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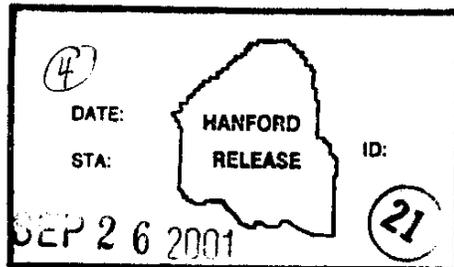
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ACRONYMS AND ABBREVIATIONS

BY-109	single-shell tank 241-BY-109
ChargeBal	charge balance (ratio of cation equivalents to anion equivalents)
CSol	centrifuged solids
EDS	energy dispersive x-ray spectrometer
ESP	Environmental Simulation Program
FY	fiscal year
GEA	gamma energy analysis
IC	ion chromatography
ICP	inductively coupled plasma spectroscopy
kgal	kilogallons (thousand gallons)
MassBal	mass balance
ML	mega-liters (million liters)
mL	milliliters
MSU	Mississippi State University
PLM	polarized light microscope (or microscopy)
RPP	River Protection Project
S-110	single-shell tank 241-S-110
SEM/EDS	scanning electron microscopy with energy dispersive x-ray spectroscopy
TGA	thermogravimetric analysis
TIC	total inorganic carbon
TOC	total organic carbon
U-107	single-shell tank 241-U-107
Vol	volume
Vol%	volume percent
Wt	weight
Wt%	weight percent

1.0 INTRODUCTION AND EXECUTIVE SUMMARY

Laboratory tests were completed on the dissolution characteristics of Hanford saltcake waste from single-shell waste tanks 241-BY-109, 241-S-110, and 241-U-107 (henceforth referred to as BY-109, S-110, and U-107). This work was funded by the Department of Energy's Tanks Focus Area (EM-50) under Technical Task Plan Number RL0-8-WT-41, "PHMC Pretreatment – Saltcake Dissolution". The tests performed were similar in scope to those completed in previous years on waste from tanks BY-102, BY-106, B-106, A-101, S-102, and TX-113 (Herting 1998, 1999, 2000).

The River Protection Project (RPP) is tasked with retrieving waste from double-shell and single-shell tanks to provide feed for vitrification. The RPP organization needs chemical and physical data to evaluate technologies for retrieving the waste. Little relevant laboratory testing has been done to evaluate in-tank dissolution parameters for the various types of saltcake wastes that exist in single-shell tanks.

A computer modeling program known as the Environmental Simulation Program (ESP), produced by OLI Systems, Inc. of Morris Plains, New Jersey, is being used by the RPP organization to predict solubilities during dilution and retrieval of all tank waste types. Data from this task are provided to ESP users to support evaluation, refinement, and validation of the ESP model.

1.1 RESULTS SUMMARY

Cascade (or "stepwise") dissolution tests were performed on composite saltcake samples from all three tanks. The purpose of these tests is to evaluate the solid/liquid phase distribution of chemicals and radionuclides during tank waste retrieval operations. **Feed stability** tests were done on mixtures of "retrieved" waste from all three tanks to evaluate the potential for -precipitation of solids in receiver tanks during tank waste retrieval. **Solid phase identification** tests were performed on saltcake from tank BY-109.

Weight percent (Wt%) dilution is defined in this report as 100 times the weight of diluent (water in all cases for the tests this year) divided by the weight of undiluted saltcake composite sample. A 50% dilution, for example, would be 50 g of H₂O added to 100 g of undiluted composite sample. Volume percent (Vol%) dilution is defined as 100 times the volume of diluent divided by the calculated volume of undiluted sample. Weight percent dilutions are measured directly by weighing the undiluted sample and diluent. Volume percent dilutions are estimated from the known volume of diluent and calculated volume of undiluted sample. Vol% dilutions are emphasized throughout this report because they are more useful to the tank farm process engineers.

1.1.1 Cascade Dissolution Tests

The Cascade dissolution test is a very small scale, batch-wise representation of a continuous in-tank retrieval process. A single saltcake sample is contacted several times with fresh water, removing the equilibrated liquid phase after each contact. Progress of the saltcake dissolution is followed through volume/weight measurements and chemical analysis of the removed liquid samples. See Section 2.4 for a detailed procedure description.

Some of the dissolution data for tanks BY-109, S-110, and U-107 are plotted in Figure 4-1 as Vol% Saltcake Dissolved vs. Vol% Dilution at two temperatures. The Vol% Saltcake Dissolved is a measure of how much saltcake could be removed from a tank by adding water to the saltcake and pumping saturated brine (dissolved saltcake) from the tank.

The data in Figure 4-1 show how much the dissolution can vary from one tank to another. Tank BY-102 saltcake was still dissolving at 600% dilution (six volumes of water per volume of original saltcake). Tank S-110 saltcake had reached nearly terminal dissolution at 200% dilution. (That is, little additional dissolution occurred beyond 200% dilution.) Increasing the temperature of the water/waste mixture from 21 °C to 50 °C provided a slight improvement to the dissolution for tanks BY-109 and S-110, but a dramatic increase in dissolution for tank U-107. The dramatic increase comes with a price, though, in the increased potential for pipe plugging due to phosphate precipitation when the solution cools during transport.

Data are presented in Section 5 that explain the gross dissolution behavior in terms of solubilities of individual components in the waste. Evidence is provided there of the importance of double salts (sodium fluoride sulfate and others) in controlling the equilibrium concentrations of sulfate, phosphate, and fluoride in the dissolved waste solutions. Identification of solid phases in equilibrium with the ions in solution is important for computer modeling of the thermodynamics of the system.

Computer simulations of the Cascade dissolution tests on tanks BY-109, S-110 and U-107 were conducted by investigators at Mississippi State University using ESP (to be published). For the major analytes, major trends in the experimental data were also seen in the simulation results. However, discrepancies between experimental data and simulation predictions were evident for those species involved in double salt formation (phosphate, fluoride, sulfate) at low levels of dilution. In particular, for tank BY-109, the ESP simulation predictions indicated an overestimation of phosphate solubility for the first dilution in the cascade and an underestimation of phosphate solubility for the second dilution in the cascade. ESP predictions indicated that the sodium fluoride phosphate double salt will dissolve, followed by the sodium fluoride sulfate double salt, then finally, the remaining sodium fluoride will dissolve. The ESP results indicated a need to qualitatively determine the solid phase speciation during the cascade dissolutions.

Information gathered from the solid phase speciation will be used to improve the ESP prediction of these fluoride-containing species.

1.1.2 Feed Stability Tests

In-tank dissolution and subsequent retrieval of saltcake is envisioned, in one scenario, to occur by sprinkling water or inhibited water (0.01 M NaOH, 0.01 M NaNO₂) over the saltcake, allowing the water to course through the salt, and retrieving the salt-saturated solution from the tank bottom. The composition of the retrieved solution will vary with time in such a scenario. Components like sodium fluoride sulfate and sodium fluoride phosphate will not dissolve to any appreciable extent until the sodium nitrate and other more soluble components are removed from the system, because the solubilities of the former are very dependent on total sodium ion concentration (common ion effect).

The question arises, then, what would happen if the retrieved solution is pumped into a staging tank where the liquid removed from the source tank late in the retrieval process (which is high in sulfate, phosphate, etc.) is allowed to mix with the early-retrieved solution (which is high in nitrate and has a high ionic strength) from the same or a different tank? The Feed Stability Tests were designed to evaluate whether the late-retrieved fractions would re-precipitate when mixed with the early-retrieved fractions.

The tests were carried out using composite saltcake samples from tanks BY-109, S-110 and U-107. The **Cascade Dissolution** method was used, with a total of five contacts for each saltcake, just as described in the previous section. This time, instead of analyzing the liquid phase from each contact, the liquid phase was saved in a collection vial. When all of the contacts were complete, the following combinations were prepared in 15 mL centrifuge cones labeled Cone A – Cone F by mixing equal volumes of clear solution from the collection vials indicated (major anions shown in parentheses):

- Cone A: BY-109 Contact 4 (F⁻, SO₄²⁻) + S-110 Contact 1 (NO₃⁻)
- Cone B: BY-109 Contact 4 (F⁻, SO₄²⁻) + U-107 Contact 1 (NO₃⁻)
- Cone C: S-110 Contact 4 (C₂O₄²⁻) + U-107 Contact 1 (NO₃⁻)
- Cone D: U-107 Contact 3 (PO₄³⁻) + S-110 Contact 1 (NO₃⁻)
- Cone E: U-107 Contact 3 (PO₄³⁻) + BY-109 Contact 5 (F⁻, SO₄²⁻)
- Cone F: U-107 C4 (PO₄³⁻) + BY-109 C5 (F⁻, SO₄²⁻) + S-110 C1 (NO₃⁻)

The results of the test found solids precipitating in all six cones, ranging from a few volume percent settled solids up to complete gelling of the mixture (Cone D). In all cases, the precipitated solid phases were identified by microscopy. See Section 3.2 for detailed results.

Tank farm operators need to be aware of the potential for solids formation in receiver tanks during saltcake retrieval operations.

1.1.3 Solid Phase Identification

Accurate chemical modeling of the tank waste systems (e.g., with ESP) depends in part on proper identification of the solid phases in equilibrium with the ions in solution. Analysis of solids from tank BY-109 were performed this fiscal year using the three-pronged approach of polarized light microscopy (PLM), scanning electron microscopy with energy dispersive x-ray spectroscopy (SEM/EDS), and x-ray diffraction (XRD).

Most of the major solid phases were identified by all three techniques. They included Na_3FSO_4 , $\text{Na}_7\text{F}(\text{PO}_4)_2 \cdot 19\text{H}_2\text{O}$, $\text{Na}_2\text{C}_2\text{O}_4$, and $\text{Na}_2\text{CO}_3 \cdot \text{H}_2\text{O}$. Sodium fluoride, NaF, was identified primarily by XRD as another major component. Aluminum hydroxide was suspected by PLM, confirmed by SEM/EDS, and identified by XRD as the specific crystalline form of $\text{Al}(\text{OH})_3$ known as bayerite. Some undefined aluminosilicate phase was also observed by SEM/EDS. There were also some surprises. Cryolite, Na_3AlF_6 , was identified by SEM/EDS and confirmed by XRD. This phase has not been observed before in tank waste, based on a recent review document (Rapko 2000). Other undefined but surprising phases observed by SEM/EDS included an apparent calcium strontium carbonate and discrete uranium-rich particles.

1.2 COMPOSITION OF TANK WASTES

All of the tests described in this report were performed on composite saltcake samples made from archived core segment samples. One composite sample was prepared for each tank, and tests performed on aliquots taken from the composite sample. Tables 5-1 and 5-2 show the overall composition of the composite sample from each tank. The total sodium content of all three tanks was similar, but there were major differences in the anions associated with the sodium cations.

1.2.1 Tank BY-109

Two core samples were taken from Tank BY-109 in 1997. The two cores (core 201 and core 203) were very similar to one another, but showed stark vertical layering, both in physical appearance and in chemical composition. The composite sample was created by mixing segment samples from both cores. Those segments that could be identified from their chemical analyses (DOE 2001) as composed primarily of saltcake were selected for the composite. Those that contained primarily sludge were not used.

The composite sample was brown, fluid enough to pour though rather cohesive, and contained a trace of free-standing liquid when allowed to settle for prolonged periods. The notable features of the chemical make-up of the composite sample (Table 5-1) are the very high fluoride and sulfate levels and low nitrate level.

