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SRNL Radionuclide Field Lysimeter Experiment: Baseline Construction and Implementation

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ABBREVIATIONS

SRNL	Savannah River National Laboratory
SRS	Savannah River Site
CLSM	Controlled Low-Strength Material
DCB	Dithionite, Carbonate, Bicarbonate
RPD	Radiological Protection Department
ID	Inner Diameter
LBC	Lime Buffer Capacity
OM	Organic Matter
PVC	Polyvinyl chloride

1 OBJECTIVES

The purpose of this document is to compile information regarding experimental design, facility design, construction, radionuclide source preparation, and path forward for the ten year Savannah River National Laboratory (SRNL) Radionuclide Field Lysimeter Experiment at the Savannah River Site (SRS). This is a collaborative effort by researchers at SRNL and Clemson University. The work instructions for the establishment, maintenance and sampling of the lysimeters are located in Appendix A.

The scientific objectives of this study are to:

1. Study long-term radionuclide transport under conditions more representative of vadose zone conditions than laboratory experiments.
2. Provide more realistic quantification of radionuclide transport and geochemistry in the vadose zone, providing better information pertinent to radioactive waste storage solutions, than presently exists.
3. Reduce uncertainty and improve justification for geochemical models such as those used in performance assessments and composite analyses.

2 PLANNING AND CONSTRUCTION

2.1 Site Planning

The lysimeter project team selected a site near the A-01 Constructed Wetland (Figure 1). The advantages of this location include SRNL custodianship,proximity to the SRNL Technical Area and a non-remote worker location.

In accordance with SRS Environmental Compliance Manual 3Q, potential environmental implications of the project were evaluated (EEC TC-A-2011-0051); DOE-SR determined that the project was categorically excluded (CX B3.1) from additional NEPA documentation.

The project team obtained a Site Use Permit (SU-11-08-O) and a Site Clearance Permit (6659) in accordance with SRS Site Infrastructure and Services Manual 1D, Procedure 3.02, Site Real Property Configuration Control.

SRS Design Engineering staff developed the site plan (C-CT-A-00045; Figure 2) and designed the modified roll-off pans (C-SK-A-RC001; Figure 3) to contain the lysimeter experiments. Baker Waste Equipment built the roll-off pans in accordance with the design sketch.

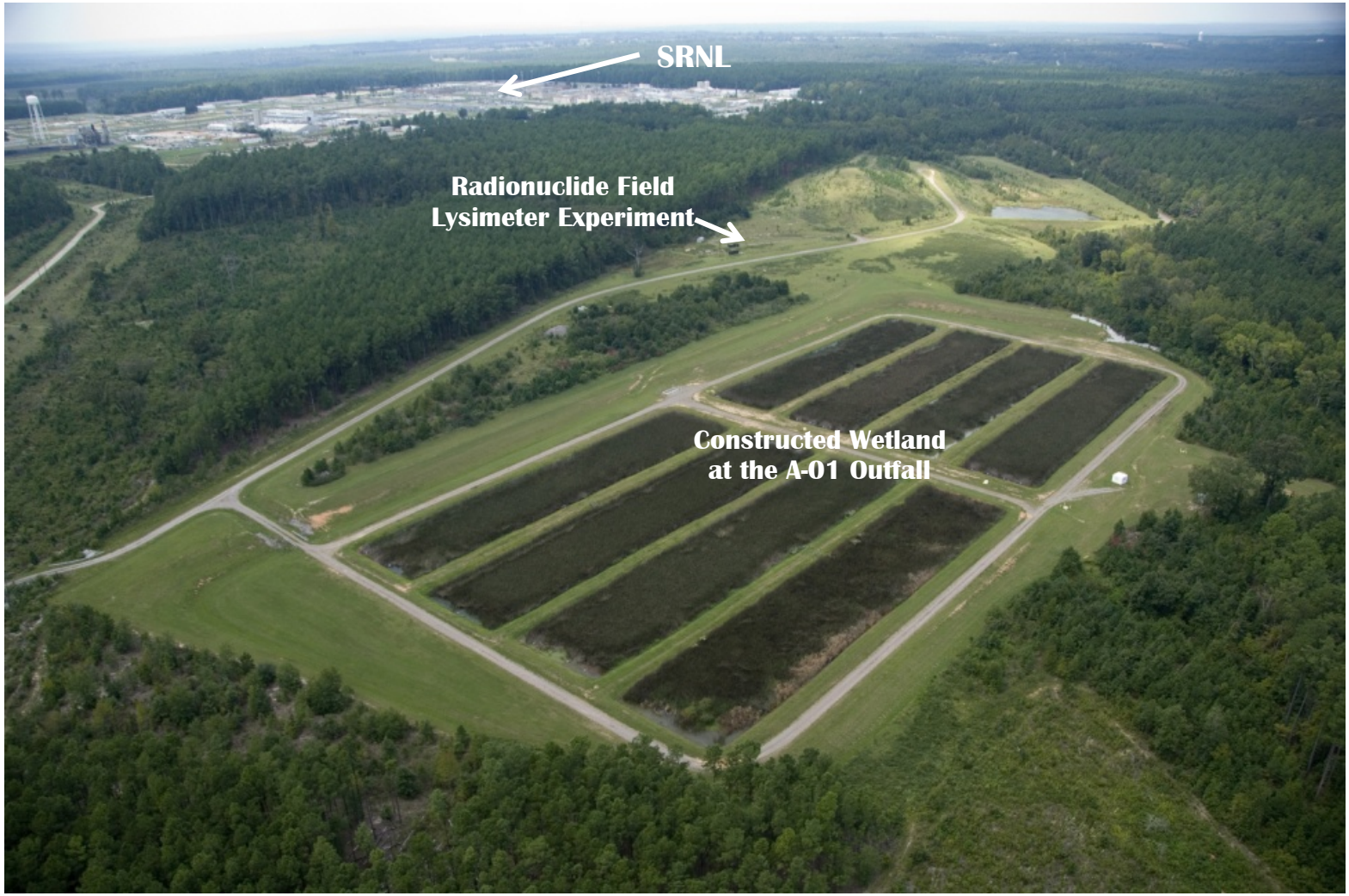

Figure 1 Aerial photo showing the lysimeter site relative to A-01 Constructed Wetland and SRNL Technical Area.

