water. These water additions could have dissolved the sodium nitrate in the top layer, and left behind a layer containing the remaining salts, including those of aluminum, although it is not possible to confirm this with this sample. Conversely, if this high aluminum composition is similar throughout the tank, more interstitial liquid may be released upon draining, and more will be retained by the remaining saltcake. The draining characteristics of a high aluminum composition waste are also expected to be different from those of sodium nitrate, although the impact is not known. The high concentration of aluminum species led to precipitation of aluminum hydroxide during a draining/dissolution test using deionized water. If this occurs during tank retrieval, it has significant impacts for tank waste retrieval schedules. This phenomenon would likely manifest as slow dissolution and percolation rates, depending on the volume of the high aluminum region, and the method of water addition. Further study is recommended to examine the quantity and conditions for sodium hydroxide additions needed to avoid precipitation of aluminum hydroxide. Even if this sample is not representative of the tank, the presence of layers of high aluminum-containing phases could cause formation of low permeability layers during subsequent dissolutions.

Only about 66% (by mass) of the original saltcake sample dissolved during the drain/dissolution demonstration using deionized water. The sum of aluminum that was removed in all the dissolution steps was only 27% of the total calculated present in the bulk sample. The volume of aluminum hydroxide solids remaining after dissolution was 76% of the original saltcake sample volume. Although the Pu was largely removed from the dissolved salt by filtration, this may not represent the behavior in a tank environment due to the slow kinetics of Pu dissolution in salt solution. The short contact times and low temperatures used in the experiments will not mimic the conditions in a waste tank. Furthermore, if sodium hydroxide is added to facilitate aluminum dissolution, this will impact the Pu solubility.

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List of Abbreviations

AA	Atomic Absorption spectroscopy
ADDS	Adjusted Drained Dissolved Salt
ADS	Analytical Development Section
DDS	Drained Dissolved Salt
DI	de-ionized
EDS	X-ray Emission Dispersive Spectroscopy
FL	Free Liquid supernatant to a saltcake sample upon receipt
ICP-ES	Inductively-Coupled Plasma – Emission Spectroscopy
ICP-MS	Inductively-Coupled Plasma – Mass Spectroscopy
IC	Ion Chromatography
I.D.	inner diameter
IL	Interstitial Liquid drained from a saltcake sample
ILD	Interstitial Liquid Displacement
LCS	Low-Curie Salt
LWD	Liquid Waste Disposition
O.D.	outer diameter
PIL	Partition into Interstitial Liquid
PLM	Polarized-Light Microscopy
SEM	Scanning Electron Microscopy
SIL	Simulated (non-radioactive) Interstitial Liquid
SL	Supernatant Liquid from a tank supernate sample
SPF	Saltstone Processing Facility
SRS	Savannah River Site
SRTC	Savannah River Technology Center
TIC	Total Inorganic Carbon
TOC	Total Organic Carbon
UDS	Un-drained Dissolved Salt
WCS	Waste Characterization System
XRD	X-Ray Diffraction spectroscopy

Introduction

Recently, the Savannah River Site (SRS) has taken interest in potential processes that could be utilized for accelerated processing of a fraction of High-Level Waste saltcake inventory. In support of this, Liquid Waste Disposition (LWD) has undertaken a program of tank characterization, including salt tank sampling.[1,2] As part of this initiative, the surface of the Tank 29H saltcake was sampled using a 12" long sample tube. The primary objective of the characterization is to gather information that will be useful to the selection and process sequencing of salt tanks. Key process parameters include the ^{137}Cs concentration and liquid retention properties of the Tank 29H saltcake. Additional chemical analyses are performed to provide information on salt elemental, ionic, and radiological composition to aid in assessment of the suitability of treated material in the disposal facility.

This document serves as the final and comprehensive record of the handling and analyses of the Tank 29H saltcake core and supernate samples.

Background

Tank 29H was the drop tank for the 242-H (1H) evaporator from 1971 through 1984. Additional, minor amounts of 1H evaporator waste were introduced in 1986 – 1988. The waste to the tank was characterized as high heat waste. Tank 29H received steam condensate from the 3H evaporator separator pot vent line from March, 1999 through January, 2003. Also during that period, nearly all of the free liquid above the saltcake (293 inches) was removed in three transfers, ending in December, 2001. The condensate continued to be added to the tank for more than a year following removal. In addition, about 21,000 gallons of rainwater from the 1H evaporator CTS pit has been added to the tank in the past three years.

This task documents characterization and dissolution studies performed on the top portion of the saltcake and a supernate surface dip sample that was collected separately.

Samples

Saltcake Core Samples

On August 26, 2003, one salt core sample was collected from riser B6 of Tank 29H. The sample was taken from the B6 riser at approximately 206 inches, which is 7 feet lower than the measured tank saltcake level (~290 inches). The reason it was 7 feet below the recognized tank salt cake level was because the area beneath the B6 riser had been mined out with water in preparation of installing a slurry pump. The temperature of the saltcake in the tank that day was 47 °C, with a supernate temperature of 37 °C. The tank liquid level was 315 inches. Additional samples were planned, but difficulty was encountered in recovering the second sample. This sample was contained in a 0.95 in. I.D., 1.13 in. O.D., 12.7 in. long stainless steel tube with capped ends. The sample was delivered to SRTC, placed into the shielded cells, and weighed on August 27.

Sample T29H-B6-1 contained 315.3 grams of saltcake that occupied 147.8 cm^3, for a bulk saltcake density of 2.13 g/cm^3. Figure 4 displays the approximate contents of the Tank 29H sample. The sample contained no supernatant free liquid. In order to complete the required analyses, the sample was divided into two approximately equal halves by cutting the filled tube, and the two halves of the sample tube were renamed (top half is T29H-B6-1-A, and the bottom half is T29H-B6-1-B). Salt for chemical analysis and water content measurement was taken from the bottom of the top half of sample T29H-B6-1-B.

Figure 2 shows the salt that was visible from the ends of the sample tube. From left to right, the images are of the top of the sample, the bottom of the top half of the severed sample, the top of the bottom half of the severed sample, and the bottom of the sample. The salt was white to light yellow/brown in color. The

middle of the sample glistened and was wetter in appearance than the top and bottom of the sample. Note that the top of the sample tube was filled to the point that no mast threads were visible.

Figure 3 shows the ~50 g of salt removed from the bottom of the top half of the sample. The salt appeared wet and exhibited thixotropic behavior. For example, when the salt was allowed to sit for ~15 minutes, the salt set up and resisted flow, and a spatula inserted vertically in the salt could be supported. After mixing the salt and not allowing it to set, a spatula could not be supported.
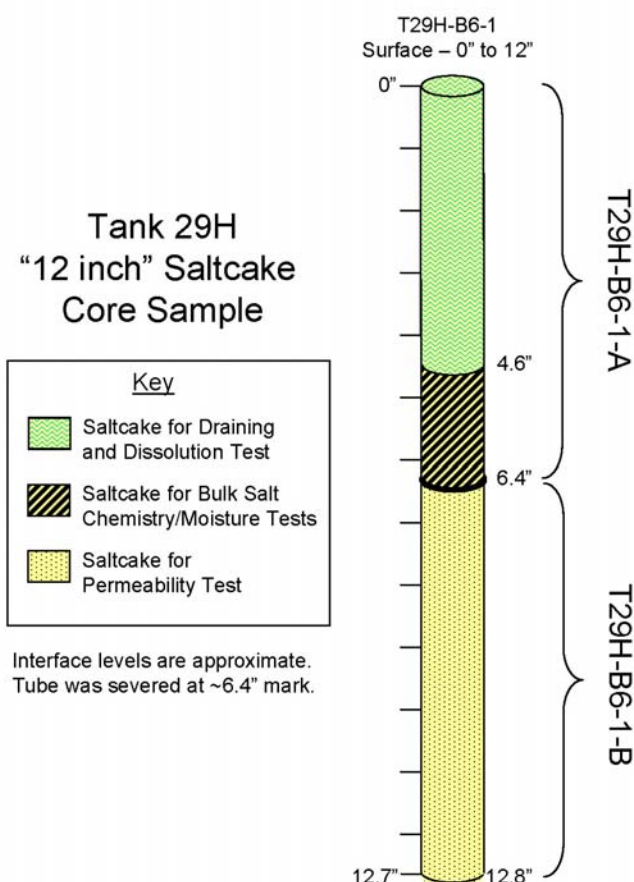
Figure 1: Material contained in Tank 29H core sample
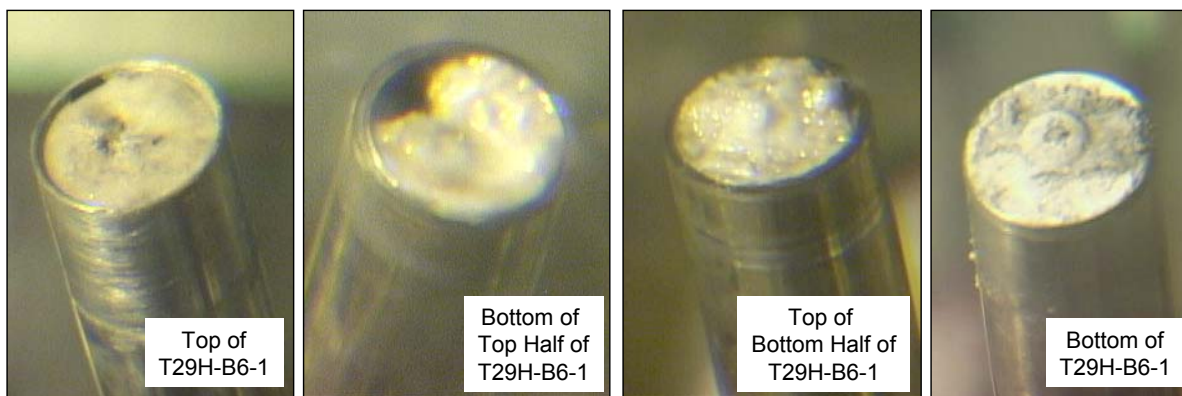
Figure 2: The top, middle (cut), and bottom of the Tank 29H saltcake core sample
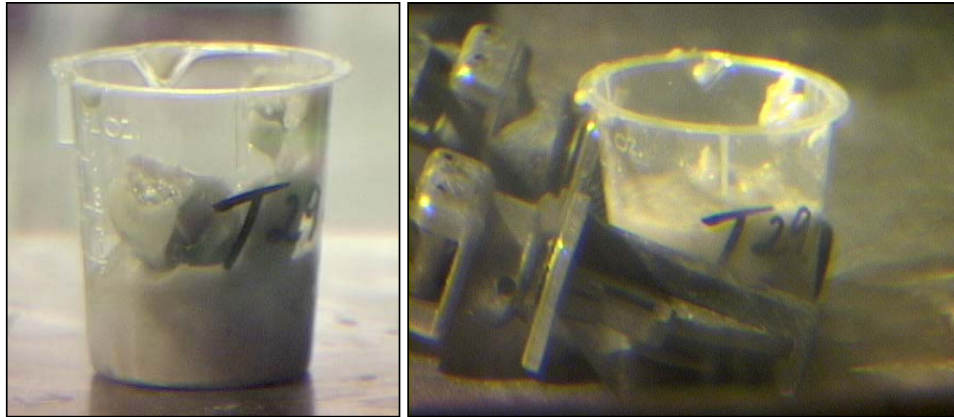
Figure 3: Salt collected from the bottom of the top half of the Tank 29H sample

Supernate Sample

On September 11, 2003, SRTC received a supernate sample from Tank 29H. The surface dip sample (HTF-588A) contained ~80 mL of supernate, pictured in Figure 4. The sample was cloudy with brown insoluble solids.
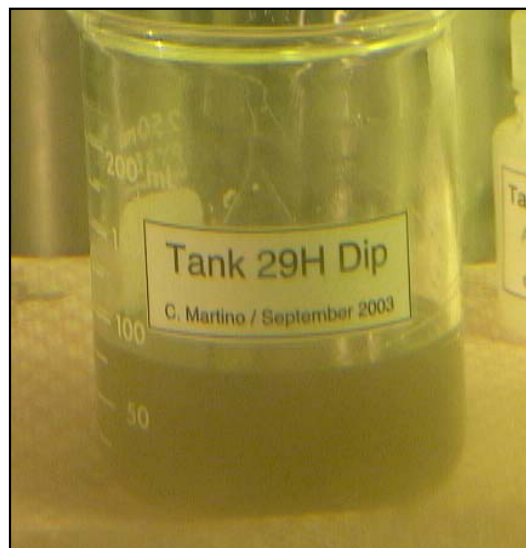
Figure 4: Tank 29H supernate sample HTF-588A, as received (unfiltered)

Experimental

Saltcake Core Sample

The core sample (T29H-B6-1) was weighed, uncapped, and visually inspected upon receipt in the shielded cells. The sample was cut into two approximately equal segments and salt was removed from the bottom of the top half of the sample for undrained bulk salt analysis. The remaining portion of the top half of the sample was vacuum drained for two days and the drained interstitial liquid was filtered and analyzed. The drained saltcake was subsequently dissolved with deionized (DI) water using the

drain/dissolve protocol. Details and analysis of the interstitial liquid and dissolved drained saltcake are provided herein.