1.2.2 Tank S-110

Two core samples (cores 240 and 241) were taken from Tank S-110 in 1998. A partial core sample (core 140) was taken in 1996. Physical appearance and chemical analyses of the individual segments (DOE 2001) showed wide variations, both between segments within a core and between cores. The composite sample was created by mixing segment samples from all three cores. Those segments that could be identified from their chemical analyses as composed primarily of saltcake were selected for the composite. Those that contained primarily sludge were not used.

The saltcake segment samples varied in appearance from greenish-white to nearly black. The composite sample was very dark brown, was fluid enough to pour while the jar was being vibrated, was fairly cohesive (similar to BY-109), and contained no free-standing liquid when allowed to settle for prolonged periods. The notable features of the chemical make-up of the composite sample (Table 5-1) are the very high nitrate level, relatively high carbonate and Cr, and low levels of most other analytes.

1.2.3 Tank U-107

Seven core samples have been taken from Tank U-107 (cores 129, 134, 135, 242, 242R, 245, 245R). The cores were essentially similar to one another, but showed dramatic vertical layering. Individual segments ranged in appearance from bright white to shiny black. The composite sample was created by mixing segment samples from the three cores that still had appropriate archived saltcake segments available, namely cores 135, 242R, and 245. Those segments that could be identified from their chemical analyses (DOE 2001) as composed primarily of saltcake were selected for the composite. Those that contained primarily sludge were not used.

The composite sample was almost black, barely fluid enough to pour while the sample jar was being vibrated, was somewhat cohesive (less than S-110 or BY-109), and contained no free-standing liquid when allowed to settle for prolonged periods. The notable features of the chemical make-up of the composite sample (Table 5-1) are the very high nitrate and phosphate levels with almost no fluoride.

2.0 DESCRIPTION OF LABORATORY STUDIES

Composite samples of tank waste were prepared in a hotcell. Smaller samples were handled in fume hoods. The procedures, data, and observations are recorded in controlled laboratory notebook HNF-N-70-2. The procedures are described in this section, and the raw data are shown in the following section (3.0).

All dissolution tests were done on composite samples made by combining representative core segment samples from each tank. Details of the **composite preparation** are given in Section 2.1.

In prior years, two types of dissolution tests were done. The **sequential dissolution tests** were done to establish an overall saltcake waste composition for each tank, as well as to define basic dissolution behavior. The **series dissolution tests** were done to establish dissolution characteristics of individual salts as a function of the amount of diluent added. This year, **cascade dissolution tests** (previously called stepwise dissolution tests) were done exclusively. The cascade tests are really an extension of the sequential tests. They incorporate the merits of the series tests, and are more directly related to in-tank behavior than the previous tests. Details of all three test procedures are provided in Sections 2.2 – 2.4.

Feed stability tests were done on saltcake from all three tanks. These tests were meant to evaluate whether solids might form during tank waste retrieval when late-retrieved liquid from one tank was mixed with early-retrieved liquid from another tank. These tests are described in section 2.5.

Solid phase identification tests were performed on saltcake from tank BY-109. Information about solid phase speciation is needed to improve the input data to ESP. These tests are described in section 2.6.

A “cold” (nonradioactive) study was done to evaluate methods for measuring the **rate of saltcake dissolution**. These tests, which are important in understanding the kinetic, non-equilibrium aspects of saltcake dissolution, are described in section 2.7.

2.1 PREPARATION OF COMPOSITE SAMPLES

The saltcake composite samples were made by combining archived fractions of individual core segment samples into a single composite jar for each tank. The waste in the completed composite jar was mixed thoroughly before taking subsamples for the individual tests. Weights of individual core segment samples used for each composite sample are shown in Tables 2-1 through 2-3. One aliquot of each composite sample was analyzed for percent water by thermogravimetric analysis (TGA) and total inorganic carbon/total organic carbon (TIC/TOC). A second aliquot was analyzed for anions by ion chromatography (IC) and for metals by inductively coupled plasma spectroscopy (ICP)

following a water-digest of the sample. A third aliquot was analyzed for radionuclides (by various methods) and for metals by ICP following a potassium hydroxide fusion digest. Results of these analyses are shown in Tables 5-1 and 5-2.

Table 2-1. Segment Samples Used to Create BY-109 Core Composite Sample

Core	Segment ^a	Jar #	Weight, g
201	2 U	13030	25.1
201	2 L	13029	25.1
201	3R1 U	13035	25.0
201	3R1 L	13034	25.0
203	3 U	13039	25.1
203	3 L	13040	25.5
203	4 U	13042	25.0
203	4 L	13041	25.0
201	5 U	12726	25.1
	Composite	Total Wt =	225.9

^a Segment 1 is at top of waste; U = upper half of segment; L = lower half of segment

Table 2-2. Segment Samples Used to Create S-110 Core Composite Sample

Core	Segment ^a	Jar #	Weight, g
140	1	9903	25.1
140	2	9905	25.0
140	3	9907	25.0
240	3	14600	25.3
241	3	14973	25.0
140	4B	9809	25.4
241	4	14976	25.0
240	6	14624	51.4
240	7	14625	25.0
	Composite	Total Wt =	252.2

Table 2-3. Segment Samples Used to Create U-107 Core Composite Sample

Core	Segment ^a	Jar #	Weight, g
245	1 L	14975	24.8
135	2R U	9708	24.8
135	2R U	9709	24.9
245	3 U	14957	24.9
245	3 L	14958	24.9
245	4 U	14955	24.9
245	4 L	14956	25.0
245	6 L	14954	50.4
242R	7 L	14733	24.8
242R	7 U	14734	24.7
	Composite	Total Wt =	274.1

2.2 SEQUENTIAL DISSOLUTION TESTS

Sequential dissolution tests were done in prior years (FY 1998-2000) to establish the overall composition of each saltcake composite sample. To describe the test briefly, 30 g of tank composite sample was transferred into a 50 mL centrifuge cone, and an equal weight of water was added. After mixing for a day or more (tumbling end-over-end), the cone was centrifuged, and the supernatant liquid was analyzed.

The weight and volume of centrifuged solids were recorded, and then 30 g of water was added to the solids. The cone was mixed again, centrifuged, and the supernatant liquid analyzed for the same components as the first wash solution. The volume and weight of undissolved centrifuged solids were recorded, and the solids were analyzed. The analytical results from the first wash, second wash, and undissolved solids were combined to determine the overall composition of the composite sample. This overall composition was used as input to the ESP modeling done at Mississippi State University (MSU).

In the FY 2001 tests, the overall composite sample composition was determined through direct analysis of the solid saltcake composite sample, as described in Section 2.1. The results, presented in Tables 5-1 and 5-2, were used as input to the ESP modeling done at MSU.

2.3 SERIES DISSOLUTION TESTS

Series dissolution tests were performed in prior years to establish the dissolution behaviors of the individual saltcake components. To briefly describe this test, a small aliquot (four to six grams) of composite sample was placed into each of six 15 mL centrifuge cones. An amount of water was added to each cone representing 50% to 300% by weight of the composite sample in the cone. Each cone was mixed to establish equilibrium, then centrifuged. The liquid phase from each cone was analyzed.

In FY 2001, the series dissolution tests were replaced by the cascade dissolution tests, which should more accurately reflect the dissolution profile for each saltcake component during the retrieval process.

2.4 CASCADE DISSOLUTION TESTS

Cascade dissolution tests, which have also been called **stepwise** dissolution tests (Herting 1999, 2000), employ a single saltcake sample that is contacted multiple times with water. The process is similar to the sequential dissolution tests, but with more contacts and smaller contact volumes. The cascade test is a very small scale, batch-wise representation of a continuous in-tank retrieval process. The tests were done once at ambient hotcell temperature (21 °C) and again at 50 °C.

For each tank, a 40-gram aliquot of saltcake composite sample was placed into a tared 50-mL graduated centrifuge cone. The cone plus saltcake was weighed, then 20 g of water was added, and the cone was re-weighed. Then the cone was vortex-mixed, then placed on a tumbler to tumble end-over-end for at least 20 hours. After the mixing/equilibration period, each cone was centrifuged for 30 minutes. Total volume and centrifuged solids volume were recorded, and the liquid was decanted into a sample vial for analysis. (For the 50 °C tests, the liquid samples had to be diluted with water to prevent precipitation of salts when the samples cooled to room temperature.) The cone with residual solids was weighed to determine the weight of centrifuged solids and (by difference) the weight of solution decanted. Then a fresh 20 g portion of water was added and the cycle of mixing/centrifuging/decanting was repeated for a total of five water contacts (fewer in some cases at 50 °C).

The liquid samples were analyzed by ICP, IC, TIC/TOC, OH⁻, %H₂O by oven drying at 120°C, density, and radionuclide analyses. Radionuclides were analyzed by GEA (⁶⁰Co, ¹²⁵Sb, ¹³⁷Cs, ¹⁵⁴Eu, ¹⁵⁵Eu, and ²⁴¹Am), and extraction (^{89/90}Sr and ⁹⁹Tc).

Residual solids were treated differently for the 21 °C and 50 °C tests. For the 21 °C tests, the solids were treated with acid (25% by weight HNO₃, 25% HCl, 50% H₂O, with a density of 1.162 g/mL) in the same cone used for the water contacts. The mixed slurry was heated to 60 °C for at least two hours, then cooled and centrifuged. The liquid phase was submitted for analysis, and the weight of acid-insoluble solids was recorded (see Table 3-1).

For the 50 °C tests, one aliquot of the residual solids (following the final water contact) was analyzed for TIC/TOC and %H₂O by TGA. Fusion digest preparations of a second aliquot were analyzed for metals by ICP and radionuclides by the same methods listed for the liquid samples. No IC analyses were performed on residual solids because any reasonable digestion method (acid or fusion) following what was essentially an extensive water digest would have destroyed or obscured the anion analytes of interest.

2.5 FEED STABILITY TESTS

The objective of this test series was to determine if salt precipitation occurs by mixing clear tank waste solutions “retrieved” from different tanks. Dissolution of a composite sample from each tank was performed using the **Cascade dissolution** method just described.

For each tank, a 40-gram aliquot of saltcake composite sample was placed into a tared 50-mL graduated centrifuge cone. The cone plus saltcake was weighed, then 20 g of water was added, and the cone was re-weighed. Then the cone was mixed and tumbled end-over-end for at least 20 hours, then centrifuged. Total volume and centrifuged solids volume were recorded, and the liquid was decanted into a collection vial labeled with a tank identifier and contact number (e.g. ‘BY9C1’ for tank BY-109 Contact 1). The cone with residual solids was weighed to determine the weight of centrifuged solids and (by difference) the weight of solution decanted. The same number of contacts and contact volumes were used as for the Cascade test, so the composition of liquid in each Feed Stability collection vial should be approximately equal to the corresponding sample vials from the Cascade test.

At the conclusion of the five contacts for all three of the tank composite samples, mixtures were prepared representing tanks that might potentially be receiving waste from an active on-going retrieval process. These mixtures were prepared by pipetting equal volumes of clear solution from selected collection vials into 15 mL graduated centrifuge cones. For the specific mixtures that were prepared, and the resulting observations, see section 3.4.

2.6 SOLID PHASE CHARACTERIZATION TESTS

The three-pronged approach of using polarized light microscopy (PLM), scanning electron microscopy with energy dispersive x-ray spectroscopy (SEM/EDS), and x-ray diffraction (XRD) was used to identify solid phases in BY-109 saltcake. The specific instruments used were:

PLM – Leitz Laborlux[®] 12 polarized light microscope with Colorview 12 digital color camera.

SEM/EDS – Aspex[®] Personal Scanning Electron Microscope, Model II, with Noran Light Element EDS detector.

XRD – Rigaku[®] MiniFlex x-ray diffractometer.

