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**THE ACTINIDE SOURCE-TERM WASTE TEST PROGRAM (STTP)**

**Final Report**

**Prepared for the DOE-Carlsbad Field Office**

**Summer 2001**

**VOLUME IV**

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## Introduction

Volume IV of the STTP Final Report is a compilation of the Liter-scale Disassembly Observations Checklists and the Results of Observations for each test container. The D&D of the liter-scale test containers was conducted according to an established procedure, but each test container required unique attention and visual characterization that was hand-written onto a checklist. Therefore, the handwritten portion of the checklist is not in electronic form and will be in hard copy only. The Results of Observations for each test container is a summary of the major parameters for the test phase and incorporates the data obtained from the checklist.

## Liter-Scale No. 01 Results of Observations

### 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 pH 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.

### 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 pH 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<sub>2</sub> content in the headspace of the test container.

## **LITER-SCALE #1 D&D**

- 1. Construct a ratchet for LS disassembly**
- 2. Brine level 1" below the screen**
- 3. Rad reading 0 at screen top**
- 4. Rad reading < 0.1 mR at glove port**
- 5. Brine solution clear**
- 6. Very little radiation activity throughout the process**
- 7. Cement solid at the bottom of the LS container**
- 8. Portland cement ~ 3", packed but loose**
- 9. Need longer tongs for reaching into the container to remove mass**
- 10. Screen was intact – black in color at removal (FeO ?), undissolved**
- 11. Core sample taken, next time the core sample will be taken first**
- 12. Analysis will be done to determine form of Pu**
- 13. Mesh screen turned back to gray after ~ 15 minutes**
- 14. Glass vial was recovered, PN 00011126**
- 15. Need Danny to spot weld screen onto hole in lid**

**16. Add Zeolite – Stir – Add Zeolite to ¼” from top of container – Tighten lid using two bolts**

**17. Rad readings:**

**Bottom of container: 0.3 mR**

**Middle of container: 0.2 mR**

**Top of container: 0.1 mR**

**Overall Assessment:**

LS-02 had been a test container that was difficult to sample. The D&D showed that the screen was filled with Portland Cement and was the reason for the sampling difficulty. There was no corrosion on the SS feedthroughs and no visible corrosion of the Fe mesh. There was no cemented solid at the bottom of the test container.

There was essentially no solubilization of Pu or any other actinide and <1 ppm of Fe throughout the test. The pcH of 10.3 to 10.6 precipitated the Mg which increased the solids within the sludge. The Ca did not precipitate at this pcH range. There was Fe identified on the 5 micron filters on 11 of 13 filters despite the fact that there was < 1 ppm of soluble Fe throughout the test period. There was Sr and S identified on all filters. The origin of the Sr is not known.

opened door went up to 5.0% O<sub>2</sub>  
started @ 1.2% O<sub>2</sub>

Page 1 of 2

Attachment 1

25°C

### Liter Scale Disassembly Observations Checklist

The STTP Observer fills out and signs this form. The C-AAC operator signs this form. NMT-7 waste management personnel may sign this form.

Liter-Scale Container # LS-02 Date: 3-28-01  
Hazardous matrix: Portland Brine A  
Videotape recorded? yes  no If yes, record ID# of tape \_\_\_\_\_

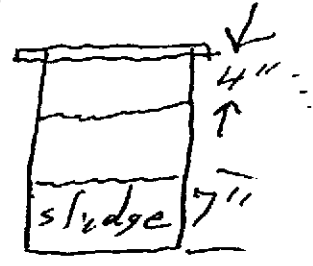
Dose rate reading of screen 0.7 mR

Lid and screen observations. (e.g. condition of screen, impaction, corrosion, etc.)  
Lid has no rust. Screen filled with solid gray Portland cement and no rust.

Note the color/turbidity (muddy, cloudy, opaque, thick) of main brine pool. Note presence of suspensions or crystals. The brine has a slight grayish tint to it. No suspensions or crystals noted.

Depth of the brine pool. \_\_\_\_\_  
Took out about 800 mls of brine.

Hardness of solids at the bottom of the container. Soft liquidy sludge



RAD (mR/hr contact) reading of the brine in the 1-liter bottles. 0 mR

Core sample taken?  
 NA

Fe mesh container recovered? If yes, describe condition of brine and solid material surrounding the container for the mesh.  
 no Soft gray liquid

Dose rate reading of container holding the mesh (mR/hr contact) 0.1 mR



### Liter Scale Disassembly Observations Checklist

Liter-Scale Container # LS-02 Date: 3-28-01

Was the mesh container embedded?

no It was easy to remove though.

