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THE ACTINIDE SOURCE-TERM WASTE TEST PROGRAM (STTP)

Final Report

Prepared for the DOE-Carlsbad Field Office

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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: H₂ = 0.8 v/o; O₂ = 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 H₂ 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% O₂
started @ 1.2% O₂

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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.)

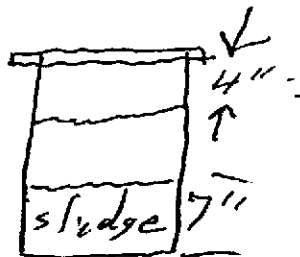
Lid has no rust. Screen filled with solid gray Portland cement and no rust.

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?
 yes NA

Fe mesh container recovered? If yes, describe condition of
 yes no brine and solid material surrounding the container for the mesh.
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: H₂ = 7.2 v/o; O₂ = 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 O₂
@ 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 _____

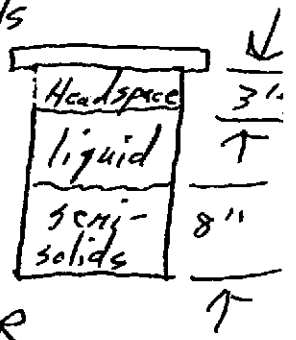
Dose rate reading of screen 0.7 mR

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 O₂
@ 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 _____

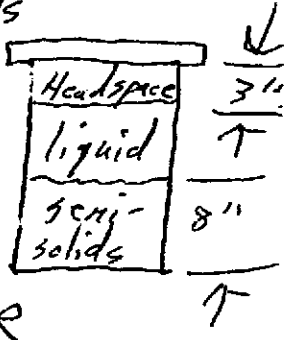
Dose rate reading of screen 0.7 mR

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 mls then the vac line started to plug up.

Hardness of solids at the bottom of the container. Atole 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 oatmeal consistency
Dose rate reading of container holding the mesh (mR/hr contact) 0.4 mR
Mesh is in good condition, no deterioration noted.

5.28 O₂
@ 24°C

Page 1 of 2

Attachment 1, continued

checklist

Liter Scale Disassembly Obse

is this form. NMT-7

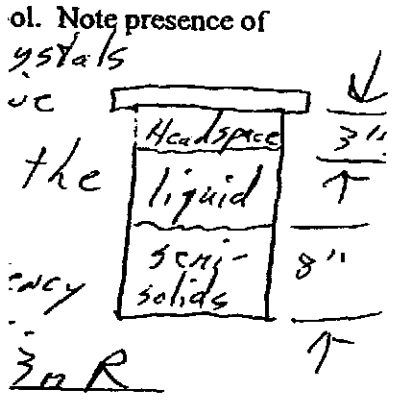
Liter-Scale Container # LS-03 Date: 4-12-01

sh

Was the mesh container embedded?
yes no But had to pry it ¹⁰/_{10g}

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 ---



After cleaning the mesh with water, note the color and p
Grayish color with good
No corrosion noted shiny cutl
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 aqueousorb to 9" of brine/solids/zeo mixture to absorb remain moisture.
Plastic locking grayish aqueousorb 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) CO₂ 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: 1×10^{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 CO₂; 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) CO₂ 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 CO₂ 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 O₂
↓ 3.58 O₂ 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 0.1 mR

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

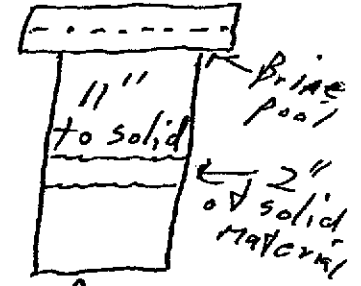
Lid nice and ~~sepe~~ clean, good condition. No corrosion noted. A little white mass noted around the screen.

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 crusty -

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? NA

Fe mesh container recovered? If yes, describe condition of _____
 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) CO₂, 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: 1×10^{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 CO₂ 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) CO₂ 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

Page 1 of 2

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

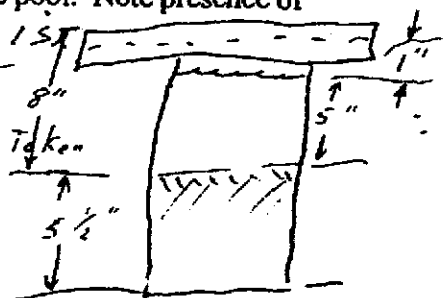
Lid and screen observations. (e.g. condition of screen, impaction, corrosion, etc.)
Cement solids on top of screen - looks in good shape.

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?
 NA

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

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) CO₂, 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: 9×10^{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 CO₂ 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) CO₂ pressure in Castile Brine. LS-4,5, and 6 were a set of three test containers with CO₂ 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 CO₂ pressure at 60 Bar (870 psig).

2.28 O₂
 28°C
 went to 2.58 O₂
 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

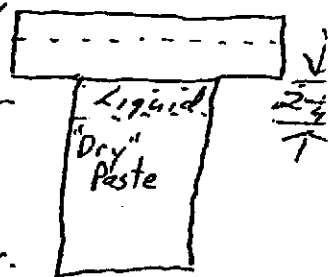
Lid and screen observations. (e.g. condition of screen, impaction, corrosion, etc.)

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 Corn starch 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

yes 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
O₂ 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 If yes, record ID# of tape ✓

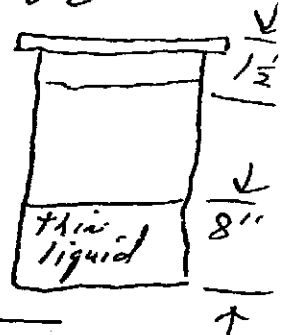
Dose rate reading of screen 0.2 mR

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 James (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 O₂

Attachment 1

27°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 # 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?

yes 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: [Signature] (C-AAC) date 4/3/01
[Signature] (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: H₂ = 3.4 v/o, O₂ = 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 H₂ concentration in the headspace was relatively low (3.4 v/o) and the O₂ was 0.17 v/o.

5.8% O₂

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 _____

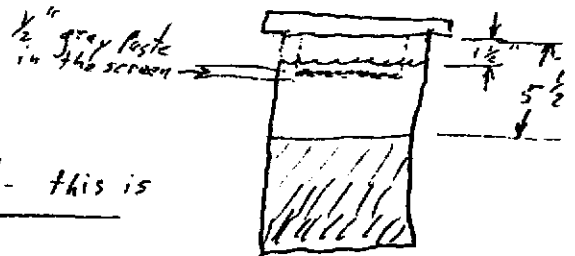
Dose rate reading of screen 0.4 mR/hr contact

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: ²⁴¹Am (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: 10⁹ to 10¹⁰ 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.

H₂ Headspace Gas Content: 32.5 v/o H₂, 1.5 v/o O₂

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 H₂ concentration in the headspace in this test container (~ 32.5 v/o H₂) which was higher than similar Portland cement tests w/o Am-241 (typically < 5% H₂). 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 ²⁴¹Am 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

*O₂ 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.)

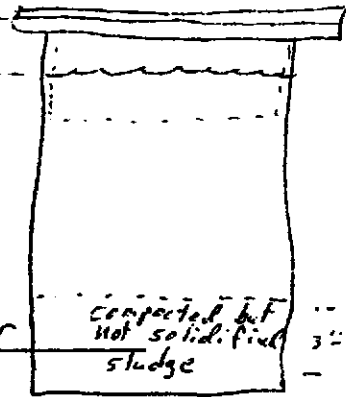
*Lid - original metal mostly but rusticles around scrubbing port, level probe and gauge po.
small amount of screen impaction near ports but nowhere else
at Rust & sludge in screen. sides of vessel are darkened & coated w/ "Rust"*

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? yes NA Retrieved

Fe mesh container recovered? If yes, describe condition of brine and solid material surrounding the container for the mesh.

*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: ²⁴¹Am (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: 10⁹ to 10¹⁰ 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.

H₂ Headspace Gas Content: 22% H₂

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 pch 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 ²⁴¹Am 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, 10⁹ to 10¹⁰ 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 H₂ 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.

O₂ not a concern
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-11 Date: 2-6-01

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

Dose rate reading of screen 1.4 mR

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

Lid - original metal. Rust around sampling port, level probe & gauge part. Rust around top of vessel also. Rust near top of screen ~ 20%

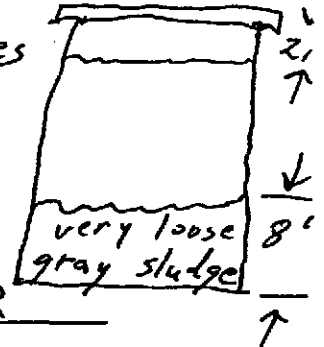
Note the color/turbidity (muddy, cloudy, opaque, thick) of main brine pool. Note presence of suspensions or crystals. Brine is grayish.

Sediment on screen with black particles of rust from the lid.

Depth of the brine pool. _____

Hardness of solids at the bottom of the container. ~ 1 inch solids

Stirred cement like at bottom - into the liquid layer 0.3 mR



Core sample taken? yes Retrieved level probe for surface analysis

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

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

Liter Scale Disassembly Observations Checklist

Liter-Scale Container # LS-11 Date: 2-6-01

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

Pumped out ~ 1 liter of brine.

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

Waste dispositioned according to NMT-7 instructions:

[Signature] (NMT-7) date 2/6/01

(NMT-7) date _____

Liter Scale Container disassembly complete:

[Signature] (C-AAC) date 2/6/01
[Signature] (STTP) date 2-6-01

Liter-Scale No. 12 Results of Observations

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
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 pH 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 H₂ - 1.2 v/o O₂,
<<1% N₂O

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 ²⁴¹Am added as a soluble chloride complex for a theoretical concentration of 37,500 ppb if the Am remained soluble. The pH 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 H₂ 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 that the ²⁴¹Am produced this effect. The high pH 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 10⁹ to 10¹⁰ particles per liter in each test.