In the hotcell, approximately two grams of the BY-109 composite sample was transferred to a sample vial labeled “**BY9 Orig**” to be analyzed by PLM, SEM/EDS, and XRD. A much larger aliquot of the composite sample (43.1 g) was transferred to a 50 mL centrifuge cone.

Water was added to the cone containing the BY-109 composite sample to raise the total volume to 45 mL. The slurry was mixed intermittently (vortex mixer) for at least one hour at 21 °C. Then, before the slurry could settle, a small sample of the slurry (~4.5 mL, or 10% of the total slurry volume) was transferred to a sample vial labeled “**BY9Dil1**” for PLM, SEM/EDS, and XRD. The remaining slurry was centrifuged, the liquid was decanted into a collection jar, and the solids were weighed. Then water was added to the centrifuge cone to raise the total slurry volume again to 45 mL. The process – add water/mix/sample the slurry/centrifuge/decant/weigh solids – was repeated until a total of six slurry samples had been taken (**BY9Dil1** – **BY9Dil6**). The residual solids after the exhaustive dissolution were collected for analysis and labeled “**BY9Resi**”.

Next, the decanted liquid from the collection jar was evaporated slowly. Each time the volume of precipitated solids reached ~20% of the total volume, the solids were collected, either by allowing the slurry to settle or by filtering the slurry, before continuing the evaporation. These samples were labeled **BY9Evap2** – **BY9Evap6**. Sample **BY9Evap1** had been reserved for solids that formed in the collection jar before any evaporation was started, but no such solids formed.

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2.7 DISSOLUTION KINETICS TESTS

If the retrieval water added to the saltcake in the single shell tanks percolates through the salt bed, solid/liquid equilibrium is virtually assured. If the water tends to run quickly across the surface of the saltcake, or runs through channels in the salt bed, it may not be in physical contact with the salt long enough to establish equilibrium. Some type of yet-to-be-established kinetics tests are needed to establish how much solid/liquid contact is required to saturate the liquid phase (i.e., reach equilibrium). Some scouting tests were performed with reagent sodium nitrate salt to evaluate some different techniques for performing these kinetics tests. A method for rapid analysis of samples by measuring the refractive index of the solution proved to be adequate for establishing the nitrate concentration in the solution.

Technique #1: A beaker of water was stirred rapidly while solid NaNO_3 was added. Liquid phase samples were taken as a function of time by drawing up liquid through a syringe filter. The technique demonstrated that the salt dissolved more quickly than the samples could be drawn. The salt dissolved to the saturation point within seconds of the time the salt was added to the rapidly-stirred water.

Technique #2: This was a modification of Technique #1 in which the stirring rate was very slow. This technique showed the development of very strong layering in the liquid phase, being salt-saturated near the bottom of the beaker and pure water at the top. Despite modifications to the stirrer and the sampling process, representative samples could not be taken.

Technique #3: Solid NaNO_3 was placed into the upper chamber of a vacuum filter apparatus, followed by a volume of water. After allowing 'x' seconds for dissolution to occur, without stirring or mixing, the vacuum was turned on to separate the phases. This technique shows the most promise of those tested so far.

3.0 RESULTS – RAW DATA

“Raw data” include values of net weight, volume, and chemical and physical analyses of the samples. Visual observations of the test materials are also presented in this section. The raw data provide a reference record of the tests. However, sample-to-sample comparisons of raw data generally should not be made because of variations in initial sample weights and volumes. Comparisons based on normalized data, accounting for initial volume and weight differences, are presented in later sections of the report.

3.1 CASCADE DISSOLUTION TESTS

The procedure for the Cascade dissolution tests was described in Section 2.4. Weights, volumes, and analytical sample identification numbers of the various fractions for the ambient temperature tests are shown in Table 3-1, with sample analytical results shown in Tables 3-3 through 3-8. Corresponding data for the 50 °C tests are shown in Tables 3-2 and 3-9 through 3-14, respectively.

Mass balance and charge balance calculations can be done on the supernatant liquid fractions, because all of the known components of the liquid were analyzed. The formulas used for these calculations are:

$$\text{MassBal} = \%H_2O + (\sum_i C_i + 3C_{\text{TOC}} + 5C_{\text{TIC}} + 2.2C_{\text{Al}})/(10,000 \times \text{Density})$$

where the C_i values are concentrations expressed in $\mu\text{g/mL}$ as shown in Table 3-3 and subsequent tables; Density is in g/mL ; and $i = \text{OH}^-$, Cr, K, Na, F⁻, Cl⁻, NO₂⁻, NO₃⁻, PO₄³⁻, SO₄²⁻. The TOC concentration is multiplied by 3 to account for the mass of oxygen, nitrogen, and hydrogen associated with the carbon in typical organic complexants. This is an approximate factor. For the centrifuged solids samples in which virtually all of the TOC comes from oxalate, the oxalate concentration is used instead of the TOC in the mass balance calculation. (The same is true for the liquid samples in which oxalate can be shown to represent all or nearly all of the TOC.) The TIC concentration is multiplied by 5 and the Al concentration is multiplied by 2.2 to account for the mass of oxygen in carbonate and aluminate (AlO₂⁻), respectively. These are known factors. The aluminate is used as the anhydrous AlO₂⁻ rather than the hydrated Al(OH)₄⁻ because the hydration water is included in the %H₂O factor.

$$\text{ChargeBal (cation/anion)} = \sum_+(C_+/W_+)/\sum_-(C_-Z_-/W_-)$$

where the C_+ values are the concentrations of Na and K in $\mu\text{g/mL}$; W_+ are the respective atomic weights; C_- are the concentrations of anionic species (OH⁻, TOC, TIC, Al, F⁻, Cl⁻, NO₂⁻, NO₃⁻, PO₄³⁻, SO₄²⁻); Z_- are the charges on the respective anions; and W_- are the formula weights of the anions. The Z_- for TOC is taken to be 0.5, assuming a typical mixture of organic compounds in solution that have an average of two carbon atoms per negative charge (e.g., acetate). The Z_- for TIC is 2. Aluminum is assumed to be present in the liquid phase as aluminate ion, Al(OH)₄⁻, but 27 is used as the formula weight because the concentration is expressed in the tables as Al, not Al(OH)₄⁻.

Table 3-1. Raw Data, Cascade Dissolution Test at 21 °C.
(weights in grams, volumes in mL)

	BY-109	S-110	U-107
Composite Sample Wt, g	40.496	41.022	41.644
Wt H ₂ O Added (1 st wash)	21.905	20.474	21.167
Total Vol, mL	43.0	41.0	43.0
1 st Water Wash Sample ID ^a	BY9C1	S110C1	U107C1
CSol ^b Vol	19.0	10.0	15.0
CSol Wt	31.903	17.858	23.111
Wt H ₂ O Added (2 nd wash)	19.517	20.025	19.621
Total Vol, mL	38.0	29.5	34.0
2 nd Water Wash Sample ID ^a	BY9C2	S110C2	U107C2
CSol ^b Vol	16.0	5.0	11.0
CSol Wt	26.040	7.827	16.518
Wt H ₂ O Added (3 rd wash)	17.795	19.373	19.161
Total Vol, mL	33.0	25.0	30.0
3 rd Water Wash Sample ID ^a	BY9C3	S110C3	U107C3
CSol ^b Vol	13.5	4.0 ^c	7.5
CSol Wt	21.744	5.645	10.574
Wt H ₂ O Added (4 th wash)	20.096	19.492	20.000
Total Vol, mL	33.5	23.5	27.0
4 th Water Wash Sample ID ^a	BY9C4	S110C4	U107C4
CSol ^b Vol	11.0	3.0 ^c	3.5 ^c
CSol Wt	17.213	4.258	5.152
Wt H ₂ O Added (5 th wash)	19.835	19.267	20.442
Total Vol	30.0	22.5	23.5
5 th Water Wash Sample ID	BY9C5	S110C5	U107C5
CSol Vol ^c	7.5	3.0 ^c	3.0 ^c
CSol Wt	12.581	4.275	3.596
Vol Acid-dissolved CSol	34.5	26.5	21.5
CSol Sample ID	BY9ACID	S110ACID	U107ACID

^a decanted supernatant liquid customer ID number

^b CSol = centrifuged solids

^c volumes below 5 mL are estimates only.

Table 3-2. Raw Data, Cascade Dissolution Test at 50 °C.
(weights in grams, volumes in mL)

	BY-109	S-110	U-107
Composite Sample Wt, g	42.192	42.528	42.833
Wt H ₂ O Added (1 st wash)	19.441	18.590	20.031
Total Vol, mL	41.5	42.0	43.5
1 st Water Wash Sample ID ^a	50BY9C1 / 103	50S10C1 / 104	50U07C1 / 105
CSol Vol ^b	17.5	7.5	13.5
CSol Wt	31.635	12.885	20.151
Wt H ₂ O Added (2 nd wash)	17.066	19.901	19.751
Total Vol, mL	35.0	27.5	32.0
2 nd Water Wash Sample ID ^a	50BY9C2 / 106	50S10C2 / 107	50U07C2 / 108
CSol Vol	15.0	4.5 ^c	3.0 ^c
CSol Wt	26.037	6.221	3.675
Wt H ₂ O Added (3 rd wash)	18.359	18.516	22.124
Total Vol, mL	33.0	22.5	24.0
3 rd Water Wash Sample ID ^a	50BY9C3 / 109	50S10C3 / 110	50U07C3 / 111
CSol Vol	12.5	3.5	2.0
CSol Wt	20.421	4.638	2.900
Wt H ₂ O Added (4 th wash)	28.684	30.316	U-107 Stopped After 3 rd Wash
Total Vol, mL	40.0	33.0	
4 th Water Wash Sample ID ^a	50BY9C4 / 112	50S10C4 / 113	
CSol Vol	8.0	3.0	
CSol Wt	13.654	4.291	
Wt H ₂ O Added (5 th wash)	36.991	S-110 Stopped After 4 th Wash	
Total Vol	43.5		
5 th Water Wash Sample ID	50BY9C5 / 115		
CSol Vol	5.0		
CSol Wt	6.203		

^a decanted supernatant liquid; customer ID number / Labcore ID number S01R000...

^b CSol = centrifuged solids

^c volumes below 5 mL are estimates only.