Undrained bulk salt characterization was performed on small portions of salt removed from the bottom of the sample tubes. Approximately 50 grams of salt was removed from the bottom of the top half of T29H-B6-1 and analyzed for water content and salt chemistry. Undrained bulk salt subsamples were prepared for analysis either by aqua regia (acid) dissolution or by DI water dissolution. The dissolutions were performed by dissolving approximately 2 grams of the material into 100 mL of liquid. For the DI water dissolutions, aliquots were taken from the liquid portion after agitation and settling, although no significant solids were visible. The aqua regia dissolutions were submitted to radiological chemistry for ^{137}Cs (gamma scan), Pu isotopics, ^{241}Am, and ^{90}Sr, 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/dissolutions were submitted for ion chromatography (IC) for anion characterization, wet chemistry/titration for total base, free hydroxide, and CO_3^{2-} analysis, and total inorganic and organic carbon (TIC/TOC) analysis. An additional portion of the water-dissolved sample was submitted for ^{14}C analysis.

Several analytes require assumptions to be made or calculations and adjustments that require several analytical methods. The ^{99}Tc is reported as a maximum value based on the entire mass 99 data, and thus ignores any contribution of ^{99}Ru. The mass 101 values are reported in the appendix in case adjustment of ^{99}Tc for the contribution of natural Ru is desired. Total Cs is calculated as the sum of ^{133}Cs from ICP-MS and ^{137}Cs from radiochemistry. The ^{135}Cs is reported as a maximum value based on the entire mass 135 data, and thus ignores any contribution of ^{135}Ba. This provides a good measurement for the filtered liquid samples because of the limited Ba solubility, but mass 138 values are reported in the appendix in case adjustment of ^{135}Cs for the contribution of natural Ba is desired. Total Co is reported as mass 59, and thus may be biased high in the presence of organic components that fragment at mass 59. An alternative free hydroxide estimate is provided as a calculated value by taking the total base and subtracting the contribution of aluminate and carbonate ions. Total inorganic carbon is reported as mass of CO_3^2 and total organic carbon is reported as mass of carbon.

Supernate Sample

Upon entry into the SRTC shielded cells, the Tank 29H supernate sample (HTF-588A) was 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. An additional filtration was applied to the 29H samples using 0.1-micron filters and portions of the filtrates were analyzed.

Unfiltered and filtered supernate samples were prepared for analysis either by pressurized HNO_3/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 HNO_3/HF dissolutions of the filtered and unfiltered samples were submitted to radiological chemistry for ^{137}Cs (gamma scan), Pu isotopics, ^{241}Am, and ^{90}Sr, ICP-ES for elemental analysis, ICP-MS for certain actinide and fission product quantification, and AA for the measurement of As, Se, Hg, and K. The water dilutions of the filtered samples were submitted to IC for anion characterization, wet chemistry/titration for total base and free hydroxide, TIC/TOC analysis, and radiological chemistry for ^{14}C.

Draining and Permeability

The saltcake bulk density of the as-received material was calculated from a direct measurements of the sample weight and an estimate of the filled volume while the sample was still in the thin walled sample tube. Densities of the interstitial, free, and supernatant liquids were measured as the weight of material required to fill 2 mL Class A volumetric flasks. The water content of the saltcake (θ_{water}) and liquid

samples were estimated gravimetrically: small portions (~1g) of saltcake or liquid were dried at 115 °C (±5 °C) to drive off water until a constant weight had been achieved. The solids collected from the filtration of the supernate samples were washed with water and dried for determination of insoluble solids content.

The porosity and saturation of saltcake samples were estimated using the following equations.

$$\phi = \frac{V_{voids}}{V_{total}} = \frac{V_{total} - V_{solid}}{V_{total}}$$

$$V_{solid} = \frac{W_{solid}}{\rho_{solid}} = \frac{W_{total} - W_{liquid}}{\rho_{solid}}$$

$$W_{liquid} = \left(\frac{W_{total} * \theta_{water}}{\chi_{wil}} \right)$$

$$S = \frac{V_{liquid}}{V_{voids}} = \frac{\left(\dfrac{W_{liquid}}{\rho_{liquid}} \right)}{\left(V_{total} * \phi \right)}$$

S = saturation (fraction)
ϕ = porosity (fraction)
ρ_{soild} =density of solids
χ_{wil}= mass fraction of water in interstitial liquid
θ_{water} = gravimetric water content
V_{total} = total volume of sample
V_{solid} = volume of solids in sample
V_{voids} = volume of sample that does not correspond to solids
W_{total} = total weight of sample
W_{solid} = weight of solids in sample
W_{liquid} = weight of interstitial liquid in sample

Uncertainty in the density of the interstitial liquid, weight percent water, density of solids, and mass fraction of water in interstitial liquid was incorporated into the porosity estimate by performing a Monte Carlo analysis using Crystal Ball 2000 (by Decisioneering, Inc.), an add-in for MS Excel.

A falling head permeability test was conducted on sample T29H-B6-1-B. The permeability test method was similar to that used on saltcake samples from Tank 41.[3] The sample was not drained prior to initiating saturation with simulated interstitial liquid (SIL). The SIL used to saturate the sample was a 10.8 M [Na^+] solution with a density of 1.4 g/ml, as shown in Table 1.

Table 1: Chemical composition of simulated interstitial liquid used to saturated saltcake samples for falling head permeability test.

Component	Concentration
NaOH	6.8 M
NaNO3	1.5 M
NaNO2	2.5 M

Interstitial liquid (IL) was extracted from sample T29H-B6-1-A using 10" Hg vacuum for 160 hours. Ten inches of Hg corresponds to 98 inches of interstitial liquid (assuming a density of 1.4 gm/cc) and was selected to drain the sample to average saturation based on the conceptual model for drainage with sandy loam as a porous media analog (**Figure 5**).[4] Figure 6 shows the apparatus used to extract the IL. No screen was placed in the bottom shipping cap during vacuum extraction.
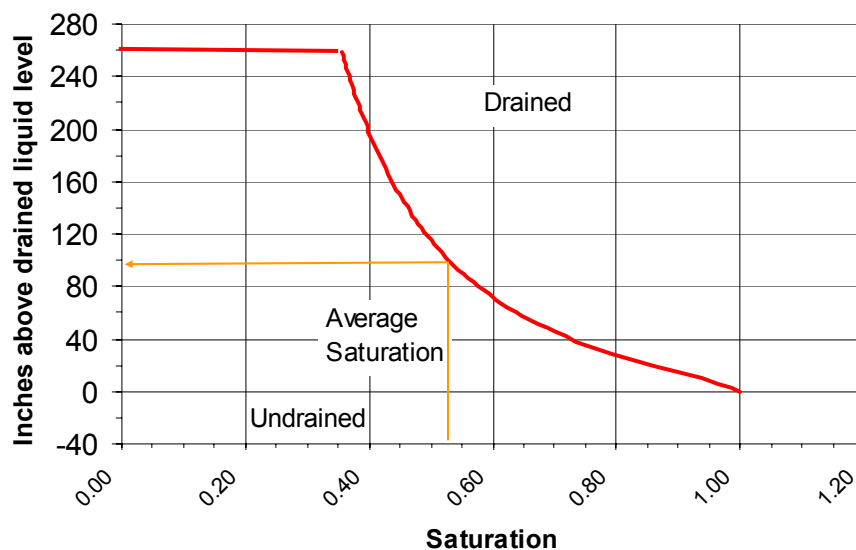
Figure 5: Theoretical drainage curve for salt cake in tank 29H with the top of salt at 300 inches, final liquid level in caisson of 40 inches, interstitial liquid density of 1.39 gm/cc, and sandy loam drainage curve.
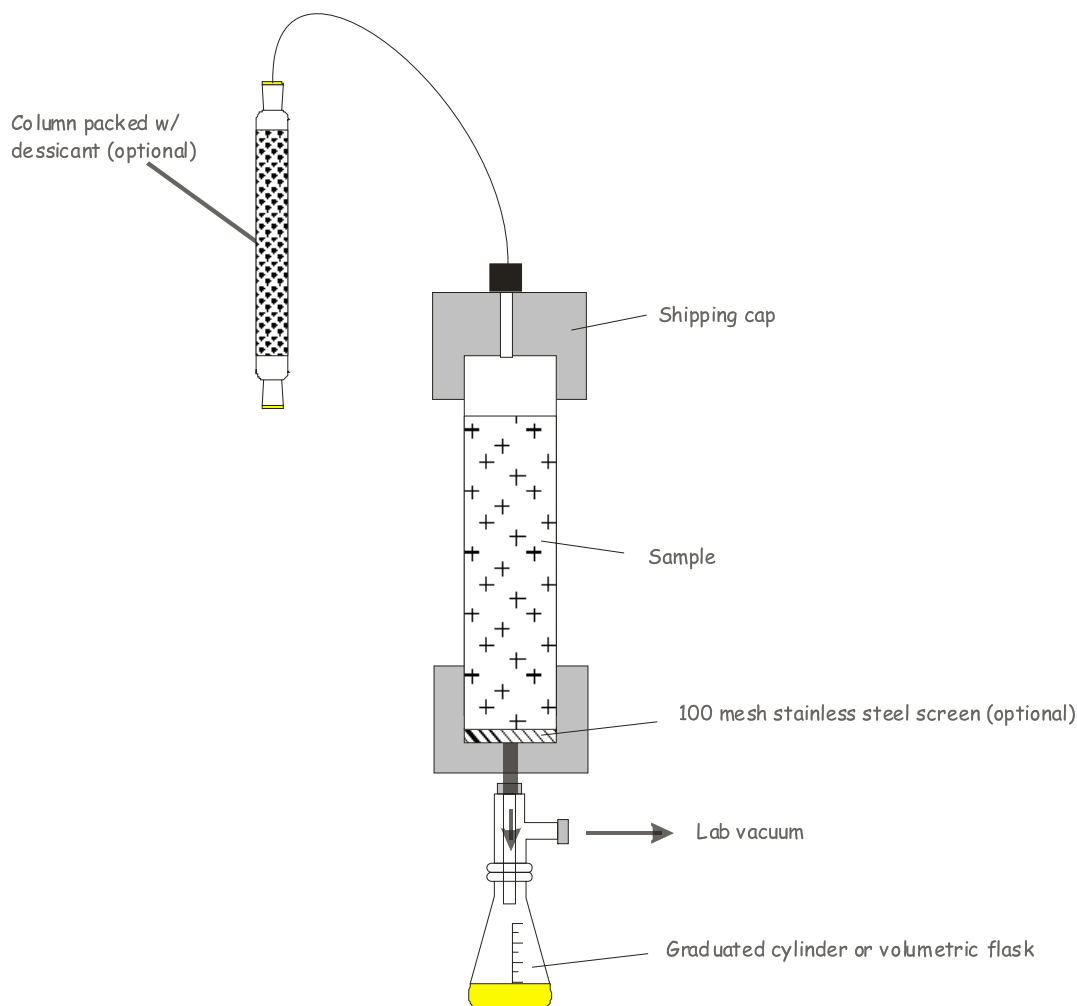
Figure 6: Schematic of apparatus used to extract interstitial liquid from saltcake samples

Dissolution of Drained Saltcake

Figure 7 contains a generalized illustration of the apparatus used during dissolution testing. The apparatus utilized the remaining drained portion of T29H-B6-1-A (6.4 inch tube with 4.6 inches of salt) in the original ~1 inch I.D., 1/16 inch wall thickness sampling tube. The dissolution apparatus was essentially the same as the vacuum-extraction apparatus (Figure 6), with the exception of the media bag for water introduction. The orientation of the sample was adjusted so that the larger void in the sample tube would be at the top of the dissolution apparatus. Because salt was removed from the bottom of T26H-B6-1-A for initial testing, the dissolution of T26H-B6-1-A was performed in an orientation that was inverted from the in-tank orientation of the sample. No screen was used at the bottom of the sample during the dissolution protocol.
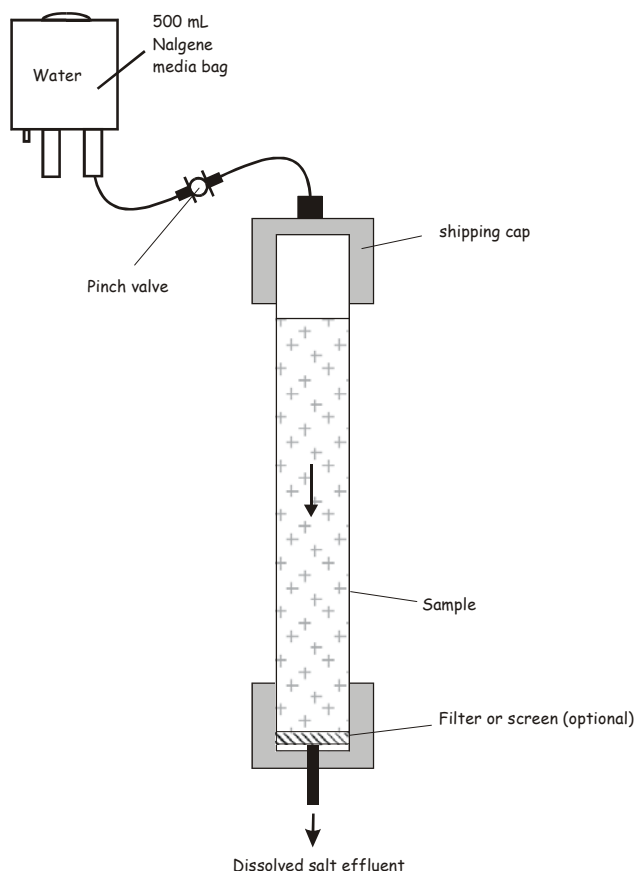
Figure 7: Schematic of dissolution test apparatus

Water was introduced to the top of the sample tube via a media bag and the dissolved salt effluent was collected from the bottom of the sample. The rate of water introduction can be roughly controlled by a pinch valve, but the rate of effluent elution can be limited by the saltcake physical properties. In the case of this Tank 29H sample, liquid was not evident at the bottom of the sample after several hours of dissolution by gravity draining alone. To assist the flow of dissolution fluid through the sample, vacuum was applied to the bottom of the sample in a manner similar to the process for draining interstitial liquid. During later stages of the dissolution of the Tank 29H sample, vacuum was no longer needed.