O₂ Not important

Liter Scale Disassembly Observations Checklist 24°C

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-12 ~~LS-33~~ Date: 1-31-00

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.)
* → Black coating on lid & O-ring (Hard - doesn't scratch screen driver)
Screen black but not impacted.
Thin coatings. - will retrieve level probe

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

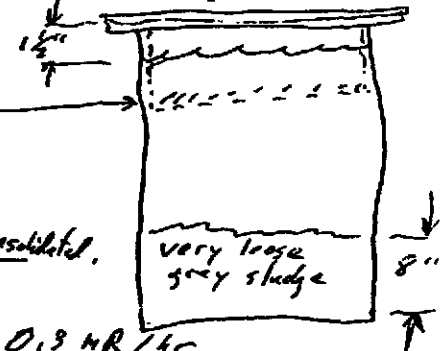
pale gray/green, quite clear (no sediment)

Depth of the brine pool. _____

Sludge — 1/4"

Hardness of solids at the bottom of the container. Soft - unconsolidated.

RAD (mR/hr contact) reading of the brine in the 1-liter bottles. 0.3 mR/hr



Core sample taken? — Retrieval Level Probe as a sample for surface analysis - what is Black coating?
yes N/A

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

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

Liter Scale Disassembly Observations Checklist

Liter-Scale Container # L5-12 Date: 1-31-01

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 - we removed only 1 liter of brine and absorbed the rest/200
Black coating on lid.

one rusticle the size & shape of a chocolate chip on the
sampling fitting.

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

Waste dispositioned

according to NMT-7 instructions:

[Signature] (NMT-7) date 1/31/01

(NMT-7) date _____

Liter Scale Container disassembly
complete:

[Signature] (C-AAC) date 1/31/01
[Signature] (STTP) date 1-31-01

Liter-Scale No. 13 Results of Observations

Test Characteristics

Waste: Envirostone
80-90 % CaSO₄ with 10-20 % melamine-formaldehyde and
0.1% NH₄Cl
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 pH 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: 10¹² to 10¹³ particles/Liter
Filter Paper-WDXRF: Fourteen of 17 filters contained Pu; all filters had Fe; only 2 had Sr. This was a high Pu bearing colloids or microprecipitates. The Fe in the filters was consistent with the soluble Fe throughout the test.
Headspace Gas Content: H₂ = 56.6 v/o; O₂ = 0.20 v/o; CO₂ = 4.4 v/o; TOC = 3300

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 pH 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 O₂ content (0.20 v/o), and measurable CO₂ (4.4 v/o). Of note is that soluble Fe, Pu, and U were correlative in this test.

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.

28°C 2% O₂

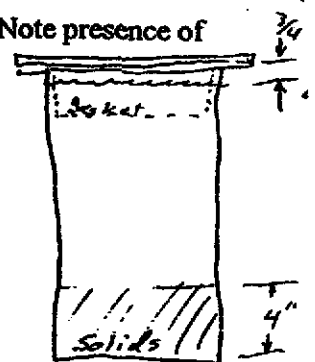
Liter-Scale Container # LS-13 Date: 1-22-01

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

Dose rate reading of screen 0.4 mR/hr

Lid and screen observations. (e.g. condition of screen, impaction, corrosion, etc.)
Lid & screen uncorroded & clean. A small amount of floating material adhered to each.

Note the color/turbidity (muddy, cloudy, opaque, thick) of main brine pool. Note presence of suspensions or crystals. Yellowish, pasty goo in screen, it is a thin layer of (flocculated clay?) floating atop the thin "clear" brine



Depth of the brine pool. 9" above solids

Hardness of solids at the bottom of the container. Soft.
Like peanut butter w/ oil separated. Hardest at bottom but the entire mass can be reconstituted.

RAD (mR/hr contact) reading of the brine in the 1-liter bottles. 0.0 mR/hr

Core sample taken?
 NA

Fe mesh container recovered? If yes, describe condition of no brine and solid material surrounding the container for the mesh.
Solid material looks like wet powdered ongirostone except for small amount of green color close to mesh. All solids washed away easily.
Dose rate reading of container holding the mesh (mR/hr contact) 0.2 mR/hr

Liter Scale Disassembly Observations Checklist

Liter-Scale Container # LS-13 Date: 1-22-01

Was the mesh container embedded?

yes no

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 Greenish black - Greenish yellow sediment washed easily off screen

thickness and hardness of the deposited material 0 / hard

After cleaning the mesh with water, note the color and physical appearance of the mesh.
Original Black Paline and some cut ends of mesh are still SHINY.

Other notes

Waste container number for disposal of the D&D liter scale container 5708T

Waste dispositioned according to NMT-7 instructions:

[Signature] (NMT-7) date 1/22/01
____ (NMT-7) date _____

Liter Scale Container disassembly complete:

[Signature] (C-AAC) date 1/22/01
[Signature] (STTP) date 1-22-01

Liter-Scale No. 14 Results of Observations

Test Characteristics

Waste: Envirostone
80-90% CaSO₄ with 10-20% melamine-formaldehyde and
0.1% NH₄Cl
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

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 pH 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: 3×10^{13} to 8×10^{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: H₂ 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, powdered 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 pH 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. 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 pH 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 3×10^{13} to 8×10^{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 H₂ concentration in the headspace of the test container but this was lower than LS-13 with a TOC of ~ 4000 ppm with a H₂ concentration of ~ 57 v/o.

Attachment I

Liter Scale Disassembly Observations Checklist

2.1% O₂ 32°C

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-14 Date: 1-18-01

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

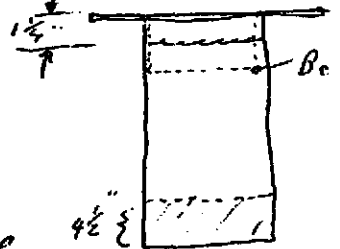
Dose rate reading of screen 0.1 mR/hr PX → 0.6 mR/hr

Lid and screen observations. (e.g. condition of screen, impaction, corrosion, etc.)
Screen & lid free of corrosion. Screen removed easily. Lid and Container looked new. No fitting degradation at all!

Note the color/turbidity (muddy, cloudy, opaque, thick) of main brine pool. Note presence of suspensions or crystals. *Yellowish (Baby shit) goo-light floaties (floculent) on top of "Clear" Brine. Non viscous liquid.*
1.5 liters of Brine extracted

Depth of the brine pool. _____

Hardness of solids at the bottom of the container.
Like peanut butter with the oil separated and floating on top.



RAD (mR/hr contact) reading of the brine in the 1-liter bottles. 0.8 mR/hr

Core sample taken?
 NA

Fe mesh container recovered? If yes, describe condition of
 no brine and solid material surrounding the container for the mesh.
Solids were wet powdered enivirostone. Mesh was original black color with original SHINY cut ends. No extra corrosion.

Dose rate reading of container holding the mesh (mR/hr contact) 0.7

Liter Scale Disassembly Observations Checklist

Liter-Scale Container # LS-14 Date: _____

Was the mesh container embedded?
yes no

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 ✓

After cleaning the mesh with water, note the color and physical appearance of the mesh.
Original black w/ original SHINY cut ends

Other notes

Waste container number for disposal of the D&D liter scale container 57089

Waste dispositioned according to NMT-7 instructions: [Signature] (NMT-7) date 4/8/01

(NMT-7) date _____

Liter Scale Container disassembly complete: JR Halun (C-AAC) date 1/18/01
[Signature] (STTP) date 1-18-01

Liter-Scale No. 15 Results of Observations

Test Characteristics

Waste: Envirostone
80-90% CaSO₄ with 10-20% melamine-formaldehyde and
0.1% NH₄Cl

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 pH 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
Mg ~1,000 ppm
Na ~88,000 ppm
Pb ~4.5 ppm (final analyses)

TIC/TOC: 40/1,400 ppm

Particle Concentration: 10¹² to 10¹³ 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; O₂ = 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 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 H₂ in the headspace was low at 3.0 V/o; LS-13 had a H₂ level of ~57 v/o and LS-14 had a H₂ concentration of 37 v/o. There was no corrosion observed on the SS feedthroughs and the Fe mesh did not have observable corrosion.

1.88 O₂
29°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.

Non-Haz
Liter-Scale Container # LS-15 Date: 4-19-01 Environment Castille Fe mesh

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

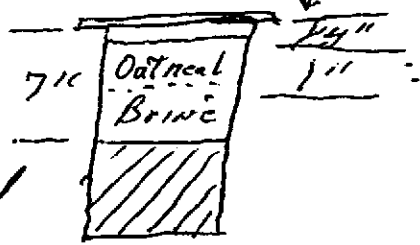
Dose rate reading of screen 0.3 mR

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

Lid has a cream color to it, but it seems to be in good condition. Pasty texture. The screen seems to be in good condition with about 2" of oatmeal solids. Note the color/turbidity (muddy, cloudy, opaque, thick) of main brine pool. Note presence of suspensions or crystals. Brine is a cream color.

Oatmeal suspension throughout the vessel.

Depth of the brine pool. 7"



Hardness of solids at the bottom of the container. Not hard at all.

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

Took out ~950 mls of brine.

Core sample taken? yes NA

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

Semi-solid soft material

Dose rate reading of container holding the mesh (mR/hr contact) 0.15 mR

Liter Scale Disassembly Observations Checklist

Liter-Scale Container # LS-15 Date: 4-19-01

Was the mesh container embedded?

yes no

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 NA

thickness and hardness of the deposited material NA

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

Black and gray mesh that seems to be in good condition.

Other notes

Added aquasorb since the zeolite didn't absorb all the solution.

Waste container number for disposal of the D&D liter scale container LS-15.

Waste dispositioned

according to NMT-7 instructions: John V. May (NMT-7) date 4/19/01

_____ (NMT-7) date _____

Liter Scale Container disassembly complete:

FR Behm (C-AAC) date 4/19/01

Wh-R-V (STTP) date 4-19-01

Liter-Scale No. 16 Results of Observations

Test Characteristics

Waste: Envirostone
80-90% CaSO₄ with 10-20% melamine-formaldehyde and
0.1% NH₄Cl
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 pH 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: 10⁹ to 10¹⁰ particles/Liter
Filter Paper-WDXRF: Pu, Fe, and SrSO₄ identified as colloids or microprecipitates
Headspace Gas content: ~62 v/o; which is relatively high for Envirostone; N₂O = 2.5 v/o. NO₃ 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.