Table 3-3. Chemical Analyses, Tank BY-109 Cascade Dissolution Test, 21 °C.
(Chemical analytes in g/L except as noted)

Analyte	H ₂ O Contact					Acid-Dissolved Solids
	1	2	3	4	5	
Vol% Diln	104	196	280	375	469	--
Sample ID	BY9C1	BY9C2	BY9C3	BY9C4	BY9C5	BY9ACID
S01R000...	231	235	239	243	247	251
Density (g/mL)	1.238	1.127	1.095	1.095	1.093	--
Al	15.5	5.98	1.98	0.553	0.134	0.329
Cr	0.693	0.287	0.110	0.042	0.022	1.40
Fe	< 0.02	< 0.01	< 0.01	< 0.01	< 0.01	0.87
K	2.45	0.99	0.347	0.106	< 0.10	0.24
Na	120	68.8	51.1	47.0	49.3	55.3
P	0.476	0.796	1.02	1.24	1.33	7.39
S	1.97	8.23	13.6	17.4	18.5	9.92
Si	0.229	0.428	0.409	0.129	0.063	0.133
F	4.17	11.1	14.2	16.0	17.3	--
Cl	1.88	0.787	0.328	< 0.17	< 0.17	--
NO ₂ ⁻	31.8	12.4	3.35	1.14	< 1.10	--
NO ₃	132	50.8	14.1	3.80	< 1.42	--
PO ₄ ³⁻	1.29	2.66	2.98	3.71	3.88	--
SO ₄ ²⁻	4.84	25.1	36.5	46.4	50.0	--
C ₂ O ₄ ²⁻		2.46	3.58	4.35	4.76	--
TIC	3.58	1.44	0.578	0.221	0.116	--
TOC	1.14	1.03	1.01	1.21	1.35	--
OH ⁻	18.3	7.44	0.238	0.815	--	--
%H ₂ O	69.4	80.5	88.1	85.9	87.7	--
MassBal	99.5	98.6	100.3	97.4	99.2	--
ChargeBal	0.95	0.93	1.02	97.4	99.2	--

Table 3-4. Chemical Analyses, Tank S-110 Cascade Dissolution Test, 21 °C.
(Chemical analytes in g/L except as noted)

Analyte	H ₂ O Contact					Acid-Dissolved Solids
	1	2	3	4	5	
Vol% Diln	95	188	278	369	459	--
Sample ID	S110C1	S110C2	S110C3	S110C4	S110C5	S110ACID
S01R000...	232	236	240	244	248	252
Density (g/mL)	1.477	1.221	1.047	1.002	1.001	--
Al	11.3	2.09	0.325	0.076	0.073	4.14
Cr	4.31	1.05	0.188	0.062	0.011	15.8
Fe	< 0.02	< 0.01	< 0.01	< 0.01	< 0.01	1.48
K	0.732	0.217	0.029		0.003	< 0.05
Na	208	103	22.4	11.4	1.79	2.46
P	0.549	1.42	1.28	0.179	0.084	0.265
S	4.17	2.74	0.451	0.065	0.011	< 0.01
Si	0.361	0.097	0.025	0.006	0.022	0.381
F	0.340	0.921	0.450	< 0.12	< 0.02	--
Cl	2.40	0.512	0.246	0.354	0.023	--
NO ₂	26.1	6.34	< 1.10	< 1.10	< 0.12	--
NO ₃	294	200	28.3	3.46	0.457	--
PO ₄ ³⁻	1.67	4.01	3.91	< 1.20	0.251	--
SO ₄ ²⁻	10.3	7.00	< 1.40	< 1.40	< 0.15	--
C ₂ O ₄ ²⁻	< 1.10	1.24	8.68	17.7	1.88	--
TIC	9.18	2.40	0.423	0.109	0.042	--
TOC	0.848	0.345	2.08	4.18	0.521	--
OH	15.6	4.08	1.08	0.294	--	--
%H ₂ O	54.6	69.8	94.3	96.4	99.7	--
MassBal	97.7	98.1	100.9	99.9	100.2	--
ChargeBal	1.06	1.01	0.99	0.94	1.14	--

Table 3-5. Chemical Analyses, Tank U-107 Cascade Dissolution Test, 21 °C.
(Chemical analytes in g/L except as noted)

Analyte	H ₂ O Contact					Acid-Dissolved Solids
	1	2	3	4	5	
Vol% Diln	95	183	269	358	450	--
Sample ID	U107C1	U107C2	U107C3	U107C4	U107C5	U107ACID
S01R000...	233	237	241	245	249	253
Density (g/mL)	1.391	1.138	1.081	1.088	1.018	--
Al	8.06	1.89	0.317	0.098	0.059	6.01
Cr	0.305	0.232	0.053	0.018	0.007	2.59
Fe	< 0.02	< 0.01	< 0.01	< 0.01	< 0.01	1.78
K	1.17	0.259	< 0.10	< 0.10	< 0.02	< 0.01
Na	179	60.4	37.7	34.7	13.9	2.16
P	1.98	6.08	13.9	15.2	6.06	0.613
S	0.677	0.167	0.029	< 0.02	< 0.01	0.004
Si	0.298	0.082	0.049	0.031	0.050	0.061
F	0.535	0.199	< 0.12	0.165	0.103	--
Cl	2.65	0.808	1.83	< 0.17	0.028	--
NO ₂	34.9	7.89	1.38	< 1.10	0.145	--
NO ₃	367	80.5	10.3	2.00	0.589	--
PO ₄ ³⁻	5.35	17.6	39.6	46.6	18.6	--
SO ₄ ²⁻	1.42	< 1.40	< 1.40	< 1.40	< 0.15	--
C ₂ O ₄ ²⁻	< 1.10	2.36	< 1.10	< 1.10	< 0.12	--
TIC	2.50	0.657	0.032	0.048	0.024	--
TOC	1.39	0.97	< 0.04	0.047	0.080	--
OH	8.36	3.34	1.62	1.12	--	--
%H ₂ O	49.9	83.6	88.0	87.3	96.6	--
MassBal	95.6	99.6	96.6	95.1	99.9	--
ChargeBal	0.95	1.06	1.02	0.94	0.98	--

Table 3-6. Radionuclide Analyses, Tank BY-109 Cascade Dissolution Test, 21 °C.
(Radionuclides in µCi/mL except as noted)

Analyte	H ₂ O Contact					Acid-Dissolved Solids
	1	2	3	4	5	
Vol% Diln	104	196	280	375	469	--
⁶⁰ Co	< 1.2E-3	< 4.0E-4	< 1.5E-4	< 5.0E-5	< 3.0E-5	< 2.9E-2
^{89/90} Sr	2.14E-1	9.97E-2	4.2E-2	3.7E-2	2.2E-2	1.79E+1
⁹⁹ Tc	7.2E-2	2.2E-2	7.2E-3	2.6E-3	8.0E-4	9.4E-2
¹²⁵ Sb	< 5.9E-2	< 2.3E-2	< 6.8E-3	< 1.8E-3	< 9.0E-4	< 8.3E-2
¹³⁷ Cs	68.3	25.3	8.80	2.62	0.80	1.37
¹⁵⁴ Eu	< 8.3E-3	< 3.0E-3	< 1.1E-3	< 3.1E-4	< 1.0E-4	< 9.3E-2
¹⁵⁵ Eu	< 3.9E-2	< 1.5E-2	< 4.4E-3	< 1.0E-3	< 2.4E-4	< 4.8E-2
²⁴¹ Am	< 9.4E-2	< 3.6E-2	< 1.1E-2	< 2.4E-3	< 2.3E-4	< 4.9E-2
Total Alpha	< 5.0E-5	< 7.5E-4	< 4.1E-4	< 6.0E-5	< 5.0E-5	0.075

Table 3-7. Radionuclide Analyses, Tank S-110 Cascade Dissolution Test, 21 °C.
(Radionuclides in µCi/mL except as noted)

Analyte	H ₂ O Contact					Acid-Dissolved Solids
	1	2	3	4	5	
Vol% Diln	95	188	278	369	459	--
⁶⁰ Co	< 1.6E-3	< 4.0E-4	< 6.0E-5	< 2.0E-5	< 3.0E-5	< 3.4E-2
^{89/90} Sr	2.26E-1	7.0E-2	1.3E-2	1.0E-2	2.0E-3	8.45E+1
⁹⁹ Tc	9.5E-2	1.8E-2	3.3E-3	6.2E-4	8.0E-5	4.6E-3
¹²⁵ Sb	< 7.0E-2	< 2.1E-2	< 2.1E-3	< 1.0E-3	< 8.0E-4	< 9.7E-2
¹³⁷ Cs	93.5	20.7	3.25	0.84	0.63	3.08
¹⁵⁴ Eu	< 1.1E-2	< 2.5E-3	< 4.1E-4	< 1.1E-4	< 9.0E-5	0.26
¹⁵⁵ Eu	< 4.5E-2	< 1.4E-2	< 1.1E-3	< 5.7E-4	< 2.2E-4	< 6.9E-2
²⁴¹ Am	< 1.1E-1	< 3.3E-2	< 2.7E-3	< 1.4E-3	< 2.0E-4	0.28
Total Alpha	< 6.0E-4	< 4.0E-4	< 4.0E-5	< 2.5E-5	< 2.6E-5	0.35

Table 3-8. Radionuclide Analyses, Tank U-107 Cascade Dissolution Test, 21 °C.
(Radionuclides in $\mu\text{Ci/mL}$ except as noted)

Analyte	H ₂ O Contact					Acid-Dissolved Solids
	1	2	3	4	5	
Vol% Diln	95	183	269	358	450	--
⁶⁰ Co	< 1.9E-3	< 5.4E-4	< 7.0E-5	< 3.0E-5	1.7E-4	< 3.2E-2
^{89/90} Sr	2.12E-1	3.1E-2	1.3E-2	1.7E-2	2.8E-3	1.22E+1
⁹⁹ Tc	8.2E-2	2.4E-2	6.1E-3	3.0E-3	8.0E-4	9.5E-3
¹²⁵ Sb	< 6.7E-2	< 2.0E-2	< 2.0E-3	< 1.0E-3	< 7.0E-4	< 9.3E-2
¹³⁷ Cs	85.0	20.3	3.03	0.73	0.46	3.07
¹⁵⁴ Eu	< 1.0E-2	< 2.4E-3	< 3.8E-4	< 1.2E-4	4.5E-4	< 1.0E-1
¹⁵⁵ Eu	< 4.3E-2	< 1.3E-2	< 1.1E-3	< 5.0E-4	< 2.1E-4	< 5.0E-2
²⁴¹ Am	< 1.1E-1	< 3.2E-2	< 2.6E-3	< 1.3E-3	< 1.7E-4	0.27
Total Alpha	5.5E-4	< 4.0E-4	2.5E-4	1.5E-4	8.0E-5	0.25

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Table 3-9. Chemical Analyses, Tank BY-109 Cascade Dissolution Test, 50 °C.
(Chemical analytes in g/L except as noted)

Analyte	H ₂ O Contact					Residual Solids
	1	2	3	4	5	
Vol% Diln	88	166	249	379	561	--
Sample ID	50BY9C1	50BY9C2	50BY9C3	50BY9C4	50BY9C5	50BY9RS
S01R000...	103	106	109	112	115	121/124
Density (g/mL)	1.32	1.23	1.12	1.13	1.11	--
Al	17.2	6.19	2.01	0.41	0.08	21.5
Cr	0.89	0.36	0.15	0.04	0.02	12.6
Fe	< 0.02	< 0.01	< 0.01	< 0.01	< 0.01	7.9
K	2.78	1.04	0.36	0.09	0.06	--
Na	125	70.0	60.0	48.6	44.5	199
P	2.32	4.47	6.31	2.48	0.43	< 4.0
S	1.92	5.97	11.6	14.9	15.8	30.5
Si	0.40	0.44	0.67	0.05	0.07	3.7
F ⁻	4.16	10.1	13.0	14.6	16.1	--
Cl ⁻	1.97	0.76	0.30	0.18	< 0.04	--
NO ₂	33.1	11.8	3.61	< 1.35	0.26	--
NO ₃	83.8	30.0	9.05	1.78	0.30	--
PO ₄ ³⁻	6.47	12.3	17.7	7.11	1.39	--
SO ₄ ²⁻	4.49	15.4	30.7	39.0	48.1	--
C ₂ O ₄ ²⁻	1.19	3.58	6.10	7.02	5.74	--
TIC	4.65	1.81	0.76	0.19	0.09	0.78
TOC	1.53	1.50	2.07	1.46	0.85	2.90
OH	20.3	7.11	2.56	1.74	0.74	--
%H ₂ O	62.9	79.9	83.5	87.4	89.3	39.4
MassBal	89.3	95.1	97.0	97.9	99.6	--
ChargeBal	1.04	1.02	1.00	0.99	0.92	--

Table 3-10. Chemical Analyses, Tank S-110 Cascade Dissolution Test, 50 °C.
(Chemical analytes in g/L except as noted)