After removing the mesh from the container, note:

percent dissolved 0%

amount of deposited material on the Fe mesh Zero

color of the deposited material ✓

thickness and hardness of the deposited material ✓

After cleaning the mesh with water, note the color and physical appearance of the mesh.

Mesh looks in good shape, nice and sturdy.

Other notes

Mesh is grayish looking with little orange spots, but it is intact. Fe III condition.

Waste container number for disposal of the D&D liter scale container \_\_\_\_\_.

Waste dispositioned

according to NMT-7 instructions: Ronald C. [Signature] (NMT-7) date 3/28/01

\_\_\_\_\_  
(NMT-7) date \_\_\_\_\_

Liter Scale Container disassembly complete:

[Signature] (C-AAC) date 3/28/01

[Signature] (STTP) date 3-28-01

## Liter-Scale No. 03 Results of Observations

### 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 pH 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  
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<sub>2</sub> = 7.2 v/o; O<sub>2</sub> = 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 pryed 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 pH range of 12.8-13.1 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 of this sample; 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.

**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 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 of this sample; 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.

5.28 O<sub>2</sub>  
@ 24°C  
Page 1 of 2

Attachment 1

Liter Scale Disassembly Observations Checklist

The STTP Observer fills out and signs this form. The C-AAC operator signs this form. NMT-7 waste management personnel may sign this form.

Liter-Scale Container # LS-03 Date: 4-12-01  
Haz Portland Castile Fe Mesh  
Videotape recorded? yes  no If yes, record ID# of tape \_\_\_\_\_

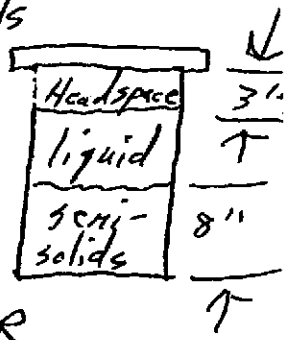
Dose rate reading of screen 0.7 mR

Lid and screen observations. (e.g. condition of screen, impaction, corrosion, etc.)  
Lid looks good, no visible rust. Screen basket not rusted, half full of Portland cement.

Note the color/turbidity (muddy, cloudy, opaque, thick) of main brine pool. Note presence of suspensions or crystals. No suspensions or crystals noted. Slight grayish tinge in the brine pool.

Depth of the brine pool. Took out ~ 800 ml then the vac line started to plug up.

Hardness of solids at the bottom of the container. Atale Gray consistency of solids at the bottom of the container.



RAD (mR/hr contact) reading of the brine in the 1-liter bottles. 0.3 mR  
~ 900 ml taken out-

Core sample taken?  
yes  NA

Fe mesh container recovered? If yes, describe condition of  
yes no brine and solid material surrounding the container for the mesh.  
Fe Mesh lodged in the container. Had to pry out. Mesh is gray colored - Material surrounding mesh is oatmeal consistency  
Dose rate reading of container holding the mesh (mR/hr contact) 0.4 mR  
Mesh is in good condition, no deterioration noted.

5.2B O<sub>2</sub>  
@ 24°C

Attachment 1

Liter Scale Disassembly Observations Checklist

The STTP Observer fills out and signs this form. The C-AAC operator signs this form. NMT-7 waste management personnel may sign this form.

Liter-Scale Container # LS-03 Date: 4-12-01  
Haz Portland Castile Fe Mesh

Videotape recorded? yes  no If yes, record ID# of tape \_\_\_\_\_

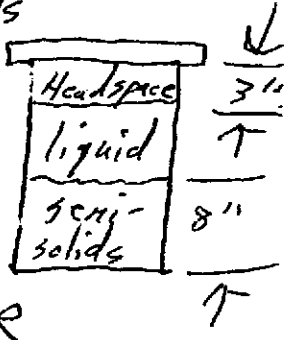
Dose rate reading of screen 0.7 mR

Lid and screen observations. (e.g. condition of screen, impaction, corrosion, etc.)  
Lid looks good, no visible rust. Screen basket not rusted, half full of Portland cement.

Note the color/turbidity (muddy, cloudy, opaque, thick) of main brine pool. Note presence of suspensions or crystals. No suspensions or crystals noted. Slight grayish tinge in the brine pool.

Depth of the brine pool. Took out ~ 800 mls then the vac line started to plug up.

Hardness of solids at the bottom of the container. At a <sup>gray</sup> consistency of solids at the bottom of the container.



