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THE ACTINIDE SOURCE-TERM WASTE TEST PROGRAM (STTP)

Final Report

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VOLUME II

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Selection of Wastes for the Actinide Source-Term Waste Test Program (STTP)

I. Selection of Wastes

The technical requirements for the STTP were detailed in the Sandia National Laboratories document, "Technical Requirements for the Contact-Handled Transuranic Waste Source-Term Test Program," SAND91-2111. The primary criteria for waste to be selected for the STTP tests were determined by the test matrix for the STTP and the additives added to specific liter-scale and drum-scale tests. The test matrix is given in Table 1 and Table 2 of section III. (a). The principal criteria applied to all tests were as follows:

1) For the 15 drum-scale tests, the entire contents of a standard 55-gallon waste drum of a specified Transuranic content code (TRUCON code) must be used for each of the drum-scale tests;

2) For the 39 liter-scale tests, a portion of the homogeneous (Solidified, sludge, or salt) waste must be obtained from a different drum for each test. That means that 39 waste drums must be sampled so that each test will have a unique waste and no test will be from a single waste drum; and

3) Each waste drum used for the drum-scale tests and sampled for the liter-scale tests must have at least 5 grams of ^{239}Pu. Five grams of ^{239}Pu per 210 liter of brine will give a concentration of 2.38×10^{-2} gm/L or approximately 1×10^{-4} M solution of ^{239}Pu if the Pu were to dissolve completely.

The Waste drums containing the available TRUCON codes were selected from the inventory of waste drums already present at Los Alamos National Laboratory. The wastes were located at TA-54, TA-55, and CMR Building. A preliminary list of waste drums that met the TRUCON code requirements and the 5 gm specification was devised and a final selection was made based on TRUCON code, ^{239}Pu content, and access to the waste drums.

Loading of Test Containers for the Actinide Source-Term Waste Test Program (STTP)

II. (a) Loading of Test Containers: Introduction

This section documents the method used to prepare, package, and label the actinide isotopes requested for the WIPP Actinide Source-Term Waste Test Program (STTP). The following table summarizes the sources that were requested as well as the quantity of each nuclide needed.

Table 1. Number of Containers Containing Each Actinide

Number of Containers	^{232}Th	^{238}U	^{237}Np	^{241}Am
15	5g	5g	5g	0
41	75mg	75mg	75mg	0

The original actinide starting materials include:

1) High purity depleted uranium metal, which had a purity of about 100.0%. The metal's I.D. is CLS-1-LC314.

2) Reagent grade (96.2% Th $(NO_3)_4 \bullet 4H_2O$) thorium nitrate from Baker and Adamson Products, General Chemical Division, Allied Chemical Corporation.

3) High purity (88.11% Np) neptunium oxide from sample NMT-3-ERP6947CF2S.

4) High purity (99.26% Np) neptunium metal from sample NMT-3-ERP6749T2BC.

In some cases it was not possible to prepare the requested level of material in the container because of limitations caused by the total amount of starting material in possession.

Each of the starting actinide material was treated as follows:

1) Two thorium nitrate master solutions were prepared. An accurately weighed portion of thorium nitrate was dissolved in water for each solution. The lower concentrated solution was filtered into a calibrated 100ml volumetric flask. The higher concentrated solution was filtered into a calibrated 250ml volumetric flask. Using a "Rainin" pipettor, equal portions of about 75mg of the lower concentrated solution were added to 41, 5 dram glass vials having pop-top caps, and equal portions of about 5g were added to 15, 4oz wide mouth, screw cap jars.

Two portions of each master solution were sent for analysis by IDMS (Isotope Dilution Mass Spectrometry). The concentration as determined by IDMS was used to calculate the thorium content for each master solution.

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2) Two uranium master solutions were prepared. Uranium metal was dissolved in concentrated nitric acid with a trace of hydrofluoric acid. The lower concentration solution was transferred to a calibrated 100ml volumetric flask while the higher concentration solution was transferred to a calibrated 250ml volumetric flask. Using a "Rainin" pipettor, equal portions of about 75mg of the lower concentrated solution were added to 5-dram vials containing the 75mg of thorium. About 5g of the higher concentrated uranium solution were added to the 15, 4oz jars containing the 5g of thorium. Two portions of each master solution were sent for analysis by IDMS (Isotope Dilution Mass Spectrometry). The concentration as determined by IDMS was used to calculate the uranium content of each master solution.

3) Accurately weighed portions of neptunium oxide were dissolved using the sealed-reflux dissolution system (LA-5776). The resulting solutions were combined into a calibrated 100ml volumetric flask. Using a "Rainin" pipettor, equal portions of about 70mg were added to the same 5-dram vials containing the thorium and uranium. With all the actinides added to the vials, the solutions were left to air dry to near dryness. The neptunium concentration was determined by coulometry.

4) Fifteen accurately weighed portions of neptunium metal, each weighing 5.00g ± 0.05g were dissolved in a 100ml beaker using concentrated hydrochloric acid and hydrofluoric acid. The resulting solutions were quantitatively transferred with water to the 15 jars containing the 5g portions of thorium and uranium. The solutions were allowed to air dry to near dryness.

Table 2 shows the amount of thorium, uranium, and neptunium in each 5-dram container.

Table 2. Amount of Neptunium, Thorium, and Uranium in Each Source Container

Container No.	$^{*232}Th$	$^{*238}U$	$^{**237}Np$
NTU75-1 to 41	78.9mg	80.4mg	65.2mg

* The calculations for this value are found in LA notebook #5768, page 67.

** The calculations for this value are found in LA notebook #5768, page 68.

Table 3 shows the amount of neptunium, thorium, and uranium in each 4oz jar.

Table 3. Amount of Neptunium, Thorium, and Uranium in Each Source Container

Container No.	$^{*237}Np$	$^{**232}Th$	$^{**238}U$
NTU 5-1 to 15	4.97g	4.77g	4.97g

* The calculations for these values are found in LA notebook #5768, page 75.

** The calculations for these values are found in LA notebook #5768, pages 71-73.

II. (b) Loading of Liter-Scale Test Containers

The loading of the liter-scale test containers occurred at the LANL Waste Characterization, Reduction, and Repackaging Facility. The Pyrochemical salt waste, including MgO-Y_2O_3 crucible, chloride salts, and calcium oxide, was comminuted at LANL's TA-55. Added to the titanium test vessels was 800 to 1,340g of the Pyrochemical salt waste. Figure 1 is a flowchart of the process used for loading the liter-scale test containers (refer to page 9 for Figure 1).

Listed below are summaries of the appropriate influencing variables for the Pyrochemical salt test containers.

L25, 26, and 27:

Comminuted waste: 1,338g; 1,315g; 1,315g
Fe mesh: ~110g
Nd: 45mg; Th, U, and Np: 75mg each
Brine: solid ratio = 2:1

L28, 29, and 30: (pressurized)

Comminuted waste: 907g; 907g; 930g
Fe mesh: ~55g
Nd: 30mg; Th, U, and Np: 75mg each
Brine: solid ratio = 2:1
CO_2 at 60 bars (870 psig)

L31, 32, and 33:

Comminuted waste: 1,315g; 1,315g, 1,338g
Fe mesh: ~110g
Nd: 45mg; Th, U, and Np: 75mg each
Brine: solid ratio = 2:1
Bentonite: brine equilibrated; 120g each

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L34, 35, and 36:

Comminuted waste: 862g; 885g; 885g
Fe mesh: ~110g
Nd: 45mg, Th, U, and Np: 75mg each
Brine: solid ratio = 3:1

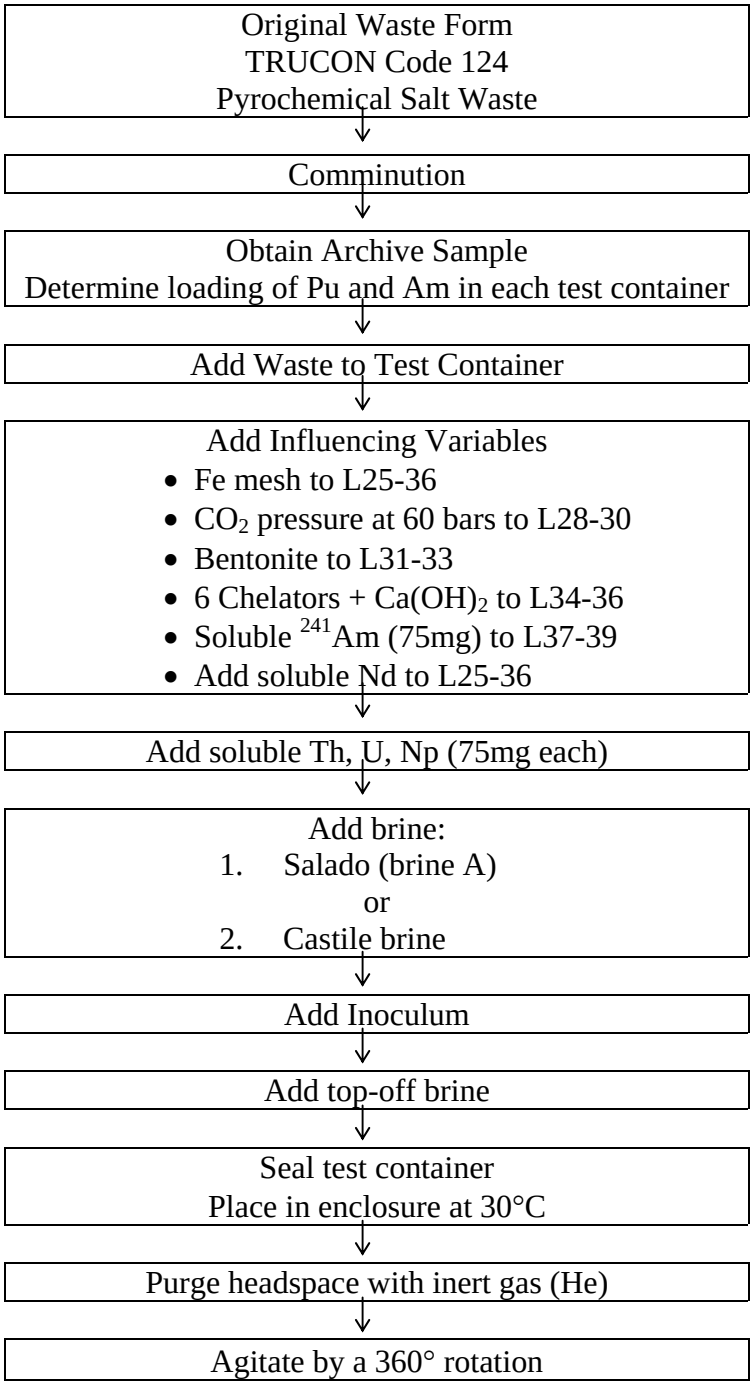
Chelators added:	Acetamide	100ppm
	Sodium acetate	139ppm
	Ascorbic acid	101ppm
	Trisodium citrate dihydrate	154ppm
	Oxalic acid dihydrate	143ppm
	Ammonium thiocyanate	148ppm

$Ca(OH)_2$: 96g each

L37, 38, and 39:

Comminuted waste: 1,315g; 1,338g, 1,338g
Fe mesh: none
Nd: 45mg; Th, U, and Np: 75mg each
Brine: solid ratio = 2:1
^{241}Am added: 75mg as soluble salt

Figure 1.
Flowchart of process used for loading Pyrochemical salt tests.



After loading the test containers with all the influencing variables and Brine A or Castile brine, the containers were transferred to the Chemistry and Metallurgy Research (CMR) nuclear facility. At the CMR approximately 100-150cc of inoculum containing microbes from the WIPP environs were added.

The inoculum consisted of WIPP brine combined with anaerobic brine-containing sediments from around the WIPP. The brine extract from a combination of WIPP brine and WIPP sediments were allowed to settle, then decanted and added to all the test containers. A portion of top-off brine was added to each test container to adjust total volume, leaving a headspace volume of 5 to 12%. The inoculum used for the STTP contained organic colloids, organisms, and microorganisms. The inoculum was maintained in an anaerobic condition. The inoculum was expected to represent an initial reducing condition to the test containers.

After the test containers were loaded with all the additives including inoculum and brine, they were sealed to maintain the anaerobic condition for the length of the testing. Any oxic conditions that might develop from radiolysis would have to overcome the initial reducing condition of the test container's contents.

II. (c) Loading of Drum-Scale Test Containers

The STTP Drum-Scale Test containers will each contain the contents of a single waste drum and several additives as illustrated in Figures 2 and 3 (pages 12 and 13). There will be 15 drum-scale test containers; twelve to be loaded with TRUCON Code 116/216 (combustibles) waste types and three will be loaded with TRUCON Code 117/217 (metals) waste types. Two drum-scale test containers will be designated as QA/QC blank Brine A and Castile Brine containers.

Each set of triplicate experiments will include two drum-scale test containers with Brine A and the third test container will be filled with Castile Brine. Nonradioactive additives will be added prior to attachment of the DS test containers to the Waste Characterization, Reduction, and Repackaging Facility (WCRRF) glovebox. The quantities of actinides to be added are given in the following table (Table 4):

Table 4. Loading of STTP Drum-Scale Test Containers

TRUCON Code 116[*] (Combustibles)

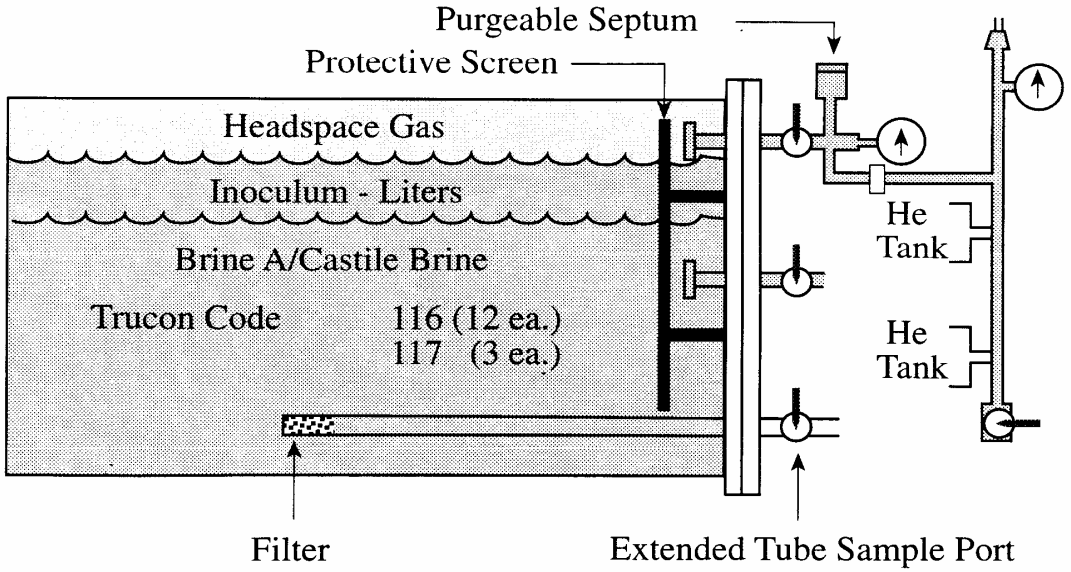
Test Container	*Brine Type*	*Additives*
D1	Brine A	
D2	Brine A	
D3	Castile Brine	
D4	Brine A	10kg Brine – Equilibrated Bentonite
D5	Brine A	10kg Brine – Equilibrated Bentonite
D6	Castile Brine	10kg of Non-Equilibrated Bentonite
D7	Brine A	Chelating Agents
D8	Brine A	Chelating Agents
D9	Castile Brine	Chelating Agents
D10	Brine A	$NaNO_3/NaH_2PO_4$
D11	Brine A	$NaNO_3/NaH_2PO_4$
D12	Castile Brine	$NaNO_3/NaH_2PO_4$

TRUCON Code 117[**]

D13	Brine A	Metals
D14	Brine A	Metals
D15	Castile Brine	Metals
D16	Brine A	Blank
D17	Castile Brine	Blank

[*] Fe mesh and added actinides (D1 – D5)
[**] $NdCl_3$ added (D1 – D17)

Figure 2.
Layout of Loaded Drum-Scale Test Container



Given: Volume = 65 gal = 246 liters
Headspace Gas Volume = 12% - Inoculum = 29.5 L - Inoculum
Inoculum = ~5% (~12.3 L)
Min Free Volume > 40 gal = > 151.4 liters

Neodymium chloride (D1 thru D17)

Add 6.42 gm $NdCl_3$ per each DS test container
Add 6.42 gm $NdCl_3$ to 2 ea QA DS test containers

Chelators (D7,D8,D9)

Add low-MW major chelators 100-200 mg/L
Add High-MW minor chelators 30-50 mg/L

Brine-Equilibrated Bentonite

Add 10 kg Brine A - Equilibrated Bentonite D4, D5
Add 120 kg of non-equilibrated Bentonite to D6

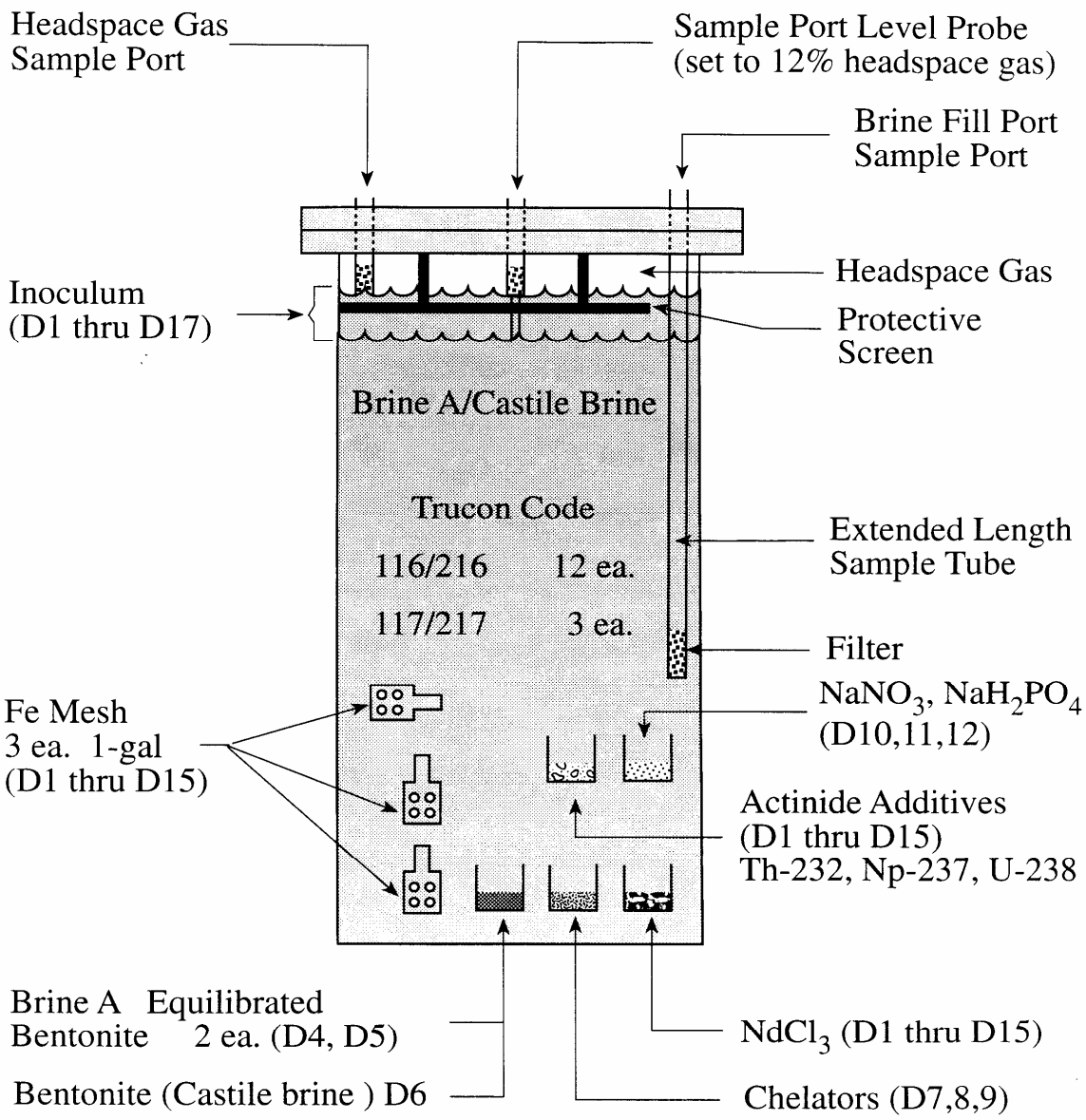
Microbial Fertilizer

Add 0.01 M $NaNO_3$ to D10, D11, D12
Add 0.01 M $NaH_2PO_4 \cdot H_2O$ to D10, D11, D12

Actinides

Add ~ 5gm Th-232, U-238, and Np-237 to D1 thru D15

Figure 3.
Illustration of STTP Drum-Scale Test Container Loading



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II. (d) Calculation of Concentration of Chelators in STTP Liter and Drum-Scale Test Containers

The following chelators were added to three liter-scale and three drum-scale test containers.

Liter-scale test containers with added chelators and 96.2 gm of $Ca(OH)_2$ are:

L-34 Oxygen sparging Pyrochemical salts
L-35 Direct oxide reduction Pyrochemical salts
L-36 Direct oxide reduction Pyrochemical salts

The concentration of chelators added to the liter-scale test containers assume a brine volume of 2000 ml.

The drum-scale containers with added chelators are D7, D8, and D9, which are loaded with TRUCON Code 116/216, combustibles. The concentration of chelators added to the drum-scale test containers assume a brine volume of 200L.

Chelator	***LS 34 (mg)***	***LS 35 (mg)***	***LS 36 (mg)***
Acetamide	200	200	202
Sodium Acetate	277	276	277
Trisodium Citrate Dihydrate	308	307	311
Oxalic Acid Dihydrate	285	288	286
Ascorbic Acid	201	202	198
Ammonium Thiocyanate	295	294	295

Chelator	***D 7 (mg)***	***D 8 (mg)***	***D 9 (mg)***
Acetamide	30.2	30.2	30.3
Sodium Acetate	42.0	42.0	42.0
Trisodium Citrate Dihydrate	46.6	46.6	46.6
Oxalic Acid Dihydrate	43.3	43.3	43.3
Ammonium Thiocyanate	44.4	44.4	44.4

Calculations for Liter-Scale Test Containers

Acetamide

$$\frac{(200\ \text{mg})\,(1000\ \text{ug/mg})}{2000\ \text{ml}} = 100\ \text{ppm}$$

Sodium Acetate

$$\frac{(277\ \text{mg})\,(1000\ \text{ug/mg})}{2000\ \text{ml}} = 139\ \text{ppm}$$

Trisodium Citrate Dihydrate

$$\frac{(308\ \text{mg})\,(1000\ \text{ug/ml})}{2000\ \text{ml}} = 154\ \text{ppm}$$

Oxalic Acid Dihydrate

$$\frac{(285\ \text{mg})\,(1000\ \text{ug/mg})}{2000\ \text{ml}} = 143\ \text{ppm}$$

Ascorbic Acid

$$\frac{(201\ \text{mg})\,(1000\ \text{ug/mg})}{2000\ \text{ml}} = 101\ \text{ppm}$$

Ammonium Thiocyanate

$$\frac{(295\ \text{mg})\,(1000\ \text{ug/mg})}{2000\ \text{ml}} = 148\ \text{ppm}$$

Calculations for Drum-Scale Test Containers

Acetamide

$$\frac{(30.2\ \text{gm})\,(1000\ \text{mg/gm})}{200\ \text{L}} = 151\ \text{ppm}$$

Sodium Acetate

$$\frac{(42.0\ \text{gm})\,(1000\ \text{mg/gm})}{200\ \text{L}} = 210\ \text{ppm}$$

Trisodium Citrate Dihydrate

$$\frac{(46.6\ \text{gm})\,(1000\ \text{mg/gm})}{200\ \text{L}} = 233\ \text{ppm}$$

Oxalic Acid Dihydrate

$$\frac{(43.3\ \text{gm})\,(1000\ \text{mg/gm})}{200\ \text{L}} = 217\ \text{ppm}$$

Ammonium Thiocyanate

$$\frac{(44.4\ \text{gm})\,(1000\ \text{mg/gm})}{200\ \text{L}} = 222\ \text{ppm}$$

II. (e) Addition of Influencing Variables

Addition of Actinides to STTP Test Containers

The waste streams added to the STTP test containers were all from the Los Alamos National Laboratory inventory of wastes. The criteria for waste drums to be used for the STTP matrix is that each waste drum must contain at least 5 grams of Pu. ^{241}Am should accompany the total Pu but a criteria is not established for Am. There is no criteria to limit the quantity of ^{240}Pu and ^{241}Pu. There is no need to include waste containing ^{238}Pu because of the added difficulty in handling, sampling, and analyzing samples with the very high radioactivity levels of ^{238}Pu. The chemical effects expected to result from ^{238}Pu in the wastes will be induced by addition of about 75 mg of ^{241}Am to six different liter-scale test containers. Because the WIPP is expected to have wastes contaminated with Th, U, and Np, all the liter-scale and drum-scale tests are spiked with soluble salts of these actinides. Neodymium was also added to select test containers as a chemical analogue or surrogate for ^{241}Am.

The following is a list and description of the added actinides and neodymium:

Neodymium

Neodymium was added as $NdCl_3$ directly to the test containers at the WCRRF. The glass vial containing the $NdCl_3$ was not added to the test container.

Thorium

Thorium was added as a water solution of $Th(NO_3)_4 \bullet 4H_2O$ to a glass vial containing other actinides. The glass vial was placed in each test container. The Th was 99.99% pure and was present as ^{232}Th.

Uranium

Uranium was added as a nitric acid solution from a high purity specimen of depleted U metal (100%). The metal was dissolved in 16 N HNO_3 heated with a trace of HF. An aliquot of the U solution was placed in a glass vial containing other actinides. The glass vial was placed in each test container. The U was ^{238}U.

Neptunium

Neptunium was added as a Np salt solution after dissolution of NpO_2 by a sealed reflux dissolution system. Concentrated HCl with a few drops of HNO_3 and HF was used to dissolve the NpO_2. Aliquots of the dissolved oxide were added to glass vials containing other actinides. An aliquot of the solution was placed in a glass vial with other actinides. The glass vial was placed in each test container. The Np was ^{237}Np. The Np solution from the dissolved NpO_2 was added to each liter-scale test container, the Np added to the drum-scale tests was dissolved from specimens of Np metal. The metal was dissolved in concentrated HCl with a few drops of HNO_3 and HF. The 15 specimens of Np metal weighed 5 gm.

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Americium

Americium was added as an Am solution after dissolution of AmO_2 in 0.5 ml 12 N HCl with two drops 16 N HNO_3 and two drops of HF. An aliquot of the solution was placed in a glass vial and evaporated to damp dryness. Each of the six glass vials had a radiation reading of about 800 mR/hr β, γ at contact. The AmO_2 was about 82.2% Am and remainder Pu. The vials with Am were added to each select test container along with the vials containing the other actinides.

Table 5. Actinides Added to STTP Liter-Scale Test Containers

Test Containers	*Nd (mg)* *	*^{232}Th (mg)*	*^{238}U (mg)*	*^{237}Np (mg)*	*^{241}Am (mg)*
1, 2, 3	45	75	75	75	0
4, 5, 6	30	75	75	75	0
7, 8, 9	45	75	75	75	0
10, 11, 12	0	75	75	75	75
13, 14, 15	45	75	75	75	0
16, 17, 18	45	75	75	75	0
19, 20, 21	45	75	75	75	0
22, 23, 24	45	75	75	75	0
25, 26, 27	45	75	75	75	0
28, 29, 30	30	75	75	75	0
31, 32, 33	45	75	75	75	0
34, 35, 36	45	75	75	75	0
37, 38, 39	0	75	75	75	75

* Nd added as $NdCl_3$ for a concentration of about 15 mg/L for nonpressurized tests, assumption of 3L; 15 mg/L for pressurized tests, assumption of 2L.

Table 6. Actinides Added to STTP Drum-Scale Test Containers

Test Containers	*Nd (mg)* *	*^{232}Th (mg)*	*^{238}U (mg)*	*^{237}Np (mg)*	*^{241}Am (mg)*
1, 2, 3	3.69	5	5	5	0
4, 5, 6	3.69	5	5	5	0
7, 8, 9	3.69	5	5	5	0
10, 11, 12	3.69	5	5	5	0
13, 14, 15	3.69	5	5	5	0

* Nd added as $NdCl_3$ for a concentration of about 24.37 mg/L, assuming 151.4 L (40 gal).

Addition of Mixed Inoculum to STTP Test Containers

A batch of mixed inoculum was prepared for addition to each of the 39 liter-scale tests and 15 drum-scale tests. The inoculum consisted of a mixture of ingredients sampled from the WIPP site that contained a diverse mixture of halotolerant microorganisms that survive in the surfacial and supersaline conditions of the WIPP environments. The components making up the mixed inoculum batch are found in the table below.

Table 7. Components of the Mixed Inoculum Batch

Component	***Quantity %***	***Liters/drum***
Lake Brine and sediment mixture	20 %	1.06
Muck-pile salt solution	30 %	1.59
G-seep Brine	50 %	2.65
	Total	5.30

The inoculum solution was to be added to the test containers to establish and maintain an anaerobic environment. The anaerobic environment was established by purging the mixed inoculum for 30 minutes with N_2. The mixed inoculum was continually mixed and maintained under a N_2 atmosphere prior to addition to the liter-scale and drum-scale tests. About 100-150 ml of inoculum was added to the liter-scale tests, and about 5.3 liters was added to the drum-scale tests. For the pressurized liter-scale tests, 80-100 ml of inoculum was added to each of the 6 pressurized tests. The quantity of chemical reducing agents added as part of the inoculum injection was not known.

Addition of Fe to STTP Test Containers

An important parameter that could have a significant effect on the Redox potential in the repository is the presence of iron from the mild steel 55-gallon drums containing the waste. The STTP experiments were designed to be conducted in Ti vessels because high ionic strength brine was expected to dissolve to different degrees many of the proposed containment vessels that might be used to contain the experiments with brine at 30 °C for up to 5 years. Because titanium was not dissolved by the synthetic Brine A and Castile Brine formulations, a means of trying to determine the effect of Fe in the mild steel drums had to be tested. Consequently, all test containers were fabricated from Ti metal and a high surface area mesh was added to each test to simulate the amount of Fe that would be present if the waste were in a 55-gallon drum. The corrodable surface area in a 55-gallon drum was calculated to be about 4 m^2. To simulate this surface area of Fe, an Fe mesh with wire strands of a small diameter and therefore a large surface area was procured and prepared by Sandia National Laboratory for addition to the STTP test containers. The procured material was a No. 10 mesh made with 20 gauge wire and procured from Aggregate and Mining Supply Company of Albuquerque, New Mexico. The material came in rolls three feet wide and had a nominal analyses of elements as follows:

Fe	98.453 %
C	0.679 %
Mn	0.620 %
Si	0.230 %
P	0.012 %
S	0.006 %

It was determined that 2748 in^2 of this mesh would provide about 4 m^2 of surface area in a 55-gallon or 210-liter test drum. For the liter-scale vessels, about 39.25 in^2 would be required. The Fe mesh was cut in approximately 1.25 inch width and 30.0 inch length sections, with a target mass of 102.01 g selected to provide the necessary surface area for the experiments in three liter-scale test containers. This amounted to 39.25 in^2 with an average weight to surface area ratio of 2.599 g/in^2. These cut samples were packaged in 4 ounce Qorpak polypropylene jars with one inch diameter holes cut on the top and bottom of each jar to allow inflow and outflow of brine.

The Fe mesh for the 6 pressurized test containers with a two-liter volume were cut to a target mass of about 68 g and placed in 2-ounce Qorpak polyethylene jars.

The Fe mesh prepared and packaged for the drum-scale tests were cut into 6 pieces that were 36 inches by 12 inches plus one additional piece that was 12 inches by 12.9 inches for a target surface of 2748 square inches. The target weight of Fe mesh was about 7142 g to achieve about 4 m^2 of corrodable steel surface. The accumulated Fe mesh sections were placed in a 5 gallon polyethylene bucket that had 5-inch diameter holes cut in the top and bottom to allow flow of brine through the Fe mesh.

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A spectroscopic analysis of the Fe mesh with a Wavelength Dispersive X-ray Fluorescence Spectrometer showed an average constituent analysis of five wires, giving the following data:

Fe	95.4 %
Zn	1.5 %
P	1.43 %
Mn	1.28 %
Si	0.29 %
Cr	0.14 %
C	not analyzed

The presence of zinc was disturbing because it might be a galvanizing constituent or a protective layer. A surface analysis was performed to determine the property of the zinc in the wire by Auger Electron Spectroscopy. The zinc was determined to be a loose protective layer that was discontinuous over the Fe based wire strands. Corrosion on the unprotected Fe strands was visible where the zinc layer was very thin (~200 angstroms) and did not provide total protection of the wire.

II. (f) Nitrates in STTP Waste Solutions

A concern has been raised concerning the oxidative strength of nitrates in STTP wastes. This memo responds to that concern. Nitrates can be considered to be oxidants when they are in solid form or in concentrated acid solutions. Nitrates in dilute solutions or in neutral basic solutions are very weak oxidants or are not oxidants at all unless heated in the presence of strong reductants.

STTP waste does not contain concentrations of nitrates in solid form. The samples from the test containers contain varying concentrations of nitrates according to Table 8.