Analyte	H ₂ O Contact					Residual Solids
	1	2	3	4	5	
Vol% Diln	83	173	256	392	--	--
Sample ID	50S110C1	50S110C2	50S110C3	50S110C4	--	50S110CRS
S01R000...	104	107	110	113	--	122/125
Density (g/mL)	1.52	1.16	1.04	1.021	--	--
Al	10.7	2.23	0.32	0.11	--	58.7
Cr	4.47	1.12	0.31	0.06	--	88.9
Fe	< 0.03	< 0.01	< 0.01	< 0.01	--	7.78
K	0.79	0.17	0.045	0.004	--	--
Na	223	71.7	20.0	2.98	--	14.4
P	1.54	1.08	0.09	0.025	--	< 4.0
S	2.88	3.84	0.55	0.04	--	< 2.0
Si	0.25	0.09	0.034	0.036	--	3.58
F	0.047	1.44	0.202	0.021	--	--
Cl	2.26	0.55	0.08	0.009	--	--
NO ₂	26.4	6.13	0.80	0.06	--	--
NO ₃	351	82.3	11.3	0.93	--	--
PO ₄ ³⁻	4.76	3.49	0.54	0.08	--	--
SO ₄ ²⁻	7.09	9.94	1.48	0.12	--	--
C ₂ O ₄ ²⁻	0.51	3.47	21.5	3.30	--	--
TIC	7.53	4.90	0.90	0.09	--	0.44
TOC	0.88	1.08	5.17	0.75	--	1.04
OH	15.4	4.03	0.80	0.23	--	--
%H ₂ O	42.5	78.9	93.3	99.1	--	58.6
MassBal	88.6	97.4	99.2	99.9	--	--
ChargeBal	1.06	1.01	0.91	0.99	--	--

Table 3-11. Chemical Analyses, Tank U-107 Cascade Dissolution Test, 50 °C.
(Chemical analytes in g/L except as noted)

Analyte	H ₂ O Contact					Residual Solids
	1	2	3	4	5	
Vol% Diln	87	173	270	--	--	--
Sample ID	U7C1	U7C2	U7C3	--	--	U7RS
S01R000...	105	108	111	--	--	123/126
Density (g/mL)	1.55	1.25	1.02	--	--	--
Al	7.63	2.11	0.23	--	--	101
Cr	0.36	0.15	0.06	--	--	36.3
Fe	< 0.03	< 0.01	< 0.01	--	--	21.9
K	1.28	0.36	0.02	--	--	--
Na	196	92.4	9.95	--	--	27.8
P	12.7	20.6	2.96	--	--	< 10.0
S	0.63	0.18	0.002	--	--	< 5.0
Si	0.30	0.11	0.04	--	--	22.5
F	< 0.33	0.54	0.59	--	--	--
Cl	2.41	0.67	0.04	--	--	--
NO ₂	28.2	8.08	0.04	--	--	--
NO ₃	328	93.0	4.86	--	--	--
PO ₄ ³⁻	43.9	61.0	9.02	--	--	--
SO ₄ ²⁻	1.22	0.38	0.04	--	--	--
C ₂ O ₄ ²⁻	0.64	1.85	0.13	--	--	--
TIC	2.72	0.86	0.06	--	--	0.49
TOC	1.59	1.14	0.35	--	--	0.71
OH ⁻	10.1	3.11	0.70	--	--	--
%H ₂ O	36.8 ^a	71.9	97.3	--	--	45.9
MassBal	78.3 ^a	93.6	100.0	--	--	--
ChargeBal	0.97	0.98	0.90	--	--	--

^a Anomalously low %H₂O analysis results in low MassBal.

Table 3-12. Radionuclide Analyses, Tank BY-109 Cascade Dissolution Test, 50 °C.
(Radionuclides in $\mu\text{Ci/mL}$ except as noted)

Analyte	H ₂ O Contact					Acid-Dissolved Solids
	1	2	3	4	5	
Vol% Diln	88	166	249	379	561	--
⁶⁰ Co	< 2.0E-3	< 2.0E-3	< 1.0E-3	< 1.0E-3	< 2.0E-4	< 2.0E-2
^{89/90} Sr	1.6E-1	5.0E-3	6.0E-3	3.0E-3	5.0E-3	1.34E+2
⁹⁹ Tc	7.4E-2	3.0E-2	1.1E-2	2.1E-3	5.0E-4	6.3E-2
¹²⁵ Sb	< 6.9E-2	< 3.8E-2	< 2.6E-2	< 9.0E-3	< 4.0E-3	< 9.0E-2
¹³⁷ Cs	80	28	9.2	2.0	1.3	11.8
¹⁵⁴ Eu	< 3.0E-3	< 3.0E-3	< 4.0E-3	< 2.0E-3	< 1.0E-3	3.3E-1
¹⁵⁵ Eu	< 1.6E-2	< 1.1E-2	< 1.6E-2	< 6.0E-3	< 3.0E-3	< 9.0E-2
²⁴¹ Am	< 1.6E-2	< 1.1E-2	< 3.6E-2	< 1.5E-2	< 6.0E-3	< 2.1E-1
Total Alpha	< 2.0E-3	< 5.0E-4	4.0E-3	1.0E-3	4.0E-4	6.4E-1

Table 3-13. Radionuclide Analyses, Tank S-110 Cascade Dissolution Test, 50 °C.
(Radionuclides in $\mu\text{Ci/mL}$ except as noted)

Analyte	H ₂ O Contact					Acid-Dissolved Solids
	1	2	3	4	5	
Vol% Diln	83	173	256	392	--	--
⁶⁰ Co	< 5.0E-4	< 6.0E-4	< 1.1E-3	< 2.0E-4	--	0.094
^{89/90} Sr	2.3E-1	1.3E-2	1.4E-2	4.0E-3	--	406
⁹⁹ Tc	6.2E-2	2.0E-2	3.0E-3	3.0E-4	--	0.023
¹²⁵ Sb	< 4.0E-2	< 2.1E-2	< 1.7E-2	< 4.0E-3	--	< 0.11
¹³⁷ Cs	93.3	21.3	3.5	1.3	--	14.5
¹⁵⁴ Eu	< 2.0E-3	< 1.0E-3	< 3.0E-3	< 6.0E-4	--	1.2
¹⁵⁵ Eu	< 1.0E-2	< 5.0E-3	< 1.1E-2	< 3.0E-3	--	0.6
²⁴¹ Am	< 8.0E-3	< 5.0E-3	< 2.5E-3	< 6.0E-3	--	1.1
Total Alpha	< 1.0E-3	< 1.0E-3	1.0E-3	4.0E-4	--	1.5

Table 3-14. Radionuclide Analyses, Tank U-107 Cascade Dissolution Test, 50 °C.
(Radionuclides in $\mu\text{Ci/mL}$ except as noted)

Analyte	H ₂ O Contact					Acid-Dissolved Solids
	1	2	3	4	5	
Vol% Diln	87	173	270	--	--	--
⁶⁰ Co	< 2.0E-3	< 2.0E-3	< 5.0E-4	--	--	0.11
^{89/90} Sr	3.5E-1	1.7E-2	4.0E-3	--	--	54.0
⁹⁹ Tc	7.8E-2	2.4E-2	2.0E-3	--	--	0.08
¹²⁵ Sb	< 7.8E-2	< 3.7E-2	< 8.0E-3	--	--	< 0.30
¹³⁷ Cs	83.4	23.2	2.4	--	--	47.0
¹⁵⁴ Eu	< 4.0E-3	< 4.0E-3	< 1.0E-3	--	--	0.87
¹⁵⁵ Eu	< 2.0E-2	< 1.2E-2	< 4.0E-3	--	--	< 0.23
²⁴¹ Am	< 1.6E-2	< 8.0E-3	< 1.2E-2	--	--	2.0
Total Alpha	5.0E-3	5.0E-4	8.0E-4	--	--	0.25

3.2 FEED STABILITY TESTS

The **Feed Stability** test procedure was described in Section 2.5. Weights of diluent added and residual solids after each contact are shown in Table 3-15. Liquid phase compositions are assumed to be similar to those presented in Tables 3-3 through 3-5.

Table 3-15. Raw Data, Feed Stability Test at 21 °C.
(weights in grams, volumes in mL)

	BY-109	S-110	U-107
Composite Sample Wt, g	33.529	38.607	40.637
Wt H ₂ O Added (1 st wash)	21.836	21.419	20.980
Total Vol, mL	40.0	41.5	43.0
1 st Water Wash Sample ID	BY9C1	S110C1	U107C1
CSol Vol	16.5	7.0	13.5
CSol Wt	27.848	12.168	20.698
Wt H ₂ O Added (2 nd wash)	18.147	17.752	19.733
Total Vol, mL	34.5	25.0	32.5
2 nd Water Wash Sample ID	BY9C2	S110C2	U107C2
CSol Vol	14.0	5.0	10.0
CSol Wt	23.963	6.806	14.681
Wt H ₂ O Added (3 rd wash)	17.359	17.982	19.879
Total Vol, mL	32.5	23.5	30.0
3 rd Water Wash Sample ID	BY9C3	S110C3	U107C3
CSol Vol	12.5	3.5	10.5
CSol Wt	19.785	4.803	8.496
Wt H ₂ O Added (4 th wash)	17.777	16.895	17.852
Total Vol, mL	30.0	21.0	23.5
4 th Water Wash Sample ID	BY9C4	S110C4	U107C4
CSol Vol	9.5	3.5	3.5
CSol Wt	15.399	4.019	3.395
Wt H ₂ O Added (5 th wash)	17.881	17.622	16.701
Total Vol	27.5	21.5	19.5
5 th Water Wash Sample ID	BY9C5	S110C5	U107C5
CSol Vol	7.5	3.0	2.5
CSol Wt	11.303	3.933	1.899

Six mixtures were prepared, labeled Cone A – Cone F. Each mixture contained equal volumes (7.0 mL from each tank for cones A-E, 5.0 mL each for cone F) of clear supernatant liquid from the collection vials resulting from the water contacts shown in Table 3-15. The mixtures were prepared as follows (where major anions in each collection vial are shown in parentheses):

Cone A: BY9C4 (F^- , SO_4^{2-}) + S110C1 (NO_3^-)
 Cone B: BY9C4 (F^- , SO_4^{2-}) + U107C1 (NO_3^-)
 Cone C: S110C4 ($C_2O_4^{2-}$) + U107C1 (NO_3^-)
 Cone D: U107C3 (PO_4^{3-}) + S110C1 (NO_3^-)
 Cone E: U107C3 (PO_4^{3-}) + BY9C5 (F^- , SO_4^{2-})
 Cone F: U107C4 (PO_4^{3-}) + BY9C5 (F^- , SO_4^{2-}) + S110C1 (NO_3^-)

In all six cases, solids formed within minutes after mixing. In cone D, a virtual 'gel' formed – not a true gel, but a slurry of long needles that immobilized the solution, acting like a gel. The 'gel' could be broken by very vigorous shaking, after which the slurry settled out somewhat.

Cones A – E had a total volume of 14 mL; cone F had a total volume of 15 mL. All six cones were left to sit for three days. Then the solids that formed were identified by polarized light microscopy (PLM), and the following observations were made:

Cone A contained approximately 0.5 mL solids, strongly adhering to the walls of the cone and to each other. Solids were identified by PLM as containing three phases, which were, in order of abundance, Na_3FSO_4 , $Na_7F(PO_4)_2 \cdot 19H_2O$, and $Na_2C_2O_4$.

Cone B was very similar in appearance to Cone A. The PLM showed two phases in approximately equal proportion, Na_3FSO_4 and $Na_7F(PO_4)_2 \cdot 19H_2O$. These solids were also examined by SEM/EDS, with the same finding. Example images are shown in Figures 3-1, 3-2, and 3-3.