The dissolved salt effluent was periodically sampled in 5 mL aliquots. The collection of one of these aliquots was timed to obtain an estimate of the effluent flow rate. These aliquots were weighed to obtain the effluent density. During periods of salt dissolution when an aliquot was not collected, the dissolved salt effluent was collected in a larger graduated bottle. For the remainder of this report, the small aliquots are referred to as sub-samples of drained/dissolved saltcake and the larger bottles of effluent are referred to as the bulk drained/dissolved saltcake.

Analysis of the composite bulk drained/dissolved saltcake was performed after filtration of the material through a 0.45-micron filter. Analysis of sub-samples taken at various stages throughout the flow-through dissolution process was performed without filtration. Subsamples were prepared for chemical and radiochemical analyses at 20 to 50-times dilutions with 2 M nitric acid and DI water. The nitric acid dilutions were submitted for the same analyses as were the acid dissolutions of the core samples. The analyses included radiological chemistry, ICP-ES, ICP-MS, and AA. The water dilutions of the filtered samples were submitted for IC, wet chemistry titration, and TIC/TOC. The filter was analyzed by microscopy and spectroscopy techniques to identify solid phases and particle size. Two values are reported for ^{99}Tc by ICP-MS: a maximum based on the entire mass 99 data as ^{99}Tc, and a minimum number adjusting for the potential ^{99}Ru assuming that the mass 101 result is from natural ^{101}Ru.

Results

Sample Density and Moisture Content

The salt removed from the bottom of T29H-B6-1-A had a water content of 21.4 wt % with a standard deviation of 3.1 wt %, as shown in Table 2. The half-width of the 95% confidence interval for the water content was 3.2 wt %. The density of the bulk salt was 2.13 g/cm^3.

Table 2: Density and water content of Tank 29H Samples

Analyte	Unit	T29H-B6-1				HTF-588A	
		undrained saltcake		FL (filtered)		Supernate	
		average	stdev	average	stdev	average	stdev
Density	g/mL	2.13	--	1.48	--	1.26	0.02
Water	wt %	21.4	3.1	*54.7*	--	69.8	--
Liquid	vol %	*56*	--	100	--	100	--

note: the wt% water in the filtered liquid was calculated based on summing individual analyses of cations and anions;

note the vol% liquid in the undrained saltcake is calculated based on the wt% water in saltcake and wt% water in filtered liquid

Several (~9) milliliters of interstitial liquid (IL) had been vacuum drained from the remaining portion of T29H-B6-1-A. The density of the interstitial liquid was 1.48 g/cm^3. There was not enough interstitial liquid to determine its viscosity.

The Tank 29H supernate sample (HTF-588A) had a density of 1.26 g/cm^3 with a standard deviation from four measurements of 0.02 g/cm^3. The samples had a total solids content (which is approximately all soluble solids) of 30.2 wt % with a standard deviation from three measurements of 0.4 wt %.

Chemical and Radiological Analyses

Tables 3 through 6 contain the characterization information for the undrained bulk saltcake, interstitial liquid, and dip samples. Results are reported as the average of the individual analyses. Details of the analysis results, along with standard deviations, are shown in the appendix (Tables 10 – 14 for undrained bulk saltcake from T29H-B6-1, Tables 15 – 18 for interstitial liquid drained from T29H-B6-1, and Tables 19 – 23 for supernate sample HTF-588A). Values reported at less than their detection limits are preceded by "<" and values that contain an average of detectable concentrations and detection limits are preceded by "</=".

The last column in Tables 3-5 shows the percentage of each component that is partitioned into the interstitial liquid Subtracting the "Partition into IL" from 100% yields an estimate for each component of the percentage contained in the solid phase of the original saltcake sample. To calculate the "Partition into IL", the wt % of the component measured in the filtered interstitial liquid (drained from T3F-1-4) is multiplied by the wt % of IL in the undrained saltcake and divided by the wt % of the component measured in the undrained saltcake.

The portion of undrained saltcake that was analyzed contained a relatively large amount of aluminum (Al of 13.3 wt %) and a relatively small amount of sodium nitrate (NaNO$_3$ of 7.4 wt %). The ^{137}Cs activity of the sample was 6.40E+8 pCi/g, which corresponds to 5.2 Ci per gallon of saltcake.

The total strontium in the undrained saltcake sample was calculated by adjusting the ICP-MS observation of ^{88}Sr and the radiochemistry observation of ^{90}Sr. The total strontium in the Tank 29H sample was 5.35E-5 wt %, of which ^{90}Sr had an 18.5 wt % isotopic contribution to the total strontium.

The interstitial liquid had a ^{137}Cs activity of 1.69E+9 pCi/g (2.50E+9 pCi/mL), which is 9.5 Ci per gallon of IL. This ^{137}Cs content is significantly higher than the 4.6 Ci/gal expected from the average of Tank 29H supernate samples from 2001, but it is within the realm of expectations based on Tank 29H supernate samples from the mid 1980's through mid 1990's.[5] The interstitial liquid also contained 38.1 mg/L of ^{99}Tc, corresponding to 6.5E+5 pCi/mL of ^{99}Tc.

The ^{137}Cs activity of the as-received supernate sample was 6.92E+8 pCi/mL, which corresponds to 2.6 Ci/gal. The as-received sample contained 1.05 pCi/g (1.32E+5 pCi/mL) of ^{238}Pu, of which approximately 72% was removed upon filtration with a 0.45-micron filter. Due to the uncertainty with these empirical data, it can not be concluded whether additional filtration with a 0.1-micron filter provided additional removal of actinide components.

Table 3: Saltcake, Interstitial liquid, and Dip Sample Radiological Analysis Comparison

		T29H-B6-1		Tk 29 Dip (HTF-588A)			
		Undrained saltcake	Interstitial liquid	Unfiltered Supernate	Filtered (0.45 µm) Supernate	Filtered (0.1 µm) Supernate	PIL
Analyte	Units	Average	Average	Average	Average	Average	%
^{14}C	(pCi/g)	9.9E+02	--	--	1.20E+03	--	--
^{90}Sr	(pCi/g)	1.35E+07	1.14E+06	1.14E+04	1.26E+05	9.64E+04	3.3
^{99}Tc	(pCi/g)	2.01E+05	4.36E+05	1.40E+05	1.45E+05	1.38E+05	84.7
^{135}Cs	(pCi/g)	2.76E+03	6.55E+03	2.18E+03	2.25E+03	2.15E+03	92.7
^{137}Cs	(pCi/g)	6.40E+08	1.69E+09	5.50E+08	5.68E+08	5.72E+08	103.3
^{230}Th	(pCi/g)	< 5.7E+02	< 6.8E+02	< 1.3E+03	< 1.3E+03	< 1.2E+03	--
^{232}Th	(pCi/g)	6.23E-02	< 3.7E-03	< 7.2E-03	< 7.2E-03	< 6.4E-03	< 2.3
^{233}U	(pCi/g)	1.89E+03	< 3.3E+02	< 6.4E+02	< 6.3E+02	< 5.7E+02	< 6.7
^{234}U	(pCi/g)	2.43E+03	< 2.1E+02	< 4.1E+02	< 4.1E+02	< 3.7E+02	< 3.4
^{235}U	(pCi/g)	6.62E+00	1.59E-01	3.96E-01	2.51E-01	2.29E-01	0.9
^{236}U	(pCi/g)	8.91E+01	2.66E+00	4.54E+00	< 4.2E+00	< 3.8E+00	1.2
^{237}Np	(pCi/g)	7.46E+01	< 2.4E+01	< 4.6E+01	< 4.6E+01	< 4.1E+01	< 12.4
^{238}U	(pCi/g)	4.95E+00	9.43E-01	4.47E-01	3.54E-01	3.03E-01	7.5
^{238}Pu	(pCi/g)	1.12E+06	7.61E+03	1.05E+05	2.90E+04	2.70E+04	0.3
$^{239/240}$Pu	(pCi/g)	1.79E+04	2.13E+03	2.11E+03	8.76E+02	< 6.60E+02	4.7
^{239}Pu	(pCi/g)	9.53E+03	< 2.1E+03	< 4.1E+03	< 4.1E+03	< 3.6E+03	< 8.6
^{240}Pu	(pCi/g)	< 6.4E+03	< 7.6E+03	< 1.5E+04	< 1.5E+04	< 1.3E+04	--
^{241}Pu	(pCi/g)	< 2.9E+06	< 3.5E+06	< 6.8E+06	< 6.7E+06	< 6.0E+06	--
^{241}Am	(pCi/g)	1.13E+04	1.82E+03	< 5.00E+02	< 5.43E+02	< 3.83E+02	6.3
^{242}Pu	(pCi/g)	< 1.1E+02	< 1.3E+02	< 2.6E+02	< 2.6E+02	< 2.3E+02	--
Total U	wt%	2.0E-03	< 3.0E-04	--	--	--	--
U-235 enrichment	wt%	1.6E+01	2.47E+00	--	--	--	--

Table 4: Saltcake, Interstitial liquid, and Dip Sample Principal Component Comparison

Analyte			T29H-B6-1		Tk 29 Dip	
			Undrained saltcake	Interstitial liquid	Filtered (0.45 µm) Supernate	
		Units	Average	Average	Average	PIL %
Na^+		wt%	2.13E+01	1.83E+01	9.98E+00	33.7
NO_3^-		wt%	5.38E+00	7.71E+00	8.30E+00	56.1
NO_2^-		wt%	3.03E+00	8.25E+00	2.57E+00	106.6
SO_4^{2-}		wt%	2.19E+00	4.40E-02	5.36E-01	0.8
PO_4^{3-}		wt%	7.91E-02	2.96E-01	3.10E-02	146.5
Cl^-		wt%	2.02E-01	</=1.22E-02	4.77E-03	< 2.4
F^-		wt%	< 9.68E-03	< 9.79E-03	1.43E-03	--
$C_2O_4^{2-}$		wt%	< 4.84E-02	< 4.89E-02	7.15E-03	--
CHO_2^-		wt%	< 4.84E-02	< 4.89E-02	< 4.8E-02	--
Free OH-		wt%	7.71E+00	5.32E+00	2.04E+00	27.0
AlO_2^-		wt%	2.9E+01	3.84E+00	1.47E+00	5.2
CO_3^{2-}		wt%	3.02E+00	8.48E-01	1.88E+00	11.0
K^+		wt%	8.06E-02	2.95E-01	< 7.66E-02	143.3
TOC		wt%	3.9E-01	2.55E-01	3.00E-01	25.5
Total Base		wt% as OH⁻	1.49E+01	9.90E+00		25.9
Mass Balance			94.0	--	97.0	
Charge balance		% (cation-anion)	-35.3	10.6	6.1	
wt% solid		calculated wt%	7.26E+01	4.53E+01	2.72E+01	
wt% water		calculated wt%	2.74E+01	5.47E+01	7.28E+01	