Generally, there is an average of 7 to 8 samples taken each week from the STTP test containers. Taken as a whole, the average concentration of nitrates in 50-ml samples taken from STTP test containers is about 4150 μg/ml or ppm. The amount of nitrates that would be disposed of, without consideration of dilution, would be as follows:

$$\frac{\left(50\ ml/sample\right)\left(8\ sample/week\right)\left(4150\ \mu g/ml\right)}{1\times 10^{6}\ \mu g/gm} = 1.7\ gm\ \ per\ \ week$$

Analyses of elemental analytes and actinides requires that the samples be acidified with nitric acid (HNO_3) to assure the actinides and elemental analytes are solubilized and remain stable or do not precipitate. Consequently, each sample is acidified by adding concentrated nitric acid (16N) to a final concentration of 2N HNO_3. A total of 200 to 250 ml of 2N HNO_3 results from analyses of each sample for ICP-MS and ICP-AES analyses of samples. This results in generating about 2 liter of 2N HNO_3 waste per week. 2N HNO3 is considered to be a dilute acid relative to concentrated nitric acid (16N) and does not have appreciable oxidative strength at that concentration. If the samples are added to the CMR outflow of waste water, the dilution is such that there is no oxidative strength of the resultant solution. In summary, the nitrates and nitric acid solutions to be disposed of from STTP waste solutions are:

1.) 1.7 gram of nitrates in solution per week

2.) About 2 liter of 2N HNO_3 per week

3.) There are essentially no solid nitrates to be disposed as waste from STTP samples

Table 8. Nitrate Concentrations in STTP Waste Solutions

Liter-Scales:

LITER-SCALE NUMBER	CONCENTRATION, µG/ML
L01	200
L02	600
L03	1,000
L04	200
L05	600
L06	2,000
L07	200
L08	800
L09	1,000
L10	200
L11	700
L12	1,000
L13	100
L14	100
L15	200
L16	100
L17	13,000
L18	1,000
L19	36,000
L20	30,000
L21	32,000
L22	36,000
L23	30,000
L24	32,000
L25	<100
L26	<100
L27	<100
L28	<100
L29	<100
L30	<100
L31	<100
L32	<100
L33	<100
L34	<100
L35	<100
L36	<100
L37	<100
L38	<100
L39	200

Drum-Scales:

DRUM-SCALE NUMBER	CONCENTRATION, μ/G/ML
D1	<50
D2	<50
D3	<50
D4	<50
D5	<50
D6	150
D7	<50
D8	800
D9	500
D10	100
D11	<50
D12	500
D13	<50
D14	<50
D15	200

II. (g) Evaluation of Hydrogen as a Potential Hazard in STTP Test Containers

Summary

Safety assessment of hydrogen in headspace of STTP test containers

An assessment of the details of gas generation in STTP test containers has resulted in a conclusion that a hazard does not exist for rapid combustion or an explosion to occur in the headspace of the test containers due to the reaction of H_2 and O_2 or N_2O. The conditions for the reactions to occur are not present when consideration is given to the following influencing parameters:

- volume of reactant gas – very small (30 cc max. in LS and 120 ml max. in DS)
- concentration of reactant gas – unbalanced
- stoichiometry – unbalanced fuel to oxidant ratio
- pressure – low to ambient
- temperature – 30 ^{O}C
- ignition sources
  - continuous: none
  - single spark: none
- presence of moisture – 31 torr over pool of brine

The thermodynamic or thermochemical reactions governing the combination of H_2 with O_2 or N_2O were calculated to show that the total amount of energy theoretically available for release under optimal conditions and stoichiometry would be about 0.09 kcal for liter-scale test containers and 0.36 kcal for the drum-scale test containers. The dilute concentration of oxidant will favor non-propagation of the reaction. Titanium does not spark when contacted by other metals and the large volume of highly conductive brine does not allow a static charge to develop in the test containers. The test containers are purged with He at least once a year and for some test containers is the predominant gaseous component in the headspace. The pressure of each test container is released before each sampling event.

Consideration of the major influencing parameters show that the combustion of H_2 and oxygen or some other oxygen containing gas is highly unlikely and even if a reaction was to occur the theoretical energy available for release within the test container headspace would be easily contained. The hazard associated with the hydrogen gas generation in the headspace of the STTP test containers is predictable, contained, and will not result in spread of contamination or present a hazard to the samplers.

Introduction

This section provides a discussion on the generation and accumulation of hydrogen in the headspace of STTP test containers and the potentiality of hydrogen as a hazard or non-hazard under the conditions of the experiments.

Hydrogen is generated within the test containers by four major processes:

- radiolysis of water molecules in brine;
- radiolytic decomposition of organics dissolved in brine;
- radiolytic degradation of solid organic materials; and
- corrosion reactions of brine on metals including electrolytic enhancement of corrosion.

The first three processes are the result of the energy given off by the decay of radioactive actinides entrained in the waste or dissolved in the brine. The most prevalent actinides with high specific activity in transuranic wastes destined for the WIPP are ^{238}Pu, ^{239}Pu, and ^{241}Am. The specific activity of the three most active actinides is dependent on the half-life of the particular radioisotope according to the following equations.

$$dn/dt = N\lambda$$

where dn/dt = decay rate as a function of time
N = number of atoms
λ = decay constant of specific radioisotope
where $\lambda = 0.693 / t_{1/2}$
$t_{1/2}$ = half-life of radioisotope
N = Avogadro's number/gm-atomic weight

For ^{239}Pu: $t_{1/2} = 2.410 \times 10^4$ annum

$$dn/dt = \frac{(6.02\times10^{23})(0.693)}{(239)(2.410\times10^{4})(365.25)(1440)}$$

$$= 1.38 \times 10^{11} \text{ disintegrations/min/gm}$$

For ^{241}Am: $t_{1/2} = 432.7$ annum

$$dn/dt = \frac{(6.02\times10^{23})(0.693)}{(241)(432.7)(365.25)(1440)}$$

$$= 7.60 \times 10^{12} \text{ dis/min/gm}$$

For ^{238}Pu: $t_{1/2} = 87.7$ annum

$$dn/dt = \frac{(6.02\times10^{23})(0.693)}{(238)(87.7)(365.25)(1440)}$$

$$= 3.80 \times 10^{13} \text{ dis/min/gm}$$

Continued on next page

The primary radioactivity in STTP test containers arise from ^{239}Pu and ^{241}Am because ^{238}Pu containing waste was not included in the STTP test matrix. However, the specific activity from ^{239}Pu and ^{241}Am is high enough to generate radiolytically produced gases in the test containers. Radiolytically produced gases include H_2, CO, CO_2, CH_4, N_2O, ethane, and ethylene. Very small and insignificant quantities of O_2 and N_2 are generated that accumulate in the headspace. Oxygen is known to be generated as free radicals but recombination reactions preclude significant quantities of oxygen from accumulating in the headspace as oxygen in a standard state. Liter-scale test containers No. 36, 39, 38, 26, 27, and 37 are high activity test containers that show generated O_2. The primary source of oxygen is from air originally present in the headspace or solubilized in the brine. Also, air can be introduced as part of the brine and gas sampling that is conducted on each test container. However, the greatest concentration of gases in the headspace are generally H_2, CO_2, CO, and N_2O. The N_2O is highest in test containers that contain very high concentrations of nitrate. The highest concentrations of H_2 are from test containers that have a high concentration of soluble organics and high radioactivity such as ^{241}Am.

Hydrogen from corrosion is primarily shown in drum-scale tests (DS 13, 14, and 15) selected because of their metal content.

Hydrogen, oxygen, and other gases in the headspace of an STTP test container can be considered to be in their standard state because they exist in their most stable form as molecules of O_2, H_2, CO_2, H_2O, etc. and at a pressure of almost 1 atmosphere and 30 °C. To determine the energy evolved from a reaction of stoichiometric quantities of H_2 and O_2, the reactants which are H_2 and O_2 are considered to react to form the product in a straight-chain or one-step path as in the following reaction.

$$H_2\,(g) + 1/2\,O_2 \rightarrow H_2O\,(\ell)$$

The standard enthalpies of formation, or more common, standard heats of formation of the reactants are combined to form the product of the thermochemical reaction which is liquid H_2O. The standard enthalpy change for this reaction at 25 °C and about 1 atm is the standard heat of formation of $H_2O_{(l)}$. Calculation of the energy evolved from the thermochemical reaction according to Hess's Law Equation gives the following result.

Given:

The standard Enthalpy of Formation $(\Delta H°_f)$ for:

$$H_2(g) = 0.00\ kcal/mol$$

$$O_2(g) = 0.00\ kcal/mol$$

$$H_2O(\ell) = -68.32\ kcal/mol$$

Continued on next page

$$\Delta H° = [Sum\,of\ \Delta H^{\circ}{}_{f}\ of\ products - sum\,of\ \Delta H^{\circ}{}_{f}\ of\ reactants\ ]$$

For Reaction:

$$2H_2 + O_2 \rightarrow H_2O(\ell)$$

$$\Delta H° = \left[1mol\left(\frac{-68.32kcal}{mole}\right)\right] - \left[2mol\left(\frac{0.00kcal}{mole}\right) + 1mol\left(\frac{0.00kcal}{mole}\right)\right]$$

$$= -68.32\ \tfrac{kcal}{mole}\ per\,mole\,of\ H_2O\ formed$$

For Reaction:

$$H_2 + N_2O \rightarrow H_2O + N_2$$

$$\Delta H^{\circ} = -88\,kcal/mol$$

From the above calculation for H_2 and O_2 and assuming standard conditions, the reaction is exothermic by

–68.32 kcal/mol.

Because the reaction is at near standard conditions and it is assumed to be a one step reaction, the standard entropy change (ΔS°) from the standard entropy of formation does not need to be considered.

The STTP test containers have an internal volume of approximately 3 liters and were loaded with enough brine, waste, and other influencing variables to leave a headspace volume of 5 to 10% of the total volume of the test container. Assuming the headspace volume is 10% then the volume is

(3000 mL)(.10) = 300 mL

The vapor pressure of water at 30 °C is 31.82 torr. To determine the partial pressure of each gas in the headspace of the test container would require subtration of the influence of the vapor pressure of H_2O.

$$P_{o2} = P_{total} - P_{water}$$

Continued on next page

The atmospheric pressure at Los Alamos is about 570 mm Hg therefore the partial pressure of O_2 is

$$P_{O_2} = 570\,torr - 31.82\ torr$$
$$= 538.2\,torr$$

Correcting for volume

$$V_2 = V_1\,(\tfrac{P1}{P_2})$$

$$V_2 = V_1\,\tfrac{538.2}{570}$$

$$V_2 = 0.94 V_1$$

The partial pressure of $H_2O(g)$ is about 6% of the total gas pressure in the headspace of a test container.

Conditions of Flammability

The hazard associated with containing an ignitable gas or mixture of gases is governed by the conditions, which enhance or suppress the possibility of ignition or uncontrolled rapid reaction. The conditions or parameters that influence the possibility and intensity of an ignition are described below.

Volume of Gas

The energy expelled by igniting a volume of gas is governed by the volume of gaseous components that will react and the kinetics of the reaction. The potential flammability of a gas or mixture of gases is a safety concern when the gas(es) support propagation of a flame and provide conditions that promote a high speed of flame propagation. The energy released from exothermic reactions is given in thermodynamic terms as kjoul/mol or kcal/mol. The greater the number of moles of a gas, the greater the energy released and alternatively, the smaller the volume, the less the energy released.

Stoichiometry

The rate of reaction of a mixture of gases is controlled by the fuel to oxidant ratio. The ratio of fuel (H_2) to oxidizing media (O_2 or N_2O) influences the combustibility of the gas mixture. If too much fuel is present, the fuel rich mixture may combust but flame propagation rate will be slow.

Continued on next page

If too little fuel is present, the lean flammability limit will have a higher threshold of ignition and the flame propagation rate will be limited. If the fuel (H_2) and oxygen (O_2) concentrations are optimal (stoichiometric) at 2 mole of H_2 and 1 mole of O_2, then ignition can occur with a single spark rather than a continuous spark source and flame propagation is maximized. However, impurity gases such as N_2, CO, CO_2, and H_2O (g) can upset stoichiometric ratios and make gaseous mixtures less likely to ignite and combust.

Pressure

The pressure of a container containing ignitable gases has a great influence on the rate of reaction because the concentration of gases is greater within a given volume. The higher the pressure, the greater the number of reactant molecules and the greater the energy that can be released from a single source. Reactions that occur at high pressures create the greatest "shock' wave during the reaction because of the greatest potential energy available for reaction in a confined space. Reactions occurring at ambient pressure are generally slower unless the stoichiometry of the mixtures are near perfect on a mole to mole basis.

Temperature

The temperature of a gas can influence the rate of reaction of two gases by maintaining a stoichiometric mixture and providing a convection of ideal gases with molecules with higher or lower kinetic velocity. The higher the temperature for a given volume and mass of gas, the greater the pressure of the gases and reaction between gases is more rapid and complete. Temperatures higher than about 500 ^{o}C will have a decided influence on increasing reaction rates while temperatures less than 100 ^{o}C will generally have a lesser influence on the reaction rate of gases. The Kelvin temperature of a gas is directly proportional to the average kinetic energy of its molecules and hence the reaction rate in a fixed volume. If pressure and temperature are at near ambient conditions, the rate of reactions are slowed and stoichiometry becomes a greater factor in the combustibility of gases.

Ignition Sources

Sources of ignition can be considered to be of two types; single spark or continuous spark. Single spark ignition sources can be a spark initiated because of a discharge between positively and negatively charged items, especially in a dry atmosphere. Striking two metals together can also create a single spark. A continuous can spark result from an electric current arcing between two conductive contacts or a sporadic arc between contacts. The presence of water in an enclosed system tends to ground out or eliminate the possibility of arcing between two contacts due to static electricity because water is a good conductor of electricity. In the case of STTP, brine is an excellent conductor of electricity and eliminates sparking due to static electricity.

Continued on next page

Presence of Moisture

The presence of moisture mitigates the possibility of ignition in gaseous mixtures because it eliminates the possibility of ignition from a single spark and maintains a cooling effect on any heated surface. Also, moisture has a vapor pressure that dilutes and upsets achieval of a stoichiometric gaseous mixture. The presence of moisture within an enclosed gas system will mitigate the possibility of ignition and eliminate flame propagation because of the cooling effect of the partial pressure of moisture especially if the moisture arises from a relatively large volume of water.

STTP Liter-Scale Test Containers

The STTP Liter-Scale test containers are all-titanium metal vessels that have a total volume of 3 liters. The test containers have been loaded with comminuted homogeneous wastes, namely, Portland cement, Envirostone, and Pyrochemical salt wastes. Certain test containers have added influencing variables such as Fe mesh, chelators, $Ca(OH)_2$, and ^{241}Am. After addition of Brine A or Castile brine, the test containers were topped-off with an inoculum brine mixture containing microbes from the WIPP site. The remaining headspace in the liter-scale test containers is about 5-10% of the total volume or 150 to 200 ml. The gas composition measured in the headspace of the liter-scale test containers after the headspace has been purged and several weeks have elapsed is given in Table 9 on page 32.

As shown in Table 9, the concentration of gases generated due to radiolysis dilutes and replaces the air atmosphere gases that were soluble in the brine in equilibrium with the headspace. The concentration of H_2 predominates in most liter-scale test containers except where N_2O predominates in test containers that contain high nitrate in the brine solution. The concentration of O_2 and nitrogen is depleted to less than 10% except for LS 36 and 39. A classic method for determination of O_2 in the headspace of an enclosed container has been to add an excess of H_2 and initiate a reaction between H_2 and available O_2 by providing an intermittent spark until most of the O_2 was consumed in the following reaction:

$$2H_2 + O_2 \rightarrow 2H_2O$$

I have conducted this type of analysis many times and generally tried to maintain a volume of 0_2 less than 50 ml. An intermittent spark source was required to complete the reaction. In STTP test containers, the total volume of O_2 is generally less than 1% of the 150-200 ml headspace volume or between 1-2 ml. One test container (LS 36) was measured to have about 15% O_2 or 22 to 30 ml. The number of kilocalories calculated for this reaction is about 68 kcal/mol. With one mole of O_2 equal to 22.4 liters, the energy released for the reaction of 30 ml of O_2 with excess hydrogen under ideal conditions and a continuous spark would be:

$$68{,}000\,cal/mole)\left(\frac{30\,cc}{22{,}400\,\frac{cc}{mol}}\right) = 91\,cal$$

Continued on page 33

Table 9. Headspace Gas Analysis for all Test Containers

LS	PU Total G	Solution Activity (100 ev/min)	H_2 (vol. %)	O_2 (vol.%)	N_2 (vol.%)	CO_2 (vol.%)	N_2O (vol.%)	NO_3 (ppm)	TOC (ppm)	pcH
L01	0.017	1E11	0.8	0.03	0.3	<0.1	<0.1	200	40	8.9
L02	0.105	6E10	3.9	0.64	2.2	<0.1	0.1	600	40	10.5
L03	0.124	1E11	7.2	0.57	3.8	0.1	<0.1	1000	50	13.0
L07	0.026	1E11	1.2	0.16	1.1	<0.1	<0.1	200	30	8.9
L08	0.118	6E10	1.8	0.03	0.2	<0.1	<1.0	800	70	9.4
L09	0.108	6E10	3.4	0.17	0.5	<0.1	<0.1	1000	60	13.0
L10*	0.021	1E12	**32.5**	1.50	0.2	<1.0	0.2	200	40	8.9
L11*	0.131	6E10	**22.2**	0.90	2.0	0.1	0.1	700	40	10.4
L12*	0.107	6E10	**~30.0**	1.20	0.3	<0.1	<0.1	1000	50	13.0
L13	**3.386**	1E11	**56.6**	0.20	0.5	404	<0.1	100	**4300**	7.2
L14	**3.468**	2E12	**37.4**	0.04	0.4	2.7	0.6	100	**4400**	7.3
L15	**0.041**	2E11	**3.0**	0.01	2.6	0.5	<0.1	200	**1300**	7.1
L16	0.618	5E12	61.6	0.06	0.6	0.7	2.5	100	400	7.8
L17	1.474	7E10	7.7	0.05	2.4	0.3	**20.4**	**13000**	300	7.8
L18	2.591	6E10	--	0.09	1.3	0.4	>1.0	1000	400	7.6
L19	0.508	3E11	15.8	0.10	5.9	1.8	**32.0**	**36000**	700	8.1
L20	0.080	6E10	2.9	0.06	2.0	0.4	**20.0**	**30000**	400	7.2
L21	0.250	1E11	7.3	0.02	4.1	0.2	**19.0**	**32000**	320	8.0
L22	0.233	4E11	6.1	0.15	6.0	0.6	**39.0**	**36000**	480	7.0
L23	0.500	6E10	8.0	0.06	6.4	0.9	**31.3**	**30000**	400	7.3
L24	0.309	6E10	12.7	0.02	25.0	0.2	**22.5**	**32000**	350	7.5
L25	0.381	8E11	13.0	0.08	0.8	<0.1	0.2	<100	20	7.8
L26	1.044	7E14	72.7	5.40	0.6	<0.1	0.1	<100	30	7.8
L27	3.400	1E15	65.2	4.80	5.0	<0.1	<0.1	<100	60	10.8
L31	0.806	2E11	32.7	0.80	1.5	<0.1	0.5	<100	30	9.0
L32	**4.083**	3E14	**44.6**	2.00	1.0	<0.1	<0.1	<100	30	8.9
L33	1.151	1E12	34.5	2.20	2.5	<0.1	0.3	<100	30	9.7
L34	2.004	3E11	28.6	0.66	2.2	0.1	1.1	<100	60	8.8
L35	0.451	1E13	20.9	0.07	1.7	<0.1	<1.0	<100	50	8.3
L36	**11.130**	3E14	**69.9**	14.90	0.2	<0.1	<0.1	<100	120	11.0
L37*	**4.333**	2E11	**50.0**	4.70	0.2	<0.1	<0.1	<100	20	7.7
L38*	2.736	1E11	51.3	8.70	0.2	<0.1	<0.1	<100	30	7.7
L39*	**4.482**	7E14	**45.6**	11.90	0.3	0.1	<0.1	200	40	9.8

* Am added to test container

This amount of energy is quite small and would not present a hazard. The assumption that this reaction would occur presupposes optimal conditions and a continuous or intermittent spark. The conditions in the STTP liter-scale test containers are not optimal because there are other gases in the mixture including water vapor from the brine at 30 ^{O}C. The partial pressure of $H_2O(g)$ at 30 ^{O}C is about 31.8 torr in the headspace of the test containers. The test containers are totally fabricated from titanium metal which does not spark when brought in contact with another metal so the potential for an ignition source is not present.

Seven liter-scale test containers contain a high concentration of N_2O from the radiolytic decomposition of nitrate. Typically, for test containers that have high N_2O content (up to 39%), the oxygen concentration is very low (<0.1%) and the H_2 concentration ranges from about 3% to 16%. This would make available about 8 to 40 ml of H_2 to react with excess oxidant.

The reaction kinetics of the reaction of H_2 with N_2O is slower than with O_2 but the total energy released is greater. The total energy released when a stoichiometric concentration of H_2 reacts with N_2O to form H_2O is about 88 kcal/mole. For 40 ml of H_2, the total energy released would be about 157 cal. The reaction of H_2 with O_2 or N_2O would result in an initial energy release and a subsequent negative pressure. A test conducted by STTP analysts consisted of dissociating a quantity of H_2O into a stoichiometric quantity of H_2 and O_2 in the headspace of a liter-scale test container and applying a direct electrical spark, which resulted in a small pop and a negative pressure. This actual test provided the samplers with an example of a worst case reaction under the most ideal conditions for the reaction to occur. The actual conditions in a test container do not favor stoichiometric ratios and the presence of brine and water vapor without a spark source makes this potential hazard quite innocuous if it should happen to occur and quite improbable because of the non-ideal conditions.

Drum-Scale Test Containers

The STTP Drum-Scale test containers are fabricated from titanium metal with a total volume of about 65 gal (246 liter). The test containers have been loaded with a mixture of heterogeneous wastes including combustibles (DS 1 through 12) and metals (DS 13, 14, 15). DS 4, 5, and 6 have added brine equilibrated bentonite, DS 7, 8, and 9 have added chelaters, and D 10, 11 and 12 have added sodium nitrate and phosphates as influencing variables. The drums were filled with Brine A or Castile brine and topped off with a microbial inoculum brine mixture. The headspace volume after addition of all additives is approximately 17.2 liters or about 7% of the total volume. The present gas composition in the headspace of the drum-scale test containers after the headspace has been purged with He and allowed to set for several weeks shows that O_2, CO, and N_2O are all less than 0.7%. The concentration of H_2 predominates in the headspace of the drum-scale test containers but the highest oxygen concentration is 0.7% (120 ml) and most drums are at <0.3% (52 ml). As with the discussion on liter-scale test containers, the amount of H_2 that is stoichiometrically available to react with O_2 is very small and in the drum scale test containers the oxygen concentration is diluted so ignition will not occur.

Continued on next page

Also, the drum-scale test containers are filled with brine and maintained at 30 ^{O}C, which results in water vapor (g) being present in the headspace with a partial pressure of about 31.8 torr. The drum-scale test containers are totally fabricated from titanium metal, which does not tend to spark when brought in contact with another metallic object. The headspace of each test container is purged with He at least once a year. The pressure developed in the headspace of the drum-scale test containers is released to less than 2 psig each week or prior to sampling. The remaining very low concentration of oxygen (<0.7% or $< 7 \times 10^{-3}$ mole)will not result in propagation of a H_2 and O_2 reaction and require several ignition events to react the O_2 with the excess H_2.

Test Matrix for the
Actinide Source-Term Waste Test Program (STTP)

III. (a) Test Matrix

Overview of the STTP Test Matrix

Liter-Scale:

L01-L12	Portland Cement	12 containers
L13-L24	Envirostone	12 containers
L25-L39	Pyrochemical Salt	15 containers

Drum-Scale:

D01-D12	Combustibles	12 drums
D13-D15	Envirostone	3 drums

* Most containers filled with Brine A (Salado formation brine).
* Containers divisible by 3 (L03, L06, etc.) contain Castile Brine

Table 1. STTP Liter-Scale Test Matrix

	L-01	L-02	L-03		
TRUCON	111/211	111/211	111/211	Solidified aqueous inorganic process sludge	
Brine	A	A	CASTILE	Portland Cement	10:1 / 2:1 / 2:1
	L-04	L-05	L-06		
TRUCON	111/211	111/211	111/211	Solidified aqueous inorganic sludge with CO_2	
Brine	A	A	CASTILE	Portland Cement	10:1 / 3:1 / 2:1
	L-07	L-08	L-09		
TRUCON	111/211	111/211	111/211	Solidified aqueous inorganic sludge without Fe	
Brine	A	A	CASTILE	Portland Cement	10:1 / 2:1 / 2:1
	L-10	L-11	L-12		
TRUCON	111/211	111/211	111/211	Solidified aqueous inorganic sludge w/o Fe; Am-241 added	
Brine	A	A	CASTILE	Portland Cement	10:1 / 2:1 / 2:1
	L-13	L-14	L-15		
TRUCON	112/212	112/212	112/212	Absorbed organic liquids	
Brine	A	A	CASTILE	Envirostone	2:1 / 2:1 / 2:1

Continued on next page

	L-16	L-17	L-18		
TRUCON	113/213	113/213	113/213	Absorbed aqueous laboratory waste	
Brine	A	A	CASTILE	Envirostone	2:1 / 2:1 / 2:1
	L-19	L-20	L-21		
TRUCON	114/214	114/214	114/214	Cemented inorganic particles	
Brine	A	A	CASTILE	Envirostone	2:1 / 2:1 / 2:1
	L-22	L-23	L-24		
TRUCON	126/226	126/226	126/226	Cemented organic sludge	
Brine	A	A	CASTILE	Envirostone	2:1 / 2:1 / 2:1
	L-25	L-26	L-27		
TRUCON	124/224	124/224	124/224	Pyrochemical salts	
Brine	A	A	CASTILE		2:1 / 2:1 / 2:1
	L-28	L-29	L-30		
TRUCON	124/224	124/224	124/224	Pyrochemical salts with CO_2	
Brine	A	A	CASTILE		2:1 / 2:1 / 2:1
	L-31	L-32	L-33		
TRUCON	124/224	124/224	124/224	Pyrochemical salts with brine – equilibrated bentonite	
Brine	A	A	CASTILE		2:1 / 2:1 / 2:1
	L-34	L-35	L-36		
TRUCON	124/224	124/224	124/224	Pyrochemical salts with $Ca(OH)_2$ and chelators	
Brine	A	A	CASTILE		3:1 / 3:1 / 3:1
	L-37	L-38	L-39		
TRUCON	124/224	124/224	124/224	Pyrochemical salts without Fe; Am-241 added	
Brine	A	A	CASTILE		2:1 / 2:1 / 2:1

Table 2. STTP Drum-Scale Test Matrix

	D-01	D-02	D-03	
TRUCON	116/216	116/216	116/216	Combustibles
Brine	A	A	CASTILE	
Waste (lbs)	43	59.1	47.2	
Total (lbs)	740.2	728.4	720	
	D-04	D-05	D-06	
TRUCON	116/216	116/216	116/216	Combustibles and brine – equilibrated bentonite
Brine	A	A	CASTILE	
Waste (lbs)	72.6	85.5	76.6	
Total (lbs)	765	723	746	
	D-07	D-08	D-09	
TRUCON	116/216	116/216	116/216	Combustibles with chelators
Brine	A	A	CASTILE	
Waste (lbs)	122.6	51.55	56.65	
Total (lbs)	783.4	726	720.6	
	D-10	D-11	D-12	
TRUCON	116/216	116/216	116/216	Combustibles and sodium nitrate/phosphates
Brine	A	A	CASTILE	
Waste (lbs)	66.4	48.6	66.4	
Total (lbs)	728	754.6	717	
	D-13	D-14	D-15	
TRUCON	117/217	117/217	117/217	Metals
Brine	A	A	CASTILE	
Waste (lbs)	171.4	136.8	162.2	
Total (lbs)	867.4	846.2	860	

III. (b) Composition of Brines

Table 3. Composition of Brine A and Castile Brine

Compound	*Brine A (g/L)*	*Castile Brine (g/L)*
$MgCl_2 \bullet 6H_2O$	292.10	3.86
NaCl	100.10	261.64
KCl	57.20	7.23
Na_2SO_4	6.20	23.70
$Na_2B_4O_7 \bullet 10H_20$	1.95	6.00
$CaCl_2$	1.66	1.33
$NaHCO_3$	0.96	0.00
NaBr	0.52	1.13
LiCl	0.125	0.00
RbCl	0.027	0.00
$SrCl_2 \bullet 6H_2O$	0.015	0.00
KI	0.013	0.00
$FeCl_2 \bullet 6H_2O$	0.0125	0.00
CsCl	0.00125	0.00

III. (c) Liter-Scale Test Matrix

Portland Cement: L01-L12

Test Container	Description of Contents	Ratios
L01, L02, L03	Solidified aqueous inorganic sludge	10:1 / 2:1 / 2:1
L04, L05, L06	Solidified aqueous inorganic sludge with CO_2, (pressurized)	10:1 / 3:1 / 2:1
L07, L08, L09	Solidified aqueous inorganic sludge without Fe	10:1 / 2:1 / 2:1
L10, L11, L12	Solidified aqueous inorganic sludge without Fe, ^{241}Am added	10:1 / 2:1 / 2:1

Envirostone: L13-L14

Test Container	Description of Contents
L13, L14, L15	Adsorbed organic liquids
L16, L17, L18	Absorbed aqueous laboratory waste
L19, L20, L21	Cemented inorganic particulates
L22, L23, L24	Cemented organic sludge

Pyrochemical Salts: L25-L39

Test Container	Description of Contents	Ratios
L25, L26, L27	Only Pyrochemical salts	
L28, L29, L30	Pressurized with CO_2 to 60 bars, 870 psig	
L31, L32, L33	Brine equilibrated with bentonite	
L34, L35, L36	Added $Ca(OH)_2$ and chelators	3:1 / 3:1 / 3:1

III. (d) Summary of Portland Cement Liter-Scale Tests

Summary of all Liter-Scale Tests, Excluding Pressurized Containers (L04-L06)

Actinides (Th, Np, Pu, U, and Am) and Nd remained very low (typically <20 ppb), except for L07 where Np increased from 4 to 310 ppb and is currently at 264 ppb. Pu, typically <20 ppb, reached a high of 40 ppb (in July 1997). In liter-scale 10, Np ranged from 20-45 ppb and Pu started at 26 ppb but decreased to 11 ppb. For TOC tests, TOC readings were very low (≤ 100 ppm). pcH tests where the highest of all the liter-scale tests (6.9 – 13.2).

Summary of Liter-Scale Pressurized Containers L04-L06

Nd, Th, Np, and Am remained less than 30 ppb, except liter-scale 04, which started at 421 ppb and then dropped. U was high and stayed high, the following chart illustrates this:

- L04: 10,000-15,000 ppb
- L05: 320-700 ppb
- L06: 2,900-4,900 ppb

pcH was comparable to the non-pressurized test containers. It ranged from 7.0-7.9.

III. (e) Summary of Envirostone Liter-Scale Tests

Nd, Th, Np, and Am typically were less than 10 ppb with Am always maintaining 3 ppb. The U count was the highest in Envirostone test containers than all the other liter-scale test containers (170 – 27,000 ppb). Np was also the highest in Envirostone test containers (7753 ppb). Pu was typically low (less than 20 ppb for L17 – L24) with the exception of L13-L16 in which Pu was up to 1700 ppb. pcH was also typically low with the narrow range of 6.4 – 8.6. All tests were neutral to slightly basic (pcH 6.8 – 8.2) and U solubilized from 10 to 20000 ppb whereas Th and Nd did not solubilize. Pu and Am did not solubilize to > 1100ppb. Most tests were <10 ppb. Pu was incorporated into 16% of colloids filtered and Fe identified on 27% of the filters. Organic solvents increased the population of particles by a factor of 100 and increased Pu-bearing colloids but not Th, Np, and U. High nitrate waste yielded 20-40 v/o N_2O in headspace but did not increase Pu concentrations. Envirostone test tended to be reducing as evidenced by presence of green ferrous chloride in sludge. Fe mesh was not fully available to brine because of compaction.

III. (f) Summary of Pyrochemical Salt Liter-Scale Tests

The Pyrochemical salt liter-scale tests are characterized by high Pu loadings with a high radiolytic degradation of brine and low Fe. All tests were basic (pcH 7.7-11.3). Three non-pressurized tests had high peak Pu (L26, 27, 36) concentrations (up to 14% of inventories) and one test (L26) showed high Eh and low Fe concentrations, meaning a prominent oxidizing environment was displayed. Bentonite had relatively low soluble Pu and low Fe concentrations but higher Pu and Fe colloidal populations. Chelators with $Ca(OH)_2$ were not effective in solubilizing actinides in Brine A and were more effective in Castile Brine (only in liter-scale tests, drum-scale tests showed solubilization in both brines). Soluble Am added to L37, 38 and 39 precipitated and did not resolubilize, in fact six tests resulted in cemented solids. Fe in Pyrochemical salt tests was effective in maintaining a non-oxidative environment. Comminution of waste had an important role in Pyrochemical salt waste chemistry and Fe had limited availability to brine because of compaction and cementation.