Cone C contained 1.0 mL of uniformly-white, fine (small particle size) settled solids, which were identified by PLM as $Na_2C_2O_4$. No other phases were present.

Cone D, which had initially formed a 'gel', contained 4.5 mL of clear-to-white settled solids after vigorous shaking and subsequent settling. PLM showed the solids to be about 99% $Na_3PO_4 \cdot 12H_2O$ and 1% $Na_7F(PO_4)_2 \cdot 19H_2O$.

Cone E contained 1.5 mL of settled solids with some apparent layering. PLM showed just one solid phase, $Na_7F(PO_4)_2 \cdot 19H_2O$.

Cone F was identical to Cone E (1.5 mL settled solids, single phase $Na_7F(PO_4)_2 \cdot 19H_2O$), except that no layering was noted in the settled solids.

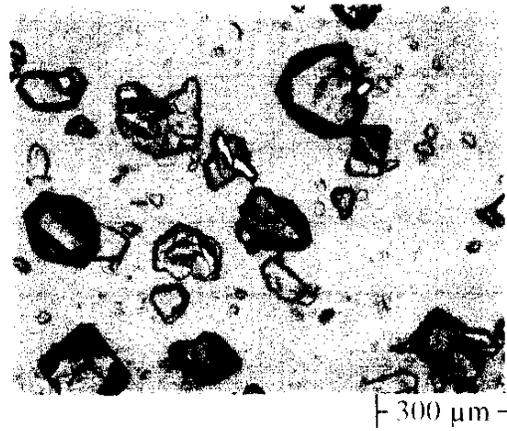


Figure 3-1. PLM image of Na_3FSO_4 and $\text{Na}_7\text{F}(\text{PO}_4)_2 \cdot 19\text{H}_2\text{O}$ crystals from Cone B

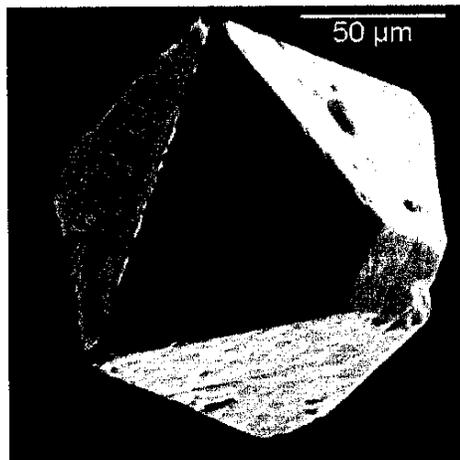


Figure 3-2. SEM image of $\text{Na}_7\text{F}(\text{PO}_4)_2 \cdot 19\text{H}_2\text{O}$ crystal from Cone B.

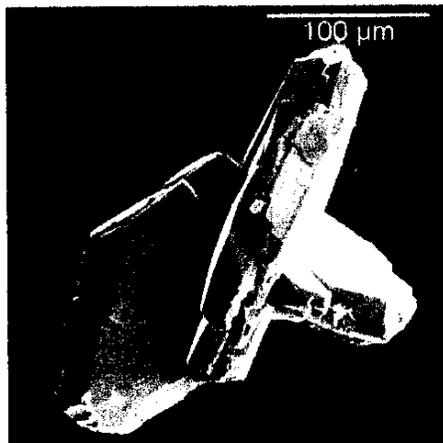


Figure 3-3. SEM image of Na_3FSO_4 crystals from Cone B.

4.0 DILUTION RESULTS, VOLUME AND WEIGHT MEASUREMENTS

All results quoted in this section have been “normalized” to a common basis for comparison. The common basis is defined in each sub-section as appropriate.

One way to measure the effectiveness of the dissolution brought about by the stepwise addition of water to the saltcake is to measure the weight percent or volume percent of centrifuged solids remaining after each dissolution contact. Although the weight percent numbers may be more accurate (having been measured directly by weighing the samples), the volume percent numbers (which are in-part calculated from density and weight measurements) are more useful to tank farm engineers, so the volume percent numbers are emphasized here.

The volume of centrifuged solids relative to the original sample volume gives the “Vol% CSol” values presented in Tables 4-1 through 4-3 for the Cascade dissolution tests at 21 and 50 °C, plus the Feed Stability tests at 23 °C. The cumulative “Vol% Dilution” is the sum of the volumes of water added relative to the original sample volume.

If the assumption is made that the “Vol% CSol” after 30 minutes in the centrifuge is roughly equivalent to the volume of settled and drained solids in a tank after some months of settling and saltwell pumping, then the relative volume of saltcake that can be removed by pumping liquid from the tank should be roughly equivalent to the complement of the “Vol% CSol” (i.e., 100 minus that number). That complement could be called “Vol% Saltcake Dissolved” (using a loose definition of the word “dissolved”). Those data are plotted in Figure 4-1 for all three tanks at each of the three temperatures.

The graphs in Figure 4-1 can be used to predict how much saltcake can be retrieved at a given volume of diluent. For example, if tank U-107 contains 1.4 ML (360 kgal) of saltcake (Hanlon 1999), then addition of 2.8 ML (720 kgal) of water (200 Vol% dilution) would dissolve about half of the saltcake at 21 °C, or 80% of the saltcake at 50 °C.

It is clear from the plots shown in Figure 4-1 that 500% dilution was not sufficient to dissolve all of the soluble salt in tank BY-109 (curve still increasing at 500% dilution), whereas dissolution was nearly complete at 200% dilution for tank S-110; tank U-107 falls between those extremes. It is also evident that temperature had a small but measurable effect for tanks BY-109 and S-110, and a relatively large effect for tank U-107.

The relatively large discrepancy between the curves at 21 °C (Cascade test) and 23 °C (Feed Stability test) for tank BY-109 is one indication that the composite sample was not well homogenized. The nitrate ion analyses of the 21 °C and 50 °C Cascade tests also point to some sample inhomogeneity for the BY-109 composite sample.

Table 4-1. Volumetric Dissolution Profiles, Tank BY-109
 [all values in Vol% (volume percent); CSol = centrifuged solids]

Contact	21 °C Cascade Test		23 °C Feed Stability Test		50 °C Cascade Test	
	Dilution	CSol	Dilution	CSol	Dilution	CSol
1	104	90	125	94	88	80
2	196	76	229	80	166	68
3	280	64	328	71	249	57
4	375	52	429	54	379	36
5	469	36	531	43	561	23

Table 4-2. Volumetric Dissolution Profiles, Tank S-110

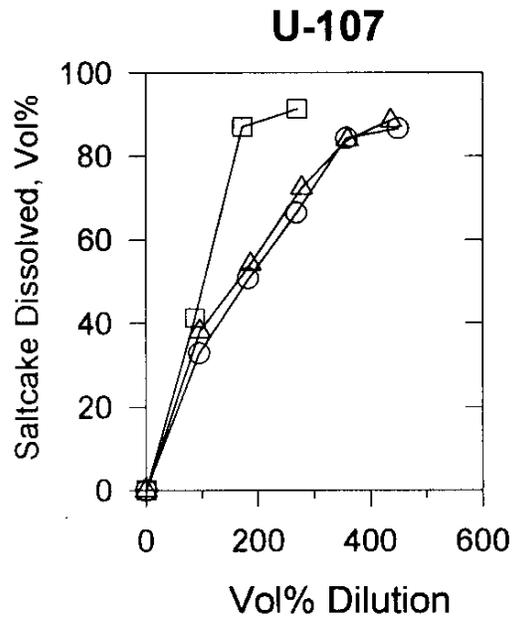
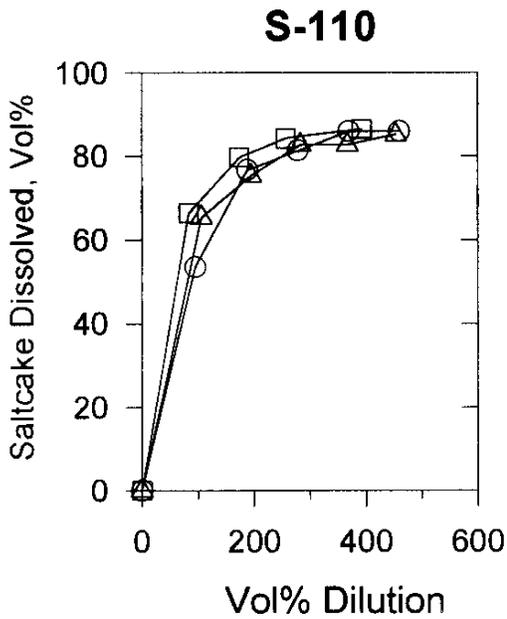
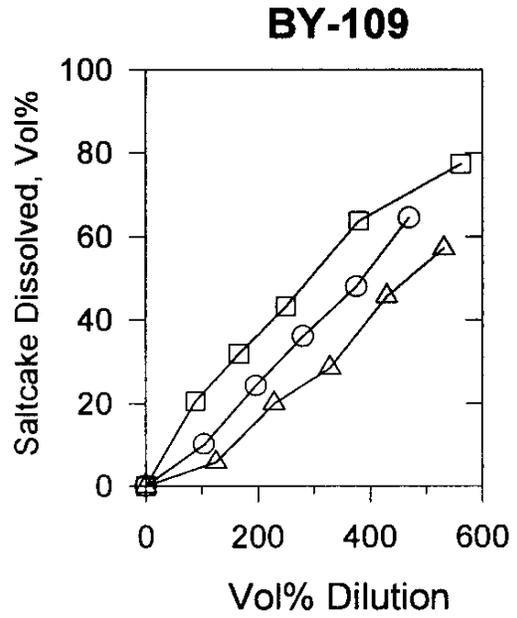
Contact	21 °C Cascade Test		23 °C Feed Stability Test		50 °C Cascade Test	
	Dilution	CSol	Dilution	CSol	Dilution	CSol
1	95	46	106	35	83	34
2	188	23	194	25	173	20
3	278	19	282	17	256	16
4	369	14	366	17	392	14
5	459	14	453	15	--	--

Table 4-3. Volumetric Dissolution Profiles, Tank U-107

Contact	21 °C Cascade Test		23 °C Feed Stability Test		50 °C Cascade Test	
	Dilution	CSol	Dilution	CSol	Dilution	CSol
1	95	67	96	62	87	59
2	183	49	187	46	173	13
3	269	34	278	28	270	9
4	358	16	360	16	--	--
5	450	13	437	12	--	--

Figure 4-1. Amount of Saltcake Dissolved as a Function of Temperature and Volume of Water Added

- 50 °C
- △—△ 23 °C
- 21 °C



5.0 CHEMICAL COMPOSITION TRENDS

The gross sample behavior explored in the previous section (i.e., volume of centrifuged solids as a function of dilution) can be understood in terms of what happens to individual saltcake components. These trends are discussed in Section 5.

The overall composition of the composite sample from each tank was determined by direct analysis of the solid plus water-digest and fusion-digest preparations. The results are summarized in Tables 5-1 (chemical) and 5-2 (radionuclides).

Each of the three saltcakes has its unique characteristics. Tank BY-109 saltcake is unusually low in nitrate and high in fluoride and sulfate. Tank S-110 saltcake is high in nitrate and unremarkable in all other anions, though it does have more oxalate than the other two samples. Tank U-107 saltcake is also high in nitrate, and it has an unusual combination of high phosphate / low fluoride.

5.1 LIQUID PHASE COMPOSITION TRENDS

Figure 5-1 (tank BY-109), Figure 5-2 (tank S-110), and Figure 5-3 (tank U-107) show the dissolution behaviors of the major analytes of interest. These are plots of the analytical data from Tables 3-3 through 3-5.