RAD (mR/hr contact) reading of the brine in the 1-liter bottles. 0.3 mR  
~ 900 ml taken out-

Core sample taken?  
 NA

Fe mesh container recovered? If yes, describe condition of  
yes no brine and solid material surrounding the container for the mesh.  
Fe mesh lodged in the container. Had to pry out. Mesh is gray colored. Material surrounding mesh is orange consistency  
Dose rate reading of container holding the mesh (mR/hr contact) 0.4 mR  
Mesh is in good condition, no deterioration noted.

5.28 O<sub>2</sub>  
@ 24°C

Page 1 of 2

Attachment 1, continued

checklist

### Liter Scale Disassembly Obse

is this form. NMT-7

Liter-Scale Container # LS-03 Date: 4-12-01

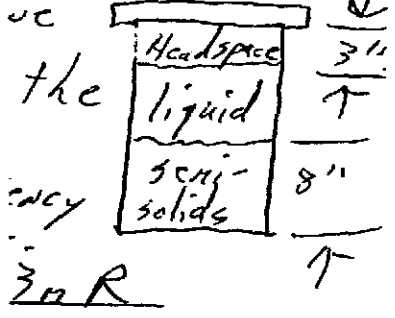
sh

Was the mesh container embedded?  
yes  no  But had to pry it <sup>10</sup>/<sub>10g</sub>

on, etc.)  
en basted not

After removing the mesh from the container, note:  
percent dissolved 0%  
amount of deposited material on the Fe mesh 0  
color of the deposited material ---  
thickness and hardness of the deposited material ---

ol. Note presence of  
ystals



After cleaning the mesh with water, note the color and p  
Grayish color with good  
No corrosion noted shiny cut  
Other notes  
No level probe in vesse  
Gross particulate in brine sample settled immediate  
Waste container number for disposal of the D&D liter s

pry out -  
mesh is orange/  
MR consistency  
ation noted

Waste dispositioned  
according to NMT-7 instructions: Seluh VNT

Liter Scale Container disassembly  
complete: [Signature]

### Liter Scale Disassembly Observations Checklist

Liter-Scale Container # LS-03 Date: 4-12-01

Was the mesh container embedded?  
yes  no  But had to pry it out since it was logged in -

After removing the mesh from the container, note:

percent dissolved 0%

amount of deposited material on the Fe mesh 0

color of the deposited material /

thickness and hardness of the deposited material /

adding 100 ml aqueous brine to 9" of brine/solids/zeo mixture to absorb remaining moisture. Plastic looking grayish aqueous brine after allowing to set for an hour

After cleaning the mesh with water, note the color and physical appearance of the mesh.

Grayish color with good physical appearance  
No corrosion noted. Shiny cut ends. Light gray wires

Other notes

No level probe in vessel.  
Gross particulate in brine sample settled immediately.

Waste container number for disposal of the D&D liter scale container \_\_\_\_\_.

Waste dispositioned according to NMT-7 instructions: [Signature] (NMT-7) date 4-12-01

\_\_\_\_\_  
(NMT-7) date \_\_\_\_\_

Liter Scale Container disassembly complete: [Signature] (C-AAC) date 4/12/01

[Signature] (STTP) date 4-12-01



## Liter-Scale No. 04 (Pressurized) Results of Observations

### 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<sub>2</sub> pressure in headspace.

### 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 pH 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:  $1 \times 10^{11}$  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<sub>2</sub>; 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 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<sub>2</sub> 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 pH did not vary due to CO<sub>2</sub> 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.

Temp 22°C  
 ↓ 5.62 O<sub>2</sub>  
 ↓ 3.58 O<sub>2</sub> after  
 Page 1 of 2 screen  
 removed

Attachment 1

Liter Scale Disassembly Observations Checklist

The STTP Observer fills out and signs this form. The C-AAC operator signs this form. NMT-7,  
 waste management personnel may sign this form.

Liter-Scale Container # LS-04 Date: 5-9-01

Haz, Portland Brine A  
 Fe mesh

Videotape recorded? yes (no) If yes, record ID# of tape \_\_\_\_\_

Dose rate reading of screen 0.14R

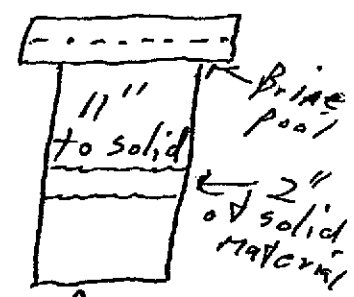
Lid and screen observations. (e.g. condition of screen, impaction, corrosion, etc.)  
 Lid nice and ~~see~~ clean, good condition. No corrosion noted. A little white mass noted around the screen.