Table 5: Saltcake, Interstitial liquid, and Dip Sample Elemental Comparison

		T29H-B6-1		Tk 29 Dip (HTF-588A)			
		Undrained saltcake	Interstitial liquid	Unfiltered Supernate	Filtered (0.45 µm) Supernate	Filtered (0.1 µm) Supernate	
Analyte	Units	Average	Average	Average	Average	Average	PIL %
Ag	wt %	< 6.94E-04	< 7.81E-05	< 7.35E-04	< 7.33E-04	< 6.53E-04	--
Al	wt %	1.33E+01	1.76E+00	1.98E-01	6.73E-01	5.11E-01	5.2
B	wt %	< 7.50E-02	1.02E-02	< 7.94E-02	< 7.91E-02	< 7.06E-02	--
Ba	wt %	< 6.94E-04	2.64E-04	< 7.35E-04	< 7.33E-04	< 6.53E-04	--
Ca	wt %	< 2.10E-02	< 2.29E-03	< 2.22E-02	< 2.21E-02	< 1.97E-02	--
Cd	wt %	< 9.25E-04	< 1.01E-04	< 9.80E-04	< 9.77E-04	< 8.71E-04	--
Ce	wt %	< 1.15E-02	< 1.25E-03	< 1.22E-02	< 1.21E-02	< 1.08E-02	--
Cr	wt %	2.13E-02	2.96E-02	6.45E-03	1.02E-02	8.58E-03	54.5
Cu	wt %	< 1.44E-03	2.03E-03	< 1.52E-03	< 1.51E-03	< 1.35E-03	--
Fe	wt %	1.08E-02	1.17E-02	< 1.08E-03	< 1.07E-03	< 9.58E-04	42.6
Gd	wt %	< 1.25E-03	< 1.38E-04	< 1.32E-03	< 1.32E-03	< 1.18E-03	--
K	wt %	< 4.41E-01	2.95E-01	< 4.67E-01	< 4.65E-01	< 4.15E-01	--
La	wt %	< 9.25E-04	</=1.1E-04	< 9.80E-04	< 9.77E-04	< 8.71E-04	--
Li	wt %	< 3.94E-03	6.00E-03	< 4.16E-03	< 4.15E-03	< 3.70E-03	--
Mg	wt %	</=3.99E-03	1.31E-03	< 3.04E-03	< 3.03E-03	< 2.70E-03	--
Mn	wt %	</=2.20E-03	2.86E-03	< 1.08E-03	< 1.07E-03	< 9.58E-04	--
Mo	wt %	1.97E-02	2.65E-02	< 1.00E-02	< 9.97E-03	9.13E-03	52.5
Na	wt %	2.13E+01	1.83E+01	8.91E+00	9.98E+00	9.17E+00	33.7
Ni	wt %	< 3.47E-03	1.2E-03	< 3.67E-03	< 3.66E-03	< 3.27E-03	--
P	wt %	4.45E-02	1.03E-01	< 2.38E-02	< 2.37E-02	</=2.12E-02	90.5
Pb	wt %	< 1.14E-02	< 1.25E-03	< 1.21E-02	< 1.20E-02	< 1.07E-02	--
S	wt %	6.67E-01	7.38E-02	1.95E-01	1.33E-01	1.91E-01	4.3
Sb	wt %	3.67E-02	5.35E-03	< 7.40E-03	< 7.38E-03	< 6.58E-03	5.7
Si	wt %	2.24E-02	1.77E-02	6.86E-02	6.50E-02	5.38E-02	30.9
Sn	wt %	1.30E-02	2.79E-03	< 1.20E-02	< 1.20E-02	< 1.07E-02	8.4
Sr	wt %	< 4.63E-03	< 5.06E-04	< 4.90E-03	< 4.89E-03	< 4.36E-03	--
Ti	wt %	< 2.78E-04	< 3.22E-05	< 2.94E-04	< 2.93E-04	< 2.61E-04	--
Zn	wt %	< 2.55E-03	4.18E-03	< 2.69E-03	< 2.69E-03	< 2.40E-03	--
Zr	wt %	< 5.56E-04	1.1E-04	< 5.88E-04	< 5.86E-04	< 5.23E-04	--

Table 6: Likely major components of dry Tank 29H salt solids, normalized

Species	estim. wt%
$NaNO_3$	2.26
Na_2SO_4	4.98
NaCl	0.50
NaF	0.01
$Na_2C_2O_4$	0.05
$NaCHO_2$	0.04
$NaAlO_2 \cdot 2H_2O$	83.95
$Na_2CO_3 \cdot H_2O$	8.20

Dissolution of Drained Saltcake

Approximately 46.3 g of salt was removed from the bottom of the top half of T29H-B6-1-A for as-received characterization, leaving about 112.2 g (52.7 mL, calculated) of undrained saltcake in the top half of T29H-B6-1-A. The remaining salt was drained under 10 inches Hg vacuum for 6.8 days, removing 13.3 g of interstitial liquid. At the conclusion of this draining (and the start of the dissolution test), approximately 98.9 g of drained saltcake remained in T29H-B6-1-A.

As water was added during dissolution, only several drops of effluent emerged from the sample initially. Vacuum was applied to the bottom of the apparatus to force liquid to flow through the sample. Only 5 mL was removed during the remaining 6 hours of the first dissolution shift, which became the first sub-sample. The following day, water was again applied to the sample while vacuum was used to assist flow through the sample. After 2 hours, a 61 mL slug of liquid and solids suddenly emerged from the bottom of the sample and was collected. At this time, the vacuum was stopped and a 5 mL portion of the liquid and solids became the second sub-sample. The remaining 56 mL of the material that was pulled through the sample became the beginning of the bulk dissolved salt effluent. When water was reapplied to the saltcake, the remainder of the dissolution proceeded without assistance of vacuum. The effluent did not reach the target density of 1.3 g/mL, likely due to the relatively fast water addition rate (~9 mL/min) and the possible channeling through the sample. Salt was completely removed from the sample during the test.

Tables 7 through 9 contain the T29H-B6-1 dissolution information and analysis of the unfiltered sub-samples and filtered bulk dissolved salt effluent. The anion-cation balance is in poor agreement for the second sub-sample and the density of the second sub-sample is estimated from the sodium concentration. The ^{137}Cs content of the first sub-sample was 9.0 Ci/gal at 11.5 M Na^+, indicating that the first 5 mL of effluent vacuum extracted was essentially the same as the original interstitial liquid. Although the Tank 29H test did not run smoothly, as expected, the ^{137}Cs concentration of subsequent sub-samples again declined more quickly than the Na^+. The bulk dissolved salt effluent had a ^{137}Cs of 0.32 Ci/gal at 3.5 M Na^+. The ^{238}Pu content of the filtered bulk salt effluent was less than the ^{238}Pu content of the unfiltered sub-samples, indicating that ^{238}Pu can be removed by filtration (although it was not proven that the Pu was at thermodynamic equilibrium when filtered). The total alpha content of the filtered bulk salt effluent was < 2 nCi/mL..

Figure 8 contains a photo of the four sub-samples (glass graduated bottles in foreground, ordered left to right) and the bulk salt effluent (larger polyethylene bottle) collected during the dissolution of the top half of T29H-B6-1. The insoluble solids were not re-suspended before the photo was taken. The second sub-sample, corresponding to a portion of the slug of material that broke through the saltcake, contained 30 to 50 vol. % of white insoluble solids. The bulk sample contained about 40 mL of insoluble solids in the 220 mL sample. The solids were not included in the analysis of that liquid.

Figure 9 contains the XRD analysis of the insoluble solids filtered from the Tank 29H bulk dissolved salt effluent. The only crystalline material identified in the insoluble solids was the aluminum hydroxide $(Al(OH)_3)$ polymorphs: gibbsite and bayerite. From the estimated composition of the saltcake, these materials may have been present initially, but more likely formed from disproportionation of sodium aluminate. The fine particulate aluminum hydroxide likely built up at the top of the saltcake during dissolution, prohibiting the water from contacting additional salt. This phenomenon was noted by Mississippi State University DIAL during similar dissolution testing of Hanford high-aluminum saltcake simulant.[6]

Figures 10 and 11 contain plots of the analysis of selected components during the dissolution tests. Because of the difficulty with dissolution in the second step, the results are not representative of a fully saturated solution and may not be in thermodynamic equilibrium with the saltcake. The cation-anion balance indicates a problem with the analysis or sample preparation.

Figures 12 and 13 contain two views from the Scanning Electron Microscope photograph of the insoluble solids remaining from the dissolution test, and the Energy Dispersive Spectra (X-ray) analysis of the first photograph is shown in Figure 14. The solids appear homogeneous, but not very crystalline. The minimal amount of sodium present indicates that the solids are almost entirely aluminum hydroxide or oxides. The particle size is not easily discernible from these photographs. Although a bulky particle is shown in Figure 12 that is 30+ microns across, it appears to be a conglomeration of submicron-sized particles. Figure 13 indicates suggests particles that are 2-3 microns in diameter, but the photo does not exhibit discreet particles.

Table 7: Tank 29H drained saltcake dissolution information and analysis of dissolved saltcake for radiochemical and ionic components.

Sample T29H-B6-1	1st	2nd	3rd	4th	Filtered Bulk	
Time (min)	0 to 360	360 to 480	488 to 489	502 to 503	0 to 503	
Flow Rate (mL/min)	N/A	N/A	9.0	N/A	--	
Volume (mL)	0.0 to 5.0	5.0 to 66	116 to 121	181 to 186	0 to 216	
Density (g/mL)	1.43	1.3 *	1.18	1.07	1.12	
Radioisotopic Composition (pCi/mL)					avg.	st.dev.
^{90}Sr	--	--	--	--	7.37E+03	1.0E+03
^{99}Tc minimum	--	--	--	--	2.03E+04	1.0E+03
^{99}Tc maximum	--	--	--	--	2.10E+04	1.1E+03
^{137}Cs	2.38E+09	4.37E+07	1.11E+07	3.55E+06	8.40E+07	1E+06
^{238}Pu	2.21E+04	1.24E+03	2.39E+04	1.82E+03	< 7.02E+02	--
$^{239/240}Pu$	6.83E+02	< 4.45E+02	6.69E+02	< 3.55E+02	< 1.08E+03	--
^{241}Am	--	--	--	--	< 2.26E+02	--
Ionic Composition (M)					avg.	st.dev.
Na^+	1.15E+01	5.99E+00	2.09E+00	1.62E+00	3.48E+00	5E-02
NO_3^-	1.15E+00	2.81E-02	9.23E-02	1.99E-02	3.90E-01	4E-03
NO_2^-	1.60E+00	1.47E-02	5.64E-02	5.38E-03	8.42E-02	1E-03
OH^-	3.17E+00	4.23E-01	9.78E-01	2.42E-01	8.66E-01	1.65E-01
AlO_2^-	1.01E+00	7.52E-01	3.49E-01	1.68E-01	4.25E-01	1E-03
CO_3^{2-}	8.24E-02	2.21E-01	7.45E-01	3.25E-01	4.91E-01	3.3E-02
SO_4^{2-}	1.43E-02	5.33E-02	1.69E-01	5.15E-02	9.53E-02	2.6E-03
PO_4^{3-}	2.42E-02	5.09E-04	< 1.03E-03	< 5.21E-04	1.45E-03	5.9E-04
Cl^-	4.14E-03	< 2.73E-04	< 2.76E-04	< 2.79E-04	< 1.11E-03	--
F^-	< 5.16E-03	< 5.09E-04	5.15E-04	< 5.21E-04	< 2.08E-03	--
$C_2O_4^{2-}$	< 5.56E-03	< 5.49E-04	5.56E-04	< 5.62E-04	< 4.48E-04	--
CHO_2^-	< 1.09E-02	< 1.07E-03	< 1.09E-03	< 1.10E-03	< 4.38E-03	--
Total Base	5.09E+00	1.63E+00	3.59E+00	6.43E-01	2.68E+00	1.1E-01
K^+	< 1.73E-01	< 1.57E-01	< 2.14E-01	< 1.30E-01	2.93E-03	1.9E-04

N/A: Not Applicable. The flow rates in some cases could not be determined due to their irregular rates or the presence of solids.

Table 8: Other Elemental Analysis of the Dissolution of Drained Tank 29H Saltcake

Sample T29H-B6-1	1st	2nd	3rd	4th	Filtered Bulk	
Other Elemental Composition (mg/L)					avg.	st.dev.
Ag	< 1.06E+01	< 9.65E+00	< 1.32E+01	< 7.99E+00	< 3.33E+00	--
As	--	--	--	--	< 1.11E+00	--
B	< 1.15E+03	< 1.04E+03	< 1.43E+03	< 8.64E+02	< 3.60E+02	--
Ba	1.27E+01	< 9.65E+00	< 1.32E+01	< 7.99E+00	< 3.33E+00	--
Ca	< 3.22E+02	< 2.91E+02	< 3.98E+02	< 2.41E+02	< 1.00E+02	--
Cd	< 1.42E+01	< 1.29E+01	< 1.76E+01	< 1.07E+01	< 4.44E+00	--
Ce	< 1.76E+02	1.60E+02	2.18E+02	1.32E+02	< 5.50E+01	--
Co	--	--	--	--	< 1.85E-01	--
Cr	4.58E+02	< 1.09E+01	< 1.50E+01	< 9.07E+00	1.75E+01	3.96E-01
Cs	--	--	--	--	2.63E+00	
Cu	4.24E+01	< 1.99E+01	< 2.72E+01	< 1.65E+01	< 6.88E+00	--
Fe	3.02E+01	< 1.42E+01	< 1.93E+01	< 1.17E+01	< 4.88E+00	--
Gd	< 1.92E+01	< 1.74E+01	< 2.37E+01	< 1.44E+01	< 5.99E+00	--
Hg	--	--	--	--	< 2.44E+00	--
La	< 1.42E+01	< 1.29E+01	< 1.76E+01	< 1.07E+01	< 4.44E+00	--
Li	< 6.04E+01	< 5.46E+01	< 7.47E+01	< 4.53E+01	< 1.88E+01	--
Mg	< 4.41E+01	< 3.99E+01	< 5.46E+01	< 3.30E+01	< 1.38E+01	--
Mn	< 1.56E+01	< 1.42E+01	< 1.93E+01	< 1.17E+01	< 4.88E+00	--
Mo	3.61E+02	< 1.31E+02	< 1.79E+02	< 1.08E+02	< 4.52E+01	--
Ni	< 5.32E+01	< 4.82E+01	< 6.60E+01	< 4.00E+01	< 1.66E+01	--
P	1.29E+03	< 3.12E+02	< 4.27E+02	< 2.58E+02	< 1.08E+02	--
Pb	< 1.75E+02	< 1.59E+02	< 2.17E+02	< 1.31E+02	< 5.45E+01	--
S	1.37E+03	8.10E+03	2.25E+03	1.91E+03	3.80E+03	7.13E+01
Sb	< 1.07E+02	< 9.71E+01	< 1.33E+02	< 8.05E+01	3.66E+01	1.58E+00
Se	--	--	--	--	< 1.11E+00	--
Si	< 2.63E+01	< 2.38E+01	< 3.25E+01	< 1.97E+01	< 8.21E+00	--
Sn	< 1.75E+02	< 1.57E+02	< 2.16E+02	< 1.31E+02	< 5.43E+01	--
Sr	< 7.10E+01	< 6.44E+01	< 8.79E+01	< 5.34E+01	< 2.22E+01	--
Ti	< 4.26E+00	< 3.86E+00	< 5.28E+00	< 3.20E+00	< 1.33E+00	--
V	< 7.81E+00	< 7.07E+00	< 9.68E+00	< 5.86E+00	< 2.44E+00	--
Zn	7.57E+01	< 3.54E+01	< 4.83E+01	< 2.94E+01	< 1.22E+01	--
Zr	< 8.51E+00	< 7.71E+00	< 1.06E+01	< 6.40E+00	< 2.67E+00	--