Overall Assessment of the
Actinide Source-Term Waste Test Program (STTP)

IV. (a) Test Results and Interpretation

Results of Observations of Liter-scale Test Containers

Liter-Scale No. 01

Test Characteristics:

Waste:	Portland Cement
Total Waste Weight:	264 g
Initial Actinide Content:	Pu 68 mg/g; Total Pu = 0.018 g
	Am 1.12 μg/g; Total Am = 0.296 mg
Brine:	Brine A (10:1 brine/solid ratio)
Additives:	Fe Mesh, Th, U, Np, and Nd

Soluble Actinide Histories: (4/17/95 – 4/5/99)

Pu - Ranged from < 1 ppb to 6 ppb at end of test.
Am- Was generally < 1 ppb for time period of test.
U - Was generally < 1 ppb for time period of test.
Th - Was < 1 ppb for entire test.
Np - Was generally less than 12 ppb with no trend.
Nd - Was < 6 ppb for entire test; no trend observed.

Other Analyses (nominal):

Typical pcH Range:	8.7 – 9.0
Fe Concentrations:	< 1 ppm for entire test.
Other Analytes:	Ca 17,000 ppm
	K 20,000 ppm
	Mg 22,000 ppm
	Na 40,000 ppm
TIC/TOC:	20/40 ppm
Particle Concentration:	10^9 to 10^{10} particles/Liter
Filter Paper-WDXRF:	No Pu, no Fe, very low Sr with some S
Headspace Gas Content:	H_2 = 0.8 v/o; O_2 = 0.03 v/o

D&D Observations (1-24-01):

Corrosion:	No corrosion observed on feedthroughs.
Brine:	Clear
Bottom Solids:	Approximately 3” of loose cement sludge.
Fe Mesh:	Not corroded; black in color.

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Overall Assessment:
Liter-scale test container No. 1 was a Brine A experiment with a 10:1 brine/solid ratio (264 g of solid). The pcH ranged from 8.7 – 9.0 and the D&D process revealed that the brine was clear and that the comminuted Portland Cement was loosely packed at the bottom of the test container (~ 3 inches of compacted solids that was not cemented). There was essentially no actinides, Nd, or Fe that were solubilized in this test. There was no colloids or microprecipitates that contained Pu or Fe on the filter papers. The Fe mesh was coated with a hard black coating that prevented observable corrosion. There was a relatively low Pu and Am inventory (0.018 g and 0.296 mg, respectively) and a very low H_2 content in the headspace of the test container.

Liter-Scale No. 02

Test Characteristics:

Waste:	Portland Cement
Total Waste Weight:	1320 g
Initial Actinide Content:	Pu 80 μg/g; Total Pu = 0.105 g
	Am 1.41 μg/g; Total Am = 1.86 mg
Brine:	Brine A (2:1 brine/solid ratio)
Additives:	Fe Mesh, Nd, Th, U, Np added

Soluble Actinide Histories: (4/17/95 – 12/14/98)

Pu - Generally less than 2 ppb; 3 analyses <10 ppb.
Am- Less than 1 ppb.
U - Less than 2 ppb.
Th - Less than 1 ppb.
Np - Less than 1 ppb.
Nd - Less than 2 ppb.

Other Analyses (nominal):

Typical pcH Range:	8.7 – 9.0
Fe Concentrations:	< 1 ppm for entire test.
Other Analytes:	Ca 17,000 ppm
	K 20,000 ppm
	Mg 22,000 ppm
	Na 40,000 ppm
TIC/TOC:	20/40 ppm
Particle Concentration:	10^9 to 10^{10} particles/Liter
Filter Paper-WDXRF:	No Pu, no Fe, very low Sr with some S
Headspace Gas Content:	H_2 = 0.8 v/o; O_2 = 0.03 v/o

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D&D Observations (1-24-01):

Corrosion:	No corrosion observed on feedthroughs.
Brine:	Clear
Bottom Solids:	Approximately 3" of loose cement sludge.
Fe Mesh:	Not corroded; black in color.

Overall Assessment:

Liter-scale test container No. 1 was a Brine A experiment with a 10:1 brine/solid ratio (264 g of solid). The pcH ranged from 8.7 – 9.0 and the D&D process revealed that the brine was clear and that the comminuted Portland Cement was loosely packed at the bottom of the test container (~ 3 inches of compacted solids that was not cemented). There was essentially no actinides, Nd, or Fe that were solubilized in this test. There was no colloids or microprecipitates that contained Pu or Fe on the filter papers. The Fe mesh was coated with a hard black coating that prevented observable corrosion. There was a relatively low Pu and Am inventory (0.018 g and 0.296 mg, respectively) and a very low H_2 content in the headspace of the test container.

Liter-Scale No. 03

Test Characteristics:

Waste:	Portland Cement
Total Waste Weight:	1320 g
Initial Actinide Content:	Pu 93 μg/g; Total Pu = 0.123 g
	Am 1.45 μg/g; Total Am = 1.91 mg
Brine:	Brine A (2:1 brine/solid ratio)
Additives:	Fe Mesh, Nd, Th, U, Np added

Soluble Actinide Histories: (4/17/95 – 2/22/98)

Pu - Generally < 6 ppb and mostly < 2 ppb. No trend observed.
Am – Generally < 1 ppb with no trend observed.
U - Generally < 6 ppb with no trend observed.
Th - < 1 ppb throughout test.
Np - < 1 ppb throughout test.
Nd - < 1 ppb throughout test.

Other Analyses (nominal):

Typical pcH Range:	12.8 – 13.1
Fe Concentrations:	< 1 ppm for entire test period.
Other Analytes:	Ca 120 ppm
	K 8,000 ppm
	Mg < 10 ppm
	Na 83,000 ppm
TIC/TOC:	30/40 ppm

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Particle Concentration: 10^9 to 10^{11} particles/Liter
Filter Paper-WDXRF: One filter paper had a barely detectable amount of Pu and 15 of 17 filters had Fe. Most filters had Sr and S.
Headspace Gas Content: H_2 = 7.2 v/o; O_2 = 0.6 v/o

D&D Observations (4-12-01):

Corrosion: No visible corrosion on SS feedthroughs; screen was not corroded. Screen about half full of finely divided cement.
Brine: Castile Brine was fairly clear with slight grayish tinge. Solids in brine settled out readily.
Bottom Solids: About 8 inches of loose but settled cement and other solids in the bottom of the test container. Color of solids is gray with a consistency of hardened oatmeal.
Fe Mesh: The Fe mesh was lodged in gray solid material but was pried off. The mesh was in good condition and was not corroded visibly. After rinsing with alcohol, the Fe mesh appeared to be totally untouched by corrosion. The wire was gray in color with shiny ends.

Overall Assessment:

LS-03 was the Castile brine part of the triplet on LS-01, 02, 03. As in LS-01 and 02, LS-03 did not solubilize Pu or any other actinide. There was no soluble Fe detected in this test. The pcH range of 12.8 to 13.1 was high enough to precipitate both Mg and Ca which led to greater solids. There was no soluble Fe detected in any sample during the test period and yet 15 of 17 filters had Fe. There was essentially no Pu detected on the filter papers taken from this test; actually one filter has a barely detectable amount of Pu. The wide range of particle concentrations of 10^9 to 10^{11} was similar to LS-02. There was no corrosion of the SS feedthroughs or the Fe mesh. The brine was clear of suspensions but had a gray color. Any suspensions stirred up were rather quickly settled.

Liter-Scale No. 04 (Pressurized)

Test Characteristics:

Waste: Portland Cement
Total Waste Weight: 184 g
Initial Actinide Content: Pu 103 μg/g; Total Pu = 18.95 mg
Am 1.27 μg/g; Total Am = 0.233 mg
Brine: Brine A (10:1 brine/solid ratio)
Additives: Fe Mesh, Nd, Th, U, Np, 60 Bar (870 psig) CO_2 pressure in headspace.

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Soluble Actinide Histories: (8/28/95 – 9/21/98)

Pu - Pu started at 1.6 ppb and steadily increased to 225 ppb on 9/21/98. Total alpha activity was 15.29 nCi/ml on 9/21/98 and decreased to 0.08 nCi/ml on 5/15/01 (a factor of 191).

Am – Am was less than 0.2 ppb at the beginning of the test, Am-241 total alpha activity decreased from 0.92 (9/21/98) to 0.23 nCi/ml (5/15/01).

U - U concentration of 10,000 ppb at the beginning of the test remained fairly constant and ended up at 8705 ppb at the end of the test.

Th - Th was < 10 ppb for the entire test period. There were no trends observed.

Np –Concentrations of Np were generally less than 21.0 ppm and showed no trend during the entire test period.

Nd - Nd concentrations were < 1.1 ppb for the entire test period.

Other Analyses (nominal):

Typical pcH Range:	7.21 – 7.43
Fe Concentrations:	Started at 3.1 ppm and increased to 162.4 ppm at the end of the test.
Other Analytes:	Ca 6,000 ppm K 6,000 ppm Mg 26,000 ppm Na 40,000 ppm
TIC/TOC:	1100/70 ppm
Particle Concentration:	1×10^{11} particles/Liter
Filter Paper-WDXRF:	No Pu; 1 filter of 4 showed Fe. No Sr identified. No Al identified.
Headspace Gas Content:	60 Bar of CO_2; no other gas analyzed.

D&D Observations (5-9-01):

Corrosion:	No corrosion on lid or screen.
Brine:	Clear, non-viscous brine with light gray color. Brine level was at the top of the screen.
Bottom Solids:	The Fe mesh holder was filled with light gray colored compacted sludge.
Fe Mesh:	The Fe mesh was holder was embedded in gray-colored solids. The Fe mesh after washing had a gray-green color. The Fe mesh wire strands were black.

Overall Assessment:

LS-04 was a Portland cement test with 60 Bar (870 psig) CO_2 pressure. The Pu never achieved a high concentration but had a definite upward trend to 255 ppb. U concentrations were relatively high at ~ 10,000 – 11,000 ppm. The Fe concentration started at 3.1 ppm and increased to 162.4 ppm, which is quite high. There was no corrosion observed on the lid, screen, or feedthroughs. There were no colloids or microprecipitates that contained Pu. The pcH did not vary due to CO_2 pressure and had a range of 7.21 to 7.43. This test was a 10:1 ratio brine to solid ratio and only contained 184 grams of waste.

Liter-Scale No. 05 (Pressurized)

Test Characteristics:

Waste:	Portland Cement
Total Waste Weight:	613 g
Initial Actinide Content:	Pu 83.5 μg/g; Total Pu = 51.2 mg
	Am 0.970 μg/g; Total Am = 0.595 mg
Brine:	Brine A (3:1 brine/solid ratio)
Additives:	Fe Mesh, Nd, Th, U, Np, 60 Bar (870 psig) CO_2, pressure in headspace.

Soluble Actinide Histories: (8/28/95 – 9/21/98)

Pu - Pu started at 1.7 ppb and increased to a peak of 184 ppb with an average of about 15 ppb. Total alpha activity was 0.71 nCi/ml on 9/21/98 and decreased to 0.03 nCi/ml on 5/15/01. There was no rotation during that final period of time.

Am – Am was less than 0.8 ppb during the entire test. Am-241 total alpha activity was < 0.14 nCi/ml on 9/21/98 and was < 0.12 nCi/ml on 5/15/01.

U - U concentration started at 696 ppb and remained quite steady to end up at 479 ppb at the end of the test. No apparent trend was observed.

Th - Th was < 5.1 ppb for the entire test period.

Np - Concentrations of Np were less than 27.0 ppm.

Nd - Nd concentrations were < 0.9 ppb.

Other Analyses (nominal):

Typical pcH Range:	7.0 – 7.44
Fe Concentrations:	Started at 7 ppm and increased to 57.1 ppm. Ended at 16.4 ppm.
Other Analytes:	Ca 12,000 ppm
	K 25,000 ppm
	Mg 22,000 ppm
	Na 40,000 ppm
	Ni 7 ppm
TIC/TOC:	700/70 ppm
Particle Concentration:	1 x 10^{11} particles/Liter
Filter Paper-WDXRF:	No Pu identified on 3 filters analyzed. One filter showed Fe. No Sr identified.
Headspace Gas Content:	60 Bar of CO_2 pressure.

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D&D Observations (5-10-01):

Corrosion: No corrosion on lid or screen.
Brine: Clear, non-viscous brine with light brown tinge.
Bottom Solids: Yellowish-brown mass that was compact and about 5-1/2 inches in depth.
Fe Mesh: The Fe mesh holder was embedded in solids with a peanut butter texture and color. After rinsing the material of the mesh, the solution had a bluish tint. There was a hard coating on the mesh that was black with a blue tint. No corrosion was visible but the Fe concentration in the brine seemed to imply some corrosion took place at the pcH 7.0 – 7.4 range.

Overall Assessment:

Liter-scale test container LS-05 was a Portland cement test with 60 Bar (870 psig) CO_2 pressure. The Pu did not appreciably solubilize and other actinides remained rather low. The Fe concentration started at 7.0 ppm and increased to 57.1 ppm. There were no Pu colloids or microprecipitates that were filtered out. The pcH did not change significantly (pcH 7.4 – 7.35) during the test period. There was no corrosion observed on the lid, screen, or Fe mesh. LS-5 was the second in a set of three pressurized test containers; LS-4 has a 10:1 brine-to-solid ratio, LS-5 had a 3:1 brine-to-solid ratio and LS-6 had a 2:1 brine-to-solid ratio.

Liter-Scale No. 06 (Pressurized)

Test Characteristics:

Waste: Portland Cement
Total Waste Weight: 920 g
Initial Actinide Content: Pu 95 μg/g; Total Pu = 97.4 mg
Am 1.08 μg/g; Total Am = 0.994 mg
Brine: Castile (2:1 brine/solid ratio)
Additives: Fe Mesh, Nd, Th, U, Np, 60 Bar (870 psig) CO_2, pressure in headspace.

Soluble Actinide Histories: (8/28/95 – 9/21/98) then 4/27/01

Pu - Pu started at 25 ppb and peaked at 638 ppb before settling down to 319 ppb on 9/21/98. Pu was at an apparent trend upwards near the end of the test. The alpha activity on 9/21/98 was 28 nCi/ml which decreased to 0.07 nCi/ml (a decrease factor of 400) on 4/27/01. There was no rotation during the last period.

Am – Am was less than 1 ppb (except 1.4 ppb on 1/13/97) for the entire test period. Am total alpha on 9/21/98 was 1.12 nCi/ml and 0.21 nCi/ml on 4/27/01.

U - U started at 2934 ppb and ended up at 3675 ppb. Not much variation and rather low concentrations for a high carbonate system.

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Th - Th was < 3 ppb for the entire test period.
Np –Concentrations of Np were less than 15 ppb for the entire test period.
Nd - Nd was < 4 ppb for the entire test period.

Other Analyses (nominal):

Typical pcH Range:	7.49 – 7.87
Fe Concentrations:	Ranged from 2 ppm to 41.4 ppm.
Other Analytes:	Ca 1,000 ppm K 10,000 ppm Mg 600 ppm Na 40,000 ppm Ni 8 ppm
TIC/TOC:	1700/100 ppm
Particle Concentration:	9×10^{10} particles/Liter No correlation with L04 and L05.
Filter Paper-WDXRF:	No Pu or Fe identified on 4 of 4 filters.
Headspace Gas Content:	60 Bar (870 psig) of CO_2 pressure.

D&D Observations (4/24/01):

Corrosion:	No corrosion on lid or screen.
Brine:	The brine was a milky color with no suspensions or crystals. The brine pool was ~ 2 inches deep that led to the top of a brownish-gray soft solid.
Bottom Solids:	Brownish-gray soft solid that was compacted and took up about ¾ of the test container.
Fe Mesh:	Embedded in a clay-like sludge that was compacted in the Fe mesh holder. The compacted material would have limited brine flow to the Fe mesh. After washing, the Fe mesh did not appear to be corroded and the ends of the wire were shiny. The Fe mesh strands were dark-colored. The Fe concentration in the brine varied from 2 to 41.4 ppm at a pcH around 7.5 – 7.9. This is higher than expected for an Fe mesh that is impacted with solid material.

Overall Assessment:

LS-06 was a Portland Cement test with 60 Bar (870 psig) CO_2 pressure in Castile Brine. LS-4,5,and 6 were a set of three test containers with CO_2 pressure and Portland Cement. LS-4, 5, and 6 had a brine-to-solid ratio of 10:1, 3:1, and 2:1, respectively. This is evident during the D&D because of the increase in bottom solids with the lower ratio. Pu did not solubilize in this test to a high level (638 ppb peak) but was greater than LS-4 and LS-5 as should be expected because of the greater amount of Pu as the brine/solid ratio was smaller. No other actinides were significantly solubilized and Nd, Th, and Np were essentially very low. Nd (< 4 ppb, Th (< 3 ppb) and Np (< 14 ppb). There were no Pu or Fe colloids identified on the 4 filter papers. There was no corrosion observed on the lid, screen, or Fe mesh. The overall observations of the LS4, 5, and 6 is that there was very little solubilization of actinides on these three Portland Cement tests with added CO_2 pressure at 60 Bar (870 psig).

Liter-Scale No. 07

Test Characteristics:

Waste:	Portland Cement
Total Waste Weight:	264 g
Initial Actinide Content:	Pu 94.5 μg/g; Total Pu = 0.025 g
	Am 1.198 μg/g; Total Am = 0.316 mg
Brine:	Castile (10:1 brine/solid ratio)
Additives:	Th, U, Np
	No Fe mesh

Soluble Actinide Histories: (4/17/95 – 3/15/99)

Pu - There were four analyses of 22.9, 40, 10.5, and 14 ppb and the remainder were < 5 ppb. There was no trend of concentrations. Final concentration was 4.1 ppb.
Am - All analyses were < 1 ppm.
U - Analyses were generally < 15 ppb with one as high as 20 ppb.
Np - Started at 3.7 ppb and increased to ~250 ppb.
Th - Generally < 2 ppb.
Nd - Generally < 2 ppb.

Other Analyses (nominal):

Typical pcH Range:	8.7 – 8.9
Fe Concentrations:	<2 ppm
Other Analytes:	Ca 14,000 ppm
	K 21,000 ppm
	Mg 23,000 ppm
	Na 44,000 ppm
	Pb 4 – 5 ppm (last 9 analyses)
TIC/TOC:	12/30 ppm
Particle Concentration:	10^9 to 10^{10} particles/Liter
Filter Paper-WDXRF:	None of the filters had Pu or Fe. Only 3 of 17 had Sr.
Headspace Gas Content:	1.2 v/o H_2, ~ 1.2 v/o O_2. This was the lowest H_2 in STTP.

D&D Observations (3/28/01):

Corrosion:	Corrosion was noted around SS feedthroughs; screen was clean.
Brine:	The brine was colorless with no suspensions or crystals.
Bottom Solids:	About 8 inches of loose muddy solids that could be stirred up; this 10:1 brine to solid test had much liquid left after removal of 1 liter sample. Added AquaSorbe – 2212 to absorb brine.
Fe Mesh:	No Fe mesh added to this test container.

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Overall Assessment:
There was essentially no solubilization of Pu, Am, U, Th, or Nd during the test period. There was a rather consistent 250-300 ppb of Np after 6 months into the test. Np appeared to be more apt to solubilize in 10:1 ratios. This level of Np solubilization is negligible compared to theoretical concentration of 37,500 ppb if the amount added was totally solubilized.

There was corrosion noted on SS feedthroughs on the lid. The screen was not corroded. The brine was clear with no suspensions. The precipitates seemed to settle readily to the bottom and mix with 8 inches of sludge. There was no Fe mesh added to LS-07 and no Fe was detected on any filter paper.

Liter-Scale No. 08

Test Characteristics:

Waste:	Portland Cement
Total Waste Weight:	1320 g
Initial Actinide Content:	Pu 88.5 μg/g; Total Pu = 0.117 g
	Am 1.095 μg/g; Total Am = 1.45 mg
Brine:	Castile (12:1 brine/solid ratio)
Additives:	Nd, Th, U, Np
	No Fe mesh

Soluble Actinide Histories: (4/17/95 – 3/15/99)

Pu - There were 8 analyses from 10-26 ppb and the remainder were <5 ppb. There was no trend of concentrations.
Am - All analyses were < 0.5 ppb except last analyses was 1 ppb.
U - Analyses were generally < 2 ppb, which is quite low for U. No trend is apparent.
Np - Less than1 ppb for entire test period.
Th - Generally < 1 ppb for entire test period.
Nd - Generally < 2 ppb for entire test period.

Other Analyses (nominal):

Typical pcH Range:	9.1 – 9.5
Fe Concentrations:	<1 ppm
Other Analytes:	Ca 36,000 ppm
	K 30,000 ppm
	Mg 2,500 ppm
	Na 43,000 ppm
TIC/TOC:	10/50 ppm
Particle Concentration:	10^9 to 10^{10} particles/Liter
Filter Paper-WDXRF:	None of the filters had Pu; two of 16 showed Fe. Essentially all showed Sr and S.
Headspace Gas Content:	H_2 = 1.8 v/o (2nd lowest in STTP); O_2 = 0.03 v/o.

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D&D Observations (04/03/01):

Corrosion: No corrosion observed in SS feedthroughs. Screen was clear, had ~1/8-inch of sediment.
Brine: Clear with grayish coloration; no suspensions or crystals noted.
Bottom Solids: About 8 inches of solids; 4 inches of loose fluffy solids and then 4 inches of hard solids that may have been cemented.
Fe Mesh: No Fe mesh added to this test container.

Overall Assessment:

There was essentially no solubilization of Pu or any other actinide in LS-08 at pcH 9.1– 9.5 in a Brine A environment. Most of the Mg precipitated at the pcH range of 9.1 – 9.5. There was no soluble Fe throughout the test nor was there any filterable Pu or Fe (mostly) in LS-08. There was no Fe mesh added. There was about 4 inches of hard solids at the bottom of the test container. The brine was fairly clear for a 2:1 Brine/solid ratio test. There was no corrosion of the SS feedthroughs.

Liter-Scale No. 09

Test Characteristics:

Waste: Portland Cement
Total Waste Weight: 1320 g
Initial Actinide Content: Pu 81 μg/g; Total Pu = 0.107 g
Am 0.995 μg/g; Total Am = 1.26 mg
Brine: Castile (2:1 brine/solid ratio)
Additives: Nd, Th, U, Np
No Fe mesh

Soluble Actinide Histories: (4/17/95 – 3/15/99)

Pu - All results were < 10 ppb except two analyses at 13.0 and 16.3 ppb. Most analyses were < 5 ppb.
Am - All analyses were < 1 ppb; no trend observed.
U - All results were < 10 ppb, except one at 13.4 ppb. No trend is apparent.
Np - Less than 1 ppb, except one result (1.7 ppb).
Th - Less than 2 ppb for entire test period.

Other Analyses (nominal):

Typical pcH Range: 12.9 – 13.1
Fe Concentrations: <1 ppm for the entire test period.

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Other Analytes:	Ca 150 ppm K 8,400 ppm Mg <10 ppm Na 80,000 ppm Pb 4 ppm average for last nine results.
TIC/TOC:	15/50 ppm
Particle Concentration:	10^9 to 10^{10} particles/Liter
Filter Paper-WDXRF:	No Pu colloids or microprecipitates detected. Seven out of 17 filters had Fe. Twelve out of 17 contained Sr. Although there was < 10 ppm Mg, there was much Mg and Ca in all the filters. The Ca, Mg, and Fe were mostly on the 5 micron filters.
Headspace Gas Content:	H_2 = 3.4 v/o; O_2 = 0.17 v/o.

D&D Observations (04/19/01):

Corrosion:	No corrosion observed in SS feedthroughs. (Sampling port, level probe, pressure gauge). No coloration was noted.
Screen:	No corrosion on the o-ring; ~1/2-inch of gray paste in the screen.
Brine:	Clear except for Portland cement suspensions.
Bottom Solids:	Loose solids (6-8 inches) that settled readily, no cementation.
	Fe Mesh: No Fe mesh added to this test container.

Overall Assessment:

LS-09 was a Castile brine experiment with a 2:1 Brine/solid ratio with a very basic pcH (12.9 – 13.1) that precipitated both Ca and Mg as hydroxides. There was essentially no solubilization of Pu, Am, other actinides and Fe. There was no Fe mesh added to this test but seven of 17 filters contained Fe, perhaps from the ferric sulfate added to the original Portland Cement mix. There was no corrosion of the three SS feedthroughs at the highly basic pcH. There was 6-8 inches of loose solids that probably contained Ca and Mg hydroxides. There was no Pu found in any filter paper. The dilution of Pu within a Ca and Mg hydroxide matrix could have been substantial. The H_2 concentration in the headspace was relatively low (3.4 v/o) and the O_2 was 0.17 v/o.

Liter-Scale No. 10

Test Characteristics:

Waste:	Portland Cement
Total Waste Weight:	264 g
Initial Actinide Content:	Pu 86 μg/g; Total Pu = 0.021 g Am 1 μg/g; Total Am 0.25 mg
Brine:	Brine A (10:1 Brine/Solid ratio)
Additives:	Th, U, Np, No Fe mesh added; no Nd addition
Other:	^{241}Am (75 mg) added as soluble salt

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Soluble Actinide Histories:

Pu - Pu concentrations ranged from about 10 to 20 ppb for most of the test. No trend was observed .

Am - Am concentration varied from about 0.3 to 1.9 ppb for the life of the test. No trend was observed.

U - U concentrations ranged from 0.3 to 12 ppb for the lifetime of the test. No trend was observed.

Np - Np concentrations ranged from 20 to 48 ppb for the test and no trend was observed.

Th - Concentrations were generally less than 5 ppb with no trends observed.

Nd - Neodymium was not added to LS 10.

Other Analyses (Nominal):

Typical pcH Range:	8.2 to 8.9
Fe Concentration:	There was no Fe mesh added to this test container and Fe was typically less then 1 ppm.
Other Analytes:	Ca 13 k K 23 k Mg 23 k Na 40 k
Other:	Al, Ni, and Pb < 5 ppm
TIC/TOC:	10/30 ppm
Particle Concentration:	10^9 to 10^{10} particles/L
Filter Paper-WDXRF:	No Pu or other actinides identified on 5 micron, 1 micron and < 10 nm filter papers. Sr was not identified on 5 and 1 micron filter paper but was identified on < 10 nm filters. No Fe identified; high Ca and Mg.
H_2 Headspace Gas Content:	32.5 v/o H_2, 1.5 v/o O_2

D&D Observations (2/5/01):

Corrosion:	Some corrosion visible around sampling port, level probe, and gauge port. Some rust colored corrosion product on screen and sides of vessel.
Brine:	Brine is clear.
Bottom Solids:	About 3 to 4 inches of compacted but soft solids that were not solidified.
Fe Mesh:	No Fe mesh in LS10.

Overall Assessment:

LS10 was a comminuted Portland Cement test with 75 mg of added Am-241 to increase alpha activity and radiolysis. All actinides and Nd were precipitated immediately in the Portland Cement matrix at pcH 8-9. The added Am-241 was also immediately precipitated in the Portland Cement matrix and did not show an increase beyond 1 ppb during the entire test.

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The presence of the precipitated Am-241 was evident from the H_2 concentration in the headspace in this test container (~ 32.5 v/o H_2) which was higher than similar Portland cement tests w/o Am-241 (typically < 5% H_2). This test exemplified the effectiveness of Portland Cement in immobilizing or precipitating actinides in a high radiolytic environment. The 10:1 brine to solid ratio may have been the reason that the brine was clear and that there was a definite phase separation between the comminuted Portland cement and the brine. Although there was 75 mg of added ^{241}Am equivalent to 37,500 ppb, if totally dissolved, there was no Pu or Am detected on the filter papers indicating that at this brine to solid ratio that there was no Pu or Am colloids or microprecipitates that did not settle to the bottom of the Brine A after each rotation. There was no Fe identified on any filter.

Liter-Scale No. 11

Test Characteristics:

Waste:	Portland Cement
Total Waste Weight:	1320 g
Initial Actinide Content:	Pu 98 μg/g; Total Pu = 0.129 g
	Am 1.195 μg/g; Total Am 1.58 mg
Brine:	Brine A (2:1 Brine/Solid ratio)
Additives:	Th, U, Np No Fe mesh added; no Nd addition
Other:	^{241} Am (75 mg) added as soluble salt.

Soluble Actinide Histories:

- Pu - Pu concentrations less than 5 ppb with 4 exceptions. Low Pu with no visible trend.
- Am - Less than 0.5 ppb with no apparent trend.
- U - U concentrations less than 3 ppb with no visible trend.
- Np - Np concentrations less than 1 ppb with no trend observed.
- Th - Concentrations were less than 1 ppb with no visible trend.

Other Analyses (Nominal)

Typical pcH Range:	9.0 to 10.8
Fe Concentration:	Generally less than 1 ppm. There was no Fe mesh added to this test container.
Other Analytes:	Ca 40,000 ppm
	K 30,000 ppm
	Mg < 200 ppm (this seems like Castile Brine)
	Na 40,000 ppm
Other:	Al, Ni, and Pb < 5 ppm
TIC/TOC:	10/40 ppm
Particle Concentration:	10^9 to 10^{10} particles/L

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Filter Paper-WDXRF: No Pu identified in any filter paper. Seven of 15 filters had Fe; all filters had Sr and S at 5 and 1 micron and < 10 nm filters. High Ca and Mg found on all filters.

H_2 Headspace Gas Content: 22% H_2

D&D Observations (2/6/01):

Corrosion: SS fittings and feedthroughs were slightly rusted. There was also the appearance of rust around the top of the vessel.

Brine: Brine is gray color; there was sediment on screen with black particles of rust.

Bottom Solids: There was approximately 8 inches of very loose gray sludge at the bottom of the test container. Approximately one-inch at the very bottom of the test container was compacted "hard" solid but not cemented.

Fe Mesh: No Fe mesh in LS11.

Overall Assessment:

LS11 was a typical Portland Cement test at pcH 9.0 to 10.8 that had very low concentrations (< 5 ppb) of all actinides. There was no Fe Mesh or Nd added to this test container but there was 75 mg of Am-241. If all the ^{241}Am added as a soluble salt remained soluble the concentration would have been 37,500 ppb. The Fe concentration was also very low at < 1 ppm. The particle concentration was generally low, 10^9 to 10^{10} particles/liter. No Pu was identified on the filter papers and Fe was identified on the 5 micron filter. All filters (5 micron, 1 micron, 10 nm) showed Sr and S. There was rust observed around the S.S. fittings and feedthroughs. The H_2 was about 22 v/o which was rather low for a test with added Am-241. There was a great deal of sludge in the test container and the Mg was low for a Brine A test. Perhaps the Mg precipitated and led to a higher sludge content but the main reason was that this was a 2:1 brine to solid ratio as opposed to a 10:1 in LS10. Both tests were with Brine A. Mg begins to precipitate as $Mg(OH)_2$ at a pH of about 10.5. The absence of Pu or Am on any filter papers indicates that the Pu did not dissolve initially or that any soluble Pu or Am was immediately precipitated and settled down to the bottom of the test container after each rotation. The sludge was not solidified or cemented but was mostly loose. Overall, the high pcH (9.0 – 10.8) and high sludge content led to a test with essentially no soluble actinides.

Liter-Scale No. 12

Test Characteristics:

Waste: Portland Cement
Solidified dewatered aqueous process sludge

Total Waste Weight: 1,320 g

Initial Actinide Content: Pu 81.5 μg/g; Total Pu = 0.108 g
Am 1.021 μg/g; Total Am = 1.347 mg

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Brine:	Castile (2:1 brine/solid ratio)
Additives:	Th, U, Np, and Am-241 No Fe mesh; no Nd added

Soluble Actinide Histories:

Pu -	Remained typically <10 ppb.
Other -	Nd, Th, Np, U, and Am remained ≤4 ppb throughout the experiment.

Other Analyses (nominal):

Typical pcH Range:	12.7-13.0
Fe Concentrations:	<1 ppm
Other Analytes:	Ca 200 ppm K 8,000 ppm Mg 25 ppm Na 80,000 ppm
TIC/TOC:	40/50 ppm
Particle Concentration:	10^9 to 10^{10} particles/Liter
Filter Paper-WDXRF:	No actinides found on the filter papers. Seven of 13 filters had Fe even though Fe was not detected as a soluble cation. Sr and S were detected on 8 filters.
Headspace Gas Content:	~30 v/o H_2, ~ 1.2 v/o O_2, <<1% N_2O

D&D Observations (February 1, 2001):

Corrosion:	There was a black coating on the lid and O-ring that was hard, (i.e., could not be scratched with a screwdriver). The screen was black, but not impacted.
Brine:	The brine was a pale green color, that was clear, (i.e., did not contain sediment.
Bottom Solids:	These solids were soft and non-consolidated. The color of the very loose sludge was grayish.
Fe Mesh:	No Fe mesh added to this test container.

Overall Assessment:

Liter-scale 12 was the Castile Brine experiment of the set LS 10, 11, and 12. Whereas L10 was an experiment with a 10:1 ratio, L11 had a 2:1 ratio, and L12 had a 2:1 ratio. All three had 75 mg of ^{241}Am added as a soluble chloride complex for a theoretical concentration of 37,500 ppb if the Am remained soluble. The pcH of LS 10, 11, and 12 ranged from 8.2 - 8.9, 9.7 – 10.7, and 12.7 – 13.0, respectively. The Pu and Am were typically less than 5-10 ppb for all three tests and no Fe was identified as a soluble cation. No Pu or any other actinide was found as filterable colloids or microprecipitates. No Fe was found in L10 filters but L11 and L12 filters contained Fe even though Fe mesh was not added to the three tests. There was corrosion of SS feedthroughs in L10 and L11. There was no visible corrosion in L12 (only a black coating). The H_2 content of L10, 11, and 12 was 32.5 v/o, 22.2 v/o, and 30 v/o respectively for the three containers. There was ~1.5, 0.90, and 1.2 v/o oxygen for this three test containers that indicates

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that the ^{241}Am produced this effect. The high pcH in L12 may have been the cause for the very low concentrations of Ca and Mg found in this Castile Brine test. There were about 10^9 to 10^{10} particles per liter in each test.