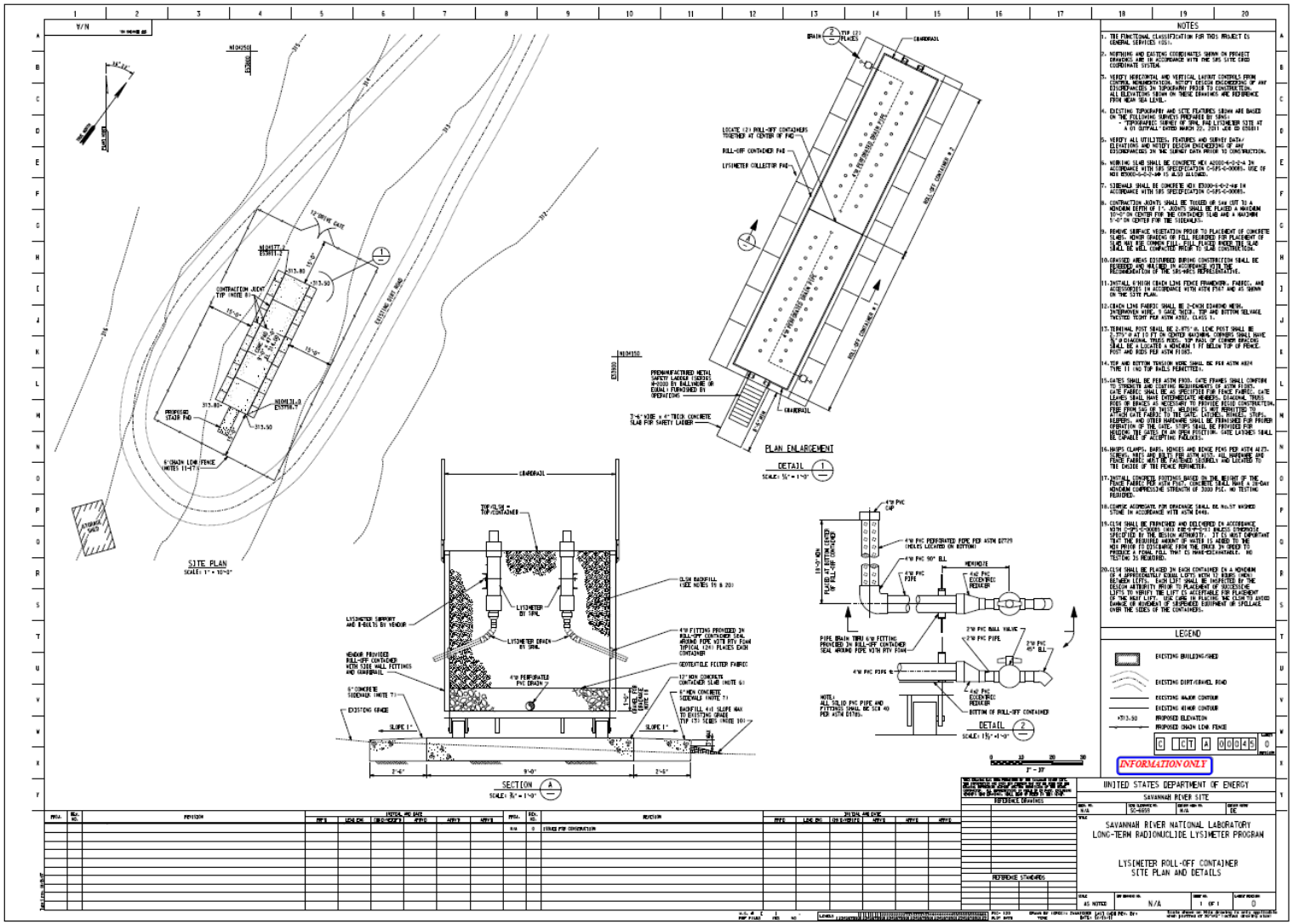

Figure 2 Lysimeter Roll-off Container: Site Plans and Details, C-CT-A-00-45

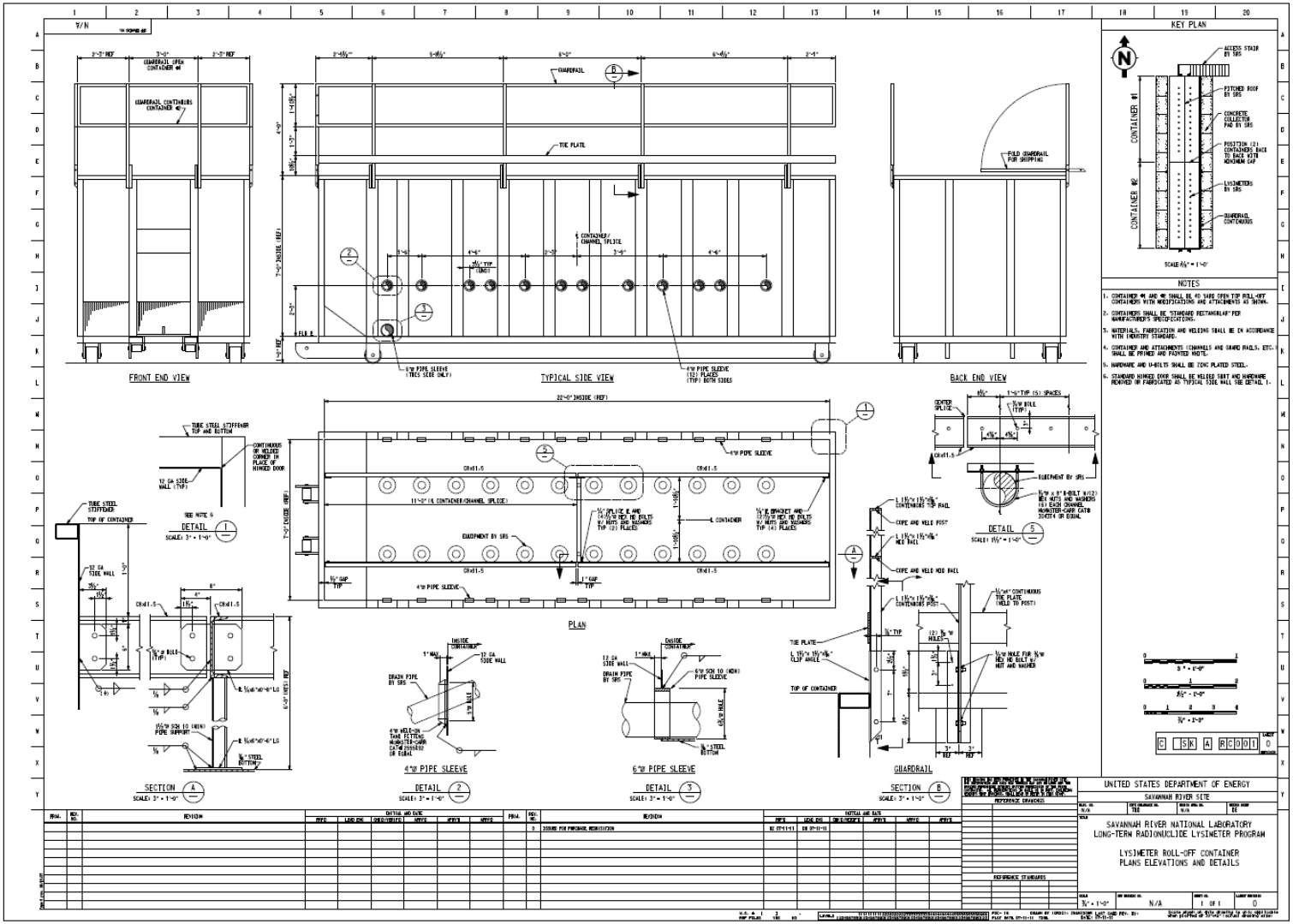

Figure 3 Plans, Elevations and Details, C-SK-A-RC001

2.2 Site Construction

SRS Construction poured concrete pads to support the roll-off pans and installed portable safety stairs. During construction, temporary scaffolding (Figure 4) provided access to the interior of the containers. Construction personnel installed a drainage system (perforated pipe in gravel, covered with filter fabric) in the bottom of each roll-off pan. After the lysimeter apparatus had been installed, the roll-off containers were backfilled with Controlled Low-Strength Material (CLSM; Figure 5), placed in successive lifts allowing each lift to set before adding more CLSM.

Figure 4 Lysimeter facility during construction with scaffolding for access to container interior

Figure 5 Backfilling the lysimeter pans with CLSM

3 LYSIMETER CONSTRUCTION AND INSTALLATION

The SRNL-Clemson team installed the field lysimeters on SRS property near the A-01 Outfall, located about 1 km from the SRNL. Lysimeters which were constructed at Clemson University were transported to SRS and installed over a three day period in the roll-off pans already in place. Three graduate students (Emerson, Hixon, Jablonski) and two undergraduate students (Waterhouse and Buchannan) assisted with the construction and installation of the lysimeters.

3.1 Lysimeter Design

Each lysimeter is a 24" x 4" (ID) PVC pipe. The pipe has a 4" to 2" reducer on one end which holds in place a perforated polypropylene grid supporting a nylon mesh screen (80 x 80 mesh, McMaster Car part # 9318T17). The grid and screen hold sediment in place during the experiment. The 4" x 2" reducer provides a junction to a 2" bushing with a ¾" barbed nipple. Nylon Tygon tubing is attached to the nipple to facilitate transport of the effluent water into a collection bottle. This lysimeter setup described above is housed within secondary containment consisting of 6" diameter PVC pipe with a 6" x 4" reducing bushing on one end. A short section of 4" PVC glued into the bushing is coupled to a 4" x 2" reducer and subsequently to a 2" PVC pipe used for effluent. In order to direct the effluent tubing out of the roll-off pan, a 45^{o} PVC elbow is used. The flexible Tygon tubing is passed through the inside of the 2" PVC from the secondary containment (Figure 6). The secondary containment serves several purposes. First, the annular space provides a means of draining the lysimeter in the unlikely event the lysimeter becomes inundated with water. Second, the PVC provides physical protection to the actual lysimeter. Third, this configuration creates a modular design in which the extraction and/or replacement of the lysimeters can be easily accomplished as well as installation of new lysimeters. Fourth, double containment is a typical practice for radiological protection.

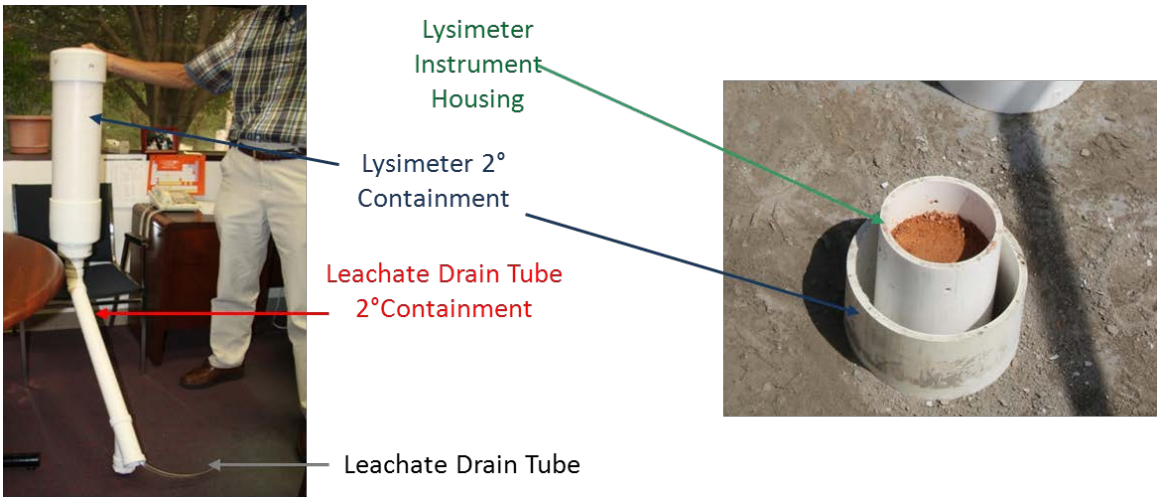

Figure 6 Lysimeter components

The soil used to fill the lysimeters is from the Dry Branch Formation (+/- Upland Unit) obtained from SRS's Central Shops Borrow Pit (Figure 7); these mixed sand and clay soils are representative of the vadose zone environment across much of SRS, including the low-level waste disposal areas. The lysimeters were packed in 2" lifts to halfway (10") with 1 x 1 cm sieved soil. After each lift the lysimeter was tapped a minimum of 10 times on a concrete block to facilitate settling. If needed, the top layer of soil was "roughened" with the end of a trowel to provide a better "junction" with the next lift of soil. A photo of soil packing and height testing is shown in Figure 8. Characterization of the soil is tabulated in Table 1

Figure 7 Map indicating the location of the central shops borrow pit where lysimeter soil was collected.

Table 1 Characterization of Soil from Central Shops Borrow pit used in the Radionuclide Field Lysimeter Experiment

	Analysis	Lysimeter Soil	Lysimeter Soil +OM[3]	OM[3]
	Total % C	0.085	1.704	13.750
	Total % N	0.008	0.062	0.456
	Total % S	0.0121	0.0184	0.0575
	LBC [1] (ppm $CaCO_3$/ pH)	150	390	
	pH $_{CaCl2}$ [2]	4.67	4.29	
	Equiv. water pH	5.27	4.89	
	% Base Saturation	27.62	14.21	
	CEC (meq/100g)	1.73	5.56	
Total Acid Digestion	**Al (ppm)**	12660	12750	
	B (ppm)	1.22	<1	
	Ca (ppm)	219	127	
	Cd (ppm)	0.98	1.71	
	Cr (ppm)	8.00	13.01	
	Cu (ppm)	<0.5	<0.5	
	Fe (ppm)	8101	13180	
	K (ppm)	129	130	
	Mg (ppm)	105	122	
	Mn (ppm)	7.29	7.72	
	Mo (ppm)	<0.5	<0.5	
	Na (ppm)	29.2	8.8	
	Ni (ppm)	<1	<1	
	P (ppm)	36.6	<6	
	Pb (ppm)	<2.5	<2.5	
	S (ppm)	112.2	111.5	
	Zn (ppm)	9.19	12.07	

1. Soil Testing: Measurement of Lime Buffer Capacity (http://www.caes.uga.edu/Publications/displayHTML.cfm?pk_id=7335)
2. Soil Testing: Soil pH and Salt Concentration (http://www.caes.uga.edu/Publications/displayHTML.cfm?pk_id=7336)
3. OM= organic matter; used for amendment of soil in lysimeters #21-23.

Figure 8 Photo of lysimeter soil packing

Two lysimeters (#24 and #37) were selected as controls. The controls have probes to measure soil moisture, temperature, and electrical conductivity. The probes have housing and wires which protrude out of the 4" lysimeter but would not fit inside the 6" lysimeter. Therefore, an 8" diameter PVC secondary housing was used instead of the 6" housing used for the other lysimeter columns. Each control lysimeter was fitted with three Decagon 5TM, measures moisture and temperature, and 5TE sensors, measures moisture, temperature and electrical conductivity, (Decagon Devices Inc., Pullman, WA). Three sensors were placed in each of the control 4" lysimeters. Three ~1" gaps were milled into each lysimeter at 8", 14" and 20" below the top of the lysimeter. The 5TM sensors were placed at the top (8") and the bottom (20") and a 5TE sensor was placed in the middle (14"). The gaps were covered with duct tape and the soil was packed into the lysimeter. Then the probes (each probe has three tapered prongs) were inserted into the lysimeter through the milled gaps. The probes were glued in place using Gorilla Glue. This is waterproof glue that worked well during prototype testing at Clemson University. The probes were glued into place and left for two days to cure. A photo of the control lysimeters during the glue curing stage is shown in Figure 9

Figure 9 Control lysimeters with decagon 5TM (top and bottom) and 5TE (middle) probes glued in place

3.2 Lysimeter Layout

The lysimeters numbered 1 through 48 for ease of identification and accounting during installation are shown in Figure 10. An arrow approximating true north is shown in the diagram below to indicate the orientation of the pans.

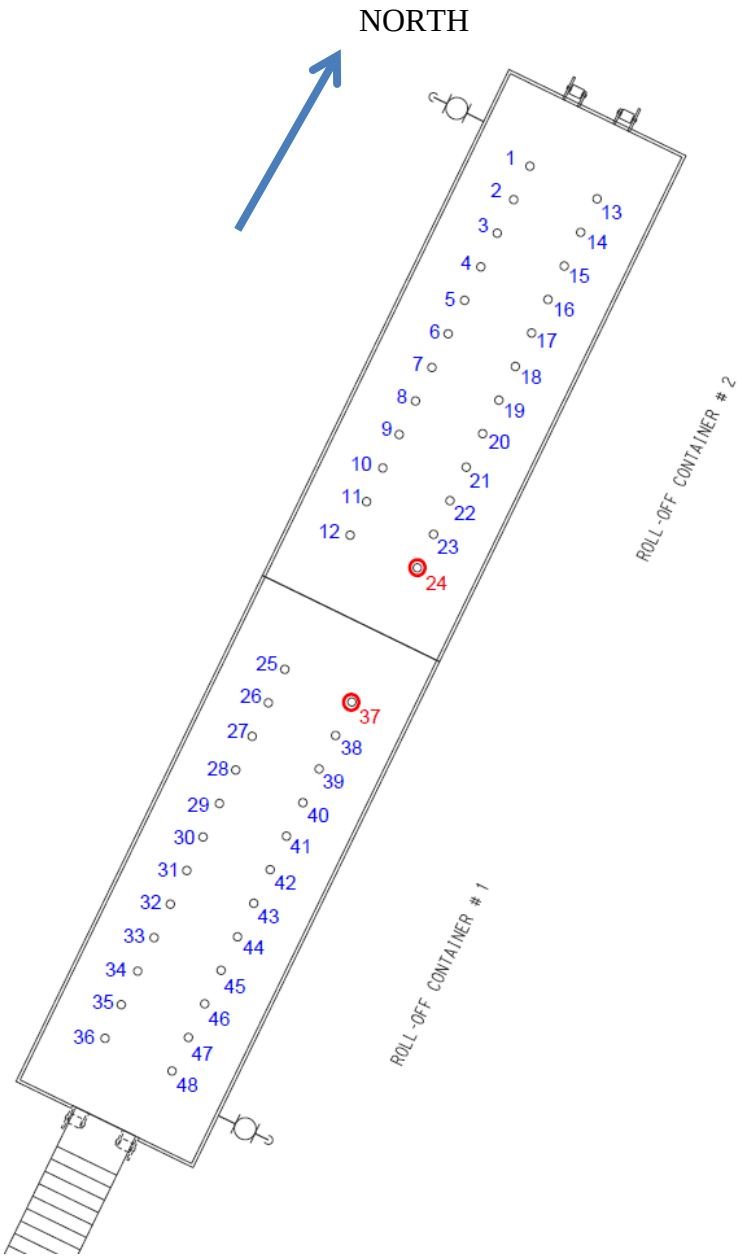

Figure 10 Numbering sequence and orientation of lysimeters within roll-off pans.

