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Tank 241-S-112 Saltcake Dissolution Laboratory Test Report

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Abstract: Laboratory tests were performed on core composite samples from tank 241-S-112 to support engineering plans to retrieve saltcake from the tank. Test results show that greater than 98% by weight of the saltcake dissolves in water at 150% cumulative dilution. Data are presented showing bulk dissolution as a function of dilution as well as the composition of the liquid phase as a function of dilution.

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TANK 241-S-112 SALTCAKE DISSOLUTION LABORATORY TEST REPORT

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ACRONYMS AND ABBREVIATIONS

ChargeBal	charge balance (ratio of cation equivalents to anion equivalents)
cP	centipoise
CSol	centrifuged solids
EDS	energy dispersive x-ray spectrometer
ESP	Environmental Simulation Program
GEA	gamma energy analysis
IC	ion chromatography
ICP	inductively coupled plasma spectroscopy
kgal	kilogallons (thousand gallons)
MassBal	mass balance
min	minutes
ML	megaliters (million liters)
mL	milliliters
mPa-s	milliPascal-seconds
PLM	polarized light microscope (or microscopy)
RPP	River Protection Project
S-112	single-shell tank 241-S-112
s^{-1}	reciprocal seconds
sec	seconds
SEM/EDS	scanning electron microscopy with energy dispersive x-ray spectroscopy
TGA	thermogravimetric analysis
TIC	total inorganic carbon
TOC	total organic carbon
Vol	volume
Vol%	volume percent
Wt	weight
Wt%	weight percent
XRD	x-ray diffraction

1.0 INTRODUCTION AND EXECUTIVE SUMMARY

Laboratory tests were completed on the dissolution characteristics of Hanford saltcake waste from single-shell waste tank 241-S-112 (S-112). The tests were designed to model the Low Volume Density Gradient approach to saltcake retrieval, in which water (or other solvent) is distributed onto the surface of the saltcake while saturated brine is pumped from the bottom of the tank [CHG 2000].

The River Protection Project (RPP) is tasked with retrieving waste from double-shell and single-shell tanks to support reducing the risk to workers, the public and the environment. The S-112 Saltcake Waste Retrieval Demonstration Project (S-112 Project) is one of three accelerated retrieval demonstrations that support this mission. This project is planned to fulfill the requirements specified in TPA Milestone M-45-03C.

The S-112 Project is proposing dissolution as the primary means of waste retrieval. The S-112 Project needs chemical and physical data on dissolution to support design of systems for retrieving the waste [Mahoney 2001]. Dissolution data will also be used in developing and refining a process control and in-tank leak detection strategy for the retrieval of tank S-112.

The dissolution test program was developed based on previous saltcake dissolution testing performed at Hanford under the Tanks Focus Area program. Enhancements to previous testing were made based on S-112 project needs and uncertainties through the development of a dissolution test plan [Herting 2001b] and several follow-on discussions with project personnel.

1.1 RESULTS SUMMARY

1.1.1 Overall Results

Tank S-112 saltcake was very soluble in water, with approximately 50% of the saltcake dissolving at 50% (w/w) dilution with water, >90% dissolving at 100% dilution, and >98% dissolving at 200% dilution. The core 291 composite sample – which contained more sodium nitrate and correspondingly less sodium carbonate, sulfate, and oxalate than the core 292 composite sample – dissolved more readily than the core 292 material, but both samples were very soluble.

Increasing the temperature from 22 °C to 50 °C caused some improvement in the dissolution of core 291 sample at low dilution, but had no significant effect at high dilution. It had virtually no effect on core 292 material at either low or high dilution. Changing the solvent from water to 2 M NaOH caused a significant worsening in the extent of dissolution for both core samples, especially core 292, where there was a significant amount of undissolved sodium oxalate even at 200% dilution.

1.1.2 Description of Tests

Dissolution testing encompassed five different types of testing on composite samples of S-112 waste. **Cascade (or "stepwise") dissolution** tests were performed on composite saltcake samples from each of two risers. The purpose of these tests is to evaluate the solid/liquid phase distribution of chemicals and radionuclides during tank waste retrieval operations. The tests were performed at ambient temperature (22 °C) and at 50 °C, using water or 2 M NaOH as the diluent. **Feed stability** tests were done to evaluate the potential for re-precipitation of solids in receiver tanks during tank waste retrieval. **Solid phase identification** tests were performed to identify specific chemical salts responsible for controlling solubility of the various anions and cations during retrieval. **Kinetics** tests were done to evaluate the rate at which the saltcake dissolves. **Viscosity** tests were done to evaluate the pumpability of retrieved solutions.

Weight percent (Wt%) dilution is defined in this report as 100 times the weight of diluent (water or 2 M NaOH) divided by the weight of undiluted saltcake composite sample. A 25% dilution, for example, would be 25 g of H_2O added to 100 g of undiluted composite sample.

1.1.3 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 (or 2 M NaOH), 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.

Tank S-112 has the highest overall saltcake solubility of any of the nine saltcake tanks tested thus far [Herting 1998, 1999, 2000, 2001a], with less than 2% by weight residue (centrifuged solids) after additions of water to approximately 150% by weight cumulative dilution. Assuming similar contact with tank wastes and a direct scaling up to the tank waste volume, this corresponds to a residual waste volume in tank S-112 of approximately 10-12 kgal.

1.1.4 Feed Stability Tests

In-tank dissolution and subsequent retrieval of saltcake is envisioned, in the scenario planned for tank S-112, to occur by sprinkling water or inhibited water (or perhaps 2 M NaOH) 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. To the extent that the system

reaches equilibrium before the brine is pumped from the tank, one would expect that components like sodium sulfate and sodium 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). This effect would be reduced if the system failed to reach equilibrium before the solution was pumped from the tank, as might happen, for example, if there were extensive channeling.

The question arises, then, what would happen if the retrieved solution were 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)? The Feed Stability Tests were designed to evaluate whether the late-retrieved fractions would re-precipitate when mixed with the early-retrieved fractions.

In some tanks that have been tested previously (e.g. tanks BY-102 and TX-113, as described in [Herting 2000]), extensive re-precipitation occurred in the receiver tank. In the current study, there was only a small amount of sodium oxalate precipitate that formed in the test with water diluent, and there was no re-precipitation in the test with 2 M NaOH diluent.

1.1.5 Solid Phase Identification

Accurate chemical modeling of the tank waste systems (e.g., with the Environmental Simulation Program, ESP) depends in part on proper identification of the solid phases in equilibrium with the ions in solution. Analysis of solids from tank S-112 were performed 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).

The major phases identified in the saltcake include, in approximate relative order of abundance: $NaNO_3$ >> Na_2CO_3 H_2O > anhydrous Na_2CO_3 > $Na_6CO_3(SO_4)_2$ (Burkeite) >> Na_3PO_4 $12H_2O$, and miscellaneous unidentified phases including a Na-S-rich phase, an Al-rich phase with no associated Si or Cr; and a trace phase containing U-Cr-Mn-Fe.

1.1.6 Dissolution Kinetics

The double shell tank space available to receive the dissolved waste is limited. To insure that the salt solutions are near saturation it is important to understand something about the kinetics of dissolution. If the water sprinkled on top of the saltcake in the tank percolates through the salt bed, the long contact time will assure that it becomes saturated in dissolving salts, as demonstrated in previous bench-scale simulant tests [Bechtold 200]. If the water runs across the top of the bed without percolating into the bed, or if it runs through open channels in the bed, it may not become saturated if the

water/salt contact time is too short. Dissolution kinetics tests were done to evaluate how long the water and saltcake must be in close physical contact to assure saturation. This is a concern because of waste volume projections, and the need to send near-saturated solution to the double shell tanks to conserve tank space.

In duplicate tests, about 9 g of water was added to 8 g of saltcake sample. The length of time allowed for the water to contact the salt was set at 30 sec, 2 min, 5 min, 23 min, or 60 min. Approximately 40% of the saltcake dissolved in 30 sec, increasing non-linearly to about 70% dissolved in 60 min, compared to 95% dissolved at "infinite" mixing time at the same diluent:saltcake weight ratio.

1.1.7 Viscosity

Solution viscosity data are needed to define the pumping and pipeline requirements for transfers of waste from S-112 to the double shell tanks. Viscosity measurements (shear rate vs. shear stress rheograms) were performed on dissolved saltcake solutions under various conditions to evaluate the pumpability of the brines created during retrieval. The brines were created from a mixed saltcake sample containing equal parts of material from both Core 291 and Core 292.

The first brine was made by mixing 82 g of the mixed saltcake with 50 g of water at 50 °C, producing 102 g (70.5 mL) of centrifuged supernatant liquid (brine) representing a solution produced during the "early" part of the retrieval operation, with a high nitrate concentration. The second brine was made by mixing the 30 g of residue from the first contact with 70.5 g of fresh water at 50 °C, producing 98 g (84 mL) of brine representing a solution produced during the "late" part of the retrieval operation, with relatively high sulfate concentration. Each of the brines was diluted 20% by volume with water before running the viscosity at 20, 35, and 50 °C.

Both brines showed Newtonian behavior at all temperatures. Viscosities for the "early" brine ranged from 6 mPa-s (6 cP) at 20 °C to 3 mPa-s (3 cP) at 50 °C. Viscosities for the "late" brine ranged from 2.0 mPa-s (2.0 cP) at 20 °C to 1.4 mPa-s (1.4 cP) at 50 °C. These results are well within the pumpable range for the project.

1.2 COMPOSITION OF TANK WASTES

Two core samples were taken from Tank S-112 in 2001. The two cores (core 291 and core 292) were not very similar to one another in appearance or chemical composition. One composite sample was created for each core by mixing representative amounts of each segment from the core (subject to the limitations of sample recovery). All of the tests described in this report were performed on the composite saltcake samples. A full description of the make-up of the composite samples can be found in [Prilucik 2002].

Table 5-1 shows the overall composition of each core composite sample, as determined in [Prilucik 2002]. Core 291, which was taken near the outer wall of the tank, was relatively dry, and was made up mainly of $NaNO_3$, with relatively small amounts of other salts. Core 292, taken near the center of the tank, was much wetter. It contained about half as much $NaNO_3$ by weight as Core 292, with correspondingly higher amounts of all other salts, especially sodium carbonate and Burkeite, $Na_6CO_3(SO_4)_2$.

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-70-3. 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 core (subject to the limitations of sample recovery). Instructions for preparing the composite samples were provided in the Tank 241-S-112 Core Sampling and Analysis Plan [Lauricella 2001].

2.1 PREPARATION OF COMPOSITE SAMPLES

The saltcake composite samples were made by combining fractions of individual core segment samples into two (duplicate) composite jars for each core [Prilucik 2002]. Table 2-1 shows a summary of the composite sample preparation. The waste in each completed composite jar was mixed thoroughly before taking subsamples for the individual tests.

Core 291 Composite Sample S01T001750 was contained in two 500 mL jars labeled 18813 and 18816. Both jars were approximately half full of saltcake, with no visible liquid phase. The saltcake was dry, crumbly, chunky, and not-quite-white, with green, yellow, and gray tints.

Core 292 Composite Sample S01T001759 was contained in two 500 mL jars labeled 18782 and 18820. Both jars were approximately half full of saltcake, with approximately 0.5 cm (roughly 5-10% by volume) of supernatant liquid. The liquid was blended with the saltcake before taking any aliquot samples. After the blending, the saltcake was fluid enough that aliquots could be removed by pouring from the jar while the jar was being vibrated with a vortex mixer. Without the aid of the vibration, the sample was too thick (viscous) to pour from the jar.

Table 2-1. Segment Samples Used to Create S-112 Core Composite Samples

Core 291			Core 292		
Segment	Jar #	Weight, g	Segment	Jar #	Weight, g
1	18833	5.0	2A	18839	67.4
2	18829	75.0	2B	18831	38.0
3A	18834	57.5	3	18870	89.6
3B	18838	57.0	4	18738	90.0
5	18842	62.8	5A	18872	90.2
5A	18827	36.0	5B	18871	90.1
6	18826	79.4	6	18821	90.1
7A	18664	27.9	7	18739	90.2
7B	18652	39.5	8	18828	65.6
8	18592	29.1	10	18733	127.3
9	18837	67.5	11	18741	62.0
10	18737	144.0	11A	18840	28.1
11	18731	75.2			
12	18540	76.0			
13	18537	39.1			
Composite	Total Wt =	871.0	Composite	Total Wt =	928.6

2.2 CASCADE DISSOLUTION TESTS

Cascade dissolution tests, which have also been called **stepwise** dissolution tests, employ a single saltcake sample that is contacted multiple times with water. The cascade test is a very small scale, batch-wise representation of a continuous in-tank retrieval process. The tests were done at ambient hotcell temperature (21-22 °C) and again at 50 °C using water as diluent, and at ambient temperature using 2 M NaOH as diluent.

For each test, a 60-gram aliquot of saltcake composite sample was placed into a tared 50-mL graduated centrifuge cone. The cone plus saltcake was weighed, then 15 g of water was added (or 15 mL of 2 M NaOH), and the cone was re-weighed. 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, samples were vortex-mixed periodically rather than tumbling end-over-end. 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 15 g portion of water was added and the cycle of mixing/centrifuging/decanting was repeated for a total of six water contacts.

The liquid samples were analyzed by:
- ICP (procedure number LA-505-161 Rev. D-0)
- IC (LA-533-107 Rev. B-0)
- TIC/TOC (LA-342-100 Rev. F-6)
- OH^- (LA-211-102 Rev. D-4)
- %H_2O by oven drying at 120 °C (LA-564-101, Rev G-4)
- density (LA-510-112 Rev. F-0)

and the following radionuclide analyses:
- GEA (^{60}Co, ^{125}Sb, ^{137}Cs, ^{154}Eu, ^{155}Eu, and ^{241}Am, LA-548-121 Rev. F-3, H-0)
- $^{89/90}Sr$ (LA-220-101 Rev. E-5, E-6),
- ^{99}Tc (LA-438-101 Rev. E-5, F-0),
- total alpha (LA-508-101 Rev. G2, H-0),
- ^{14}C (LA-348-104 Rev. D-1),
- 79Se (LA-365-132 Rev. D-6, E-0)
- ICP/MS (LA-506-101 Rev. B-0).

The Test Plan [Herting 2001b] called for the analysis of the residual solids from each test. However, the amount of solids remaining after the six water contacts was too small, in all cases, to allow samples to be analyzed. Instead, the residual solids were combined from all of the dissolution tests done with Core 291 material into a single sample for analysis. Likewise, the residual solids were combined from all of the dissolution tests done with Core 292 material into a single sample. Both residual solids samples were analyzed for TIC/TOC and %H_2O on direct sample, plus ICP and radionuclides on a fusion digest preparation.

2.3 FEED STABILITY TESTS

The objective of this test series was to determine if salt precipitation occurs by mixing clear tank waste solutions "retrieved" from early and late fractions. Dissolutions were performed using the **cascade dissolution** method just described. Both samples were from core 292, one using water as diluent, the other using 2 M NaOH, both at ambient hotcell temperature.

For each test, a 60-gram aliquot of saltcake composite sample was placed into a tared 50-mL graduated centrifuge cone. The cone plus saltcake was weighed, then 15 mL

of diluent 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 jar representing the receiver tank. 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 analogous Cascade test, so the composition of liquid in each Feed Stability collected fraction should be approximately equal to the corresponding sample vials from the Cascade test.

The centrifuged liquid from the second contact was decanted into the same collection jar as the liquid from the first contact (separate jar for the water dissolution test and the NaOH dissolution test), and the collection jar was monitored for at least 20 hours for the formation of solids. If solids formed, they were collected to measure the volume, and then they were analyzed by PLM, SEM/EDS, and XRD to identify the solids. The liquid was returned to the collection jar.

The centrifuged liquid from the third contact was decanted into the collection jar containing the liquid from the first two contacts. The collection jar was monitored as above. This process was repeated for each of the remaining contacts.

A small volume of solids formed in the receiver jar only following the 5th contact with water. The solids were identified as sodium oxalate. For details of the dissolution data and resulting observations, see section 3.2.

2.4 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 S-112 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.

® Laborlux is a registered trademark of Ernst Leitz Wetzlar GmbH, Germany.

® Rigaku is a registered trademark of Rigaku/USA, Inc., Danvers, Massachusetts.

® Aspex is a registered trademark of Alpex, Trafford, Pennsylvania.

Aliquots were taken of individual segment samples before any of the samples were homogenized or composited. The segments used for analysis were:

Core 291 Segments 1, 10, and 12
Core 292 Segments 2A (upper half), 8, and 10.

2.5 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. Sample aliquots from Core 291 were used for the dissolution kinetics tests. Two types of tests were run, called "surface dissolution" and "percolation" tests.

In a surface dissolution test, approximately 8 g of composite saltcake sample was placed into a 30 mL coarse glass frit Buchner filter funnel in such a way as to cover about half the surface of the glass frit, and sloping upward toward the outside wall (Figure 2-1). With the sample under constant vacuum, 10 mL water was dribbled onto the sample near the top so that the water would run down the slope across the surface of the sample and onto the glass frit, where it would be quickly drawn through the frit. The rate of water addition was varied so that the entire addition took 30 sec, 2 min, or 5 min. The filter apparatus was weighed before and after the water addition to determine the change in weight of the saltcake. The collected filtrate was analyzed for anion concentrations by IC and metal ion concentrations by ICP.

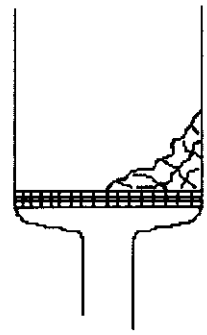


Figure 2-1.
Surface Dissolution

The sample was observed closely during the water addition. When a drop of water struck the surface of the saltcake, it had much more of a tendency to be absorbed into the saltcake, despite the slope, than to run across the surface. Liquid began to flow through the filter frit within a few (~5) seconds after the water addition was started, and stopped flowing through the frit within a few seconds after the water addition was stopped. During the course of the water addition, the sample maintained its general shape and slope, but developed significant (millimeter-sized) channels across the surface from top to bottom. The water addition point was moved intermittently to avoid developing a "hole" in the sample directly below the addition point. During the longer addition times, especially at 23 min and 60 min (see below), a dark brown to black "halo" developed on the surface of the frit extending out ~0.5 cm from the edge of the saltcake sample. This "halo" was caused by water-insoluble solids that washed out of the saltcake onto the surface of the frit, but did not go through the frit.

In a percolation test, approximately 8 g of composite saltcake sample was placed into a 15 mL coarse glass frit Buchner filter funnel, covering the entire glass frit surface to a uniform depth of about 1.5 cm. Water was added to the surface of the saltcake while the vacuum was constantly adjusted to maintain a steady flow through the saltcake, attempting to keep the saltcake bed wet, but without building up any liquid above the surface of the saltcake. As with the surface dissolution test, 10 mL water was added over a period of 30 sec, 2 min, or 5 min. The filter apparatus was weighed before and after water addition, and the filtrate was analyzed by IC and ICP.

Two (duplicate) control samples were prepared by mixing 18 g saltcake with 22 mL water and mixing (tumbling end-over-end) for three days. The samples were centrifuged. The supernatant liquid was decanted and analyzed by IC and ICP, and the centrifuge cones were re-weighed to determine the weight of centrifuged solids.

Based on the results of the initial tests, the surface dissolution tests were repeated at water addition times of 5 min, 23 min, and 60 min, all in duplicate.

2.6 VISCOSITY MEASUREMENTS

Composite saltcake samples were received from cores 291 and 292, and 20g portions of each of them were placed into cones V1 and V2. The detailed data appear in Table 2-2.

Table 2-2. Tank S-112 Viscosity Saltcake Sample Origins

Core No.	**291**	**292**
Composite Jar No.	18813	18820
Labcore No.	S01T001750	S01T001759
Subsample Jar No.	S112/291	S112I-2
Net Grams from Composite Jar to Subsample Jar	58.96	59.171
Description	Moist Gray Solids, No Excess Liquid	Wet Gray Solids, ~5mL Standing Liquid
Net Grams from Subsample Jar to Cone V1	20.117	20.846
Net Grams from Subsample Jar to Cone V2	20.43	21.062

First Contact

Each cone received nominally 25 g water and was placed in a 50 °C bath for an hour (temperature verified by a thermometer in a water-filled dummy cone), with brief removal for mixing six times. The cones were then centrifuged for five minutes, and the supernate (with dark fines) was decanted into new cones V1L and V2L. Table 2-3 contains the detailed data.

Table 2-3. First Water Contact Solids Dissolution

Cone No.	V1	V2	T (°C)
Total Grams Composite	40.963	41.492	27
Grams H_2O Added	24.918	24.943	27
Total Bulk Volume (mL)	44	44	50
Volume (mL) Centrifuged Solids	8	9	50
Grams Centrifuged Solids	14.913	15.267	50
1st Contact Supernate Cone No.	V1L	V2L	50

The supernate in each cone was held at 50 °C overnight before reading its settled solids volume. The settled solids volume was then monitored while cooling the supernates to 20 °C for one hour, wherein no real change was noted. Centrifuging the supernate brought the total solids volume up to 6 mL per cone, but still left some fines in suspension. Mixing in water to dilute 20% by volume, then holding at 20 °C for an hour with six brief mixings, and finally centrifuging, produced a very small amount of centrifuged solids and suspended fines in the supernate. These fines settled overnight to produce ~5mL settled solids in 42 mL total volume. Table 2-4 presents this detailed data.

Second Contact

The remaining solids in cones V1 and V2 from the first contact were then contacted again with portions of water, this time 35g each, and equilibrated at 50 °C, centrifuged, decanted to cones V1S and V2S, cooled, diluted, re-equilibrated and re-centrifuged in the same manner as the first contact to produce the 2nd contact supernates. These supernates were clear, yellow, having very little residual dark fines, and a bulk density of 1.15 g/mL. Tables 2-5 and 2-6 contain the relevant detailed data.

Table 2-4. First Water Contact Supernate and Dilution

1st Contact Supernate Cone No.	V1L	V2L	T (°C)
Grams Supernate Decanted to Supernate Cone	50.94	51.142	50
Total Supernate Volume (mL)	35	35.5	50
Settled Solids Volume (mL) before Cooling	4.5	5	50
Settled Solids Volume (mL) after Cooling	4.5	4.5	20
Volume (mL) Centrifuged Solids after Cooling	6	6	20
Volume (mL) after Dilution	42.5	42	20
Volume Percent Dilution	21.4	20	20
Grams H_2O Added	7.255	5.962	20
Volume (mL) Centrifuged Solids	2	3	20
Volume (mL) Solids after Fines Settled	5	5	20

Table 2-5. Second Water Contact Solids Dissolution

Cone No.	V1	V2	T (°C)
Total Grams Remaining Composite	14.913	15.267	27
Grams H_2O Added	35.432	35.058	27
Total Bulk Volume (mL)	42.5	42.5	50
Volume (mL) Centrifuged Solids	2	2	50
Grams Centrifuged Solids	1.383	1.303	50
Supernate Cone No.	V1S	V2S	50

Rheology Measurements

To measure the rheology of the diluted first contact slurry, the cones V1L and V2L were shaken, and equal portions were poured into the graduate used to administer slurry to the rheometer sample cup. In a like manner, equal portions of the cones V1S and V2S were administered to the cup to measure the rheology of the diluted 2nd contact supernate. Each of these two rheology samples was measured at 20, 35 and 50 °C. Various repeat measurements were made at 20 °C also.

Table 2-6. Second Water Contact Supernate and Dilution

Supernate Cone No.	V1S	V2S	T (°C)
Grams Supernate Decanted to Supernate Cone	48.728	48.932	50
Total Supernate Volume (mL)	42	42	50
Settled Solids Volume (mL) before Cooling	1	1	50
Settled Solids Volume (mL) after Cooling	0.5	0.5	20
Volume (mL) Centrifuged Solids after Cooling	0.1	0	20
Volume (mL) after Dilution	50	50	20
Volume Percent Dilution	19	19	20
Grams H_2O Added	8.391	8.949	20
Volume (mL) Centrifuged Solids	0	0	20
Volume (mL) Solids after Fines Settled	0	0	20

Laboratory Technical Procedure LT-519-106, Revision A-0, was used to conduct the shear rheology measurements. The shear rheology measurements were performed with shear rates ($\dot{\gamma}$) increasing from 0 s^{-1} to 1100 s^{-1}, then decreasing from 1100 s^{-1} to 0 s^{-1} to establish reversibility. The measurements were taken using a MV1 coaxial cylinder sensor system installed on a Haake M5 rheometer in the 11A/3 hot cell. A check standard tested before and after the actual samples achieved 106% recovery of the expected value of 36.3 mPa-s (cP) in both instances. Information associated with these analyses was recorded in controlled laboratory notebook HNF-N-153-1.

The solids in the first contact liquid (from cones V1L and V2L) were found to have remained effectively suspended in the rheometer throughout the temperature series and 20 °C re-measurement, because no accumulation of solids was found at the bottom of the rheometer cup at the end of the series. Therefore the 9% disparity between the first and last measurement at 20 °C must be due to factors other than settling of the solids, and lends support to the adequacy of the intervening measurements at higher temperatures. The second contact liquid had virtually no suspended solids, and so the 22% range of variability of its 20 °C rheology is required to be assigned to factors other than settling. Instrument noise is a good candidate, for example.

The results of the rheometry interpretations are found in Section 3.4.

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, direct 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 2.2. Weights, volumes, and analytical sample identification numbers of the various fractions for the Core 291 dissolution tests are shown in Table 3-1. Corresponding data for the Core 292 tests are shown in Tables 3-2 and 3-3. Analytical data for all of the tests are shown in Tables 3-4a (chemicals) and 3-4b (radionuclides) through 3-10a and 3-10b.

For the tests at 50 °C (Tests F and G), some evaporation occurred during the time the samples were held at the elevated temperature. Each sample was weighed immediately after the water was added and weighed again after the equilibration period. Typically, the weight loss during the equilibration period was about one gram, and was assumed to be water loss only. The post-equilibration weight was used to determine the weight of water added, as reported in the tables.