5.1.1 Nitrite, Chloride, Carbonate

The amount of nitrite, chloride, and carbonate in all of the saltcake samples was so low that they were completely dissolved at the lowest dilution level in all cases. Their concentrations in the liquid phase during the cascade dissolution tests fit the standard dilution pattern of a soluble salt that is completely dissolved at the lowest dilution. In subsequent dilutions, the only source of nitrite, chloride, and carbonate is the interstitial liquid remaining with the centrifuged solids from the previous dilution. Therefore, no new information about these analytes is learned from these tanks. Figure 5-3 shows the carbonate data for tank U-107, which is typical of all of the data for these analytes.

Table 5-1. Overall Composition (in wt%) of Composite Saltcake Samples
(As Determined by Direct Analysis)

	BY-109	S-110	U-107
Al (fusion) ^a	1.97	1.60	1.25
Al (water) ^b	1.48	0.93	0.79
Cr (fusion)	0.27	1.29	0.24
Cr (water)	0.07	0.36	0.03
Fe (fusion)	< 0.10	< 0.10	0.11
K (water)	0.23	0.07	0.10
Na (fusion)	24.9	22.9	21.8
Na (water)	22.7	22.4	21.0
P (fusion)	0.87	< 0.39	2.14
P (water)	0.84	0.21	1.74
S (fusion)	4.25	0.44	< 0.21
S (water)	3.92	0.48	0.06
Si (fusion)	< 0.10	< 0.10	< 0.10
Si (water)	0.73	0.05	0.06
F ⁻	5.32	0.10	0.03
Cl ⁻	0.19	0.23	0.25
NO ₂ ⁻	2.80	2.14	2.96
NO ₃ ⁻	6.94	34.5	35.8
PO ₄ ³⁻ by IC	2.37	0.69	5.16
PO ₄ ³⁻ by ICP ^c	2.57	0.63	5.33
SO ₄ ²⁻ by IC	11.5	1.24	< 0.27
SO ₄ ²⁻ by ICP ^c	11.8	1.45	0.20
C ₂ O ₄ ²⁻	1.96	1.47	< 0.20
CO ₃ ²⁻	2.34	4.90	1.31
"acetate" ^d	0.69	0.03	0.59
OH ⁻	1.54	1.23	2.26
H ₂ O	33.2	16.6	24.5
Mass Bal ^d	99.3	92.3	98.3
Chg Bal (+/-) ^d	1.02	1.03	0.92

^a (fusion) = result from analysis of KOH fusion digest preparation.

^b (water) = result from analysis of water digest preparation.

^c calculated from water digest ICP result

^d see Section 3.1 for definitions of "acetate", Mass Bal, and Chg Bal (+/-)

^e probably erroneously low due to analytical error; should be closer to 24%

Table 5-2. Radionuclide Activities (in $\mu\text{Ci/g}$) of Composite Saltcake Samples
(from fusion digest preparation except as noted)

	BY-109	S-110	U-107
^{60}Co	< 0.042	< 0.088	< 0.022
$^{89/90}\text{Sr}$	19.1	38.1	5.80
$^{89/90}\text{Sr}$ (water)	0.37	0.09	0.09
^{99}Tc	0.080	0.076	0.092
^{99}Tc (water)	0.072	0.076	0.092
^{125}Sb	< 0.44	< 0.75	< 0.35
^{137}Cs	74.5	83.8	95.1
^{137}Cs (water)	70.4	86.9	89.7
^{154}Eu	< 0.13	< 0.34	< 0.07
^{155}Eu	< 0.26	< 0.46	< 0.20
^{241}Am	< 0.60	< 1.08	< 0.48
Total Alpha	0.081	0.134	0.104

5.1.2 Nitrate

The concentration of nitrate follows a simple dilution pattern for tanks BY-109 (not shown) and U-107 (Figure 5-3). For tank S-110 (Figure 5-2), the apparent discrepancy between the 21 °C curve and the 50 °C curve is explainable. At 50 °C, all of the nitrate is in solution at the lowest dilution level, and the nitrate concentration in the liquid phase for subsequent dilutions follows the usual pattern. At 21 °C, some of the nitrate remains in the solid phase at the lowest dilution, so the second dilution shows a concentration much higher than what would otherwise result from simply the contribution from the interstitial liquid. Thus, the nitrate concentration at 21 °C is lower than at 50 °C for the first dilution (at about 100 vol% dilution), and higher for the second (at about 180 vol% dilution).

5.1.3 Fluoride

As indicated in Table 5-1, fluoride is a significant contributor only to the saltcake from tank BY-109. Solid phase analyses (Section 6) show that the major solid phase in tank BY-109 saltcake is the sodium fluoride sulfate double salt, Na_3FSO_4 . Figure 5-1 shows that the fluoride concentration in the liquid phase is closely parallel to the sulfate concentration. Both fluoride and sulfate are lower in concentration at 50 °C than at 21 °C, especially at higher dilution levels. This inverse temperature effect on solubility is common for other anhydrous multi-valent sodium salts, such as Na_2CO_3 and Na_2SO_4 .

Figure 5-1. Concentrations in Supernatant Liquid, Tank BY-109
(open symbols 21 °C, filled symbols 50 °C)

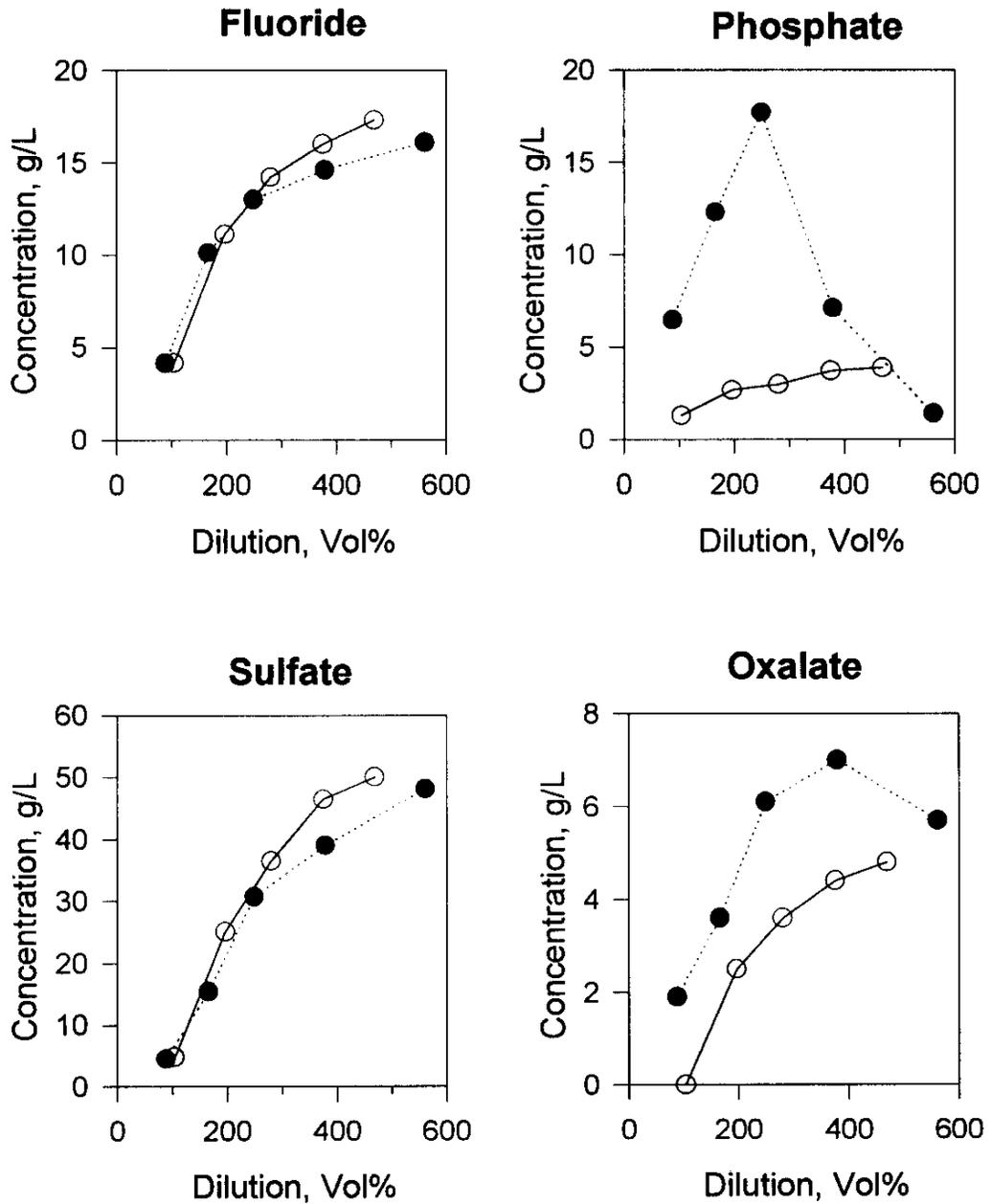


Figure 5-2. Concentrations in Supernatant Liquid, Tank S-110
 (open symbols 21 °C, filled symbols 50 °C)