Table 9: ICP-MS Analysis of the Dissolution of Drained Tank 29H Saltcake (Bulk Filtered)

T29H-B6-1 Bulk Dissolution		
(mg/L)	Average	StDev
Mass 59 (Co)	< 1.85E-01	--
Mass 88 (Sr)	< 2.49E-02	--
Mass 99 (Tc,Ru)	1.39E+00	7E-02
Mass 101 (Ru)	6.10E-02	3.5E-03
Mass 133 (Cs)	1.66E+00	2E-02
Mass 135 (Cs,Ba)	2.88E-01	1.3E-02
Mass 137 (Cs,Ba)	9.16E-01	1.7E-02
Mass 138 (Ba)	< 3.55E-02	--
Mass 230 – 234	< 2.28E-02	--
Mass 235 (U)	4.69E-02	1.08E-02
Mass 236 (U)	2.36E-02	8E-04
Mass 237 (Np)	< 2.28E-02	--
Mass 238 (U,Pu)	3.95E-01	9.2E-02
Mass 239 – 245	< 2.28E-02	--
Total U (mg/L)	4.66E-01	1.0E-01
^{235}U enrichment (%)	10.1%	0.1%
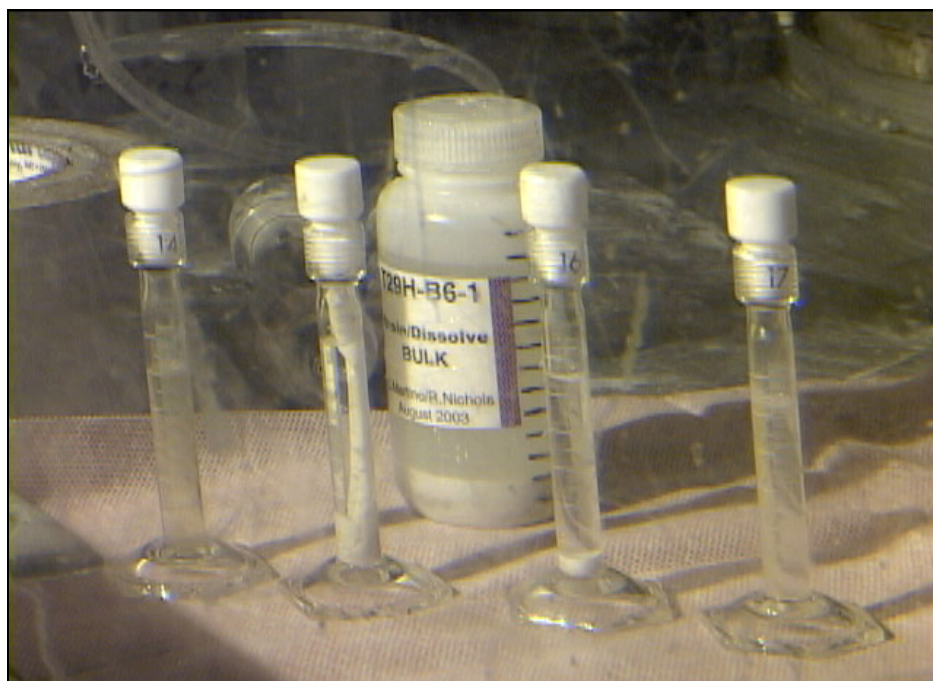
Figure 8: Tank 29H Dissolved Samples
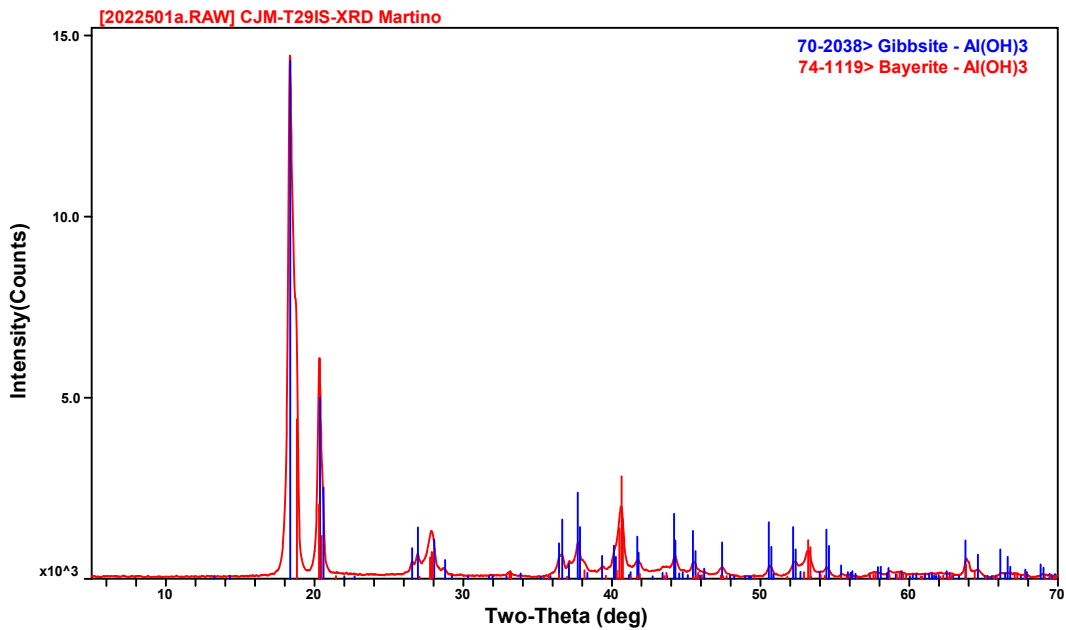
Figure 9: XRD analysis of insoluble solids resulting from dissolution of Tank 29H saltcake
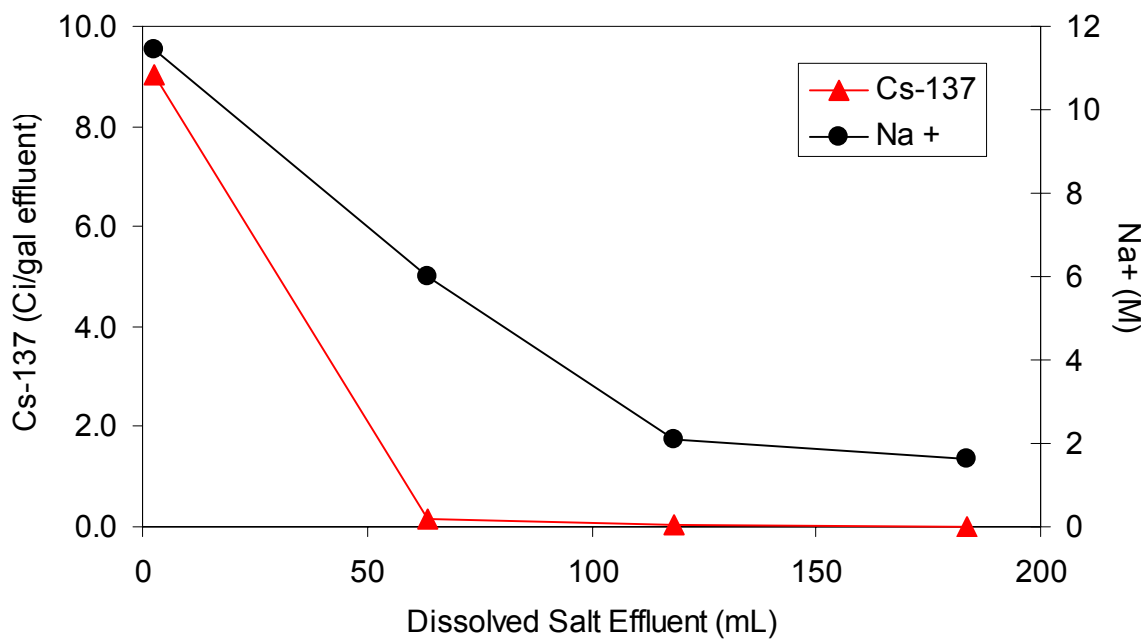
Figure 10: Tank 29H flow-through dissolution profiles for ^{137}Cs and Na$^+$
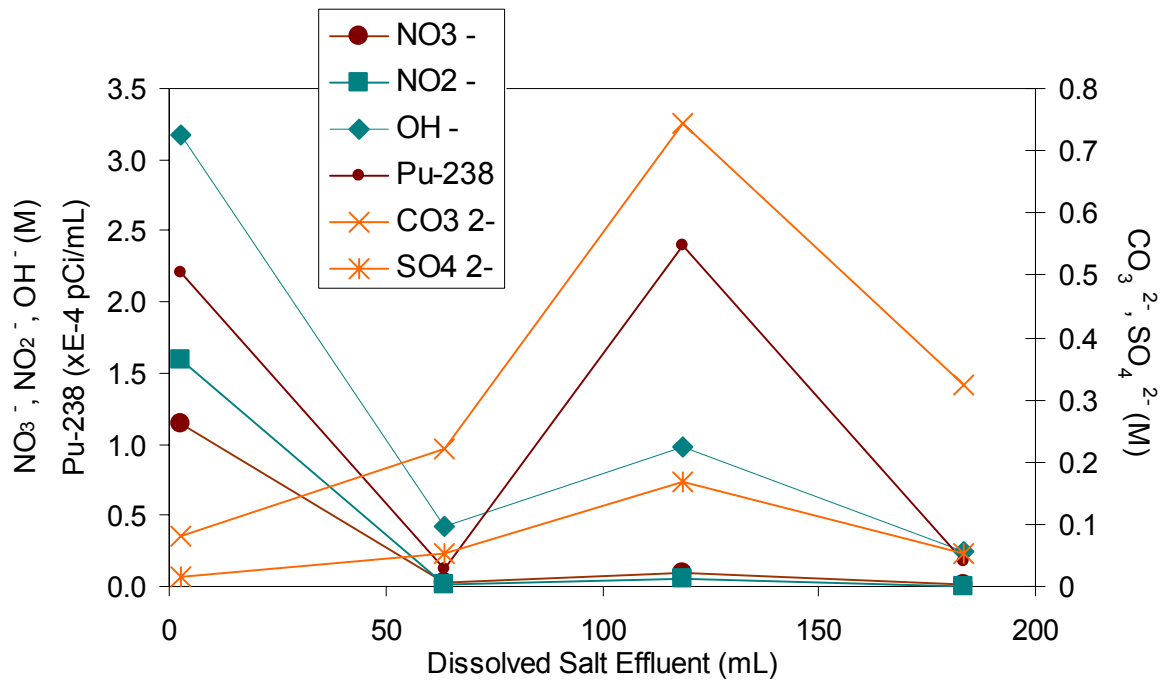
Figure 11: Tank 29H flow-through dissolution profiles for anions and Pu-238
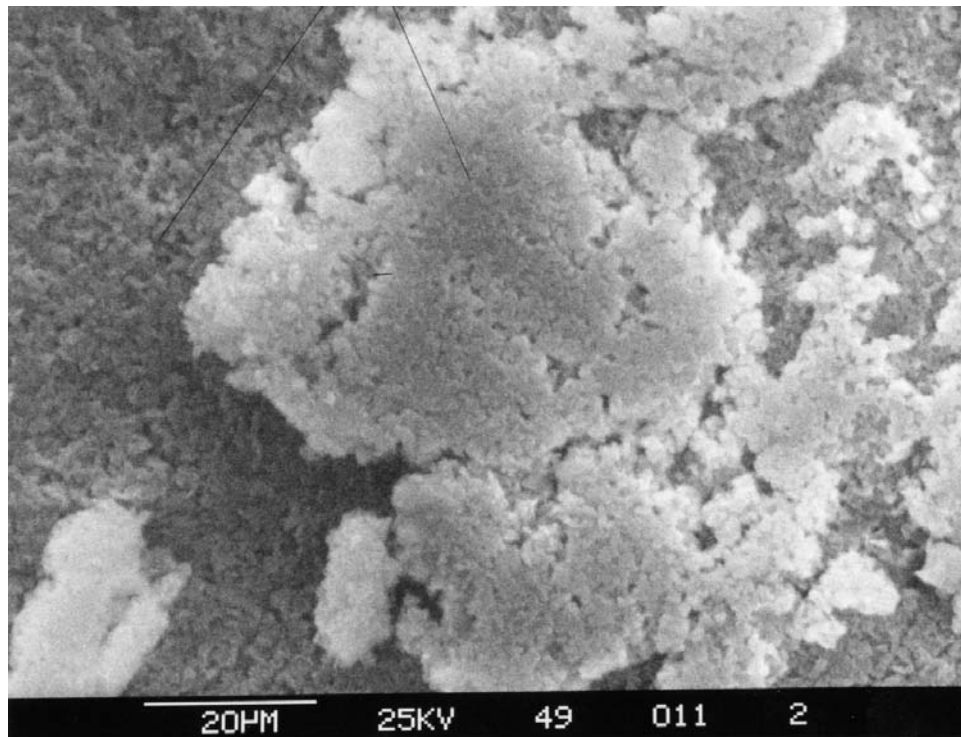
Figure 12: SEM photograph of solids remaining from dissolution tests
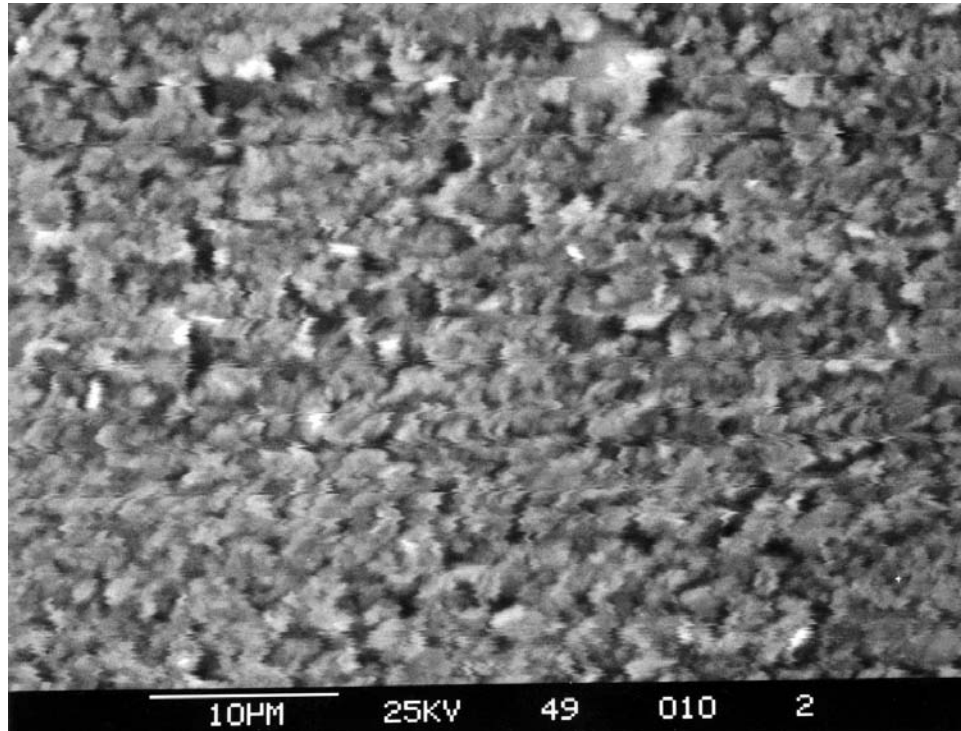
Figure 13: SEM photograph of second area of solids remaining from dissolution tests
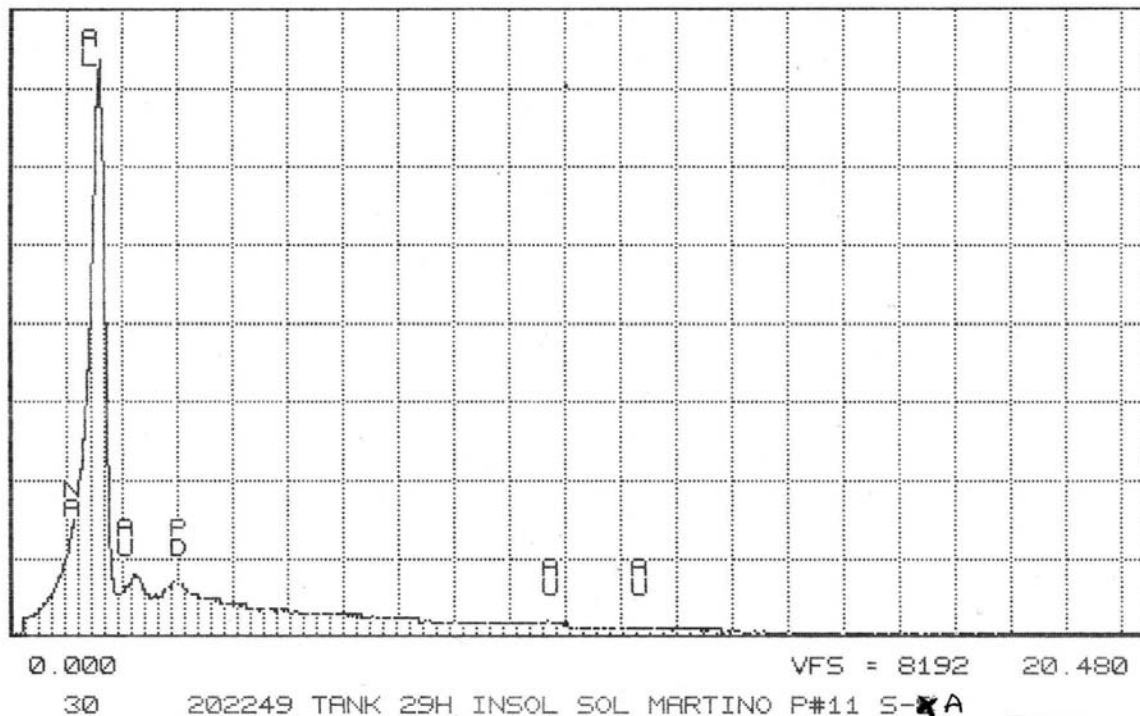
Figure 14: X-ray EDS of solids remaining from dissolution tests