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:

$$\text{MassBal} = \%H_2O + (0.1 \times \Sigma_i S_i C_i / \text{Density})$$

where the C_i values are concentrations expressed in g/L as shown in Table 3-4a and subsequent tables; S_i is the stoichiometric factor used to convert the analyte weight into species weight; Density is in g/mL; the factor 0.1 converts g/L to g/mL and changes the result into weight percent; and i = OH^-, Al, Cr, K, Na, F^-, Cl^-, NO_2^-, NO_3^-, PO_4^{3-}, SO_4^{2-}, TIC, and TOC. The PO_4^{3-} and SO_4^{2-} values are calculated from the P and S results from the ICP analysis, because they tend to be more consistent than the IC results. Some of the stoichiometric factors include: $S_i = 1.0$ for most analytes, $S_{Al} = 2.2$ to convert Al into AlO_2^-, $S_{TIC} = 5.0$ to convert inorganic carbon (as TIC) to carbonate. The total organic carbon (TOC) value is adjusted by subtracting the contribution of carbon derived from oxalate. For the residual TOC not associated with oxalate, the stroichiometric factor is $S_{TOC} = 2.46$ to account for the mass of oxygen, nitrogen, and hydrogen associated with the carbon in typical organic complexants. This is an approximate factor; the number chosen is based on the formula weight of acetate as a stand-in. The S_i values for AlO_2^- and CO_3^{2-}, for example, are exact factors. Aluminate is used as the anhydrous AlO_2^-

rather than the hydrated $Al(OH)_4^-$ because the hydration water is included in the $\%H_2O$ factor.

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

where the C_+ values are the concentrations of Na and K in μg/mL; W_+ are the respective atomic weights; C_- are the concentrations of anionic species (OH^-, TOC, TIC, Al, Cr, Si, F^-, Cl^-, NO_2^-, NO_3^-, PO_4^{3-}, SO_4^{2-}, $C_2O_4^{2-}$); Z_- are the charges on the respective anions; and W_- are the formula weights of the anions. The Z_- for TOC (the fraction not associated with oxalate) 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 $Al(OH)_4^-$.

Each of the dissolution tests was given an alpha designator for tracking purposes. The following list shows the conditions for each of the Cascade dissolution tests:

Test A – Core 291, H_2O, ambient temperature

Test B – Core 292, H_2O, ambient temperature

Test C – Core 292, H_2O, ambient temperature (duplicate of Test B)

Test D – Core 291, 2 M NaOH, ambient temperature

Test E – Core 292, 2 M NaOH, ambient temperature

Test F – Core 291, H_2O, 50 °C

Test G – Core 292, H_2O, 50 °C

Test H – Core 292, H_2O, ambient temperature (Feed Stability Test)

Test I – Core 292, 2 M NaOH, ambient temperature (Feed Stability Test)

Table 3-1. Raw Data, Cascade Dissolution Tests, Core 291.
(weights in grams, volumes in mL)

	H_2O, 22 °C (Test A)	2M NaOH, 22 °C (Test D)	H_2O, 50 °C (Test F)
Composite Sample Wt, g	59.374	59.288	62.036
Wt Diluent Added (1st wash)	15.012	15.753	15.278
Total Volume	42.0	43.0	45.0
1st Contact Sample ID[a]	S112AC1	S112DC1	S112FC1
CSol[b] Vol	27.5	31.5	23.0
CSol Wt	54.513	58.755	46.034
Wt Diluent Added (2nd wash)	14.533	16.404	13.172
Total Volume	42.5	46.5	37.5
2nd Contact Sample ID[a]	S1122AC2	S112DC2	S112FC2
CSol[b] Vol	18.0	22.5	12.5
CSol Wt	35.257	41.229	23.620
Wt Diluent Added (3rd wash)	17.400	17.317	9.558
Total Volume	36.0	37.5	23.5
3rd Contact Sample ID[a]	S112AC3	S112DC3	S112FC3
CSol[b] Vol	7.5	12.5	2.0
CSol Wt	12.943	22.778	1.717
Wt Diluent Added (4th wash)	22.671	27.207	21.461
Total Volume	29.5	37.0	22.5
4th Contact Sample ID[a]	S112AC4	S112DC4	S112FC4
CSol[b] Vol	~ 1	~ 1	~ 1
CSol Wt	1.027	1.558	0.965
Wt Diluent Added (5th wash)	23.670	23.999	22.207
Total Vol	24.5	23.0	23.5
5th Contact Sample ID	S112AC5	S112DC5	S112FC5
CSol Vol[c]	~ 0.5	~ 0.5	~ 1
CSol Wt	0.636	0.589	1.033
Wt Diluent Added (6th wash)	30.036	33.049	29.516
Total Vol	31.0	31.5	30.0
6th Contact Sample ID	S112AC6	S112DC6	S112FC6
CSol Vol[c]	~ 0.5	~ 0.5	~ 1
CSol Wt	0.742	0.395	1.115

[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 Tests, Core 292, "Baseline" Tests. (weights in grams, volumes in mL)

	H_2O, 22 °C (Test B)	H_2O, 22 °C (Test C)	H_2O, 22 °C (Test H)
Composite Sample Wt, g	57.903	61.792	59.389
Wt H_2O Added (1st wash)	13.851	16.102	15.346
Total Volume	43.0	47.0	45.5
1st Contact Sample ID[a]	S112BC1	S112CC1	--
CSol[b] Vol	21.5	23.0	25.5
CSol Wt	41.711	45.236	47.262
Wt H_2O Added (2nd wash)	15.775	14.318	15.522
Total Volume	37.5	38.0	41.5
2nd Contact Sample ID[a]	S112BC2	S112CC2	--
CSol[b] Vol	11.0	13.5	21.5
CSol Wt	20.750	26.234	35.564
Wt H_2O Added (3rd wash)	14.831	14.536	15.419
Total Volume	25.5	28.0	36.5
3rd Contact Sample ID[a]	S112BC3	S112CC3	--
CSol[b] Vol	4.5	7.5	6.5
CSol Wt	6.378	12.854	9.239
Wt H_2O Added (4th wash)	23.297	22.040	22.945
Total Volume	27.0	29.5	28.0
4th Contact Sample ID[a]	S112BC4	S112CC4	--
CSol[b] Vol	~ 2	~ 3	~ 2.5
CSol Wt	1.856	3.384	2.644
Wt H_2O Added (5th wash)	22.928	22.065	22.979
Total Vol	24.5	25.0	25.0
5th Contact Sample ID	S112BC5	S112CC5	--
CSol Vol[c]	~ 0.5	~ 0.5	~ 1
CSol Wt	0.876	0.924	0.992
Wt H_2O Added (6th wash)	30.579	29.727	29.009
Total Vol	31.5	31.0	30.5
6th Contact Sample ID	S112BC6	S112CC6	--
CSol Vol[c]	~ 0.5	~ 0.5	~ 1
CSol Wt	0.900	0.962	0.997

[a] decanted supernatant liquid customer ID number
[b] CSol = centrifuged solids
[c] volumes below 5 mL are estimates only.

Table 3-3. Raw Data, Cascade Dissolution Tests, Core 292, "Non-Baseline" Tests. (weights in grams, volumes in mL)

	2M NaOH, 22 °C (Test E)	2M NaOH, 22 °C (Test I)	H_2O, 50 °C (Test G)
Composite Sample Wt, g	61.227	59.302	61.424
Wt Diluent Added (1st wash)	16.506	16.719	13.500
Total Volume	47.0	47.0	45.0
1st Contact Sample ID[a]	S112EC1	--	S112GC1
CSol[b] Vol	28.0	25.0	20.5
CSol Wt	52.305	47.113	40.479
Wt Diluent Added (2nd wash)	15.024	17.708	12.232
Total Volume	42.5	47.5	34.0
2nd Contact Sample ID[a]	S112EC2	--	S112GC2
CSol[b] Vol	21.0	16.5	14.0
CSol Wt	36.363	28.695	25.855
Wt Diluent Added (3rd wash)	15.458	15.849	14.424
Total Volume	35.0	31.0	28.0
3rd Contact Sample ID[a]	S112EC3	--	S112GC3
CSol[b] Vol	12.5	11.5	6.5
CSol Wt	21.925	18.766	11.190
Wt Diluent Added (4th wash)	23.429	23.656	20.916
Total Volume	33.5	33.0	27.0
4th Contact Sample ID[a]	S112EC4	--	S112GC4
CSol[b] Vol	~ 4.0	~ 3.5	~ 2.0
CSol Wt	6.736	3.838	2.072
Wt Diluent Added (5th wash)	22.537	24.644	22.229
Total Vol	25.0	26.0	24.5
5th Contact Sample ID	S112EC5	--	S112GC5
CSol Vol[c]	~ 2	~ 2	~ 1
CSol Wt	2.902	2.340	1.160
Wt Diluent Added (6th wash)	32.567	33.024	30.841
Total Vol	32.5	32.5	32.5
6th Contact Sample ID	S112EC6	--	S112GC6
CSol Vol[c]	~ 2	~ 2	~ 1
CSol Wt	2.233	1.933	1.185

[a] decanted supernatant liquid customer ID number
[b] CSol = centrifuged solids
[c] volumes below 5 mL are estimates only.

Table 3-4a. Chemical Analyses, **Test A**, Core 291, H_2O Diluent, 22 °C.
(Chemical analytes in g/L except as noted)

Analyte	H_2O Contact					
	1	2	3	4	5	6
Wt% Diln	25	50	79	117	157	208
Sample ID	S112AC1	S112AC2	S112AC3	S112AC4	S112AC5	S112AC6
S02T000...	017	030	033	036	039	043
Density (g/mL)	1.430	1.390	1.377	1.243	1.004	1.004
Al	9.83	3.31	0.88	0.11	0.016	0.015
Cr	3.36	1.30	0.39	0.037	0.002	0.003
K	0.69	0.36	<0.30	<0.20	<0.004	<0.05
Na	223	186	185	101	3.83	0.155
P	1.66	1.91	1.63	0.54	0.009	<0.02
S	6.85	8.20	2.17	0.27	0.018	<0.01
Se	0.18	0.17	0.16	0.081	0.003	<0.010
Si	0.93	0.27	0.083	<0.020	0.003	0.007
F^-	<0.12	<0.03	<0.03	0.20	<0.013	<0.001
Cl^-	1.64[a]	0.96	0.26	<0.036	<0.019	0.009
NO_2^-	14.7[a]	8.36	2.38	0.595	<0.12	0.002
NO_3^-	168[a]	301	342	232	6.41	0.137
PO_4^{3-}	3.06[a]	5.26	3.59	2.23	<0.133	0.004
SO_4^{2-}	11.5[a]	22.8	4.69	0.694	<0.153	0.024
$C_2O_4^{2-}$	0.16[a]	0.40	0.45	1.65	2.31	0.03
TIC	14.6	9.08	2.16	0.245	0.006	0.006
TOC	0.72	0.42	0.43	0.32	0.55	<0.04
OH^-	14.4	7.01	1.99	0.265	<0.042	0.045
%H_2O	54.4	49.0	52.9	70.6	98.7	100.0
MassBal	92.3	91.4	93.2	98.0	99.9	100.0
ChargeBal	1.35	1.04	1.27	1.11	1.09	0.85

[a]Apparent error in IC analysis causes IC results to be low by a factor of approximately 1.5, causing a low MassBal and anomalously high ChargeBal for this sample; PO_4^{3-}:P and SO_4^{2-}:S ratios are also anomalously low.

Table 3-4b. Radionuclide Analyses, **Test A**, Core 291, H_2O Diluent, 22 °C.
(Radionuclides in µCi/mL)

Analyte	H_2O Contact					
	1	2	3	4	5	6
Wt% Diln	25	50	79	117	157	208
^{14}C	9.5E-03	5.5E-03	1.4E-03	na[1]	na	na
^{60}Co	<3.9E-03	<5.5E-04	<2.7E-04	<2.1E-05	<2.3E-05	<2.1E-05
^{79}Se	3.0E-04	1.5E-04	4.4E-05	na	na	na
$^{89/90}Sr$	3.2E-01	1.4E+00	5.2E-01	6.0E-02	2.9E-02	3.4E-02
^{99}Tc	7.0E-02	2.6E-02	7.5E-03	<9.4E-04	<7.8E-04	<8.0E-05
^{125}Sb	<1.1E-01	<2.4E-02	<8.1E-03	<1.1E-03	<5.3E-04	<3.9E-04
^{129}I	3.5E-05	1.4E-05	<8.9E-06	na	na	na
^{137}Cs	6.5E+01	2.4E+01	6.3E+00	8.2E-01	2.0E-01	1.0E-01
^{154}Eu	<1.4E-02	<2.2E-03	<9.0E-04	<9.3E-05	<5.3E-05	<7.4E-05
^{155}Eu	<3.2E-02	<6.3E-03	<2.2E-03	<6.3E-04	<3.1E-04	<2.4E-04
^{234}U	5.2E-02	4.5E-02	2.7E-02	na	na	na
^{235}U	5.7E-02	7.2E-02	5.4E-02	na	na	na
^{238}U	4.0E+00	3.4E+00	2.2E+00	na	na	na
^{241}Am	<2.7E-02	<5.9E-03	<2.0E-03	<1.6E-03	<7.7E-04	<5.8E-04
Total Alpha	<2.2E-03	<1.4E-03	<6.2E-04	<3.0E-05	2.5E-05	7.2E-05

[1]na = not analyzed

Table 3-5a. Chemical Analyses, **Test B**, Core 292, H_2O Diluent, 22 °C.
(Chemical analytes in g/L except as noted)

Analyte	H_2O Contact					
	1	2	3	4	5	6
Wt% Diln	24	51	77	117	157	209
Sample ID	S112BC1	S112BC2	S112BC3	S112BC4	S112BC5	S112BC6
S02T000…	018	031	034	037	040	044
Density (g/mL)	1.440	1.415	1.383	1.090	1.021	1.016
Al	15.2	5.28	1.6	0.18	0.025	0.018
Cr	9.3	3.3	1.05	0.13	0.007	0.015
K	0.88	<0.30	<0.30	<0.05	<0.01	<0.001
Na	229	222	195	38.4	6.38	0.28
P	1.79	1.63	1.76	0.757	0.038	0.004
S	4.77	8.53	27.1	8.00	0.38	0.017
Se	0.18	0.17	0.17	0.034	0.005	<0.001
Si	0.48	0.17	0.07	0.011	0.005	0.012
F^-	<0.013	<0.026	0.23	0.23	<0.013	0.001
Cl^-	3.14	1.18	0.38	0.064	<0.019	0.011
NO_2^-	29.1	10.9	3.3	0.37	<0.12	0.004
NO_3^-	232	246	104	11.5	0.47	0.03
PO_4^{3-}	4.31	3.74	4.11	1.81	<0.133	0.009
SO_4^{2-}	11.1	20.1	63.5	22.6	0.91	0.044
$C_2O_4^{2-}$	0.19	0.23	0.45	7.4	8.9	0.23
TIC	11.4	18.8	24.1	4.35	0.22	<0.005
TOC	0.75	0.46	0.69	1.53	2.49	<0.04
OH^-	20.5	7.22	2.55	0.35	0.056	<0.042
$\%H_2O$	54.1	54.5	62.8	90.1	98.2	100.0
MassBal	98.8	98.7	99.1	99.7	100.0	100.1
ChargeBal	1.13	1.11	1.13	1.01	1.03	1.45

Table 3-5b. Radionuclide Analyses, **Test B**, Core 292, H_2O Diluent, 22 °C.
(Radionuclides in µCi/mL)

Analyte	H_2O Contact					
	1	2	3	4	5	6
Wt% Diln	24	51	77	117	157	209
^{14}C	7.2E-03	1.1E-02	1.1E-02	na	na	na
^{60}Co	<9.0E-03	<6.7E-04	<2.8E-04	<3.2E-05	<2.5E-05	<2.9E-05
^{79}Se	4.3E-04	2.2E-04	2.1E-04	na	na	na
$^{89/90}Sr$	1.6E-01	4.9E-01	2.0E+00	6.4E-01	7.7E-02	4.3E-01
^{99}Tc	1.1E-01	3.8E-02	1.2E-02	1.4E-03	<7.8E-04	1.1E-04
^{125}Sb	<2.0E-01	<2.8E-02	<1.1E-02	<1.4E-03	<5.4E-04	<2.8E-04
^{129}I	5.3E-05	2.3E-05	<9.0E-06	na	na	na
^{137}Cs	9.8E+01	3.4E+01	1.0E+01	1.4E+00	2.0E-01	4.9E-02
^{154}Eu	<2.9E-02	<2.1E-03	<9.6E-04	<1.3E-04	<6.2E-05	5.4E-04
^{155}Eu	<5.9E-02	<7.5E-03	<2.8E-03	<8.4E-04	<3.2E-04	<2.3E-04
^{234}U	9.2E-02	5.6E-02	0.0E+00	na	na	na
^{235}U	1.4E-01	1.4E-01	0.0E+00	na	na	na
^{238}U	1.7E+01	1.2E+01	0.0E+00	na	na	na
^{241}Am	<5.0E-02	<6.9E-03	<2.6E-03	<2.1E-03	<7.8E-04	<5.3E-04
Total Alpha	<1.3E-03	<6.7E-04	1.3E-03	3.9E-04	1.0E-04	1.7E-04

Table 3-6a. Chemical Analyses, **Test C**, Core 292, H_2O Diluent, 22 °C.
(Chemical analytes in g/L except as noted)

Analyte	H_2O Contact					
	1	2	3	4	5	6
Wt% Diln	26	49	73	108	144	192
Sample ID	S112CC1	S112CC2	S112CC3	S112CC4	S112CC5	S112CC6
S02T000…	019	032	035	038	042	045
Density (g/mL)	1.455	1.449	1.413	1.188	1.063	1.010
Al	14.3	5.78	1.98	0.31	0.037	0.021
Cr	8.6	3.6	1.3	0.22	0.02	0.004
K	0.86	<0.30	<0.30	<0.10	<0.02	0.001
Na	234	236	197	79	14.6	0.47
P	1.89	1.71	1.64	1.30	0.096	0.005
S	5.12	8.87	24.2	14.7	1.28	0.035
Se	0.19	0.19	0.16	0.066	0.013	<0.001
Si	0.45	0.33	0.08	0.016	0.005	0.015
F^-	<0.013	<0.03	<0.03	0.37	0.034	0.001
Cl^-	2.54	1.20	0.46	0.080	0.034	0.008
NO_2^-	24.2	11.1	3.98	0.58	<0.12	<0.002
NO_3^-	208	251	155	20.2	1.94	0.057
PO_4^{3-}	4.51	4.17	3.91	3.54	0.19	0.013
SO_4^{2-}	10.8	21.2	56.3	35.6	3.31	0.10
$C_2O_4^{2-}$	<0.12	0.27	0.36	3.47	15.0	0.42
TIC	12.6	18.1	21.5	11.6	1.12	0.006
TOC	0.78	0.66	0.67	0.63	4.25	0.04
OH^-	17.8	8.17	3.02	0.66	0.12	<0.042
$\%H_2O$	54.4	54.0	60.2	81.2	95.9	100.0
MassBal	97.0	98.6	98.1	98.3	99.8	100.1
ChargeBal	1.22	1.17	1.09	1.05	0.98	1.46

Table 3-6b. Radionuclide Analyses, **Test C**, Core 292, H_2O Diluent, 22 °C.
(Radionuclides in µCi/mL)

Analyte	H_2O Contact					
	1	2	3	4	5	6
Wt% Diln	26	49	73	108	144	192
^{14}C	6.0E-03	1.0E-02	1.2E-02	na	na	na
^{60}Co	<4.0E-03	<1.2E-03	<3.0E-03	<3.7E-05	<2.8E-05	<2.5E-05
^{79}Se	4.4E-04	2.3E-04	2.5E-04	na	na	na
$^{89/90}Sr$	1.3E-01	4.1E-01	2.0E+00	2.8E-01	2.1E-01	2.8E-01
^{99}Tc	9.3E-02	4.1E-02	1.6E-02	<9.0E-04	4.0E-04	<7.9E-05
^{125}Sb	<1.3E-01	<3.9E-02	<1.1E-02	<1.8E-03	<6.9E-04	<3.5E-04
^{129}I	4.8E-05	<4.1E-05	8.0E-06	na	na	na
^{137}Cs	9.1E+01	3.2E+01	1.2E+01	2.3E+00	3.3E-01	8.0E-02
^{154}Eu	<1.4E-02	<3.8E-03	<9.7E-04	<2.1E-04	<6.6E-05	3.5E-04
^{155}Eu	<3.7E-02	<1.0E-02	<3.1E-03	<1.1E-03	<4.1E-04	<2.6E-04
^{234}U	9.9E-02	6.4E-02	3.2E-02	na	na	na
^{235}U	1.7E-01	1.4E-01	1.4E-01	na	na	na
^{238}U	1.7E+01	1.4E+01	1.7E+01	na	na	na
^{241}Am	<3.1E-02	<9.5E-03	<2.8E-03	<2.7E-03	<1.0E-03	<6.0E-04
Total Alpha	<1.9E-03	<8.1E-04	7.0E-04	6.1E-04	5.6E-04	2.8E-04

Table 3-7a. Chemical Analyses, **Test D**, Core 291, 2M NaOH Diluent, 22 °C.
(Chemical analytes in g/L except as noted)

Analyte	2M NaOH Contact					
	1	2	3	4	5	6
Wt% Diln	27	54	83	129	170	226
Sample ID	S112DC1	S112DC2	S112DC3	S112DC4	S112DC5	S112DC6
S02T000...	097	098	099	100	101	102
Density (g/mL)	1.430	1.480	1.410	1.360	1.120	1.080
Al	12.9	4.2	1.3	0.22	0.13	0.07
Cr	4.3	1.5	0.74	0.34	0.03	0.01
K	0.82	0.39	0.31	0.24	0.07	0.04
Na	250	198	197	170	51	45
P	1.18	1.15	1.44	1.42	1.03	<0.02
S	3.95	5.90	6.83	0.86	0.028	<0.01
Se	0.19	0.17	0.17	0.15	0.04	0.04
Si	1.39	0.43	0.19	0.19	0.26	0.18
F^-	<0.03	<0.03	<0.03	<0.025	<0.001	<0.001
Cl^-	2.78	1.00	0.37	0.040	<0.002	<0.002
NO_2^-	25.2	8.9	3.3	0.76	0.014	<0.011
NO_3^-	212	223	276	266	4.6	0.1
PO_4^{3-}	4.7	3.4	4.6	4.2	1.5	0.1
SO_4^{2-}	13.5	16.4	19.2	2.2	0.04	<0.01
$C_2O_4^{2-}$	<0.2	<0.2	<0.2	0.5	1.5	1.5
TIC	8.7	11.6	4.2	0.60	0.06	0.03
TOC	0.64	0.47	0.16	0.15	0.68	0.37
OH^-	42.8	35.0	27.8	27.8	37.4	35.8
$\%H_2O$	55.6	55.6	55.2	58.5	89.6	91.6
MassBal	99.7	93.2	94.5	93.4	98.3	99.3
ChargeBal	1.22	1.02	1.15	1.19	0.93	0.91

Table 3-7b. Radionuclide Analyses, **Test D**, Core 291, 2M NaOH Diluent, 22 °C.
(Radionuclides in µCi/mL)

Analyte	2M NaOH Contact					
	1	2	3	4	5	6
Wt% Diln	27	54	83	129	170	226
^{14}C	7.0E-03	8.9E-03	3.4E-03	5.2E-04	3.3E-04	2.0E-04
^{60}Co	<1.8E-03	<1.8E-03	<4.2E-04	<1.6E-04	<5.6E-05	<2.5E-05
^{79}Se	<9.3E-02	<6.0E-02	<1.0E-02	<2.6E-03	<1.0E-03	<2.2E-04
$^{89/90}Sr$	2.1E-02	4.8E-01	1.4E+00	8.9E-01	4.7E-02	3.7E-02
^{99}Tc	7.4E-02	3.4E-02	8.7E-03	2.0E-03	4.1E-04	4.3E-04
^{125}Sb	<9.3E-02	<6.0E-02	<1.0E-02	<2.6E-03	<1.0E-03	<2.2E-04
^{129}I	4.3E-05	2.2E-05	6.0E-06	<4.6E-06	<5.0E-06	<4.6E-06
^{137}Cs	8.2E+01	3.4E+01	9.7E+00	1.5E+00	5.0E-01	4.0E-02
^{154}Eu	<5.7E-03	<6.2E-03	<1.5E-03	<5.7E-04	<1.7E-04	<7.9E-05
^{155}Eu	<2.3E-02	<1.5E-02	<2.6E-03	<6.6E-04	<2.6E-04	<5.9E-05
^{234}U	<2.6E-02	<2.6E-02	<2.6E-02	<2.6E-02	<2.6E-02	<2.6E-02
^{235}U	5.0E-02	5.4E-02	6.9E-02	6.7E-02	2.8E-01	2.8E-01
^{238}U	7.2E+00	7.9E+00	9.8E+00	9.9E+00	4.2E+01	4.3E+01
^{241}Am	<2.0E-02	<1.3E-02	<2.2E-03	<6.0E-04	<2.2E-04	<5.2E-05
Total Alpha	<9.0E-04	1.0E-03	9.6E-04	3.1E-04	1.5E-04	1.5E-04

Table 3-8a. Chemical Analyses, **Test E**, Core 292, 2M NaOH Diluent, 22 °C.
(Chemical analytes in g/L except as noted)

Analyte	2M NaOH Contact					
	1	2	3	4	5	6
Wt% Diln	27	52	77	115	152	205
Sample ID	S112EC1	S112EC2	S112EC3	S112EC4	S112EC5	S112EC6
S02T000…	104	105	106	107	108	109
Density (g/mL)	1.410	1.440	1.400	1.360	1.160	1.100
Al	14.3	6.6	2.5	0.71	0.26	0.12
Cr	8.7	4.0	1.6	0.48	0.10	0.02
K	0.93	0.46	<0.30	<0.30	<0.10	<0.05
Na	241	240	229	205	84	50
P	1.22	1.49	1.36	1.55	1.18	0.06
S	3.79	5.45	6.73	25.1	9.31	0.48
Se	0.20	0.20	0.19	0.19	0.07	0.04
Si	0.53	0.24	0.13	0.10	0.12	0.08
F^-	<0.03	<0.03	<0.03	0.23	0.33	<0.03
Cl^-	2.82	1.32	0.52	0.23	0.15	0.06
NO_2^-	28.2	13.1	5.0	1.3	0.28	<0.2
NO_3^-	212	234	204	53	7.2	0.4
PO_4^{3-}	3.6	5.2	4.7	3.4	2.8	<0.3
SO_4^{2-}	10.2	15.0	18.5	55.7	24.5	1.2
$C_2O_4^{2-}$	<0.2	0.23	0.40	0.36	1.7	2.8
TIC	8.5	11.0	15.6	18.8	3.6	0.21
TOC	0.93	0.53	0.46	0.44	0.51	0.75
OH^-	39.1	31.7	29.9	27.8	35.5	34.2
$\%H_2O$	53.7	54.9	57.1	66.6	84.1	90.9
MassBal	97.9	97.7	98.4	99.2	99.1	99.0
ChargeBal	1.20	1.22	1.18	1.28	1.05	1.01

Table 3-8b. Radionuclide Analyses, **Test E**, Core 292, 2M NaOH Diluent, 22 °C.
(Radionuclides in µCi/mL)