Liter-Scale No. 16 Results of Observations

Test Characteristics

Waste: Envirostone
80-90% CaSO₄ with 10-20% melanine-formaldehyde and 0.1% NH₄Cl
Total Waste Weight: 1,315 g
Initial Actinide Content: Pu 470.0 µg/g; Total Pu = 0.618 g
Am 0.25 µg/g; Total Am = 0.329 mg
Brine: Brine A (2:1 brine/solid ratio)
Additives: Fe Mesh; Th, U, Np, and Nd

Soluble Actinide Histories:

- 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 pH 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: 10⁹ to 10¹⁰ particles/Liter
Filter Paper-WDXRF: Pu, Fe, and SrSO₄ identified as colloids or microprecipitates
Headspace Gas content: ~62%, which is relatively high for Envirostone H₂
<3% N₂O

- 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.
- 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 in tact. 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 pH (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 pH 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 N_2O 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.

Attachment I

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-16 Date: 12/19/00

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

Container: Rad level 0.3 mR/hr.
ON inner lid NO CORROSION - very few crystals
Dose rate reading of screen < 0.3

" " 0.1 mR/hr. at face of glove box
Lid and screen observations. (e.g. condition of screen, impaction, corrosion, etc.)

→ NO Lead ON Bottom ~~NO LEAD~~
Lead ON outside Lid clean

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

Tan color - finely divided suspensions.
cloudy ← (above screen)
Screen like new - off easy - NO CORROSION
Depth of the brine pool. _____
brine thick, may have to scoop out.

Hardness of solids at the bottom of the container. very solid tan to brown - plugging apparatus - must scoop out as much as possible.

RAD (mR/hr contact) reading of the brine in the 1-liter bottles. 0.3 mR/hr

Core sample taken? NO

NA

Fe mesh container recovered? If yes, describe condition of brine and solid material surrounding the container for the mesh.

VERY thick

Dose rate reading of container holding the mesh (mR/hr contact) 0.7
2" away

[Found vial with SNM]

Liter Scale Disassembly Observations Checklist

Liter-Scale Container # _____ Date: _____

Was the mesh container embedded?
 no IN SLUSH

After removing the mesh from the container, note:
percent dissolved totally intact appearance
amount of deposited material on the Fe mesh totally intact
color of the deposited material black

thickness and hardness of the deposited material thick/hard
screen intact greenish/black color -
only inside the container
After cleaning the mesh with water, note the color and physical appearance of the mesh.
Totally intact - black

Other notes
Screen although black - not brittle - retained spri
could not rub off black material, quite embedd

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

Waste dispositioned according to NMT-7 instructions: _____ (NMT-7) date _____

_____ (NMT-7) date _____

Liter Scale Container disassembly complete: _____ (C-AAC) date _____

_____ (STTP) date _____

Step	Action	Check
14	Remove septum holder assembly and replace with suitable plug. Monitor for contamination and clean if required.	
15	Remove annular space purge fitting and cap or plug as required. Monitor and clean if required.	
16	Remove thermocouple.	
17	Place lab jack under test container clamp.	
18	Loosen and remove test container clamp.	
19	Monitor for loose contamination and clean as required.	
20	Place test container in plastic bag, tape shut and mark appropriately. Monitor outside of bag for contamination. If any is found, double bag and check again as above.	
21	Place bagged test container in an insulated transport container in the upright position.	
22	After standard RCT check and release, transport container to wing 5.	
23	Turn over test container to appropriate NMT-1 personnel.	

Record the following:

Date: 12-18-00

Liter Scale Container #: LS-16

Time Taken Off Line: 1500

Individuals Involved: David Yeaman, Walt Sandoval

Liter-Scale No. 17
Results of Observations

Test Characteristics

Waste: Envirostone
80 - 90 % CaSO₄ with 10-20 % melamine- formaldehyde
and 0.1 % ammonium chloride.
~~XXXXXXXXXXXXXXXXXXXX~~
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.
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 pH 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: 10⁹ to 10¹⁰ particles/Liter
Filter Paper-WDXRF: Pu precipitates were found, which appear to be associated
with Fe and Ca.
H₂ Headspace Gas Content: ~7.7 v/o H₂; ~20 v/o N₂O; NO₃ 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 pH 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 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 N₂O concentration of ~ 20 v/o in the headspace of the test container. The H₂ 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 production of N₂O was probably from solid or precipitated species of Pu and Am. The activity of ³nCi is about 110 d/s is probably too low to generate 20 v/o of N₂O 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 N₂O. The Fe wire was not corroded in this test.

O₂ = 3.8
21°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-17 Date: 2-13-01

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

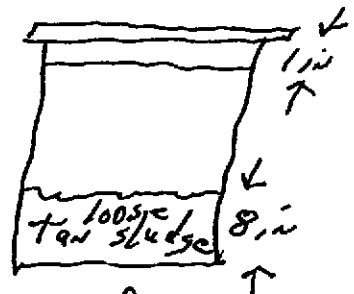
Dose rate reading of screen 0.4 nR

Lid and screen observations. (e.g. condition of screen, impaction, corrosion, etc.)
Lid - original metal. No rust, fairly clean, good condition
No sediment on screen.

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

Depth of the brine pool. _____

Hardness of solids at the bottom of the container. Soft and mushy



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

Core sample taken?
 yes NA

Fe mesh container recovered? If yes, describe condition of
 no brine and solid material surrounding the container for the mesh.
Good condition - not much solid material.

Dose rate reading of container holding the mesh (mR/hr contact) 0.3 nR

Liter Scale Disassembly Observations Checklist

Liter-Scale Container # LS-17 Date: 2-13-01

Was the mesh container embedded?
 yes no But only slightly -

After removing the mesh from the container, note:

percent dissolved 0

amount of deposited material on the Fe mesh Small amount only

color of the deposited material Tan

thickness and hardness of the deposited material Soft

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

Black and no shiny edges apparent.

Other notes

Pumped out ~1 liter of brine.

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

Waste dispositioned

according to NMT-7 instructions:

[Signature] (NMT-7) date 2/13/01

(NMT-7) date _____

Liter Scale Container disassembly complete:

[Signature] (C-AAC) date 2/13/01

[Signature] (STTP) date 2/13/01

Liter-Scale No. 18 Results of Observations

Test Characteristics

Waste: Envirostone, 80 – 90% CaSO₄ with 10 – 20% melamine formaldehyde and 0.1% ammonium chloride

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

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 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 pH 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: 10⁹ to 10¹⁰ 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 H₂; 1-2 v/o N₂O; NO₃ was ~ 100 ppm.

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 pH 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 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 near colorless and the crystals throughout the test container gave a color like butterscotch in this 2:1 brine/solid ratio test. There did not appear to be corrosion of the lid or feedthroughs.

Attachment 1

32.2 °C

2.7% O₂

Liter Scale Disassembly Observations Checklist

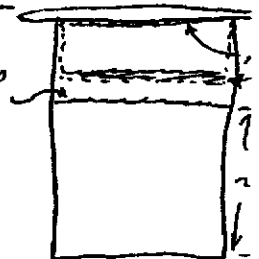
The STTP Observer fills out and signs this form. The C-AAC operator signs this form. NMT-7 waste management personnel may sign this form.

Liter-Scale Container # LS-18 Date: 1-9-00

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

Dose rate reading of screen 1 mR/hr P8

1/8" From top of brine



Lid and screen observations, (e.g. condition of screen, impaction, corrosion, etc.)
other-colored (butterscotch) mud solid in screen and stuck to lid.

Note the color/turbidity (muddy, cloudy, opaque, thick) of main brine pool. Note presence of suspensions or crystals. *Muddy liquid - much suspended clay-like material. Clear brine but looks yellowish due to suspended mat'l.*

Depth of the brine pool. ~ 9"

Hardness of solids at the bottom of the container. Soft / None

RAD (mR/hr contact) reading of the brine in the 1-liter bottles. 7 mR/hr P8

Core sample taken?
 NA

Fe mesh container recovered? If yes, describe condition of brine and solid material surrounding the container for the mesh.
 no *container full of Black Clay surrounding the mesh - washes off easily*

Dose rate reading of container holding the mesh (mR/hr contact) 1.3 mR/hr P8

Liter Scale Disassembly Observations Checklist

Liter-Scale Container # LS-18 Date: 1-9-01

Was the mesh container embedded? No
yes no

After removing the mesh from the container, note:

percent dissolved 10%

amount of deposited material on the Fe mesh ~ 2 grams

color of the deposited material Black -

thickness and hardness of the deposited material Can be scraped off But it's hard when thin. Thicker masses can be broken up w/ abrasion.

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

Black - Strong - solid - basically uncompromised.

Other notes

we were unable to pump about half the residual sludge so we absorbed. the "Brine" sample was only 500 ml.

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

Waste dispositioned

according to NMT-7 instructions: Paul Chris (NMT-7) date 1/9/01

(NMT-7) date _____

Liter Scale Container disassembly complete:

JRH (C-AAC) date 1/9/01
David J... (STTP) date 1-9-01

Liter-Scale No. 19 Results of Observations

Test Characteristics

Waste: Envirostone 80–90% CaSO₄ 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.

Other Analyses (nominal):

Typical pH 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: 10⁹ to 10¹⁰ 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 H₂; 32 v/o N₂O; 36,000 ppm NO₃

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, which is typical for Envirostone tests. This test had a high concentration of nitrites (36,000 ppm) and a resulting high concentration of N₂O (32 v/o) that precluded a high concentration of H₂ (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.

Attachment 1

3.6% O₂
27.7°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-19 Date: 2-14-01

Videotape recorded? yes 10 If yes, record ID# of tape _____

Dose rate reading of screen 0.6 mR/hr

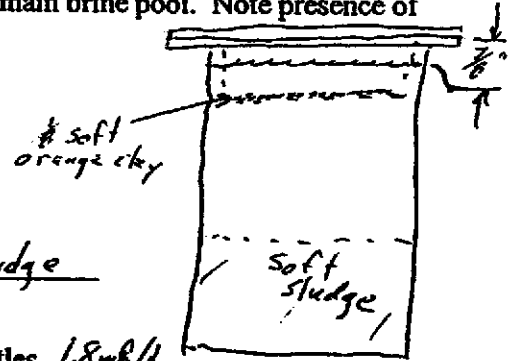
Lid and screen observations. (e.g. condition of screen, impaction, corrosion, etc.)

*clean lid except for thin orange coating around sampling port.
screen looked like new. No impaction - removed easily. had 1/2" sludge in it.*

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

Brine very clear above screen but yellow cloudy below.

Depth of the brine pool. _____



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

RAD (mR/hr contact) reading of the brine in the 1-liter bottles. 1.8 mR/hr

Core sample taken? NA

Fe mesh container recovered? If yes, describe condition of

no brine and solid material surrounding the container for the mesh. Nothing adhered to container except as though it had been dropped in the mud.