Liter-Scale No. 13

Test Characteristics:

Waste:	Envirostone 80–90 % $CaSO_4$ with 10-20 % melamine-formaldehyde and 0.1% NH_4Cl Solid Absorbed Organic Liquid Waste
Total Waste Weight:	1,320 g
Initial Actinide Content:	Pu 2575 μg/g; Total Pu = 3.40 g Am 1.92 μg/g; Total Am = 2.53 mg
Brine:	Brine A (2:1 brine/solid ratio)
Additives:	Fe Mesh; Nd, Th, U, Np Added organic solvents

Soluble Actinide Histories: (5/1/95 – 3/1/99)

Pu -	Started at 40 ppb and varied between 10-90 ppb until final concentration of 26 ppb. There did not seem to be a trend.
Am -	Generally < 1 ppb except for two results < 2 ppb.
U -	Started at 2353 ppb and varied between about 1700 to 6000 ppb and leveled off at 1000 – 2000 with a final result at 1295 ppb. There may have been a trend downward.
Th -	Varied between 2 to 24 ppb for entire test with no apparent trend.
Np -	< 4 ppb for entire test period; no trend.
Nd -	Generally < 5 ppb with no apparent trend.

Other Analyses (nominal):

Typical pcH Range:	7.0-7.2
Fe Concentrations:	Started at 14 ppm and increased to 326 ppm after six months and slowly decreased to 35 ppm. Organics seemed to solubilize Fe in this set of tests.
Other Analytes:	Ca 1,300 ppm K 1,200 ppm Mg 32,000 ppm Na 41,000 ppm Pb 4-5 ppm average
TIC/TOC:	60/3,300 ppm
Particle Concentration:	10^{12} to 10^{13} particles/Liter
Filter Paper-WDXRF:	Fourteen of 17 filters contained Pu; all filters had Fe; only 2 had Sr. This was a high number of Pu bearing colloids or microprecipitates.

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The Fe in the filters was consistent with the soluble Fe throughout the test.

Headspace Gas Content: H_2 = 56.6 v/o; O2 = 0.20 v/o; CO_2 = 4.4 v/o; TOC = 3300 ppm.

D&D Observations (1-22-01):

Corrosion: No corrosion on lid or screen.

Brine: Clear, with thin layer of suspensions floating atop the brine.

Screen: Yellowish pasty material in the screen.

Bottom Solids: Approximately 4 inches of solids with a consistency of peanut butter that was harder at the bottom but not cemented.

Fe Mesh: The plastic holder was full of greenish-yellow sediment. There was a green color next to the mesh. The compacted sediment washed away readily. The washed wire mesh was black and did not appear corroded and the cut ends were still shiny. The Fe results indicate that there was some dissolution of Fe but it must have been uniform and not noticeable.

Overall Assessment:

LS-13, 14, and 15 had a variety of added organics that had a significant effect on the chemistry of these tests. For LS-13 with Brine A and a pcH that was about neutral to slightly basic, there was relatively low levels of Pu solubilized (10-90 ppb) but the concentration of Pu persisted throughout the test. The concentration of Fe in this test was relatively high and persisted throughout the test period. There was no corrosion of the SS feedthroughs in the lid but a green coloration around the Fe mesh attests to the presence of ferrous chloride in the brine. The presence of organics increased the particle concentration in the brine and Pu and Fe were present in the filter papers, which indicated colloids or microprecipitates of both Pu and Fe. The added organics (TOC = 3300 ppm), resulted in a high H2 content (57 v/o), low O_2 content (0.20 v/o), and measurable CO_2 (4.4 v/o). Of note is that soluble Fe, Pu, and U were correlative in this test.

Liter-Scale No. 14

Test Characteristics:

Waste: Envirostone
80-90% $CaSO_4$ with 10-20% melamine-formaldehyde and 0.1% NH_4Cl

Total Waste Weight: 1,320 g

Initial Actinide Content: Pu 2592 μg/g; Total Pu = 3.42 g
Am 2.54 μg/g; Total Am = 3.35 mg

Brine: Brine A (2:1 brine/solid ratio)

Additives: Fe Mesh; Th, U, Np, and Nd

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Soluble Actinide Histories: (5/1/95 – 4/5/99)

Pu - Started at 488 ppb and leveled at ~ 150 ppb until 12/2/96 and then decreased to ~ 75 ppb.
Am - Was generally less than 2 ppb with no trend.
U - Increased to ~ 5500 ppb after 6 months and then slowly decreased to ~ 1500 ppb at the end of the experiment (4/5/99).
Np - Ranged from 2-4 ppb for the length of the test. No trend was apparent.
Th - Ranged from 10-20 ppb for the length of the test. No trend was apparent.
Nd - Ranged from 5-25 ppb for the length of the test. No trend was apparent.

Other Analyses (nominal):

Typical pcH Range:	6.9 - 7.3
Fe Concentrations:	Ranged from 130 ppm to a high of ~466 ppm after 1 year and slowly decreased to ~ 100 ppm at end of the test.
Other Analytes:	Ca ~1,300 ppm K ~28,000 ppm Mg ~30,000 ppm Na ~40,000 ppm Pb ~3-6 ppm
TIC/TOC:	80/4000 ppm
Particle Concentration:	$3x10^{13}$ to $8x10^{13}$ particles/Liter
Filter Paper-WDXRF:	Pu found on 11 of 15 filter papers; Fe found on essentially all filter papers; Sr found on 4 filter papers and S was on essentially all filter papers. Fe was present in all pore-sized filters.
Headspace Gas content:	H_2 was 37.4 v/o; no other major gases quantified.

D&D Observations (January 18, 2001):

Corrosion: There was no visible corrosion of the SS fittings or feedthroughs. There was no apparent corrosion on the lid or screen, in fact, it looked new.
Brine: There were yellowish suspensions above a pool of brine. The liquid was non-viscous. About 1.5 liters was extracted.
Bottom Solids: The sludge near the bottom was the consistency of peanut butter, probably, powered Envirostone.
Fe Mesh: The Fe mesh was a dark color without corrosion and the cut ends were still shiny.

Overall Assessment:

This test had the highest TOC of the STTP experiments with a relatively high Pu content (3.42 g) and Am content (3.35 mg) but at a pcH of 6.9-7.3 (about neutral) the Pu was generally less than 200 ppb and Th, U, Np, and Nd less than 25 ppb. Uranium concentrations were as high as 5500 ppb but decreased to ~ 1500 ppb near the end of the test. Am was generally less than 2 ppb. Soluble Fe was prevalent for most of the test at about 100-470 ppm.

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There was no apparent corrosion of the SS feedthroughs or fittings in the headspace of the test containers. The Fe mesh showed no signs of corrosion in the Brine A at pcH 6.9-7.3. Pu was identified on most filter papers along with Fe. The particle concentration in this high organic solution was one of the highest in the STTP at $3x10^{13}$ to $8x10^{13}$ particles per liter. Apparently, the high particle concentration was associated with Pu and Fe as colloids or microprecipitates. The high Pu and Am content with TOC of 4000 ppm resulted in a 37 v/o H_2 concentration in the headspace of the test container but this was lower than LS-13 with a TOC of ~ 4000 ppm with a H_2 concentration of ~ 57 v/o.

Liter-Scale No. 15

Test Characteristics

Waste:	Envirostone 80-90% $CaSO_4$ with 10-20% melamine-formaldehyde and 0.1% NH_4Cl
Total Waste Weight:	1,320 g
Initial Actinide Content:	Pu 31.5 μg/g; Total Pu = 0.042 g Am 0.019 μg/g; Total Am = 0.025 mg
Brine:	Castile (2:1 brine/solid ratio)
Additives:	Fe Mesh; Th, U, Np, and Nd Added organic solvents

Soluble Actinide Histories: (5/1/95 – 4/5/99)

- Pu - Started at 57.5 ppb, and leveled at ~ 20 ppb and increased to ~ 165 ppb (peak) and then decreased to a final result of 22 ppb.
- Am - Less than 1.5 ppb for entire test period.
- U - Followed trend of Pu; started at 441 ppb and remained between 300 and 400 until 5/18/98 when the U increased to 2,600 ppb and peaked at 3454 ppb then finally decreased to 1,052 ppb at the end of the test.
- Np - Started at ~ 5,400 ppb, increased to 7753 ppb after 2-3 months and gradually decreased to 60 ppb at the end of the test period.
- Th - Generally < 10 ppb for the entire test period.
- Nd - Started at 544 ppb and slowly decreased to 22 ppb at the end of the test period. This was one of the few tests that solubilized Nd.

Other Analyses (nominal):

Typical pcH Range:	6.8 - 7.1
Fe Concentrations:	Started low at 1-5 ppm and slowly increased to 36 ppm at the end of the test. Mostly in the 20-36 ppm range.
Other Analytes:	Ca ~700 ppm K ~4,000 ppm

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	Mg ~1,000 ppm
	Na ~88,000 ppm
	Pb ~4.5 ppm (final analyses)
TIC/TOC:	40/1,400 ppm
Particle Concentration:	10^{12} to 10^{13} particles/Liter
Filter Paper-WDXRF:	No Pu identified on any filter paper. Fe identified on 5 of 14 filters. One filter had a very high Fe concentration (1128) on the 5 micron filter that also showed Th and Nd. This was unique in the STTP and must have been a sample with a large precipitate of Fe hydroxide that scavenged the Th and Nd. There was one high Sr result.
Headspace Gas content:	H2 = 3.0 v/o; O_2 = 0.01 v/o; TOC = 1,400 ppm.

D&D Observations (April 19, 2001):

Corrosion:	There was no visible corrosion of the SS fittings or feedthroughs. (Sample port, level probe, press gauge). Cream colored material on lid.
Screen:	Contained about 2 inches of oatmeal consistency solution. There was no corrosion on the metal o-ring.
Brine:	Cream colored brine with oatmeal consistency suspensions.
Bottom Solids:	Very loose material about 5-7 inches thick for this 2:1 ratio test.
Fe Mesh:	The plastic holder was full of fine loose solids but the Fe mesh after being washed was in good condition. It was gray to black in color with no visible evidence of corrosion.

Overall Assessment:

LS-15 was part of the set of LS 13, 14, and 15 that had added organic solvents in the Envirostone. This test was conducted at pcH 6.8-7.1 which is one of the few tests (except for pressurized tests L28, 29, and 30) conducted at a pcH level on the acid side of neutral. The Pu was not substantially solubilized (up to 165 ppb), Am remained very low (<1.5 ppb) and U followed the trend of Pu and increased to 3,454 ppb and then decreased to 1,052 ppb at the end of the test. Np followed a different trend by starting at 5,400 ppb and then decreasing for the rest of the test period to 60 ppb. Th was < 10 ppb for the entire test period. This is one of the few tests that showed Nd at a starting concentration of 544 ppb. The concentration of Fe was high throughout the test but only 5 of 14 filters had Fe. No filter paper showed Pu. The TOC was 1,400 ppm, but the H_2 in the headspace was low at 3.0 V/o; LS-13 had a H_2 level of ~57 v/o and LS-14 had a H_2 concentration of 37 v/o. There was no corrosion observed on the SS feedthroughs and the Fe mesh did not have observable corrosion.

Liter-Scale No. 16

Test Characteristics:

Waste:	Envirostone 80-90% $CaSO_4$ with 10-20% melamine-formaldehyde and 0.1% NH_4Cl
Total Waste Weight:	1,320 g
Initial Actinide Content:	Pu 470.0 μg/g; Total Pu = 0.620 g Am 0.25 μg/g; Total Am = 0.330 mg
Brine:	Brine A (2:1 brine/solid ratio)
Additives:	Fe Mesh; Th, U, Np, and Nd

Soluble Actinide Histories: (5/1/95 – 2/22/99)

Pu - Concentration initially was at 150-400 ppb for about 2 years and then it went into a steady increase for the remainder of the test. **This was one of the only tests that ended up with Pu increasing, and it increased to about 1,200 ppb.**

Am - Started at <0.5 ppb for about 2 years and then it increased similar to Pu, to about 3.0 ppb at the end of the experiment.

U - Started at 14,600 ppb and slowly decreased to 182 ppb during the life of the experiment

Np - Started at ~100 ppb and increased to ~200 ppb at end of test

Th - Started at 10 ppb and increased to about 280 ppb at end of test

Nd - Started at ~20 ppb and increased to ~50 ppb at end of test

Other Analyses (nominal):

Typical pcH Range:	7.3-8.0
Fe Concentrations:	Started very low (<0.1 ppm) and remained low for about 2 years, and then began a slow increase to ~33 ppm at end of test
Other Analytes:	Ca ~1,700 ppm K ~26,000 ppm Mg ~35,000 ppm Na ~40,000 ppm
TIC/TOC:	80/430 ppm
Particle Concentration:	10^9 to 10^{10} particles/Liter
Filter Paper-WDXRF:	Pu, Fe, and $SrSO_4$ identified as colloids or microprecipitates
Headspace Gas content:	H_2 = ~62 v/o; which is relatively high for Envirostone; N_2O = 2.5 v/o. NO_3 was ~ 100 ppm.

D&D Observations (December 19, 2000):

Corrosion: There was no visible corrosion of the SS fittings or feedthroughs. There was no apparent corrosion of the screen, in fact, it was like new.

Brine: The brine was tan colored and very cloudy and became thicker near the bottom of the container. Near the bottom the brine had to be scooped out, not poured, because of its high density of solids.

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Bottom Solids: The sludge near the bottom was tan to brown colored, but it was not cemented.

Fe Mesh: The Fe mesh was retrieved and appeared to be totally intact. The Fe mesh holder was totally full of thick sludge that was impacted. There must have been some corrosion or dissolution of the Fe mesh because there was a greenish-black color adjacent to the screen, but only inside the Fe mesh holder. The Fe mesh strands had a black coating that was quite hard. The Fe mesh with its black coating seemed to have retained spring and was not brittle.

Overall Assessment:

This test, L-16, with Envirostone in Brine A at a slightly basic pcH (7.3-8.0) was one of the only STTP tests that was terminated with Pu, Am, Np, Th, and Nd concentrations trends increasing. U was different, it began at 14,983 ppb and trended downwards 182 ppb during the test. The presence of a tinge of green color within the Fe mesh holder indicated that some Fe had dissolved as $FeCl_2$, but was not being released to the brine until near the end of the test. The soluble Fe concentration at pcH 7.3 – 8.0 could have been present as a Fe^{+2} cation.

The high hydrogen concentrations may have been responsible for the corrosion- free SS fittings in the headspace region. The tan color of the brine and suspensions indicate that there was little communication with the Fe mesh and the brine. The brine would have been green colored if there had been more dissolution of the Fe wire. The reason for the eventual increase in all the actinides, except U, is not known. U was high in most of the Envirostone tests, but decreased with time, except L-15. Pu as a filtered colloid or microprecipitate was present along with Fe and $SrSO_4$. Low nitrate concentration gave a low N2O content (<2.5 v/o). The increase in Fe concentration in the brine was concurrent with the increase in all the actinide concentrations. The Fe mesh was not dissolved and had a hard black coating.

Liter-Scale No. 17

Test Characteristics:

Waste:	Envirostone 80 – 90 % $CaSO_4$ with 10-20 % melamine- formaldehyde and 0.1 % ammonium chloride. Solidified absorbed aqueous waste
Total Waste Weight:	1,320 g
Initial Actinide Content:	Pu 1,140 μg/g; Total Pu = 1.50 g Am 2.05 μg/g; Total Am = 2.706 mg
Brine:	Brine A (2:1 brine/solid ratio)
Additives:	Fe Mesh; Th, U, Np, and Nd

Soluble Actinide Histories: (5/1/95 – 2/2/99)

Pu - Was always <20 ppb, and typically remained <3 ppb.

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U - Began at 1,300 ppb, increased to a high of 20,000 ppb by July 1995, and then steadily decreased to a low of 270 ppb as of the last sampling, February 1999.

Np - Began at 4 ppb, increased to a high of 25 ppb, March 1996, and then steadily decreased to a low <4 ppb, August 1997 through February 1999.

Other - Nd was typically <10 ppb, and Th and Am were ≤1 ppb.

Other Analyses (nominal):

Typical pcH Range:	7.7-8.0
Fe Concentrations:	<1 ppm
Other Analytes:	Ca ~2,000 ppm K ~25,000 ppm Mg ~30,000 ppm Na ~43,000 ppm
TIC/TOC:	70/350 ppm
Particle Concentration:	10^9 to 10^{10} particles/Liter
Filter Paper-WDXRF:	Pu precipitates were found, which appear to be associated with Fe and Ca.

H_2 Headspace Gas Content: ~7.7 v/o H_2; ~ 20 v/o N_2O; NO_3 was ~ 13,000 ppm.

D&D Observations (February 16, 2001):

Corrosion: There was no visible corrosion on the lid, and everything appeared in clean and good condition.

Brine: The brine was tan in color and there were no suspensions or crystals noted.

Bottom Solids: The sludge near the bottom was tan and loose. The solids at the bottom were loose and 'mushy'.

Fe Mesh: The iron mesh was recovered, and it was in good condition. It was only slightly embedded and there was not much solid material surrounding it. There was a small amount of tan colored material deposited on the Fe mesh screen, which easily rinsed off. After rinsing the screen, it was black with no shiny edges.

Overall Assessment:

LS 17 was a Brine A test with a 2:1 brine/solid ratio at pcH 7.7 – 8.0 that showed essentially no soluble Nd, Th, Np, Pu, and Am but did have high U early in the test (20,000 ppb) which diminished with time to ~260 ppb at the end of the test. The reason for the behavior of U is not known. LS 17 had a high nitrate concentration that yielded an N_2O concentration of ~ 20 v/o in the headspace of the test container. The H_2 concentration of ~ 7.7 v/o was quite low relative to other test containers. There were 14 of 16 filters that contained Pu which was surprising because the soluble Pu concentration was < 3 ppb for most of the test. All filters had Fe which was also surprising because the soluble Fe was generally less than 1 ppm. The Pu and Fe in the filter papers attests to the presence of Pu and Fe bearing colloids or microprecipitates throughout the test. The nitrate in this test container had to be totally soluble in brine but the radiolytic

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production of N_2O was probably from solid or precipitated species of Pu and Am. The activity of 3nCi/ml is about 110 d/s/ml is probably too low to generate 20 v/o of N_2O but a precipitated activity of 1.5 g of Pu and 2.7 mg of Am would be sufficient activity to generate that amount of N_2O. The Fe wire was not corroded in this test.

Liter-Scale No. 18

Test Characteristics:

Waste:	Envirostone, 80 – 90% $CaSO_4$ with 10 – 20% melamine formaldehyde and 0.1% ammonium chloride Solidified absorbed aqueous waste
Total Waste Weight:	1,320 g
Initial Actinide Content:	Pu 1,970 μg/g; Total Pu = 2.60 g Am 26.48 μg/g; Total Am = 34.9 mg
Brine:	Castile (2:1 brine/solid ratio)
Additives:	Fe Mesh; Th, U, Np, and Nd

Soluble Actinide Histories: (5/1/95 – 3/8/99)

Pu - Was typically ≤10 ppb, however spiked to 210-220 ppb on two separate occasions, Oct. 1996 and Aug. 1998.

U - Began at a low of 2,000 ppb, increased to a high of 27,000 ppb by Jan. 1996, and has since steadily decreased to a low of 9,500 ppb as of the last sampling, March 1999.

Np - Began at a low of 14 ppb, increased to a high of 1,600 ppb by Nov. 1995, and has since steadily decreased to typically 19-22 ppb, Aug. 1998 to Jan. 1999.

Other - Nd was typically <5 ppb, Th and Am were typically <3 ppb.

Other Analyses (nominal):

Typical pcH Range:	7.0-7.8
Fe Concentrations:	Increased to about 50 ppm after one year and then decreased to <2 ppm for remainder of test.
Other Analytes:	Ca ~660 ppm K ~4,400 ppm Mg ~900 ppm Na ~90,000 ppm
TIC/TOC:	40/420 ppm
Particle Concentration:	10^9 to 10^{10} particles/Liter
Filter Paper-WDXRF:	Twelve of 14 filter papers had significant Pu colloids or precipitates. Two of three filter papers had Np and U precipitates. These precipitates did not appear to be directly related to Fe or Sr precipitates.
Headspace Gas Content:	~50 v/o H_2; 1 –2 v/o N_2O; NO_3 was ~ 100 ppm.

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D&D Observations (January 9, 2001):

Corrosion: There were orange-brown colored solids attached to the lid and screen.

Brine: The brine was clear with suspended clay-like particles that gave the brine a yellow color in appearance.

Bottom Solids: The solids at the bottom of the container were soft and not compacted or cemented.

Fe Mesh: The Fe mesh was surrounded by a black clay-like material that easily rinsed off. After rinsing the mesh, a hard black surface coating was noted. The mesh was still intact.

Overall Assessment:

LS 18 was the test with Envirostone in Castile brine from pcH 7.0 – 7.8 which is just basic. This test had ~ 2.6 g of Pu and 35 mg of Am which is relatively high for Envirostone tests. Soluble Pu and Am were fairly low (< 10 ppb and 1 ppb, respectively) and Fe was generally < 2 ppm after the first year of the test. U generally at the 22,000 ppb level had decreased to about 9,000 ppb near the end of the test. Although the soluble Pu had been low for most of the test, there was Pu in the filter papers indicating that Pu had precipitated or formed colloids that were filtered during the test. The Fe mesh was still intact and black colored and did not dissolve. There was a hard black coating around each strand. The bottom solids were loose and not compacted. The brine was nearly colorless and the crystals throughout the test container had a color like butterscotch in this 2:1 brine/solid ratio test. There did not appear to be corrosion of the lid or feedthroughs.

Liter-Scale No. 19

Test Characteristics:

Waste: Envirostone 80–90% $CaSO_4$ with 10–20% melamine formaldehyde and 0.1% ammonium chloride
Solidified inorganic sludge

Total Waste Weight: 1,320 g

Initial Actinide Content: Pu 380 μg/g; Total Pu = 0.502 g
Am 12.75 μg/g; Total Am = 16.8mg

Brine: Brine A (2:1 brine/solid ratio)

Additives: Fe Mesh; U, Th, Np, and Nd

Soluble Actinide Histories: (5/1/95 – 3/1/99)

Pu - Typically ≤10 ppb throughout the life of the experiment.

U - Began high at 21,000 ppb, increased a little higher to 26,000 ppb in July 1995, and has since steadily decreased to a low of 570 ppb as of the last sampling, March 1999.

Np - Typically varied from 20-45 ppb throughout the life of the experiment.

Nd - Was typically 15 ppb for the first few months of testing and then decreased to <5 ppb for the last two years of sampling.

Other - Th was typically <5 ppb and Am was typically <1 ppb.

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Other Analyses (nominal):

Typical pcH Range:	7.9-8.2
Fe Concentrations:	<1 ppm for entire test period
Other Analytes:	Ca 1,100 ppm K 23,000 ppm Mg 28,000 ppm Na 50,000 ppm
TIC/TOC:	70/700 ppm
Particle Concentration:	10^9 to 10^{10} particles/Liter
Filter Paper-WDXRF:	Six of eleven filter papers had low levels of Pu. Essentially all filters had Fe and Sr.
Headspace Gas Content:	~16 v/o H_2; 32 v/o N_2O; 36,000 ppm NO_3

D&D Observations (February 16, 2001):

Corrosion:	No corrosion was noted on lid and the screen looked like new. There was a thin orange coating noted around SS sampling port.
Brine:	The brine was very clear above the screen, but it was cloudy and yellow colored beneath the screen.
Bottom Solids:	The solids at the bottom of the container were a soft sludge.
Fe Mesh:	The Fe mesh container was coated with a soft sludge. The inside of the container consisted of a blackened sludge. The Fe mesh was slightly dissolved, and had a very thin hard black coating.

Overall Assessment:

LS 19 was an Envirostone test in Brine A at a pcH of 7.9 – 8.2 and a brine–to-solid ratio of 2:1. Pu did not solubilize to more than 10 ppb during the test period and Am was <1 ppb. There was no corrosion on the lid or SS feedthroughs. The brine above the screen was clear but had a yellowish color below the screen. There had been difficulty in sampling this test container because finely divided waste would plug the sampling needle. The orange color on the lid and the slightly yellow color in the brine may indicate an oxidizing condition for this test container. This test had a high concentration of nitrites (36,000 ppm) and a resulting high concentration of N_2O (32 v/o) that precluded a high concentration of H_2 (16 v/o). Although the soluble Pu concentration was low (0-10 ppb) and Fe was < 1 ppm for this test.

The filtered brine showed Pu and Fe on many of the filter papers indicating the presence of colloids or microprecipitates containing Pu and Fe in the Brine A. The Fe mesh was surrounded by a black sludge and was slightly corroded by the Fe wire strands which were covered with a hard coating.

Liter-Scale No. 20

Test Characteristics:

Waste:	Envirostone Solidified inorganic sludge
Total Waste Weight:	1,320 g
Initial Actinide Content:	Pu 60.5 μg/g; Total Pu = 79.86 mg Am 7.07 μg/g; Total Am = 9.33mg
Brine:	Brine A (2:1 brine/solid ratio)
Additives:	Fe Mesh; U, Th, Np, and Nd

Soluble Actinide Histories:

Pu - Started at 1.3 ppb and peaked at 12.5 ppb with no apparent trend.
Am - Was < 0.8 ppb for entire test.
U - Started at 11,101 ppb and has steadily decreased to a low of 230 ppb on last sample taken (10/18/99).
Th - Was < 2.0 ppb for entire test.
Np - Was generally < 2 ppb for entire test.
Nd - Was < 4 ppb for entire test.

Other Analyses (nominal):

Typical pcH Range:	7.0-7.8
Fe Concentrations:	Started at average of about 80 ppm and ended with about an average of 20 ppm.
Other Analytes:	Ca 1,200 ppm K 25,000 ppm Mg 30,000 ppm Na 50,000 ppm Pb 8 ppm
TIC/TOC:	50/350 ppm
Particle Concentration:	4×10^{10} particles/Liter
Filter Paper-WDXRF:	No Pu or Fe in filters. This is not surprising because Pu concentrations were very low but Fe was actually quite high (20-80 ppm) during the test period.
Headspace Gas Content:	H_2 was 2.9 v/o from a nitrate concentration of 30,000 ppm.

D&D Observations): *No available data.*

Overall Assessment:

This Envirostone test had a very low Pu concentration for the entire test. U started at 11,101 ppb but steadily decreased during the entire test period. The high nitrate concentration resulted in a high N_2O concentration in the headspace. There was no filterable colloids containing Pu or Fe at a pcH of ~ 7.5.

Liter-Scale No. 21

Test Characteristics:

Waste:	Envirostone Solidified inorganic sludge
Total Waste Weight:	1,320 g
Initial Actinide Content:	Pu 190.0 μg/g; Total Pu = 0.251 g Am 24.75 μg/g; Total Am = 32.67 mg
Brine:	Castile (2:1 brine/solid ratio)
Additives:	Fe Mesh; U, Th, Np, and Nd

Soluble Actinide Histories: (5/1/95 – 11/9/99)

Pu -	Generally less than 2 ppb, with occasional 8 ppb.
U -	Began around 5,000 ppb, and slowly decreased to 750 ppb at the end of testing.
Np -	Began at 5,000 ppb and quickly dropped to ~10 ppb after six months, and typically remained <10 ppb.
Am -	Typically less than 1 ppb
Th and Nd -	Typically less than 1 ppb.

Other Analyses (nominal):

Typical pcH Range:	7.5-8.1
Fe Concentrations:	<0.1 ppm
Other Analytes:	Ca 600 ppm K 5,000 ppm Mg 900 ppm Na 100,000 ppm
TIC/TOC:	25/300 ppm
Particle Concentration:	10^9 to 10^{10} particles/Liter
Filter Paper-WDXRF:	No Pu or actinide precipitates; 3 of 16 filters contained Fe; no Sr but high S in filters.
Headspace Gas Content:	H_2 = 8 v/o; N_2O = 19 v/o; NO_3 = 32,000 ppm

D&D Observations (November 9, 1999):

Corrosion:	No visible corrosion.
Brine:	Clear with an orange tinge.
Bottom Solids:	Loose solids.
Fe Mesh:	No corrosion; black colored coating.

Overall Assessment:

LS 21 was one of the earliest test containers to be deactivated and decommissioned because it had become so difficult to sample. This was a Castile brine test with a brine to solid ratio of 2:1 as was L 19 and L 20. The pcH of this test ranged from 7.5 – 8.1 which was similar to L 19 and L 20. The brine was clear with an orange tinge and the bottom solids were loose with an orange tinge.

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The Fe mesh had a black coating and did not show signs of corrosion. There was essentially no solubilization of Pu, Am, Th, Nd (< 1 ppm) and U started at ~5000 ppb and decreased to ~750 ppb while Np began at about 5000 ppb and decreased to <10 ppb. There was no soluble Fe (< 1 ppm) detected in this test. There were no colloids or microprecipitates containing Pu or other actinides on filters. Fe was detected on only 3 filters at a low level. H_2 was relatively low at ~ 8 v/o but N_2O was high at ~ 19 v/o (NO_3 was at 32,000 ppm). Overall, this test showed essentially no solubilization of actinides or Fe and the Fe mesh was essentially untouched.

Liter-Scale No. 22

Test Characteristics:

Waste:	Envirostone 80-90 % $CaSO_4$ with 10-20% melamine-formaldehyde and 0.1% NH_4Cl
Total Waste Weight:	1,320 g
Initial Actinide Content:	Pu 180 μg/g; Total Pu = 0.238 g Am 6.51 μg/g; Total Am = 8.59 mg
Brine:	Brine A (2:1 brine/solid ratio)
Additives:	Fe Mesh; Nd, Th, U, Np

Soluble Actinide Histories: (5/8/95 – 2/22/99)

Pu -	<5 ppb except for one analyses
Am -	<1 ppb except one analyses of 1.4 ppb
U -	Started at 8822 ppb and decreased to ~1100 ppb where it remained for ~3 years and then decreased to 619 ppb.
Np -	Generally < 1 ppb.
Th -	Generally < 1 ppb.
Nd -	Generally < 5 ppb.

Other Analyses (nominal):

Typical pcH Range:	6.8 – 7.2
Fe Concentrations:	Generally 50 to 120 ppb and then down to 16 ppb on last analyses.
Other Analytes:	Ca 2,100 ppm K 26,000 ppm Mg 30,000 ppm Na 46,000 ppm
TIC/TOC:	35/460 ppm
Particle Concentration:	10^{10} to 10^{11} particles/Liter
Filter Paper-WDXRF:	Six of 15 filters had Pu and all filters had Sr and S. There seemed to be a collaboration between Pu and Sr.
Headspace Gas Content:	H_2 = 6.1 v/o; N_2O = < 39 v/o; O_2 = 0.15 v/o; NO_3 = 36,000 ppm; TOC = 480 ppm

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D&D Observations (1-24-01):

Corrosion: No corrosion noted on SS fittings and feedthroughs; no corrosion around screen; screen had ~ ¾ - inch of gold colored sediment.

Brine: Brine has much sludge and was gold colored but still a liquid with some clarity, but has a light brown color tending to gold.

Bottom Solids: About 7 inches of a gold colored sludge or mud that was easily stirred.

Fe Mesh: The Fe mesh was in 3 pieces within the plastic holder. The rinse water was avocado green. This implies a reducing condition and may have been the reason for the high Fe content throughout the test period. Also, the Fe in the filters can be explained from leaching of the Fe wire strands. After rinsing, the Fe mesh was black in color and did not seem to be corroded. The Fe wire strands did not seem to be corroded.

Overall Assessment:

LS-22 did not solubilize Pu (< 5 ppb) over the test period. U was solubilized to a certain extent but other actinides were not. Fe was solubilized during the entire test period at the 50 to 120 ppm level. The 5 micron sized filter papers picked up Fe in all the samples taken (15 each) and Pu at a low level was found in 6 of 15 filters. Both Pu and Fe were found in the 5 micron filters and not the 1 micron or 10 nm sized filters. The TOC at ~460 ppm was higher than most and the particle concentration at 10^{10} to 10^{11} was on the high side of average. There was no corrosion on the SS feedthroughs on the lid or the screen. There was no cemented solids but about 7 inches of loose solids with a consistency of mud. There was a green color around the Fe mesh that indicates some solubilization of Fe as ferrous chloride. The nitrate concentration that was ~ 3,600 ppm led to an N_2O concentration that was much lower or at about 6.1 v/o. The Fe mesh was black and did not appear to be corroded but the green coloration within the plastic holder, which indicates differently.

Liter-Scale No. 23

Test Characteristics:

Waste: Envirostone
80 – 90 % $CaSO_4$ with 10-20 % melamine- formaldehyde and 0.1 % ammonium chloride.
Solidified organic waste

Total Waste Weight: 1,320 g

Initial Actinide Content: Pu 380 μg/g; Total Pu = 0.502 g
Am 9.55 μg/g; Total Am = 12.61 mg

Brine: Brine A (2:1 brine/solid ratio)

Additives: Fe Mesh; Th, U, Np, and Nd

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Soluble Actinide Histories: (5/8/95 – 3/1/99)

Pu - <10 ppb except one result at 14 ppb. Generally < 2 ppb. No trend observed..
Am - < 0.5 ppb entire test period.
U - Started at 1767 ppb and increased to a peak of 6247 ppb after 2 months and then decreased steadily to a final result of 261 ppb.
Np - Generally < 1 ppb except 7.2 and 1.7 ppb.
Th - Generally < 1 ppb except 2.6 ppb. No trend observed.
Nd - Generally < 5 ppb for test period. No trend observed.