3.3 Flow Testing

The inner 4” lysimeter unit and the secondary 6” housing were both flow tested to ensure that the PVC connections were secure (Table 2). A 2” sealing cap was used to plug the effluent lines and enough water was added to fill the units above all of the joints. The water was left in the unit for at least 30 seconds and then the joints were inspected for potential leaks. This took approximately 2L of water for the 4” lysimeters and 10L of water for the 6” secondary housing. The goal was to test a minimum of one third of the lysimeters (i.e., 16 out of 48). The testing results are listed in Table 1 below. On one of the 4” lysimeters water was observed at the joint. The testing was being performed in the early morning when morning dew had covered the lysimeters prior to testing. It was determined that the water observed at the joint was condensation and not from the inner water during the testing. Therefore, all 4” lysimeters that were tested passed. One secondary 6” housing failed testing as indicated by a small leak of two drops per minute from the 45^{o} elbow of the effluent PVC pipe. The housing was removed from the roll-off pan and reglued. After the glue set, the lysimeter was tested again and passed. Based on these actions, all lysimeters and secondary housing units which were tested passed the flow test.

Table 2 Lysimeter Flow Testing Results

Lysimeter #	4" Inner Lysimeter	6" Secondary Housing
1	passed	not tested
2	not tested	passed
3	not tested	not tested
4	passed	passed
5	passed	passed
6	not tested	passed
7	not tested	passed
8	passed	passed
9	not tested	passed
10	not tested	passed
11	not tested	passed
12	passed	passed
13	not tested	not tested
14	not tested	not tested
15	not tested	not tested
16	passed	not tested
17	not tested	not tested
18	passed	not tested
19	passed	not tested
20	passed	not tested
21	passed	not tested
22	passed	not tested
23	passed	not tested
24 (Control)	passed	passed
25	passed	passed
26	passed	Initial fail (passed after regluing)
27	passed	passed
28	passed	not tested
29	passed	not tested
30	passed	passed
31	passed	not tested
32	passed	passed
33	passed	not tested
34	passed	passed
35	passed	passed
36	passed	passed
37 (Control)	passed	passed
38	passed	passed
39	passed	passed
40	passed	not tested
41	passed	passed
42	not tested	passed
43	not tested	not tested
44	not tested	not tested
45	not tested	not tested
46	not tested	passed
47	not tested	passed
48	not tested	passed
Number Tested	30	27
Percent tested	63%	56%

3.4 Lysimeter Installation

Two I-beams were welded in place down the center of each roll-off pan. The lysimeters were installed by first bolting the 6” diameter secondary housing to the I-beam using an 8” U-bolt. The U-bolt threads were too short to fully tighten the bolt around the lysimeter. Therefore, 12” long 2”x4” boards were cut and holes were drilled through for the bolt to pass through. Essentially, the 2”x4” board served as a large washer to fix the secondary housing in place. A photo of the secondary housing being lifted into the roll-off pan and the first two units bolted in place is shown in Figure 11.

Figure 11 Transfer of secondary housing into roll-off pan (top left and right). The first two secondary housing units in place with effluent plumbing exiting container wall shown (bottom).

After the secondary housing was bolted into place, the 4” lysimeter was installed. In order to install the ¾” Tygon tubing, the tubing had to be passed into the roll-off pan by pushing it through the effluent 2” pipe of the secondary housing. The tubing was pushed through and an excess amount was pulled out of the top of the 6” secondary housing. The tubing was then pushed onto the ¾” barbed nipple up to the final barb. Then an 8” zip tie was secured around the tubing to provide additional pressure on the tubing to keep it attached to the barb. The lysimeter was then lowered down into the 6” secondary housing and the flexible tubing was pulled back out by a worker on the outside of the roll-off pan. Photographs of this process are shown in Figure 12. A 6” diameter cap with a 4” diameter hole drilled into the top was placed on top of the 6” secondary housing leaving the 4” lysimeter exposed. The length of the secondary housing and

4” diameter lysimeter are such that the top 0.5 cm of the lysimeter fit into the 4” diameter hole drilled in the cap.

The control lysimeters had to be installed in a slightly different manner. The lysimeter was brought into the pan and connected to the ¾” Tygon tubing as described above. Then as the unit was lowered, care was taken to prevent the probe wires from impacting the walls of the 8” diameter secondary housing. An 8” cap was required due to the 8” diameter secondary housing of the controls. Three holes were drilled in the cap to pass the wires through so that they could be connected with the Decagon datalogger. The holes were labeled top, middle, and bottom. Also, each cable was labeled near the base of the probe and near the input to note the top, middle, or bottom probe. The labels were covered with clear packing tape to prevent water damage. A photo of the control lysimeter installation is shown in Figure 13. In order to protect the cables as the backfill material is placed in the roll-off pan, the cap was removed and the wires were stored inside the secondary housing. After the roll-off pans were filled, the cap was replaced and the wires were pulled through the cap as shown in the photo in the bottom right of Figure 13.

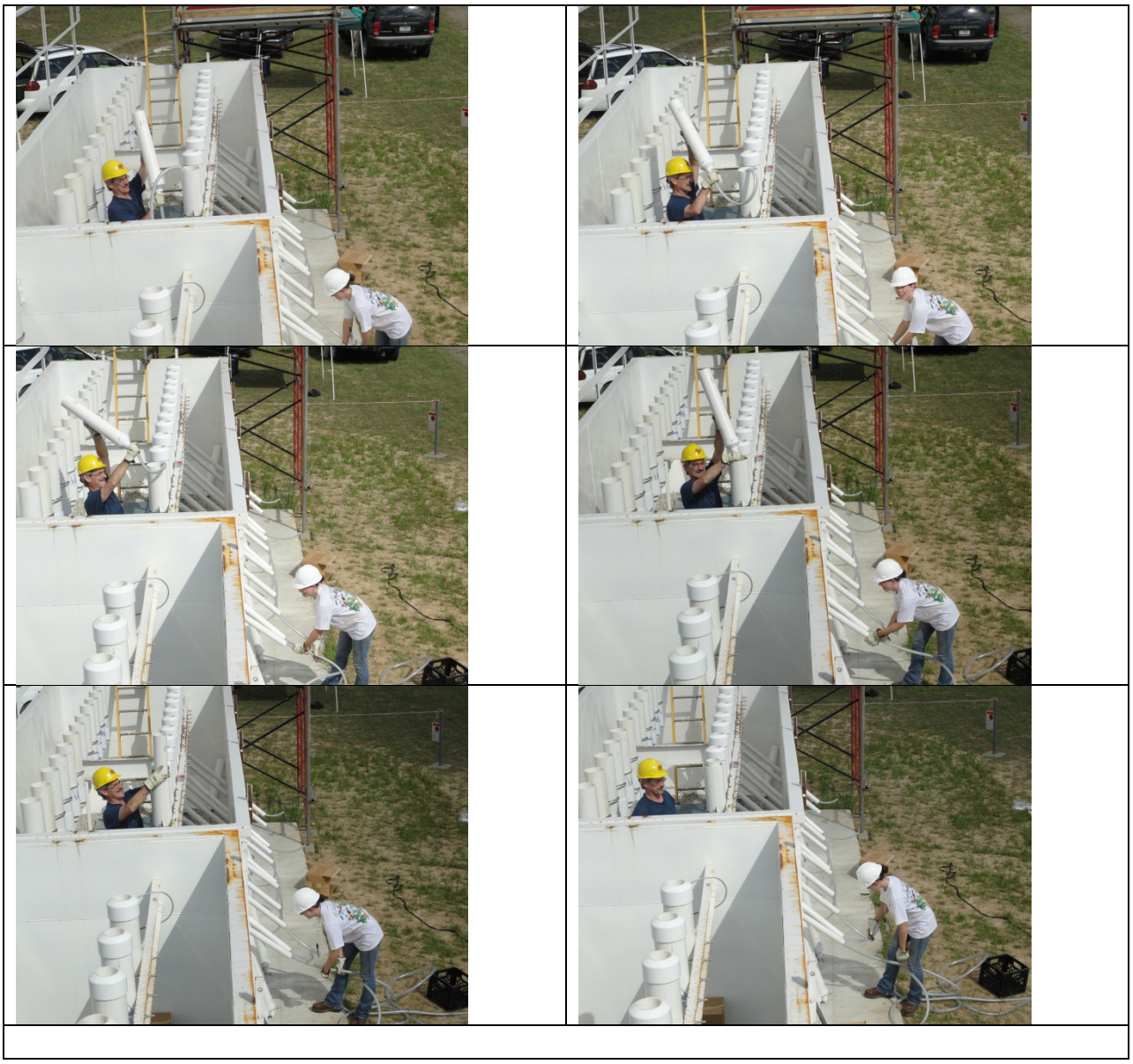

Figure 12 Picture sequence of lysimeter installation into secondary housing.

Figure 13 Installation of control lysimeters. Top right photo shows wires coming out the top of the 8" diameter secondary housing. The bottom right photo shows the cap installed with the wires coming out of the respective holes. The wires were stored in the secondary housing for protection.

4 RADIOACTIVE SOURCE PREPARATION AND INSTALLATION

4.1 Description

A total of 39 radioactive sources were prepared in the laboratory for installation in the Radionuclide Field Lysimeter Experiment. There were two physical forms utilized as radiological sources: filter "pita pockets" and cementitious pucks. A Whatman 47mm GFF (glass fiber filter) was used for the pita pockets solely as a means of material introduction. These filters were selected to be chemically inert and intended to have limited physical interference and therefore representative of soil contamination. The cementitious puck, either cement or saltstone (cement with reducing slag), was used to study behavior in commonly used solid wasteforms. In addition to the different nuclide treatments, three lysimeters (9-12 plus control 13) will have grass planted to study the effect of vegetation on plutonium mobility and three lysimeters (21-23) contain soil amended with 3% organic matter to assess the reducing ability of organic matter and the effect on plutonium mobility.

4.1.1 Filter "Pita Pockets"

The glass fiber filter pita pockets were fabricated by placing two filters on top of each other and stitching the sides with Teflon thread (dental floss). Liquid radionuclide standards (Tc-99, Ba-133, Co-60, Cs-137, Eu-152 and stable iodine were pipetted to the inside of the top pocket filter. More detail on the preparation of the filter pita pocket radiological sources is located in Appendix B. Figure 14a shows a sample filter in a half-filled mini-core delivery system used for the Pu and Np compound containing filters.