Dose rate reading of container holding the mesh (mR/hr contact) 1.2 mR/hr

Liter Scale Disassembly Observations Checklist

Liter-Scale Container # 19 Date: 2-14-01

Was the mesh container embedded?
yes - well, sort of, soft blackened (black) sludge inside container
rinses off easily.

After removing the mesh from the container, note:

percent dissolved ~ 5%

amount of deposited material on the Fe mesh Moderate - ~ 10% of wire

color of the deposited material Black

thickness and hardness of the deposited material Very thin - Hardness - 5 (apate) (force)

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

Other notes
pumped 750cc sample before the mat'l left was too thick to pump,
too thin to be solid.

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

Waste dispositioned

according to NMT-7 instructions: Ronald Chavez (NMT-7) date 2/14/01

(NMT-7) date _____

Liter Scale Container disassembly
complete:

[Signature] (C-AAC) date 2/14/01

[Signature] (STTP) date 2/14/01

Liter-Scale No. 20
Results of Observations

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 pH 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: H₂ was 2.9 v/o from a nitrate concentration of 30,000 ppm.

D&D Observations): No available data.

Corrosion:
Brine:
Bottom Solids:
Fe Mesh:

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 N₂O concentration in the headspace. There was no filterable colloids containing Pu or Fe at a pH of ~ 7.5.

**Disassembly Observations Checklist
LS-20**

NOT AVAILABLE

Liter-Scale No. 21 Results of Observations

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 pH 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 D&D'd 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 pH 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. 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. H₂ was relatively low at ~ 8 v/o but N₂O was high at ~ 19 v/o (NO₃ was at 32,000 ppm). Overall, this test showed essentially no solubilization of actinides or Fe and the Fe mesh was essentially untouched.

**D & D Liter Scale 21 Castile Brine/Envirostone
November 8, 1999**

- **O₂ 3.7%**
- **Brine appeared clear in color, but actually had an orange tinge / did not appear thick at the top**
- **Fe mesh screen did not dissolve/ appeared fairly clean**
- **Mesh screen fairly clean/ easily removed**
- **Core sample also was an orange color**
- **Actinide vial removed rad reading 5 mR**
- **Rad reading on mesh screen 0.7 - 0.8 mR**

Liter-Scale No. 22 Results of Observations

Test Characteristics

Waste: Envirostone
80-90 % CaSO₄ with 10-20% melamine-formaldehyde and
0.1% NH₄Cl
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 pH 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: 10¹⁰ to 10¹¹ particles/Liter
Filter Paper-WDXRF: Six of 15 filters had Pu and all filters had Fe. All filters had
Sr and S. There seemed to be a collaboration between Pu and
Sr.
Headspace Gas Content: H₂ = 6.1 v/o; N₂O = < 39 v/o; O₂ = 0.15 v/o; NO₃ = 36,000
ppm; TOC = 480 ppm

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

Corrosion: No corrosion noted on SS fittings and feedthroughs; no corrosion
around screen; screen had ~ ¼ - 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.

O₂ — 4.26
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-22 Date: 3-21-01

Environment Hazardous

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

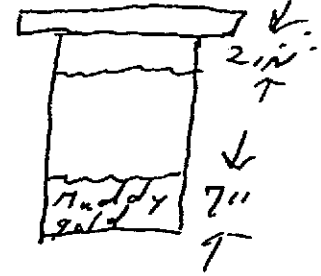
Note: When brine valve opened a slight spray ensued as it the container was still under a slight pressure -
Dose rate reading of screen 1.8 mR

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

Lid - amber colored and in good shape. Screen - no rust and about 3/4" of gold sediment in it. No probe on lid. No corrosion noted around parts.

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

Depth of the brine pool. _____



Hardness of solids at the bottom of the container.

Muddy texture that allows mixture readily.

RAD (mR/hr contact) reading of the brine in the 1-liter bottles. ~0.5 mR

1 liter of brine taken out quite easily. Brine is a light brown color.

Core sample taken? NA No lead probe on lid.

Fe mesh container recovered? If yes, describe condition of

no brine and solid material surrounding the container for the mesh.

The mesh was in 53 separate pieces but it didn't seem to be broken. The rinse water was ~0.5 mR avocado green

Liter Scale Disassembly Observations Checklist

Liter-Scale Container # LS-22 Date: 3-21-01

Was the mesh container embedded?

yes no

After removing the mesh from the container, note:

percent dissolved 06

amount of deposited material on the Fe mesh Not much

color of the deposited material Avocado green & rinsed easily off.

thickness and hardness of the deposited material Not hard - less than peanut buttery texture.

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

Black. Mesh seems to have held up well over time.

Other notes

Whoever packaged the mesh seemed to have folded up different pieces of mesh into this batch for some reason.

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

Waste dispositioned

according to NMT-7 instructions: Raul Chis (NMT-7) date 3/21/01

(NMT-7) date _____

Liter Scale Container disassembly complete:

[Signature] (C-AAC) date 3/21/01

[Signature] (STTP) date 3/21/01

Liter-Scale No. 23 Results of Observations

Test Characteristics

Waste: Envirostone
80 - 90 % CaSO₄ 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

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 pH 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: 10¹⁰ to 10¹¹ 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..
H₂ Headspace Gas Content: H₂ = 8.0 v/o; N₂O = 31.3 v/o; N₂ = 6.4 v/o; NO₃ 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 pH 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 NO₃ at 30,000 ppm and N₂O 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.

O₂ at 2.7

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.

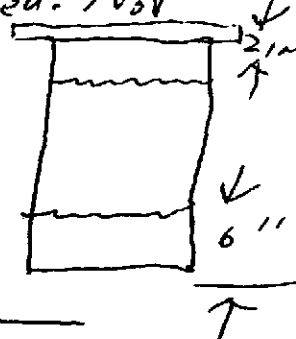
Liter-Scale Container # 5-23 Date: 3-12-01

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

Dose rate reading of screen 3.0 mR

Lid and screen observations. (e.g. condition of screen, impaction, corrosion, etc.)
Not much rust at all. Screen basket easy to remove.

Note the color/turbidity (muddy, cloudy, opaque, thick) of main brine pool. Note presence of suspensions or crystals.
Gold brine pool - ~ 1 liter brine removed. Not much in the form of suspensions or crystals.
Depth of the brine pool. 6 inches



Hardness of solids at the bottom of the container. Not much hardness of solids. Can easily be stirred

RAD (mR/hr contact) reading of the brine in the 1-liter bottles. 8.0 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.
no

The brine is water thin and the container is in very good shape.
Dose rate reading of container holding the mesh (mR/hr contact) 2.0 mR

Liter Scale Disassembly Observations Checklist

Liter-Scale Container # LS-23 Date: 3-12-01

Was the mesh container embedded?

yes no

After removing the mesh from the container, note:

percent dissolved 0%

amount of deposited material on the Fe mesh Not much

color of the deposited material Gold

thickness and hardness of the deposited material None

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

Black and good physical appearance.

Other notes

Hazardous batch - Need to dispose of correctly.

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

Waste dispositioned

according to NMT-7 instructions: [Signature] (NMT-7) date 3/12/01

(NMT-7) date _____

Liter Scale Container disassembly complete:

[Signature] (C-AAC) date 3/12/01

[Signature] (STTP) date 3-12-01

Liter-Scale No. 24 Results of Observations

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 $\mu\text{g/g}$; Total Pu = 0.305 g
Am 8.19 $\mu\text{g/g}$; 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 pH 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.
- Bottom Solids:** There was about 6 inches of a clay like mass that 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 silty clay mass that can 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. There appeared to be some corrosion of the wire and no corrosion where the lather had been.

Overall Assessment:

LS-24 was a Castile brine test at pH 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 silty 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 N₂O concentration in the headspace gas. H₂ was only 12.7 v/o but there must have been some radiolytic generation of N₂ 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.

4.6% O₂
25°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-024 Date: _____
Haz Envirostone Castile Feresh
Videotape recorded? yes NO If yes, record ID# of tape _____

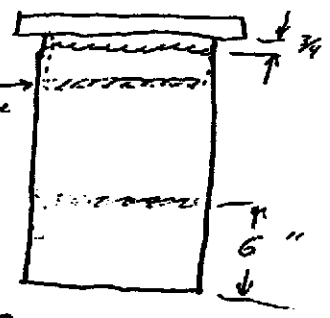
Dose rate reading of screen NA

Lid and screen observations. (e.g. condition of screen, impaction, corrosion, etc.)
Slight pressure noted when lid opened vent opened.
Lid mostly uncorroded - original color - Light rust ring around sampling port

Note the color/turbidity (muddy, cloudy, opaque, thick) of main brine pool. Note presence of suspensions or crystals.
Black specks floating on yellowish murky non-viscous liquid.
1000-ml brine sample was turbid (chalky) but yellowish-olive drab
Semi-gelatinous cohesive mass

Depth of the brine pool. _____

Hardness of solids at the bottom of the container. increases with depth.
soft - can be stirred to homogeneity. Compact at bottom



RAD (mR/hr contact) reading of the brine in the 1-liter bottles. 4.7 mR/hr

Gore sample taken?
yes NA

Fe mesh container recovered? If yes, describe condition of brine and solid material surrounding the container for the mesh.
yes no compact clay-li mass that could easily be rinsed away with H₂O.

Dose rate reading of container holding the mesh (mR/hr contact) 2.7 mR/hr

Liter Scale Disassembly Observations Checklist

Liter-Scale Container # LS-24 Date: 4/12/01

Was the mesh container embedded?
 no *unconsolidated silty/clay mess that could be rinsed (if one were to try) off w/water.*

After removing the mesh from the container, note:

percent dissolved 10%

amount of deposited material on the Fe mesh 0.010"

color of the deposited material greenish black - 1/8" boundary layer of very dark silt - remainder of solids in mesh container were undisclored ~~greenish~~

thickness and hardness of the deposited material softer than steel. can be rubbed to powder by pliers. *yellow/brown.*

After cleaning the mesh with water, note the color and physical appearance of the mesh.
Black (mineralogic "streak" is greenish black) somewhat corroded and very heavily coated.

Other notes

Waste container number for disposal of the D&D liter scale container LS-24

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. 25 Results of Observations

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.
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 pH 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.

H₂ Headspace Gas Content: H₂ = 13 v/o; O₂ = 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 H₂ concentration in the pyrochemical salt tests of 13 v/o. The H₂ 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.