Physical Analyses

The bulk density of the saltcake sample from tank 29H was greater than the bulk density reported for saltcake samples collected previously from Tanks 2F, 3F, and 41H, as shown in Table 10. The increased bulk density is most likely due to a higher aluminum content (13 wt%) in the tank 29H sample when compared to the saltcake in the Tanks 2F, 3F, and 41H (0.1-1 wt%) samples. Porosity and saturation were not estimated for the sample due to a lack of information on the crystal phases of aluminum species in the sample. However, if it is assumed that the sample was saturated, the estimated porosity would be 56% and the density of salt particles would be 2.96 gm/cc.

Saturation of the tank 29H sample with Simulated Interstitial Liquid (SIL) was initiated on November 19, 2003 to prepare the sample for a falling head test to determine the permeability of the sample. Approximately 1 mL of SIL entered the sample after 7 days. The falling head test was subsequently terminated due to the slow saturation process. Several adjustments were made to the falling head apparatus to ensure that the test equipment was not preventing SIL from entering the sample during the saturation step of the procedure. These results suggest that either the sample had a very low permeability or that the SIL was unable to displace the residual IL that was in the sample. Additionally, the sample had likely cooled from the tank temperature of 47 °C to the laboratory temperature of ~25 °C reducing the solubility of some salts in the residual IL and resulting precipitation of salts in existing pores, which ultimately reduced the permeability of the sample.

Interstitial liquid extraction of sample T29H-B6-1-A removed 13.3 grams of IL from 112 gm of saltcake, Figure 13. Based on as-received sampling results this means 28% (vol/vol) of the IL in the sample was removed during IL extraction using 10" Hg vacuum, Figure 14 (17% of the total sample volume).

Table 10. Physical properties of saltcake samples.

Sample ID	Sample Interval (ft)	Bulk Density (g/cm^3)	Water Content θ_{water} (Wt %)	Porosity ϕ (vol %)	Saturation S (vol %)
T29H-B6-1	0-1	2.13	21	nd	nd
T2F-1-1	0-1	2.04	6.2 ± 0.6	23	77
T3F-1-3 (Tank 3)	1-2	2.07	5.1 ± 2.6	19	76
T41H	1-2	1.92[a]	3.2[a]	22	34

Note: θ_{water} reported as average ± 1 Std. Dev.
[a] average of results reported in ref. 4.
nd not determined due to lack of data on salt particles
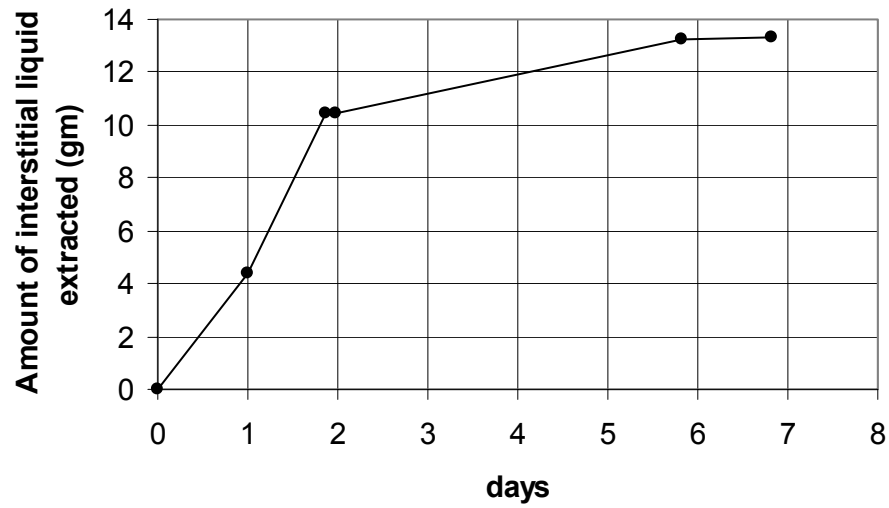
Figure 15: Extraction of interstitial liquid from T29H B6-1-A
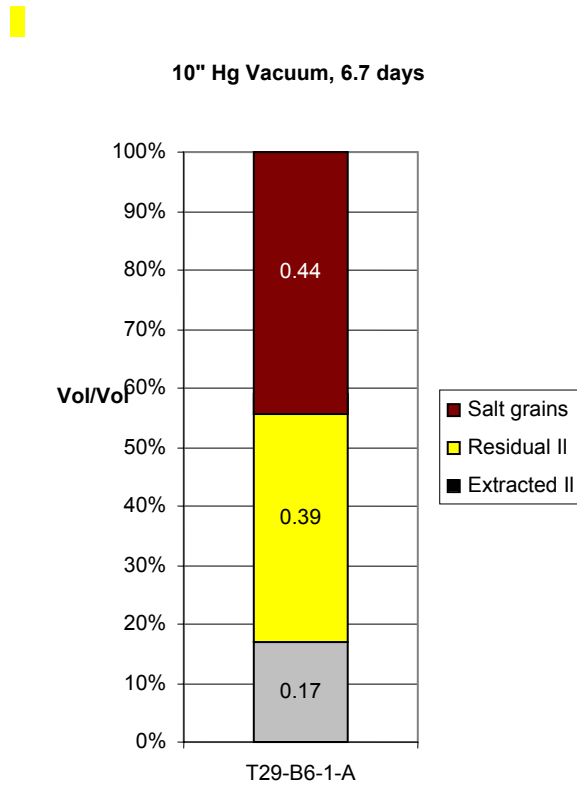
Figure 16: Volume percent of residual and extracted interstitial liquid in T29H-B6-1-A following vacuum extraction

Discussion

As evidenced by data shown in Tables 3 – 5, the supernate dip sample from Tank 29H is substantially different from the saltcake and interstitial liquid. This is not surprising since the last liquids added to the tank were low ionic strength solutions (condensate and rainwater) onto the top of the saltcake. The density of the dip sample is lower than the interstitial liquid, but it does not just appear to be a difference in saturation level. The ratios of nitrate, nitrite, hydroxide, and aluminate are different for the two liquids, with high nitrate in the dip sample, and high nitrite in the interstitial liquid.

Filtering the as-received dip sample removed approximately 72% of the Pu with a 0.45-micron filter. Filtering with a 0.1 micron filter did not further reduce the Pu content, within experimental accuracy. Conversely, less than half of the U is removed by filtration (21-37%). These data suggest that most of the Pu and some of the U is in suspension in the supernate, consistent with the observed (brown) turbidity of the solution (Figure 4). The results for Sr-90 are not explainable. An error in sample handling is suspected.

In the bulk, undrained saltcake and interstitial liquid, almost all (>90%) of the actinides are partitioned to the saltcake phase. Dissolution of this phase, and the partitioning of radionuclides are discussed later in the text.

As expected, the radionuclides C-137 and Tc-99 are entirely soluble in the interstitial liquid within experimental error. This is shown in the last column of Table 3, where the percent of each component that is distributed in the liquid is shown. To indirectly confirm the volume % of the liquid phase in the saltcake sample, the Cs-137 concentration and densities can be used. This indicates that the liquid phase is 54 vol% of the bulk saltcake, in good agreement with the 56 vol% calculated from the quantity of water determined by drying, and the mass of cations and anions in the liquid.   The calculations for DIL that exceed 100% (e.g. phosphate) are not tenable and indicate experimental and analytical variability in these minor species.

The percent volume of liquid in this sample is at the high end of the range of typical saturated saltcakes. This may be related to the high aluminum content of this sample, versus most saltcakes which are overwhelmingly sodium nitrate crystals. The difference may have profound impacts on the draining characteristics of this waste tank, although it is unlikely that this sample represents the average contents of this tank. If this composition is similar throughout the tank, more interstitial liquid may generated upon draining, and more may be retained by the remaining saltcake. Tank-wide modeling studies indicate that the best estimate for saltcake contains around 40% total void space, with 30% liquid and 10% gas.[4] A tank with higher liquid volume would be expected to have different percolation and draining characteristics, although the magnitude of the impact is not known.

Table 4 shows the principal component distribution of the saltcake, interstitial liquid, and supernate dip samples. Table 5 shows the minor elemental compositions, and Table 6 identifies the likely major components of a theoretically completely dry saltcake, shown schematically in Figure 17. The saltcake composition is calculated by excluding the liquid phase components, in proportion to the mass of each and the phase ratio. Compositions of salts, and the number of waters of hydration, are assumed and have not been independently analyzed. As can be seen, the sample is almost entirely sodium aluminate.