Analyte	2M NaOH Contact					
	1	2	3	4	5	6
Wt% Diln	27	52	77	115	152	205
^{14}C	6.2E-03	8.1E-03	9.6E-03	1.2E-02	2.4E-03	3.6E-04
^{60}Co	<9.0E-04	<2.7E-04	<3.2E-04	<1.0E-04	<1.0E-04	<9.0E-05
^{79}Se	3.3E-04	2.0E-04	1.4E-04	1.2E-04	6.9E-05	<2.1E-06
$^{89/90}Sr$	2.2E-01	1.6E-01	1.6E-01	1.5E+00	2.6E-01	8.0E-02
^{99}Tc	1.0E-01	4.8E-02	1.6E-02	4.7E-03	7.4E-04	1.3E-04
^{125}Sb	<6.4E-02	<2.0E-02	<1.5E-02	<4.1E-03	<1.9E-03	<9.8E-04
^{129}I	5.3E-05	2.5E-05	1.3E-05	<9.6E-06	<1.0E-05	<8.3E-06
^{137}Cs	9.5E+01	4.2E+01	1.6E+01	4.1E+00	9.2E-01	2.3E-01
^{154}Eu	<2.5E-03	<9.0E-04	<1.1E-03	<3.0E-04	<2.7E-04	<2.8E-04
^{155}Eu	<1.9E-02	<5.2E-03	<4.9E-03	<1.2E-03	<5.9E-04	<3.3E-04
^{234}U	<1.3E-02	<1.3E-02	<1.3E-02	<1.3E-02	<1.3E-02	<1.3E-02
^{235}U	1.1E-01	9.0E-02	9.0E-02	1.3E-01	2.8E-01	3.8E-01
^{238}U	1.5E+01	1.3E+01	1.3E+01	1.8E+01	4.1E+01	5.6E+01
^{241}Am	<1.7E-02	<4.4E-03	<4.1E-03	<1.1E-03	<5.0E-04	<2.8E-04
Total Alpha	2.0E-02	<1.3E-02	2.6E-04	3.8E-04	2.2E-04	9.8E-04

Table 3-9a. Chemical Analyses, **Test F**, Core 291, H_2O Diluent, 50 °C.
Dilution-Corrected (to account for H_2O added to prevent precipitation on cooling)
(Chemical analytes in g/L except as noted)

Analyte	H_2O Contact					
	1	2	3	4	5	6
Wt% Diln	25	48	73	115	155	206
Sample ID	S112FC1	S112FC2	S112FC3	S112FC4	S112FC5	S112FC6
S02T000...	123	125	127	129	131	133
Density (g/mL)	1.38	1.41	1.40	0.97	0.97	0.95
Al	7.8	2.5	0.59	0.04	0.02	0.01
Cr	2.6	0.8	0.21	0.01	0.00	0.00
K	0.63	0.32	<0.21	<0.01	0.002	0.002
Na	227	212	189	11	0.5	0.1
P	3.63	1.67	0.46	0.009	0.003	<0.0004
S	2.43	2.66	8.88	0.70	0.044	0.019
Se	0.20	0.18	0.16	0.009	0.0004	<0.0002
Si	0.61	0.21	0.07	0.009	0.006	0.004
F^-	<0.05	<0.05	<0.13	<0.03	<0.14	<0.002
Cl^-	2.02	0.64	0.36	<0.04	<0.20	0.01
NO_2^-	18.5	6.2	1.8	<0.3	<1.2	0.0
NO_3^-	335	387	414	17	13	0
PO_4^{3-}	9.6	4.5	2.0	<0.3	<1.4	0.0
SO_4^{2-}	6.3	7.9	29.8	1.7	0.00	0.05
$C_2O_4^{2-}$	<0.5	<0.5	<1.1	3.5	<1.2	<0.02
TIC	6.9	9.3	4.5	0.31	0.06	0.06
TOC	0.94	0.43	0.31	0.96	<0.09	<0.09
OH^-	16.0	5.7	1.2	<0.1	<0.1	<0.1
$\%H_2O$	nm[1]	nm[1]	nm[1]	nm[1]	nm[1]	nm[1]
MassBal[2]	96.8	96.8	98.4	99.9	100.6	100.0
ChargeBal	1.13	1.06	0.99	1.08	0.10	0.25

[1]nm – not measured on undiluted samples
[2]MassBal calculations made with %H2O and analytical results from diluted samples

Table 3-9b. Radionuclide Analyses, **Test F**, Core 291, H_2O Diluent, 50 °C. Dilution-Corrected (to account for H_2O added to prevent precipitation on cooling) (Radionuclides in µCi/mL)

Analyte	H_2O Contact					
	1	2	3	4	5	6
Wt% Diln	25	48	73	115	155	206
^{14}C	4.4E-03	6.2E-03	3.1E-03	na	na	na
^{60}Co	<4.2E-04	<1.9E-04	<2.3E-04	<5.6E-05	<5.1E-05	<5.8E-05
^{79}Se	2.7E-04	1.4E-04	5.2E-05	na	na	na
$^{89/90}Sr$	9.9E-02	2.2E-01	2.5E-01	5.8E-02	3.8E-02	6.4E-02
^{99}Tc	6.1E-02	2.0E-02	4.2E-03	3.2E-04	<1.7E-04	<1.5E-04
^{125}Sb	<3.6E-02	<1.3E-02	<6.2E-03	<9.5E-04	<8.4E-04	<4.1E-04
^{129}I	4.2E-05	<1.9E-05	<1.7E-05	na	na	na
^{137}Cs	5.9E+01	1.8E+01	4.3E+00	3.9E-01	2.9E-01	6.8E-02
^{154}Eu	<1.4E-03	<6.4E-04	<5.6E-04	<1.6E-04	<1.5E-04	<1.6E-04
^{155}Eu	<9.0E-03	<3.4E-03	<1.6E-03	<2.4E-04	<2.2E-04	<1.1E-04
^{234}U	3.6E-02	3.0E-02	<2.7E-02	na	na	na
^{235}U	6.3E-02	8.3E-02	9.5E-02	na	na	na
^{238}U	4.4E+00	4.0E-01	7.4E+00	na	na	na
^{241}Am	<7.6E-03	<2.8E-03	<1.3E-03	<2.0E-04	<1.9E-04	<9.6E-05
Total Alpha	<1.2E-03	<6.4E-04	<2.5E-04	9.5E-05	1.5E-05	<9.0E-06

Table 3-10a. Chemical Analyses, **Test G**, Core 292, H_2O Diluent, 50 °C.
Dilution-Corrected
(Chemical analytes in g/L except as noted)

Analyte	H_2O Contact					
	1	2	3	4	5	6
Wt% Diln	22	45	71	107	148	203
Sample ID	S112GC1	S112GC2	S112GC3	S112GC4	S112GC5	S112GC6
S02T000…	124	126	128	130	132	134
Density (g/mL)	1.37	1.34	1.32	1.12	1.02	0.95
Al	12.3	5.0	1.7	0.26	0.028	0.020
Cr	7.4	3.1	1.1	0.21	0.024	0.002
K	0.87	0.49	<0.21	<0.11	<0.01	0.002
Na	218	189	176	87	11	0.45
P	2.88	2.31	0.78	0.13	<0.004	0.001
S	1.87	4.92	14.0	23.5	1.07	0.050
Se	0.18	0.16	0.15	0.069	0.009	0.0005
Si	0.38	0.18	0.07	0.015	0.007	0.012
F^-	0.49	0.81	<0.13	<0.05	<0.01	<0.002
Cl^-	2.83	1.52	0.44	0.000	0.03	0.01
NO_2^-	29.0	12.0	3.5	0.5	<0.1	<0.02
NO_3^-	334	267	94	13	0.62	0.04
PO_4^{3-}	7.6	7.7	1.9	0.56	<0.2	<0.03
SO_4^{2-}	5.9	14.4	48.0	7.6	2.6	0.12
$C_2O_4^{2-}$	<0.5	<1.1	<1.1	4.5	11.6	<0.02
TIC	5.7	16.1	31.2	12.1	0.72	0.07
TOC	0.98	0.58	0.50	1.08	3.32	0.15
OH^-	20.3	8.2	2.8	0.64	<0.1	<0.1
$\%H_2O$	nm[1]	nm[1]	nm[1]	nm[1]	nm[1]	nm[1]
MassBal[2]	97.3	99.0	99.8	97.1	100.0	100.1
ChargeBal	1.04	0.96	0.94	1.47	1.07	0.86

[1]nm – not measured on undiluted samples
[2]MassBal calculations made with %H2O and analytical results from diluted samples

Table 3-10b. Radionuclide Analyses, **Test G**, Core 292, H_2O Diluent, 50 °C.
Dilution-Corrected
(Radionuclides in µCi/mL)

Analyte	H_2O Contact					
	1	2	3	4	5	6
Wt% Diln	22	45	71	107	148	203
^{14}C	4.0E-03	9.6E-03	1.8E-02	na	na	na
^{60}Co	<4.4E-04	<2.9E-04	<4.2E-04	<2.3E-04	<7.5E-05	<8.2E-05
^{79}Se	4.0E-04	3.1E-04	1.8E-04	na	na	na
$^{89/90}Sr$	7.9E-02	3.3E-01	1.3E+00	1.8E+00	8.8E-02	1.5E-01
^{99}Tc	9.9E-02	4.0E-02	1.3E-02	2.1E-03	<1.5E-04	<1.5E-04
^{125}Sb	<4.6E-02	<1.8E-02	<1.1E-02	<2.1E-03	<8.6E-04	<5.0E-04
^{129}I	5.7E-05	<3.1E-05	<1.5E-05	na	na	na
^{137}Cs	9.0E+01	3.6E+01	1.2E+01	2.0E+00	3.2E-01	8.6E-02
^{154}Eu	<1.8E-03	<9.4E-04	<1.3E-03	<7.4E-04	<2.1E-04	<2.5E-04
^{155}Eu	<1.1E-02	<4.4E-03	<2.7E-03	<5.9E-04	<2.1E-04	<1.4E-04
^{234}U	5.9E-02	<2.7E-02	3.6E-02	na	na	na
^{235}U	1.2E-01	7.5E-02	2.6E-01	na	na	na
^{238}U	1.5E+01	9.6E+00	3.4E+01	na	na	na
^{241}Am	<8.8E-03	<3.8E-03	<2.3E-03	<5.3E-04	<1.9E-04	<1.3E-04
Total Alpha	<2.2E-03	<8.3E-04	5.3E-04	6.7E-03	4.7E-04	5.2E-05

The residual solids (solids left undissolved after the 6th contact) from Tests A, D, and F, all from Core 291, were combined into a single analytical sample labeled S112ASOL. Similarly, the residual solids from the Core 292 Tests B, C, E, G, H, and I were combined into a single analytical sample labeled S112ESOL. Analytical results are shown in Tables 3-11a (chemicals) and 3-11b (radionuclides). The relatively high value for TOC in Core 292 reflects the presence of undissolved sodium oxalate stemming mainly from the poor dissolution in the two tests (H and I) with 2 M NaOH as the diluent. Other limitations of the accuracy of the analytical results for the residual solids are discussed in Section 5.2, where evidence is presented that the results shown in Tables 3-11a and 3-11b are anomalously low.

Table 3-11a. Chemical Analyses, Combined Residual Solids.
(Chemical analytes in mg/g except as noted)

Analyte	Core 291	Core 292
Sample ID	S112ASOL	S112ESOL
S02T000...	341-343	111-113
Al	15.0	17.6
Ca	6.0	9.1
Cd	0.1	0.4
Cr	26.6	41.8
Fe	4.6	15.3
K	na	na
Li	1.3	< 0.2
Mn	1.7	4.8
Na	8.3	37.1
P	< 5.0	< 4.5
S	< 2.5	< 2.3
Si	3.8	4.7
U	11.1	39.2
Zn	0.6	0.4
TIC	2.1	2.0
TOC	0.6	12.4
$\%H_2O$	85.2	48.8

Table 3-11b. Radionuclide Analyses, Combined Residual Solids.
(Radionuclides in μCi/g except as noted)

Analyte	Core 291	Core 292
^{14}C	na	4.3E-03
^{60}Co	< 1.4	< 1.6
^{79}Se	< 1.5E-03	< 1.3E-03
$^{89/90}Sr$	930	863
^{99}Tc	< 4.2E-02	1.8E-01
^{125}Sb	< 3.6	< 4.0
^{129}I	na	< 5.3E-04
^{137}Cs	< 2.2	8.1
^{154}Eu	< 4.4	< 5.2
^{155}Eu	< 1.7	< 2.0
^{234}U (μg/g)	< 2.9	< 2.7
^{235}U (μg/g)	67	195
^{238}U (μg/g)	9,740	28,700
^{241}Am	< 1.6	< 1.9
Total Alpha	0.88	2.70

3.2 FEED STABILITY TESTS

The **Feed Stability** test procedure was described in Section 2.3. Weights of diluent added and residual solids after each contact are shown in Table 3-2 (Test H) and Table 3-3 (Test I). Liquid phase compositions for Test H are assumed to be similar to those in Test B (Table 3-5); compositions for Test I are assumed to be similar to those in Test E (Table 3-8).

The only time that significant solids formation occurred in the receiver jar was in Test H (water diluent) following the fifth contact. The solids were identified as sodium oxalate.

3.3 Kinetics Tests

The **Kinetics** test procedure was described in Section 2.5. Sample weight (Table 3-12) was determined by weighing the entire test apparatus before and after adding sample to the filter. Weight of water added was determined by weighing the entire apparatus before and after water addition. Weight of filtrate was determined by weighing the filtrate collection vial before and after the test. Weight of sample dissolved is the difference between the filtrate weight and weight of water added. Weight percent (Wt%) dissolved is 100 times the weight of sample dissolved divided by the original composite sample weight.

Table 3-12. Kinetics Test Weight Measurements (in grams)

time, minutes →	0.5	2	5	5	23	60
Surface Dissolution						
Composite Sample	7.21	7.69	no data; vacuum problem	7.88	9.21	8.77
Water Added	9.38	9.51		9.11	9.51	9.81
Filtrate	11.28	11.96		11.63	14.57	15.15
Sample Dissolved	1.90	2.45		2.52	5.06	5.34
Wt% Dissolved	26	32		32	55	61
Surface Dissolution, Duplicate Tests						
Composite Sample	9.43	7.95	8.01	7.77	9.00	8.46
Water Added	9.16	9.64	9.03	9.07	9.39	9.91
Filtrate	11.59	12.41	11.89	11.58	13.62	15.12
Sample Dissolved	2.43	2.77	2.86	2.51	4.23	5.21
Wt% Dissolved	26	35	36	32	47	62
Percolation Dissolution				Control A (3-day mix)		
Composite Sample	8.18	no data; filter plugged	no data; water delivery problem	19.74		
Water Added	8.90			23.42		
Filtrate	11.17			42.10[a]		
Sample Dissolved	2.27			18.68		
Wt% Dissolved	28			94.6		
Percolation Dissolution, Duplicate Tests				Control B (3-day mix)		
Composite Sample	8.36	8.36	8.03	18.00		
Water Added	9.16	9.03	9.28	22.54		
Filtrate	11.06	11.60	12.80	39.58[a]		
Sample Dissolved	1.90	2.57	3.52	17.04		
Wt% Dissolved	23	31	44	94.7		

[a]Controls were centrifuged rather than filtered. "Filtrate", in this case, means weight of supernatant liquid decanted after centrifuging.

Sample weight (actual 8.3 ±1.1 g) and water weight (9.4 ±0.5 g) were intended to be relatively constant, with the only variable being the time allowed for the water addition. Water addition time is not the same as contact time, the latter being much shorter and relatively constant from one run to another. During the surface dissolution tests, for example, the filter was under constant vacuum (except during the 60 minute tests, during which the vacuum was turned on for one minute out of every five minutes). The water was dripped onto the upper edge of the slanted sample, and as soon as the dissolved brine reached the filter frit, it was sucked into the receiver vial.

The original set of tests included the surface dissolution and percolation tests at 0.5 min, 2 min, and 5 min, all in duplicate (left half of Table 3-12). There was no clear difference in results between the two types of tests. Indeed, despite the slope of the sample in the surface dissolution test, the water still appeared to penetrate the salt bed more than it ran across the surface, so it was not surprising that the results were similar.

The second set of tests (right half of Table 3-12) was added because of the incomplete dissolution at the short times of the first set of tests. The second set was done using only the surface dissolution method, to prevent the filter plugging problem encountered during the percolation tests. The 5 min water addition was repeated, followed by water additions lasting 23 min and 60 min.

Analytical data from the collected filtrate solutions from the surface dissolution tests are shown in Table 3-13. Corresponding data for the percolation tests and the control samples are shown in Table 3-14. These data provide a second way to evaluate the rate of approach to equilibrium (the first being the Wt% Dissolved values shown in Table 3-12). In Tables 3-15 and 3-16, the analytical data for each test are expressed in terms of a percentage of the average value for the two control samples, which represent "infinite" mixing and contact time, and are assumed to have reached equilibrium. A value of 100% would indicate that the sample at water addition time 't' had reached equilibrium (i.e., the sample had dissolved as much as possible for the amount of water added). A lower value serves as a "percent dissolved" for each individual analyte. The Wt% Dissolved values from Table 3-12 are repeated here for comparison.

Some general observations about the analytical results are noteworthy. With a few exceptions, the percent dissolved is relatively constant for all components, i.e, most of the components are very close to the Average percent dissolved near the bottom of Tables 3-15 and 3-16. The exceptions are noted in the footnotes below the tables.

The first exception, nitrite, is consistently higher than the average. This appears to be due to an anomalously low value for nitrite in the control samples. In comparing the control sample analytical results (Table 3-14a) with the Test A results (Table 3-4a), the somewhat coincidental agreement of the control average with Test A at 50% dilution is rather striking. In fact, Cl^-, NO_3^-, Al, Cr, Na, Se, and Si all match within 10% between the two sets of data. The species that don't match include NO_2^-, PO_4^{3-}, SO_4^{2-}, P, and S, all of which are significantly lower in the control samples than in Test A at 50% dilution. This could explain the discrepancies for nitrite and [see below] S/SO_4^{2-}, but not P/PO_4^{3-}.

Table 3-13a. Analytical Results (g/L) for Surface Dissolution Tests

time →	0.5 min	2 min	5 min	5 min	23 min	60 min
S02T000…	535	534	no data; vacuum problem	1079	1081	1083
Cl^-	0.45	0.50		0.44	0.67	0.63
NO_2^-	2.33	3.05		3.37	5.32	5.15
NO_3^-	117	152		156	236	214
PO_4^{3-}	< 1.2	< 1.2		0.93	1.62	1.90
SO_4^{2-}	4.28	5.93		6.40	10.6	10.7
Al	1.34	1.54		1.61	2.50	2.58
Cr	0.49	0.54		0.57	0.89	0.92
Na	68.2	83.3		86.0	135	142
P	0.32	0.35		0.36	0.59	0.75
S	1.89	2.33		2.32	4.02	4.20
Se	0.06	0.07		0.07	0.11	0.11
Si	0.12	< 0.2		< 0.2	0.21	0.22

Table 3-13b. Analytical Results (g/L) for Surface Dissolution, Duplicate Tests

time →	0.5 min	2 min	5 min	5 min	23 min	60 min
S02T000…	538	537	536	1080	1082	1084
Cl^-	0.42	0.51	0.61	0.38	0.48	0.64
NO_2^-	2.58	3.15	3.52	2.92	3.69	4.87
NO_3^-	139	159	164	157	214	233
PO_4^{3-}	< 1.2	< 1.2	< 1.2	1.14	1.53	2.05
SO_4^{2-}	5.35	5.79	6.35	6.86	9.41	10.3
Al	1.52	1.65	1.98	1.58	2.09	2.36
Cr	0.55	0.60	0.72	0.56	0.74	0.85
Na	79.6	84.2	96.4	88.6	126	132
P	0.35	0.41	0.44	0.40	0.57	0.73
S	2.29	2.30	2.77	2.63	3.68	3.69
Se	0.07	0.08	0.08	0.08	0.11	0.11
Si	0.14	0.15	0.18	0.13	0.18	0.20

Table 3-14a. Analytical Results (g/L) for Percolation Dissolution Tests and Control Samples (representing "infinite" time)

time →	0.5 min	2 min	5 min	Control-A	Control-B	Average
S02T000…	541	no data; filter plugged	no data; water delivery problem	545	546	(545+546)/2
Cl^-	0.41			1.13	0.81	0.97
NO_2^-	3.15			6.08	4.80	5.44
NO_3^-	127			305	287	296
PO_4^{3-}	0.88			4.06	3.72	3.89
SO_4^{2-}	4.44			10.8	10.9	10.85
Al	1.58			3.63	3.09	3.36
Cr	0.55			1.31	1.15	1.23
Na	69.4			189	192	190.5
P	0.34			1.58	1.64	1.61
S	1.76			4.79	5.35	5.07
Se	0.06			0.16	0.16	0.16
Si	0.14			0.26	0.26	0.26

Table 3-14b. Analytical Results (g/L) for Percolation Dissolution, Duplicate Tests

time →	0.5 min	2 min	5 min
S02T000…	544	543	542
Cl^-	0.36	0.48	0.58
NO_2^-	2.52	3.63	4.49
NO_3^-	107	149	174
PO_4^{3-}	0.80	1.01	1.18
SO_4^{2-}	3.83	5.45	6.56
Al	1.24	1.66	2.17
Cr	0.42	0.56	0.74
Na	58.2	79.7	96.9
P	0.28	0.38	0.44
S	1.42	2.09	2.59
Se	0.06	0.07	0.08
Si	0.08	0.13	0.17

Table 3-15a. Percent Dissolved by Component, Surface Dissolution Tests

time →	0.5 min	2 min	5 min	5 min	23 min	60 min
Cl^-	47	52	no data; vacuum problem	45	69	65
NO_2^-	43	56		62	98	95
NO_3^-	40	51		53	80	79
PO_4^{3-}	< 31	< 31		24	42	49
SO_4^{2-}	39	55		59	98	99
Al	40	46		48	74	77
Cr	40	44		46	72	75
Na	36	44		45	71	75
P	20	22		22	36	47
S	37	46		46	79	83
Se	38	43		43	69	70
Si	49	< 78		< 78	82	84
Average[1]	40	47		47	74	75
Wt%[2]	26	32		32	55	61

[1]Average percent dissolved, not including NO_2^-, PO_4^{3-}, SO_4^{2-}, P, or S.
[2]Wt% Dissolved from Table 3-12.

Table 3-15b. Percent Dissolved by Component, Surface Dissolution, Duplicate Tests

time →	0.5 min	2 min	5 min	5 min	23 min	60 min
Cl^-	44	53	63	40	49	66
NO_2^-	47	58	65	54	68	90
NO_3^-	47	54	55	53	72	79
PO_4^{3-}	< 31	< 31	< 31	29	39	53
SO_4^{2-}	53	53	59	63	87	95
Al	49	49	59	47	62	70
Cr	49	49	59	46	60	69
Na	44	44	51	47	66	69
P	25	25	27	25	35	45
S	45	45	55	52	73	73
Se	48	48	51	48	69	69
Si	48	48	51	50	70	80
Average[1]	45	49	56	47	65	71
Wt%[2]	26	35	36	32	47	62

[1]Average percent dissolved, not including NO_2^-, PO_4^{3-}, SO_4^{2-}, P, or S.
[2]Wt% Dissolved from Table 3-12.

Table 3-16. Percent Dissolved by Component, Percolation Dissolution Tests

time →	0.5 min	0.5 min dup	2 min	2 min dup	5 min	5 min dup
Cl^-	42	37	no data; filter plugged	49	no data; water delivery problem	60
NO_2^-	58	46		67		83
NO_3^-	43	36		50		59
PO_4^{3-}	23	21		26		30
SO_4^{2-}	41	35		50		60
Al	47	37		49		65
Cr	44	34		46		60
Na	36	31		42		51
P	21	18		24		27
S	35	28		41		51
Se	38	34		42		51
Si	54	34		42		51
Average[1]	41	34		46		57
Wt%[2]	28	23		31		44

[1]Average percent dissolved, not including NO_2^-, PO_4^{3-}, SO_4^{2-}, P, or S.
[2]Wt% Dissolved from Table 3-12.

The same discrepancy noted between the control samples and Test A at 50% dilution accounts for the consistently higher-than-average percent dissolved values for sulfate, as noted above, and to a lesser extent, sulfur. The same argument does **not** hold for phosphate and phosphorus, in fact the reverse is true. Both PO_4^{3-} and P are consistently much lower than the average percent dissolved in spite of the fact that the control sample concentrations are lower than the corresponding Test A concentrations at 50% dilution. Phosphate appears to be the only component in the saltcake that truly dissolves at a different rate, i.e. more slowly, than all other components.

The average percent dissolved based on analytical data for the individual components is invariably higher than the percent dissolved based on overall weight data (last row in Tables 3-15 and 3-16). The difference between the two values decreases with time. The ratio is highest at 0.5 min (45/26 = 1.7 in Table 3-15b) and lowest at 60 min (71/62 = 1.1). Recall that the weight of sample dissolved is calculated indirectly from the difference between the weight of water added and the weight of filtrate recovered. Any liquid phase trapped in the filter frit or within the sample itself is not accounted for. Thus, the percentages dissolved based on the analytical results are assumed to be more accurate.

3.4 Rheology Tests

The procedure for the rheology tests for the composite sample representing a mixture of Core 291 and Core 292 saltcake was described in Section 2.6. The contact and protracted mixing-equilibration of the saltcake with 60% of its weight of water at 50 °C converted 63% of the saltcake into decantable supernate and fines. The supernate and fines rendered ~12 bulk volume percent of slowly settled fines after cooling to 20 °C and diluting 20% by volume with water. A second contact and mixing with 85% of original sample weight of water at 50 °C left only 3% of the original solids by weight and a supernate which, after cooling to 20 °C and diluting 19% by volume with water, had essentially no centrifugable or settleable solids.

Within the sensitivity of the rheometer, the diluted supernates both exhibited simple Newtonian rheology, though the more dilute supernate was too fluid to permit good measurements at temperatures above ambient. This sample yielded shear rheology curves below the nominal sensitivity limit of the instrument used to measure them. Furthermore, both samples suffered from unwanted turbulence in the sensor that reduces the amount of useable data at the higher shear rates. Nonetheless, the samples generally displayed rheologies having the expected trends with temperature and solids loadings. Both samples evidenced simple Newtonian behavior with viscosities less than 10 mPa-s (cP) at every temperature measured. The results are presented in Table 3-17, along with sample identifications and available data file identifications. Appendix A contains plots of shear rate ($\dot{\gamma}$) *vs.* time, temperature *vs.* time, shear stress (τ, Pa) *vs.* shear rate ($\dot{\gamma}$), apparent viscosity (η, or $\tau/\dot{\gamma}$ at $\dot{\gamma}$, mPa-s) *vs.* shear rate ($\dot{\gamma}$), and "instantaneous" viscosity ($d\tau/d\dot{\gamma}$ at $\dot{\gamma}$, -s) *vs.* shear rate ($\dot{\gamma}$) for each of the measurements. The latter plot type is mislabeled "$\eta(t)$" in the attachment.