1.88 Oz

Attachment 1

27°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-25 Date: 4-3-01
Pyrochem Non-Haz Brine A
Videotape recorded? YES NO If yes, record ID# of tape _____

Dose rate reading of screen 0.25 mR

Lid and screen observations, (e.g. condition of screen, impaction, corrosion, etc.)
Crystals on the level probe. Green color on the penetrations. Rust around half of the lip of the screen.
Note the color/turbidity (muddy, cloudy, opaque, thick) of main brine pool. Note presence of suspensions or crystals. About 1/2 full of white crystals in the screen.

Depth of the brine pool. _____

Hardness of solids at the bottom of the container. Pretty hard solids about 5 inches thick.

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.
 YES NO Salt crystals all over the container.
Light blue color

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

Liter Scale Disassembly Observations Checklist

Liter-Scale Container # LS-25 Date: 4-3-01

Was the mesh container embedded?

yes no

After removing the mesh from the container, note:

percent dissolved 0%

amount of deposited material on the Fe mesh Small amount

color of the deposited material Light blue

thickness and hardness of the deposited material Not very thick or hard.

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

The mesh looks in good shape.

Other notes

Waste container number for disposal of the D&D liter scale container LS-25

Waste dispositioned

according to NMT-7 instructions: [Signature] (NMT-7) date 4.03.01

(NMT-7) date _____

Liter Scale Container disassembly complete:

[Signature] (C-AAC) date 4/3/01

[Signature] (STTP) date 4-3-01

Liter-Scale No. 26 Results of Observations

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 pH 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 21,000 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.

H₂ Headspace Gas Content: ~73 v/o H₂.

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

Corrosion: lid and SS fittings were corroded, the high H₂ 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.

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 SrSO₄ 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 H₂ 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 H₂ 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 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-26 Date: 1-24-01

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

Dose rate reading of screen 1.5 mR/hr

Lid and screen observations. (e.g. condition of screen, impaction, corrosion, etc.)
on lid -> No corrosion, yellowish crystals & solids adhered to lid and screen, screen encrusted

Note the color/turbidity (muddy, cloudy, opaque, thick) of main brine pool. Note presence of suspensions or crystals. *Brine is not viscous (>10" pour) Crystals throughout*

Depth of the brine pool. _____

Hardness of solids at the bottom of the container. 3/2 (softer in middle)

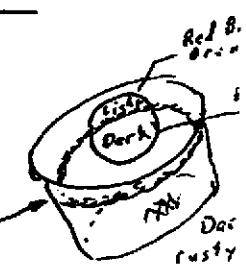
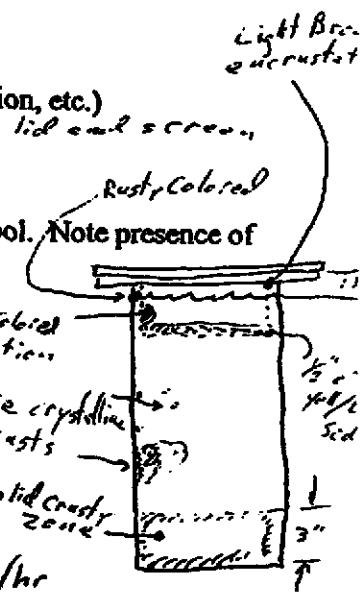
RAD (mR/hr contact) reading of the brine in the 1-liter bottles. 0.1 mR/hr

Core sample taken? yes NA

Fe mesh container recovered? If yes, describe condition of brine and solid material surrounding the mesh.
yes no Slushy !! - rinses easily

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

Bring level crusty 3mm white xls. varicored 1.0 mR/hr



Liter Scale Disassembly Observations Checklist

Liter-Scale Container # LS-26 Date: 1-24-01

Was the mesh container embedded? *Usual salt muck that rinses off except the crystals that are too big (may be crucible shards). Some of this is white crust or precip or crystals.*
yes *about the screen was surrounded by blackened salt slush.*

After removing the mesh from the container, note:

percent dissolved *one spot has 50% dissolution over a 4cm diameter. otherwise it is not dissolved but it is encrusted.*

amount of deposited material on the Fe mesh *Thin (<0.1mm) layer + white 1/2 mm*

color of the deposited material *Black or white.*

thickness and hardness of the deposited material *white = soft
Black = hard*



After cleaning the mesh with water, note the color and physical appearance of the mesh.
Encrusted. Black. mostly white except as noted above.

Other notes

Waste container number for disposal of the D&D liter scale container 57092

Waste dispositioned according to NMT-7 instructions: [Signature] (NMT-7) date 1/24/01
____ (NMT-7) date _____

Liter Scale Container disassembly complete: F.R. John (C-AAC) date 1/24/01
[Signature] (STTP) date 1-24-01

Liter-Scale No. 27 Results of Observations

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 CO₂.
- 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 pH 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 pH 10.7 to 11.2. This is surprising because there was soluble Fe in the brine most of the test.

H₂ Headspace Gas Content: 65 v/o H₂; O₂ 4.8 v/o O₂.

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 was 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 FeCl₂ · H₂O (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. 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 pH (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 H₂ 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 10¹¹ to 10¹² 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 Fe²⁺ state rather than the more insoluble Fe³⁺ state. The high H₂ 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 Fe²⁺. 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 Fe²⁺ 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 Fe³⁺, 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. Pressure gauge was probably inoperative.

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.

O₂ = 2.1%
31°C

Liter-Scale Container # LS-27 Date: 1-9-01

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

Dose rate reading of screen 4 mR/hr PX

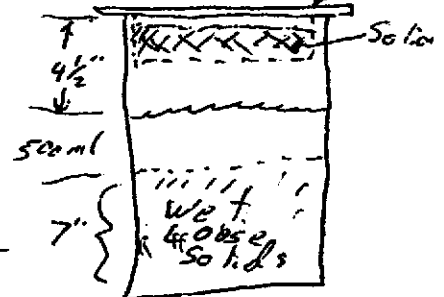
Lid and screen observations. (e.g. condition of screen, impaction, corrosion, etc.)
*Screen full of dried pea soup - thick greenish wet mass
Lid has big rust colored deposits around fittings. - Gauge inoperative
Crucible shards stuck to outside of screen.*

Severe corrosion on fittings.

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

white plati

Depth of the brine pool. 3"



Hardness of solids at the bottom of the container. Soft

RAD (mR/hr contact) reading of the brine in the 1-liter bottles. 1.5 mR/hr PX

Core sample taken?

yes NA

Fe mesh container recovered? If yes, describe condition of brine and solid material surrounding the container for the mesh.

yes no

= Soft loose wet sludge, washes easily.

Dose rate reading of container holding the mesh (mR/hr contact) 5 mR/hr PX

Liter Scale Disassembly Observations Checklist

Liter-Scale Container # LS-27 Date: 1-9-00

Was the mesh container embedded?
yes no

After removing the mesh from the container, note:

percent dissolved 0

amount of deposited material on the Fe mesh Color only

color of the deposited material black

thickness and hardness of the deposited material 0 and hard

when wet, we see shiny cut ends. Bleached but not changed in dimensions.
After cleaning the mesh with water, note the color and physical appearance of the mesh.

Other notes

Level Probe was shiny where it was immersed but coated w/ rust (probably

Waste container number for disposal of the D&D liter scale container 57089.

Waste dispositioned

according to NMT-7 instructions: Ronald Chisley (NMT-7) date 1/9/01

(NMT-7) date _____

Liter Scale Container disassembly complete:

T.R. Adam (C-AAC) date 1/9/01

Ronald Chisley (STTP) date 1-9-01

Liter-Scale No. 28 (Pressurized) Results of Observations

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 CO₂ 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 pH 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. 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 pH 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 pH 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 pH 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: 1×10^{13} particles/L

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

H₂ 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 Pyro Chemical 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, CO₂ 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 CO₂ 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 CO₂ 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.

temp 29°C
18.2 g O₂

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-28 Date: 4-26-01 *NON haz pyrochem Brine A Fe mesh*

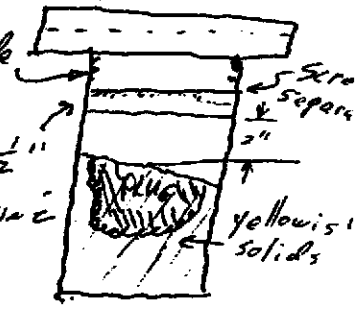
Videotape recorded? yes no If yes, record ID# of tape ✓

Dose rate reading of screen 0.6 mR

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

Lid has some small white and greenish-black crystalline mass. Screen is separated with a pesty mass and material stuck to the side.
Note the color/turbidity (muddy, cloudy, opaque, thick) of main brine pool. Note presence of suspensions or crystals. *crucible shards & other material stuck to side and stuck to screen*

The brine is 1/2 inch below the screen
Depth of the brine pool. 2 1/2 inches
Non viscous brine - cloudy (yellowish)



Hardness of solids at the bottom of the container. Hard.
Difficult to poke a screwdriver through it. Can hammer a screwdriver through many places attempting to remove a 3/4\"/>

RAD (mR/hr contact) reading of the brine in the 1-liter bottles. 2.2
350 ml of brine taken out.

Core sample taken? NA *A sample from the plug was like gypsum from the Todillo formation.*

Fe mesh container recovered? If yes, describe condition of no
brine and solid material surrounding the container for the mesh. Liquid with much solids - like oatmeal (thin oatmeal - like stone)

Dose rate reading of container holding the mesh (mR/hr contact) 2.7 mR/hr

Liter Scale Disassembly Observations Checklist

Liter-Scale Container # LS-28 Date: 4-26-01

Was the mesh container embedded?
 no impacted with crucible shards and coarse "beach sand"
Discolored Black, Not Cemented.

After removing the mesh from the container, note:

percent dissolved 0-5%

amount of deposited material on the Fe mesh Very thin - very little

color of the deposited material Black mostly, some white on ends, a 2x6 orange spot and a 1cm² orange spot. Very faint powder blue patina on top of it
thickness and hardness of the deposited material hard - thin coating

After cleaning the mesh with water, note the color and physical appearance of the mesh.
structurally → Nearly new - mostly black with a few colors (noted above)

Other notes
see back of page 1 for more sketches

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

Waste dispositioned according to NMT-7 instructions: Heretta Ortega (NMT-7) date 4/30/01

Liter Scale Container disassembly complete: J.R. Haffa (C-AAC) date 4/26/01
David Zeeman (STTP) date 4-26-01

**Liter-Scale No. 29 (Pressurized)
Results of Observations**

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 CO₂ 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 pH 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).
H₂ 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.