Analysis of the bulk saltcake for total base analysis indicated the presence of 14.9 wt% (calculated as hydroxide ion), and 7.71 wt% free hydroxide. The interstitial liquid analysis indicates 5.32 wt % free hydroxide, suggesting insoluble sodium hydroxide. The presence of insoluble sodium hydroxide is unlikely in tank waste, and an alternate explanation is needed. A possible explanation for the observation is that some of the insoluble sodium aluminate disproportionated to sodium hydroxide and aluminum hydroxide during the sample preparation in water. Preparation of the sample for analysis of the bulk undrained saltcake was done by dissolving the sample in water and analyzing for anions. Using the independent analyses of aluminum and carbonate in the bulk waste, more than 95% of the total base in the bulk waste can be accounted for as sodium aluminate. This indicates that the free hydroxide

measurement in bulk saltcake is correct, but the apparent insolubility is an anomaly of the chemistry of the sample preparation with this high aluminum matrix. This likely has implications for the dissolution of this tank, as additional sodium hydroxide would be needed to dissolve the aluminum hydroxide (discussed further below).
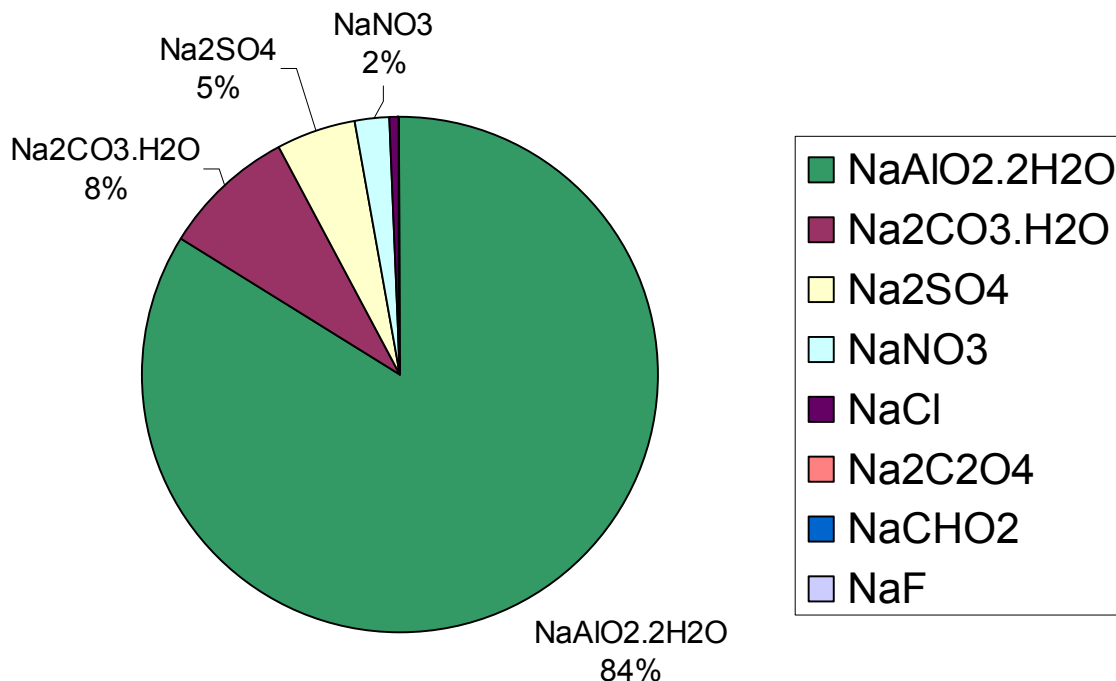
Figure 17: Likely major components of Tank 29H saltcake solids, normalized

The saltcake sample was partially drained of interstitial liquid prior to dissolution testing. Out of a total of 112.2 g (52.7 mL) of undrained saltcake, draining under 10 inches Hg vacuum for 6.8 days, removed 13.3 g (8.99 mL) of interstitial liquid. This left approximately 98.9 g of saltcake and residual interstitial liquid at the start of the dissolution test. The liquid that was removed during vacuum draining was only 17 vol%, versus a calculated 56 vol% liquid present (based on total sample volume). While the amount drained is consistent with tank 41H draining behavior, the residual liquid volume (39 vol%) is not. This calculation can be compared with the sum of cesium from the dissolution aliquots. The total Cs-137 in the residual 39 vol% interstitial should be 5.14E+10 pCi. Summing the Cs-137 in the filtered bulk sample and four subsamples is 3.31E+10 pCi, a 64% recovery. It is possible that the discrepancy is due to differences in the saturation of the dissolution sample versus the analyzed sample, which were from different sections of the tube. However, these results, if representative of the full tank composition, indicate that this saltcake will retain more liquid, and hence more Cs-137 and Tc-99, than other tanks. This behavior is likely related to the unique composition of this waste, which is very low in sodium nitrate and very high in aluminate species. This is could be because the low ionic strength liquids added to the top of the saltcake over the past few years, coupled with fact that this sample was from the bottom of a mined well, dissolved the top layer of sodium nitrate and left sodium aluminate behind by some mechanism. Even if this sample is not representative of the tank contents, it indicates that a wide range of compositions and physical properties that can be present, and current models need to be adjusted to accommodate the variable nature of the tanks. If it is assumed that this is a layer of aluminum above a typical salt tank, this observation also exemplifies the formation of a phase with relatively low solubility and possibly low permeability on top of a highly soluble phase. Settling of an aluminum-rich phase in the bottom of the mined well may also have contributed to the non-representative nature of this sample.

From the estimated composition of the saltcake, the white solids likely formed during the dissolution demonstration from disproportionation of sodium aluminate to aluminum hydroxide and sodium hydroxide. The fine particulate aluminum hydroxide likely built up at the top of the saltcake during dissolution, prohibiting the water from contacting additional salt. This phenomenon was noted by Mississippi State University DIAL during similar dissolution testing of Hanford high-aluminum saltcake simulant.[6] In a tank, this phenomenon may manifest as slow dissolution and percolation rates, depending on the water addition method, impacting tank waste retrieval schedules. Even if this is not representative of the tank, the presence of layers of high aluminate phases would likely cause formation of low permeability layers during subsequent dissolutions. Further study is recommended to examine the quantity and conditions for sodium hydroxide additions needed to avoid precipitation of aluminum hydroxide during tank waste retrieval, and to examine the water addition methods that mitigate slow percolation rates.

Although the second dissolution aliquot has a relatively low sodium molarity, compared to typical salt solutions, it may be saturated with respect to equilibrium with the "atypical saltcake" which is present after the first dissolution step. Since the anion-cation balance is poor, it is not possible to determine if the solution is saturated. Further laboratory testing and computer modeling are needed to determine the degree of saturation of this solution.

The Pu-238 solubility behavior during dissolution is interesting. Between the first and second dissolution, the Pu decreases, trending with hydroxide ion, but increases in the third sample, trending with carbonate ion. This suggests that two Pu species are present or form during dissolution. This observation does not exclude a possible role for other anions also, such as oxalate, that are near the detection limits. It is not proven here that this dissolution solution was saturated to its solubility limit with Pu or U, and further testing would be needed to determine if more of these radionuclides dissolve on prolonged contact of the salt solution with saltcake solids at tank temperatures. Although the Pu was largely removed by filtration, this may not represent the behavior in a tank environment due to the slow kinetics of Pu dissolution in salt solution. Settling may not remove the Pu to the same degree as filtration, as the species may be present as colloids. In this experiment, the dissolution test subsamples were settled, and the liquid withdrawn from above the settled solids with a pipette, indicating that the Pu remains in suspension to some extent. The short contact times and low temperatures used in the experiments will not mimic the conditions in a waste tank. Furthermore, if sodium hydroxide is added to facilitate aluminum dissolution, both the hydroxide content and aluminum content will impact the Pu solubility.[7]

Solubility of the saltcake for this tank sample is relatively low. The total yield of dissolved salt is estimated at 66% of the mass of original saltcake. The sum of aluminum that dissolved in all the dissolution steps was only 27% of the total calculated present in the bulk sample. The volume of aluminum hydroxide solids remaining after dissolution (~40 mL) approaches the volume of the original saltcake sample (52.7 mL). Addition of sodium hydroxide to dissolve the aluminum species would likely increase the yield of retrievable waste. The kinetics of redissolution can also be slow at ambient temperature, depending on the degree of crystallinity, temperature, and particle size.

Conclusions

The supernate dip sample from Tank 29H is substantially different from the saltcake and interstitial liquid. This is thought to be because low ionic strength liquids were added to the top of the saltcake during the past few years, and some of the dissolved salt was removed. Approximately 72% of the Pu and ~25% of the U is in suspension in the supernate and can be removed by filtration.

As expected, the radionuclides C-137 and Tc-99 are entirely soluble and the actinides are largely (>90%) insoluble, in the interstitial liquid, within experimental error.

It is not known how well this sample represents the average contents of the entire tank. Addition of low ionic strength liquid to the top of the saltcake/supernate and well mining operations probably dissolved some species and left behind others. There is considerably more aluminum and liquid present in this sample than in most other tank samples examined thus far. If this high aluminum composition is similar

throughout the tank, more interstitial liquid may be released upon draining, and more will be retained by the remaining saltcake than current computer models predict. The draining and percolation characteristics are also expected to be different, although the magnitude of the impact is not known. If this aluminum phase forms a low permeability layer on top of the saltcake, this may impact the waste dissolution rate, and thus the retrieval schedule. Different water addition methods would likely be impacted in different ways by the presence of a low permeability layer. The high aluminum content of this sample has implications for the dissolution chemistry also, as additional sodium hydroxide would be needed to dissolve the aluminum hydroxide. The high concentration of what appears to be sodium aluminate led to precipitation of aluminum hydroxide during the draining/dissolution test, and only about 66% of the original salt (by mass) partitioned to the aqueous phase during the drain/dissolution demonstration.   The sum of aluminum that dissolved in all the dissolution steps was only 27% of the total calculated present in the bulk sample. The volume of aluminum hydroxide solids remaining after dissolution was 76% of the original saltcake sample volume. Although the Pu was largely removed from the dissolved salt by filtration, this may not represent the behavior in a tank environment due to the slow kinetics of Pu dissolution in salt solution.

Additional characterization of this tank is recommended to determine if this sample is representative of the tank contents. Further study is also recommended to examine the quantity and conditions for sodium hydroxide additions needed to avoid precipitation of aluminum hydroxide, and to examine the kinetics of Pu and U dissolution.

Quality Assurance

This work satisfies the requirements of the original task technical and quality assurance plan.[8] Laboratory Notebooks WSRC-NB-2003-00072, WSRC-NB-2003-00177, WSRC-NB-2003-00199, and various ADS notebooks contain the experimental data.

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Appendix

Table 10: Undrained Bulk Saltcake rad. chem. results (pCi/g)

Sample:	T29H-B6-1	
(pCi/g)	Average	St. Dev.
^{14}C	9.9E+02	--
^{90}Sr	1.35E+07	4.9E+06
^{137}Cs	6.40E+08	2.0E+07
^{238}Pu	1.12E+06	1.0E+05
$^{239/240}Pu$	1.79E+04	1.5E+03
^{241}Am	1.13E+04	2.2E+03

Table 11: Undrained Bulk Saltcake IC Anions, wet chemistry titration, and TIC/TOC results (g/100g)

Sample:	T29H-B6-1	
(g/100g)	Average	St. Dev.
NO_3^-	5.38E+00	7.7E-01
NO_2^-	3.03E+00	1.5E-01
SO_4^{2-}	2.19E+00	4E-02
PO_4^{3-}	7.91E-02	9.0E-03
Cl^-	2.02E-01	2.68E-01
F^-	*< 9.68E-03*	--
$C_2O_4^{2-}$	*< 4.84E-02*	--
CHO_2^-	*< 4.84E-02*	--
Free OH^-	7.71E+00	1.67E+00
CO_3^{2-} (titration)	*< 5.81E+00*	--
CO_3^{2-} (TIC)	3.02E+00	2.1E-01
TOC	3.9E-01	1.4E-01

Table 12: Undrained Bulk Saltcake ICP-ES results (g/100g)

Sample:	T29H-B6-1	
(g/100g)	Average	St. Dev.
Ag	< 6.94E-04	--
Al	1.33E+01	2E-01
B	< 7.50E-02	--
Ba	< 6.94E-04	--
Ca	< 2.10E-02	--
Cd	< 9.25E-04	--
Ce	< 1.15E-02	--
Cr	2.13E-02	9E-04
Cu	< 1.44E-03	--
Fe	1.08E-02	1.2E-03
Gd	< 1.25E-03	--
K	< 4.41E-01	--
La	< 9.25E-04	--
Li	< 3.94E-03	--
Mg	</=3.99E-03	1.62E-03
Mn	</=2.20E-03	1.94E-03
Mo	1.97E-02	2.3E-03
Na	2.13E+01	1E-01
Ni	< 3.47E-03	--
P	4.45E-02	2.4E-03
Pb	< 1.14E-02	--
S	6.67E-01	1.04E-01
Sb	3.67E-02	2.3E-03
Si	2.24E-02	2.9E-03
Sn	1.30E-02	2.0E-03
Sr	< 4.63E-03	--
Ti	< 2.78E-04	--
Zn	< 2.55E-03	--
Zr	< 5.56E-04	--