Note the color/turbidity (muddy, cloudy, opaque, thick) of main brine pool. Note presence of suspensions or crystals. Clear, non-viscous brine. Light gray color.

Depth of the brine pool. \_\_\_\_\_  
Brine pool exactly at the top of the screen

Hardness of solids at the bottom of the container. \_\_\_\_\_  
On a scale of 1 to 10 it's an 8 and cruddy -

RAD (mR/hr contact) reading of the brine in the 1-liter bottles. 0.2 mR  
Took out 1 liter of brine



Core sample taken?  
 yes NA

3581

Fe mesh container recovered? If yes, describe condition of \_\_\_\_\_  
 yes no brine and solid material surrounding the container for the mesh.  
Compacted solids but not reacted cement

Dose rate reading of container holding the mesh (mR/hr contact) 0.2 mR

### Liter Scale Disassembly Observations Checklist

Liter-Scale Container # LS-04 Date: 5-9-01

Was the mesh container embedded?  
 no The container was full of gray compacted solids. Maybe (maybe) a slight greenish tint on entire mass within plastic container

After removing the mesh from the container, note:

percent dissolved Slight possibility  
amount of deposited material on the Fe mesh Small amount of portland cement.  
color of the deposited material Grayish green  
thickness and hardness of the deposited material N/A

After cleaning the mesh with water, note the color and physical appearance of the mesh.  
Black and white mesh

Other notes plastic vial lid looked melted.  
Aquasorb added and then zeolite also.

Waste container number for disposal of the D&D liter scale container LS.04

Waste dispositioned according to NMT-7 instructions: [Signature] (NMT-7) date 5-09-01  
\_\_\_\_\_  
(NMT-7) date \_\_\_\_\_

Liter Scale Container disassembly complete: [Signature] (C-AAC) date 5/9/01  
[Signature] (STTP) date 5-9-01

## **Liter-Scale No. 05 (Pressurized) Results of Observations**

### **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<sub>2</sub>, 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 pH 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:  $1 \times 10^{11}$  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<sub>2</sub> pressure.

### **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 pH 7.0 – 7.4 range.

**Overall Assessment:**

Liter-scale test container LS-05 was a Portland cement test with 60 Bar (870 psig) CO<sub>2</sub> 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 pH did not change significantly (pH 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.

5.2802

temp = 28°C

Attachment 1

Liter Scale Disassembly Observations Checklist  
Portland, Hazard, Brine A, Fe mesh, Pressure, Sporg sldge

The STTP Observer fills out and signs this form. The C-AAC operator signs this form. NMT-7 waste management personnel may sign this form.

Liter-Scale Container # LS-05 Date: 5-10-01

Videotape recorded? yes  no If yes, record ID# of tape \_\_\_\_\_

Dose rate reading of screen 0.1 mR

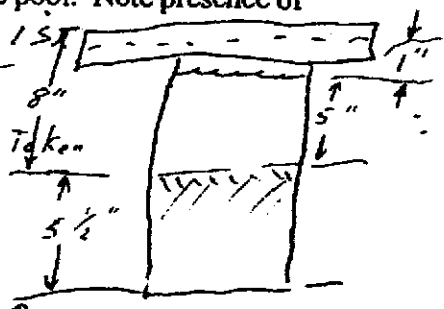
Lid and screen observations. (e.g. condition of screen, impaction, corrosion, etc.)  
Cement solids on top of screen - looks in good shape.

Note the color/turbidity (muddy, cloudy, opaque, thick) of main brine pool. Note presence of suspensions or crystals. The brine pool is clear and thin with a slight light brown tinge to it.

Depth of the brine pool. 500 ml. sample taken  
About 1/2" below the screen.

Hardness of solids at the bottom of the container.  
Yellowish-Dark brown. Compact pasty yellowish mass like unreacted wet envirostone.

RAD (mR/hr contact) reading of the brine in the 1-liter bottles. 0 mR



Core sample taken? yes NA

Fe mesh container recovered? If yes, describe condition of brine and solid material surrounding the container for the mesh.  
peanut butter texture and color and appearance.

Dose rate reading of container holding the mesh (mR/hr contact) 0.25 mR

The rinse water had a bluish tint to it after the solids ppt'd out.