Other Analyses (nominal):

Typical pcH Range:	7.0 – 7.4
Fe Concentrations:	10 to 102 ppm with an average of ~50ppm and a final at 3.2 ppm.
Other Analytes:	Ca ~2,700 ppm K ~28,000 ppm Mg ~36,000 ppm Na ~60,000 ppm Pb 3-12 ppm.
TIC/TOC:	30/480 ppm
Particle Concentration:	10^{10} to 10^{11} particles/Liter
Filter Paper-WDXRF:	Pu found in 6 of 12 filters. Fe was found at high levels in all 12 filters in the 5 micron filters and to a lesser extent in the 10 micron filters. Sr was found on 9 of 12 filters and correlation is made with the Pu filters..
H_2 Headspace Gas Content:	H_2 = 8.0 v/o; N_2O = 31.3 v/o; N_2 = 6.4 v/o; NO_3 was 30,000 ppm; TOC = 400 ppm.

D&D Observations (April 12, 2001):

Corrosion:	Fairly clear of corrosion, perhaps discoloration at SS feedthroughs.
Brine:	Gold colored brine pool about 6 inches in depth.
Screen:	No corrosion; thin coating; easy to remove.
Bottom Solids:	There was about 6 inches of loose solids that could be easily stirred. Solids are gold colored.
Fe Mesh:	The iron mesh was easily removed and totally exposed to brine pool. Fe wires were black colored and appeared to be in good shape.

Overall Assessment:

There was little solubilization of Pu (< 2 ppb) over the test period and Pu was found as colloid or microprecipitation on 6 of 12 filter papers. U was solubilized to a peak of 6,247 ppb after two months and gradually decreased to 261 ppb at the end of the test period. The other actinides did not solubilize. The concentration of Fe at pcH 7.0-7.4 in the Brine A was quite high (10 to 102 ppm) and all filters had Fe. Pu seemed to correlate with Sr on the filters. This was a high nitrate concentration test with NO_3 at 30,000 ppm and N_2O at 31.3 v/o. There was essentially no corrosion of the SS feedthroughs and the Fe wire was not impacted like most other Fe wire holders. The bottom solids were not cemented and quite loose and easily stirred.

Liter-Scale No. 24

Test Characteristics:

Waste:	Envirostone 80 – 90 % $CaSO_4$ with 10-20 % melamine- formaldehyde and 0.1 % ammonium chloride. Solidified organic sludge
Total Waste Weight:	1,320 g
Initial Actinide Content:	Pu 231 μg/g; Total Pu = 0.305 g Am 8.19 μg/g; Total Am = 10.8 mg
Brine:	Castile Brine (2:1 brine/solid ratio)
Additives:	Fe Mesh; Th, U, Np, and Nd

Soluble Actinide Histories: (5/8/95 – 2/2/99)

Pu - Generally <5 ppb with one result of 16 ppb.
Am - All results < 0.7 ppb.
U - Began at 1,575 ppb and varied between 1,500 and 2,000 ppb and then decreased slowly to 267 ppb.
Np - Started at 730 ppb and decreased to <5 ppb for the remainder of the test period.
Nd - < 1 ppb for test period.

Other Analyses (nominal):

Typical pcH Range:	7.6 – 7.9
Fe Concentrations:	Generally <1 ppm with four analyses between 1 and 13 ppm.
Other Analytes:	Ca ~800 ppm K ~5,000 ppm Mg ~2,000 ppm Na ~90,000 ppm Pb < 0.1 ppm until final 8 analyses showed 2-6 ppm.
TIC/TOC:	20/380 ppm
Particle Concentration:	10^9 to 10^{10} particles/Liter
Filter Paper-WDXRF:	Five of 14 filters showed just detectable Pu and all filters showed relatively high levels of precipitated or colloidal Fe in the 5 micron-sized filters.
H_2 Headspace Gas Content:	12.7 v/o H_2; 22.5 v/o N_2O; 25.0 v/o N_2; 0.02 v/o O_2; NO_3 was 32,000 ppm (this was the highest N_2 content of STTP)

D&D Observations (April 12, 2001):

Corrosion:	Clear of corrosion except perceptible corrosion ring around the sampling port.
Brine:	High turbidity with white chalky liquid that contained floating black specks. The color of the murky brine pool was yellow-olive drab.

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Bottom Solids: There was about 6 inches of a clay like mass that was loose on the top and hardened near the bottom. It was not cemented. There was a semi-gelatinous cohesive mass on the bottom of the screen.

Fe Mesh: The iron mesh plastic holder was full of a silt clay mass that could be washed off with water. After washing off the impacted solids, the Fe mesh was covered by a greenish-black deposit that was very heavy and the wire was somewhat corroded under the heavy coating.

Overall Assessment:

LS-24 was a Castile brine test at pcH 7.6 – 7.9 with a 2:1 brine to solid ratio in Envirostone. There was very limited solubilization of Pu (< 5ppb) and essentially no solubilization of Np, Th, Nd, and Am. U, as typical of Envirostone tests, had 1575 ppb in solution that slowly decreased to ~ 267 ppb at the end of the experiment. The soluble Fe concentration was < 1 ppm throughout the test. Although Pu was just barely detected in the brine as a soluble cation, 5 of 14 filters had low levels of Pu and all filters had low levels had Fe as colloids or microprecipitates.

The SS feedthroughs appeared not to be corroded but there was a perceptible corrosion ring around the sampling port. The screen was intact and not corroded. The Fe mesh holder was full of a silt-like material and after washing the Fe strands were covered with a greenish-black coating. There appeared to be corrosion of the Fe wire. This was a high nitrate (32,000 ppm) content in this test that yielded a high N_2O concentration in the headspace gas. H_2 was only 12.7 v/o but there must have been some radiolytic generation of N_2 at 25 v/o. There was about 6 inches of clay like mud that thickened as it approached the bottom.

Overall, there was essentially no solubilization of Pu and other actinides and any actinides and Fe that did solubilize were immediately precipitated. There was not much SS corrosion and the corrosion led to Fe precipitates that were identified in the filter paper samples.

Liter-Scale No. 25

Test Characteristics:

Waste:	Pyrochemical salts (Direct Oxide Reduction - DOR)
Total Waste Weight:	1320 g
Initial Actinide Content:	Pu 285 μg/g; Total Pu = 0.376 g
	Am 0.30 μg/g; Total Am 0.396 mg
Brine:	Brine A (2:1 Brine/Solid ratio)
Additives:	Fe Mesh: Nd, Th, U, Np

Soluble Actinide Histories: (5/8/95 – 12/6/99)

Pu - Started at 106 ppb and varied from 10 to 127 ppb for entire test period. Final result was 40 ppb but no trend was observed.

Am - Less than 1 ppb for entire test period.

U - Generally < 10 ppb for entire test period. No trend was observed.

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Np - Started low at 4.7 ppb and remained low for 7-8 months and increased steadily to 348 ppb and then began a slow decrease to 154 ppb at the end of the test period.
Th - Generally < 3 ppb; no trend observed.
Nd - Generally < 7 ppb; no trend observed.

Other Analyses (Nominal):

Typical pcH Range: 7.7 to 8.1
Fe Concentration: Typically < 1 ppm with about 50% of results that varied from 1-20 ppm. The trend seemed to be towards < 1 ppm.
Other Analytes: Ca ~80,000 ppm
K 22,000 ppm
Mg 22,000 ppm
Na 8,000 ppm
TIC/TOC: 15/25 ppm
Particle Concentration: 10^{10} to 10^{11} particles/L
Filter Paper-WDXRF: Pu was identified at a low level in 9 of 15 filters. No Fe was detected on any filter paper. There was high Sr and S identified on all filters. There was a correlation between Sr and Pu.
H_2 Headspace Gas Content: H_2 = 13 v/o; O_2 = 0.08 v/o

D&D Observations (4/3/01):

Corrosion: Green color of corrosion around SS feedthroughs.
Screen: Corrosion around half of the o-ring; screen about 1/3 full of white crystals and sludge.
Brine: Clear with much suspensions.
Bottom Solids: Compacted solids ~ 5 inches in depth.
Fe Mesh: Salt crystals all over plastic holder. Compacted salt in holder was a light blue.

Overall Assessment:

LS-25 was part of a set of Pyrochemical salt tests of LS-25, 26, and 27. Pyrochemical salt tests had much higher Pu loadings than other waste forms but LS-25 had the lowest Pu loading of the Pyrochemical salts. There was limited solubilization of Pu (10-127 ppb) in this Brine A test at a pcH range of 7.7 to 8.1. There was Fe solubilization at a low level during the test but no Fe was found in any filter paper. There was Pu at a low level in 9 of 15 filter papers so there was some colloidal species of Pu in the particle population. The lower Pu loading led to the lowest H_2 concentration in the Pyrochemical salt tests of 13 v/o. The H_2 generation rate can be related to the radiolytic effectiveness of the alpha activity.

There was evidence of corrosion on the SS feedthroughs and there was a green coloration that is a reduced form of Fe. The screen o-ring also had corrosion on half of the o-ring. The brine was rather clear but loaded with suspensions that settled. The Fe mesh showed some indication of dissolution based on the light blue color on the salts adjacent to the Fe mesh. This test seemed to have a reducing environment based on the color of the Fe compounds.

Liter-Scale No. 26

Test Characteristics:

Waste:	Pyrochemical salts (Direct Oxide Reduction - DOR)
Total Waste Weight:	1320 g
Initial Actinide Content:	Pu 3075 μg/g; Total Pu = 4.06 g Am 1.71 μg/g; Total Am 2.25 mg
Brine:	Brine A (2:1 Brine/Solid ratio)
Additives:	Fe Mesh: Nd, Th, U, Np
Other:	None

Soluble Actinide Histories: (5/8/95 – 12/6/99)

Pu - Started at 42.9 ppb on 5/8/95 and increased to 70,000 ppb on 12/2/96 slowly decreasing to 3287 ppb at the end of the test (12/6/99). Pu (VI) was identified on 3/4/97 and was present until 8/24/98 (~ 1-1/2 years).

Am - Followed general trend of Pu up to 165 ppb on 12/2/96 and decreased to 9.9 ppb at end of test (12/6/99).

Other - Nd, Th, and Nd were less than 5 ppb and U was less than 15 ppb.

Other Analyses (Nominal):

Typical pcH Range:	7.6 to 8.2
Fe Concentration:	Very low, 0.1 ppm generally with an occasional sample up to 3 ppm.
Other Analytes:	Ca 100,000 ppm K 21000 ppm Mg 25,000 ppm Na 11,000 ppm
TIC/TOC:	20/30 ppm
Particle Concentration:	10^{11} to 10^{12} particles/L
Filter Paper-WDXRF:	Pu, Sr, and S correlate on the 5 micron filter paper data. Fe was not detected. Pu was identified in 5 micron filter papers but not in 1 micron or < 10μm filters. Ca was ~1000 to 5000 units.

H_2 Headspace Gas Content: ~73 v/o H_2.

D&D Observations (1/30/01):

Corrosion:	Lid and SS fittings were corroded, the high H_2 gas content (73%) in the headspace might have had an influence on this.
Brine:	Fairly clear, with a light yellow or straw color.
Bottom Solids:	3 to 3-1/2 inches uncemented solids.
Fe Mesh:	Recovered; one spot with about a 4-cm diameter, was ~ 50% corroded with remainder of wire being essentially uncorroded. Black deposit on coating prevented corrosion of wire.

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Overall Assessment:

There appeared to be early corrosion of the Fe wire mesh in one spot. The Pyrochemical salt sludge and encrustations may have prevented free brine flow through Fe mesh plastic holder. The Fe mesh was impacted in the holder and developed a hard black coating that prevented further dissolution. Throughout the history of this test container all the Pyrochemical waste sludge was mostly available for exposure to the brine. Also, the soluble Fe concentrations were very low in this container. These two reasons could explain why Pu was so high in the container. Those two reasons could explain why Pu was so high in this container and the presence of Pu (VI) for such a long interval.

Nd, Th, Np, and U concentrations were generally less than 5 ppb and U was less than 15 ppb during the history of L26. Pu and Am were the only actinides that solubilized in this experiment. Because most of the 1320 grams of waste, including actinides, was available to the brine, radiolytic activity in the brine would be expected to be high for this experiment. The straw yellow color of the brine would indicate that Fe did not have a major influence in this test. This may also be the reason for the high Pu (VI) concentration in this test container. The particle concentration in L26 was relatively high.

The comminution of the Pyrochemical salt waste could have had a significant impact on the chemistry of this test because of the limited flow of brine to contact the Fe mesh. The percentage of Pu in the waste that solubilized in LS26 was as high as 0.5%, one of the highest in the STTP.

All the 5 micron sized filter papers (17 each) contained Pu as well as Sr and Sulfate, but no Fe was found in any filter papers regardless of size (5 micron, 1 micron, or <10μm). Apparently, there may be Pu colloids or microprecipitates associated with $SrSo_4$ or hydrated Pu, since Fe was not present as a filterable precipitate. The Pu was associated with microprecipitates larger than 5 micron but was not associated with precipitates in the 1 micron or <10μm range.

The H_2 gas content (~ 73 v/o) was very high for this experiment and there was no corrosion of the SS fittings; however, LS27 had much corrosion at a H_2 concentration of 65 v/o. Perhaps the major differences in the two containers is, the pcH for LS26 was 7.6 – 8.2 whereas the pcH for LS27 was 10.7 – 11.2.

Overall, this test had high concentrations of Pu and Am and very low concentrations of Nd, Th, Np, U, and Fe. The Fe mesh in the plastic container was essentially isolated from the brine by compaction of comminuted waste in the plastic holder. All the actinides were available to the brine because there was no cementation. This had to be an oxidizing environment.

Liter-Scale No. 27

Test Characteristics:

Waste:	Pyrochemical salts (DOR)
Total Waste Weight:	1320 g
Initial Actinide Content:	Pu 2585 μg/g; Total Pu = 3.41 g
	Am 1.18 μg/g; Total Am 1.56 mg
Brine:	Castile (2:1 Brine/Solid ratio)
Additives:	Fe Mesh: Nd, Th, U, Np
Other:	None

Soluble Actinide Histories:

Pu - Pu concentrations began at 47,673 ppb and peaked at 243,438 ppb on 10/30/95 and began a steady decrease to 13,390 ppb on 10/18/99. LS27 had the highest soluble concentration of any test container. Pu (VI) was not identified in LS27.

Am - Am concentration began at 147 ppb and peaked at 825 ppb on 10/30/95 (same as Pu) and then slowly decreased to 64 ppb on 10/18/99. Am concentrations followed the general trend of Pu concentrations.

U - U concentrations started at 33 ppb which was the lowest concentration of any of the other actinides or Nd. U concentrations increased to a high of 197 ppb on 5/6/96 and then started to decrease slowly to a final concentration of 50 ppb on 10/18/99.

Th - Th concentrations began relatively high for Th at 2842 ppb and increased to about 6000 ppb where it seemed to remain for about one year and then slowly decreased to a final concentration of 148 ppb on 10/18/99. LS27 had the highest long term concentrations of Th than any other STTP test container. LS25 and LS26 each had Th concentrations of < 5 ppb.

Np - Np concentrations started at 425 ppb and increased to about 1100 ppb on the next two sampling periods (6/12/95, 7/17/95) and then decreased to a final concentration of 68 ppb. Np concentrations seemed to increase in the presence of CO_2 in the pressurized tests but l27 did not have added CO_2.

Nd - Nd concentrations started at 122 ppb (5/8/95) and peaked at 5350 ppb on 10/30/95 which was the peak for Pu, U, Am, and Th (generally). Nd decreased similarly to Th, Pu, and Am to a final concentration of 167 ppb (10/18/99). LS27 had the highest concentrations of Nd than any other test container.

Other Analyses (Nominal):

Typical pcH Range:	10.7 to 11.2
Fe Concentration:	Fe concentrations ranged from 43 to 243 ppm and were generally in the 100 ppm level until 1/4/99 and the Fe decreased to a final concentration of 16 ppm (12/6/99).
Other Analytes:	Ca 80,000 ppm K 5000 ppm Mg < 10 ppm Na 44,000 ppm
Other:	Al, Ni, and Pb were generally less than concentrations
TIC/TOC:	10/50 ppm
Particle Concentration:	10^{11} to 10^{12} particles/L
Filter Paper-WDXRF:	There was relatively high Pu content on all filter papers analyzed (16 of 16). There was significantly high Sr and S content and no detectable Fe content on the filter papers. There were Pu colloids or microprecipitates but no Fe in this Castile brine experiment at pcH 10.7 to 11.2. This is surprising because there was soluble Fe in the brine most of the test.

H_2 Headspace Gas Content: 65 v/o H_2; O_2 = 4.8 v/o.

D&D Observations (1/30/01):

Corrosion:	There was severe corrosion on stainless steel fittings and feedthroughs.
Brine:	The main pool of brines was about 3 inches and was quite clear which is surprising because the sludge is green.
Bottom Solids:	There were no hardened or cemented solids. All the solids were loose and exposed to the brine. The solids in the screen were a pea soup consistency and green.
Fe Mesh:	The Fe mesh was totally recovered and did not appear to have dissolved. There was a blackish color around Fe mesh strands. The ends of the Fe mesh were shiny as if they had just been cut. The green color on the sludge above the screen shows that Fe dissolved from the top fittings and perhaps from the Fe mesh.

Overall Assessment:

LS27 was unique in that Pu, Am, Th, Np, and Nd were high on the initial sample.
All peak concentrations occurred about the same time and then decreased slowly until the end of the test. There was never any indication of Pu (VI) in LS27 which is not surprising because the green sludge shows that $FeCl_2 \cdot H_2O$ (reduced form of Fe) was present throughout the test. There was no solidified or cemented mass at the bottom of the test container and all the comminuted Pyrochemical salt waste was available to the brine, which may have been the reason for the very high actinide (except U) concentrations in this test container. The soluble Fe concentration was around 100 to 240 ppm early in the test and decreased to 20 to 100 ppm late in the test which indicated a depletion of soluble Fe with time.

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The green color on the sludge in the screen attests to the presence of Fe in the sludge but the clear color in the brine indicates that the Castile brine leached soluble Fe from the sludge at a slow rate at the very basic pcH (10.7 – 11.2). The effect of the high Fe on the nonsolidification of the Pyrochemical salt is not known but the low Mg content in this Castile brine may have been the reason for this. The high H_2 concentration in the headspace did not eliminate corrosion of the SS fittings. The Pu on the 5 micron filter papers and the centriprep filter papers (< 10 nm) showed that colloids or microprecipitates bearing Pu were present in the brine. The particle concentration was relatively high at 10^{11} to 10^{12} particles/liter. The absence of Fe at the 5 micron, 1 micron, and < 10 nm diameter filter papers was surprising because Fe was present as a soluble cation most of the test and perhaps the Fe was always in the Fe^{2+} state rather than the more insoluble Fe^{3+} state. The high H_2 content (65%) attests to the high radiolysis rate of the brine but the reduced form of Fe in the sludge seemed to predominate in maintaining a relative low Eh. The peroxide or hypochlorite formed as a result of radiolysis could impact the Fe before the Pu. The Pu in the filter paper (5μ and < 10nm) was associated with Sr and sulfate in every case but not with Fe which was present as Fe^{2+}. This may be an important parameter to observe in other test containers.

The low magnesium content that could have led to the total availability of all the comminuted waste to the brine without cementation and the high radioactivity in the waste was probably the reason L27 had such high concentrations of all actinides. Radiolysis certainly had to have an impact on the chemistry but the oxidation of available soluble Fe^{2+} ion would use up the oxidation capacity of the radiolytically produced oxidants. The absence of Fe in the filter papers is evidence that Fe was being oxidized to Fe^{3+}, which is highly insoluble ferric hydroxide ($K_{sp} \sim 10^{-38}$) that could age and agglomerate into larger precipitates that would settle out and not be suspended as colloidal matter.

Liter-Scale No. 28 (Pressurized)

Test Characteristics:

Waste:	Pyrochemical Salts (DOR)
Total Waste Weight:	920 g
Initial Actinide Content:	Pu 11,530 μg/g; Total Pu = 10.607 g
	Am 1.35 μg/g; Total Am 1.242 mg
Brine:	Brine A (2:1 Brine/Solid ratio)
Additives:	Fe Mesh: Nd, Th, U, Np, 60 Bars (870 psig) of CO_2 pressure
Later Additives:	76.4 g of MgO added as slurry on 2/97.

Soluble Actinide Histories: (8/28/95)

Pu - Started at 5989 ppb and steadily increased to 90,942 ppb on 1/13/97. MgO was added on 2/97 which resulted in an increase in pcH to 7.70 from 4.48, and significant decrease of all actinides; Pu decreased to 18,097 ppb. After the addition of MgO and the initial reduction of Pu concentration, the Pu increased to a peak of 197,984 ppb on 9/21/98.

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Pu (VI) was identified in L28 on a sample taken 5/17/99. The total alpha activity increased from 1337 nCi/ml on 12/6/99 to 1683 nCi/ml on 5/15/01.

Am - Am started at 34.8 ppb and increased to 352 ppb prior to addition of MgO. After addition of MgO, the pcH increased from 4.48 to 7.70 and Am decreased to 40.1 ppb. Am increased to 392 ppb near the end of the test which was a level similar to that before the addition of MgO. Am followed the trend of Pu except at a much lower level. The total Am-241 activity increased from 19.3 nCi/ml on 12/6/99 to 152.5 nCi/ml on 5/15/01.

U - U started at 5,230 ppb and increased to 10,833 ppb on 1/13/97. After addition of MgO on 2/97 and the pcH increasing from 4.48 to 7.7, U decreased to 106 ppb and then increased to 4,520 ppb and the last analyses showed U at 668 ppb on 5/17/99.

Th - Th started 178 ppb and increased to 1359 ppb on 1/13/97. After the addition of MgO on 2/97 and an increase in pcH from 4.48 to 7.70, Th decreased to 36 ppb and then slowly increased to 531 ppb at the end of the test.

Np - Started at 3549 ppb and increased to 13,931 ppb on 1/13/97 and decreased to 166 ppb after addition of MgO on 2/97. Np increased to 2049 on 8/11/97 and had decreased to 744 ppb on 5/17/99.

Nd - Nd started at 15.4 ppb and increased to 31.5 ppb prior to addition of MgO. After addition of MgO and an increase of pcH from 4.48 to 7.70, Nd dropped to 5.3 ppb and then slowly increased to 57 ppb at end of the test.

Other Analyses (Nominal:)

Typical pcH Range:	Prior to MgO: 4.48 – 5.35
After MgO:	5.03 – 7.70
Fe Concentration:	Fe started at 19 ppm and increased to 165 ppm prior to MgO addition. After MgO addition, the pcH increased from 4.48 to 7.70 and the Fe decreased to 1.1 ppm and then increased to 50.8 ppb on 12/6/99.
NOTE:	It is surprising that Pu (VI) was detected in LS-28 with all the Fe unless the Fe was in the form of Fe^{+3} or if soluble Fe was not available to the top portion of the brine.
Other Analytes:	Ca 115,000 ppm K 12,000 ppm Mg 15,000 ppm Na 4,000 ppm Ni 300 ppm
TIC/TOC:	200/50 ppm
Particle Concentration:	1 x 10^{13} particles/L

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Filter Paper-WDXRF: Pu at rather high levels (29.6, 19.5, 57.1 $\mu g/cm^2$) was identified in 3 of 3 filters. Fe was identified in one filter. Sr was identified in all three filters.

H_2 Headspace Gas Content: Not analyzed.

D&D Observations (4/26/01):

Corrosion: There was no corrosion observed on feedthroughs.

Brine: The brine level was about ½ inch below the screen. The depth of the brine is about 2-1/2 inches. The brine is a cloudy and yellowish color liquid with suspensions.

Intermittent Level Solids: A very hard plug was found under the brine that essentially divided the test in two parts; one above the hard plug and one below the plug. The plug was very hard and was finally removed with a hammer and screwdriver point. The plug limited the communication between the top and bottom of the test container and could have been the reason that the chemistry of the upper brine was not long-termed. The plug probably formed when the MgO was added to the test container and consisted of sorel cement and other solidified masses that were very hard.

Bottom Solids: The bottom solids were very hard yellowish colored cement. A hammer and chisel were used to poke through the solids.

Fe Mesh: The Fe mesh was embedded in the bottom liquid, which had a oatmeal like consistency.

The Fe mesh holder was covered with a coarse layer of crucible shards that were discolored. The Fe mesh appeared to be nearly new and was mostly black covered with a thin dark coating.

Overall Assessment:

NOTE: An extended summary of the principal parameters of LS 28 is given in LA-UR-00-1606, "A Study of STTP Pyrochemical Salt Tests and Results Featuring Pu(VI)".

Liter-scale No. 28 was one of the most visible tests in the STTP because it contained such a high level of Pu, CO_2 pressure (870 psig), and added MgO and the fact that Pu (VI) was identified after the addition of MgO. The D&D showed that the MgO added to the test container immediately formed a hard solid, perhaps sorel cement, that isolated the top portion of brine from the bottom portion. This would have effectively allowed the top portion of soluble Pu and Am to form higher levels of radiolytic oxidants and oxidize soluble Pu to Pu(VI) at pcH 7.70.

The presence of high levels of soluble Pu in a test container pressurized with CO_2 gas at pcH 7.70 that dropped to pcH 5.03 (because of such a low volume of brine) after addition of MgO indicates that CO_2 was effective in solubilizing Pu and other actinides at pcH 4.48 and the addition of MgO only served to enhance the solubility and oxidative strength of the solution above the solid sorel cement mass. This finding strengthens the position that Pu(VI) will be found only in limited pockets in the WIPP.

Liter-Scale No. 29 (Pressurized)

Test Characteristics:

Waste:	Pyrochemical salts (DOR)
Total Waste Weight:	920 g
Initial Actinide Content:	Pu 4.715 μg/g; Total Pu = 4.338 g
	Am 2.59 μg/g; Total Am = 2.38 mg
Brine:	Brine A (2:1 brine/solid ratio)
Additives:	Fe Mesh; Th, U, Np, and Nd
	60 Bar (870 psig) of CO_2 pressure

Soluble Actinide Histories: (8/28/95 – 6/20/01)

Pu -	Started at 161 ppb and increased to maximum of 8446 ppb on 8/11/97 and ended at 6865 ppm. The total alpha-activity of L29 increased from 360 nCi/ml on 9/20/98 to 1822 nCi/ml on 6/20/01.
Am -	Started at 1.1 ppb and increased to ~ 37 ppb on 9/21/98. Am activity decreased from 9/21/98 (100 nCi/g) to ~ 50 nCi/g on 6/20/01.
U -	Started at 1.1 ppb and increased to 4840 on 1/22/96 and then to 9513 on 9/21/98.
Th -	Started at 2.8 ppb and increased to 377 on 9/21/98.
Np -	Increased from 17.5 ppb(8/28/95) to 8179 ppb on 9/21/98.
Nd -	Was less than 7 ppb for entire test.

Other Analyses (nominal):

Typical pcH Range:	4.73 – 5.68 (Acid side of neutral)
Fe Concentrations:	Varied from 34 to 1468 ppm during test. This Fe concentration is very high.
Other Analytes:	Ca 150,000 ppm
	K 4,000 to 12,000 ppm
	Mg 20,000 ppm
	Na 2,000 ppm
TIC/TOC:	500/50 ppm
Particle Concentration:	3×10^{12} particles/Liter
Filter Paper-WDXRF:	Pu in 3 of 4 filters; Fe on 2 of 4 filters. No Sr detected. Np and Th on one filter that had highest Pu (47.1).

H_2 Headspace Gas Content: No analyses.

D&D Observations (5/23/01):

Corrosion:	No visible corrosion.
Brine:	Fairly clear liquid with no suspensions.
Bottom solids:	Dark muddy brown solids that had texture of sand. Part of material was hard and a portion was soft.
Fe mesh:	Fe mesh was embedded in dark, muddy looking sludge. Fe mesh was coated with black thin coating. After washing, the coating was black to gray.

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Overall Assessment:
Liter-scale test container No. 29 was a Pyrochemical salt waste in Brine A and had ~870 psig CO_2 pressure placed on the headspace. The pcH started at 5.68 and decreased with time (8/28/95 – 9/21/98) to 4.73. Pu increased with time to a peak of 8446 ppb. There were 3 filters that had Pu but it did not appear to correlate with $SrSO_4$. The total alpha activity increased from 360 nCi/ml on 9/20/98 to 1822 on 6/20/01 during which no rotation was conducted on the drum. The Fe mesh did not appear to be corroded and had a black gray color. The particle concentration was relatively high at this pcH at ~ 3×10^{12} particles/liter. This test showed that even with 870 psig of CO_2 in a acid condition that actinides were not significantly solubilized.

Liter-Scale No. 30 (Pressurized)

Test Characteristics:

Waste:	Pyrochemical salts (DOR)
Total Waste Weight:	902 g
Initial Actinide Content:	Pu 2.185 μg/g; Total Pu = 2.010 g
	Am 2.59 μg/g; Total Am = 2.38 mg
Brine:	Castile brine (2:1 brine/solid ratio)
Additives:	Fe Mesh; Th, U, Np, and Nd
	60 Bar (870 psig) of CO_2 pressure

Soluble Actinide Histories: (8/28/95 – 6/20/01)

Pu -	Pu started at 2166 ppb and decreased to 1011 ppb. The total alpha-activity remained about the same from 9/20/98 (68 nCi/ml) to 6/20/01 (70 nCi/ml).
Am -	Am decreased from 20 ppb to 5.8 ppb during the test period.
U -	U decreased from 5663 ppb (8/28/95) to 2787 ppb (9/21/98).
Th -	Th started at 35 ppb and decreased to 32.9 ppb at end of test.
Np -	Np started at34.8 ppb and increased to 135 ppb during the test period.
Nd -	Nd started at 69 ppb and decreased to 6 ppb on 9/21/98.

Other Analyses (nominal):

Typical pcH Range:	5.9 – 6.6
Fe Concentrations:	Achieved a high of 1967 ppm on 1/22/96 which decreased to 9.2 ppm on 9/21/98.
Other Analytes:	Ca 30,000 ppm
	K 4,000 ppm
	Mg 20,000 ppm
	Na 22,000 ppm
TIC/TOC:	Coarse/Fine 700/80 ppm
Particle Concentration:	2×10^{11} particles/Liter
Filter Paper-WDXRF:	Pu in 3 of 3 filters; Fe in one filter. No Sr or Al detected. No other actinides.
H_2 Headspace Gas Content:	No analyses.

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D&D Observations:

Corrosion: No visible corrosion noted on lid or screen.

Brine: Light gray to brown color with a consistency of oatmeal. Depth of brine pool was about 3 inches.

Bottom solids: Very hard solid material with brown color.

Fe mesh: Fe mesh in holder was embedded in a hard brown solid. The container had to be broken to retrieve the Fe mesh.

The Fe mesh had a dark black color and did not appear to be corroded, however, the soluble Fe concentration was very high (1967 ppm) early in the test that rapidly decreased to 9.2 ppm near the end of the test. The color of the unwashed deposits on the mesh were red, yellow, and green.

Overall Assessment:

Liter-scale test container No. 30 was a Pyrochemical salt waste in Castile Brine with a pcH range of 9.5 – 6.6 under a CO_2 pressure of 60 Bars (870 psig).

Pu started at 2166 ppb and decreased to 1011 ppb. All other actinides decreased in concentration during the test except Np, which increased from 34.8 to 135 ppb. The total particle concentration was relatively low (~2 x 10^{11} particles/liter) which was surprising because the brine had an oatmeal like texture. Pu colloids or microprecipitates were found in all three of the filter papers analyzed but at relatively low levels. The Pu and other additives did not achieve high concentrations relative to other Pyrochemical salt tests.

Liter-Scale No. 31

Test Characteristics:

Waste: Pyrochemical salt waste (O_2 Sparging)

Total Waste Weight: 1,320 g

Initial Actinide Content: Pu 612.5 μg/g; Total Pu = .809 g
Am 0.49 μg/g; Total Am = 0.647 mg

Brine: Brine A (2:1 brine/solid ratio)

Additives: Fe Mesh; Th, U, Np, and Nd
Bentonite – 120 g that was brine equilibrated

Soluble Actinide Histories:

Pu - Began low <20 ppb for 2.5 years, then increased to 280 ppb for last sample (3/99).

U - Began at 3,700 ppb and continued to decrease throughout the experiment, ending at a low of 500 ppb.

Np - Began at 450 ppb then decreased to 120-200 ppb for the last 3 years of testing (9/96 – 3/99).

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Nd - Started at 71 ppb and slowly decreased to 6.4 ppb at end of test.
Other -Nd, Th, and Am were generally < 10 ppb during the test.

Other Analyses (nominal):

Typical pcH Range:	8.7 – 9.0
Fe Concentrations:	Very low at < 1 ppm
Other Analytes:	Ca 1,300 ppm
	K 50,000 ppm
	Mg 30,000 ppm
	Na 60,000 ppm
TIC/TOC:	35/25 ppm
Particle Concentration:	10^9 to 10^{10} particles/Liter
Filter Paper-WDXRF:	There were 8 of 12 filters with Pu; 1 of 12 had Np; 4 of 12 had Th. This was one of the only tests that showed Th and Np on the filters.
Headspace Gas Content:	H_2 = ~ 33 v/o; O_2 ~ 0.80 v/o; N_2O ~ 0.5 v/o

D&D Observations (November 4, 1999): No data found.