Overall Assessment:

Liter-scale test container No. 29 was a Pyrochemical salt waste in Brine A and had ~870 psig CO₂ pressure placed on the headspace. The pH 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 SrSO₄. 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 pH at ~ 3×10^{12} particles/liter. This test showed that even with 870 psig of CO₂ in an acid condition that actinides were not significantly solubilized.

Non Haz
Pyrochem
Brine A
Iron Mesh

$O_2 < 2\%$
 $29^\circ C$

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-29 Date: 5-23-01

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

Dose rate reading of screen 0.3 mR/hr

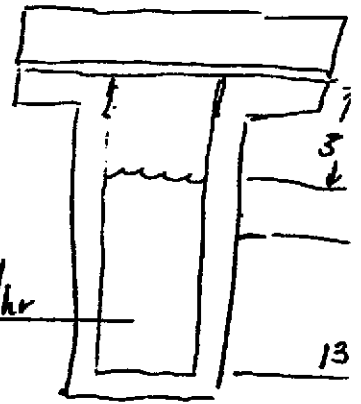
Lid and screen observations. (e.g. condition of screen, impaction, corrosion, etc.)
Brown Crystals on top of screen - 1mm size, about 4-8cc of material x ls. Screen was torn. A few black drips around sampling port & gauge port. No corrosion apparent.

Note the color/turbidity (muddy, cloudy, opaque, thick) of main brine pool. Note presence of suspensions or crystals. *Clear brine. No suspensions. Crystals have sunk to bottom or been deposited like sand on screen - not stuck, but laid on top. Some 4mm crystals adhered to sides of container.*
Depth of the brine pool. 400ml brine

Hardness of solids at the bottom of the container. 0.3 mR/hr 

Color: *dark muddy brown*
Texture: *Sandy*

RAD (mR/hr contact) reading of the brine in the 1-liter bottles. 0.3 mR/hr



Core sample taken?
yes NA Crystals from side of container

Fe mesh container recovered? If yes, describe condition of
 no brine and solid material surrounding the container for the mesh.
Consistency of sand; Appearance, dark muddy brown

Dose rate reading of container holding the mesh (mR/hr contact) < 0.2 mR/hr
Consistency of sand; dark muddy brown brine

Liter Scale Disassembly Observations Checklist

Liter-Scale Container # LS-29 Date: 5-23-01

Was the mesh container embedded?

yes no

After removing the mesh from the container, note:

percent dissolved 0

amount of deposited material on the Fe mesh coated w/black (thin coating)

color of the deposited material Salt + pepper + black

thickness and hardness of the deposited material thin and appears hard

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

black to grey. No visible corrosion

Other notes Material surrounding screen is grey and no green is visible

Waste container number for disposal of the D&D liter scale container LS-29.

Waste dispositioned

according to NMT-7 instructions: S. L. V. M. J. (NMT-7) date 5-24-01

(NMT-7) date _____

Liter Scale Container disassembly complete:

T. R. Blahm (C-AAC) date 5/23/01

David J. ... (STTP) date 5-23-01

Note: Removed a chunk of ^{material} 3" x 2" x 1"

Liter-Scale No. 30 (Pressurized) Results of Observations

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 CO₂ 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 at 34.8 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 pH 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.
H₂ Headspace Gas Content: No analyses.

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 CO₂ 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 ($\sim 2 \times 10^{11}$ 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.

1.18 O₂
24°C temp

Attachment 1

High Pressure

Liter Scale Disassembly Observations Checklist

Pyrochem Castile brine Fe mesh Non-Hez

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-30 Date: 5-30-01

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

Dose rate reading of screen 1.7 mR No corrosion noted

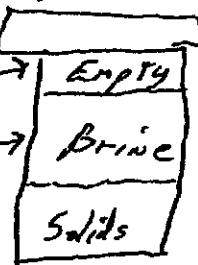
Lid and screen observations, (e.g. condition of screen, impaction, corrosion, etc.)

Lids in good condition, has a little white-gray residue. Screen is intact with the oatmeal residue on it.

Note the color/turbidity (muddy, cloudy, opaque, thick) of main brine pool. Note presence of suspensions or crystals. Main brine pool is light gray to brown and has an oatmeal texture.

Depth of the brine pool. 3" from top of vessel. 3" →

Hardness of solids at the bottom of the container. Pretty hard 10 1/2" →



RAD (mR/hr contact) reading of the brine in the 1-liter bottles. 6 mR
w 750 mls of brine obtained
A brownish color.

Core sample taken? NA Small ceramic-like pieces were found (white) in the container the mesh was in.

Fe mesh container recovered? If yes, describe condition of no brine and solid material surrounding the container for the mesh.
A hard brown solid material, had to break the container to get the mesh out

Dose rate reading of container holding the mesh (mR/hr contact) 1.6 mR

Liter Scale Disassembly Observations Checklist

Liter-Scale Container # LS-30 Date: 5-30-01

Was the mesh container embedded?
 yes no It was cemented to the container's mesh insert. The mesh is black.

After removing the mesh from the container, note:

percent dissolved N/A

amount of deposited material on the Fe mesh N/A

color of the deposited material Different colors (red, yellow & grey)

thickness and hardness of the deposited material Extremely hard deposit. Couldn't remove all of it.

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

The color of the mesh is dark black, but it seems to have held up okay -

Other notes
Will put 6 pieces of white ceramic material as a core sample.

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

~150 mls of aquasorb added to container.

Waste dispositioned

according to NMT-7 instructions: Donetta Ostry (NMT-7) date 5/30/01

(NMT-7) date _____

Liter Scale Container disassembly complete:

J. K. [Signature] (C-AAC) date 5/30/01
Al. R. Vigil (STTP) date 5-30-01

~~517055~~

Liter-Scale No. 31 Results of Observations

Test Characteristics:

Waste: Pyrochemical salt waste (O₂ 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).
- 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 pH 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: 10⁹ to 10¹⁰ 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: H₂ = ~ 33 v/o; O₂ = 0.80 v/o; N₂O = 0.5 v/o

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

Corrosion:
Brine:
Bottom solids:
Fe mesh:

Overall Assessment:

This pyrochemical salt waste test with benonite 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.

**Disassembly Observations Checklist
LS-31**

NOT AVAILABLE

Liter-Scale No. 32 Results of Observations

Test Characteristics:

Waste: Pyrochemical salt waste (O₂ 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 mg 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.
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 pH 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: 10⁹ to 10¹⁰ 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: H₂ = ~ 44.6 v/o; O₂ = 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 pH 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 D&D Process

Liter-Scale container # 32

Date of D&D: 12/1/99

Lid and screen observations/RAD reading (mR):

Lid had three rust spots/corrosion on underside.

Note condition of screen:

Screen was corroded- salt adhering at bottom.

Note color/turbidity (muddy- cloudy- opaque- thick) of main brine pool/ presence of suspensions or crystals:

Brine was fairly clear- salt crystals around the edge- pea soup color.

Depth of brine pool/hardness of solids at bottom of container:

Brine in the container was fairly liquid. More sludge toward the bottom of the container. Large salt particles on the bottom.

RAD (mR) reading after brine pumped to 1-liter bottles:

2 mR (gamma reading) – Filling up another bottle of brine- tube became clogged, had to begin dipping liquid and sludge into the bottle.

After liquids separated from solids take RAD reading (mR):

2.5 mR - 3mR sludge only

Core sample may be taken at this point at PL discretion:

No core sample was taken- will obtain sample from the bottom and sides of container.

If Fe mesh container recovered note condition of brine/solid material surrounding the container/RAD reading (mR):

3 mR (sludge) Fe mesh couldn't be located.

Was Fe mesh container embedded?

Yes, in the bottom where the mesh was, rock hard.

After removal of Fe mesh container remove screen/Note condition, color, outward appearance of screen/RAD reading mR):

After water wash/alcohol bath of Fe mesh screen note color, physical appearance of screen:

Note: An RCT was not present, Kathy Garduno called Ron Chavez who stated an RCT was not needed to witness the D&D process.

Liter-Scale No. 33 Results of Observations

Test Characteristics:

Waste: Pyrochemical salts (O₂ 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: 10⁹ to 10¹⁰ 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.
H₂ Headspace Gas Content: H₂ = 35 v/o; O₂ = 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.

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 pH 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 $5 \times 10^{-2}\%$. 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.

Attachment 1

Liter Scale Disassembly Observations Checklist 4.28 02

25°C

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-33 Date: 1-30-01

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

Dose rate reading of screen ~~2.5~~ 2.5

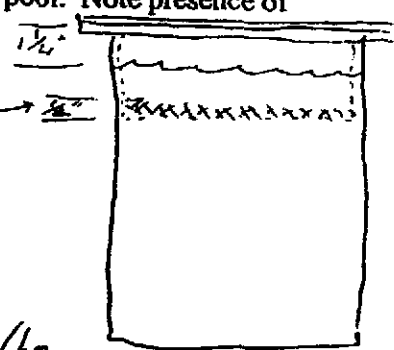
Lid and screen observations, (e.g. condition of screen, impaction, corrosion, etc.)
Fittings & feedthrus in lid have a rusty ring around them - rather small comp. to the worst, but still corroded. Nothing else stuck to or growing on lid or basket. Upper ring broke loose from basket while we were trying to pull basket out - weakened by abrasion. No encrustation on screen.

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

Depth of the brine pool.

Hardness of solids at the bottom of the container. Gravel.
2" of crucible shards - uncemented.

RAD (mR/hr contact) reading of the brine in the 1-liter bottles 2 mR/hr



Core sample taken? yes no

Fe mesh container recovered? If yes, describe condition of Loose material - no cementation; all crucible shards and fines;
no discoloration.
 yes no

Dose rate reading of container holding the mesh (mR/hr contact) 0.5 mR/hr

Liter Scale Disassembly Observations Checklist

Liter-Scale Container # LS-33 Date: 1-30-01

Was the mesh container embedded?
yes no

After removing the mesh from the container, note:

percent dissolved 0%

amount of deposited material on the Fe mesh 0-0.1 mm

color of the deposited material if any, it is black.

thickness and hardness of the deposited material Thinner than most - hardly any.
material is hard

After cleaning the mesh with water, note the color and physical appearance of the mesh.
uncorroded wires w/o encrustation.