4.1.2 Cementitious Pucks

A grout puck weighing approximately 17.6 g with a diameter of 1.25" and thickness of 0.5" was used in each of the lysimeters containing either the beta (Tc and I) or gamma suite (Ba, Co, Cs, and Eu). The cementitious material was either cement or slag-containing saltstone simulant. Recipes and methodology for making the pucks are found in Appendix C. Figure 14b displays a top down view of a puck in place in a half-filled lysimeter.

a.

b

Figure 14 Two types of radioactive sources for Radionuclide Field Lysimeter Experiment. a. Filter pita pocket in half filled mini core delivery system. b. Top view of lysimeter with a cementitious puck centered in the lysimeter before the top half is filled with soil.

4.1.3 Radionuclides

There were several groups of radiological material used in the lysimeters. The cationic gamma suite consisted of Cs-137 (as cesium chloride), Co-60 (as cobalt chloride), Ba-133 (as barium chloride), and Eu-152 (as europium chloride) These radionuclides were chosen because they were a cost effective means of evaluating the behavior of monovalent, soft divalent, divalent and trivalent cations. The results from these selected nuclides can also be used to predict behavior of other similarly charged elements. The anion group consisted of Tc-99, as ammonium pertechnetate, and stable I, as KI. Activities utilized for each lysimeter are listed in Table 3. Pu oxidations states of III, IV, and V, and Np oxidation states of IV and V were used in the following chemical formulations: Plutonium(III) oxalate, Plutonium(IV) oxalate, Ammonium Pu(V) carbonate, Pu(IV) colloids, Neptunium(IV) oxide, and Neptunium(V) nitrate. The gamma suite radionuclides and the Tc-99 were procured form Eckert and Zeigler as nominal radionuclide standards. Reagent grade KI was dissolved in deionized water as the stable iodine source. The Pu and Np compounds were provided by Eddie Keyser, Mike Bronikowski, and Philip Almond of SRNL. Details regarding the preparation of these compounds are located in Appendix C.

Table 3 Radioactive source activities in each lysimeter instrument in their respective locations in the lysimeter containment units. For descriptions containing "cement" or "saltstone", the sources were in the form of cementitious pucks. For all other samples, filter pita pockets were used. Lysimeters 9-12 will have grass planted and 21-23 will have soil amended with organic matter. Note that Pu and Np compound activities are approximates as the transfer could not be quantified during glovebox operations.

Lysimeter No.	Description	uCi	Lysimeter No.	Description	uCi
1	Empty	-	13	Empty	-
2	Cement Control	0	14	Saltstone Control	0
3	Cement Control		15	Saltstone Control	
4	Cement gamma Suite	110 ^{137}Cs; 83 ^{60}Co; 94 ^{133}Ba; 221 ^{152}Eu	16	Saltstone gamma suite	110 ^{137}Cs; 83 ^{60}Co; 94 ^{133}Ba; 221 ^{152}Eu
5	Cement gamma Suite		17	Saltstone gamma suite	
6	Cement gamma Suite		18	Saltstone gamma suite	
7	Cement Tc & I	261 ^{99}Tc; 0.07 mM I	19	Saltstone Tc & I	261 ^{99}Tc; 0.07 mM I
8	Cement Tc & I		20	Saltstone Tc & I	
9	Pu(IV)-oxalate, grass	187 ^{239}Pu; 105 ^{240}Pu; 6210 ^{241}Pu	21	Pu(V)NH4(CO3)/OM	282 ^{239}Pu; 1050 ^{240}Pu; 1600 ^{241}Pu
10	Pu(IV)-oxalate, grass		22	Pu(V)NH4(CO3)/OM	
11	Pu(IV)-oxalate, grass		23	Pu(V)NH4(CO3)/OM	
12	Grass control		24	Instrumented control	
25	Sediment Control		37	Instrumented Control	
26	gamma Suite	7.9 ^{137}Cs; 8.6 ^{60}Co; 6.7 ^{133}Ba; 12.7 ^{152}Eu	38	Pu(IV)oxalate	187 ^{239}Pu; 105 ^{240}Pu; 6210 ^{241}Pu
27	gamma Suite		39	Pu(IV)oxalate	
28	gamma Suite		40	Pu(IV)oxalate	
29	Np(V)nitrate	1.7 ^{237}Np	41	Pu(V)NH4(CO3)	362 ^{239}Pu; 92 ^{240}Pu; 3210 ^{241}Pu
30	Np(V)nitrate		42	Pu(V)NH4(CO3)	
31	Np(IV)02	1.9 ^{237}Np	43	Pu(V)NH4(CO3)	
32	Np(IV)02		44	Pu colloids	176 ^{239}Pu; 45 ^{240}Pu; 1560 ^{241}Pu
33	Pu(III)oxalate	122 ^{239}Pu; 60 ^{240}Pu; 3550 ^{241}Pu	45	Pu colloids	
34	Pu(III)oxalate		46	Pu colloids	
35	Pu(III)oxalate		47	Empty	-
36	Empty	-	48	Empty	-

4.2 Archived Radiological Sources

Duplicate samples were made and stored in two nitrogen purged vessels (Appendix E). One vessel contained non-radioactive cementitious material of each batch and the second vessel contained duplicate radioactive sources of saltstone, cement and beta suite in a filter pita pocket. Vessels containing oxygen scrubber pads and a colorimetric oxygen indicator were purged with nitrogen. Vessels will be monitored for any change in the oxygen indicator color and re-purged with nitrogen gas as necessary. The archived samples represent the initial (t_0) conditions of the sample. Preservation in an inert environment (nitrogen) will limit redox reactions of the redox sensitive Pu and Np, especially in the saltstone.

Figure 15 Inert environmental chambers for archiving lysimeter sources both non-radiological (left) and radiological (right - samples not yet loaded). The blue and white packets are oxygen scrubbers and the pink dot visible on the right is the oxygen indicator which is pink in the absence of oxygen and turns blue when oxygen present.

4.3 Radiological Source Deployment

Radiological sources were deployed in several stages. All sources were centered within the 4" lysimeter. During the handling of the pita pockets containing Pu and Np compounds a potential for contamination was identified. This is likely due to the activities used (~1 – 6000 µCi), the possibility for these alpha emitters to migrate through the porous filters, or transfer of material within the glovebox used for preparation

The delivery system is pictured in Figure 14a and described in Appendix E. Upon placement of the sources in the lysimeter, soil was packed in a series of lifts in a manner consistent with the initial packing.

5 MAINTENANCE AND MONTORING

5.1 Atmospheric Technologies Database

Rainfall and air temperature monitored by SRNL Atmospheric Technologies was extracted from their database. Data from the "SRTC" station was used, which is acquired from an instrument shelter located near 773-A, 1 km from the Radionuclide Field Lysimeter Experiment.

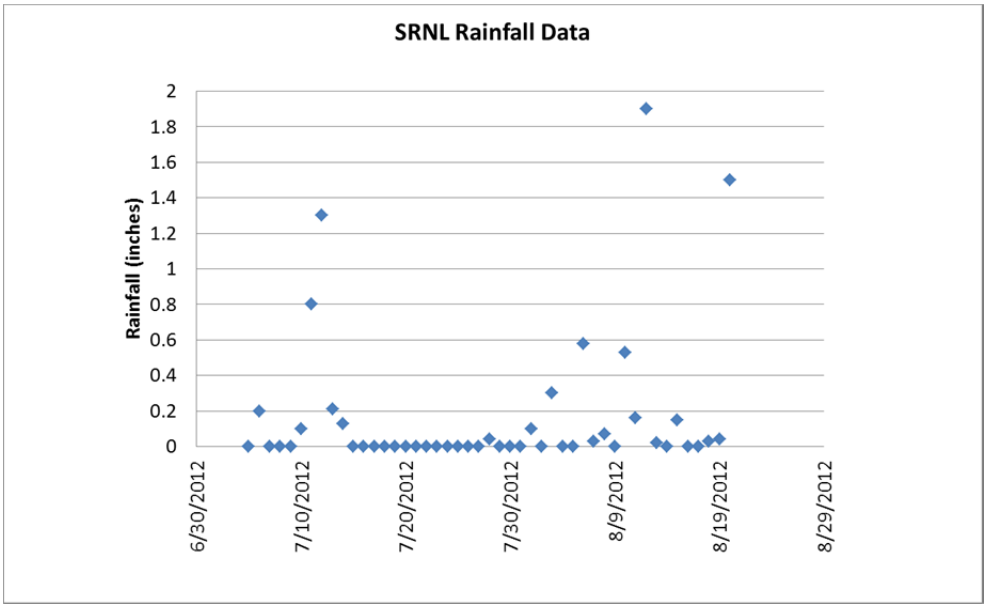

Figure 16 Atmospheric Technologies database rainfall data for "SRTC" station, located near 773-A.

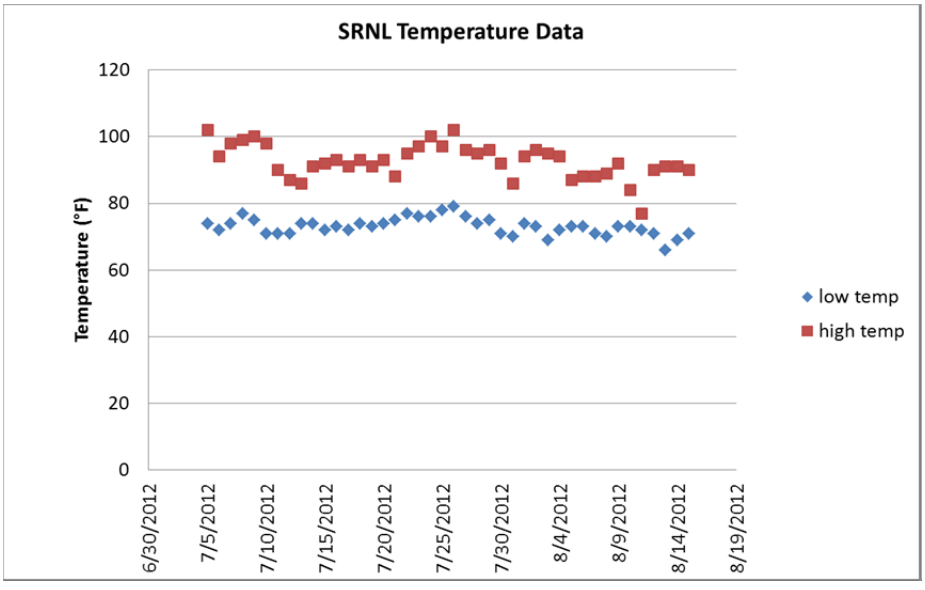

Figure 17 Low and high air temperature for SRNL station from Atmospheric Technologies database.

5.2 Leachate Capacity

Bagwell et al. (2010) calculated the 100-year rainfall volume collection for each lysimeter to be 1.8L based on a catchment area of 12.5 in^2 and a 24 hour rainfall of 8 in. Therefore, primary leachate collectors of 2 L were used.

5.3 Rainwater Mass Balance

A water mass balance of the system will be calculated as the system is monitored. A simple box model can be used for this calculation, where: rainfall = sample + overflow + evaporation. Rainfall data was collected at a location 1km from the Radionuclide Field Lysimeter and stored in the SRNL Atmospheric Technologies database. Evaporation will be calculated by temperature data alone using the Penman formula (Equation 1) described by Linacre (1977). The calculated value will be compared with the value based on measured difference.

Equation 1 Penman Equation for Evaporation

$$E_o = \frac{\frac{700 * T_m}{100 - A} + 15(T - T_d)}{80 - T}$$

where:

T_m= T+ 0.006*elevation
T= mean temperature
A= latitude (degrees)
T_d = mean dewpoint

The sample and overflow volumes will be calculated by converting measured weights of the solution in the sample bottle (collecting leachate that passed through individual lysimeters) and the overflow bottle (collecting water that entered between the 4 inch diameter and 6 inch diameter pipes through the “overflow holes” located 1 inch above the soil layer) (Figure 18).

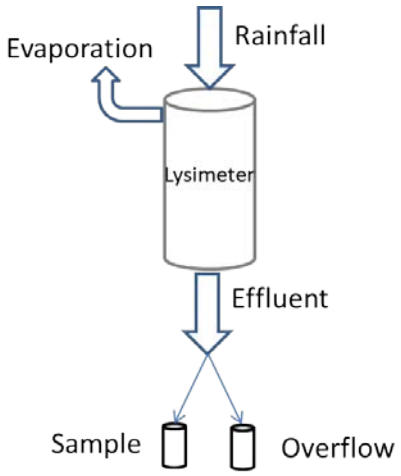

Figure 18 Pictorial representation of water mass balance.