Overall Assessment:
This Pyrochemical salt waste test with bentonite had a relatively low concentration of Pu and other actinides. The brine equilibrated bentonite may have had a role in maintaining low concentrations for Pu and all other actinides. Even though the Pu concentration was generally low but began a trend up to 275 ppb, the overall concentration was relatively low for Pyrochemical salt waste. There were 8 of 12 filters that had Pu colloids and 4 of 12 filters that had Th. The presence of Th in the test container was rather unique for the STTP. Fe was found on 3 filters but at very low levels. No Sr was found on the filter.

Liter-Scale No. 32

Test Characteristics:

Waste:	Pyrochemical salt waste (O_2 Sparging)
Total Waste Weight:	1,320 g
Initial Actinide Content:	Pu 3,105 μg/g; Total Pu = 4.10 g
	Am 2.21 μg/g; Total Am = 2.92 mg
Brine:	Brine A (2:1 brine/solid ratio)
Additives:	Fe Mesh; Th, U, Np, and Nd
	Bentonite – 120 g that was brine equilibrated

Soluble Actinide Histories: (5/15/95 – 3/8/99)

Pu - Started low <4 ppb, increased to 697 ppb after 16 months, then slowly decreased to a low of 158 ppb, reached at the end of sampling period.
Am - Started low (<0.1 ppb) and leveled off at 1-2 ppb after 16 months.
U - Began at a high of 3,173 ppb, and decreased to a low of 381 ppb at end of testing period.

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Np - Started at 161 ppb and slowly decreased to 65 ppb at end of testing period.
Nd - Started at 71 ppb and slowly decreased to 6.4 ppb at end of test.
Th - Started at 7.4 ppb and decreased to 1.3 ppb at end of testing period.

Other Analyses (nominal):

Typical pcH Range:	8.6 – 9.0
Fe Concentrations:	Very low at < 1 ppm for most of test with 1 result of 1 ppm and another at 3 ppm.
Other Analytes:	Ca 750 ppm K 50,000 ppm Mg 30,000 ppm Na 60,000 ppm Pb Up to 6.6 ppm and decreasing to 1.1 ppm
TIC/TOC:	30/30 ppm
Particle Concentration:	10^9 to 10^{10} particles/Liter
Filter Paper-WDXRF:	Pu at high level identified in 10 of 15 filter papers; Fe was identified in 5 of 15 filters; Sr was identified in the filters with highest Pu. S was in all filters.
Headspace Gas Content:	H_2 = ~ 44.6 v/o; O_2 = 2.0 v/o

D&D Observations (December 15, 1999):

Corrosion:	Lid had 3 areas of corrosion at SS feedthroughs. Screen was corroded with salt adhering to bottom.
Brine:	Brine was fairly clear with some suspensions and had a pea soup consistency and color near bottom.
Bottom solids:	Very hard cemented solid at bottom of test container.
Fe mesh:	Was embedded under hardened solid and could not be located.

Overall Assessment:

LS-32 was a test with Brine A at a pcH of 8.6 – 9.0 (same as L 31) with added bentonite, a montmorillonite clay with colloidal silica. LS-32 had a relatively heavy loading of Pu (4.10 g) but none of the actinides achieved a high level of solubility (Pu max 697 ppb, Am max 2 ppb, U max 3173 ppb, Np 161 ppb, and <75 ppb for Th and Nd. The concentration of Fe was very low (<1 ppm) but the color of the brine suspensions may be from Fe. There was a significant cemented portion at the bottom of the test container that totally covered the Fe mesh. The particle concentration was low (10^9 to 10^{10} particles/liter) and the colloid-sized particles contained Pu and some Fe. There was evidence of corrosion in the SS feedthroughs on the lid of the container. The bentonite was effective in maintaining a relatively low actinide concentration. The headspace gas concentration of H_2 was ~45 v/o which is in agreement with the relatively high concentration of Pu.

Liter-Scale No. 33

Test Characteristics:

Waste:	Pyrochemical salts (O_2 Sparging)
Total Waste Weight:	1,320 g
Initial Actinide Content:	Pu 860 μg/g; Total Pu = 1.14 g
	Am 0.83 μg/g; Total Am = 1.10 mg
Brine:	Castile (2:1 brine/solid ratio)
Additives:	Fe Mesh; Th, U, Np, and Nd
	Bentonite – 120 g that was brine equilibrated

Soluble Actinide Histories:

Pu - Began low <20 ppb for one year, then increased to 130-190 ppb for remainder of test

Np - Began at 20-40 ppb for ~2 years, then decreased to 5 ppb for remainder of test

Other - Nd, Th, U, and Am were generally <5 ppb during the test

Other Analyses (nominal):

Typical pcH Range:	9.5-9.8
Fe Concentrations:	< 0.1 ppm
Other Analytes:	Ca 1,000 ppm
	K 35,000 ppm
	Mg 500 ppm
	Na 100 ppm
TIC/TOC:	10/30 ppm
Particle Concentration:	10^9 to 10^{10} particles/Liter
Filter Paper-WDXRF:	Pu was detected as microprecipitates or colloids in 14 to 16 of the filter papers. Pu was associated with Sr on only three filter papers. Fe was associated with Pu on 6 filters. The Pu may be associated with bentonite on the other filter papers.

H_2 Headspace Gas Content: H_2 = 35 v/o; O_2 = 2.2 v/o

D&D Observations (1/30/01):

Corrosion:	SS fittings and feedthroughs were corroded rather severely; upper ring on screen corroded and was weakened.
Brine:	Milky white above screen with no evidence of floating suspensions; there was ~1/2 inch of gray sludge in screen; milky white brine below screen.
Bottom solids:	No cemented or hardened solids at bottom of test container; mostly there was about 2 inches of uncemented suspension of Pyrochemical salts and bentonite and perhaps crucible shards.

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Fe mesh: None of the Fe mesh appeared to dissolve; the wire strands have a blackish color that is fairly thin. The cut ends of the Fe mesh were still shiny. There was apparently little contact of the Fe with the brine because the bentonite and salts packed the inside of the Fe mesh holder.

Overall Assessment:

There appeared to be no corrosion of the Fe wire in the Castile Brine at pcH 9.5-9.8. There was a black film or thin coating around the wire strands. The SS fittings and feedthroughs in the Ti metal lid were quite corroded in the headspace gas region. The Fe was precipitated and did not remain soluble as evidenced by <0.1 ppm concentration in the brine throughout the test. Overall, the bentonite appeared to be effective in maintaining a very low concentration of all soluble actinides throughout the life of the test.

Nd, Th, U, and Am were generally less than 5 ppb, and Np increased from 20 to 40 ppb initially, and then decreased to <5 ppb at the end of the test. Most of the waste including actinides was available to the brine, but the actinide concentrations remained very low and radiolysis was not effective in oxidizing Pu. The particle concentration was relatively low indicating that there was an agglomeration of colloids and fine particles. The comminution of the Pyrochemical salts and crucibles could have had a significant impact on the chemistry of this experiment. Bentonite, a Montmorillonite clay with colloidal silica, could have had a role in preventing the cementation of the Pyrochemical salts in Castile Brine, but the major role may have been the low Mg (500 ppm) because there was significant cementation in L32 with Brine A and a Mg content of ~ 30,000 ppm. The percentage of Pu solubilized in L33 was about $5x10^{-2}$%. The Pu filtered as microprecipitates or colloids were associated with Fe and Sr only at the larger concentrations. The Pu may have been associated with the bentonite. The rather high corrosion of the SS fittings and feedthroughs is interesting at a headspace gas concentration of ~35 v/o.

Liter-Scale No. 34

Test Characteristics:

Waste:	Pyrochemical salts (Direct oxide reduction)
Total Waste Weight:	880 g
Initial Actinide Content:	Pu 2325 μg/g; Total Pu = 2.05 g
	Am 3.06 μg/g; Total Am = 2.69 mg
Brine:	Brine A (3:1 Brine/Solid ratio)
Additives:	Fe Mesh; Nd, Th, U, Np, chelators, and 96.2 gm of $Ca(OH)_2$
	Acetamide = 100 ppm
	Sodium Acetate = 139 ppm
	Ascorbic Acid = 101 ppm
	Trisodium Citrate Dihydrate = 154 ppm
	Oxalic Acid Dihydrate = 143 ppm;
	Ammonium Thiocyanate = 148 ppm

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Soluble Actinide Histories: (5/15/95 – 3/8/99)

Pu - Started at 5.9 ppb and increased to 33 ppb after one year and varied from 10 to 50 ppb for the remainder of the test.

U - Started at a peak of 776 ppb and has slowly decreased to a final concentration of 74 ppb.

Th - Varied between 4.2 and 38.9 ppb during the test period until a final concentration of 11.1 ppb.

Np - Started at 187 ppb and varied between 150 to 254 for the entire test period to a final concentration of 152 ppb. The trend seemed to decrease in concentration very slowly.

Nd - Generally < 2 ppb throughout the test.

Other Analyses (nominal):

Typical pcH Range: 8.6 – 9.0

Fe Concentrations: Generally < 2 ppm for the entire test.

Other Analytes: Ca 9,000 ppm
K 45,000 ppm
Mg 24,000
Na 65,000 ppm
Pb 0.1 to 6 ppm

TIC/TOC: 10/80 ppm

Particle Concentration: 10^9 to 10^{10} particles/Liter

Filter Paper-WDXRF: Essentially all filters contained Pu. No filters had Fe. No filter had Sr.

H_2 Headspace Gas Content: H_2 = 28.6 v/o; O_2 = 0.66 v/o; N_2 = 2.2 v/o; N_2O = 1.1 v/o

D&D Observations (03/26/01):

Corrosion: Sample port – Blue and brown colored corrosion spots at SS feedthroughs.

Screen: Corrosion observed on o-ring around the top of the screen. Full of white solids. Salts in screen about 1/8-inch thick crystals. Very difficult to remove.

Brine: Colorless with suspensions; 1 liter removed.

Bottom solids: About 8 inches of solids of oatmeal consistency on top and very hard on the bottom. The solids totally encompassed the Fe mesh.

Fe mesh: Could not remove the Fe mesh from the bottom solids, which were very hard.

Overall Assessment:

LS-34 was part of the test set of LS-34 (OS), LS-35 (DOR) and LS-36 (DOR). Chelators and $Ca(OH)_2$ were added to each test. LS-34 was a Brine A experiment with a Pu loading of 2.05 g in a 3:1 brine to solid ratio test. Chelators and 96.2 gm of $Ca(OH)_2$ were added to this test but not much solubilization of actinides took place as Pu was generally <30 ppb at the pcH range of 8.6-9.0. There was <2 ppm of Fe solubilization at any time during this test. LS-34 had historically been difficult to sample because the screen was loaded with crystals and particulate. Essentially all (11 of 13) filters contained Pu but no Fe or Sr was identified on any filter paper.

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There was corrosion on the SS penetrations on the Ti lid and the SS o-ring. The screen showed evidence of corrosion. The corrosion found on the lid and screen o-ring did not result in much soluble Fe or precipitated Fe. Overall, the actinides added to this Brine A test in the presence of chelators did not result in much solubilization. This is in sharp contrast to the drum-scale tests and LS-36 with added chelators, which showed much solubilization. Of course, LS-34 is an Oxygen Sparging waste (rich in Na and K chloride) rather than a DOR (rich in $CaCl_2$) experiment.

Liter-Scale No. 35

Test Characteristics:

Waste:	Pyrochemical salts (Direct oxide reduction)
Total Waste Weight:	880 g
Initial Actinide Content:	Pu 510 μg/g; Total Pu = 0.449 g
	Am 0.79 μg/g; Total Am = 0.07 mg
Brine:	Brine A (3:1 Brine/Solid ratio)
Additives:	Fe Mesh; Nd, Th, U, Np, chelators, $Ca(OH)_2$
	Acetamide = 100 ppm
	Sodium Acetate = 139 ppm
	Asorbic Acid = 101 ppm
	Trisodium Citrate Dihydrate = 154 ppm
	Oxalic Acid Dihydrate = 143 ppm;
	Ammonium Thiocyanate = 148 ppm
	Calcium Hydroxide = 96.2 g

Soluble Actinide Histories: (5/15/95 – 3/8/99)

Pu - Began at 930 ppb and increased to a high of 1458 ppb after six months and very slowly tailed off to a final 86 ppb at the end of the test. Chelators in Brine A and Pyrochemical salts had a small influence in solubilizing Pu.

U - Followed the same trend as Pu. Started at 233 ppb and increased to 423 ppb and then slowly decreased to 53 ppb at the end of the test period.

Th - Started at 271 ppb and increased to 575 ppb after six months and then slowly decreased to 62 ppb at the end of the test period. Th was minimally solubilized by chelators in Brine A at pcH 8.2 – 8.3.

Np - Started at 436 ppb and peaked at 2308 ppb after about six months and very slowly decreased to 1260 ppb at the end of the test period.

Am - Followed the same trend as Pu but at a much lower level. Started at 9.7 ppb and peaked at 18.6 ppb and slowly decreased to < 1 ppb at the end of the test period.

Nd - Generally < 5 ppb throughout the test. No trend was observed.

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Other Analyses (nominal):

Typical pcH Range:	8.2 – 8.3
Fe Concentrations:	Generally < 1 ppm with nine results between 1 and 3 ppm.
Other Analytes:	Ca 86,000 ppm K 28,000 ppm Mg 23,000 Na 20,000 ppm
TIC/TOC:	15/60 ppm
Particle Concentration:	10^{10} to 10^{11} particles/Liter
Filter Paper-WDXRF:	Only 2 filters (5 micron sized) had just detectable Pu. There was no Fe on any of the filters. There was essentially no Sr (3 each) on the filters. Apparently, the chelators complexed the Pu and Fe in this test whereas LS-34 and LS-36 had significant Pu on most filter papers. Essentially none of the filters from LS-34, 35, and 36 had Fe.

H_2 Headspace Gas Content: H_2 = 20.9 v/o; O_2 = 0.07 v/o; N_2 = 1.7 v/o

D&D Observations (03/13/01):

Corrosion:	Sample port - Blue-black coloration/corrosion Level Probe - Blue-black coloration/corrosion with large white crystals Gauge port - No coloration/corrosion Screen - Blue around outer o-ring
Brine:	Color is murky beige with the consistency of oatmeal. We were only able to retrieve about 200 ml of thick solution.
Bottom solids:	There was a lot of solids and sludge to within 3 inches of the top of the screen. The solids were voluminous and totally covered the Fe mesh but were not cemented.
Fe mesh:	The inside of the plastic holder was full of solids and salts and comminuted Pyrochemical salts. The material around the Fe mesh was a greenish-blue paste. There were blue colored salts in the holder. The Fe mesh did not seem corroded and was coated with a blue-black colored coating.

Overall Assessment:

LS-35 was a Castile brine test as part of a set of LS-34 (OS), LS-35 (DOR) and LS-36 (DOR) with added chelators and $Ca(OH)_2$. The pcH range of 8.2 to 8.3 was the least basic of this set (LS-34 8.7-8.9 and LS-36 11.0-11.4). Although, LS-34 had a cemented portion that covered the Fe mesh and LS-36 had ~ ½-inch of a cemented solid block, LS-35 had voluminous solids that was not cemented. LS-35 was unique in the greenish-blue coloration/corrosion on the three SS feedthroughs and the metal o-ring around the screen. There was little solution (~200 ml) retrievable from the main brine pool. There was a greenish-black color around the Fe mesh. The Fe mesh did not appear to be corroded but the vivid green-black color around the mesh attested to the solubilization of the Fe strands.

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There was minimal Pu solubility in LS-35 and the added chelators did not solubilize much Fe or other actinides. This is in stark contrast to the drum-scale tests, which showed much solubilization of all actinides. There was minimal solubilization of Fe (1-3 ppm) and only 2 filters showed a low level of Pu. The H_2 in the headspace was ~ 21 % and CO_2 was not detectable from the added chelators that resulted in a rather low TOC of 60 ppm.

Liter-Scale No. 36

Test Characteristics:

Waste:	Pyrochemical salts (Direct oxide reduction)
Total Waste Weight:	880 g
Initial Actinide Content:	Pu 12,575 μg/g; Total Pu = 11.07 g
	Am 5.61 μg/g; Total Am = 4.94.mg
Brine:	Castile (3:1 Brine/Solid ratio)
Additives:	Fe Mesh; Nd, Th, U, Np
	Chelators: Acetamide = 100 ppm; sodium acetate = 139 ppm; ascorbic acid = 101 ppm; trisodium citrate dihydrate = 154 ppm; oxalic acid dihydrate = 143 ppm;
	ammonium thiocyanate = 148 ppm
	Calcium hydroxide (96.2 gm)

Soluble Actinide Histories: (5/15/95 – 3/8/99)

- Pu - Began very high 20,000 ppb and continued decreasing until a low of 700 ppb was reached in January 1999. Final Pu concentration was 1439 ppb.
- U - Began at a high of 300 ppb, decreased to < 5 ppb by 3/8/99.
- Th - Began at a high of 6,000 ppb, decreasing to final concentration of 53 ppb on 3/8/99.
- Np - Began at 670 ppb and continued decreasing to a final concentration of 17 ppb on 3/8/99.
- Am - Started at a high of 75-90 ppb, and decreased to a final concentration of 6.7 ppb on 3/8/99.
- Nd - Started at a high around 15-20 ppb, decreasing to a low of 1-3 ppb on 3/8/99.

Other Analyses (nominal):

Typical pcH Range:	11.0-11.4
Fe Concentrations:	Ranged from 50 ppm to 100 ppm during most of the test and ended up at 17 ppm.
Other Analytes:	Ca 66,000 ppm
	K 5,500 ppm
	Mg less than undetectable
	Na 57,000 ppm

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TIC/TOC: 10/85 ppm
Particle Concentration: 10^{10} to 10^{11} particles/Liter
Filter Paper-WDXRF: All 16 filters had relatively high Pu; one filter which had the highest Pu (315) also had Np and Th; Fe was not found on most filter papers and only two had Fe. Fourteen filters had Sr and S and correlated to high Pu. Fe did not correlate to Pu at this pcH.
H_2 Headspace Gas Content: H_2 = 70 v/o; O_2 = 15 v/o (the highest in the STTP); N_2 = 0.2
The high O_2 was due to radiolysis rather than air in-leakage.

D&D Observations (11/29/00):

Corrosion: Sample port was nearly plugged with corrosion products. There was considerable corrosion on and around all SS feedthroughs.
Brine: Brine was a greenish-gray liquid that was thickened near the bottom of the test container with suspended material.
Bottom solids: There was 3 or 4 inches of loose solids that were gray in color and appeared as gravel. Below this muck was about ½" of cemented solids that nearly encompassed the wire mesh.
Fe mesh: The plastic holder that contained the mesh was embedded in about ½" of cemented solid. Only one half of the mesh could be removed from the plastic holder. The Fe mesh appeared to not be corroded and covered with white deposits that upon washing revealed a hard black coating.

Overall Assessment:

LS 36 was a Castile Brine test with a relatively basic pcH (11.0 – 11.4) that had added chelators. The effect of the chelators was much more pronounced in LS 36 for all actinides and especially Pu and Am at the basic pcH. This pcH was effective at precipitating Mg and perhaps some Ca. This test had the greatest mass of Pu in the STTP. The colloidal particle concentration for this test was rather high (10^{10} to a maximum of 10^{12} particles per liter) and was identified in both the 5 micron filters and the centriprep or < 10 nm filters. The concentration of Pu and Am was high for most of the test period but decreased with time. The chelators were effective in maintaining a high Fe concentration most of the test period. It was surprising that there was so few (two each) filters that had Fe. All filters contained Pu to high levels and the Pu correlated with Sr and S. The Pu could have been entrained with $SrSO_4$ at this pcH and not with Fe. The Fe concentration throughout the test should have maintained a reducing environment unless the Fe was oxidized and precipitated as $Fe(OH)_3$ which has a much lower K_{sp} than $Fe(OH)_2$. The SS feedthroughs were severely corroded in the headspace which had an O_2 content of ~ 15 v/o, highest in the STTP. However, the Fe mesh did not appear to be corroded with a black coating on the surface of the Fe wire strands.

There was a cemented solid block about ½ - inch thick at the bottom of the test container that embedded about half the plastic Fe mesh holder. This was rather surprising because the soluble Mg content was less than detectable for the entire test period.

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The cemented solid could have been immediately formed by the available but limited Mg content at the beginning of the test to form a sorel cement solid. Overall, this was an experiment with many variables that only begins to make sense in the context of all the other experiments. Pu and Am are certainly solubilized by the chelators but other factors tend to reduce the soluble concentration over a long time period. The pcH of 11.0 – 11.4 certainly had a strong influence on the chemistry of this test. The Ca should have been higher because of the addition of 96 g $Ca(OH)_2$ to this test.

Liter-Scale No. 37

Test Characteristics:

Waste:	Pyrochemical salts (Direct oxide reduction)
Total Waste Weight:	1320 g
Initial Actinide Content:	Pu 3,295μg/g; Total Pu = 4.35 g
	Am 0.83 μg/g; Total Am = 1.1 mg
Brine:	Brine A (2:1 Brine/Solid ratio)
Additives:	No Fe mesh added; no Nd added; 75 mg of Am-241 added as soluble salt, equivalent to ~ 37,000 ppb

Soluble Actinide Histories: (5/15/95 – 3/15/99)

Pu -	Began low, 45 ppb, decreasing to a low of 20 ppb by September 1996 through February 1997, then increased to a high of 380 ppb by January 1999.
Other -	Nd, Th, Np, U, and Am were <5 ppb during the experiment and showed no signs of a trend.

Other Analyses (nominal):

Typical pcH Range:	7.6 – 8.3
Fe Concentrations:	< 10 ppm for part of the test and < 2 ppm for most of the test period.
Other Analytes:	Ca 150,000 ppm
	K 30,000 ppm
	Mg 27,000 ppm
	Na 7,000 ppm
TIC/TOC:	15/25 ppm
Particle Concentration:	10^9 to 10^{10} particles/Liter
Filter Paper-WDXRF:	Low levels of Pu found in last 8 filters; low levels of Fe identified in 6 of 8 filters; Sr and S identified on all high Pu filters.
H_2 Headspace Gas Content:	H_2 = ~ 50 v/o; O_2 = ~ 4.7 v/o

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D&D Observations (12/06/00):

Corrosion: SS fittings and feedthroughs were corroded; level probe fell off but was shiny. Black scale could have prevented sampling through sample port. There was much salt on the lid. Greenish colored gel around o-ring.

Brine: Clear with tan-colored fines; many crystals throughout.

Bottom solids: Compacted silt/clay at bottom that yielded to a screwdriver. Compacted solids were at an angle from ~ 1 to 4 inches at a slant.

Fe mesh: No Fe mesh.

Overall Assessment:

LS-37 was a DOR Pyrochemical salt test in Brine A at pcH 7.6 – 8.3 and had 75 mg (37,000 ppb) of Am-241 added as a soluble salt. There was no Fe mesh added to this test but there was a persistent low level of Fe (< 2 ppm) throughout the test period. Pu was stable in the range of 20-40 ppb until 3/9/98 and increased to 200-400 ppb the last 5 or 6 samples. All other actinides including Am-241 were generally < 2 ppb. The Am-241 added as a soluble salt precipitated immediately and settled out at the bottom of the test container. There was a relatively low concentration of colloids or microprecipitates (10^9 to 10^{10}) and on the last 8 filters showed Pu. The Pu appeared to be associated with Sr rather than Fe. The greenish gel near the upper o-ring of the screen attests to the presence of $Fe(Cl)_2$ and may have been the source of Fe during the test. The stainless steel feedthroughs in the headspace showed signs of corrosion. The headspace gas was ~ 50 v/o with O_2 at about 4.7 v/o.

Liter-Scale No. 38

Test Characteristics:

Waste: Pyrochemical salts (O_2 Sparging)

Total Waste Weight: 1320 g

Initial Actinide Content: Pu 2045 μg/g; Total Pu = 2.736 g
Am 5.55 μg/g; Total Am 7.43 mg

Brine: Brine A (2:1 Brine/Solid ratio)

Additives: 75 mg of Am-241 added as a soluble salt is equivalent to 37,000 ppb assuming 2000 ml; no Fe mesh added; no Nd added.

Soluble Actinide Histories: (5/95 – 3/99)

Pu - Was low, vacillating during the time of testing from 3-20 ppb with an average of about 10 ppb.

Am - Was < 1 ppb for the entire test which is surprising because 75 mg of soluble ^{241}Am was added to this test container.

Other – Th, Np, U, and Am remained ≤ 4 ppb during the test; Nd was not added.

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Other Analyses (Nominal):

Typical pcH Range:	7.5 – 8.1
Fe Concentration:	< 1.0 ppm
Other Analytes:	Ca 76,000 ppm
	K 40,000 ppm
	Mg 30,000 ppm
	Na 24,000 ppm
TIC/TOC:	15/35 ppm
Particle Concentration:	10^9 to 10^{10} particles/L
Filter Paper-WDXRF:	There was no Pu, Fe, or Sr found on filter papers.

H_2 Headspace Gas Content: 51.3 v/o H_2, O_2 = 8.7 v/o

D&D Observations (2/6/01):

Corrosion:	SS fittings and feedthroughs had light colored crystals on surfaces. Crystals also on level probe.
Brine:	Brine is color of tea or light coffee.
Bottom Solids:	Approximately ¼ inch of yellowish-brown fines on top of very hard or cemented mass.
Fe Mesh:	No Fe mesh was added to this test container.

Overall Assessment:

LS38 was a Pyrochemical salt test in Brine A. There was added ^{241}Am (75 mg) which precipitated immediately in the brine pcH 7.5 – 8.1. There was no soluble Fe over 1 ppm in the entire period of the test (5/95 – 3/99). Neodymium and Fe mesh were not added to the test. Thorium, uranium, and Np were all essentially < 1 ppb for the entire test. There was no Pu detected on the filter papers (5 μ, 1 μ, and < 10 nm), nor Fe or Sr. The precipitated Pu and Am were present as precipitates on top of a cemented solid at the bottom or was incorporated into the solid mass. It may be that the Pu and Am once precipitated was incorporated into the cemented mass which would have depleted the available inventory of 2.74 g of Pu and 7.43 mg of ^{241}Am. The 75 mg of ^{241}Am added to the 7.43 mg already present in the waste gave a total available mass of 82.43 mg of ^{241}Am . The 75 mg of Am was added as soluble chloride salt. The theoretical concentration of ^{241}Am assuming 2 liters of brine and 100% solubility would have been 41,215 ppb. The average Am concentration was less than 1 ppb or 2.4 x 10^{-3} %. The percentage of Pu that dissolved relative to what was available in the 1320 g of waste was 7.0 x 10^{-4} %. The precipitated Pu and Am resulted in a radiolytically generated hydrogen content of ~ 51.3 v/o and O_2 at 8.7 v/o.

LS-38 in Brine A showed no Pu in the filters while LS-39 in Castile Brine showed Pu in essentially all filters. There appeared to be greater colloids containing Pu in LS-39 than LS-38 but the particles per liter were about the same.

Liter-Scale No. 39

Test Characteristics:

Waste:	Pyrochemical Salts (O_2 Sparging)
Total Waste Weight:	1,320 g
Initial Actinide Content:	Pu 3350 μg/g; Total Pu = 4.42 g
	Am 8.7 μg/g; Total Am = 11.5 mg
Brine:	Castile (2:1 Brine/Solid ratio)
Additives:	Th, U, Np (No Nd and no Fe mesh) Am-241 added as 75 mg soluble salt; equivalent to 37,000 ppb at 2000 ml.

Soluble Actinide Histories: (5/95- 3/99)

Pu -	Began low, 14 ppb, and then increased to 2,600 ppb after 2.5 years, and ended at ~2,000 ppb
Am -	Started at <0.4 ppb and slowly increased to 18 ppb during the test; this is a significant increase for Am-241.
U -	Started at 470 and decreased to 30 ppb
Np -	Started at 50 ppb and decreased to 6 ppb
Th -	Was <1 ppb during the test
Nd -	Not added

Other Analyses (nominal):

Typical pcH Range:	9.4-9.9
Fe Concentrations:	<1 ppm for entire test, no Fe mesh added
Other Analytes:	Ca 6,700 ppm
	K 7,000 ppm
	Mg 2,000 ppm
	Na 70,000 ppm
TIC/TOC:	10/30 ppm
Particle Concentration:	10^9 to 10^{10} particles/Liter
Filter Paper-WDXRF:	Pu filtered in filter papers was rather high in mass and in 13 out of 14 filter papers. No Fe was associated with the Pu. Sr was associated with the high level precipitates.

H_2 Headspace Gas Content: ~46 v/o; O_2 at 11.9 v/o (this is high for O_2).

D&D Observations (11-15-00):

Corrosion:	SS fittings were corroded, but level probe was clean; rust colored deposits (~1 mm thick) surrounded the fittings. Screen impacted with brown scale.
Brine:	A pale brownish-gray colored liquid that was not viscous. The liquid was opaque.
Bottom Solids:	There was a cemented solid at the bottom of the test container that was at a slant (~2" to 1" from the bottom). There was a pasty sludge ~1-1/2 inch depth above a hard or cemented solid.
Fe mesh:	None added.

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Overall Assessment:

No Fe mesh added to this test container, and the soluble Fe concentration was less than 1 ppm for the entire test. The addition of 75 mg of ^{241}Am in soluble form did not seem to increase the concentration of Am until after 2-3 years into the test and Am increased from less than 1 ppb to ~18 ppb. The inventory of Am was about 86.6 mg or a concentration of ~ 43,300 ppb assuming 100% dissolution. However, the overall solubility of actinides in L-39 was very low compared to the inventory available to the brine. The percentage of Pu that solubilized relative to what was available in the 1,320 g of waste (4.42 g) was about $8x10^{-2}$ %. The presence of Pu in all > 5 micron filters after 11/13/95 verifies that Pu colloids or microprecipitates were present most of the test period. The pcH range 9.4 – 9.9 apparently precipitated the Pu in a form not associated with Fe since Fe was not in the filters. Sr was associated with the filters with the highest Pu content.

The Am was added as a soluble salt and immediately precipitated and was less than 1 ppb for about 6 months. The precipitated Pu and Am resulted in a radiolytically generated hydrogen concentration of ~ 46 v/o and an O_2 of 11.9 v/o, the second highest in the STTP. LS 36 was the highest.

There was a cemented solid at the bottom of the test container that was slanted from a high of ~ 2 inches to about 1 inch. There was a pasty sludge of ~ 1 ½ inches above the cemented solid.

The Pu and Am had leveled off at around 2,000 ppb for Pu and 18 ppb for Am. This was an increase for both actinides from the initiation of the experiment. The color of the brine (pale brownish-gray) has been an indication that this was not a reduced environment experiment in contrast to LS 37.

The identification of Pu in most of the filters was interesting relative to LS-38 which showed no Pu.

IV. (b) Deactivation and Decommission (D&D) of Test Containers

Plan for Deactivating and Decommissioning the Actinide Source-Term Waste Test Program

Goal:
To deactivate and decommission (D&D) the Actinide Source-Term Waste Test Program (STTP) experiments within one year, according to facility, NMT-Division, LANL (Los Alamos National Laboratory), and DOE (Department of Energy) regulations. The STTP experiment is located in Wing 9, of the CMR (Chemistry & Metallurgy Research) Building, housed in two environmental enclosures within rooms 9010A and 9010B. The CMR is a Nuclear Facility under NMT (Nuclear Materials & Technology) Division management. All safety protocols must be incorporated into the D&D process.

Description of STTP:
The STTP is an experimental program conducted by Los Alamos National Laboratory for the Department of Energy, Carlsbad Area Office (CAO). The STTP was implemented in 1995 and has been maintained for five and one-half years. The STTP is a dynamic test program, designed to:

1. Provide time sequential quantitative measurements of mobile actinide concentrations in synthetic WIPP (Waste Isolation Pilot Plant) brines that have been in continual contact with actual TRU (Transuranic) wastes for over five years;
2. Establish the influence of Salado type brine (Brine A) and Castile brine on the chemistry and concentration of mobile actinides; and
3. Allow for comparison with the hypotheses of actinide solubility models developed from laboratory tests and actinide literature.

The STTP consists of 39 liter-scale test containers and 15 drum-scale test containers. The 39 liter-scale test containers are configured as follows:

12 Portland cement containers
12 Envirostone containers; and
15 Pyrochemical salt containers.

The drum containers were divided as follows:

12 heterogeneous TRU wastes; and
3 massive metal TRU wastes.

Included in the liter-scale tests are 6 test containers that are much heavier duty and are pressurized to 60 bars (870 psig) with CO_2. Three of these contain Portland cement waste and three are Pyrochemical salt waste.

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All STTP test containers have added soluble ^{232}Th, ^{238}U, and ^{237}Np. Six liter-scale containers have added ^{241}Am. All test containers have added Fe mesh, which should be observed and examined as part of the D&D process. Most STTP containers were fabricated from titanium metal and have stainless steel hardware affixed to the lids.