SHINY cut ends.

Other notes

Waste container number for disposal of the D&D liter scale container 57092.

Waste dispositioned according to NMT-7 instructions:

[Signature] (NMT-7) date 1/30/01
____ (NMT-7) date _____

Liter Scale Container disassembly complete:

[Signature] (C-AAC) date 1-30-01
[Signature] (STTP) date 1-30-01

Liter-Scale No. 34 Results of Observations

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, 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

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 pH 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: 10⁹ to 10¹⁰ particles/Liter
Filter Paper-WDXRF: Essentially all filters contained Pu. No filters had Fe. No filter had Sr.

H₂ Headspace Gas Content: H₂ = 28.6 v/o; O₂ = 0.66 v/o; N₂ = 2.2 v/o; N₂O = 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 was 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 Ca(OH)_2 were added to this test but not much solubilization of actinides took place as Pu was generally <30 ppb at the pH 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. 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.

O₂ - 2.66
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-34 Date: 3-21-01
Pyrochemical Non-hazardous
Videotape recorded? yes no If yes, record ID# of tape _____

Probably ground penetrations only

Dose rate reading of screen 2.11 R

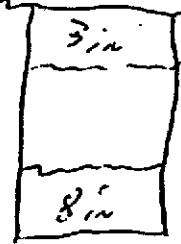
Lid and screen observations. (e.g. condition of screen, impaction, corrosion, etc.)

Lid Corroded. ground penetrations - pitting color, Outside.
More corrosion around screen. Also brown color corrosion
black in spots on top. around one side of penetrations.
Note the color/turbidity (muddy, cloudy, opaque, thick) of main brine pool. Note presence of Inside
suspensions or crystals. Screen full of white solids - 1/8" thick seen
Hard to get at to remove. Need to almost break screen
to remove it. The brine itself is colorless.

Depth of the brine pool. Brine is colorless ~~and~~ except for the reflection of the vessel.

Hardness of solids at the bottom of the container. at heel consistency. Solids at bottom of
vanner are very hard.

RAD (mR/hr contact) reading of the brine in the 1-liter bottles. 20.8 R
1 liter of clear brine taken out.



Core sample taken? yes NA

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

Will leave Fe mesh in because of the
rockhard solids surrounding the container.
Dose rate reading of container holding the mesh (mR/hr contact) N/A

Liter Scale Disassembly Observations Checklist

Liter-Scale Container # LS-34 Date: 3-21-01

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.

N/A

Other notes

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

Waste dispositioned

according to NMT-7 instructions: Ronald Chis (NMT-7) date 3/21/01

(NMT-7) date _____

Liter Scale Container disassembly complete:

[Signature] (C-AAC) date 3/21/01

[Signature] (STTP) date 3/21/01

Liter-Scale No. 35 Results of Observations

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 pH 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.

Other Analyses (nominal):

Typical pH Range: 8.2 – 8.3
Fe Concentrations: Generally < 1 ppm with nine results between 1 and 3 ppb.
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.

H₂ Headspace Gas Content: H₂ = 20.9 v/o; O₂ = 0.07 v/o; N₂ = 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 Brine A 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 pH 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 ~ 1/2-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.

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 H₂ in the headspace was ~ 21 % and CO₂ was not detectable from the added chelators that resulted in a rather low TOC of 60 ppm.

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-35 Date: 3-13-01

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

Dose rate reading of screen 0.2 mR Teal-blue ground top.

Lid and screen observations. (e.g. condition of screen, impaction, corrosion, etc.)
Teal blue ground level probe is connected to lid and sampling port. Doesn't seem to be rust.

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

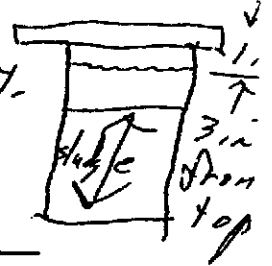
Color of main brine pool is a murky beige with murky white particles floating.

Depth of the brine pool.

Pyrochemical salts are a pain to get out.

Hardness of solids at the bottom of the container.

A lot of solids in vessel. Teal blue ground screen at top.
RAD (mR/hr contact) reading of the brine in the 1-liter bottles. 0.1 mR
Only were able to pull out 200mls.



Core sample taken?

NA Out mealy solution throughout!

Fe mesh container recovered? If yes, describe condition of

no brine and solid material surrounding the container for the mesh.
Teal-blue brine all around the container -

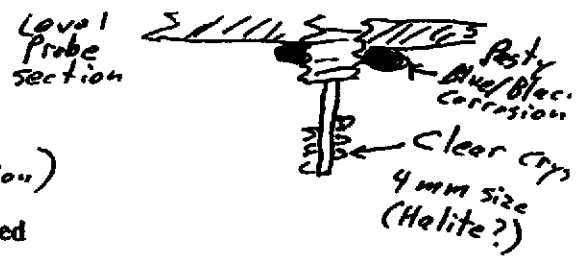
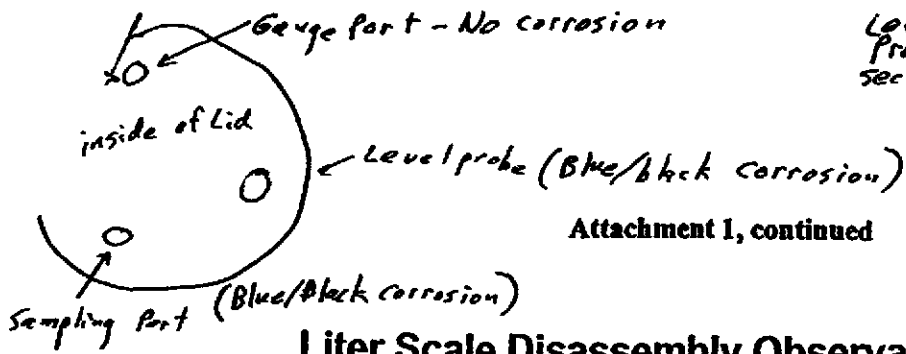
Dose rate reading of container holding the mesh (mR/hr contact) 0.2 mR

Very hard sample to work with.
Big white particle size dried salts around outer screen -

NMT11-WI-099,R0

STTP Liter Scale Container
Disassembly & Waste
Packaging

Very salty - 1.1



Attachment 1, continued

page 2 of 2

Liter Scale Disassembly Observations Checklist

Liter-Scale Container # LS-35 Date: 3-13-01

Was the mesh container embedded?
 no Oatmeal-like sludge reading ~ 0.3 nF at contact through gloves.

After removing the mesh from the container, note:
 percent dissolved Not much.

amount of deposited material on the Fe mesh Blue salts quite a bit.

color of the deposited material Blue

thickness and hardness of the deposited material Pretty hard, doesn't rinse off or soak off.

After cleaning the mesh with water, note the color and physical appearance of the mesh.
Blue-black mesh, still

Other notes
Non-hazardous vessel. Greenish Blue paste around iron mesh. & Screen not difficult to remove.

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

Waste dispositioned according to NMT-7 instructions: Sel L V M (NMT-7) date 3-14-01

_____ (NMT-7) date 3

Liter Scale Container disassembly complete: [Signature] (IC-AAC) date 3/14/01

[Signature] (STTP) date 3-13-01

Salt crystals formed around level probe!
 NMT11-WI-099,R0 STTP Liter Scale Container Disassembly & Waste Packaging
Nice big white zones

Quite a different batch from any...

We had to use aquasorb to soak up
the brine because the regular absorbent
wouldn't do it. The aquasorb seemed to
work quite well. Then we topped off
the vessel with vermiculite.

Liter-Scale No. 36 Results of Observations

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: Acetnide = 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

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 pH 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
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 pH.

H₂ Headspace Gas Content: H₂ = 70 v/o; O₂ = 15 v/o (the highest in the STTP);
N₂ = 0.2
The high O₂ 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 pH (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 pH. This pH 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 (10¹⁰ to a maximum of 10¹² 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 SrSO₄ at this pH 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 O₂ 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. The cemented solid could have been immediately formed by the available but limited mg content at the beginning of the test to form a soret 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 pH 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.

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 # 36 Date: 11-29-00

0.8% O₂ start

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

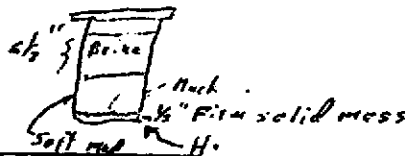
Dose rate reading of screen 0.7 mR/hr βS

Lid and screen observations. (e.g. condition of screen, impaction, corrosion, etc.)
*Sample port heavily plugged - gauge port more open - much rust & corrosion in tank as seen
Screen choked w/ gray muck-like pudding. Free brine below the screen. 1/2" deep*

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

Greenish - gray liquid coffee-like

Depth of the brine pool. 1 liter -



Hardness of solids at the bottom of the container.

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

Core sample taken?

yes NA

yes no Fe mesh container recovered? If yes, describe condition of *Mesh container was impacted. brine and solid material surrounding the container for the mesh. Gray gravelly material. The mesh impacted & recrystallized salt. Only 1/2 pieces of wire could be removed from plastic box.*

Dose rate reading of container holding the mesh (mR/hr contact) not measured

Screen rate = 1.5 βS

Liter Scale Disassembly Observations Checklist

Liter-Scale Container # 36 Date: 11-29-00

Was the mesh container embedded?

yes no lightly

After removing the mesh from the container, note:

percent dissolved 0

amount of deposited material on the Fe mesh 10 gm on lower part of lower screw

color of the deposited material white

thickness and hardness of the deposited material H=3 (calcite)

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

Black - uncorroded (based on apparent dimensions)

Other notes

Brine absorption test. 30 ml Brine added to 6 ml Agnesorho 2212. will be set overnight & inspect for absorption in morning. (11-30-00 Not much absorpt. will add 5 ml Agnesorho)

Waste container number for disposal of the D&D liter scale container 57089

Waste dispositioned according to NMT-7 instructions:

[Signature] (NMT-7) date 11/30/00

_____ (NMT-7) date _____

Liter Scale Container disassembly complete:

JR Fahn (C-AAC) date 11/30/00

[Signature] (STTP) date 11-30-00

Step	Action	Check
14	Remove septum holder assembly and replace with suitable plug. Monitor for contamination and clean if required.	
15	Remove annular space purge fitting and cap or plug as required. Monitor and clean if required.	
16	Remove thermocouple.	
17	Place lab jack under test container clamp.	
18	Loosen and remove test container clamp.	
19	Monitor for loose contamination and clean as required.	
20	Place test container in plastic bag, tape shut and mark appropriately. Monitor outside of bag for contamination. If any is found, double bag and check again as above.	
21	Place bagged test container in an insulated transport container in the upright position.	
22	After standard RCT check and release, transport container to wing 5.	
23	Turn over test container to appropriate NMT-1 personnel.	