5.4 Decagon Devices – soil sensors and data logger

Decagon Devices, Inc., (Pullman, WA) equipment was used to measure soil parameters in control lysimeters – on in each container (Figure 19). Two Em50 series Data Loggers were installed. One instrumented control was located in each of the roll off pans (lysimeter numbers 24 and 37) noted by a red circle in Figure 10. In each of the instrumented control lysimeters a 5TE sensor, measuring soil temperature, moisture and electrical conductivity, were placed in the middle of the lysimeter and one was placed in the ground approximately 1' from the concrete pad and 1' below grade. 5TM sensors, measuring soil temperature and moisture, were placed in the top and bottom of the instrumented control lysimeters. An example of the output is presented in Figure 20.

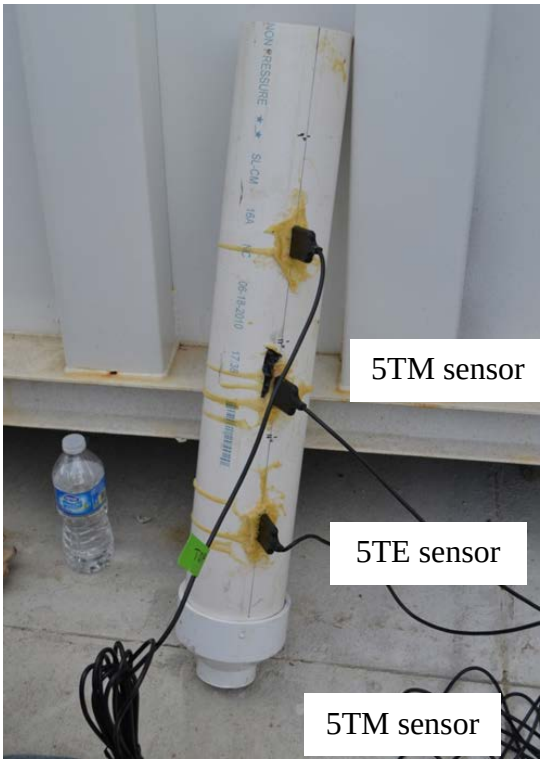

Figure 19 **Control lysimeter with soil parameter sensors. 5 TM sensor measures soil temperature and moisture and 5 TE sensor measures soil temperature, moisture and electrical conductivity.**

The system will be monitored monthly or as deemed necessary based on weather conditions and experimental results. Monitoring will include visual inspection of the entire site including: the top of the containers, the sample bottle housings, pan drains and sample and overflow bottles. The volumes of the sample and overflow bottles will be measured via bottle weights using a field scale. A water mass balance will then be calculated by comparing the rainfall to the sum of the sample bottle, overflow bottle and evaporation rate. Samples are scheduled to be taken quarterly. Sample bottles will be replaced by new ones, capped and analyzed. Overflow water will be rad screened by Radiological Protection Department (RPD) and disposed of accordingly.

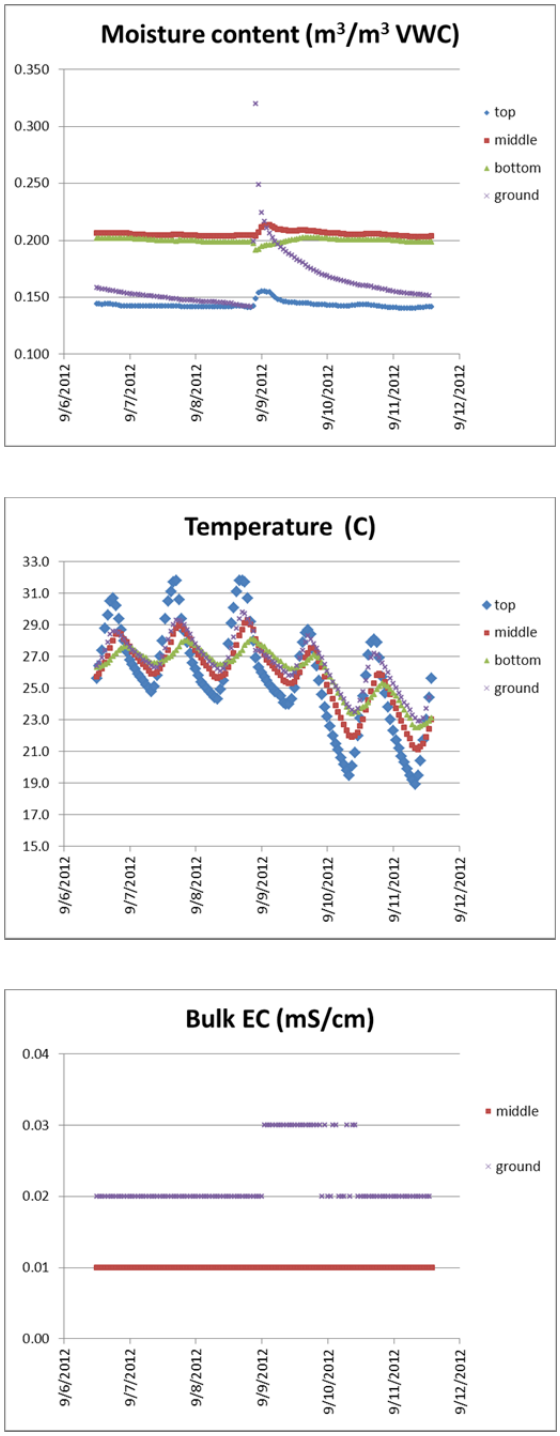

Figure 20 Soil moisture, temperature and electrical conductivity measured from a 5TE electrode recorded to a Decagon Devices data logger from the control lysimeter in the south lysimeter container.

6 REFERENCES

Bagwell, L., Kaplan, D., Roberts, K., Molz, F., Powell, B., 2010, Ten-year Radiological Lysimeter Experiment at Savannah River Site – Documentation to Accompany Environmental Evaluation Checklist, SRNL-TR-2010-00208, Rev. 2, Savannah River National Laboratory, Aiken, SC.

Linacre, E.T. 1977. A simple formula for estimating evaporation rates in various climates using temperature data alone. Agricultural Meteorology. 18(6), 409-424.

APPENDIX A: Work Instructions for establishment, maintenance and sampling of lysimeters near the A01 outfall

INTEROFFICE MEMORANDUM

April 25, 2011 SRNL-L3500-2011-00001 rev.1

TO: D.I. Kaplan, 773-43A
FROM: K.A. Roberts, 773-43A

WORK INSTRUCTIONS FOR ESTABLISHMENT, MAINTENANCE, AND SAMPLING OF LYSIMETERS AT A01 OUTFALL

Scope

These work instructions cover the establishment, maintenance, and sampling of 10-year lysimeters located near the A01 outfall (Figure 1). Detailed R&D Directions and/or worker checklists will be developed prior to establishment.

Equipment

Establishment
Radioactive sources

Maintenance
Replacement components for lysimeter e.g. tubing etc.

Monitoring
Nalgene bottles (2L)
PPE (gloves; thin mil nitrile or rad equivalent)

Safety

SRNL Radiological Protection Department (RPD) personnel will be present during the deployment of the radioactive sources in each lysimeter. PPE (as determined by RPD for incidental contact) will be utilized. During leachate quarterly sampling, PPE, as determined by HP/RPD will be utilized. If RPD deems SRWP 001 to be insufficient a new SRWP will be developed by RPD and signed in on by all workers. Although the site is not considered a remote location, workers will work in pairs and bring a two-way radio.

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Work Instructions

Establishment

The lysimeters will be constructed and moved to the experiment site (Figure 1). The lysimeters will be prepared at the experiment site by packing halfway (12") with SRS soil, placing the radioactive sources (pre-loaded on filter paper or other solid media) atop the 12" soil column, then filling the remaining 12" with SRS soil.

Monitoring

The lysimeters will be monitored at a frequency of no less than once per month. This will involve examining the site for physical damage or degradation of materials, weed removal and general housekeeping of the site. A worker checklist will be developed prior to commencement of monitoring activities. Weeds will be removed by hand and disposed of according to RPD specifications.

Sampling

Leachate sampling will occur on a quarterly basis or more frequently as needed for specific radionuclides and or as necessitated by rainfall.

When there is sufficient (actual volume TBD) aqueous sample collected in the sample bottle, the bottle will be changed as follows:

1. 2 L wide-mouth Nalgene containing nitric acid (as preservative) will be brought to the experiment site.
2. Unscrew lid of the bottle attached to the lysimeter.
3. Place and tighten cap from lysimeter on the new bottle.
4. Place and tighten cap from new bottle on the sample bottle.
5. Return samples to 773-A for analysis.

Cores taken from lysimeters for sediment analyses will follow EAS Work Instructions WI-EAS-0004 Rev. 1

cc: Dave Crowley, 773-43A
Laura Bagwell, 773-42A

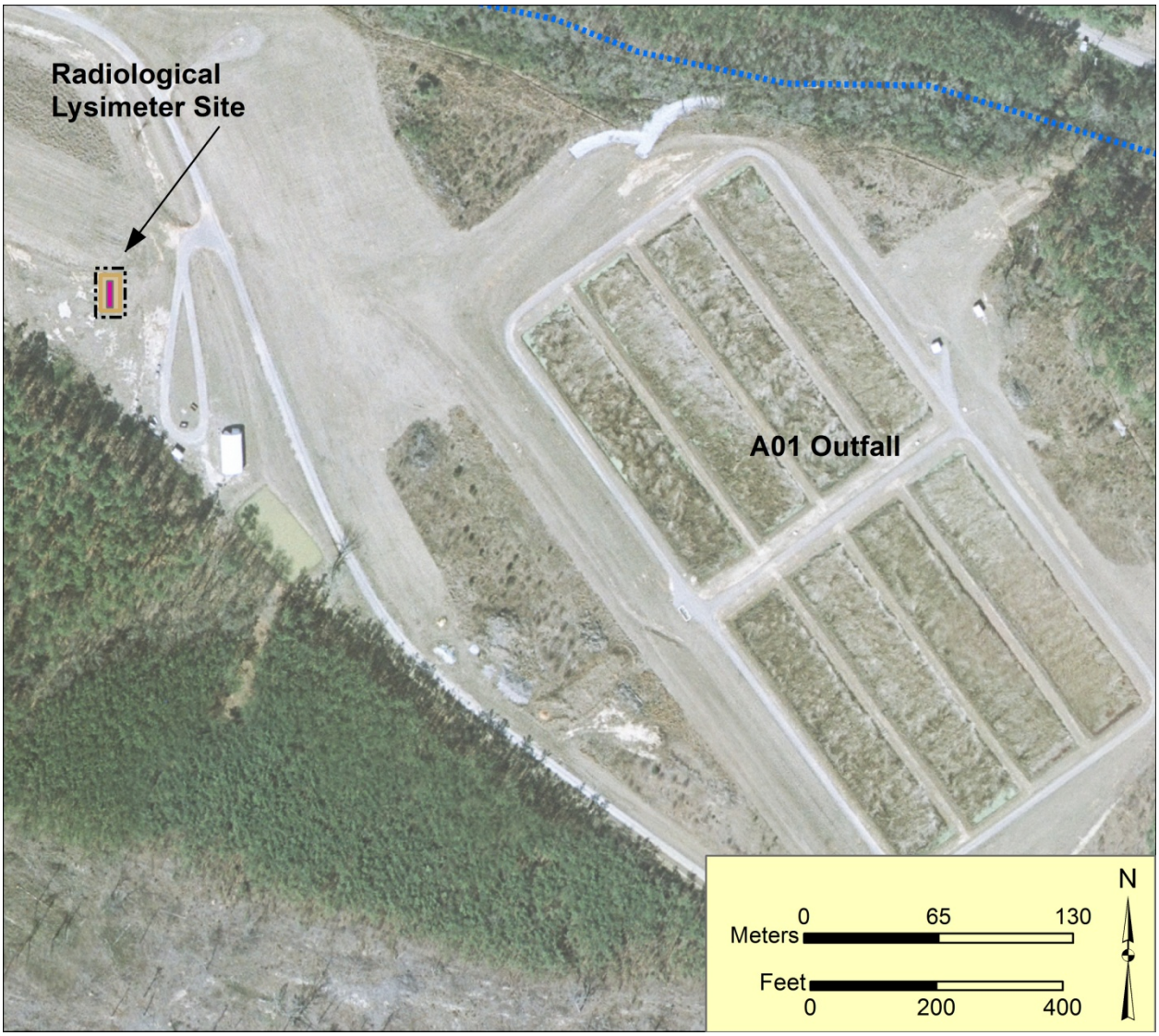

Figure 21 Location of Radiological Lysimeter Site near A01 outfall

APPENDIX B: R&D DIRECTIONS: LYSIMETER RAD SOURCE PREPARATION AND INSTALLATION

R&D Directions:
Lysimeter Rad source preparation and installation

Kim Roberts & Dan Kaplan
3/30/12

Objective:

To prepare radioactive sources and install sources in the Lysimeters located near the A01 outfall site. Three types of sources will be used: filter paper, saltstone, and concrete.