D&D Preparatory Activities:

- Establish D&D process and extent of D&D including/not including enclosures.
- Assure completion of CMR Nuclear Facility Authorization Basis Document, which includes Basis for Interim Operations (BIO).
- Initiate notification to the New Mexico Environmental Department through DOE-LAAO (Los Alamos Area Office) that D&D process will be implemented.
- Assure continuation of required EPA and RCRA documentation from daily inspections in RCRA interim storage area.
- Initiate documentation to close-out STTP as a Permitted Interim Status Storage Area
- Assure compliance with STTP Environmental Assessment Document for close out of STTP.
- Develop a Closure Plan
- Complete Hazards Analysis for D&D Operations in CMR
- Develop SOP or Work Instruction According to NMT Division Guidelines.
- Complete Hazard Control Plan (HCP) for the STTP D&D process for both liter-scale and drum-scale operations.
- Obtain approval for fabrication of any new equipment and installation into facility for the D&D process
- Assure any new equipment has passed Swagelok Testing according to NMT procedures.
- Develop path forward plan for disposal of STTP wastes generated by the D&D process.
- Develop a Transuranic Waste Interface Document (TWID)

D&D Process:

STTP Liter-scale Experiment:

- Brine from liter-scale test containers (35-50 liters)
- TRU waste brine
- Mixed TRU waste brine
- Low level brine waste
- Low level mixed waste brine
- Hardware from lid on liter-scale test containers
- Pressure relief tubing and hardware connected to facility ventilation system (Facility interface contractor maybe required)
- Rotators for liter-scale test containers
- Rotators for pressurized liter-scale test containers
- Brine sampling hardware for liter-scale containers
- Headspace gas sampling hardware and apparatus
- Disassembly and disposal of 40 temperature probes from liter-scale test containers

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- Continuous Air Monitors (CAM) disposal and/or reuse
- Disassembly of hard-piped Fixed Air Sample System (Facility interface to vacuum system, contractor maybe required)
- Eye-wash station deactivation and /or disassembly
- D&D of equipment and instrumentation to be re-used rather than going through disposal
- Packaging of STTP wastes according to LANL and NMT-Division waste packaging protocols and approved by waste packaging specialists
- Documentation of all waste packaging activities
- Assuring accountability of nuclear material in STTP test containers is transferred from STTP to waste account
- Decontaminate enclosure for transfer to facility or D&D and dispose of enclosure. For D&D and disposal operation obtain contractor to conduct D&D, which will include:
- Enclosure ventilation system
- Pumps and filtration system
- Pressure relief filtration system
- Electrical system
- Lighting system
- Enclosure walls
- Disconnecting utility system from facility
- Disconnecting telephone and emergency response system

STTP Drum-Scale Experiment:

- Brine from Drum-scale tests (750-850 gallons or 2,800-3,200 liters)
- TRU waste brine
- Mixed TRU waste brine (includes F-listed and D-listed substances)
- Low level waste brine
- Low level mixed waste brine (includes F-listed and D-listed materials)
- Fabrication of equipment to D&D 65-gallon all-titanium drums weighing about 800 lbs (360 kg)
- Obtain approval for fabricating and installing equipment in enclosure for D&D process on drums
- Determine need for contractor to install heavy-duty equipment for D&D operation
- Assure any new equipment has passed Swagelok Testing according to NMT procedures
- Hardware from STTP drums
- Rotators for drums
- Disassemble drum connections to facility utilities and dispose
- Disassemble drum-scale pressure relief tubing and hardware connected to Facility ventilation system (Facility interface contractor maybe required)

Continued on next page

- Brine sampling hardware for drum-scale containers, disposal
- Headspace gas sampling hardware and apparatus , disposal
- Disassembly and disposal of 15 temperature probes from drum-scale test containers
- Disconnect Continuous Air Monitors (CAMs) and dispose or decontaminate for reuse
- Disassembly of hard-piped Fixed Air Sample System (Facility interface to vacuum system, contractor maybe required)
- Eye-wash station deactivation and /or disassembly
- D&D of equipment and instrumentation to be re-used rather than going through disposal
- Packaging of STTP wastes according to LANL and NMT-Division waste packaging protocols and approved by waste packaging specialists
- Documentation of all waste packaging activities
- Assuring accountability of nuclear material in STTP test containers is transferred from STTP to waste account
- Decontaminate enclosure for transfer to facility or D&D and dispose of enclosure. For D&D and disposal operation obtain contractor to conduct D&D, which will include:
- Enclosure ventilation system
- Pumps and filtration system
- Pressure relief filtration system
- Electrical system
- Lighting system
- Enclosure walls
- Disconnecting utility system from facility
- Disconnecting telephone and emergency response system

Process for D&D of Drums from STTP Drum-Scale Experiment:

Several different plans were developed to D&D the drum-scale tests. To complete the D&D of the drum-scale test containers within the budget projected for this work effort, a plan was required that did not necessitate opening the lid of the drum in a glovebox environment. Of the several work plans that were developed the most straightforward and economical was not to open the drum lid but to solidify the ~50 gallons of brine by adding AQUASORBE 22-12 to the drum. Brine was sampled from several drums to establish the ratio of AQUASORBE to brine that was needed to completely solidify the brine within a 65-gal test container. The ration established for Brine A and Castile Brine was tested on two cold drums with added non-radioactive surrogate wastes. A large funnel with a wide opening ball valve was used to add the AQUASORBE. It was soon learned that the waste floating in the brine impeded the flow of adsorbent and so some of the brine from each drum was pumped out of the drum into a plastic bag contained in a second drum. After pumping out a portion of brine, AQUASORBE 22-12 was added to the drum, which was now about half full.

Continued on next page

The AQUASORBE then flowed unimpeded into the partially emptied drum and the brine that was initially pumped out was pumped back into the drum. Any excess brine was adsorbed in the bag and was disposed with the 65-gal drum containing the adsorbed brine in a standard waste box. This process was very successful in allowing disposal of the 65-gal drum and any excess brine that was adsorbed. We estimated that this process saved the project about $1-2 million. However, visual observation of the waste, additives, or the Fe mesh-contained vessels was not made. All 15 drum-scale test containers were processed through this D&D procedure without spread of contamination. Indeed there was not a single contamination incident in the entire STTP D&D process.

CMR Analytical Laboratories:

- D&D of laboratories dedicated to STTP D&D liter-scale operation
- D&D of hoods dedicated to STTP D&D activities
- D&D of analytical instrumentation dedicated to STTP analyses

Analytical Support Required for D&D of STTP:

- Radiochemistry of brine required for disposal
- pcH of brine required for disposal
- Analyses of RCRA elements required for disposal
- Analyses of actinide elements required for final disposal
- X-ray analyses of select materials may be required for disposal
- Radiochemistry required for safety examination of Fe mesh required for programmatic reasons
- Examination of unexpected crystals, residues, and salts required for programmatic reasons
- Visual observation of all liter-scale D&D efforts will be conducted (Management Safety Walk-Around required by Division Management)

Current Waste Recharge Rates at LANL TA-54

Waste Type	Dollars/Volume
Hazardous Waste (Non-DP)	11.00 per kg
Solid LLW Non-Compactible (non-DP)	2,486 per m^3
Solid LLW Compactible (non-DP)	1,250 per m^3
Mixed LLW (non-DP)	88,305 per m^3
TRU Waste (non-DP)	58,000 per m^3

Estimated Volumes for Liter-Scales:

- 39 liter-scale test containers — 1 m^3
- Solidified brine @ 10% efficiency — 0.3 m^3
- Other hardware — 0.7 m^3

Estimated Volumes for Drum-Scales:

- 15 drum-scale test containers @ 0.25 m3 each — 3.75 m^3
- Brine to be shipped to outside contractor
- Other hardware — 1.25 m^3

Estimated Waste Volume Cost*:

Liter Scale	2 m^3 × \$58,000 =	\$116,000
Drum Scale	5 m^3 × \$88,305 =	\$441,500
TOTAL COST		\$557,500

* Does not include enclosures and hardware.

Personnel and Capabilities Required for D&D Operations:

- STTP Management Team
- Project Leader (1)
- Principle Investigator (1)
- Administrative Assistant / Budget Analyst (1)
- QA Specialist (1)
- Training Specialist (1)
- Document Control Specialist (1)

Continued on next page

- Data Management / Report Writing (2)
- D&D Technicians & Staff
    - Brine and hardware (2)
    - Gas Analyses (2 @ 30%)
    - Engineer (0.6)

- NMT-1 Analytical Group
    - D&D Technicians (3)
    - D&D Staff (1)
    - Radiochemistry (1)
    - PcH (0.5)
    - RCRA (1)
    - Anions (0.5)
    - Gas (See STTP Mngt. Team)
    - LIMS (0.5)

- CST-9 Analytical Group
    - Actinides (1)

- NMT-11 Surface Science Team
    - Fe Analyses (0.3)
    - Other Analyses (0.3)

- ESA-EPE Engineering
    - Design & Implementation (0.3)
    - Fabrication & Operation (0.3)

- Waste Management
    - Waste Coordinator (0.2)
    - Waste Packaging Specialist (0.2)
    - RCRA Waste Specialist (0.2)

- Nuclear Materials Representative
    - NM Specialist (0.1)
- Transportation
    - Transportation Specialist

- Outside Contractor
    - STTP Enclosure – CMR Interface ($200.0k)
- Facility Personnel
    - Engineers

Continued on next page

  - Radiation Control Technicians (RCT)
  - Property Management
- TA-54
  - DVRS Costs for Storage and Volume Reduction
- Closure Plan
  - Costs driven by NMED

Concurrent Safety Maintenance Activities:
(During the D&D process, STTP safety maintenance must continue as part of the overall safety program and regulatory compliance work)

- STTP Management Team
- RCRA Inspections and Documentation
- EPA Inspections and Documentation
- Pressure Relief Operations for test containers that exceed 8-10 psig (pressure developed from radiolysis of brine and brine constituents)
- Preparation of test containers for D&D operations
- Decontamination of test containers that exclude brine contaminated salts
- Maintenance of test containers that exhibit plugging of pressure gages
- Maintenance of sampling port to allow depressurization of test containers
- Maintenance of all life-safety systems in enclosures
- Assuring ventilation system in enclosures maintain 5-7 turnover volumes per hour
- Assuring operation of all emergency response systems
- Replacement of pressure gages that become plugged; testing to assure proper operation
- Maintain documentation of historical maintenance of test containers
- Apply QA to all operations
- Maintain training of all workers as RCRA TSD workers

Disposal of STTP Legacy Wastes at WCRRF:
(Waste developed as part of the opening of waste drums and loading of STTP test containers still resides at the Waste Characterization, Reduction, and Repackaging Facility. STTP is responsible for characterization and repackaging of this waste for disposal.)

Activities at the WCRRF

- Identify STTP Legacy Waste
- Characterize Waste Forms
- TRU Waste

- Mixed TRU Waste
- Mixed Low Level Waste
- Low Level Waste
- Package Waste
- Dispose of Waste

STTP Budget Options & Considerations*

Cost Estimates ($M):

Option 1: Maintain STTP in Standby Condition

FY 2001	2.2
Standby Total:	**$2.2M**

Option 2: D&D Process will require 2 years

Majority of work will be in FY 20001

• Basic STTP Operational/Management Team	2.2
• D&D Liter-Scale (LS) Test Containers	2.0
• D&D Drum-Scale (DS) Test Containers	2.0
• D&D STTP Residual Wastes	0.5
Total:	**$6.7M**

Complete D&D Work in FY 2002

• Basic STTP Team	1.4
• Complete Unfinished D&D (if needed)	(1.0)
• D&D of Enclosures (if needed)	(1.0)
• D&D of Administrative Functions/Records	0.3
Total:	**$1.7 - 3.7M**

2 yr. D&D Total:	**$8.4 -10.4M**

Option 3: Conduct D&D in 3 Years (equally divided)

• Basic STTP Team	3 x 2.2 = 6.6
• D&D LS and DS Tests	4.0
• D&D Enclosures (if needed)	(1.0)
• D&D Administrative Functions	0.3
3 yr. D&D Total:	**$12.4M**

**Estimate does not include 38% space tax*

Proposal for the Deactivating and Decommissioning Plan for the Actinide Source-Term Waste Test Program

Subject: D&D Plan for STTP with Budget and Safety Considerations

The D&D of the STTP in the Chemistry & Metallurgy Research (CMR) Building must be completed according to **all** the requirements for conducting work in a DOE operated Nuclear Facility. There can be no shortcuts to Safety, Quality Assurance, Training, and Formality of Operations! The retention of the existing management and operation team is essential for the D&D operation to be completed in an effective and efficient manner.

The STTP experiment has not had a safety incident in over four years of operation. The required D&D process is a very complicated set of activities with highly radioactive test containers, brine, brine wetted components, sludge, and hardware. The potential for a contamination incident is always present, but was easier to mitigate with the sampling type work previously done. The D&D process is much more complicated, and needs to be monitored carefully at all times. A single contamination incident can set the D&D plan back for an indeterminate period, thus delaying the anticipated schedule for completion of the D&D process. Safety or security infractions by any non-STTP activity in the CMR or TA-55 can result in DOE, LANL, or the Nuclear Materials and Technology (NMT) Division placing a "Stop Work" or "Stand Down" order on all activities. These orders require lengthy and expensive resumption activities for all projects and teams within the facilities. These prevalent risks must be acknowledged by the sponsor of projects in DOE-operated Nuclear Facilities.

The D&D plan for the STTP takes into consideration this complex set of activities to be conducted in an effective and sequentially-organized manner according to rules and regulations applicable to DOE operations in a Nuclear Facility. These include:

1. Code of Federal Regulations
    - EPA mandates
    - RCRA mandates
2. DOE-Orders for Formality of Operations
3. DOE required Quality Assurance for all operations
4. Price-Anderson Act Compliance
5. LANL Safety Requirements
6. NMT Division Safety Requirements
7. DOE Authorization Basis Documentation
    - Basis of Interim Operation (BIO)
8. Hazards Analyses Documentation
9. Standard Operating Procedures
10. Hazard Control Plans
11. Work Instructions

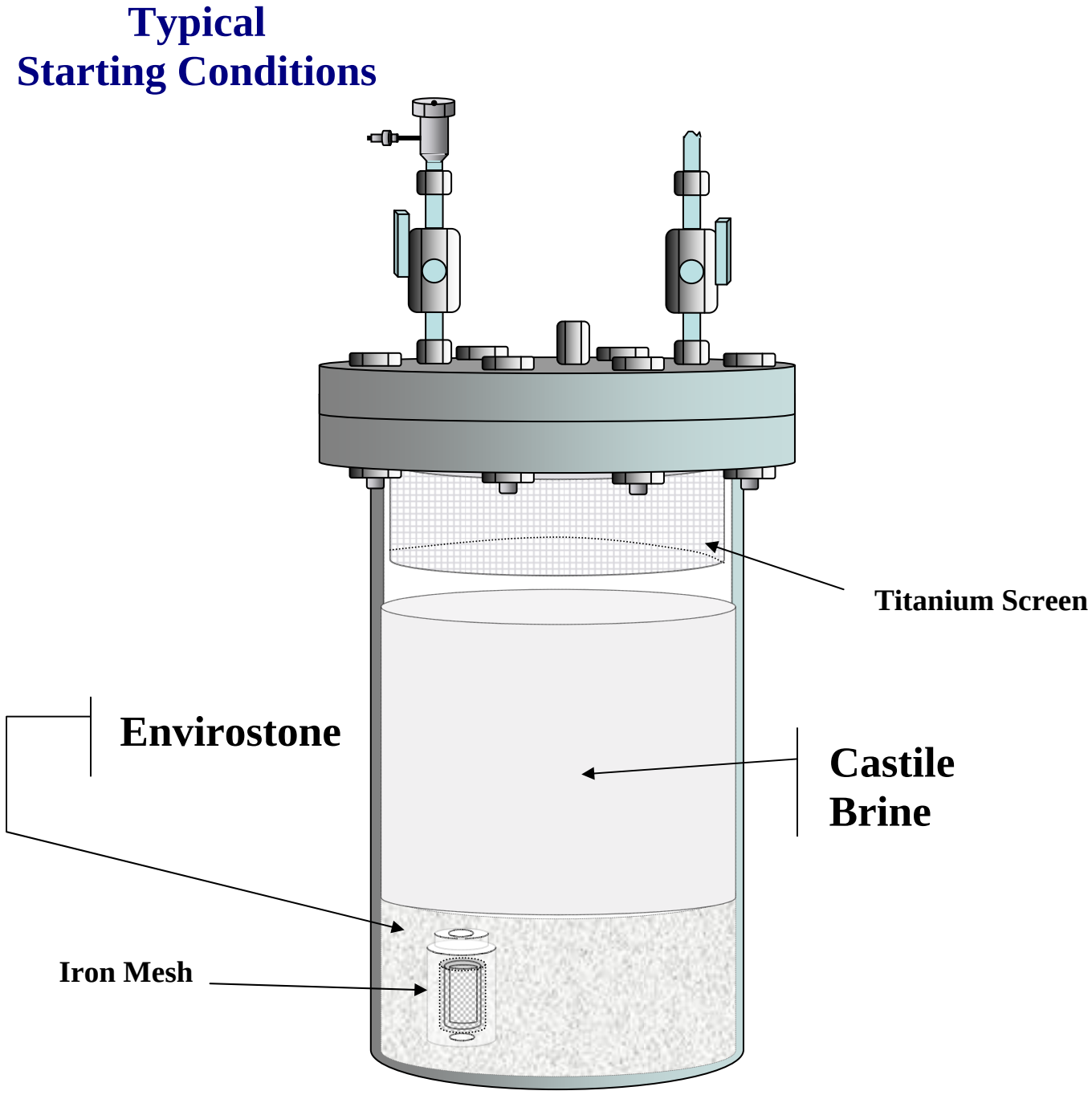
Continued on next page

12. Radioactive Work Procedures
13. Special Work Permits
14. Compliance Training
15. Technical Training
16. On-the-Job Training (OJT)

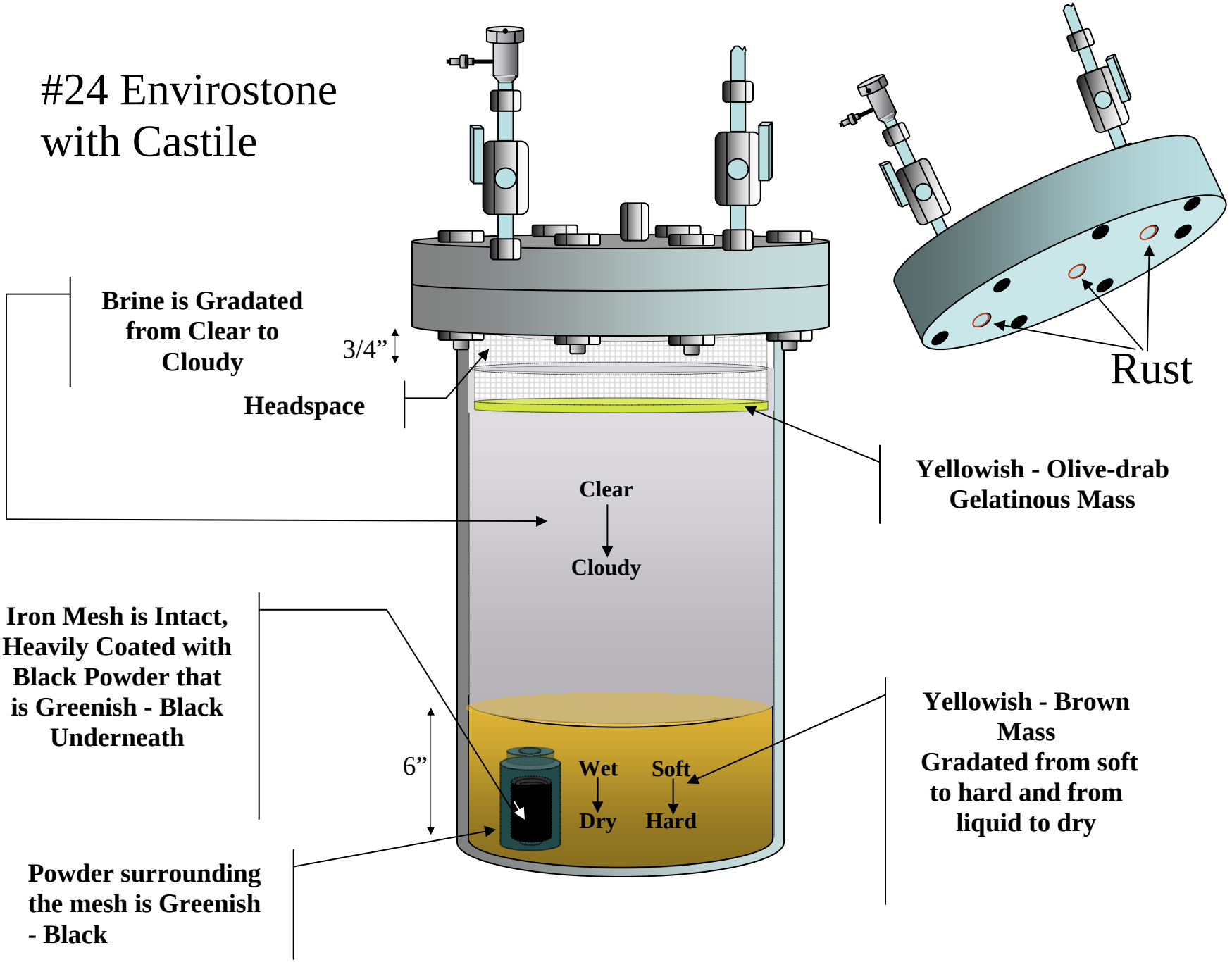
Furthermore, NMT Division will assess a 38 to 46% space tax on all non-DP (Defense Programs) projects, of which STTP is one, located within CMR, because it is a DP Nuclear Facility.

IV. (b) Deactivation and Decommission (D&D) of Test Containers

Diagrams

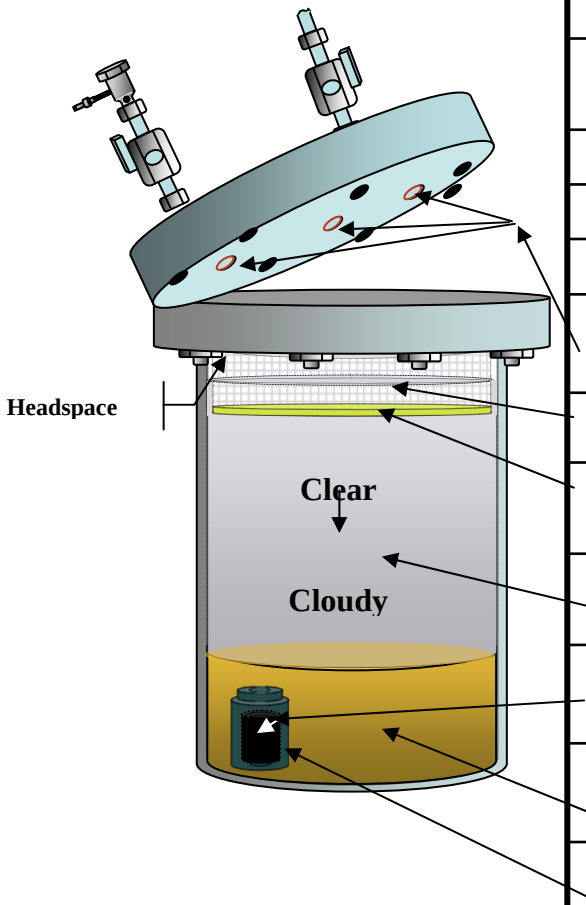


#24 Envirostone with Castile



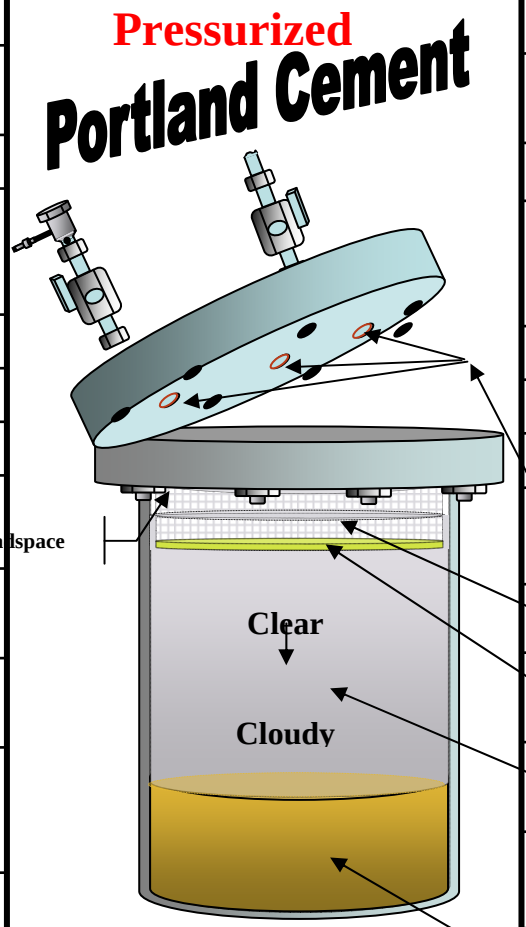
Portland Cement

	L-1	L-2
Waste Type	Portland Cement	Portland Cement
Pu Content	0.018g	0.165g
Brine	A	A
Additives	Fe Mesh Actinides	Fe Mesh, Actinides
pcH Range	8.7-9.0	10.3-10.6
Fe, ppm	< 1	< 1
Special (ratio)	10:1	2:1
Corrosion of s.s. Feed Throughs	None	None
Screen Condition	Black, No Corrosion	No Corrosion
Material in Screen	Some Solids	Filled with Solids
Brine Color/viscosity	Clear	Clear, Gray
Iron Mesh Color/Corrosion	Gray, Black No Corrosion	Gray, Black No Corrosion
Bottom Solids Loose/Cemented	3” Loose	7” Loose
Sludge	Gray, Compacted	Gray, Compacted



	L-3
Waste Type	Portland Cement
Pu Content	0.123g
Brine	Castile
Additives	Fe Mesh, Actinides
pcH Range	12.8-13.1
Fe, ppm	< 1
Special (ratio)	2:1
Corrosion of s.s. Feed Throughs	None
Screen Condition	No Corrosion
Material in Screen	1/2 Filled
Brine Color/viscosity	Clear, Gray
Iron Mesh Color/Corrosion	Gray, Black No Corrosion
Bottom Solids Loose/Cemented	8” Loose
Sludge	Gray, Compacted

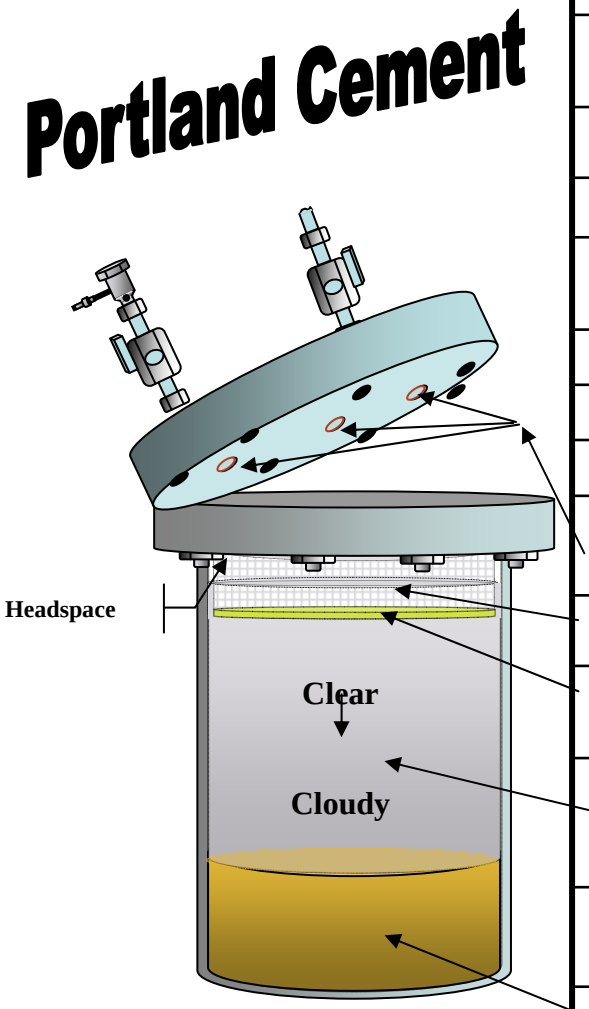
	L-4	L-5
Waste Type	Portland Cement	Portland Cement
Pu Content	Pu, 18.95 mg; Am, 0.233 mg	Pu, 51.2 mg; Am, 0.595 mg
Brine	A	A
Additives	Fe mesh, Nd, Th, U, Np, CO_2 60 Bar	Fe mesh, Nd, Th, U, Np, CO_2 60 Bar
pcH Range	7.21-7.43	7 - 7.44
Fe, ppm	3.1 - 162.4	7 - 57.1
Special (ratio)	10:1	3:1
Corrosion of s.s. Feed Throughs	None	None
Screen Condition	None	None
Material in Screen	None	None
Brine Color/viscosity	Clear, non-viscous light gray	Clear, non-viscous light brown
Iron Mesh Color/Corrosion	Green-gray	Black with blue tint
Bottom Solids Loose/Cemented	Compacted, light gray	5.5” yellow-brown Compacted
Sludge	gray	Peanut butter



STTP

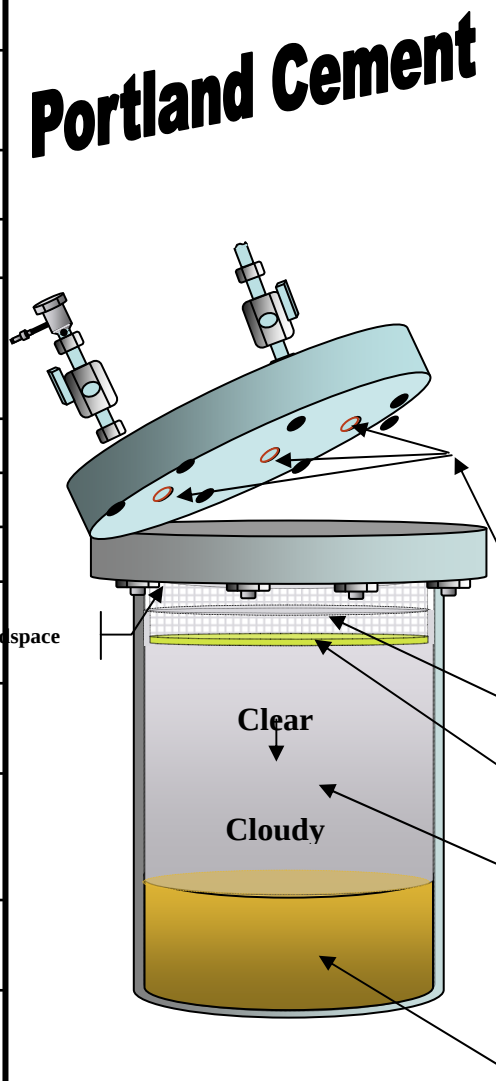
	L-6
Waste Type	Portland Cement
Pu Content	Pu, 97.4 mg; Am, 0.994 mg
Brine	Castile
Additives	Fe mesh, Nd, Th, U, Np, CO_2 60 Bar
pcH Range	7.49 - 7.87
Fe, ppm	2 - 41.4
Special (ratio)	2:1
Corrosion of s.s. Feed Throughs	None
Screen Condition	None
Material in Screen	None
Brine Color/viscosity	Milky, ~2” deep
Iron Mesh Color/Corrosion	Dark colored
Bottom Solids Loose/Cemented	~8” Brownish-gray soft solid
Sludge	Clay-like

	L-7	L-8
Waste Type	Portland Cement	Portland Cement
Pu Content	0.025g	0.117g
Brine	A	A
Additives	No Fe Mesh Actinides	No Fe Mesh, Actinides
pcH Range	8.7-8.9	9.1-9.56
Fe, ppm	< 2	< 1
Special (ratio)	10:1	2:1
Corrosion of s.s. Feed Throughs	Yes	None
Screen Condition	No Corrosion	No Corrosion
Material in Screen	Thin Coating	1/8” Solids
Brine Color/viscosity	Colorless Liquid	Gray Liquid
Iron Mesh Color/Corrosion	Not Added	Not Added
Bottom Solids Loose/Cemented	8” Loose Muddy Solids	8” Loose 4” Cemented
Sludge	N/A	N/A



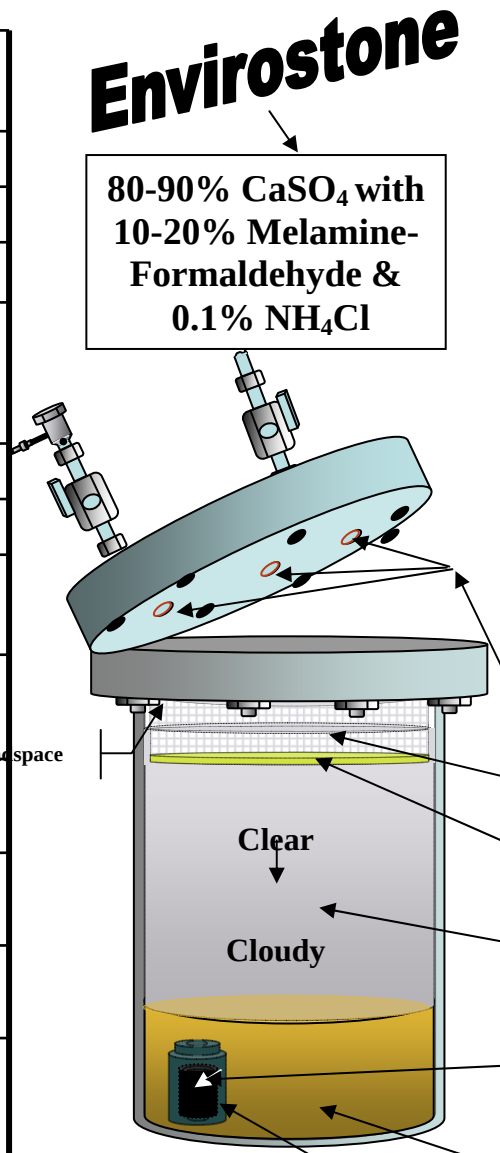
	L-9
Waste Type	Portland Cement
Pu Content	0.107g
Brine	Castile
Additives	No Fe Mesh, Actinides
pcH Range	12.9-13.1
Fe, ppm	< 1
Special (ratio)	2:1
Corrosion of s.s. Feed Throughs	None
Screen Condition	No Corrosion
Material in Screen	1/2 “ of Gray Paste
Brine Color/viscosity	Clear Liquid with Suspensions
Iron Mesh Color/Corrosion	Not Added
Bottom Solids Loose/Cemented	6-8” Loose Not Cemented
Sludge	N/A