When viewing Table 3-17 and Appendix A, it is important to keep in mind that the MV1 sensor's recommended dynamic range is bounded by shear stresses between 3.22 and 322 Pa. Also, samples of low viscosity are prone to entering a turbulent flow regime within the sensor at the higher shear rates, which invalidates the data taken there, since the method assumes laminar flow. The turbulence manifests itself as a discontinuous increase in the slope of shear stress as a function of shear rate caused by the increased drag of turbulent flow. Furthermore, operation at very low shear stress accentuates the artificial effects of mechanical torque signal measurements; including nonzero shear stress vs. shear rate intercepts and, thereby, distorted apparent viscosities at low shear stress. Finally, these samples consist of aqueous solutions and suspensions of solids; therefore, derived viscosities below that of water at the corresponding temperature would not be credible. The instrument has no special ability to maintain a suspension that may tend to settle over an hour's time frame. Settling could progressively affect the higher temperature measurements, because they are taken towards the end of the sample analysis, and the suspending medium is less viscous. The results are based on measurements close to minimum instrument sensitivity and should be examined for all these effects.

Table 3-17. Tank S-112 Dissolution Test Sample Identifications and Rheologies.

Sample I. D.	Description	T (°C)	Estimated Shear Rheological Parameter(s) ± Std. Error	Data File Name	Fit Range/Comment
V1L and V2L	1st Contact at 50°C, decanted, cooled to 20°C, diluted 20%: Slurry with ~12% bulk solids, 1.36 g/mL bulk density	20.0	Newtonian Viscosity: 5.739 ± 0.056 mPa-s	V1LV2L20	Laminar, linear τ from 50 up to 800 down to 50 s^{-1}
		35.0	Newtonian Viscosity: 3.959 ± 0.065 mPa-s	V1LV2L35	Laminar, linear τ from 50 up to 600 down to 50 s^{-1}
		50.1	Newtonian Viscosity: 2.90 ± 0.13 mPa-s	V1LV2L50	Laminar, linear τ from 50 up to 350 down to 50 s^{-1}
		20.0	Newtonian Viscosity. 6.257 ± 0.084 mPa-s	RERUN20	Rerun at 20°C after all others, approx. 3 hours. Laminar, linear τ from 50 up to 900, shifting slope down to 50 s^{-1}. Possible settling.
		20.0	Weighted Average two runs at 20°C 5.90 ± 0.24 mPa-s.	N/A	
V1S and V2S	2nd Contact at 50°C, decanted, cooled to 20°C, diluted 19%: Liquid with ~0% bulk solids, 1.15 g/mL bulk density	20.0	Newtonian Viscosity: 2.00 ± 0.18 mPa-s	V1SV2S20	Laminar, linear τ from 50 up to 250 down to 50 s^{-1}
		20.0	Newtonian Viscosity: 1.88 ± 0.16 mPa-s	RERUNS20	Re-zeroed and ran again. Laminar, linear τ from 50 up to 300 down to 50 s^{-1}.
		35.1	Newtonian Viscosity: 1.76 ± 0.31 mPa-s	V1SV2S35	Laminar, linear τ from 50 up to 200 down to 50 s^{-1}
		50.1	Newtonian Viscosity: 1.41 ± 0.57 mPa-s	V1SV2S50	Laminar, linear τ from 50 up to 150 down to 50 s^{-1}
		20.0	Newtonian Viscosity: 1.60 ± 0.13 mPa-s	RE2S20	Rerun at 20°C again, Laminar, linear τ from 50 up to 300 down to 50 s^{-1}
		20.0	Newtonian Viscosity: 1.79 ± 0.13 mPa-s	DUPS20	Fresh aliquot. Laminar, linear τ from 50 up to 300 down to 50 s^{-1}
		20.0	Weighted Average four runs at 20°C 1.781 ± 0.083 mPa-s	N/A	

The attached plots of data were examined for each measurement, and the ranges of valid τ vs. $\dot{\gamma}$ data indicated in Table 3-17 were then fitted by appropriate rheological response functions. In this study, both samples were interpreted to have given linear responses at all temperatures that suffered only from the imperfect torque-zeroing capability of the instrument, and imperfect stress measuring capability below approximately 0.2 Pa or 50 s^{-1}. Only the downward scan of the V1L-V2L sample rerun at 20 °C (file name RERUN20) exhibited a disagreement with the upward scan. Hence, the apparent intercepts of the τ axis were not taken to indicate genuine yield strengths, but rather, the true responses were assumed to be simple Newtonian in nature. A straight-line function was fitted to the valid data, and the slope was assigned to a constant Newtonian viscosity given in Table 3-17 together with its associated standard error. Note that only the 20 °C runs of the V1L-V2L sample managed to build up the minimally recommended amount of shear stress for a good measurement before suffering invalidating turbulence, and that not a single measurement of the V1S-V2S sample managed to. Nonetheless, sample responses were sufficiently clear to establish the expected general trends with solids content and temperature.

In fact, the V1L-V2L sample was viscous enough to have its Arrhenius temperature functionality extracted by fitting the results of Table 3-17 weighted by their standard errors to an Arrhenius function, as shown in Figure 3-1. Figure 3-11 displays the Arrhenius fitting function, the fitted Arrhenius parameters and their standard errors. Also plotted are the measurements for the V1S-V2S sample. These latter results were too close to minimum detectibility for extracting Arrhenius parameters. The known viscosity of water is plotted in the figure for comparison. Figure 3-11 indicates that the ability to measure Newtonian viscosity disappears into noise as the viscosity approaches 1½ mPa-s.

The successfully fitted Arrhenius function for the 1st contact supernate can be used to estimate its viscosity as a function of temperature. The recommended interpolating function is given in Table 3-18.

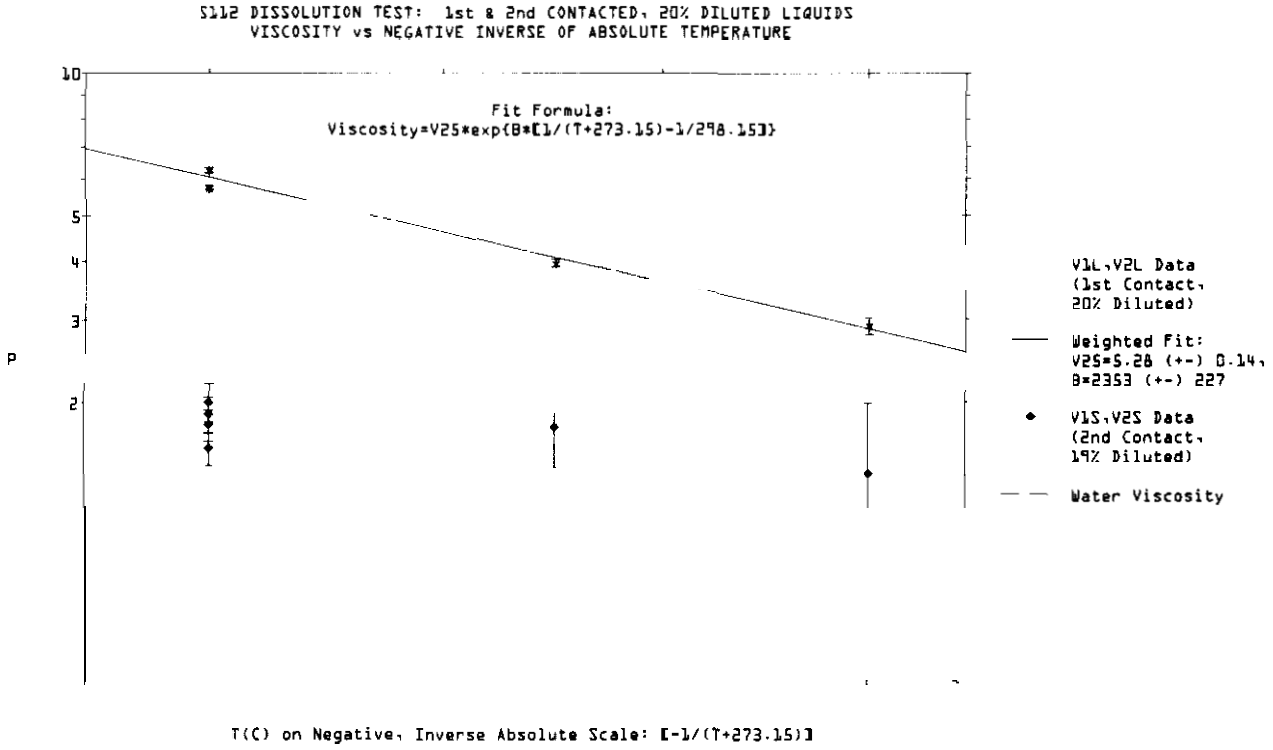


Figure 3-1. Reverse Arrhenius Plot of Viscosity Results

Table 3-18. Rheology Interpolating Functions.

SAMPLE	ARAMETER	INTERPOLATION FORMULA
V1L-V2L (1st Contact, diluted 20%)	Viscosity η (mPa-s)	$\eta(T) = 5.28 \times e^{2353\left(\frac{1}{T+273.15} - \frac{1}{298.15}\right)}$
V1S-V2S	Viscosity η (mPa-s)	Not developed

In conclusion, Newtonian viscosity value estimates less than 10 mPa-s were found for both samples at all temperatures, despite the difficulties of working at the lower limit of instrument sensitivity. The temperature dependence of the more concentrated, solids-loaded sample was found to have the expected form. Due to the aqueous, non-polymeric nature of the samples, it is recommended that the viscosities be assumed to describe their rheological flow behavior at the higher shear rates not measurable due to instrumental turbulence.

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 centrifuged solids remaining after each dissolution contact.

The weight of centrifuged solids relative to the original sample weight gives the "Wt% CSol" values presented in Tables 4-1 (core 291) and 4-2 (core 292). The cumulative "Wt% Dilution" is the sum of the weights of water or 2 M NaOH added relative to the original sample weight.

The dissolution data for Core 291 Tests A, D, and F are shown in Figure 4-1. The data are consistent with what would be expected for a sample made up mainly of sodium nitrate. Relative to the baseline conditions of Test A (water diluent, 22 °C), an increase in temperature causes more efficient dissolution, i.e., more dissolution with less water added. Similarly, changing the solvent from water to 2 M NaOH causes less efficient dissolution.

Analogous data for Core 292 Tests B, E, and G are shown in Figure 4-2. This core contains significant amounts of sodium carbonate and sulfate as well as nitrate. The effect is that temperature has very little effect on dissolution, due to the retrograde solubility of carbonate and sulfate (see Section 5). The decrease in dissolution with 2 M NaOH is again evident.

Table 4-1. Weight Percent Dissolution Profiles, Core 291
[all values in Wt% (weight percent); CSol = centrifuged solids]

Contact	H_2O, 22 °C, Test A		2M NaOH, 22 °C, Test D		H_2O, 50 °C, Test F	
	Dilution	CSol	Dilution	CSol	Dilution	CSol
1	25	92	27	99	25	74
2	50	59	54	70	48	39
3	79	22	83	38	73	3.2
4	117	1.7	129	2.6	115	1.6
5	157	1.1	170	1.0	155	1.8
6	208	1.2	226	0.7	206	1.8

Table 4-2. Weight Percent Dissolution Profiles, Core 292
[all values in Wt% (weight percent); CSol = centrifuged solids]

Contact	H_2O, 22 °C, Tests B/C/H		2M NaOH, 22 °C, Tests E/I		H_2O, 50 °C, Test G	
	Dilution	CSol	Dilution	CSol	Dilution	CSol
1	24 26 26	72 73 80	27 28	85 79	22	66
2	51 49 52	36 42 60	51 58	59 48	45	43
3	77 73 78	11 21 16	77 85	36 32	71	19
4	117 108 117	3.2 5.5 4.5	115 125	11 6.5	107	3.5
5	157 144 155	1.5 1.5 1.7	152 166	4.7 3.9	148	2.0
6	209 192 204	1.6 1.6 1.7	205 222	3.6 3.3	203	1.9

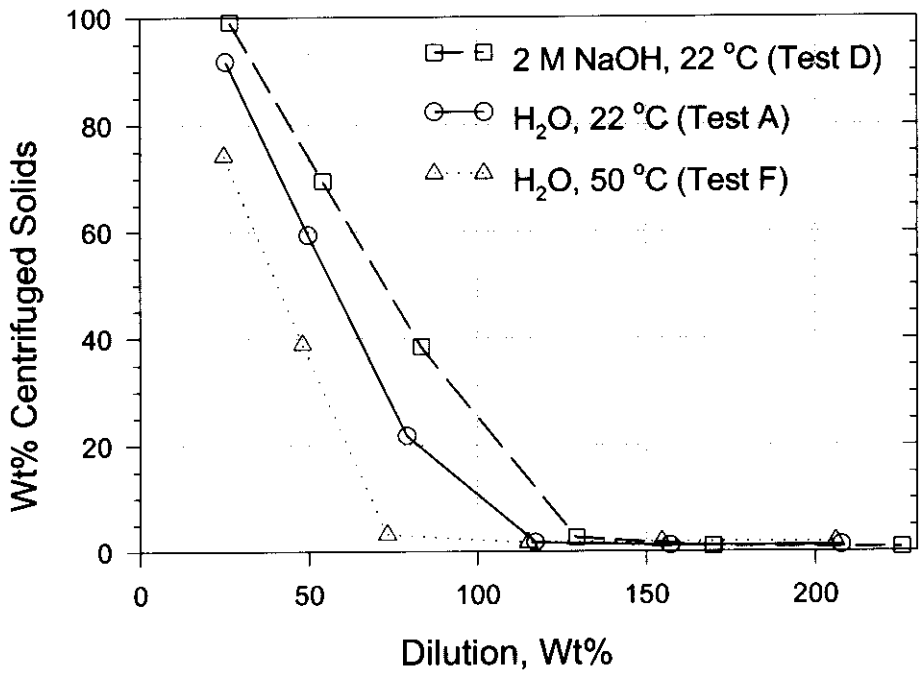


Figure 4-1. Tank S-112 Centrifuged Solids, Core 291

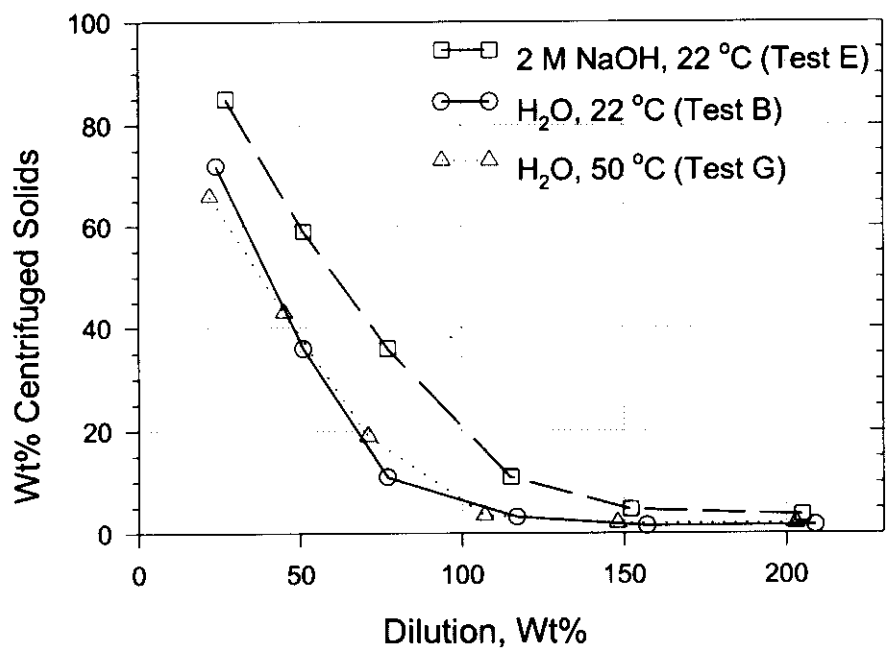


Figure 4-2. Tank S-112 Centrifuged Solids, Core 292

5.0 CHEMICAL COMPOSITION TRENDS

The gross sample behavior explored in the previous section (i.e., weight 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 core was determined by direct analysis of the solid plus water-digest and fusion-digest preparations. The results [Prilucik 2002] are summarized in Tables 5-1.

Each of the two core samples has its unique characteristics. Core 291 saltcake is high in nitrate and unremarkable in all other anions, and is very dry. Core 292 saltcake is relatively low in nitrate and high in carbonate and sulfate, and is wet enough to have a separable liquid phase.

5.1 LIQUID PHASE COMPOSITION TRENDS

Figure 5-1 (Core 291) and Figure 5-2 (Core 292) show the dissolution behaviors of the major analytes of interest. These are plots of the analytical data from Tables 3-4 and 3-9 for Core 291, and Tables 3-5 and 3-10 for Core 292.

5.1.1 Nitrate

In the case of nitrate, the most abundant anion by weight in both cores, Figures 5-1 and 5-2 both show the increase in nitrate solubility with temperature during the early part of the dissolution. (The filled circles are higher than the open circles at low wt% dilution.) In Core 291, Figure 5-1, the increase in solubility results in earlier exhaustion of the nitrate. At 50 °C, the nitrate is effectively gone at all dilutions above 100 wt%, compared to 150 wt% at 22 °C. This difference is not very pronounced in Core 292, which is much lower in total nitrate than Core 291.

Figure 5-1 clearly shows the increase in solubility at both temperatures with increasing dilution during the early stages, an effect caused by the common ion effect, or LeChatelier's Principle. As the dilution level increases, the concentrations of the fully-soluble sodium salts (hydroxide, nitrite, chloride, aluminate) decrease, allowing the solubility of the nitrate to increase. This is seen in Figure 5-2 as well, but only at 22 °C. At the higher temperature, there isn't enough nitrate in the sample to saturate the solution at any dilution level above 25 wt%, so a simple dilution curve is obtained.

Table 5-1. Overall Composition (in wt%) of Composite Saltcake Samples from [Prilucik 2002]

	Core 291		Core 292	
	Fusion Digest	Acid Digest	Fusion Digest	Acid Digest
Al	0.46	0.45	0.80	0.78
Cr	0.20	0.19	0.54	0.53
Fe	< 0.010	0.009	< 0.010	0.055
K	--	< 0.040	--	< 0.040
Na	25.7	27.1	25.5	25.7
P	< 0.40	0.15	< 0.40	0.20
S	0.70	0.72	1.75	1.83
Si	0.14	0.03	0.13	0.02
U	< 0.50	< 0.02	< 0.50	0.04
	Water Digest		Water Digest	
F^-	< 0.012		0.018	
Cl^-	0.15		0.18	
NO_2^-	0.99		1.56	
NO_3^-	51.0		26.9	
PO_4^{3-}	0.48		0.58	
SO_4^{2-}	1.93		5.18	
$C_2O_4^{2-}$	0.26		0.78	
OH^-	0.95		1.16	
	Direct		Direct	
CO_3^{2-}	4.88		12.2	
H_2O	8.7		17.0	
Mass Balance	98.1		94.3	
Charge Balance	1.03		0.99	
Radionuclides in μCi/g (fusion digest except where noted)				
^{14}C (water)	0.005		0.016	
^{60}Co	< 0.015		0.017	
^{79}Se (acid)	0.0003		0.0005	
$^{89/90}Sr$	17.0		32.0	
^{99}Tc	0.035		0.063	
^{125}Sb	< 0.14		< 0.18	
^{137}Cs	32.8		56.4	
^{154}Eu	< 0.043		< 0.056	
^{155}Eu	< 0.037		< 0.047	
^{241}Am	< 0.033		< 0.042	
Total Alpha	0.034		0.071	

Figure 5-1. Concentrations in Supernatant Liquid, Core 291, H_2O Diluent
(open symbols 22 °C, filled symbols 50 °C)

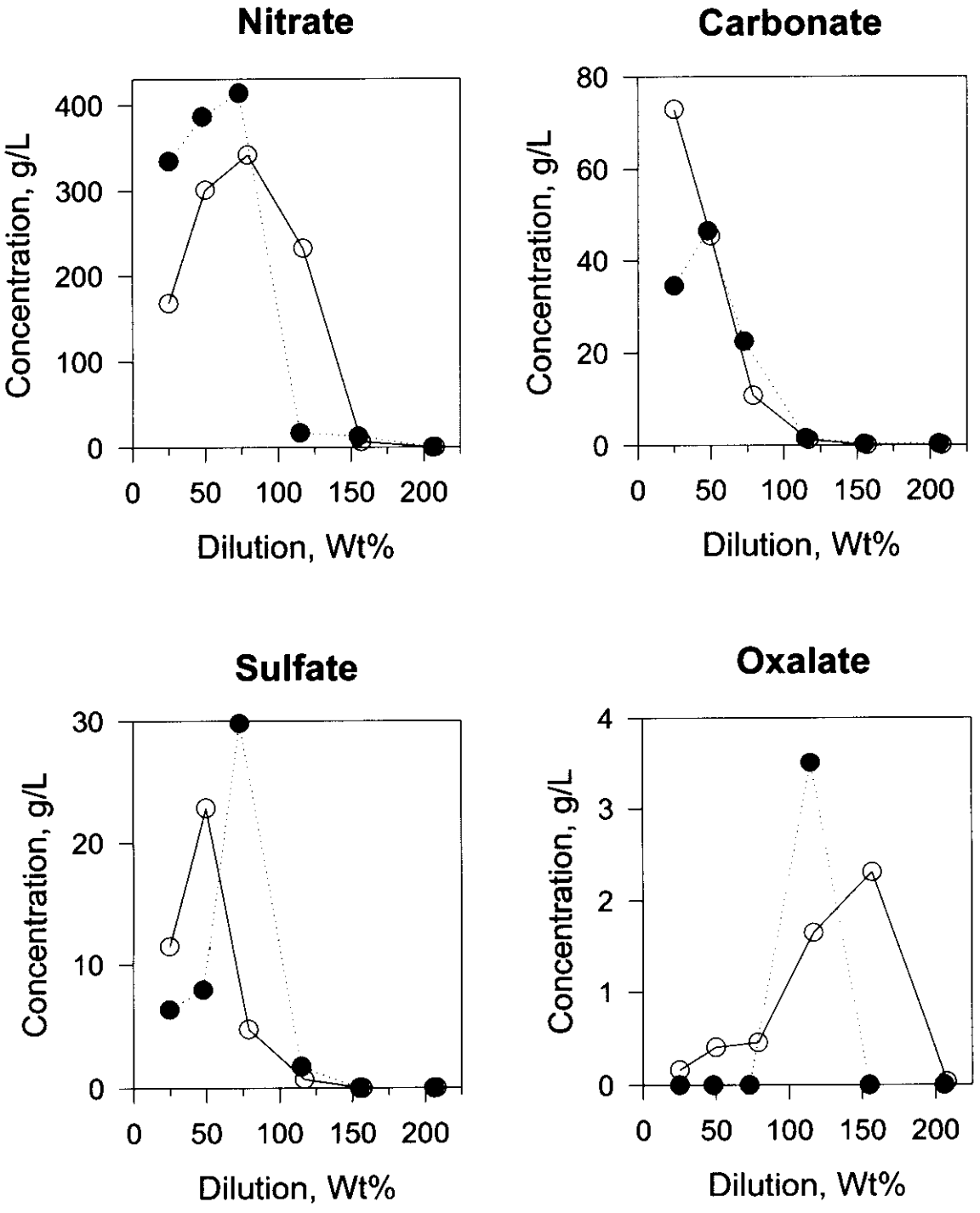
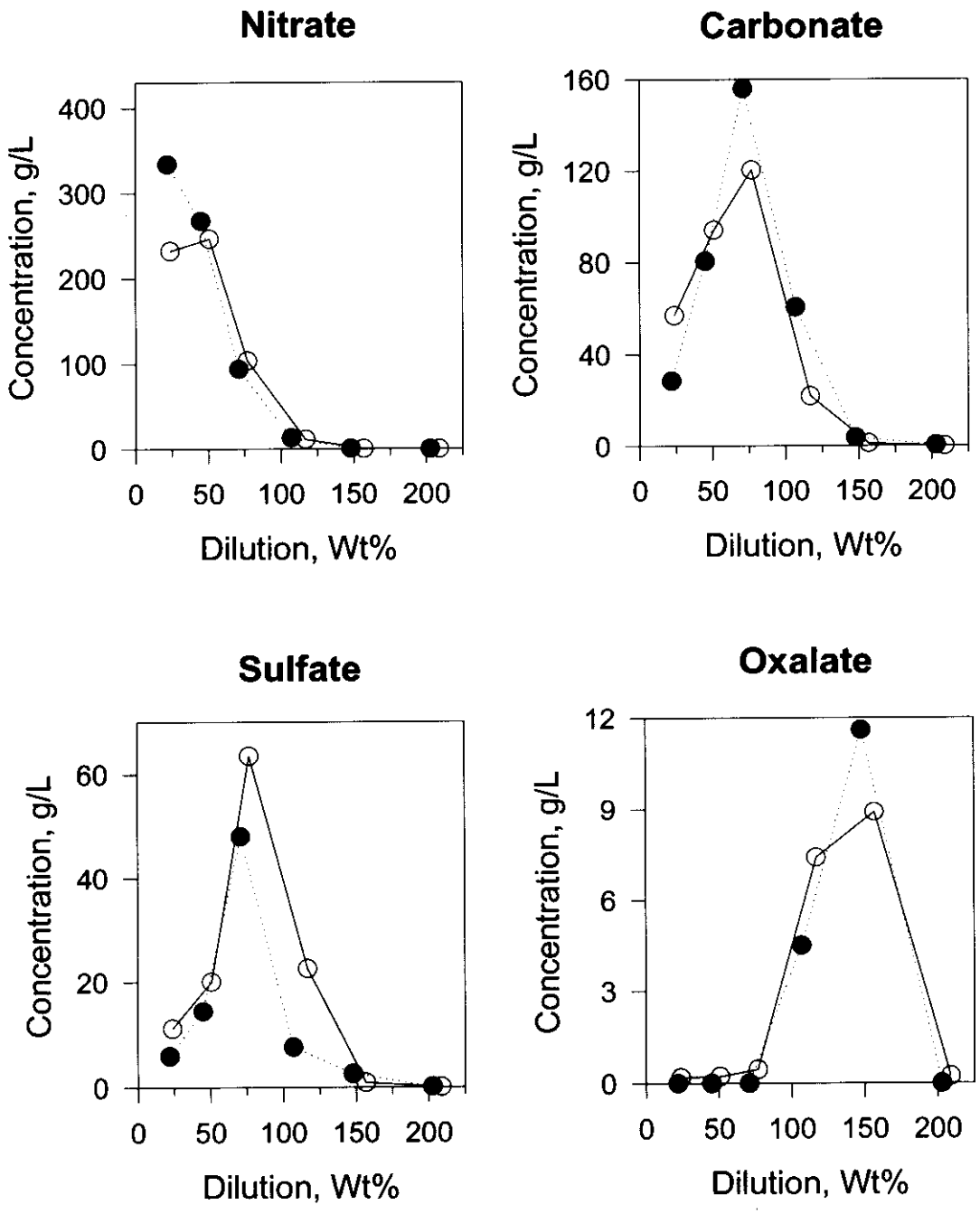


Figure 5-2. Concentrations in Supernatant Liquid, Core 292, H_2O Diluent
(open symbols 22 °C, filled symbols 50 °C)

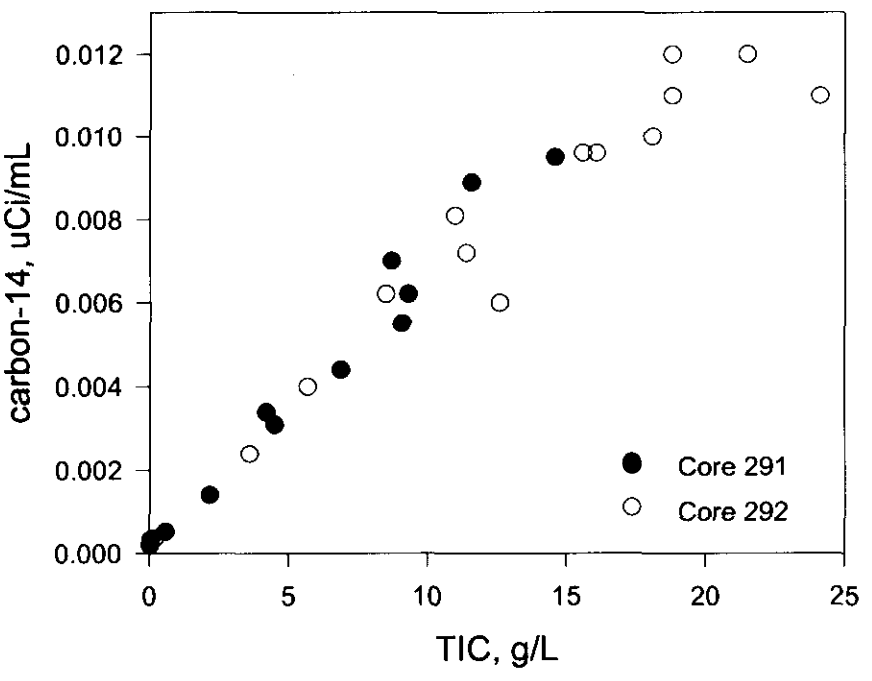


5.1.2 Carbonate and ^{14}C

Anhydrous sodium carbonate, sodium carbonate monohydrate, and sodium carbonate sulfate double salt (Burkeite), all contribute to the overall carbonate concentration in solution. All three salts were observed in the saltcake (see Section 6), and all three have retrograde, or inverse, solubility with temperature, i.e., the solubility decreases with increasing temperature. This is clearly evident at 25 wt% dilution in both figures. As dilution increases in Figure 5-1, the curves for the two temperatures converge at 50 wt% dilution, beyond which point there is insufficient carbonate present in the sample to saturate the solution. In Figure 5-2, however, the carbonate concentration increases sharply with dilution (at either temperature) up to about 80 wt% dilution, showing a strong common ion effect. The effect is much stronger for sodium carbonate than for sodium nitrate because of the two sodium ions per mole of sodium carbonate. To a first approximation, the solubility of sodium carbonate is dependent on the square of the total sodium concentration, whereas for sodium nitrate the dependence is first order.