HAP: SRNL-L3100-2009-00216-2
SRNL-L3500-2010-00011-0

Hazards: Radioactive Material

Hazards Mitigations: proper rad worker qualifications, PPE and RPD support when needed

Materials:

Radioactive materials:
- Standard solutions (Cs-137, Co-60, Ba-133, Eu-152, , Tc-99)
- Pu and Np compounds

Filter pita pockets (47mm GFF)
Glide Unwaxed dental floss
Filter holders (petri slides or petri dishes)
Saltstone ingredients
Concrete ingredients
Mold for cementitous materials
Pipettes

Methods:

1. **Prepare filter "pita pockets":** sew around the edge of two 47mm GFF(glass fiber filters) using unwaxed dental floss leaving a small opening for introduction of source. Pinch pocket once or twice to relax filter and allow easy access to interior (especially important for samples to be prepped in glovebox).
2. **Adding Liquid Spikes to Filter Paper:** Prepare standard solution dilutions for filter paper sources according to Table 1.
   a. Place filter in Millipore petri slide
   b. Pipette appropriate materials on filter trying to evenly disperse. Add dropwise to top of filter to prevent pooling up and potential of leaking through filter to petri.

c. Allow filter to dry in hood

3. **Preparing saltstone and concrete radiological sources:**
    a. Mix all dry ingredients
    b. Add calculated volume of appropriate liquid (water for cement and simulant waste for saltstone)
    c. Mix
    d. Transfer to rad hood, add radiological spike and shake
    e. Uncap and allow to set >1 week in fume hood.
4. **Adding Solid Spikes (Actinides) to Filter Paper:**
    a. As these samples are prepared in the glovebox utilize practices to minimize contamination such as using a separate plastic bag for each filter pita pocket and leaving the petri in the bag during transfer process
    b. Tap spatula in container with solid compounds (Pu or Np)
    c. Slide spatula into pita pocket opening, use petri cover to hold filter while removing spatula
5. Once radiological materials are ready for installation. Secure in transport containers (e.g. Millipore petri slides for filters, small plastic snap top containers for saltstone or concrete). Bag out sources from hood. Have RPD survey the material, package according to HMTR, Transportation services requirements and have RPD transfer survey completed.
6. For installation:
    a. Place each source into lysimeters already ½ packed with soil, try to center source in 4' tube.
        i. For saltstone/cement samples: remove lid from plastic container, lower container in lysimeter and invert releasing the puck. Use container to center if needed. Immediately cover with one beaker of dirt.
        ii. For filters (no rad dose): remove lid from petri inside the lysimeter. Lower the petri in lysimeter and invert releasing filter. Use petri container to center pita if needed. Immediately cover with one beaker of dirt.
        iii. For filters (with dose – Np and Pu): see R&D Directions: Pu and Np source delivery system preparation below.
7. Once completed, fill lysimeters with remainder of soil and pack. One beaker of dirt packed with 2" x 4" lumber until desired height from top of the tube.
8. RPD will then complete their Rad survey and post appropriately the area.

Table 4 Preparation scheme for radioactive standard solutions

Lysimeter Rad source preparation																	
decay corrected 03/29/12																	
rad dose survey 03/29/12																	
Filter paper source																	
	decay corr	stock	dose rate			for 1mL dilution				dilution dose rate				ml of dil			
	stock	volume	mrem/hr	mrem/hr	mrem/hr	dilution	vol stock	vol diluent	dil activity	mrem/hr	mrem/hr	mrem/hr	target activity*	needed	# of	total spike	
Isotope	uCi/mL		extremity	skin	WB	factor	(mL)	(mL)	uCi/mL	extremity	skin	WB	uCi	mL	treatments	vol	
^{137}Cs*	1080	0.979	44	10	10	15	0.07	0.93	72.00	2.93	0.67	0.67	7.9	0.11	4	0.44	
^{60}Co	1050	1	250	40	30	25	0.04	0.96	42.00	10.00	1.60	1.20	8.6	0.20	4	0.82	
^{133}Ba	938	1	66	14	6	15	0.07	0.93	62.53	4.40	0.93	0.40	6.7	0.11	4	0.43	
^{152}Eu	324	6	710	16	7	5	0.20	0.80	64.80	142.00	3.20	1.40	12.7	0.20	4	0.78	
^{85}Sr	2.07	0.945	2	1	1	1	1.00	0.00	2.07	2.00	1.00	1.00	0.05	0.02	4	0.10	
stable -I													10mM				
SUM			1072	81	54					161.33	7.40	4.67					
above SRWP radiation dose suspension guide limits (1000 extremity; 5WB)																	
*from Lysimeter list v5a.xls (gamma suite)																	

Cementitious source														revised				
			dose rate			target	vol stock				dose rate							
			mrem/hr	mrem/hr	mrem/hr	activity	needed	# of	total	spike	mrem/hr	mrem/hr	mrem/hr	activity	vol spike	# of	total spike	spike
Isotope	uCi/mL		extremity	skin	WB	uCi	mL	treatments	volume	remaining	extremity	skin	WB	uCi		treatments	volume	remaining
^{137}Cs*	1080	0.979	44	10	10	158	0.146	7	1.02407407	-0.11	6.44	1.46	1.46	126.4	0.117	7	0.82	0.09
^{60}Co	1050	1	250	40	30	172	0.164	7	1.14666667	-0.19	40.95	6.55	4.91	137.6	0.131	7	0.92	0.04
^{133}Ba	938	1	66	14	6	134	0.143	7	1	-0.07	9.43	2.00	0.86	107.2	0.114	7	0.80	0.13
^{152}Eu	324	6	710	16	7	254	0.784	7	5.48765432	0.31	556.60	12.54	5.49	254	0.784	7	5.49	0.31
^{85}Sr	2.07	0.945	2	1	1	1	0.483	7	3.38164251	-3.44	0.97	0.48	0.48	0.05	0.024	7	0.17	-0.22
^{99}Tc	500	7.5	2	1	1	261	0.522	7	3.654		1.04	0.52	0.52	261	0.522	7	3.65	3.85
stable -I																		
SUM			1074	82	55						615.43	23.56	13.73					

Table 5 Cementitious puck formulation - Batch 1 - cement used for lysimeters 2, 2A, 3, 4, 5, and 6. ("A" designates archive sample)

Philip Almond
Lysimeter Grout SALTSTONE
Batch1

using measured density and theory wt% solids
using measured density and wt% solids
input needed

160

Puck size 17.6 g

Targeting a Fixed Amount (Mass) of Grout	Trial Run	PreMix (grams)	Simulant (grams)		H_2O (grams)	H_2O/Premix	Grout (grams)	
		221.08	178.92		132.65	0.60	400	Targeted amount of grout and water/premix ratio
		221.43	178.57	mL less per puck	132.86			
	35.00733138		152.5260	1.146	113.48	0.852624		
	143.56		152.5260					
		Salt	Water	Salt + Water	Water	Salt	Total	
	Tank50	grams/Liter	grams/Liter	grams/Liter	g/g simulant	g/g simulant		
		312.47	895.93	1208.4	0.741	0.2586	1.000	Using theoretical wt% solids (using measured density)
		309.35	899.05	1208.4	0.744	0.2560	1.000	Using measured wt% solids and density
	Premix		Target					
	fly ash	0.45	99.4882					
	slag	0.00	0.0000					
	cement	0.55	121.5967					
	total	1.00	221.08					
	fly ash	0.45	99.6429					
	slag	0.00	0.0000					
	cement	0.55	121.7857					
	total	1.00	221.43					

Density of Simulant	1.2084
Wt % Solids (simulant)	25.60

Summary

Total grout wanted	400.0000	g
Total simulant	178.5714	g
Total premix	221.4286	g
fly ash	99.6429	g
slag	0.0000	g
cement	121.7857	g
Water to premix ratio	0.6000	

	Targeted	17.6	final puck mass
1	fill to	16.454	wet grout mass
2	add	1.146	spike volume (mL)

Original

Batch Sheet for Tank 50 Permeant - Saltstone Formulation
1L 7/18/2011

	TARGET		REFERENCE		
Compound	Molarity	MW	Amt / Liter		
	Moles/Liter	grams/mole	grams		
Water	0	18.015	**818.30**		
50% by Weight NaOH	1.551	39.998	**124.07**		
$NaNO_3$	2.116	84.995	**179.86**		
$NaNO_2$	0.336	68.996	**23.20**		
Na_2CO_3	0.148	105.990	**15.64**		
Na_2SO_4	0.059	142.042	**8.32**		
Aluminum Nitrate (9 H2O)	0.110	375.129	**41.26**		
Total Salt mass			312.47		
Total Na Molarity	4.42				

Table 6 Cementitious puck formulation - Batch 2 - Cement used for lysimeters 7, 8, 8A ("A" designates archive sample)

Philip Almond
Lysimeter Grout SALTSTONE
Batch2

using measured density and theory wt% solids
using measured density and wt% solids
input needed

160

Puck size
17.6 g

Targeting a Fixed Amount (Mass) of Grout	Trial Run	PreMix (grams)	Simulant (grams)		H_2O (grams)	H_2O/Premix	Grout (grams)	
		221.08	178.92		132.65	0.60	400	Targeted amount of grout and water/premix ratio
		221.43	178.57	mL less per puck	132.86			
	18.02297165		165.1623	0.59	122.88	0.43896		
	160.55		165.1623					
		Salt	Water	Salt + Water	Water	Salt	Total	
	Tank50	grams/Liter	grams/Liter	grams/Liter	g/g simulant	g/g simulant		
		312.47	895.93	1208.4	0.741	0.2586	1.000	Using theoretical wt% solids (using measured density)
		309.35	899.05	1208.4	0.744	0.2560	1.000	Using measured wt% solids and density

Premix		Target
fly ash	0.45	99.4882
slag	0.00	0.0000
cement	0.55	121.5967
total	1.00	221.08
fly ash	0.45	99.6429
slag	0.00	0.0000
cement	0.55	121.7857
total	1.00	221.43

Density of Simulant	1.2084
Wt % Solids (simulant)	25.60

Summary

Total grout wanted	400.0000	g
Total simulant	178.5714	g
Total premix	221.4286	g
fly ash	99.6429	g
slag	0.0000	g
cement	121.7857	g
Water to premix ratio	0.6000	

Original

Batch Sheet for Tank 50 Permeant - Saltstone Formulation
1L 7/18/2011

Compound	TARGET Molarity	MW	REFERENCE Amt / Liter		
	Moles/Liter	grams/mole	grams		
Water	0	18.015	**818.30**		
50% by Weight NaOH	1.551	39.998	**124.07**		
$NaNO_3$	2.116	84.995	**179.86**		
$NaNO_2$	0.336	68.996	**23.20**		
Na_2CO_3	0.148	105.990	**15.64**		
Na_2SO_4	0.059	142.042	**8.32**		
Aluminum Nitrate (9 H2O)	0.110	375.129	**41.26**		
Total Salt mass			312.47		
Total Na Molarity	4.42				