### Liter Scale Disassembly Observations Checklist

Liter-Scale Container # LS-05 Date: 5-10-01

Was the mesh container embedded?

yes  no

After removing the mesh from the container, note:

percent dissolved n/a

amount of deposited material on the Fe mesh Small amt

color of the deposited material Pasty yellow mass

thickness and hardness of the deposited material not very hard, peanut buttery.

After cleaning the mesh with water, note the color and physical appearance of the mesh.

Mesh seems to have held up well

Other notes

Blackish mesh with blue tint on it. Will try aquasorb first on container remains -

Waste container number for disposal of the D&D liter scale container LS-05.

Used 100 mls of aquasorb and 300 mls of rinse water -

Waste dispositioned

according to NMT-7 instructions: [Signature] (NMT-7) date 5-10-01

\_\_\_\_\_ (NMT-7) date \_\_\_\_\_

Liter Scale Container disassembly complete:

[Signature] (C-AAC) date 5/10/01

[Signature] (STTP) date 5-10-01



## Liter-Scale No. 06 (Pressurized) Results of Observations

### 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<sub>2</sub>, 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.

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 pH 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:  $9 \times 10^{10}$  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<sub>2</sub> 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 pH 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<sub>2</sub> pressure in Castile Brine. LS-4,5, and 6 were a set of three test containers with CO<sub>2</sub> 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<sub>2</sub> pressure at 60 Bar (870 psig).

2.28 O<sub>2</sub>  
28°C  
went to 2.58 O<sub>2</sub>  
Page 1 of 2  
when door opened

Attachment 1

### Liter Scale Disassembly Observations Checklist

The STTP Observer fills out and signs this form. The C-AAC operator signs this form. NMT-7 waste management personnel may sign this form.

Liter-Scale Container # LS-06 Date: 4-24-01  
Pressurized Haz. Portland Castille Fe mesh  
Videotape recorded? yes  no If yes, record ID# of tape \_\_\_\_\_

Dose rate reading of screen 0

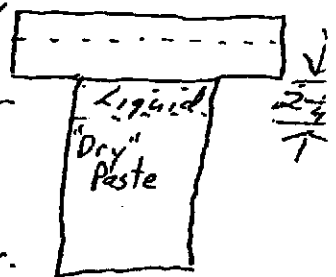
Lid and screen observations. (e.g. condition of screen, impaction, corrosion, etc.)

Lid looks clean, no corrosion or deposits noted. Screen looks clear with small ceramic shards.

Note the color/turbidity (muddy, cloudy, opaque, thick) of main brine pool. Note presence of suspensions or crystals.

The brine is a milky color. No suspensions or crystals noted. The walls of the container are clean. Depth of the brine pool. 2" deep, then

wet solids - brownish-gray.



Hardness of solids at the bottom of the container. Not soft but compacted - very dry damp paste. Like settled Cornstarch in water.

0.1 mR RAD (mR/hr contact) reading of the brine in the 1-liter bottles. ~200 ml/s of brine taken - light gray in color.

Core sample taken? yes  NA Sludge sample for Doug taken -

Fe mesh container recovered? If yes, describe condition of

no brine and solid material surrounding the container for the mesh. No brine except what was left in the very compacted clay-like cement. The cement reconstituted to wet sludge consistency after much stirring.

Dose rate reading of container holding the mesh (mR/hr contact) 0.1 mR

Metal gasket instead of O-rings in good shape -

### Liter Scale Disassembly Observations Checklist

Liter-Scale Container # LS-06 Date: 4-24-01

Was the mesh container embedded?  
 yes  no The brine couldn't flow through the container.

After removing the mesh from the container, note:

percent dissolved 0

amount of deposited material on the Fe mesh 0

color of the deposited material light gray (skin?) like all the clay would not wash off and then it dried.

thickness and hardness of the deposited material 0/NA

\* shiny cut ends!!

After cleaning the mesh with water, note the color and physical appearance of the mesh.

Clean, new, gray -> Black (original oxide coatings). Added 100 ml Aquasol to container - immediate absorption.

- used 400 ml water and added it to container.

Other notes

The compacted clay washed off easily with water but probably wouldn't have reconstituted with agitation of only 15 minutes per operating procedures.

Waste container number for disposal of the D&D liter scale container LS.06

Waste dispositioned

according to NMT-7 instructions:

S. V. King (NMT-7) date 4-24-01

(NMT-7) date \_\_\_\_\_

Liter Scale Container disassembly complete:

T. R. Halbur (C-AAC) date 4/24/01

M. V. King (STTP) date 4-24-01

## Liter-Scale No. 07 Results of Observations

### 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: Nd, 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 pH 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:  $H_2 = 1.2$  v/o;  $O_2 = 1.2$  v/o. This was the lowest  $H_2$  in STTP.