There is an excellent correlation between carbonate and ^{14}C. Figure 5-3 shows all of the data points from Tests A – G for which both TIC and ^{14}C analyses are available. The obvious correlation applies to all experimental conditions, i.e, both core samples, both temperatures, and both diluents. This correlation strongly implies that ^{14}C is present in the waste exclusively as carbonate ion. There is no correlation between ^{14}C and oxalate (see especially Contacts 4-6 in Tables 3-7a/b and 3-8a/b).

Figure 5-3. Carbonate:^{14}C Correlation



5.1.3 Sulfate and $^{89/90}Sr$

The only sulfate-containing phase positively identified in the saltcake (see Section 6) was Burkeite, $Na_6CO_3(SO_4)_2$, which has inverse solubility with temperature that is clearly evident in both figures. The very strong common ion effect is also evident in both figures.

The correlation between sulfate and $^{89/90}Sr$ is noteworthy. The correlation is not 1:1 like that between TIC and ^{14}C, i.e., the $^{89/90}Sr$ activity does not double when the sulfate concentration doubles. But it is true that, for every test (A – G), the $^{89/90}Sr$ activity reaches its peak at the same point in the dilution curve that the sulfate concentration peaks. This is a strong indication that the small soluble fraction of $^{89/90}Sr$ is associated with the Burkeite phase.

5.1.4 Oxalate

Sodium oxalate probably exists in the saltcake as the anhydrous salt, which does not show inverse solubility with temperature. The data in the Figures 5-1 and 5-2 are difficult to interpret because the oxalate was at or below detection limits in most of the samples, especially those below 100 wt% dilution. The figures make it clear, though, that the oxalate does not appreciably dissolve until most of the other sodium salts are effectively gone.

5.1.5 Other Species

Several components are either completely in the liquid phase at the lowest dilution level (Cl^-, NO_2^-, OH^-) or exist in two or more forms, one of which is completely soluble at the lowest dilution level and the others are relatively insoluble (Al, Cr, Si). All of these show concentrations in the liquid phase that drop sharply from one dilution to the next, and are essentially absent (less than one gram per liter) in the liquid phase beyond approximately 100% dilution under all test conditions for both core samples.

Phosphate was not a major component in either core. In both cores, the phosphate concentration in the liquid phase was relatively constant for the first three dilutions while the solution was saturated, then fell with further dilution as the solid phase was exhausted.

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 3-4 through 3-11) by the volume of solution (or weight of residual solids) gives the total number of grams or microcuries of analyte in each fraction.

Table 5-2 shows the amount (grams or μCi) of each major component in each fraction from the Core 292 Cascade test at 22 °C with water as the diluent (Test B). The column labeled "Total" is the sum of the six liquid fractions plus the residual solids. The column labeled "Percent Dissolved" is 100 times the sum of the six liquid samples divided by the Total. Calculations for the other tests are shown in Appendix B, and the results are summarized in Table 5-3.

The final row in Table 5-3 shows the overall weight percent dissolution, based on the weight data from Tables 4-1 and 4-2. In both cores, raising the temperature from 22 °C to 50 °C resulted in slightly poorer overall dissolution, which appears to be related to the significantly poorer dissolution of Al, Cr, and Si. Changing from water to 2 M NaOH causes some improvement in dissolution for Core 291, but a significant worsening for Core 292. In the case of Core 291, the improvement appears to be related to the improved dissolution of Al, Cr, and Si. In the case of Core 292, the worsening is clearly due to the incomplete dissolution of oxalate in the 2 M NaOH.

Figures 5-4 (Core 291 Test A) and 5-5 (Core 292 Test B) are called "dissolution profile" plots. They show the "Fraction Removed" on the y-axis, defined as the cumulative weight of constituent removed from the sample in the decanted liquid phase divided by the total amount present (liquid and solid phases) in the undiluted sample. The "Fraction Removed" at the highest dilution point matches the "Percent Dissolved" in the preceding tables, when multiplied by 100 to convert "fraction" to "percent". The legend for each figure follows the order of dissolution, with the components with the highest fraction removed listed at the top of the legend. The data for these graphs (as well as the data for the other tests not shown) are tabulated in Appendix B.

Table 5-2. Distribution by Fraction, Core 292, 22 °C, H_2O Diluent (Test B)
(original undiluted sample weight 57.9 g)
(chemicals in grams, radionuclides in μCi)

Analyte	Liquid Samples						Residual Solids	Total	Percent Dissolved
	1st Contact	2nd Contact	3rd Contact	4th Contact	5th Contact	6th Contact			
F^-	0[b]	0	0.005	0.006	0	0	not analyzed	0.011	100[a]
Cl^-	0.068	0.031	0.008	0.002	0	0		0.109	100[a]
NO_2^-	0.626	0.289	0.069	0.009	0	0		0.994	100[a]
NO_3^-	4.988	6.519	2.184	0.286	0.011	0.001		13.990	100[a]
PO_4^{3-}	0.093	0.099	0.086	0.045	0.002	0	0	0.325	100
SO_4^{2-}	0.239	0.533	1.334	0.564	0.022	0.001	0	2.692	100
$C_2O_4^{2-}$	0.004	0.006	0.009	0.184	0.215	0.007	0.041	0.467	91
CO_3^{2-}	1.226	2.491	2.531	0.544	0.026	0	0.009	6.825	99.9
Al	0.363	0.140	0.034	0.005	0.001	0.001	0.016	0.522	97
Cr	0.199	0.087	0.022	0.003	0	0	0.038	0.350	89
Fe	0	0	0	0	0.0003	0.0003	0.0138	0.0144	4
K	0.019	0	0	0	0	0	0	0.019	100
Na	4.924	5.883	4.095	0.960	0.153	0.009	0.033	16.06	99.8
P	0.038	0.043	0.037	0.019	0.001	0	0	0.139	100
S	0.103	0.226	0.569	0.200	0.009	0.001	0	1.107	100
Si	0.010	0.005	0.001	0	0	0	0.058	0.075	78
U	0	0	0	0.0008	0.0007	0.0005	0.035	0.037	5
$^{89/90}Sr$	3.50	13.09	42.84	15.90	1.85	13.33	1762[c]	1853	5
^{99}Tc	2.30	1.01	0.25	0.04	0	0	0.16	3.76	96
^{137}Cs	2098	901	216	35	5	2	7	3264	99.8

[a]100% dissolution assumed for these components; residual solids not analyzed by IC.
[b]An entry of '0' indicates analytical result was below detection limit.
[c]Based on fusion digest of undiluted composite sample [Prilucik 2002] minus sum of liquid fractions.

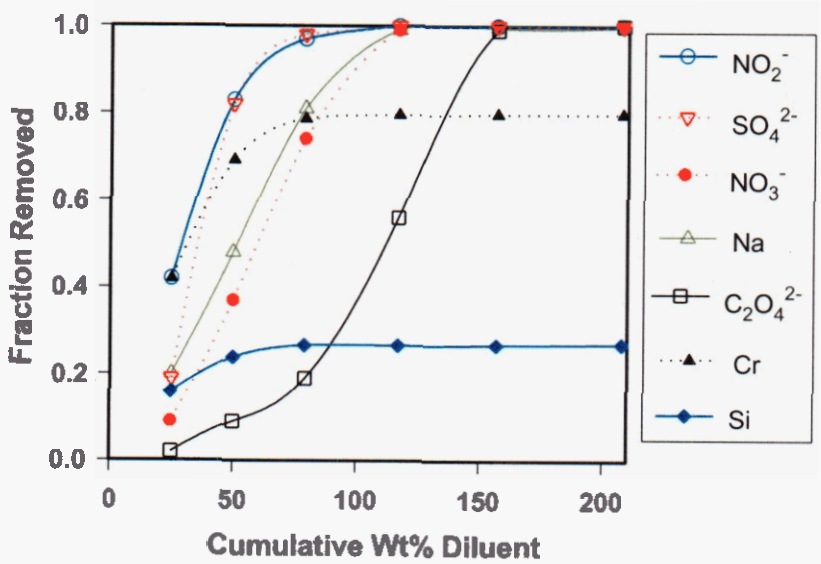
Table 5-3. Distribution by Fraction Summary
(percent dissolved at end of dissolution test)

	Core 291			Core 292		
Temp	22 °C	50 °C	22 °C	22 °C	50 °C	22 °C
Diluent	H_2O	H_2O	2M NaOH	H_2O	H_2O	2M NaOH
Test	A	F	D	B-C avg	G	E
F^-, NO_2^-, Cl^-, NO_3^-	assumed to be 100% dissolved					
PO_4^{3-}	100	100	100	100	100	100
SO_4^{2-}	100	100	100	100	100	100
$C_2O_4^{2-}$	99	97	98	92	88	31
CO_3^{2-}	99.7	99.5	99.7	99.9	99.8	99.7
Al	92	87	96	97	91	93
Cr	80	68	85	89	83	76
Na	99.7	99.9	100	99.8	99.7	99.7
P	100	100	100	100	100	100
S	100	100	100	100	100	100
Si	27	24	56	25	19	33
$^{89/90}Sr$	6	2	6	4	4	3
^{99}Tc	100	100	100	95	94	90
^{137}Cs	100	100	100	99.8	99.7	99.4
gross weight	98.8	98.2	99.3	98.4	98.1	96.4

The dissolution profiles (Figures 5-4 and 5-5) are similar for the two cores. The most significant differences are apparent in the carbonate, sulfate, and nitrate curves. In Core 291, which is high in nitrate, the carbonate and sulfate are removed relatively early in the dissolution, coming out before the nitrate. At 50% diluent, for example, over 80% of the carbonate and sulfate have been removed, compared to less than 40% of the nitrate.

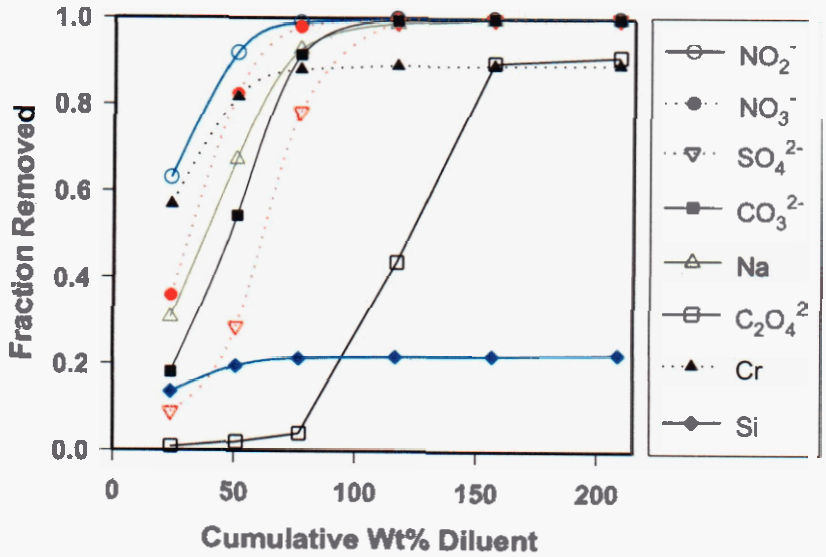
In Core 292, which is high in carbonate and sulfate, the reverse is true. At 50% diluent, over 82% of the nitrate has been removed, compared to about 54% of the carbonate and 29% of the sulfate.

Figure 5-4. Core 291 Dissolution Profile, H_2O, 22 °C



[not shown: Cs, Tc, OH^-, Cl^-, CO_3^{2-} similar to NO_2^-]

Figure 5-5. Core 292 Dissolution Profile, H_2O, 22 °C



[not shown: Cs, OH^-, Cl^- similar to NO_2^-]

The overall sum of liquid fractions and residual solids (the column labeled Total in Table 5-2) can be divided by the original undiluted sample weight (shown above the table) and multiplied by 100 to give the weigh percent of each component in the undiluted saltcake. (In the case of the radionuclides, the dividend is not multiplied by 100, and the result is expressed in μCi/g.) These values can be compared to the analytical results for the direct analysis of the composite sample [Prilucik 2002] as a test of the validity of the analysis by fractions. This comparison is shown in Table 5-4 for Core 291 and Table 5-5 for Core 292.

In general, the comparison is remarkably good, considering the number of independent analyses that are summed to provide the results for each dilution test. There are a few discrepancies worth pointing out.

All of the components that have relatively low solubility (Fe, Si, $^{89/90}$Sr) are consistently low in the sum-of-fractions compared to the direct analysis. This is an indication that the analyses of the residual solids (the solids remaining undissolved after the last water/NaOH contact) were not accurate. In every test, the amount of residual solids was too small to collect a sample for analysis. Therefore, the residual solids from all of the Core 291 Tests A, D, and F were combined into a single sample for fusion digest and analysis. Likewise, all of the residual solids from Core 292 Tests B, C, E, G, H, and I were combined into a single sample for analysis. In both cases, the analytical results gave composition values that were significantly below the values determined by direct (fusion digest) analysis of the undiluted core composite samples [Prilucik 2002].

Oxalate is expected to be low in Tests D and E due to the poor dissolution of oxalate in 2 M NaOH. (The residual solids were not analyzed for oxalate.) The low oxalate in Test F is not real. It is an artifact of the high "less-than" values reported for several of the liquid samples (see Table 3-9a). The "less-than" results are not included in the sum of fractions except where no values above detection limit were reported (i.e., fluoride in Tests D and F).

In calculating the percent dissolved (Table 5-3) and the dissolution profile graphs (Figures 5-4 and 5-5), it was necessary to choose a "best value" for the total amount of each component present in the sample used for each test. In most cases, this "best value" was taken as the sum-of-fractions (sum of liquid fractions plus the residual solids). In a few cases, the direct composite sample analysis [Prilucik 2002] was used instead. Those cases are those in which there was a significant fraction of material not accounted for due to the bias in the analysis of the residual solids, as described above. Specifically, the direct analysis result was chosen in favor of the sum-of-fractions for Al, Cr, Fe, Na, Si, and $^{89/90}$Sr, unless the sum-of-fractions was higher than the direct analysis. In the case of ^{137}Cs, no significant amount was detected in the residual solids in either core, so the bias was not an issue, and the sum-of-fractions was used as the "best total". In the case of ^{99}Tc, none was detected in Core 291 residual solids, and the sum-of-fractions was higher than the direct analysis in Core 292 Test B, so the sum-of-fractions taken as the "best value" throughout.

Table 5-4. Composition Determined by Sum of Fractions and Direct Analysis, Core 291 (chemicals in weight percent, radionuclides in μCi/g)

Analyte	Test A	Test D	Test F	Direct
	H_2O, 22 °C	2M NaOH, 22 °C	H_2O, 50 °C	[Prilucik 2002]
F^-	0.010	< 0.004	< 0.009	< 0.012
Cl^-	0.10	0.11	0.11	0.15
NO_2^-	0.85	1.02	0.97	0.99
NO_3^-	44.3	36.2	42.9	51.0
PO_4^{3-}	0.57	0.67	0.60	0.47
SO_4^{2-}	1.48	1.83	1.64	2.17
$C_2O_4^{2-}$	0.22	0.15	0.13	0.26
CO_3^{2-}	4.25	4.25	3.95	4.90
OH^-	0.75	--	0.84	0.95
Al	0.44	0.51	0.43	0.46
Cr	0.19	0.21	0.18	0.20
Fe	0.006	0.004	0.009	0.009
K	0.032	0.059	0.035	--
Na	27.0	--	23.5	27.1
P	0.22	0.23	0.21	0.15
S	0.62	0.64	0.53	0.72
Si	0.04	0.08	0.04	0.14
^{14}C	0.0053	n.a.	0.0051	0.0048
^{79}Se	0.0002	0.0002	0.0002	0.0003
$^{89/90}Sr$	12.6	7.4	17.0	17.0
^{99}Tc	0.031	0.033	0.031	0.035
^{137}Cs	29.4	34.4	30.1	32.8

Table 5-5. Composition Determined by Sum of Fractions and Direct Analysis, Core 292 (chemicals in weight percent, radionuclides in μCi/g)

Analyte	Test B	Test C	Test E	Test G	Direct
	H_2O, 22 °C	H_2O, 22 °C	2M NaOH, 22 °C	H_2O, 50 °C	[Prilucik 2002]
F^-	0.019	0.017	0.023	0.046	0.018
Cl^-	0.19	0.17	0.17	0.18	0.19
NO_2^-	1.72	1.54	1.59	1.69	1.56
NO_3^-	24.2	24.1	25.1	25.8	26.9
PO_4^{3-}	0.56	0.63	0.75	0.64	0.60
SO_4^{2-}	4.65	5.93	5.19	5.57	5.49
$C_2O_4^{2-}$	0.81	0.86	0.41	0.74	0.78
CO_3^{2-}	11.8	12.4	11.4	11.8	12.2
OH^-	1.20	1.15	--	1.18	1.16
Al	0.90	0.89	0.88	0.76	0.80
Cr	0.60	0.60	0.65	0.52	0.54
Fe	0.025	0.025	0.056	0.031	0.055
K	0.032	0.033	0.045	0.051	--
Na	27.7	29.0	--	25.0	25.5
P	0.24	0.26	0.26	0.22	0.20
S	1.91	2.03	2.14	1.72	1.83
Si	0.04	0.04	0.06	0.03	0.13
^{14}C	0.012	0.010	0.015	0.011	0.016
^{79}Se	0.0003	0.0004	0.0003	0.0003	0.0005
$^{89/90}Sr$	15.0	14.6	32.5	18.1	32.0
^{99}Tc	0.065	0.061	0.064	0.061	0.063
^{137}Cs	56.4	53.4	52.7	53.2	56.4

6.0 SOLID PHASE IDENTIFICATION

Knowledge of the speciation of the solid phases in tank waste, as opposed to the simple elemental analysis data historically available, is becoming increasingly important. Accurate chemical modeling of tank waste systems (e.g., with ESP) depends on proper identification of system components, including the chemical speciation of solids.

Methods for analyzing solid phases in saltcake samples are under development. The first attempt at a comprehensive solid phase analysis was performed on a composite saltcake sample from tank BY-102 [Herting 1999].

The solids characterization scheme employed for tank S-112 saltcake, described in Section 2.6, used a combination of polarized light microscopy (PLM), X-ray diffraction (XRD), and scanning electron microscopy with energy-dispersive X-ray spectroscopy (SEM/EDS). The three methods are complementary, and many solid phases can be identified by the combination of techniques.

Several crystalline phases can be identified by PLM alone. Crystal size and shape (morphology) and optical properties (number of unique refractive indexes, approximate birefringence, extinction position) can be determined by simple microscopic observation of the crystals with polarized light. Each crystalline phase has a "fingerprint" set of physical and optical properties that make it identifiable virtually at a glance. Several types of crystalline phases common to Hanford waste have been fingerprinted this way and catalogued [Herting 1992].

The XRD instrument identifies powdered crystalline phases by their diffraction patterns. The software for the instrument includes a large library of diffraction patterns of known compounds, and identification of an unknown is made by matching its diffraction pattern to one of the known compounds in the library.

The SEM provides high-magnification images with very good depth-of-field (which the PLM lacks). The associated EDS is basically an X-ray fluorescence technique that provides semi-quantitative elemental analysis on a particle-by-particle basis. It complements the PLM and XRD in two ways. For crystalline materials, it can provide the elemental composition of an individual crystal, so the scope of possible candidates is narrowed considerably, and identification might then be possible with PLM or XRD. It can also identify the elemental makeup of amorphous particles – something neither of the other techniques can do.

6.1 SAMPLE PREPARATION AND MOUNTING

Sample preparation is one of the most important factors for all three techniques (PLM, SEM/EDS, and XRD) in getting good results. Sample size, of course, is always a consideration for such very-small-volume techniques. The radioactive nature of the samples puts some constraints on allowable sample handling methods, in consideration of both radiation dose to personnel and the potential for radioactive contamination of the laboratory. Significant progress was made during the year in developing appropriate sample handling methods, though the methods are still evolving.

6.2 TANK S-112 SALTCAKE SAMPLES

Sample aliquots were taken for solid phase characterization from six core segment samples from tank 241-S-112, as required by the Tank Sampling and Analysis Plan [Lauricella 2001]. The six samples were analyzed by a combination of PLM, SEM/EDS, and XRD. All three segments from Core 291 (segments 1, 10, and 12) contained mainly sodium nitrate with traces of minor components. Core 292 (segments 2A, 8, and 10) contained large amounts of sodium carbonate in anhydrous and monohydrate forms, sodium carbonate sulfate double salt [a.k.a. Burkeite, $Na_6CO_3(SO_4)_2$], and sodium nitrate. The sodium nitrate content increased with increasing depth. Observations for each individual segment sample follow.

Core 291 Segment 1, Sample S01T001730 (light gray dry salt)

PLM: estimated 99% $NaNO_3$ with traces of several unidentified minor phases.
SEM/EDS: $NaNO_3$ with indications of a fine-grained aluminosilicate phase.
XRD: no phases observed other than $NaNO_3$

Core 291 Segment 10, Sample S01T001731 (bright yellow with green tint, clumpy, moist salt)

PLM: still mainly $NaNO_3$, but with significant (on the order of 5%) unidentified second phase and still traces of other minor phases.
SEM/EDS: mainly $NaNO_3$; other phases include probably Na_3PO_4 $12H_2O$ and a phase with mixed Na-Al-Cr.
XRD: $NaNO_3$ with <5% Na_2CO_3 H_2O

Core 291 Segment 12, Sample S01T001732 (same in appearance as Segment 10)

PLM: virtually identical in appearance to Segment 10.
SEM/EDS: same as Segment 10.
XRD: no phases observed other than $NaNO_3$

Core 292 Segment 2A (upper half), Sample S01T001733 (uniformly gray-brown and grainy)

PLM: little if any $NaNO_3$; multiple other phases, the most common of which has refractive indexes consistent with Na_2CO_3 xH_2O.
SEM/EDS: major phase Na_2CO_3 xH_2O; other significant phases include a Na-S-rich phase and an Al-rich phase with no associated Si or Cr; trace phase containing U-Cr-Mn-Fe.
XRD: (percentages very approximate) 60% Na_2CO_3 H_2O; 25% $Na_6CO_3(SO_4)_2$ (Burkeite); <10% $NaNO_3$; <10% Na_3PO_4 $12H_2O$.

Core 292 Segment 8, Sample S01T001738 (dark olive green "goo", consistency of biscuit dough)

PLM: very similar to Segment 2A, but with more $NaNO_3$.
SEM/EDS: mostly $NaNO_3$, with surface coating containing Al-S-Cr.
XRD: $NaNO_3$, $NaNO_2$, Na_2CO_3 H_2O, and anhydrous Na_2CO_3 all major phases; no Burkeite observed.