	L-10	L-11
Waste Type	Portland Cement	Portland Cement
Pu Content	0.021g	0.12g
Brine	A	A
Additives	No Fe Mesh Actinides ^{241}Am	No Fe Mesh, Actinides ^{241}Am
pcH Range	8.2-8.9	9.0-10.8
Fe, ppm	< 1	< 1
Special (ratio)	10:1	2:1
Corrosion of s.s. Feed Throughs	Yes	Yes
Screen Condition	Some Corrosion	Corrosion Products
Material in Screen	1/8” Sludge and Rust	1-2” Gray Sediment with Rust
Brine Color/viscosity	Clear	Gray
Iron Mesh Color/Corrosion	Not Added	Not Added
Bottom Solids Loose/Cemented	3-4” Compacted	8” Loose, 1” Compacted
Sludge	N/A	N/A



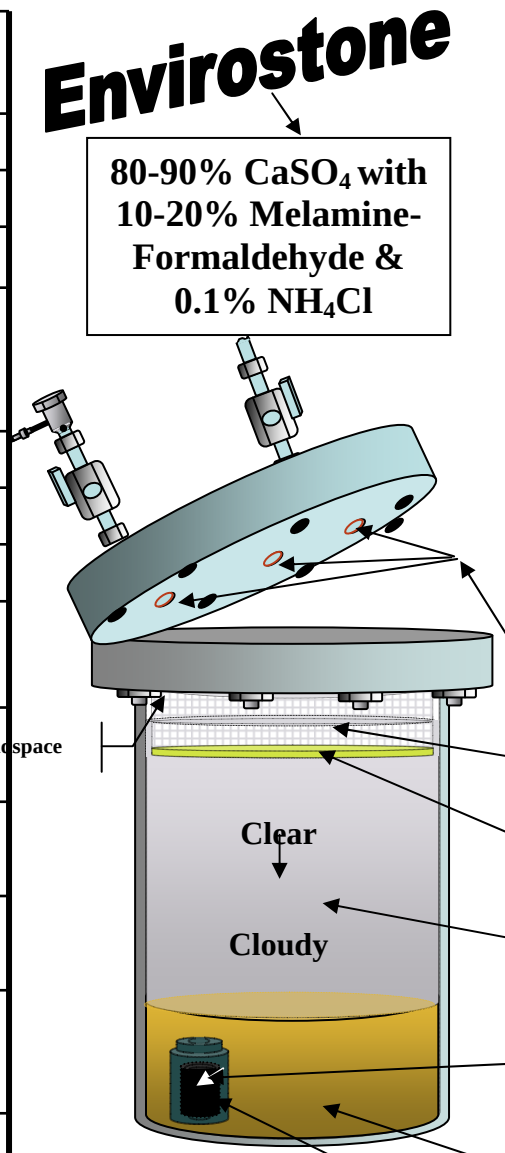
	L-12
Waste Type	Portland Cement
Pu Content	0.108g
Brine	Castile
Additives	No Fe Mesh, Actinides ^{241}Am
pcH Range	12.7-13.0
Fe, ppm	< 1
Special (ratio)	2:1
Corrosion of s.s. Feed Throughs	Yes
Screen Condition	Black and Coated
Material in Screen	~1/4” Solids, Gray
Brine Color/viscosity	Pale Gray-Green
Iron Mesh Color/Corrosion	Not Added
Bottom Solids Loose/Cemented	8” Loose gray Sludge
Sludge	N/A

	L-13	L-14
Waste Type	Envirostone	Envirostone
Pu Content	3.4g	3.42g
Brine	A	A
Additives	Fe Mesh Actinides Organics	Fe Mesh, Actinides Organics
pcH Range	7.0-7.29	6.9-7.3
Fe, ppm	14 - 326	100 - 466
Special (ratio) H_2 in vol. %	2:1, H_2 =57 %	2:1 , H_2 =37 %
Corrosion of s.s. Feed Throughs	None TOC = 3300ppm	None TOC = 4000ppm
Screen Condition	No Corrosion	No Corrosion Looked new
Material in Screen	Coating of Yellow Paste	Thin coating of white Material
Brine Color/viscosity	Clear with Suspensions	Clear with Suspensions
Iron Mesh Color/Corrosion	No Corrosion, Greenish-yellow Sludge	No Corrosion, Dark Color
Bottom Solids Loose/Cemented	~4" of peanut butter Consistency	4.5" of yellowish paste
Sludge	Impacted	Impacted



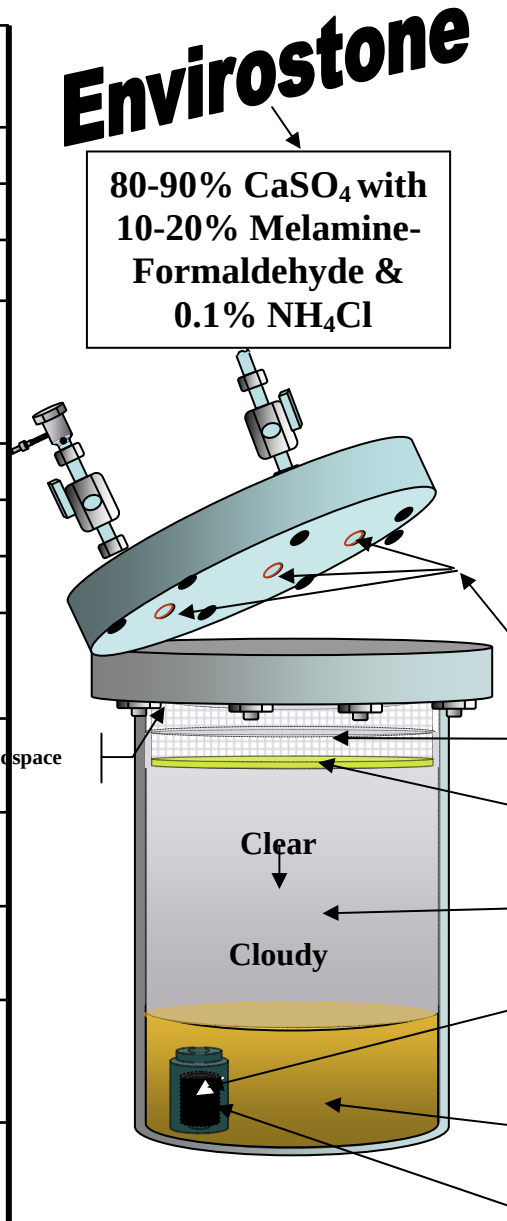
	L-15
Waste Type	Envirostone
Pu Content	0.042g
Brine	Castile
Additives	Fe Mesh, Actinides Organics
pcH Range	6.8-7.1
Fe, ppm	1 - 36
Special (ratio)	2:1 , H_2 =3 %
Corrosion of s.s. Feed Throughs	None TOC = 1400 ppm
Screen condition	No Corrosion
Material in Screen	~2" Oatmeal-like Material
Brine Color/viscosity	Cream Colored with Oatmeal Consistency
Iron Mesh Color/Corrosion	No Corrosion, Gray-Black material
Bottom Solids Loose/Cemented	5-7" of loose solids
Sludge	Impacted

	L-16	L-17
Waste Type	Envirostone	Envirostone
Pu Content	0.62g	1.5g
Brine	A	A
Additives	Fe Mesh Actinides	Fe Mesh, Actinides
pcH Range	7.3-8.0	7.7-8.0
Fe, ppm	1 - 33	< 1
Special (ratio)	2:1	2:1
Corrosion of s.s. Feed Throughs	None Like New	None Like New
Screen Condition	Like New	Like New
Material in Screen	Tan Fines in Screen	No Sediment
Brine Color/viscosity	Tan, Cloudy	Tan, Clear
Iron Mesh Color/Corrosion	No Corrosion, Greenish-Black	No Corrosion, Immersed in Tan Sludge
Bottom Solids Loose/Cemented	8” of Tan loose Sludge	8” of Black-Tan Sludge
Sludge	Tan	Black-Tan



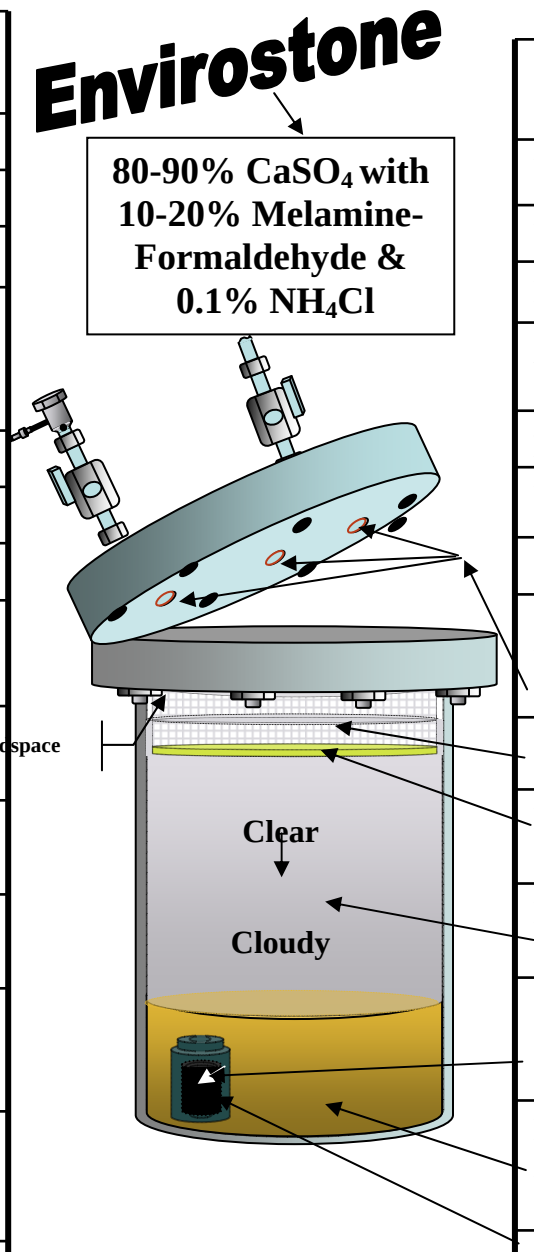
	L-18
Waste Type	Envirostone
Pu Content	2.6g
Brine	Castile
Additives	Fe Mesh, Actinides
pcH Range	7.0-7.8
Fe, ppm	2 - 50
Special (ratio)	2:1
Corrosion of s.s. Feed Throughs	No Corrosion Orange-brown Solids
Screen Condition	Thin-brown Sludge on Screen
Material in Screen	Thin Coat of Muddy Clay
Brine Color/viscosity	Yellow Tan with Suspensions
Iron Mesh Color/Corrosion	Black Clay, Black Wire, No Corrosion
Bottom Solids Loose/Cemented	Muddy Suspension
Sludge	Yellow-Tan

	L-19	L-20
Waste Type	Envirostone	Envirostone
Pu Content	0.502g	0.080g
Brine	A	A
Additives	Fe Mesh Actinides	Fe Mesh, Actinides
pcH Range	7.9-8.2	7.2-7.8
Fe, ppm	< 1	20 - 95
Special (ratio)	2:1, N_2O = 16%	2:1, N_2O = 20%
Corrosion of s.s. Feed Throughs	None	
Screen Condition	Like New	
Material in Screen	1/8" Sludge - Orange	
Brine Color/viscosity	Clear-light yellow	
Iron Mesh Color/Corrosion	Black Sludge, Black coating, Some Corrosion	
Bottom Solids Loose/Cemented	Soft Sludge	
Sludge	Black	



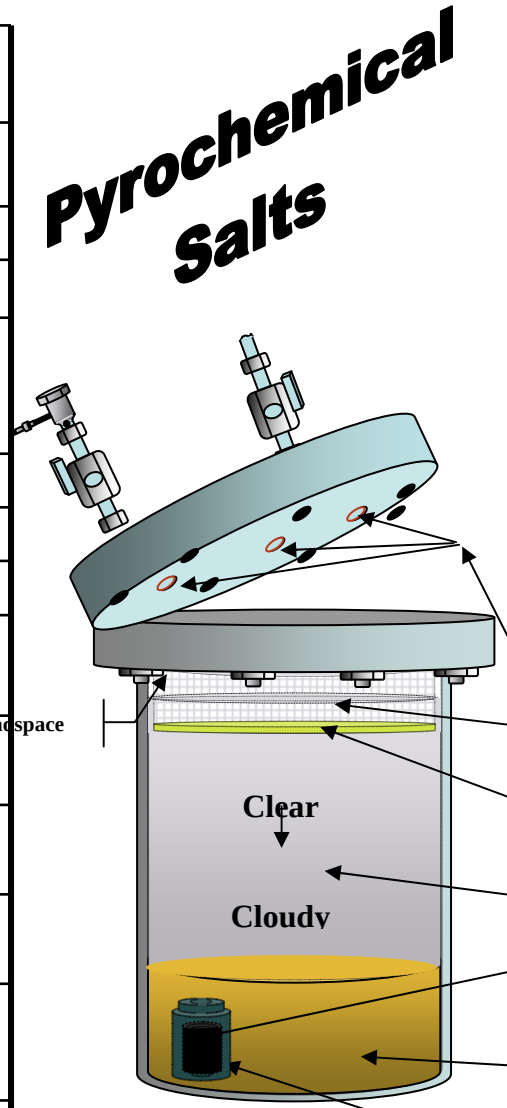
	L-21
Waste Type	Envirostone
Pu Content	0.251g
Brine	Castile
Additives	Fe Mesh, Actinides
pcH Range	7.5-8.1
Fe, ppm	< 0.1
Special (ratio)	2:1, N_2O = 19%
Corrosion of s.s. Feed Throughs	None
Screen condition	Like New
Material in Screen	Clear- Orange Tinge
Brine Color/viscosity	Clear- Orange Tinge
Iron Mesh Color/Corrosion	Black Coating, No Corrosion
Bottom Solids Loose/Cemented	Loose Solids
Sludge	White and Orange Tinge

	L-22	L-23
Waste Type	Envirostone	Envirostone
Pu Content	0.238g	0.502g
Brine	A	A
Additives	Fe Mesh Actinides	Fe Mesh, Actinides
pcH Range	6.8-7.2	7.0-7.4
Fe, ppm	50-120	10-102
Special (ratio)	2:1, N_2O = 39%	2:1, N_2O = 31%
Corrosion of s.s. Feed Throughs	None	None Discoloration
Screen Condition	No Corrosion	No Corrosion
Material in Screen	3/4" Sediment	Gold Colored Sludge, thin
Brine Color/viscosity	Light Brown to gold, Murky	Gold Colored 6" deep
Iron Mesh Color/Corrosion	3 Pieces, Greenish Corrosion	Black Colored, some corrosion
Bottom Solids Loose/Cemented	7" of Gold Colored Sludge	6" of Gold Colored Sludge Loose solids
Sludge	Black	



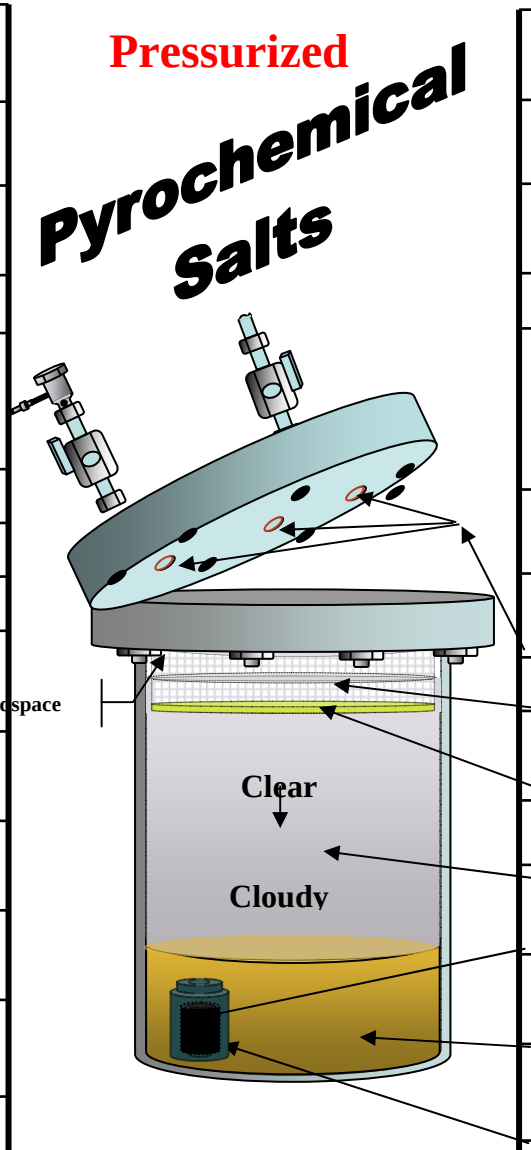
	L-24
Waste Type	Envirostone
Pu Content	0.305g
Brine	Castile
Additives	Fe Mesh, Actinides
pcH Range	7.6-7.9
Fe, ppm	< 1
Special (ratio)	2:1, N_2O = 25%
Corrosion of s.s. Feed Throughs	None Discoloration of Sampling Port
Screen condition	Trace Corrosion
Material in Screen	Thin Coating, Semi-Gelatinous
Brine Color/viscosity	Yellow-olive-drab, murky
Iron Mesh Color/Corrosion	Greenish-black deposit on wire, some corrosion
Bottom Solids Loose/Cemented	6" of Clay-like mass of loose solids
Sludge	

	L-25	L-26
Waste Type	Pyrochemical Salts (DOR)	Pyrochemical Salts (DOR)
Pu Content	0.38g	4.1g
Brine	A	A
Additives	Fe Mesh Actinides	Fe Mesh Actinides
pcH Range	7.7-8.1	7.6-8.2
Fe, ppm	< 1; 1-20 50%	< 1 -generally
Special (ratio)	2:1	2:1
Corrosion of s.s. Feed Throughs	Yes, Green Color	Yes
Screen Condition	O-ring Corrosion	No Corrosion
Material in Screen	1/3 full of crystals and Sludge	1/2" yellow-brown Sediment
Brine Color/viscosity	Whitish murky color	Whitish murky color
Iron Mesh Color/Corrosion	Embedded in light blue solids	Corrosion noted, much encrustations
Bottom Solids Loose/Cemented	5" depth, not cemented	3-31/2" Loose Solids
Sludge	Light Blue	Black-White Precipitate



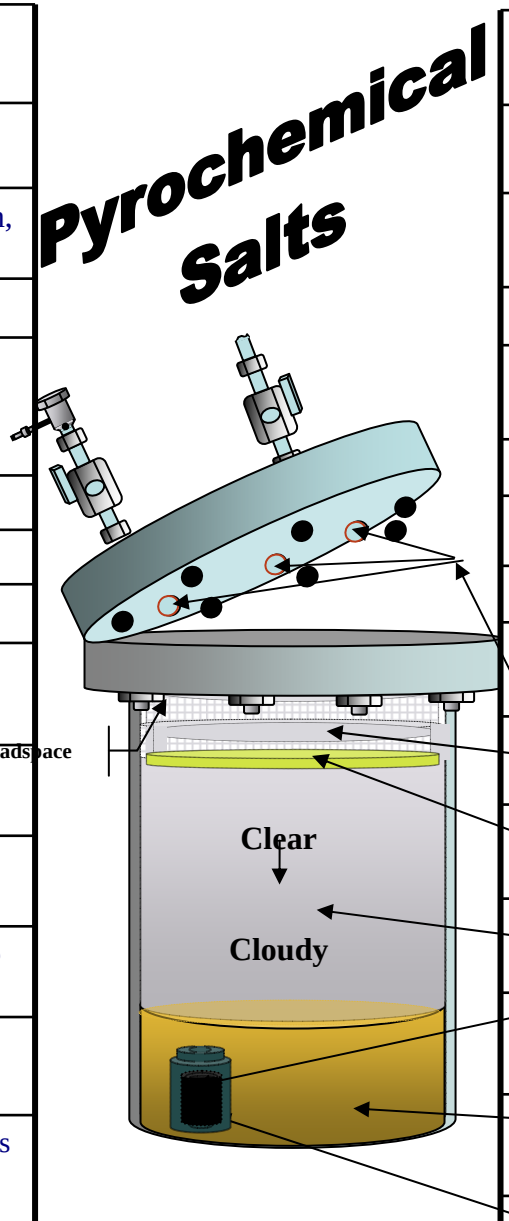
	L-27
Waste Type	Pyrochemical Salts (DOR)
Pu Content	3.41g
Brine	Castile
Additives	Fe Mesh Actinides
pcH Range	10.7-11.2
Fe, ppm	43-243
Special (ratio)	2:1
Corrosion of s.s. Feed Throughs	Yes, Severe Corrosion
Screen Condition	Rust Particles
Material in Screen	Full of Green Colored Deposits
Brine Color/viscosity	Clear Whitish color
Iron Mesh Color/Corrosion	Black Color around Fe Mesh
Bottom Solids Loose/Cemented	7" Loose Solids
Sludge	Loose Solids

	L-29	L-30
Waste Type	Pyrochemical Salts (DOR)	Pyrochemical Salts (DOR)
Pu Content	Pu, 4.338 mg; Am, 2.38 mg	Pu, 2.010 mg; Am, 2.38 mg
Brine	A	Castile
Additives	Fe mesh, Nd, Th, U, Np, CO_2 60 Bar	Fe mesh, Nd, Th, U, Np, CO_2 60 Bar
pcH Range	4.73 - 5.68	5.9 - 6.6
Fe, ppm	34 - 1468	1967 - 9.2
Special (ratio)	2:1	2:1
Corrosion of s.s. Feed Throughs	None	None
Screen Condition	None	None
Material in Screen	None	None
Brine Color/viscosity	clear	Gray to brown oatmeal
Iron Mesh Color/Corrosion	Black thin coating	Dark coating
Bottom Solids Loose/Cemented	Muddy brown - sand texture	Brown hard solid
Sludge	Dark	Hard brown solid



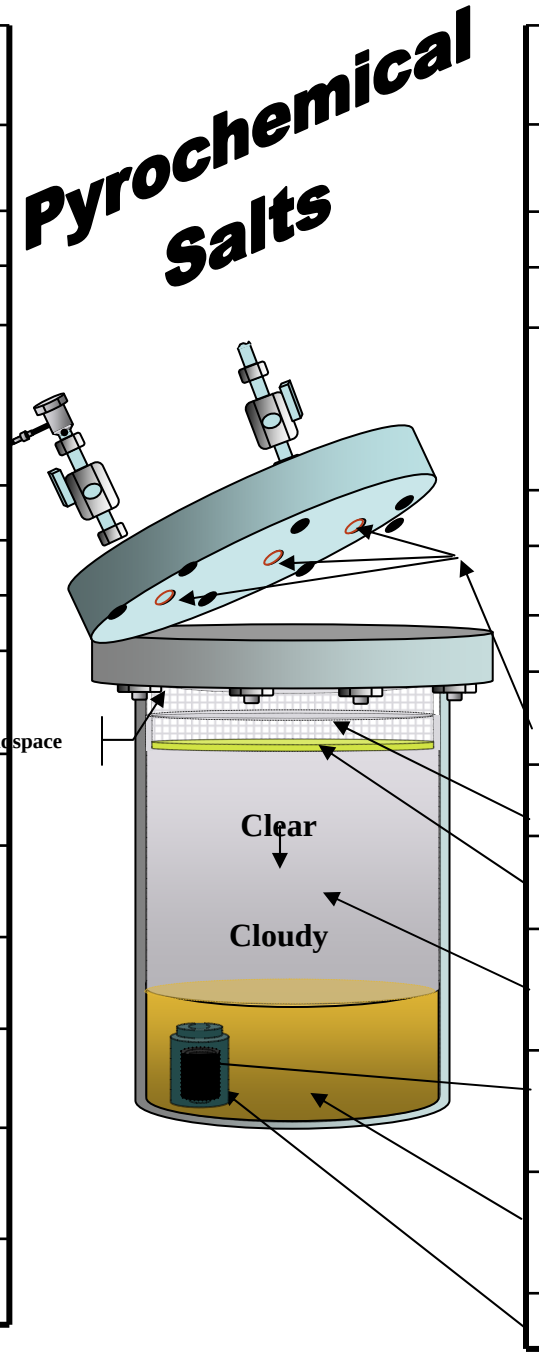
	L-28
Waste Type	Pyrochemical Salts (DOR)
Pu Content	Pu, 10.6 mg; Am, 1.24 mg
Brine	A
Additives	Fe mesh, Nd, Th, U, Np, CO_2 60 Bar; 76.4 g MgO (2/97)
pcH Range	4.48 - 5.35; 5.03 - 7.7 after MgO
Fe, ppm	19 - 165 ; 4.48 - 7.7 after MgO
Special (ratio)	2:1
Corrosion of s.s. Feed Throughs	None
Screen Condition	None
Material in Screen	None
Brine Color/viscosity	Cloudy to yellow
Iron Mesh Color/Corrosion	Thin dark coating, new like
Bottom Solids Loose/Cemented	Very hard yellow cement
Sludge	Oatmeal like

	L-31	L-32
Waste Type	Pyrochemical Salts (DOR)	Pyrochemical Salts (OS)
Pu Content	Pu, 0.809 g; Am, 0.647 mg	Pu, 4.10 g; Am, 2.92 mg
Brine	A	A
Additives	Fe mesh, Nd, Th, U, Np,	Fe mesh, Nd, Th, U, Np,
pcH Range	8.7 - 9.0	8.6 - 9.0
Fe, ppm	< 1	< 1
Special (ratio)	2:1, H_2 = 33 $^v/_o$	2:1, H_2 = 44. $^v/_o$
Corrosion of s.s. Feed Throughs	No Data	Yes
Screen Condition	No Data	Yes
Material in Screen	No Data	Salts
Brine Color/viscosity	No Data	Clear Pea soup consistency
Iron Mesh Color/Corrosion	No Data	Embedded in cement
Bottom Solids Loose/Cemented	No Data	Cemented solids
Sludge	No Data	Cemented solids



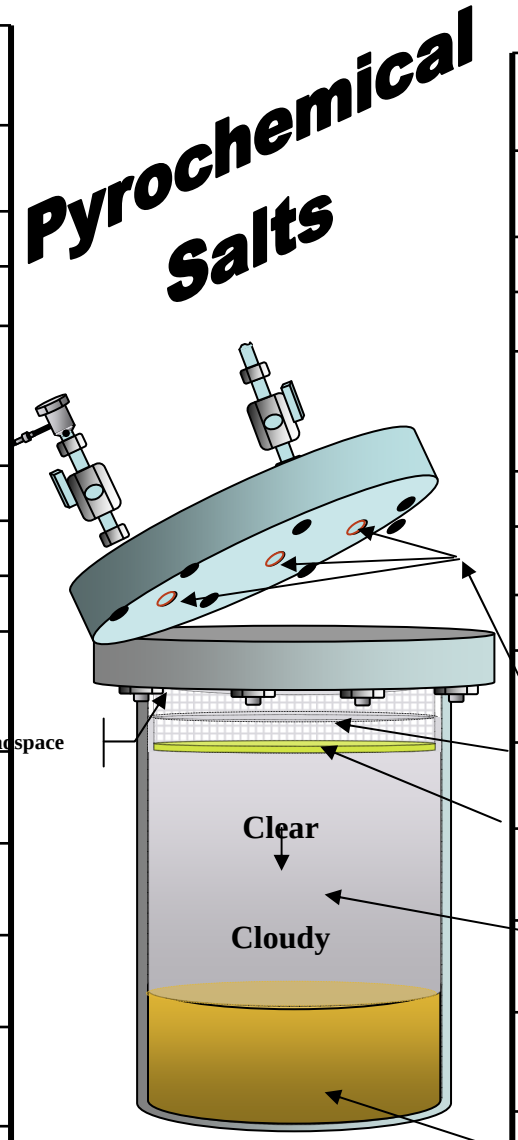
	L-33
Waste Type	Pyrochemical Salts (OS)
Pu Content	Pu, 1.14 g; Am, 1.10 mg
Brine	Castile
Additives	Fe mesh, Nd, Th, U, Np,
pcH Range	9.5 - 9.8
Fe, ppm	< 0.1
Special (ratio)	2:1, H_2 = 35 $^v/_o$
Corrosion of s.s. Feed Throughs	Severely corroded
Screen Condition	Upper ring Corroded
Material in Screen	1/2” grey sludge
Brine Color/viscosity	Milky White
Iron Mesh Color/Corrosion	Black coating, no visible corrosion
Bottom Solids Loose/Cemented	No cemented solids,~2” waste
Sludge	~ 2”

	L-34	L-35
Waste Type	Pyrochemical Salts (DOR)	Pyrochemical Salts (DOR)
Pu Content	2.1g	0.45g
Brine	A	A
Additives	Fe Mesh Actinides, Chelators $Ca(OH)_2$	Fe Mesh Actinides, Chelators $Ca(OH)_2$
pcH Range	8.6-9.0	8.2-8.3
Fe, ppm	< 2	< 1
Special (ratio)	3:1, H_2 = 29%	3:1, H_2 = 21%
Corrosion of s.s. Feed Throughs	Yes, Blue & Brown	Yes, Blue - Black
Screen Condition	O-ring Corroded	O-ring Blue
Material in Screen	Full of White Solids	Thin Coating
Brine Color/viscosity	Colorless with Suspensions	Murky Beige, very thick
Iron Mesh Color/Corrosion	Embedded in cemented solids	Greenish-blue, not corroded
Bottom Solids Loose/Cemented	5” with 3-4” cemented	8” Loose Solids
Sludge	Oatmeal Consistency	Blue colored layer



	L-36
Waste Type	Pyrochemical Salts (DOR)
Pu Content	11.1g
Brine	Castile
Additives	Fe Mesh Actinides, Chelators $Ca(OH)_2$
pcH Range	11.0-11.4
Fe, ppm	50-100
Special (ratio)	3:1, H_2 = 70%
Corrosion of s.s. Feed Throughs	Yes, Rust Colored
Screen condition	Rust Particles
Material in Screen	Full of gray Muck
Brine Color/viscosity	Greenish-gray, thickened near the bottom
Iron Mesh Color/Corrosion	Embedded in cemented solids, 1/2 retrieved
Bottom Solids Loose/Cemented	3-4” Loose Solids, 1/2 cemented
Sludge	Gray Sludge

	L-37	L-38
Waste Type	Pyrochemical Salts	Pyrochemical Salts
Pu Content	4.42g	2.74g
Brine	A	A
Additives	No Fe Mesh, Th, U, Np, Nd, ^{241}Am	No Fe Mesh, Th, U, Np, Nd, ^{241}Am
pcH Range	9.4-9.9	7.5-8.1
Fe, ppm	< 1	< 1
Special (ratio)	2:1	2:1
Corrosion of s.s. Feed Throughs	Yes, rust surrounded fittings	Some
Screen Condition	Green-gel on screen	Rust particles
Material in Screen	Brown Scale in Screen	1/8” thick Rusticles
Brine Color/viscosity	Tan, Clear	Tan, Clear (like Tea)
Iron Mesh Color/Corrosion	Not Added	Not Added
Bottom Solids Loose/Cemented	Compacted Solids at Angle	1-2” Cemented Solid
Sludge	N/A	N/A



	L-39
Waste Type	Pyrochemical Salts
Pu Content	4.35g
Brine	Castile
Additives	.No Fe Mesh, Th, U, Np, Nd, ^{241}Am
pcH Range	7.6-8.3
Fe, ppm	< 2
Special (ratio)	2:1
Corrosion of s.s. Feed Throughs	Yes
Screen Condition	O-ring has green gel
Material in Screen	Black Scale
Brine Color/viscosity	Taupe Opaque Liquid
Iron Mesh Color/Corrosion	Not Added
Bottom Solids Loose/Cemented	Cemented Solids at Angle
Sludge	N/A

Recommended Experimentation (partial list)

- Conduct proposed Fe experiment in the WIPP; this is critical; Fe in heterogeneous waste forms.
- Determine composition of brine in the disposal rooms; Sr and Ba.
- Conduct experiments in recommended glass test container.
- Investigate precipitation of Mg and Ca and impact on chelators; MgO should mitigate effect of chelators.
- Cementation occurred in both Castile and Brine A in Pyrochemical Salt; determine limits of Mg involvement in cementation.
- Non-comminution of waste should be tested.
- Slow addition of brine to MgO experiments, rather than MgO to brine.
- Conduct Pu(IV), (V), (VI) tests in wastes that include MgO.