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

Corrosion: corrosion noted around SS feedthroughs; screen was clean.  
Brine: 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 ratio 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.

**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 Pu or Fe was detected on any filter paper.

No mesh  
O<sub>2</sub> not a factor

Page 1 of 2

27°C

Attachment 1

### Liter Scale Disassembly Observations Checklist

The STTP Observer fills out and signs this form. The C-AAC operator signs this form. NMT-7 waste management personnel may sign this form.

Liter-Scale Container # LS-07 Date: 3-28-01 Brine A  
Hazardous matrix: Portland  
Videotape recorded? yes  (no) If yes, record ID# of tape ✓

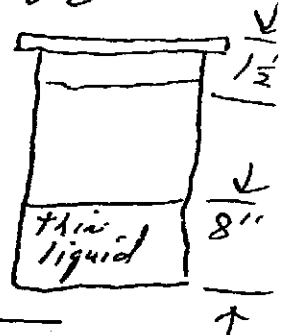
Dose rate reading of screen 0.2 mR

Lid and screen observations. (e.g. condition of screen, impaction, corrosion, etc.)  
Rust noticeable around the lid penetrations.  
Screen appears fairly clean.

Note the color/turbidity (muddy, cloudy, opaque, thick) of main brine pool. Note presence of suspensions or crystals. Colorless brine pool. No suspensions or crystals noted.

Depth of the brine pool. \_\_\_\_\_

Hardness of solids at the bottom of the container. Muddy solid  
at bottom of the container. Went into  
solution when stirred. Very watery.  
RAD (mR/hr contact) reading of the brine in the 1-liter bottles. 0.05 mR  
1 liter sample taken.



Core sample taken?  
yes  NA

Fe mesh container recovered? If yes, describe condition of  
yes  brine and solid material surrounding the container for the mesh.  
None

Dose rate reading of container holding the mesh (mR/hr contact) \_\_\_\_\_

### Liter Scale Disassembly Observations Checklist

Liter-Scale Container # LS-07 Date: 3-28-01

Was the mesh container embedded?  
yes no

N/A

After removing the mesh from the container, note:

- percent dissolved \_\_\_\_\_
- amount of deposited material on the Fe mesh \_\_\_\_\_
- color of the deposited material \_\_\_\_\_
- thickness and hardness of the deposited material \_\_\_\_\_

After cleaning the mesh with water, note the color and physical appearance of the mesh.

Other notes

Brine is very watery. We're worried about absorbing all the liquid. Added ~ 500 ml of aquasorb.

Waste container number for disposal of the D&D liter scale container

Aquasorb-2212 fully absorbed liquid. Absorbed to rubbery consistency. could have used 2 the amount.

Waste dispositioned

according to NMT-7 instructions:

Sherette Ortega (NMT-7) date 3/29/01

\_\_\_\_\_  
(NMT-7) date \_\_\_\_\_

Liter Scale Container disassembly complete:

Holly Paul (C-AAC) date 3/29/01

Paul Johnson (STTP) date 3-29-01



## Liter-Scale No. 08 Results of Observations

### 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 (2:1 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 than 1 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 pH 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.

### 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 pH 9.1– 9.5 in a Brine A environment. Most of the Mg precipitated at the pH 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.

1-88 O<sub>2</sub>

27°C

Attachment 1

Liter Scale Disassembly Observations Checklist

The STTP Observer fills out and signs this form. The C-AAC operator signs this form. NMT-7 waste management personnel may sign this form.

Liter-Scale Container # 15-08 Date: 4-3-01

Portland Videotape recorded? yes  no If yes, record ID# of tape Hazardous / Brine A

Dose rate reading of screen 0.7 m R

Lid and screen observations. (e.g. condition of screen, impaction, corrosion, etc.)  
Screen in good condition. Lid in good condition. A little sediment visible in screen.

Note the color/turbidity (muddy, cloudy, opaque, thick) of main brine pool. Note presence of suspensions or crystals. Brine is grayish looking. No suspensions or crystals noticed. ~ 1 liter pulled.

Depth of the brine pool. \_\_\_\_\_

Hardness of solids at the bottom of the container. \_\_\_\_\_  
~ 4 inches of solids at the bottom of the cont. Pretty hard stuff.