Core 292 Segment 10, Sample S01T001739 ("green apple" green moist salt)

PLM: similar to Segment 8; $NaNO_3$, Na_2CO_3 xH_2O, and traces of several unidentified minor phases; significant amount of an interesting but unidentified bi-capped prism crystal not observed with other instruments.
SEM/EDS: mainly $NaNO_3$ with traces of $Na_3PO_4 \cdot 12H_2O$, a phase containing Al-S-Cr, and a fine-grained S-rich phase.
XRD: predominantly $NaNO_3$ with smaller but significant amounts of $NaNO_2$, Na_2CO_3 H_2O and anhydrous Na_2CO_3.

7.0 ACKNOWLEDGEMENTS

The authors are indebted to Ms. Lenna Mahoney for her thorough and conscientious review of the spreadsheet used to calculate the derived data reported here. The authors also thank Dr. Jim Person, Dr. Jim Jewett, Mr. John Appel, Mr. Blaine Barton, Mr. Dave Place, and Mr. Brian Brendel for their helpful reviews of the manuscript. Viscosity measurements were performed by Mr. John Smith. X-Ray and SEM/EDS analyses were performed by Dr. Wade Warrant and Mr. Gary Cooke, respectively. The bulk of the hotcell work was performed by Mr. Wayne Edmonson. The staff at the 222-S Laboratory provided the analytical work and miscellaneous laboratory support.

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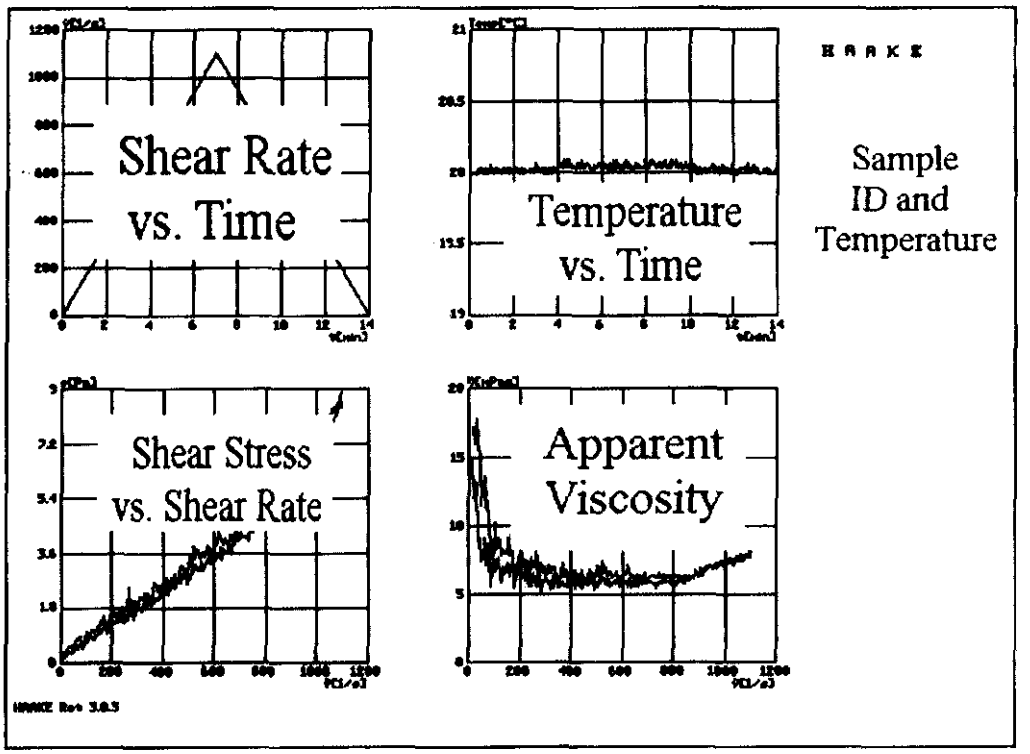
Appendix A

Rheology Figures



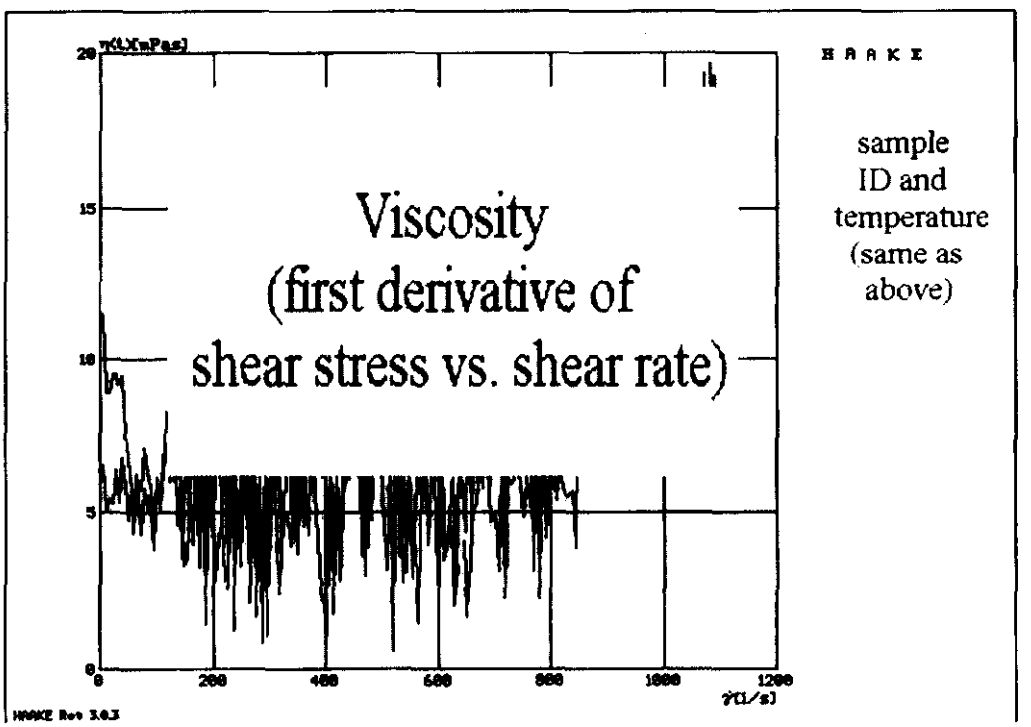
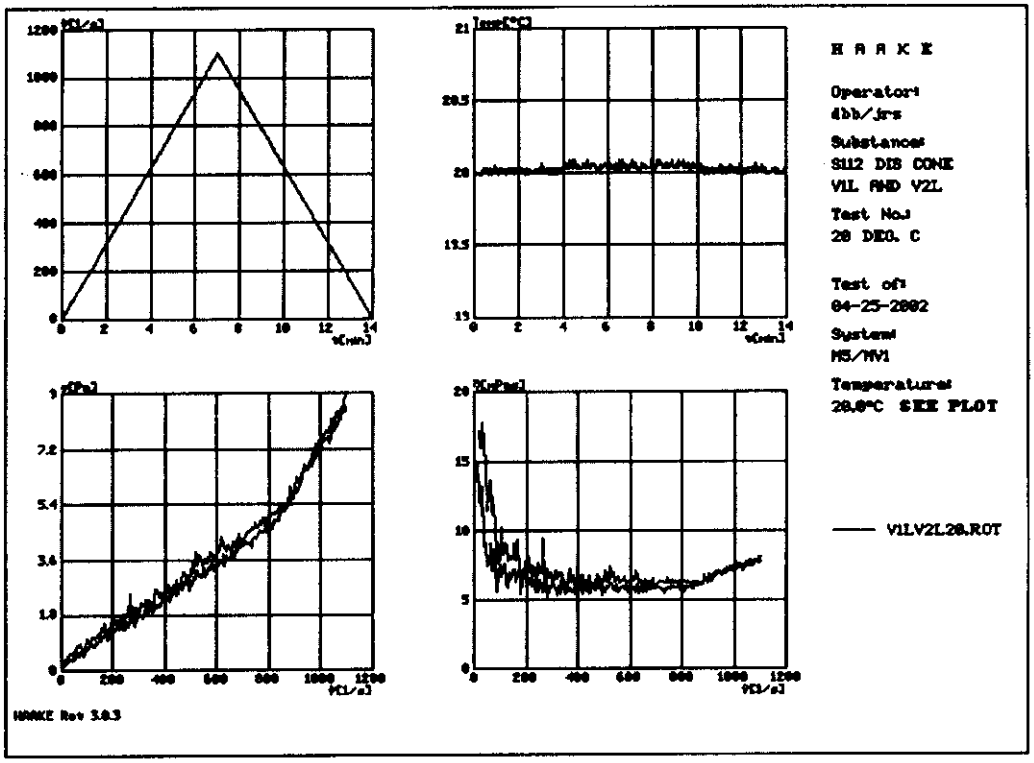


Figure A-1. Template for Rheology Figures



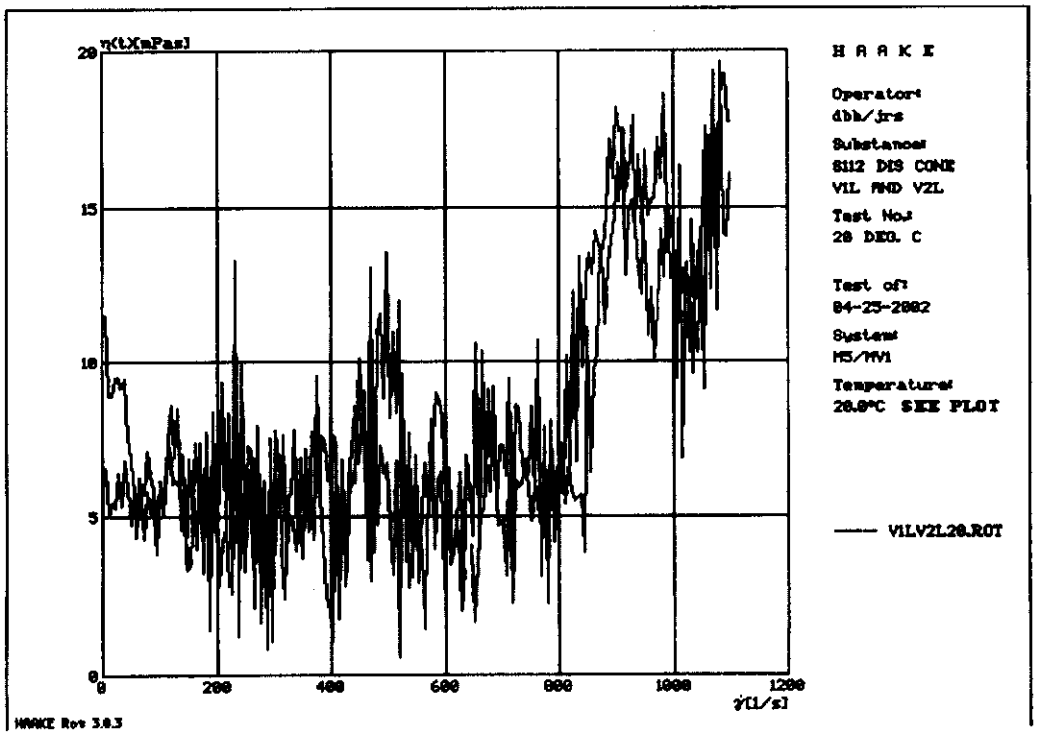
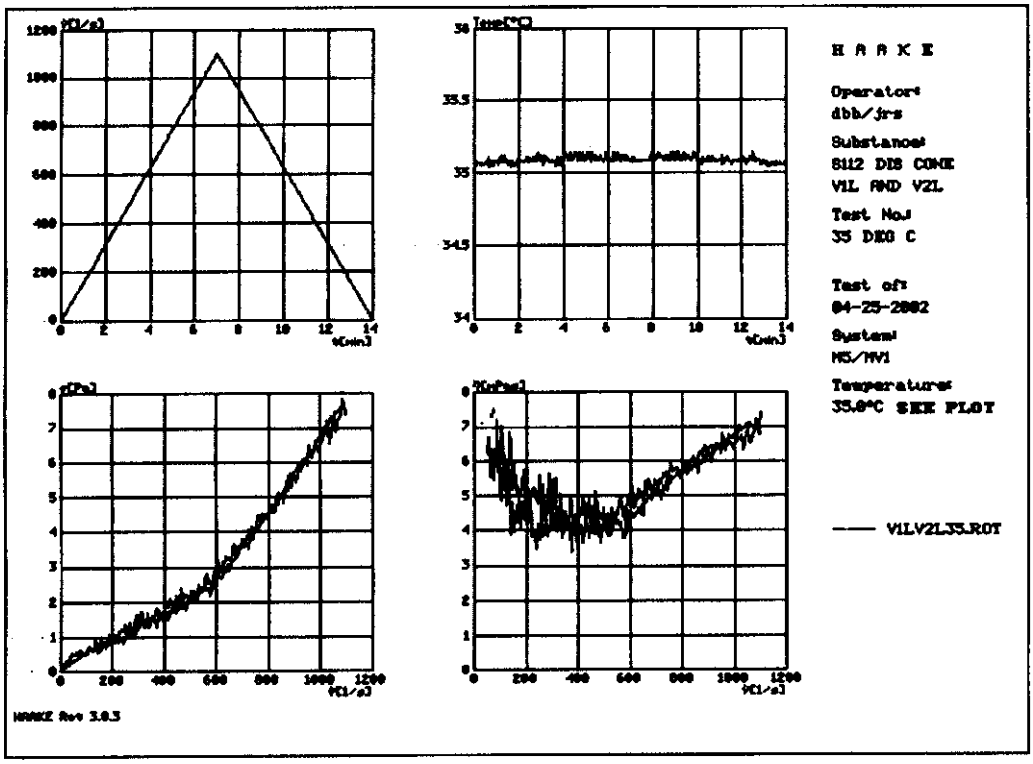


Figure A-2. Rheology Figures for Sample V1L/V2L at 20 °C



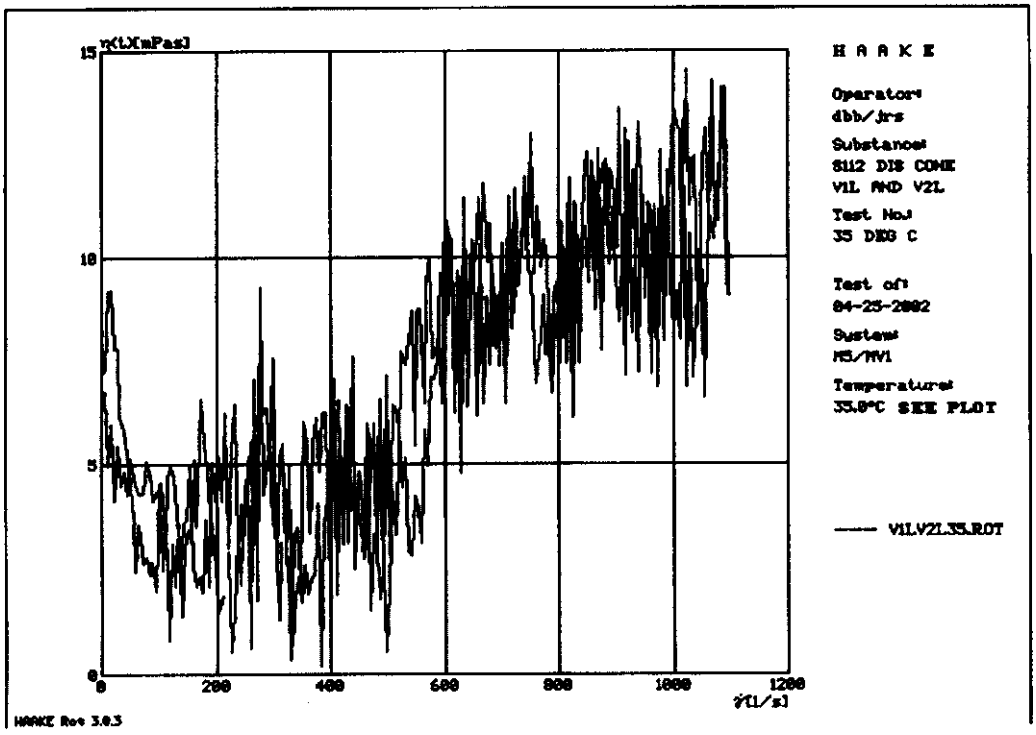
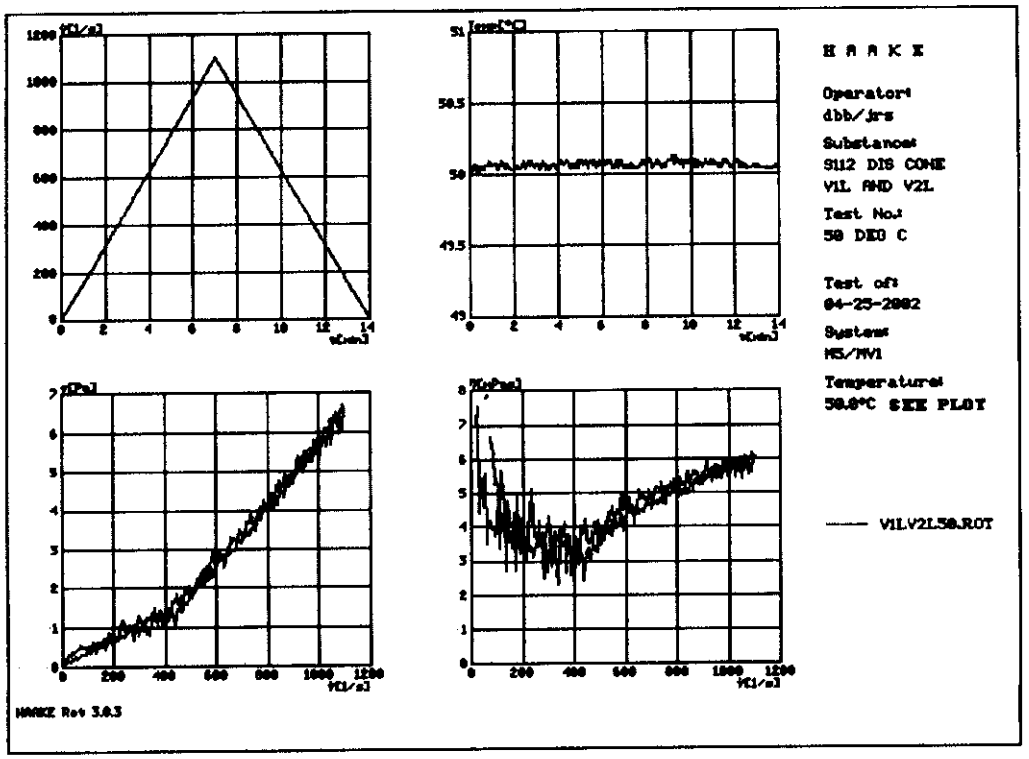


Figure A-3. Rheology Figures for Sample V1L/V2L at 35 °C



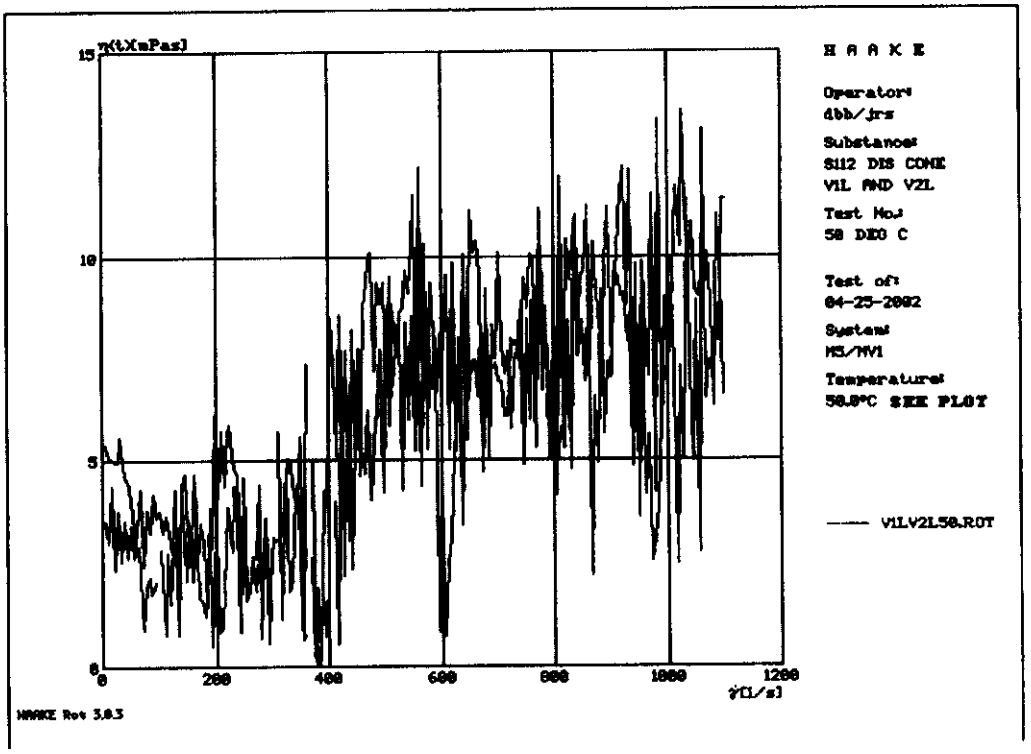
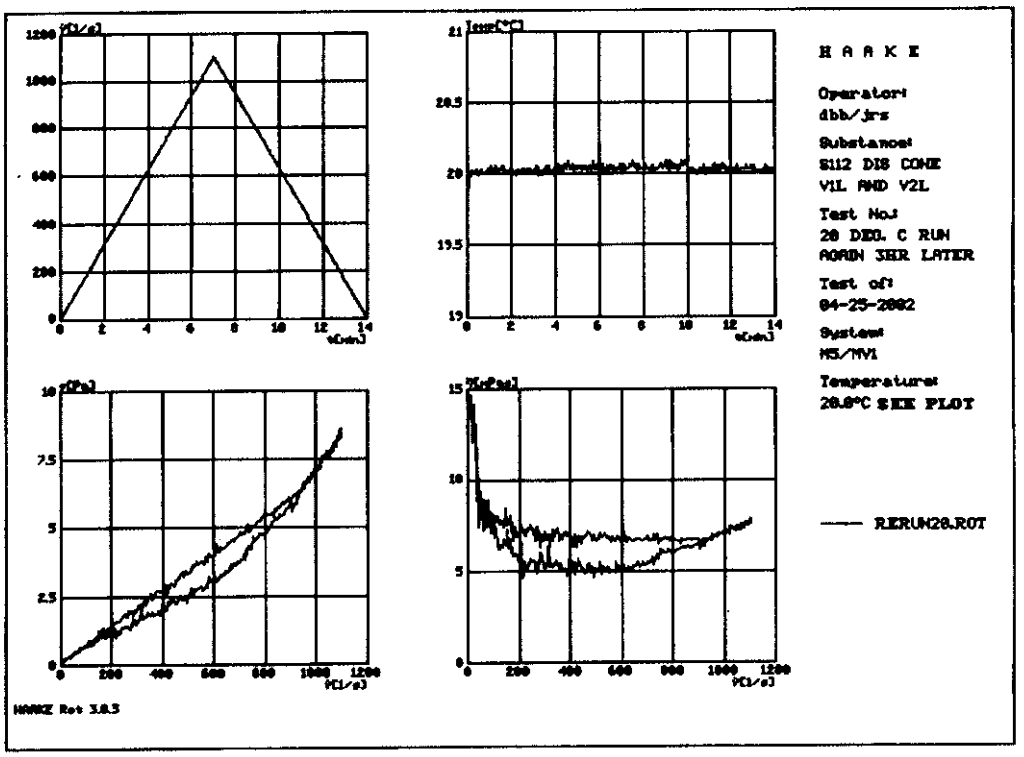


Figure A-4. Rheology Figures for Sample V1L/V2L at 50 °C



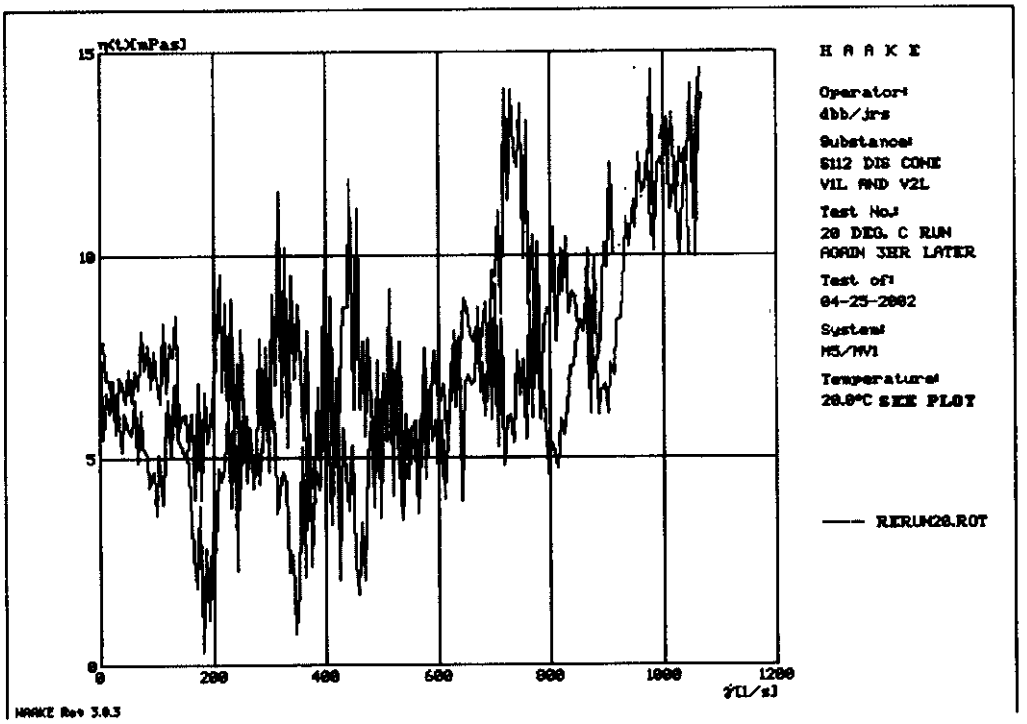
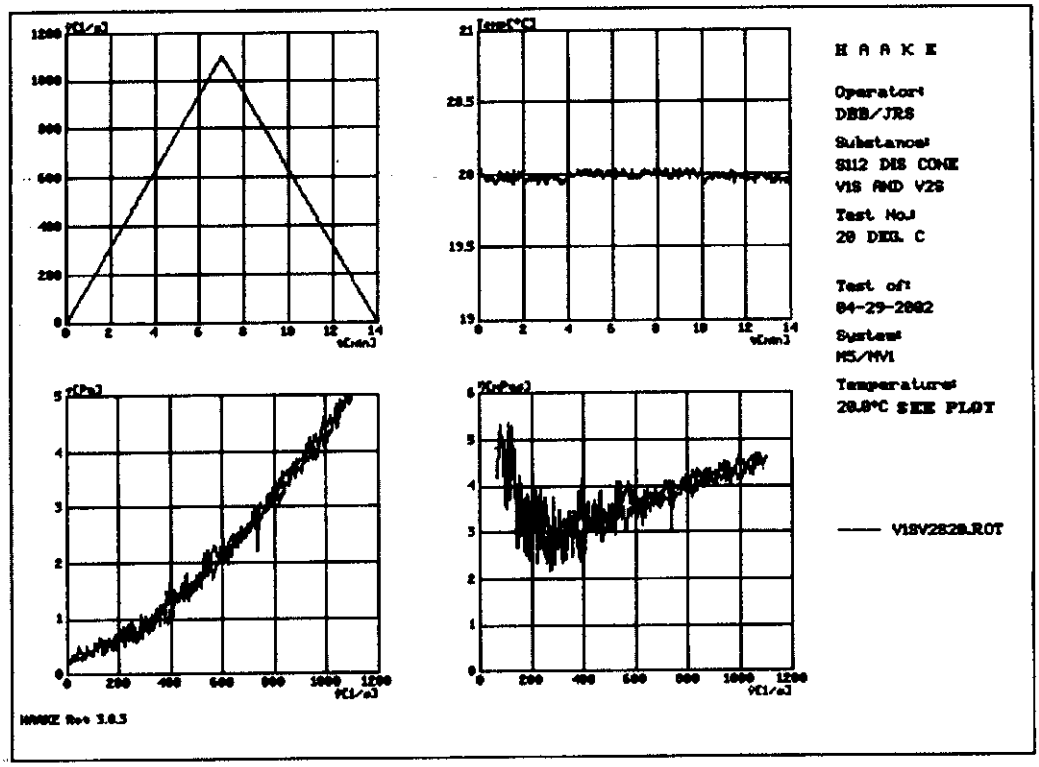