	Targeted	17.6	final puck mass
1	fill to	17.01	wet grout mass
2	add	0.59	spike volume (mL)

Table 7 Cementitious puck formulation - Batch 3 - Saltstone used for lysimeters 14, 14A, 15, 16, 17, 18 ("A" designates archive sample)

Philip Almond
Lysimeter Grout SALTSTONE — using measured density and theory wt% solids
Batch3 — using measured density and wt% solids
input needed

160

Puck size 17.6 g

Targeting a Fixed Amount (Mass) of Grout	Trial Run	PreMix (grams)	Simulant (grams)		H_2O (grams)	H_2O/Premix	Grout (grams)	
		221.08	178.92		132.65	0.60	400	Targeted amount of grout and water/premix ratio
		221.43	178.57	mL less per puck	132.86			
	35.00733138		152.5260	1.146	113.48	0.852624		
	143.56		152.5260					

	Salt	Water	Salt + Water	Water	Salt	Total	
Tank50	grams/Liter	grams/Liter	grams/Liter	g/g simulant	g/g simulant		
	312.47	895.93	1208.4	0.741	0.2586	1.000	Using theoretical wt% solids (using measured density)
	309.35	899.05	1208.4	0.744	0.2560	1.000	Using measured wt% solids and density

Premix		Target
fly ash	0.45	99.4882
slag	0.45	99.4882
cement	0.10	22.1085
total	1.00	221.08
fly ash	0.45	99.6429
slag	0.45	99.6429
cement	0.10	22.1429
total	1.00	221.43

Density of Simulant	1.2084
Wt % Solids (simulant)	25.60

Summary

Total grout wanted	400.0000	g
Total simulant	178.5714	g
Total premix	221.4286	g
fly ash	99.6429	g
slag	99.6429	g
cement	22.1429	g
Water to premix ratio	0.6000	

	Targeted	17.6	final puck mass
1	fill to	16.454	wet grout mass
2	add	1.146	spike volume (mL)

Original

Batch Sheet for Tank 50 Permeant - Saltstone Formulation
1L 7/18/2011

	TARGET		REFERENCE		
Compound	Molarity	MW	Amt / Liter		
	Moles/Liter	grams/mole	grams		
Water	0	18.015	**818.30**		
50% by Weight NaOH	1.551	39.998	**124.07**		
$NaNO_3$	2.116	84.995	**179.86**		
$NaNO_2$	0.336	68.996	**23.20**		
Na_2CO_3	0.148	105.990	**15.64**		
Na_2SO_4	0.059	142.042	**8.32**		
Aluminum Nitrate (9 H2O)	0.110	375.129	**41.26**		
Total Salt mass			312.47		
Total Na Molarity	4.42				

Table 8 Cementitious puck formulation - Batch 4 - Saltstone used for lysimeters 19, 20, 20A ("A" designates archive sample)

Philip Almond
Lysimeter Grout SALTSTONE
Batch4

using measured density and theory wt% solids
using measured density and wt% solids
input needed

160

Puck size 17.6 g

Targeting a Fixed Amount (Mass) of Grout	Trial Run	PreMix (grams)	Simulant (grams)		H_2O (grams)	H_2O/Premix	Grout (grams)	
		221.08	178.92		132.65	0.60	400	Targeted amount of grout and water/premix ratio
		221.43	178.57	mL less per puck	132.86			
	18.02297165		165.1623	0.59	122.88	0.43896		
	160.55		165.1623					
		Salt	Water	Salt + Water	Water	Salt	Total	
	Tank50	grams/Liter	grams/Liter	grams/Liter	g/g simulant	g/g simulant		
		312.47	895.93	1208.4	0.741	0.2586	1.000	Using theoretical wt% solids (using measured density)
		309.35	899.05	1208.4	0.744	0.2560	1.000	Using measured wt% solids and density

Premix		Target
fly ash	0.45	99.4882
slag	0.45	99.4882
cement	0.10	22.1085
total	1.00	221.08
fly ash	0.45	99.6429
slag	0.45	99.6429
cement	0.10	22.1429
total	1.00	221.43

Density of Simulant	1.2084
Wt % Solids (simulant)	25.60

Summary

Total grout wanted	400.0000	g
Total simulant	178.5714	g
Total premix	221.4286	g
fly ash	99.6429	g
slag	99.6429	g
cement	22.1429	g
Water to premix ratio	0.6000	

Original

Batch Sheet for Tank 50 Permeant - Saltstone Formulation
1L 7/18/2011

Compound	TARGET Molarity	MW	REFERENCE Amt / Liter		
	Moles/Liter	grams/mole	grams		
Water	0	18.015	**818.30**		
50% by Weight NaOH	1.551	39.998	**124.07**		
$NaNO_3$	2.116	84.995	**179.86**		
$NaNO_2$	0.336	68.996	**23.20**		
Na_2CO_3	0.148	105.990	**15.64**		
Na_2SO_4	0.059	142.042	**8.32**		
Aluminum Nitrate (9 H2O)	0.110	375.129	**41.26**		
Total Salt mass			312.47		
Total Na Molarity	4.42				

	Targeted	17.6	final puck mass
1	fill to	17.01	wet grout mass
2	add	0.59	spike volume (mL)

Table 9 Batch description of saltstone and cement rad sources ("A" designates replicate to be archived).

		spike vol			spike vol
spike	cement	(amount to decrease simulant)		Saltstone	(amount to decrease simulant)
none	2	1.146		14	1.146
none	2A	1.146		14A	1.146
none	3	1.146		15	1.146
cations	4	1.146		16	1.146
cations	5	1.146		17	1.146
cations	6	1.146		18	1.146
anions	7	0.59		19	0.59
anions	8	0.59		20	0.59
anions	8A	0.59		20A	0.59
	type	#'s	less simulant volume		
Batch 1:	cement	2, 2A, 3-6	1.146		
Batch 2:	cement	7, 8, 8A	0.59		
Batch 3:	saltstone	14, 14A, 15-18	1.146		
Batch 4:	saltstone	19, 20, 20A	0.59		

APPENDIX C. Preparation of Actinide Compounds for Lysimeter Experiments

HAP: SRNL-L3000-2010-00002-1

Pu stock solution is 25.6 g/L ~ 0.107M Pu in ~1M HNO3

Pu(III) Oxalate

Chemicals Needed

1 M Ascorbic Acid	50 mL
1 M Sulfamic Acid	50 mL
0.9 M Oxalic acid	50 mL
1.5M HNO_3 acid	50 mL
0.5 M Nitric Acid	50 mL
0.2 M Oxalic Acid	50 mL

Take 7.6 mL of Pu stock solution
Add 17.4 mL of 1.5 M HNO_3 to give 25 mL of 7.78 g/L Pu in ~1.5M HNO_3
Place 25 mL of 7.78 g/L Pu in a 50 mL beaker.
Add 1.32 mL of 1M Sulfamic acid. Let stir for 10 minutes.
Add 1.52 mL of 1M Ascorbic acid. Let stir for 10 minutes.
--Solution should now be Pu(III) (blue color)
Add while stirring 9.7 mL of 0.9M Oxalic acid dropwise over a 20-30 minute period.
Digest solution @ 50 °C for 30 minutes (place watch glass on top of beaker)
Let cool
Filter using a syringe filter (blue solids)
Wash 3X with 0.5 mL of 0.5 HNO_3, 0.2M Oxalic acid.
Let air dry on filter
Place sample in a pre-weighed 4-dram vial

Pu(IV) Oxalate

Take 7.6 mL of Pu stock Solution
Add 17.4 mL of 1.5 M HNO_3 to give 25 mL of 7.78 g/L Pu in ~1.5M HNO_3
Add 1.52 mL of 1M Ascorbic Acid. Let Stir for 10 minutes. (blue color)
--Solution should now be Pu(III) (blue color)
Starting with 13.9 mL in a 50 mL beaker
Add 1.46 mL 1 M Sodium Nitrite solution with stirring and heat to 50 °C.
--Will become all (Pu(IV) (brown-green in color)
Heat another beaker containing 5.7 mL 0.9M Oxalic acid to 50 °C and stir.
Slowly add, drop wise, the Pu(IV) solution over 20-30 min
Digest solution @ 50 °C for 30 min. (place watch glass on top)
Let cool
Filter using a syringe filter with 0.45µM filter (light brown solids)
Wash 3X with 0.5 mL of 0.5 HNO3, 0.2M Oxalic acid.
Let air dry on filter
Place sample in a pre-weighed 4-dram vial

Pu(V) Carbonate

53.5 g/L Pu stock solution
$AgNO_3$ 3 mg
$Na_2S_2O_8$ 91 mg
$(NH_4)_2CO_3$ 180 mg

Heat Water Bath to 70 °C.
Add 1 mL of Pu Stock solution (53.5 g/L) to 4 dram vial containing $Ag(NO_3)$ and $Na_2S_2O_8$.
Heat $Pu/Ag/S_2O_8$ in water bath to 60-80 °C for 20 minutes (by setting the closed vial in a 50 mL beaker with 5 mL H_2O on a hot plate).
Remove vial and cool to near room temperature.
(check UV-Vis to confirm all Pu(VI)
Add all 180 mg of solid $(NH_4)_2CO_3$ to precipitate $NH_4PuO_2CO_3$,that occurs over 10 minutes as CO_2 evolution is observed. (producing green solids)
Filter solution with filtering apparatus.
Wash 3X with 15 wt% NH_4CO_3 solution.
Let air dry on filter
Place sample in a pre-weighed 4-dram vial

Np(IV) oxide

The Np(IV) oxide was similarly product oxide probably from the HB-Line standard production process (anion exchange, oxalate precipitation,filtration, calcination to 650 °C).

Np(V) nitrate

Np(V) nitrate was product solution from a past anion exchange run.
Roughly 1M HNO_3 and 50 g Np(V)/L and had oxidized over time from Np(IV) to Np(V).