Table 13: Undrained Bulk Saltcake AA results (g/100g)

Sample:	T29H-B6-1	
(g/100g)	Average	St. Dev.
As	< 9.2E-04	
K	8.06E-02	4.6E-03
Se	< 9.2E-04	
Hg	7.10E-03	4.2E-04

Table 14: Undrained Bulk Saltcake ICP-MS results (g/100g)

Sample:	T29H-B6-1	
(g/100g)	Average	St. Dev.
Mass 59	< 1.9E-05	
Mass 88	3.6E-05	2.5E-05
Mass 99	1.19E-03	4E-05
Mass 101	1.0E-04	3E-07
Mass 133	1.8E-03	3E-05
Mass 135	2.4E-04	7E-06
Mass 137	1.6E-03	5E-05
Mass 138	6.0E-05	1.4E-05
Mass 230	< 2.8E-06	
Mass 231	< 2.8E-06	
Mass 232	5.7E-05	8E-06
Mass 233	2.0E-05	3E-06
Mass 234	3.9E-05	5E-06
Mass 235	3.1E-04	3E-05
Mass 236	1.4E-04	1E-05
Mass 237	1.1E-05	1E-06
Mass 238	1.47E-03	1.5E-04
Mass 239	1.5E-05	7E-07
Mass 240	< 2.8E-06	
Mass 241	< 2.8E-06	
Mass 242	< 2.8E-06	
Mass 243	< 2.8E-06	
Mass 244	< 2.8E-06	

Table 15: Interstitial Liquid rad. chem. results (pCi/mL)

Sample:	T29H-B6-1 IL	
(pCi/mL)	Average	St. Dev.
^{90}Sr	1.68E+06	2E+04
^{137}Cs	2.50E+09	5E+07
^{238}Pu	1.13E+04	3.0E+03
$^{239/240}$Pu	3.15E+03	1.48E+03
^{241}Am	2.69E+03	5E+01

Table 16: Interstitial Liquid IC Anions, wet chemistry titration, and TIC/TOC results (mg/L)

Sample:	T29H-B6-1 IL	
(mg/L)	Average	St. Dev.
NO_3^-	1.14E+05	2E+03
NO_2^-	1.22E+05	5E+02
SO_4^{2-}	6.52E+02	0E+00
PO_4^{3-}	4.38E+03	5E+01
Cl^-	</=1.81E+02	5E+01
F^-	*< 1.45E+02*	
$C_2O_4^{2-}$	*< 7.24E+02*	
CHO_2^-	*< 7.24E+02*	
Free OH^-	7.87E+04	6.1E+03
CO_3^{2-}	*< 8.69E+03*	
CO_3^{2-} (TIC)	1.26E+04	2E+02
TOC	3.77E+03	4.5E+02
Tot. Base (M)	8.62E+00	6.1E-01
OH^- (calculated)	1.27E+05	

Table 17: Interstitial Liquid ICP-ES results (mg/L)

Sample:	T29H-B6-1 IL	
(mg/L)	Average	St. Dev.
Ag	< 1.16E+00	--
Al	2.60E+04	6E+02
B	1.50E+02	0E+00
Ba	3.91E+00	4.3E-01
Ca	< 3.39E+01	--
Cd	< 1.50E+00	--
Ce	< 1.86E+01	--
Cr	4.39E+02	1.1E+01
Cu	3.01E+01	3.6E+00
Fe	1.74E+02	8.1E+01
Gd	< 2.04E+00	--
K	4.37E+03	6E+01
La	</=1.7E+00	2E-01
Li	8.89E+01	4.56E+01
Mg	1.95E+01	9.6E+00
Mn	4.23E+01	1.39E+01
Mo	3.91E+02	9E+00
Na	2.71E+05	1E+03
Ni	1.8E+01	7E+00
P	1.52E+03	1E+01
Pb	< 1.84E+01	--
S	1.09E+03	7E+01
Sb	7.92E+01	2.4E+00
Si	2.62E+02	3.0E+01
Sn	4.13E+01	1.6E+00
Sr	< 7.48E+00	--
Ti	< 4.76E-01	--
V	1.10E+01	5E-01
Zn	6.19E+01	7.7E+00
Zr	1.6E+00	5E-02

Table 18: Interstitial Liquid ICP-MS results (mg/L)

Sample:	T29H-B6-1 IL	
(mg/L)	Average	St. Dev.
Mass 59	5.6E-01	1E-02
Mass 88	< 1.4E-01	--
Mass 99	3.81E+01	3E-01
Mass 101	2.42E+00	3E-02
Mass 133	5.73E+01	9E-01
Mass 135	8.42E+00	9.7E-02
Mass 137	3.01E+01	6E-01
Mass 138	3.8E-01	2E-02
Mass 230	< 5.0E-02	--
Mass 231	< 5.0E-02	--
Mass 232	< 5.0E-02	--
Mass 233	< 5.0E-02	--
Mass 234	< 5.0E-02	--
Mass 235	1.09E-01	3.0E-02
Mass 236	6.1E-02	3E-04
Mass 237	< 5.0E-02	--
Mass 238	4.15E+00	1.1E-01
Mass 239	< 5.0E-02	--
Mass 240	< 5.0E-02	--
Mass 241	< 5.0E-02	--
Mass 242	< 5.0E-02	--
Mass 243	< 5.0E-02	--
Mass 244	< 5.0E-02	--

Table 19: Tank 29H supernate rad. chem. results (pCi/mL)

Tank 29H	As Received		0.45-μm Filtrate		0.1-μm Filtrate	
(pCi/mL)	Average	St. Dev.	Average	St. Dev.	Average	St. Dev.
^{14}C	--		1.20E+03	--	--	
^{90}Sr ^{a}	1.44E+04	3.3E+03	1.59E+05	3.3E+04	1.21E+05	8E+02
^{137}Cs	6.92E+08	0E+00	7.15E+08	8E+06	7.21E+08	8E+06
^{238}Pu	1.32E+05	2.8E+03	3.65E+04	1.4E+03	3.40E+04	2.8E+03
$^{239/240}$Pu	2.66E+03	1E+01	1.10E+03	4E+01	*< 8.31E+02*	--
^{241}Am	*< 6.30E+02*	--	*< 6.84E+02*	--	*< 4.83E+02*	--

Note a: Unexpectedly, the as-received sample had lower amounts of ^{90}Sr than the filtered samples

Table 20: Tank 29H supernate IC anions, wet chemistry titration, and TIC/TOC results (mg/L)

Tank 29H	0.45-μm Filtrate	
(mg/L)	Average	St. Dev.
NO_3^-	1.05E+05	3E+03
NO_2^-	3.24E+04	3E+02
SO_4^{2-}	6.76E+03	1.3E+02
PO_4^{3-}	3.91E+02	4.2E+01
Cl^-	6.01E+01	0E+00
F^-	1.80E+01	0E+00
$C_2O_4^{2-}$	9.01E+01	4.25E+01
CHO_2^-	*< 6.01E+02*	--
Total Base (M)	3.07E+00	4.7E-02
Free OH$^-$	2.56E+04	8.7E+02
CO_3^{2-} (TIC)	2.37E+04	6.4E+02
TOC	3.79E+03	1.3E+02
OH$^-$ (calculated)	4.08E+04	

Table 21: Tank 29H supernate ICP-ES results (mg/L)

Tank 29H	As Received		0.45-μm Filtrate		0.1-μm Filtrate	
(mg/L)	Average	St. Dev.	Average	St. Dev.	Average	St. Dev.
Ag	< 9.26E+00	--	< 9.24E+00	--	< 8.23E+00	--
Al	2.49E+03	0E+00	8.47E+03	6.19E+03	6.44E+03	2.23E+03
B	< 1.00E+03	--	< 9.97E+02	--	< 8.90E+02	--
Ba	< 9.26E+00	--	< 9.24E+00	--	< 8.23E+00	--
Ca	< 2.80E+02	--	< 2.78E+02	--	< 2.48E+02	--
Cd	< 1.23E+01	--	< 1.23E+01	--	< 1.10E+01	--
Ce	< 1.54E+02	--	< 1.52E+02	--	< 1.36E+02	--
Cr	8.13E+01	1.1E+00	1.28E+02	5.7E+01	1.08E+02	1.2E+01
Cu	< 1.92E+01	--	< 1.90E+01	--	< 1.70E+01	--
Fe	< 1.36E+01	--	< 1.35E+01	--	< 1.21E+01	--
Gd	< 1.66E+01	--	< 1.66E+01	--	< 1.49E+01	--
K	< 5.88E+03	--	< 5.86E+03	--	< 5.23E+03	--
La	< 1.23E+01	--	< 1.23E+01	--	< 1.10E+01	--
Li	< 5.24E+01	--	< 5.23E+01	--	< 4.66E+01	--
Mg	< 3.83E+01	--	< 3.82E+01	--	< 3.40E+01	--
Mn	< 1.36E+01	--	< 1.35E+01	--	< 1.21E+01	--
Mo	< 1.26E+02	--	< 1.26E+02	--	1.15E+02	4E+00
Na	1.12E+05	3E+03	1.26E+05	1.3E+04	1.16E+05	4E+03
Ni	< 4.62E+01	--	< 4.61E+01	--	< 4.12E+01	--
P	< 3.00E+02	--	< 2.99E+02	--	</=2.67E+02	2E+00
Pb	< 1.52E+02	--	< 1.51E+02	--	< 1.35E+02	--
S	2.46E+03	2E+01	1.68E+03	8.2E+02	2.41E+03	5.3E+01
Sb	< 9.32E+01	--	< 9.30E+01	--	< 8.29E+01	--
Si	8.64E+02	6.8E+01	8.19E+02	3.6E+01	6.77E+02	7.4E+01
Sn	< 1.51E+02	--	< 1.51E+02	--	< 1.35E+02	--
Sr	< 6.17E+01	--	< 6.16E+01	--	< 5.49E+01	--
Ti	< 3.70E+00	--	< 3.69E+00	--	< 3.29E+00	--
V	< 6.79E+00	--	< 6.77E+00	--	< 6.04E+00	--
Zn	< 3.39E+01	--	< 3.39E+01	--	< 3.02E+01	--
Zr	< 7.41E+00	--	< 7.38E+00	--	< 6.59E+00	--

Table 22: Tank 29H supernate ICP-MS results (mg/L)

Tank 29H	As Received		0.45-μm Filtrate		0.1-μm Filtrate	
(mg/L)	Average	St. Dev.	Average	St. Dev.	Average	St. Dev.
Mass 59	< 2.8E-01	--	< 2.8E-01	--	< 2.5E-01	--
Mass 88	< 2.4E-01	--	< 2.4E-01	--	</=2.4E-01	4E-02
Mass 99	1.04E+01	8E-01	1.08E+01	6E-01	1.03E+01	1E-02
Mass 101	4.3E-01	1E-02	3.8E-01	3E-03	3.3E-01	2E-03
Mass 133	1.67E+01	6E-01	1.69E+01	8E-01	1.61E+01	4E-01
Mass 135	2.39E+00	9E-02	2.46E+00	6E-03	2.35E+00	2E-02
Mass 137	8.95E+00	2E-01	9.04E+00	2.7E-01	8.72E+00	9E-02
Mass 138	< 1.5E-01	--	5.0E-01	1E-02	6.4E-01	6.3E-01
Mass 230	< 8.3E-02	--	< 8.3E-02	--	< 7.4E-02	--
Mass 231	< 8.3E-02	--	< 8.3E-02	--	< 7.4E-02	--
Mass 232	< 8.3E-02	--	< 8.3E-02	--	< 7.4E-02	--
Mass 233	< 8.3E-02	--	< 8.3E-02	--	< 7.4E-02	--
Mass 234	< 8.3E-02	--	< 8.3E-02	--	< 7.4E-02	--
Mass 235	2.31E-01	1.5E-02	1.46E-01	2.2E-02	1.34E-01	9.2E-03
Mass 236	8.85E-02	8.0E-03	< 8.3E-02	--	< 7.4E-02	--
Mass 237	< 8.3E-02	--	< 8.3E-02	--	< 7.4E-02	--
Mass 238	1.68E+00	1.7E-03	1.33E+00	8.8E-03	1.14E+00	2.5E-04
Mass 239	< 8.3E-02	--	< 8.3E-02	--	< 7.4E-02	--
Mass 240	< 8.3E-02	--	< 8.3E-02	--	< 7.4E-02	--
Mass 241	< 8.3E-02	--	< 8.3E-02	--	< 7.4E-02	--
Mass 242	< 8.3E-02	--	< 8.3E-02	--	< 7.4E-02	--
Mass 243	< 8.3E-02	--	< 8.3E-02	--	< 7.4E-02	--
Mass 244	< 8.3E-02	--	< 8.3E-02	--	< 7.4E-02	--

Table 23: Tank 29H supernate AA results (mg/L)

Tank 29H	As Received		0.45-μm Filtrate		0.1-μm Filtrate	
(mg/L)	Average	St. Dev.	Average	St. Dev.	Average	St. Dev.
As	< 3.1E+00	--	< 3.1E+00	--	< 2.7E+00	--
K	9.4E+02	1E+01	9.7E+02	2E+01	9.8E+02	9.6E+01
Se	< 3.1E+00	--	< 3.1E+00	--	< 2.7E+00	--
Hg	7.2E+01	9E+00	4.7E+01	3E-01	4.4E+01	5E-01