Figure A-5. Rheology Figures for Sample V1L/V2L Returned to 20 °C



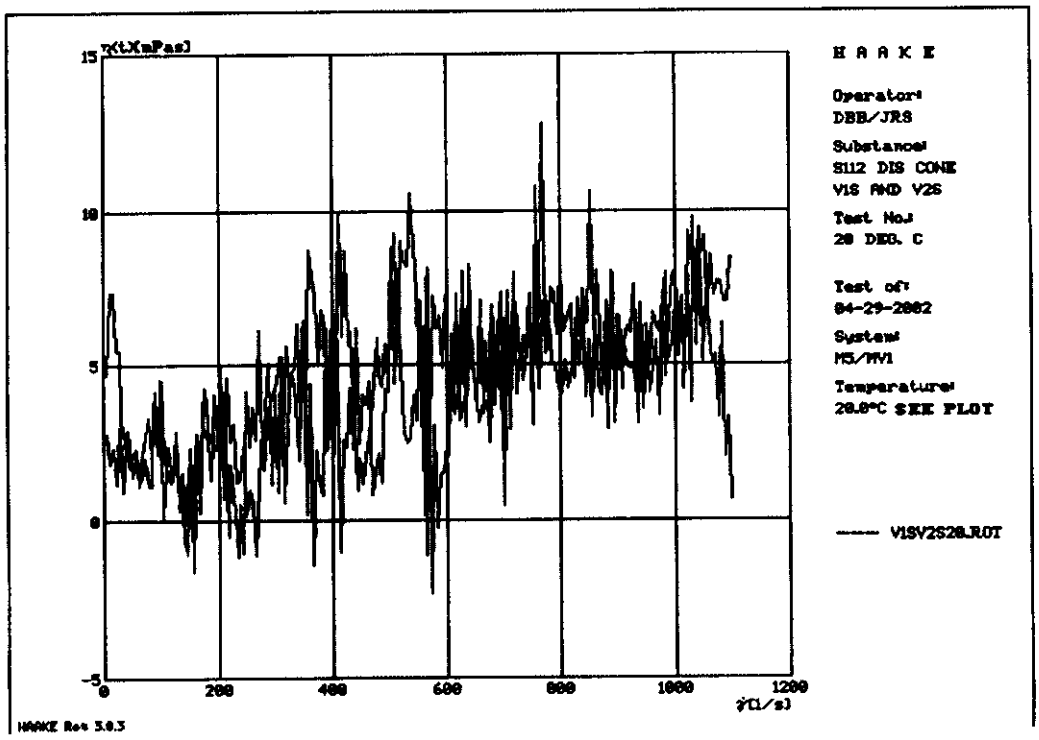
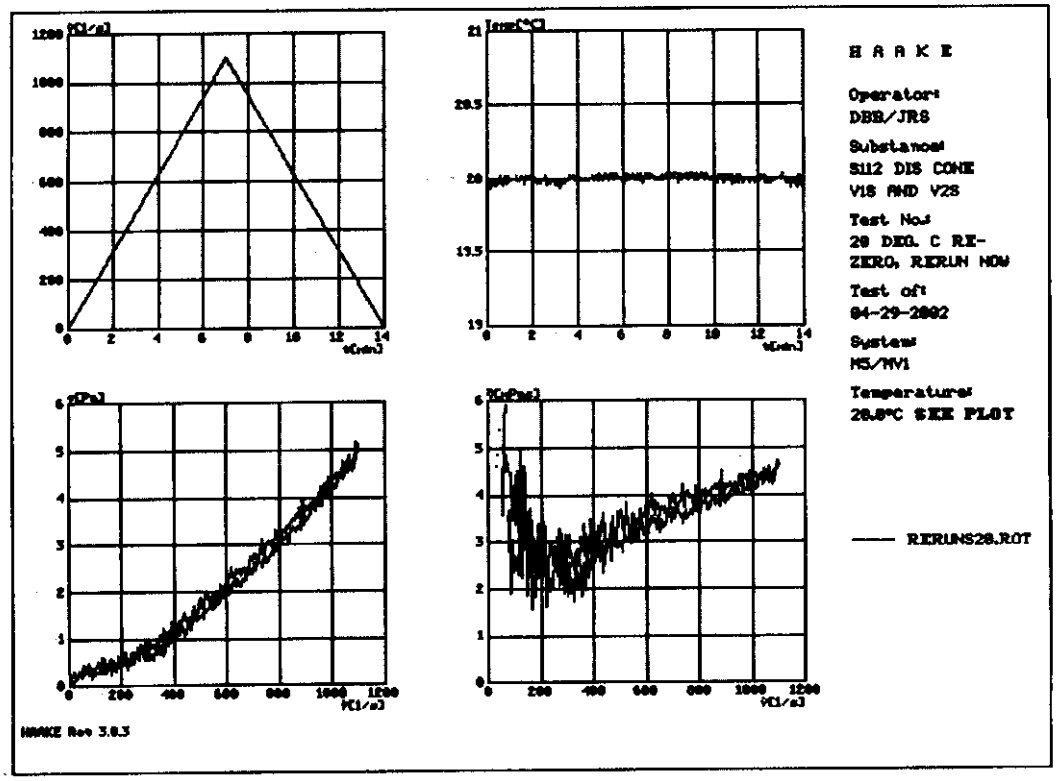


Figure A-6. Rheology Figures for Sample V1S/V2S at 20 °C



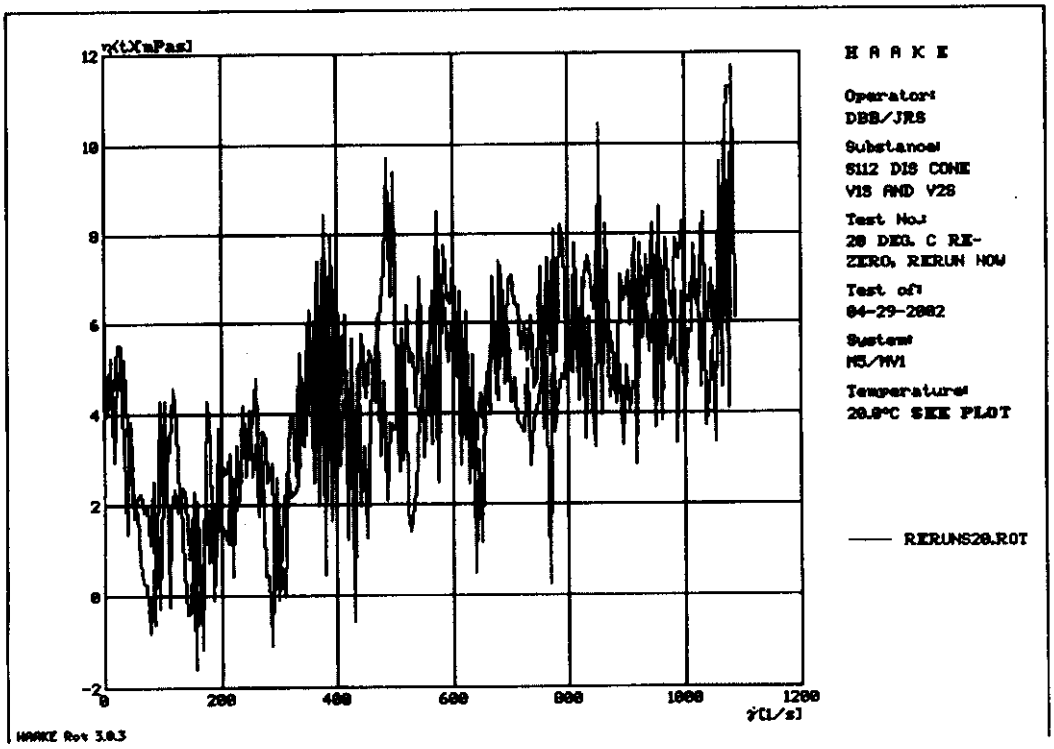
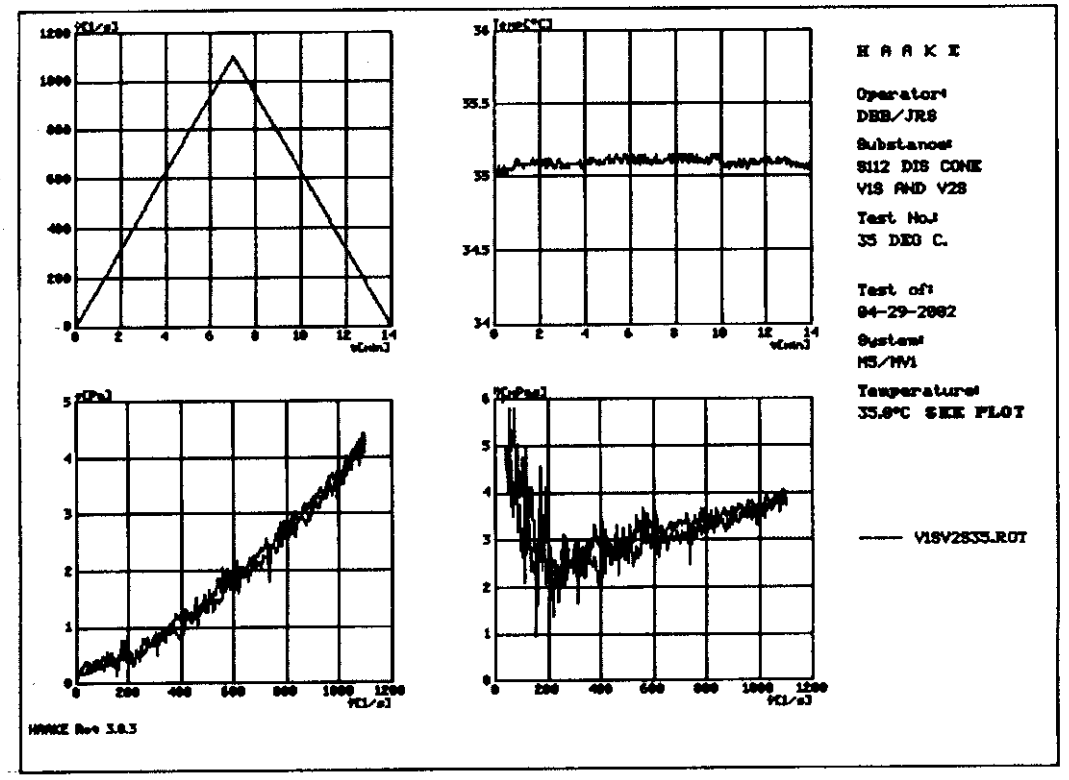


Figure A-7. Rheology Figures for Sample V1S/V2S at 20 °C (re-run)



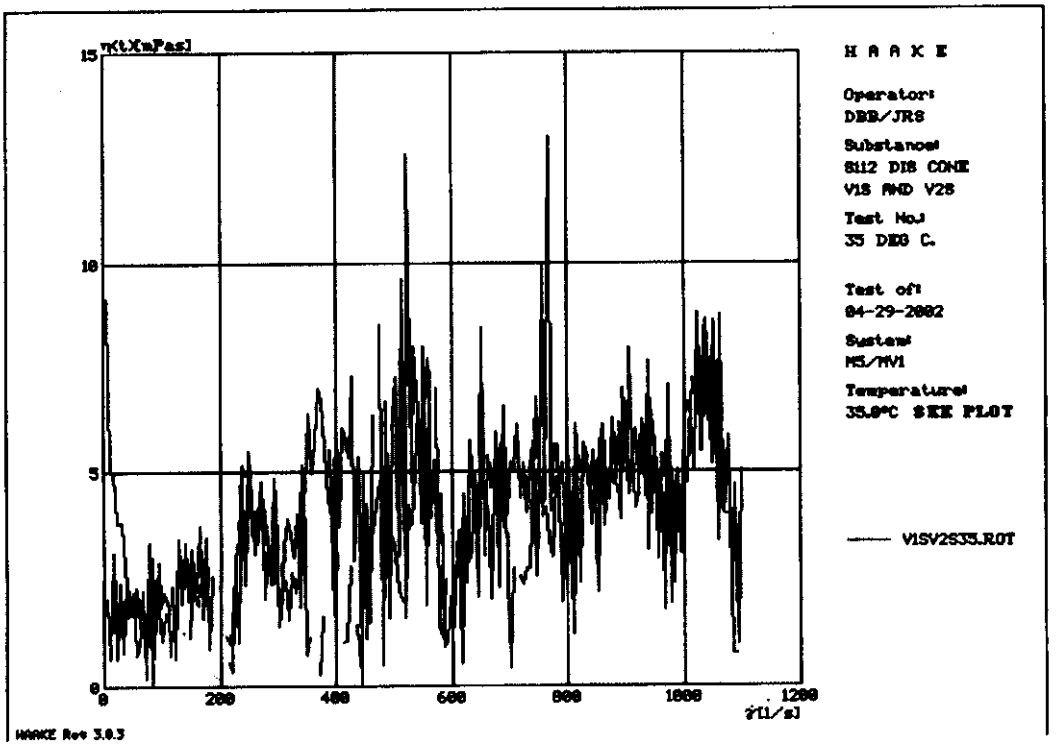
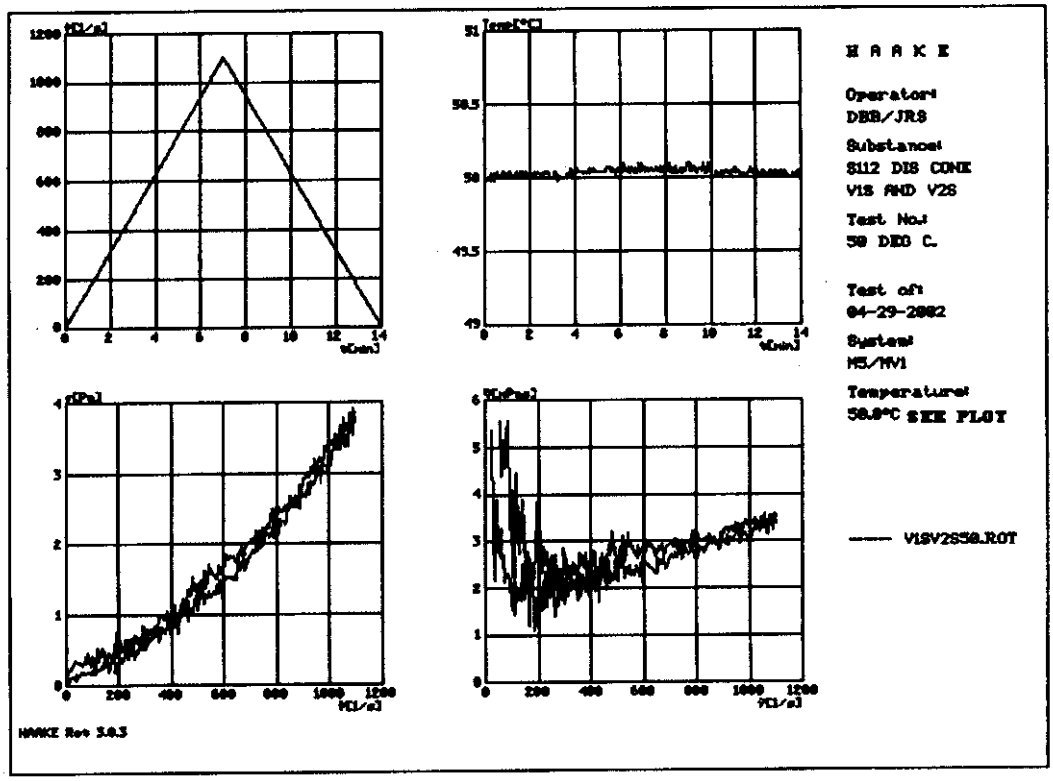


Figure A-8. Rheology Figures for Sample V1S/V2S at 35 °C



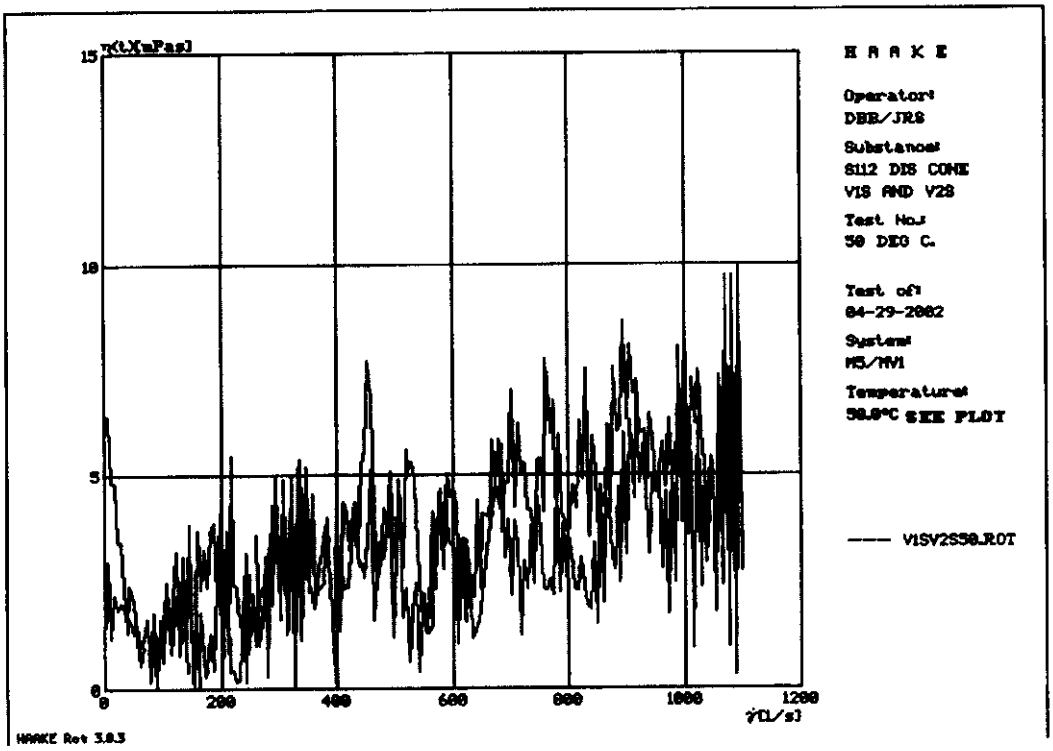
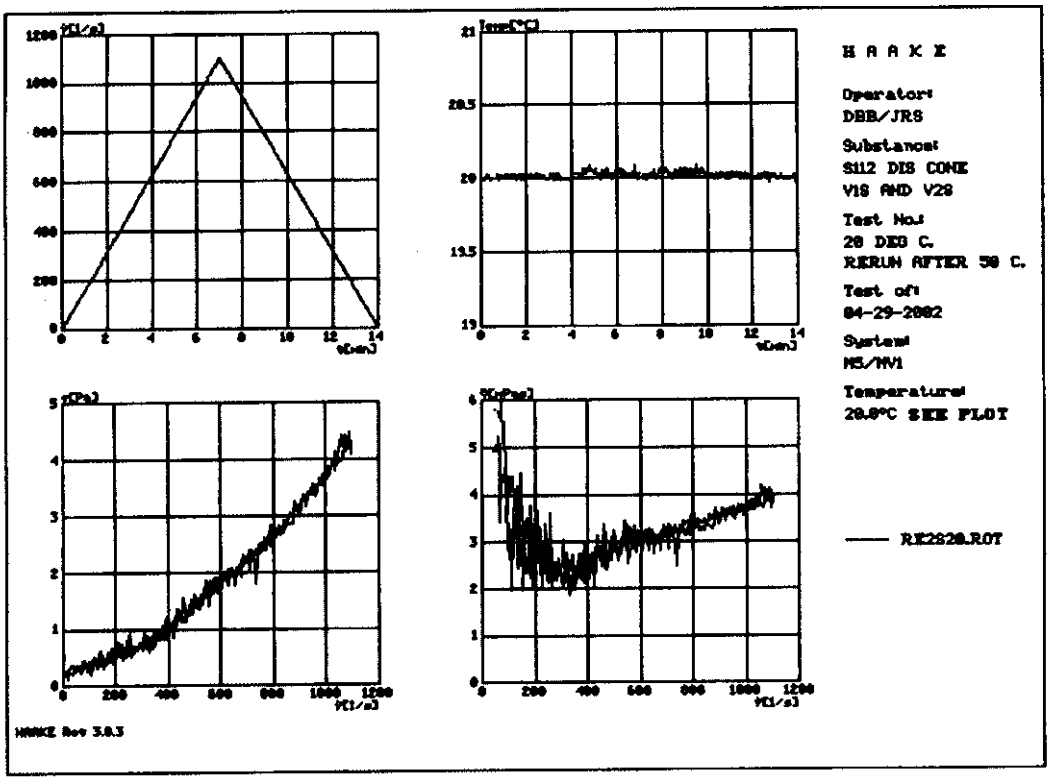


Figure A-9. Rheology Figures for Sample V1S/V2S at 50 °C



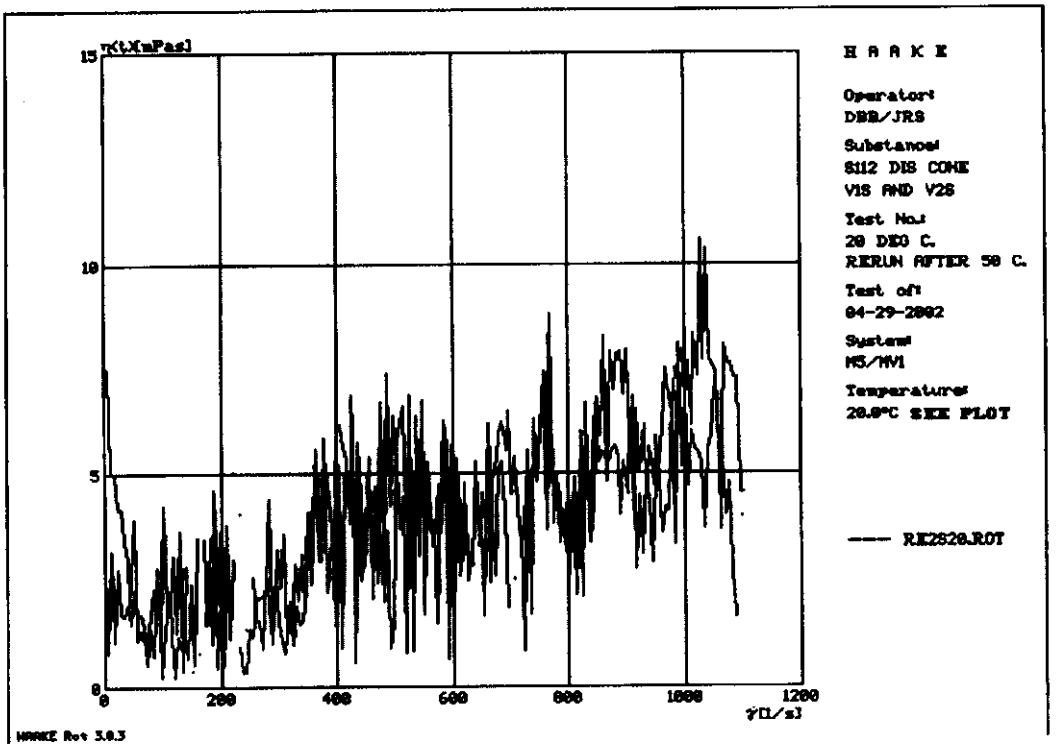
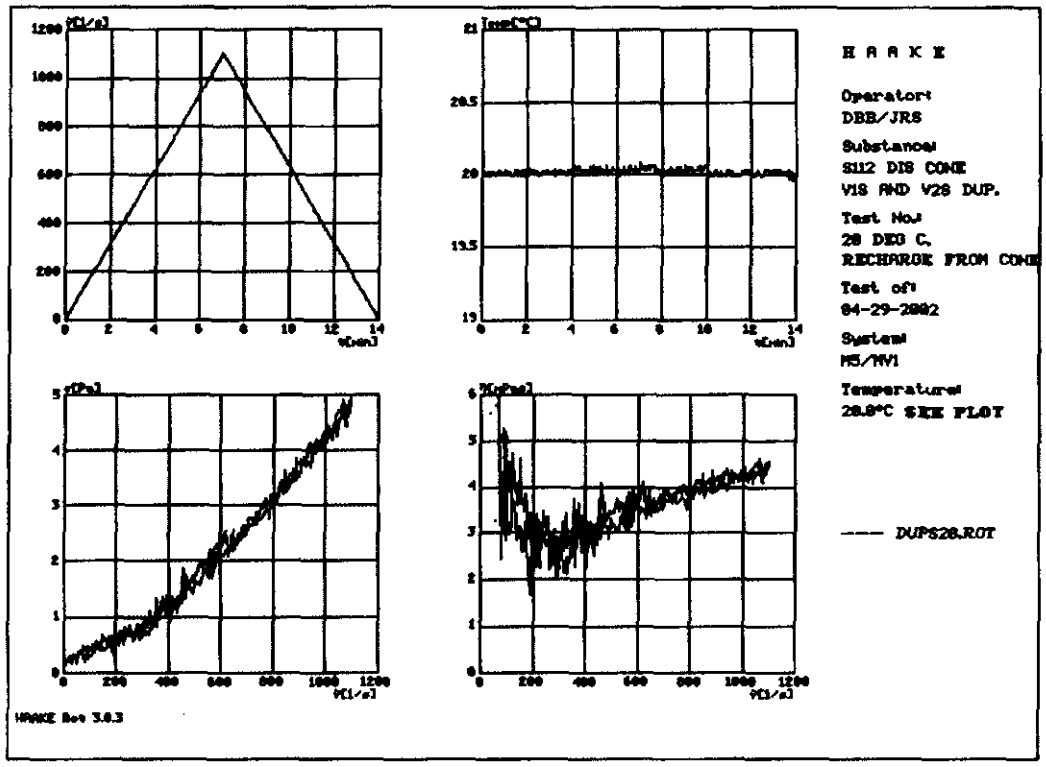