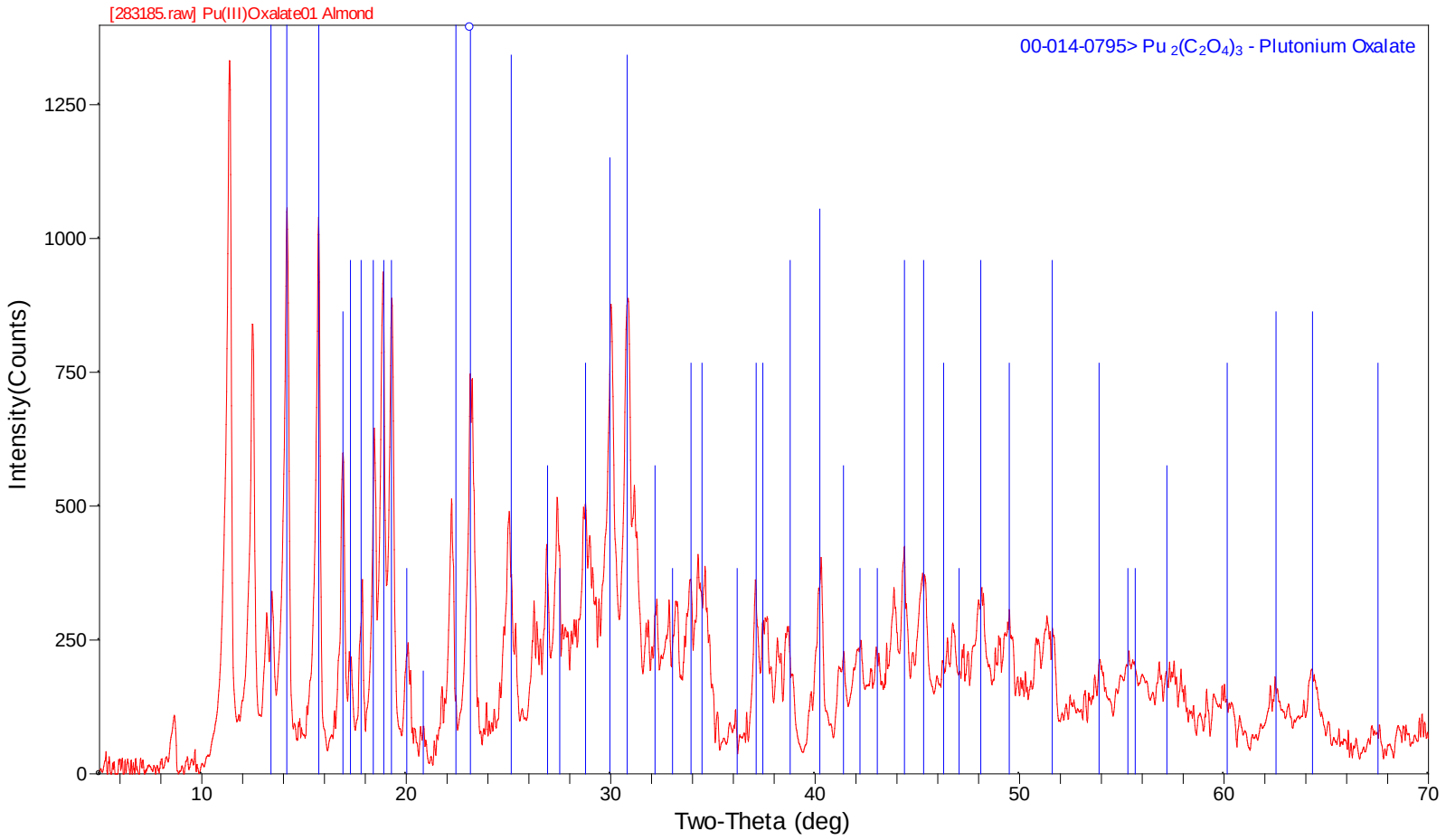

Figure 22 XRD spectrum of plutonium oxalate

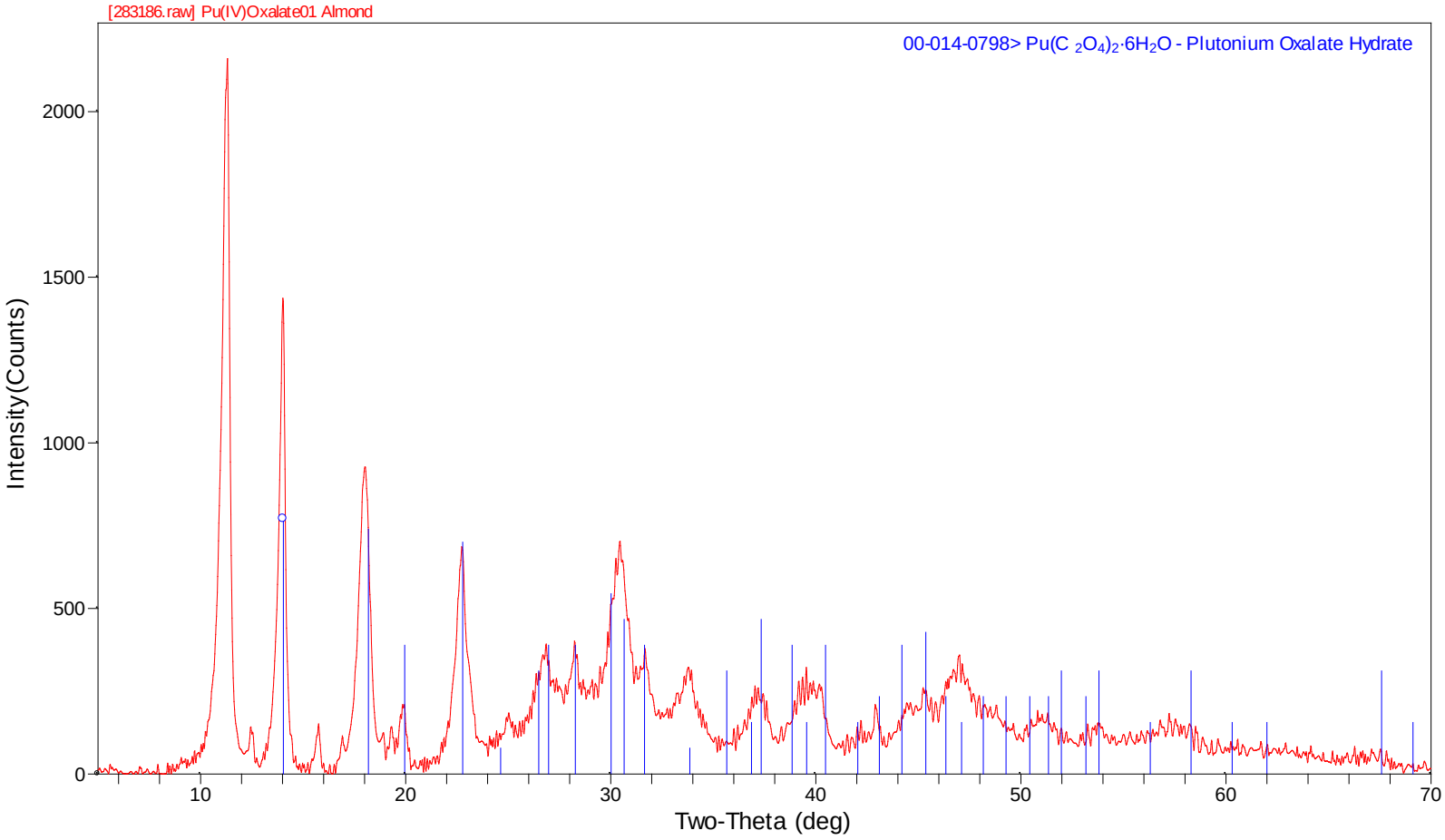

Figure 23 XRD spectrum of plutonium oxalate hydrate. (The missing peaks in the spectra above are likely from the identified phase. The missing peaks in the reference spectra are suspected to be uncollected low angle camera data.)

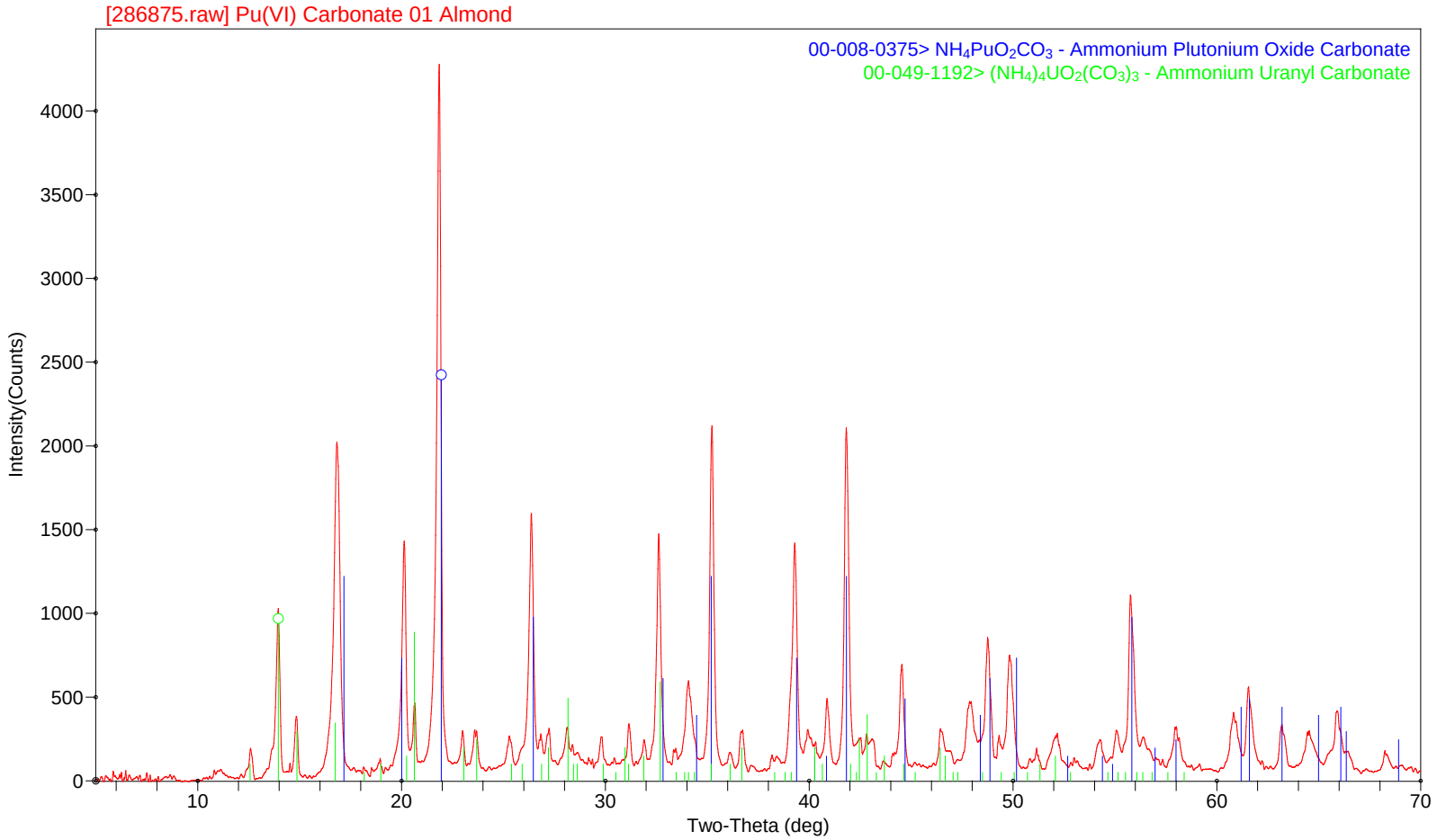

Figure 24 XRD spectrum of ammonium plutonium oxide carbonate.

APPENDIX D R&D Directions Pu and Np source delivery system preparation.

Kim Roberts
6/14/12

Materials:

3” tall 2” pvc pipe
Lysimeter soil
Cap
Foil
50mL flat topped centrifuge tube

Method

Place cap on bottom of pvc tube
Pack half full with lysimeter soil using 50mL flat topped centrifuge tube
Remove cap
Cover bottom with foil
Wrap pvc tube with foil covering the top lip

Rad source introduction

Place lexan tube inside pvc pipe
Remove filter with plastic forceps
Place filter inside lexan tube to soil level
Remove lexan tube
Pack the rest of the pvc with lysimeter soil
Remove foil and have tube smeared (top and bottom)
Label pvc pipe with lysimeter number (using marker outside the hood)
Place tube in rad bag (e.g. 4” x 12”)
Using tape from outside the hood, j-loop bag
Have bag probed and smeared and placed in second bag outside of hood

APPENDIX E R&D DIRECTION SAMPLE ARCHIVE NITROGEN PURGE

R&D Directions:
Transfer of rad samples to nitrogen purged vessel in Rad hood in B-122
Kim Roberts
8/22/12

I would like to transfer archived samples from the lysimeter project to a vessel (photo below) and purge with nitrogen gas in the hood in B-122.
The Pu and Np samples came from the glovebox in F003 and were the ones that probed for alpha and single sample had measurable smearable alpha.
The samples are in 3 bags:

- 1 bag contains archive filters of Pu(III), Pu(IV) and Pu(V)
- 1 bag contains archive filters of Pu(colloids), Np(IV), and Np(V)
- 1 bag contains archive filter of beta suite and 2 pucks (saltstone or cement) with Beta suite
- 1 puck with Tc & I

The proposed work is as follows:

1. Open J-looped bags with samples and have smear taken.
2. If smears are reasonable, transfer the sample containers to the vessel. If smears are high, transfer to clean sample containers, re-smear and transfer into vessel.
3. Once all samples loaded in vessel, purge with house nitrogen gas – using RPD approved air filter on outflow.
4. Once vessel purged close inlet and outlet and move to appropriate storage location in back of hood
5. Have air filter counted by RPD.

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