Record the following: 4 11-28-00
Date: ~~11-29-00~~ 11-28-00
Liter Scale Container #: LS 36
Time Taken Off Line: 0940
Individuals Involved: W.F. Senlove
D.R. Jeomans

Liter-Scale No. 37 Results of Observations

Test Characteristics:

Waste: Pyrochemical salts (Direct oxide reduction)
Total Waste Weight: 1320 g
Initial Actinide Content: Pu 3,295 µg/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 pH 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.

H₂ Headspace Gas Content: H₂ = ~ 50 v/o; O₂ = ~ 4.7 v/o

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 pH 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 $\text{Fe}(\text{Cl})_2$ 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.

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 # 37 Date: 12-06-00

30°C at site

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

18% O₂

Dose rate reading of screen 0.3 mR/hr

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


dy
12-06-00
Level probe
fell out of
but still shiny.
reflow insulator
still in place



Mechanical shell of "rust" that used to sit around the sample port on lid. - Effect scale would have prevented sampling w/ needle
Area of flocculated mat'l (actually a mass of crystals) Greenish gel at lip of lid near O-ring

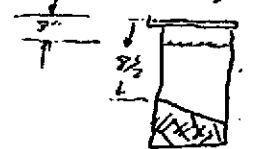
Note the color/turbidity (muddy, cloudy, opaque, thick) of main brine pool. Note presence of suspensions or crystals. Clear w/ tan fines that settle out. screen held material out to sides & bottom that packed like cream of wheat. Level probe had fallen out. We clean off the mass of cubic (Halite) ten crystals, washed & dried it & sent for surface analysis. Recombinant turned out to be a mass of cubic crystals ~1mm-7mm size each.

Depth of the brine pool. 7"

a few white masses  floating

1 1/2 liters brine recovered

Hardness of solids at the bottom of the container. Compressed silt/clay at the softest, crusty at hardest. Hardness disappears when pumped with a screwdriver.



RAD (mR/hr contact) reading of the brine in the 1-liter bottles. 0.0

Core sample taken? No
yes NA

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) NA

Liter Scale Disassembly Observations Checklist

Liter-Scale Container # 37 Date: 12.6.00

0.5 = R/hr. on Si

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

Waste container number for disposal of the D&D liter scale container 57089

Waste dispositioned according to NMT-7 instructions. [Signature] (NMT-7) date 12/6/00
_____ (NMT-7) date _____

Liter Scale Container disassembly complete: T.R. Hehner (C-AAC) date 2/6/00
David [Signature] (STTP) date 12.6.00

Step	Action	Check
14	Remove septum holder assembly and replace with suitable plug. Monitor for contamination and clean if required.	
15	Remove annular space purge fitting and cap or plug as required. Monitor and clean if required.	
16	Remove thermocouple.	
17	Place lab jack under test container clamp.	
18	Loosen and remove test container clamp.	
19	Monitor for loose contamination and clean as required.	
20	Place test container in plastic bag, tape shut and mark appropriately. Monitor outside of bag for contamination. If any is found, double bag and check again as above.	
21	Place bagged test container in an insulated transport container in the upright position.	
22	After standard RCT check and release, transport container to wing 5.	
23	Turn over test container to appropriate NMT-I personnel.	

Record the following:

Date: 12-04-00

Liter Scale Container #: 37

Time Taken Off Line: 1500

Individuals Involved: Walt Sandoval, David Yeaman

Liter-Scale No. 38
Results of Observations

Test Characteristics:

Waste: Pyrochemical salts (O₂ 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 as a soluble 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 ²⁴¹Am was added to this test container.

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

Other Analyses (Nominal)

Typical pH 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: 10⁹ to 10¹⁰ particles/L

Filter Paper-WDXRF: There was no Pu, Fe, or Sr found on filter papers.

H₂ Headspace Gas Content: 51.3 v/o H₂; O₂ = 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 pH 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 $2.4 \times 10^{-3} \%$. The percentage of Pu that dissolved relative to what was available in the 1320 g of waste was $7.0 \times 10^{-4} \%$. 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.

Attachment I

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-38 Date: 12-18-00

30°C
O₂ = NA

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

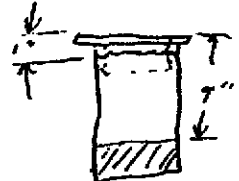
Dose rate reading of screen 0.7 mR/hr β1

Lid and screen observations. (e.g. condition of screen, impaction, corrosion, etc.)
see white/yellow/brown crystals clustered in "dead sites" on level probe. Brine is like coffee - (more like weak tea) - particles in thick & 2" across w/ 1/2" hole stuck to lid screen relatively free of rust; Brit. some light colored cement granules.

Note the color/turbidity (muddy, cloudy, opaque, thick) of main brine pool. Note presence of suspensions or crystals. *clear brine (weak tea). No floaties. Crystals growing on*

Level probe. Brine sample = 0.3 pK Dose rate of 2nd Brine bottle (more solids) = 0.8 μ crystals approx. sub millimeter size
Rate of sludge in bottle = 2.5 pK - 4.0 μ

Depth of the brine pool. 1.8 liters total



Hardness of solids at the bottom of the container. 8 μ
1/4" of fines (yellowish brown) loose on top of a very solid mass

RAD (mR/hr contact) reading of the brine in the 1-liter bottles. 0.3 → 0.8 mR/hr μ

Core sample taken? NO
yes NA

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

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

Liter Scale Disassembly Observations Checklist

Liter-Scale Container # LS-38 Date: 12-18-00

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

Waste container number for disposal of the D&D liter scale container 57089

Waste dispositioned according to NMT-7 instructions: [Signature] (NMT-7) date 12/18/00

(NMT-7) date _____

Liter Scale Container disassembly complete: [Signature] (C-AAC) date 12-18-00
[Signature] (STTP) date 12-18-00

Step	Action	Check
14	Remove septum holder assembly and replace with suitable plug. Monitor for contamination and clean if required.	
15	Remove annular space purge fitting and cap or plug as required. Monitor and clean if required.	
16	Remove thermocouple.	
17	Place lab jack under test container clamp.	
18	Loosen and remove test container clamp.	
19	Monitor for loose contamination and clean as required.	
20	Place test container in plastic bag, tape shut and mark appropriately. Monitor outside of bag for contamination. If any is found, double bag and check again as above.	
21	Place bagged test container in an insulated transport container in the upright position.	
22	After standard RCT check and release, transport container to wing 5.	
23	Turn over test container to appropriate NMT-1 personnel.	

Record the following: ⁴⁻¹²⁻²¹⁻⁰⁰
Date: ~~11-19-00~~ 12-4-00
Liter Scale Container #: 038
Time Taken Off Line: 1500
Individuals Involved: Wilt Sandoual, David Yeaman

Liter-Scale No. 39 Results of Observations

Test Characteristics

Waste: Pyrochemical Salts (O₂ 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 pH 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: 10⁹ to 10¹⁰ 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.
H₂ Headspace Gas Content: ~46 v/o; O₂ at 11.9 v/o (this is high for O₂).

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.

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 $8 \times 10^{-2} \%$. 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 pH 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 1/2 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.

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-39 Date: 11-15-00

Videotape recorded? yes If yes, record ID# of tape NA

Dose rate reading of screen 12 mR p8 - mostly 8

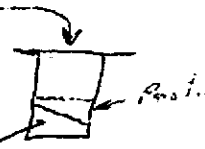
Lid and screen observations. (e.g. condition of screen, impaction, corrosion, etc.)
screen impacted w/ brown scale. Difficult to pry loose. Grey soft scale around entire
of screen.
Level probe almost pristine.
small scale (rust) for some surrounding fittings

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

Tanpep-colored opaque liquid 1 1/2" from top flange. Watery (non-vision)
No suspensions or crystals. 1/2 full of pasty sludge (40%)
by 11-15-00

Depth of the brine pool. ~60% of height
35 mR/hr at 12" p8 } estimate use mR/hr contact
25 " " " " }

Hardness of solids at the bottom of the container. pasty on top 1 1/2"
hard solid



RAD (mR/hr contact) reading of the brine in the 1-liter bottles. 12 mR/hr contact p8 in top 5 cm
29 mR/hr in sample w/ more particulate

Core sample taken? 2 cc. 1-5 ml
yes NA Do we need a sample for CST? p8
11-15-00

Fe mesh container recovered? If yes, describe condition of
yes brine and solid material surrounding the container for the mesh.

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

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Liter-Scale Container # LS-39 Date: 11-15-00

Was the mesh container embedded?
 yes no

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 C₂ Level at start = 1.1% 29.4°C
Packed container is ~ 7 mR/hr contact pos

Waste container number for disposal of the D&D liter scale container 57089

Waste dispositioned according to NMT-7 instructions:

[Signature] (NMT-7) date 11/15/00
____ (NMT-7) date _____

Liter Scale Container disassembly complete:

[Signature] (C-AAC) date 11/15/00
[Signature] (STTP) date 11-15-00

Step	Action	Check
14	Remove septum holder assembly and replace with suitable plug. Monitor for contamination and clean if required.	✓
15	Remove annular space purge fitting and cap or plug as required. Monitor and clean if required.	✓
16	Remove thermocouple.	✓
17	Place lab jack under test container clamp.	✓
18	Loosen and remove test container clamp.	✓
19	Monitor for loose contamination and clean as required.	✓
20	Place test container in plastic bag, tape shut and mark appropriately. Monitor outside of bag for contamination. If any is found, double bag and check again as above.	✓
21	Place bagged test container in an insulated transport container in the upright position.	✓
22	After standard RCT check and release, transport container to wing 5.	✓
23	Turn over test container to appropriate NMT-1 personnel.	✓

Record the following:

Date: 11-14-00

Liter Scale Container #: LS-39

Time Taken Off Line: 1500

Individuals Involved: Walt Sandover

David Yeaman