Figure A-10. Rheology Figures for Sample V1S/V2S returned to 20 °C



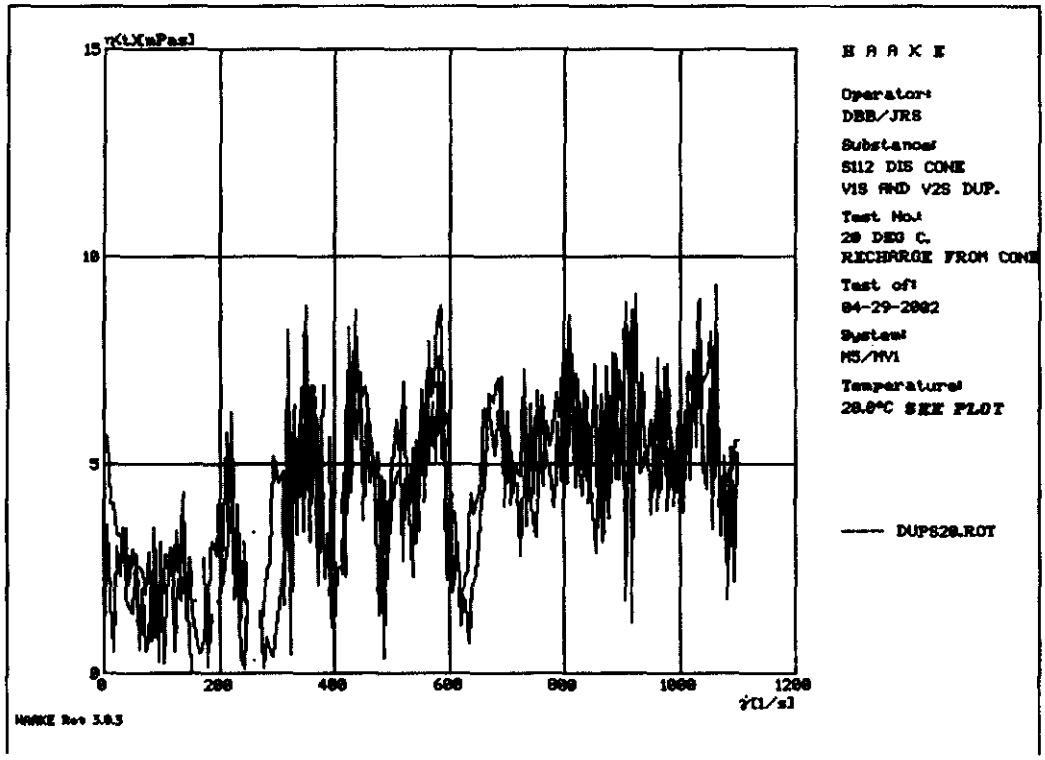


Figure A-11. Rheology Figures for Sample V1S/V2S Dup at 20 °C

Appendix B

Distribution by Fraction

Table B-1. Distribution by Fraction, Core 291, 22 °C, H_2O Diluent (Test A)
(original undiluted sample weight 59.4 g)
(chemicals in grams, radionuclides in μCi)

Analyte	Liquid Samples						Residual Solids[c]	Total	Percent Dissolved
	1st Contact	2nd Contact	3rd Contact	4th Contact	5th Contact	6th Contact			
F^-	0[b]	0	0	0.006	0.0001	0	not analyzed	0.006	100[a]
Cl^-	0.024	0.024	0.007	0.002	0	0		0.057	100[a]
NO_2^-	0.213	0.205	0.068	0.017	0	0		0.503	100[a]
NO_3^-	2.44	7.38	9.75	6.61	0.15	0.004		26.33	100[a]
PO_4^{3-}	0.044	0.129	0.102	0.064	0.001	0	0	0.340	100
SO_4^{2-}	0.167	0.559	0.134	0.020	0.001	0.001	0	0.881	100
$C_2O_4^{2-}$	0.002	0.010	0.013	0.047	0.055	0.001	0.002	0.130	99
CO_3^{2-}	1.06	1.11	0.31	0.035	0.001	0.001	0.008	2.52	99.7
Al	0.143	0.081	0.025	0.003	0.000	0.000	0.021	0.274	92
Cr	0.049	0.032	0.011	0.001	0.000	0.000	0.024	0.116	80
Fe	0	0	0	0	0.00014	0	0.0054	0.0055	3
K	0.010	0.009	0	0	0	0	na	0.019	100[a]
Na	3.23	4.56	5.27	2.88	0.092	0.005	0.052	16.09	99.7
P	0.024	0.047	0.046	0.015	0.000	0.000	0	0.133	100
S	0.099	0.201	0.062	0.008	0.000	0.000	0	0.370	100
Si	0.013	0.007	0.002	0	0.0001	0.0002	0.062	0.084	27
U	0	0	0	0	0	0	0.008	0.008	0
$^{89/90}Sr$	4.6	34.8	14.7	1.7	0.7	1.0	952	1009	6
^{99}Tc	1.02	0.64	0.21	0	0	0	0	1.87	100
^{137}Cs	941	598	178	23	5	3	0	1748	100

[a]100% dissolution assumed for these components; residual solids not analyzed by IC.
[b]An entry of '0' indicates analytical result was below detection limit.
[c]Based on fusion digest of undiluted composite sample [Prilucik 2002] minus sum of liquid fractions.

Table B-2. Distribution by Fraction, Core 292, 22 °C, H_2O Diluent (Test B)
(original undiluted sample weight 57.9 g)
(chemicals in grams, radionuclides in μCi)

Analyte	Liquid Samples						Residual Solids[c]	Total	Percent Dissolved
	1st Contact	2nd Contact	3rd Contact	4th Contact	5th Contact	6th Contact			
F^-	0[b]	0	0.005	0.006	0	0	not analyzed	0.011	100[a]
Cl^-	0.068	0.031	0.008	0.002	0	0.0003		0.109	100[a]
NO_2^-	0.626	0.289	0.069	0.009	0	0.0001		0.994	100[a]
NO_3^-	4.99	6.52	2.18	0.286	0.011	0.001		13.99	100[a]
PO_4^{3-}	0.093	0.099	0.086	0.045	0.002	0.0003	0	0.325	100
SO_4^{2-}	0.239	0.533	1.334	0.564	0.022	0.001	0	2.692	100
$C_2O_4^{2-}$	0.004	0.006	0.009	0.184	0.215	0.007	0.041	0.467	91
CO_3^{2-}	1.23	2.49	2.53	0.544	0.026	0	0.009	6.825	99.9
Al	0.363	0.140	0.034	0.005	0.001	0.001	0.016	0.522	97
Cr	0.199	0.087	0.022	0.003	0	0	0.038	0.350	89
Fe	0	0	0	0	0.0003	0.0003	0.031	0.032	2
K	0.019	0	0	0	0	0	na	0.019	100
Na	4.924	5.883	4.095	0.960	0.153	0.009	0.033	16.06	99.8
P	0.038	0.043	0.037	0.019	0.001	0	0	0.139	100
S	0.103	0.226	0.569	0.200	0.009	0.001	0	1.107	100
Si	0.010	0.005	0.001	0.0003	0.0001	0.0004	0.058	0.075	22
U	0	0	0	0.0008	0.0007	0.0005	0.035	0.037	5
$^{89/90}Sr$	3.50	13.09	42.84	15.90	1.85	13.33	1762	1853	5
^{99}Tc	2.30	1.01	0.25	0.04	0	0	0.16	3.76	96
^{137}Cs	2098	901	216	35	5	2	7	3264	99.8

[a]100% dissolution assumed for these components; residual solids not analyzed by IC.
[b]An entry of '0' indicates analytical result was below detection limit.
[c]Based on fusion digest of undiluted composite sample [Prilucik 2002] minus sum of liquid fractions.

Table B-3. Distribution by Fraction, Core 292, 22 °C, H_2O Diluent (Test C)
(original undiluted sample weight 61.8 g)
(chemicals in grams, radionuclides in μCi)

Analyte	Liquid Samples						Residual Solids[c]	Total	Percent Dissolved
	1st Contact	2nd Contact	3rd Contact	4th Contact	5th Contact	6th Contact			
F^-	0[b]	0	0	0.010	0.0008	0	not analyzed	0.011	100[a]
Cl^-	0.061	0.029	0.009	0.002	0.001	0.0002		0.103	100[a]
NO_2^-	0.581	0.272	0.082	0.015	0.001	0		0.951	100[a]
NO_3^-	4.99	6.15	3.18	0.535	0.048	0.002		14.90	100[a]
PO_4^{3-}	0.108	0.102	0.080	0.094	0.005	0.0005	0	0.389	100
SO_4^{2-}	0.259	0.652	1.488	1.165	0.094	0.003	0	3.661	100
$C_2O_4^{2-}$	0	0.007	0.007	0.092	0.368	0.013	0.044	0.530	92
CO_3^{2-}	1.51	2.22	2.20	1.54	0.137	0.001	0.009	7.617	99.9
Al	0.343	0.142	0.041	0.008	0.001	0.001	0.017	0.552	97
Cr	0.207	0.088	0.026	0.006	0.001	0.0001	0.040	0.368	89
Fe	0	0	0	0	0.0004	0.0003	0.033	0.034	2
K	0.021	0	0	0	0	0	na	0.021	100[a]
Na	5.62	5.78	4.03	2.09	0.358	0.014	0.036	17.93	99.8
P	0.045	0.042	0.034	0.034	0.002	0.0002	0	0.158	100
S	0.123	0.217	0.496	0.388	0.031	0.001	0	1.257	100
Si	0.011	0.008	0.002	0.0004	0.0001	0.0005	0.059	0.080	27
U	0	0	0	0	0.004	0.0003	0.038	0.043	11
$^{89/90}Sr$	3.1	10.0	40.2	7.4	5.1	8.5	1903	1977	4
^{99}Tc	2.23	1.00	0.33	0	0.01	0	0.17	3.74	95
^{137}Cs	2174	794	250	61	8	2	8	3298	99.8

[a]100% dissolution assumed for these components; residual solids not analyzed by IC.
[b]An entry of '0' indicates analytical result was below detection limit.
[c]Based on fusion digest of undiluted composite sample [Prilucik 2002] minus sum of liquid fractions.

Table B-4. Distribution by Fraction, Core 291, 22 °C, 2M NaOH Diluent (Test D)
(original undiluted sample weight 59.3 g)
(chemicals in grams, radionuclides in μCi)

Analyte	Liquid Samples						Residual Solids[c]	Total	Percent Dissolved
	1st Contact	2nd Contact	3rd Contact	4th Contact	5th Contact	6th Contact			
F^-	0[b]	0	0	0	0	0	not analyzed	0	100[a]
Cl^-	0.032	0.024	0.009	0.001	0	0		0.066	100[a]
NO_2^-	0.290	0.214	0.083	0.019	0.0004	0		0.607	100[a]
NO_3^-	2.44	5.35	6.90	6.65	0.116	0.003		21.46	100[a]
PO_4^{3-}	0.054	0.081	0.114	0.106	0.039	0.001	0	0.394	100
SO_4^{2-}	0.155	0.39+4	0.480	0.055	0.001	0	0	1.085	100
$C_2O_4^{2-}$	0	0	0	0.013	0.037	0.036	0.002	0.088	98
CO_3^{2-}	0.501	1.392	0.530	0.074	0.008	0.004	0.008	2.517	99.7
Al	0.148	0.102	0.034	0.006	0.003	0.002	0.011	0.305	96
Cr	0.050	0.037	0.019	0.008	0.001	0.0002	0.020	0.134	85
Fe	0	0.0001	0.0001	0.0001	0.00005	0.00005	0.005	0.005	7
K	0.009	0.009	0.008	0.006	0.002	0.001	na	0.035	100
Na	2.88	4.75	4.92	4.25	1.27	1.13	0.006	19.21	99.97
P	0.014	0.028	0.036	0.036	0.026	0	0	0.138	100
S	0.045	0.142	0.171	0.022	0.001	0	0	0.380	100
Si	0.016	0.010	0.005	0.005	0.007	0.004	0.037	0.084	56
U	0	0	0	0	0.001	0.001	0.008	0.010	20
$^{89/90}Sr$	0.2	11.5	35.3	22.3	1.2	0.9	937	1008	7
^{99}Tc	0.85	0.82	0.22	0.05	0.01	0.01	0	1.96	100
^{137}Cs	937	811	242	38	12	1	0	2042	100

[a]100% dissolution assumed for these components; residual solids not analyzed by IC.
[b]An entry of '0' indicates analytical result was below detection limit.
[c]Based on fusion digest of undiluted composite sample [Prilucik 2002] minus sum of liquid fractions.

Table B-5. Distribution by Fraction, Core 292, 22 °C, 2M NaOH Diluent (Test E)
(original undiluted sample weight 61.2 g)
(chemicals in grams, radionuclides in μCi)

Analyte	Liquid Samples						Residual Solids[c]	Total	Percent Dissolved
	1st Contact	2nd Contact	3rd Contact	4th Contact	5th Contact	6th Contact			
F^-	0[b]	0	0	0.007	0.008	0	not analyzed	0.015	100[a]
Cl^-	0.054	0.028	0.012	0.007	0.003	0.002		0.105	100[a]
NO_2^-	0.536	0.282	0.113	0.038	0.006	0		0.975	100[a]
NO_3^-	4.03	5.03	4.59	1.57	0.166	0.012		15.39	100[a]
PO_4^{3-}	0.068	0.112	0.107	0.099	0.065	0.005	0	0.456	100
SO_4^{2-}	0.194	0.323	0.416	1.643	0.564	0.037	0	3.18	100
$C_2O_4^{2-}$	0	0.005	0.009	0.011	0.038	0.085	0.327	0.475	31
CO_3^{2-}	0.809	1.18	1.76	2.77	0.415	0.031	0.022	6.99	99.7
Al	0.272	0.141	0.056	0.021	0.006	0.004	0.039	0.539	93
Cr	0.165	0.086	0.036	0.014	0.002	0.001	0.093	0.397	76
Fe	0	0	0	0	0	0	0.033	0.033	0
K	0.018	0.010	0	0	0	0	na	0.028	100[a]
Na	4.58	5.16	5.15	6.05	1.92	1.51	0.083	24.46	99.7
P	0.023	0.032	0.031	0.046	0.027	0.002	0	0.160	100
S	0.072	0.117	0.151	0.740	0.214	0.014	0	1.310	100
Si	0.010	0.005	0.003	0.003	0.003	0.003	0.054	0.080	33
U	0	0	0	0	0	0.0018	0.0875	0.089	2
$^{89/90}Sr$	4.2	3.5	3.6	42.8	6.0	2.4	1927	1990	3
^{99}Tc	1.94	1.030	0.36	0.14	0.017	0.004	0.40	3.89	90
^{137}Cs	1805	894	360	121	21	7	18	3227	99.4

[a]100% dissolution assumed for these components; residual solids not analyzed by IC.
[b]An entry of '0' indicates analytical result was below detection limit.
[c]Based on fusion digest of undiluted composite sample [Prilucik 2002] minus sum of liquid fractions.

Table B-6. Distribution by Fraction, Core 291, 55 °C, H_2O Diluent (Test F)
(original undiluted sample weight 62.0 g)
(chemicals in grams, radionuclides in μCi)

Analyte	Liquid Samples						Residual Solids[c]	Total	Percent Dissolved
	1st Contact	2nd Contact	3rd Contact	4th Contact	5th Contact	6th Contact			
F^-	0[b]	0	0	0	0	0	not analyzed	0	100[a]
Cl^-	0.044	0.016	0.008	0	0	0.0003		0.069	100[a]
NO_2^-	0.406	0.156	0.038	0.002	0.001	0		0.603	100[a]
NO_3^-	7.36	9.67	8.90	0.370	0.296	0.001		26.59	100[a]
PO_4^{3-}	0.212	0.114	0.043	0.001	0	0	0	0.370	100
SO_4^{2-}	0.139	0.197	0.640	0.038	0.003	0.001	0	1.019	100
$C_2O_4^{2-}$	0	0	0	0.076	0	0	0.003	0.079	97
CO_3^{2-}	0.760	1.158	0.485	0.033	0.007	0.008	0.012	2.46	99.5
Al	0.171	0.063	0.013	0.001	0.001	0.0002	0.038	0.286	87
Cr	0.057	0.021	0.004	0.0002	0	0	0.038	0.122	68
Fe	0	0	0	0.0002	0.0001	0	0.0055	0.0058	4
K	0.014	0.008	0	0	0.0001	0.0001	na	0.022	100[a]
Na	4.99	5.30	4.06	0.239	0.012	0.002	0.009	14.60	99.94
P	0.080	0.042	0.010	0.0002	0.0001	0	0	0.132	100
S	0.054	0.066	0.191	0.015	0.001	0.001	0	0.328	100
Si	0.013	0.005	0.001	0.0002	0.0001	0.0001	0.067	0.088	24
U	0	0	0	0.0008	0.0001	0	0.012	0.013	6
$^{89/90}Sr$	2.2	5.5	5.5	1.2	0.9	1.9	1038	1055	2
^{99}Tc	1.34	0.50	0.09	0.01	0	0	0	1.94	100
^{137}Cs	1293	432	93	8	6	2	0	1865	100

[a]100% dissolution assumed for these components; residual solids not analyzed by IC.
[b]An entry of '0' indicates analytical result was below detection limit.
[c]Based on fusion digest of undiluted composite sample [Prilucik 2002] minus sum of liquid fractions.

Table B-7. Distribution by Fraction, Core 292, 55 °C, H_2O Diluent (Test G)
(original undiluted sample weight 61.4 g)
(chemicals in grams, radionuclides in μCi)

Analyte	Liquid Samples						Residual Solids[c]	Total	Percent Dissolved
	1st Contact	2nd Contact	3rd Contact	4th Contact	5th Contact	6th Contact			
F^-	0.012	0.016	0[b]	0	0	0	not analyzed	0.028	100[a]
Cl^-	0.069	0.030	0.010	0.002	0.001	0.0004		0.112	100[a]
NO_2^-	0.710	0.240	0.076	0.013	0.001	0		1.039	100[a]
NO_3^-	8.18	5.33	2.02	0.335	0.014	0.001		15.88	100[a]
PO_4^{3-}	0.185	0.153	0.041	0.014	0.001	0	0	0.394	100
SO_4^{2-}	0.144	0.287	1.03	1.89	0.061	0.005	0	3.42	100
$C_2O_4^{2-}$	0	0	0	0.113	0.272	0.017	0.054	0.456	88
CO_3^{2-}	0.702	1.62	3.35	1.51	0.085	0.011	0.012	7.29	99.8
Al	0.302	0.101	0.036	0.007	0.001	0.001	0.046	0.493	91
Cr	0.181	0.063	0.023	0.005	0.001	0.0001	0.057	0.330	83
Fe	0	0	0	0.0006	0.0002	0	0.034	0.035	3
K	0.021	0.010	0	0	0	0	na	0.031	100[a]
Na	5.33	3.77	3.78	2.17	0.255	0.014	0.044	15.37	99.7
P	0.070	0.046	0.017	0.003	0	0	0	0.137	100
S	0.046	0.098	0.301	0.587	0.025	0.002	0	1.059	100
Si	0.009	0.004	0.001	0.0004	0.0002	0.0004	0.065	0.080	19
U	0	0	0	0.011	0.003	0.0001	0.046	0.061	23
$^{89/90}Sr$	1.9	6.7	27.2	45.3	2.1	4.6	1878	1966	4
^{99}Tc	2.42	0.79	0.29	0.05	0	0	0.21	3.77	94
^{137}Cs	2211	725	263	50	8	3	10	3269	99.7

[a]100% dissolution assumed for these components; residual solids not analyzed by IC.
[b]An entry of '0' indicates analytical result was below detection limit.
[c]Based on fusion digest of undiluted composite sample [Prilucik 2002] minus sum of liquid fractions.

Table B-8. Percent Removed by Fraction, Core 291, 22 °C, H_2O Diluent (Test A)
(PO_4^{3-} and SO_4^{2-} based on ICP results in Tables B-8 through B-14)

Wt% Dilution	25	50	79	117	157	208
Cl^-	42	83	96	99	100	100
NO_2^-	42	83	97	100	100	100
NO_3^-	9	37	74	99	100	100
PO_4^{3-}	18	53	88	100	100	100
SO_4^{2-}	27	81	98	100	100	100
$C_2O_4^{2-}$	2	9	19	55	98	99
CO_3^{2-}	42	86	98	99.6	99.6	99.7
Al	52	82	91	92	92	92
Cr	42	69	79	80	80	80
Na	20	48	81	99	99.6	99.7
Si	16	24	27	27	27	27
$^{89/90}Sr$	0	4	5	6	6	6
^{99}Tc	54	89	100	100	100	100
^{137}Cs	54	88	98	100	100	100

Table B-9. Percent Removed by Fraction, Core 292, 22 °C, H_2O Diluent (Test B)

Wt% Dilution	24	51	77	117	157	209
Cl^-	62	91	98	100	100	100
NO_2^-	63	92	99	100	100	100
NO_3^-	36	82	98	100	100	100
PO_4^{3-}	28	59	86	99	100	100
SO_4^{2-}	9	30	81	99	100	100
$C_2O_4^{2-}$	1	2	4	44	90	91
CO_3^{2-}	18	54	92	99	99.9	99.9
Al	63	89	96	97	97	97
Cr	57	82	88	89	89	89
Na	31	67	93	99	99.7	99.8
Si	14	20	21	22	22	22
$^{89/90}Sr$	0	1	3	4	4	5
^{99}Tc	61	88	95	96	96	96
^{137}Cs	64	92	99	99.6	99.7	99.8

Table B-10. Percent Removed by Fraction, Core 292, 22 °C, H_2O Diluent (Test C)

Wt% Dilution	26	49	73	108	144	192
Cl^-	59	88	97	99	100	100
NO_2^-	61	90	98	100	100	100
NO_3^-	33	75	96	100	100	100
PO_4^{3-}	29	55	77	98	100	100
SO_4^{2-}	10	27	67	97	100	100
$C_2O_4^{2-}$	0	1	3	20	90	92
CO_3^{2-}	20	49	78	98	99.9	99.9
Al	62	88	95	97	97	97
Cr	57	81	88	89	89	89
Na	31	64	86	98	99.7	99.8
Si	13	23	26	26	26	27
$^{89/90}Sr$	0	1	3	3	3	4
^{99}Tc	60	87	95	95	95	95
^{137}Cs	66	90	98	99	99.7	99.8

Table B-11. Percent Removed by Fraction, Core 291, 22 °C, 2M NaOH Diluent (Test D)

Wt% Dilution	27	54	83	129	170	226
Cl^-	48	84	98	100	100	100
NO_2^-	48	83	97	100	100	100
NO_3^-	11	36	68	99	100	100
PO_4^{3-}	10	30	56	81	100	100
SO_4^{2-}	12	49	94	99.8	100	100
$C_2O_4^{2-}$	0	0	0	15	57	98
CO_3^{2-}	20	75	96	99	99.5	99.7
Al	49	82	93	95	96	96
Cr	37	64	78	85	85	85
Na	15	40	65	87	94	100
Si	19	31	37	43	50	56
$^{89/90}Sr$	0	0	3	6	7	7
^{99}Tc	44	85	96	99	99	100
^{137}Cs	46	86	98	99	100	100

Table B-12. Percent Removed by Fraction, Core 292, 22 °C, 2M NaOH Diluent (Test E)

Wt% Dilution	27	52	77	115	152	205
Cl^-	51	78	89	95	98	100
NO_2^-	55	84	95	99	100	100
NO_3^-	26	59	89	99	100	100
PO_4^{3-}	14	34	53	82	99	100
SO_4^{2-}	5	14	26	83	99	100
$C_2O_4^{2-}$	0	1	3	5	13	31
CO_3^{2-}	12	29	54	93	99	99.7
Al	53	80	90	91	92	93
Cr	48	73	74	75	76	76
Na	19	40	61	86	94	99.7
Si	13	19	23	26	29	33
$^{89/90}Sr$	0	0	1	3	3	3
^{99}Tc	53	81	88	89	89	90
^{137}Cs	56	84	95	99	99.4	99.4

Table B-13. Percent Removed by Fraction, Core 291, 55 °C, H_2O Diluent (Test F)

Wt% Dilution	25	46	61	96	132	179
Cl^-	64	88	99	99	100	100
NO_2^-	67	93	99.5	100	100	100
NO_3^-	28	64	97	99	100	100
PO_4^{3-}	61	92	100	100	100	100
SO_4^{2-}	16	37	95	99.5	99.8	100
$C_2O_4^{2-}$	0	0	0	97	97	97
CO_3^{2-}	31	78	98	99	99	99.5
Al	60	82	86	87	87	87
Cr	47	65	68	68	68	68
Na	34	70	98	99.9	99.9	99.9
Si	15	21	23	23	23	24
$^{89/90}Sr$	0	1	1	1	1	2
^{99}Tc	69	95	99.6	100	100	100
^{137}Cs	69	94	99	99.5	99.9	100

Table B-14. Percent Removed by Fraction, Core 292, 55 °C, H_2O Diluent (Test G)

Wt% Dilution	22	42	65	99	136	186
Cl^-	62	89	97	99	100	100
NO_2^-	68	91	99	100	100	100
NO_3^-	52	85	98	100	100	100
PO_4^{3-}	52	85	98	100	100	100
SO_4^{2-}	4	14	42	97	100	100
$C_2O_4^{2-}$	0	0	0	25	84	88
CO_3^{2-}	10	32	78	99	99.7	99.8
Al	61	82	89	90	91	91
Cr	55	74	81	83	83	83
Na	35	59	84	98	99.6	99.7
Si	12	16	18	18	19	19
$^{89/90}Sr$	0	0	2	4	4	4
^{99}Tc	65	85	92	94	94	94
^{137}Cs	68	90	98	99	99.7	99.7