RAD (mR/hr contact) reading of the brine in the 1-liter bottles. 0 m R  
Another 4 inches of solids after the first 4 inches.

Core sample taken?  NA  
Fe mesh container recovered? If yes, describe condition of \_\_\_\_\_  
yes  no \_\_\_\_\_  
brine and solid material surrounding the container for the mesh.

Dose rate reading of container holding the mesh (mR/hr contact) 0 m R

### Liter Scale Disassembly Observations Checklist

Liter-Scale Container # LS-08 Date: 4-3-01

Was the mesh container embedded?  
yes no

After removing the mesh from the container, note:

percent dissolved \_\_\_\_\_

amount of deposited material on the Fe-mesh \_\_\_\_\_

color of the deposited material \_\_\_\_\_

thickness and hardness of the deposited material \_\_\_\_\_

After cleaning the mesh with water, note the color and physical appearance of the mesh.

Other notes

Waste container number for disposal of the D&D liter scale container LS-08

Waste dispositioned

according to NMT-7 instructions: Sebastian V. Martin (NMT-7) date 04/03/01

\_\_\_\_\_  
(NMT-7) date \_\_\_\_\_

Liter Scale Container disassembly  
complete:

Adrian Paul (C-AAC) date 4/3/01

Bob F. West (STTP) date 4/3/01

## Liter-Scale No. 09 Results of Observations

### 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 pH Range: 12.9 – 13.1  
Fe Concentrations: <1 ppm for the entire test period.  
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<sub>2</sub> = 3.4 v/o, O<sub>2</sub> = 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 pH (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 pH. 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<sub>2</sub> concentration in the headspace was relatively low (3.4 v/o) and the O<sub>2</sub> was 0.17 v/o.

5.8% O<sub>2</sub>

28°C

Attachment 1

Liter Scale Disassembly Observations Checklist

The STTP Observer fills out and signs this form. The C-AAC operator signs this form. NMT-7 waste management personnel may sign this form.

Haz Portland Castille no Fe mesh  
Liter-Scale Container # LS-09 Date: 4-19-01

Videotape recorded? yes  no If yes, record ID# of tape \_\_\_\_\_

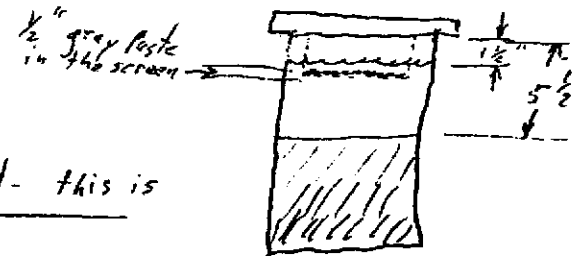
Dose rate reading of screen 0.4 mR/hr contact

Lid and screen observations. (e.g. condition of screen, impaction, corrosion, etc.)  
Lid uncorroded - no coloration. Gray paste stuck to lid. Slightly darkened screen.

Note the color/turbidity (muddy, cloudy, opaque, thick) of main brine pool. Note presence of suspensions or crystals.

Brine is clear except for powdered Portland Cement suspended in it (partly settled)

Depth of the brine pool. 5 1/2"



Hardness of solids at the bottom of the container. Soft - this is only settled solids, not reacted cement.

RAD (mR/hr contact) reading of the brine in the 1-liter bottles. 0.2  
850 ml sample taken

Core sample taken?  
yes  NA

Fe mesh container recovered? If yes, describe condition of  
yes no brine and solid material surrounding the container for the mesh. NA

Dose rate reading of container holding the mesh (mR/hr contact) NA

### Liter Scale Disassembly Observations Checklist

Liter-Scale Container # LS-09 Date: 4-19-01

Was the mesh container embedded?  
yes no NA

After removing the mesh from the container, note:

percent dissolved \_\_\_\_\_ NA

amount of deposited material on the Fe mesh \_\_\_\_\_ NA

color of the deposited material \_\_\_\_\_ NA

thickness and hardness of the deposited material \_\_\_\_\_ NA

After cleaning the mesh with water, note the color and physical appearance of the mesh.  
NA

Other notes  
Lid and level probe were absolutely pristine.

Waste container number for disposal of the D&D liter scale container LS-09.