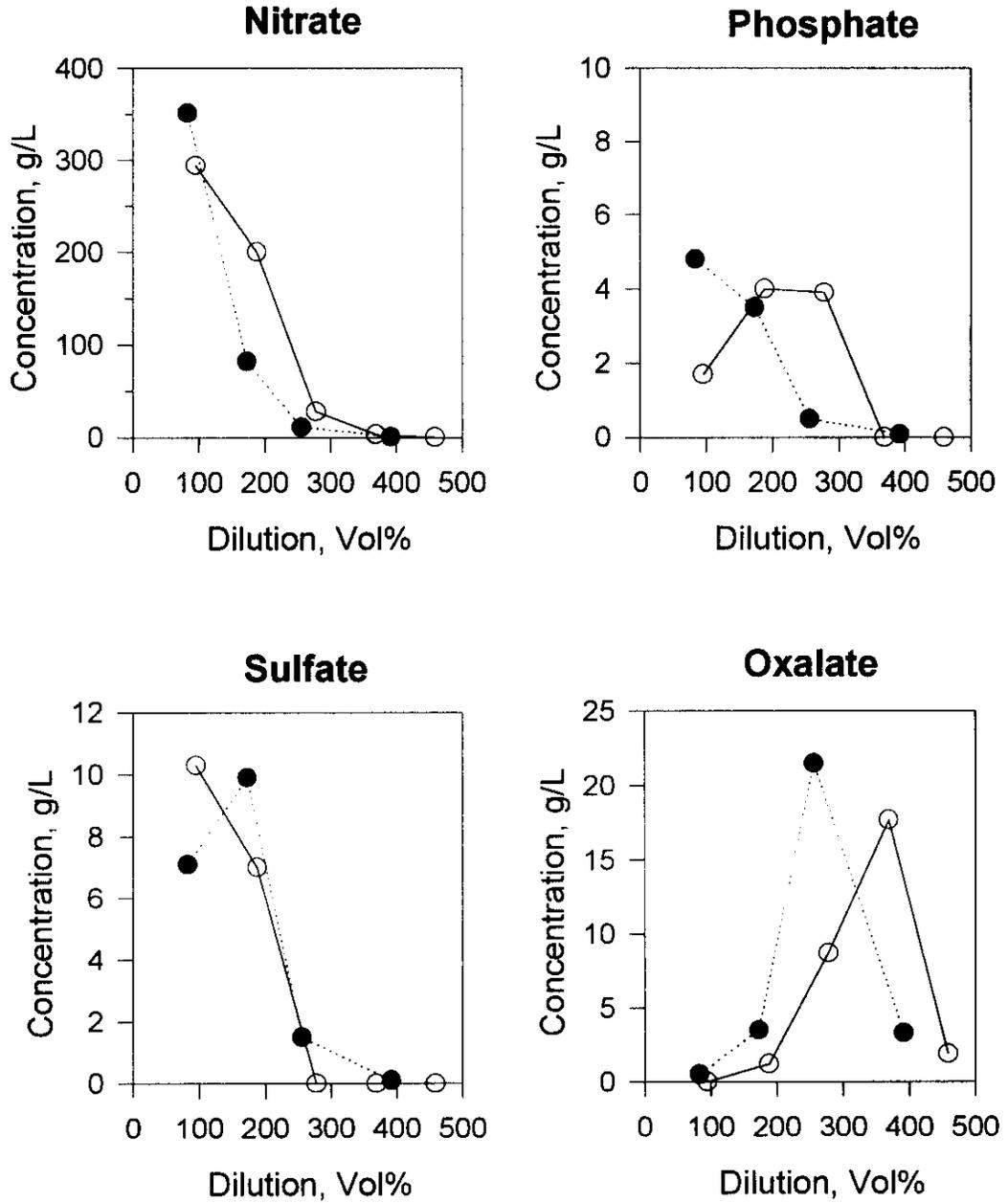
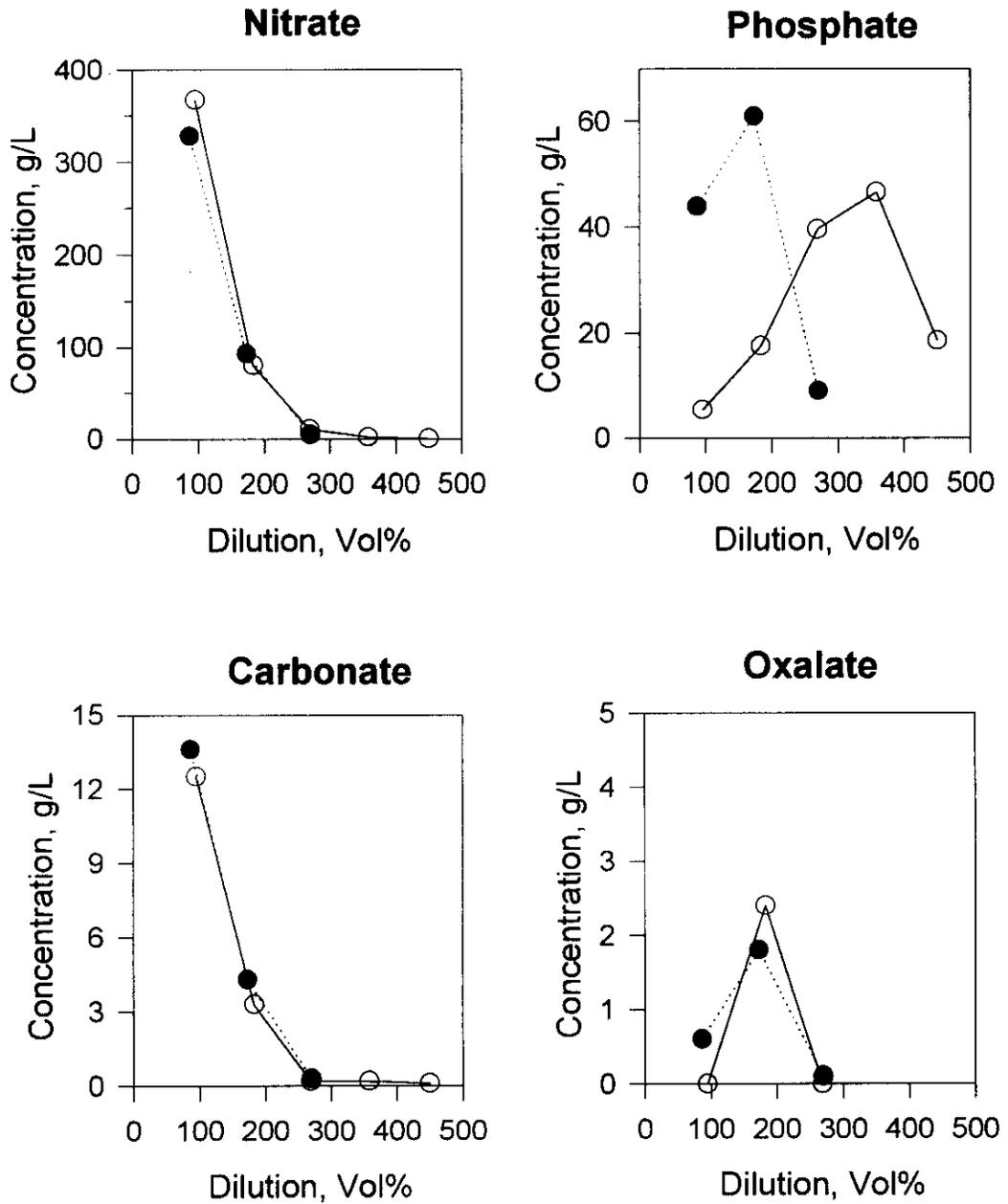


Figure 5-3. Concentrations in Supernatant Liquid, Tank U-107
(open symbols 21 °C, filled symbols 50 °C)



5.1.4 Phosphate

Phosphate is a major component of the saltcake from tanks U-107 and BY-109, though the character of the phosphate salt is quite different between the two tanks. In BY-109 saltcake, the phosphate is present as the fluoride phosphate double salt, $\text{Na}_7\text{F}(\text{PO}_4)_2 \cdot 19\text{H}_2\text{O}$. In U-107 saltcake, the solid phase is $\text{Na}_3\text{PO}_4 \cdot 12\text{H}_2\text{O}$. Both salts are very much more soluble at 50 °C than at 21 °C, as shown in Figures 5-1 and 5-3 (and is also evident in Figure 5-2). The phosphate salt is much more soluble, though, than the double salt at both temperature. Thus, the phosphate concentration peaks earlier in the dilution sequence in tank U-107, and at much higher concentration. In the BY-109 saltcake, the double salt is still present in the solid phase at 21 °C even at the highest dilution level.

5.1.5 Sulfate

Sulfate is the dominant anion in tank BY-109 saltcake, on a weight percent basis, more prevalent even than nitrate. It is present mainly as the sodium fluoride sulfate double salt, as mentioned previously. It is still present in the solid phase even after dilution to nearly 600% by volume (Figure 5-1).

In tank S-110, which contains only about one-tenth as much sulfate as BY-109, the sulfate is probably present as sodium sulfate rather than the double salt, based on the low fluoride content (Table 5-1). Here, the sulfate is not completely dissolved until the second dilution step, as shown in Figure 5-2. Also shown in Figure 5-2 is clear evidence that the solubility of the sulfate is higher at 21 °C than at 50 °C.

5.1.6 Oxalate

Oxalate is a significant component in tanks BY-109 and S-110 (Table 5-1). Its solubility is highly dependant on ionic strength and on temperature. Thus, the oxalate concentration is roughly twice as high at 50 °C as at 21 °C (Figures 5-1 and 5-2), and it is several times higher in S-110 than in BY-109 due to the lower ionic strength (lower sodium ion concentration) in S-110 after the sodium nitrate has dissolved.

5.2 DISTRIBUTION BY FRACTION

In the Cascade dissolution tests, each composite saltcake sample was dissolved by contacting it repeatedly with water, decanting the liquid phase after each contact. This was a batch-wise approximation of a continuous in-tank retrieval process. The progress of the dissolution was followed by performing chemical analyses of each 'fraction' resulting from the test, i.e., the liquid phase from each contact plus the residual solids at the end of the dissolution.

The fractional dissolution gives a good initial understanding of the saltcake dissolution process achieved by the stepwise addition of water. Multiplication of the analyte concentration or activity (Tables 3-3 through 3-14) by the volume of solution (or weight of residual solids) gives the total number of grams or microcuries of analyte in each fraction.

Table 5-3 shows the amount (grams or μCi) of each component in each fraction from the BY-109 Cascade test at 21 °C. The column labeled "Total" is the sum of the six fractions. The column labeled "Percent Dissolved" is 100 times the sum of the five liquid samples divided by the total. Calculations for the other tests are not shown, but the results are summarized in Table 5-4.

Figures 5-4 through 5-6 are called "dissolution profile" plots. They show the "Percent Removed" on the y-axis, defined as 100 times the cumulative weight of constituent removed in the decanted liquid phase divided by the total amount present (liquid and solid phases) in the undiluted sample. The "Percent Removed" at the highest dilution point matches the "Percent Dissolved" in the preceding tables. The legend for each figure follows the order of dissolution, with the components with the highest percent removed listed at the top of the legend. Each of these plots represents the 21 °C test.

There is a short list of components that are essentially 100% in the liquid phase at all levels of dilution, namely nitrite, chloride, carbonate, hydroxide, and potassium. In Tables 3-3 through 3-14, the concentrations of these components in the liquid phase are seen to drop sharply from one contact to the next in all cases. The Percent Removed for each of these components depends only on the separation of the liquid phase from the solid phase. The variation from tank to tank for these components, most notable at the lowest dilution levels, correlates with the amount of centrifuged solids – the more solids, the more interstitial liquid, and the lower the percent removed.

Table 5-3. Distribution by Fraction in Tank BY-109 at 21 °C
 (total undiluted sample weight 40.496 g)
 (chemicals in grams, radionuclides in μCi)

Analyte	Liquid Samples					Residual Solids	Total	Percent Dissolved
	1 st Contact	2 nd Contact	3 rd Contact	4 th Contact	5 th Contact			
F ⁻	0.10	0.24	0.28	0.36	0.39	Not analyzed (n.a.)	1.37	64 ^b
Cl ⁻	0.05	0.02	0.01	0.00	0.00		0.07	100
NO ₂ ⁻	0.76	0.27	0.07	0.03	0.00		1.13	100
NO ₃ ⁻	3.17	1.12	0.27	0.09	0.00		4.65	100
PO ₄ ³⁻	0.03	0.06	0.06	0.08	0.09	0.78 ^a	1.10	29
SO ₄ ²⁻	0.12	0.55	0.71	1.04	1.12	1.03 ^a	4.58	75
C ₂ O ₄ ²⁻	0.00	0.05	0.07	0.10	0.11	n.a.	0.33	38 ^b
CO ₃ ²⁻	0.43	0.16	0.06	0.02	0.01	n.a.	0.68	100
Al	0.37	0.13	0.04	0.01	0.01	0.01	0.57	70
Cr	0.017	0.006	0.002	0.001	0.001	0.048	0.075	24
Fe	0.00	0.00	0.00	0.00	0.00	0.030	0.030	0
K	0.059	0.022	0.007	0.002	0.000	0.008	0.098	92
Na	2.88	1.51	1.00	1.06	1.11	1.91	9.46	80
P	0.011	0.018	0.020	0.003	0.003	0.255	0.361	29
S	0.047	0.181	0.265	0.392	0.416	0.342	1.644	79
Si	0.005	0.009	0.008	0.003	0.001	0.005	0.032	86
^{89/90} Sr	5.14	2.19	0.82	0.83	0.50	617.6	627.0	2
⁹⁹ Tc	1.73	0.48	0.14	0.06	0.02	0.32	2.75	88
¹³⁷ Cs	1639	557	172	59	18	47	2492	98

^a calculated from ICP result for P or S.

^b based on comparison of Total (sum of liquid fractions) with Table 5-1.