Waste dispositioned according to NMT-7 instructions: [Signature] (NMT-7) date 4/19/01

\_\_\_\_\_ (NMT-7) date \_\_\_\_\_

Liter Scale Container disassembly complete: T.R. Helm (C-AAC) date 4/19/01

[Signature] (STTP) date 4-19-01



## Liter-Scale No. 10 Results of Observations

### 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: <sup>241</sup>Am (75 mg) added as soluble salt

### 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 pH Range: 8.2 to 8.9

Fe Concentration: There was no Fe mesh added to this test container and Fe was typically less than 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<sup>9</sup> to 10<sup>10</sup> 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<sub>2</sub> Headspace Gas Content: 32.5 v/o H<sub>2</sub>, 1.5 v/o O<sub>2</sub>

**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 pH 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. The presence of the precipitated Am-241 was evident from the H<sub>2</sub> concentration in the headspace in this test container (~ 32.5 v/o H<sub>2</sub>) which was higher than similar Portland cement tests w/o Am-241 (typically < 5% H<sub>2</sub>). 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 <sup>241</sup>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.

Attachment 1

O<sub>2</sub> not a concern  
26°C

### Liter Scale Disassembly Observations Checklist

The STTP Observer fills out and signs this form. The C-AAC operator signs this form. NMT-7 waste management personnel may sign this form.

Liter-Scale Container # LS-10 Date: 2-5-01

Videotape recorded? yes  no If yes, record ID# of tape \_\_\_\_\_

Dose rate reading of screen 7 mR/hr

Lid and screen observations. (e.g. condition of screen, impaction, corrosion, etc.)

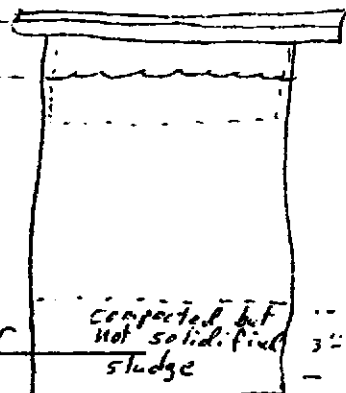
*Lid - original metal mostly but rusticles around sampling port, level probe and gauge po.  
small amount of screen impaction near ports but nowhere else  
at Rust & sludge in screen. sides of vessel are darkened & coated w/ "Rust"*

Note the color/turbidity (muddy, cloudy, opaque, thick) of main brine pool. Note presence of suspensions or crystals. *Brine is dark & cloudy but thin. Clear 2-5-01*

Depth of the brine pool. \_\_\_\_\_

Hardness of solids at the bottom of the container. Soft

RAD (mR/hr contact) reading of the brine in the 1-liter bottles. 0.7 mR/hr



Core sample taken?  NA Retrieved

Fe mesh container recovered? If yes, describe condition of brine and solid material surrounding the container for the mesh.  
yes no

*second 1/2 liter read 4 mR  
It had more particulate.*

Dose rate reading of container holding the mesh (mR/hr contact) NA

### Liter Scale Disassembly Observations Checklist

Liter-Scale Container # LS-10 Date: 2-5-01

Was the mesh container embedded?

yes no

NA

After removing the mesh from the container, note:

percent dissolved \_\_\_\_\_

amount of deposited material on the Fe mesh \_\_\_\_\_

color of the deposited material \_\_\_\_\_

thickness and hardness of the deposited material \_\_\_\_\_

After cleaning the mesh with water, note the color and physical appearance of the mesh.

Other notes

*we pumped more than 1-liter of brine out because the zeolite did not appear to absorb the brine sufficiently. The extra brine will go to 9010A for future disposition. We collected the level probe as a metal sample.*

Waste container number for disposal of the D&D liter scale container \_\_\_\_\_

Waste dispositioned

according to NMT-7 instructions

[Signature] (NMT-7) date 2/5/01

(NMT-7) date \_\_\_\_\_

Liter Scale Container disassembly complete:

[Signature] (C-AAC) date 2-5-01

[Signature] (STTP) date 2-5-01

## Liter-Scale No. 11 Results of Observations

### 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: <sup>241</sup>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 was observed.
- Th - Concentrations were less than 1 ppb with no visible trend.

### Other Analyses (Nominal)

Typical pH 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<sup>9</sup> to 10<sup>10</sup> particles/L

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<sub>2</sub> Headspace Gas Content: 22% H<sub>2</sub>

### 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 grey 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 p<sub>H</sub> 9.0 to 10.8 that had very low (< 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 <sup>241</sup>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<sup>9</sup> to 10<sup>10</sup> 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<sub>2</sub> 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)<sub>2</sub> 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 p<sub>H</sub> (9.0 – 10.8 ) and high sludge content led to a test with essentially no soluble actinides.