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Determination of Constituent Concentrations In Field Lysimeter Effluents

FY13 Final Report
Task 2: Lysimeter Leachate Chemistry

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SRR Project Title: SRR Technical Support Provided by: Clemson University
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Determination of constituent concentrations in field lysimeter effluents

Michael Witmer and Brian A. Powell

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9/4/13

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Date

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Abbreviations

BLD	Measurement was below detection limits
DNR	Data not available because the sample was not received
DO	Dissolved Oxygen
FY12Q4	Fiscal Year (YY) Quarter (Q)
HCL	Hydrochloric Acid
HDPE	High-density Polyethylene
HNO_3	Nitric Acid
HPGe	High Purity Germanium Detector
ICP-MS	Inductively Coupled Plasma Mass Spectroscopy
ID	Identification
LSC	Liquid Scintillation Counting
NA	No measurement data because sample volume was too small
$NaHSO_3$	Sodium Bisulfite
NIST	National Institute of Standards and Technology
ppb	Parts per billion
ppm	Parts per million
QAQC	Quality Assurance and Quality Control
SRNL	Savannah River National Laboratory
SRR	Savannah River Remediation
SRS	Savannah River Site

Summary

This report contains the data collected during the measurement of constituent concentrations in lysimeters from the RadFLEx facility discussed in the report "SRNL Radionuclide Field Lysimeter Experiment: Baseline Construction and Implementation" by Kaplan et al., (2012, SRNL-STI-2012-00603). Savannah river National Laboratory (SRNL) presently has 43 transport experiments underway at the RadFLEx facility. In this experiment, constituents of interest are buried in 5L containers that are open to precipitation. Leachate is collected from these lysimeters approximately every three months to provide a measure of constituent transport through the 2-foot long columns. One set of 14 lysimeters contain cementitious sources. These are:

1. radionuclide-free cementitious material (control) (lysimeters 2, 3, 14, and 15)
2. Tc-99 and stable iodine (lysimeters 7, 8, 19, and 20)
3. a suite of gamma emitters, Cs-137, Co-60, Ba-133, and Eu-152 (lysimeters 4-6 and 16-18)

A second set of 29 lysimeters contain soil sources which have been amended with constituents of interest. These include:

1. Pu(V)$NH_4(CO_3)$ (lysimeters 21-23 and 41-43).
2. a suite of beta/gamma emitters, Cs-137, Co-60, Ba-133, and Eu-152 (lysimeters 26-28)
3. Np-237 (lysimeters 29-32)
4. Pu(III)oxalate and Pu(IV) oxalate (lysimeters 9-11, 33-35, and 38-40)
5. Pu colloids (lysimeters 44-46)
6. Sediment controls with no radionuclides (lysimeters 12, 24, 25, and 37).

Effluent solutions were collected at least on a quarterly basis by SRNL staff and shipped to Clemson University for the measurements. The constituent concentrations in the effluent were measured using a variety of techniques as discussed below.

Materials and Methods

Data Recording

All measurements and observations were recorded in a laboratory notebook with the appropriate title, time, date, data taker and apparatus used. A datalog sheet that includes the sample identification (ID), dates of sample collection and receipt from the Savannah River Site (SRS) and dates of sample preparation and analysis at Clemson was created for every sample received at Clemson. An example of this datalog sheet can be found in Appendix A. Preliminary measurements and subsampling information were also recorded on these datalog sheets. A unique identification code was given to every sample and subsample.

Instruments were calibrated with National Institute of Standards and Technology (NIST) traceable standards. The calibration date, standard identification and expiration dates, and quality assurance and quality control (QAQC) spiked samples were recorded on a datalog sheet for each sampling event. An example of this datalog sheet can be found in Appendix A.

Sample Receipt and subsampling

Lysimeter effluent samples were received on a quarterly basis in 2L bottles. Approximately 250 mL, (or half of the total volume if the sample volume was less than 500 mL) of each lysimeter effluent was removed for archiving and placed into a 250mL high-density polyethylene (HDPE) pre-cleaned container. The ID given to each sample was also used for the archived containers. The pH and dissolved oxygen (DO) content of each solution was measured using a Thermo Ross semi-micro pH electrode and a VWR dissolved oxygen probe. The pH electrode was standardized with Thermo pH buffer solutions at pH values of 4.01, 7.00 and 10.04. Archive information and pH and DO measurements were recorded on datalog sheets and are provided in the data tables in Appendices B, C, and D.

Lysimeter effluent bottles, with the exception of ^{99}Tc and stable iodine bearing samples, were acidified to 2% nitric acid (HNO_3) using concentrated nitric acid. The intent of acidifying the solutions within the leachate collection bottles was to facilitate desorption of any ions sorbed to the container walls. Thus the 250 mL archived sample removed is to preserve the sample in the "field" (non-acidified) state in the event that analysis of radionuclide speciation is to be performed at a future date. Samples for analyte measurements were taken from the acidified sample. Any samples and subsamples taken from the acidified bottles were referred to as the sample ID with the suffix –Acid in sample containers and data files.

Analysis of ^{99}Tc and Stable Iodine

Lysimeter effluent bottles containing ^{99}Tc and stable iodine were received in a large cooler. ^{99}Tc activities were determined using liquid scintillation counting (LSC) and stable iodine concentrations were made using a Thermo Scientific Orion Sure-Flow 9653BN iodide selective electrode. Liquid scintillation measurements were made using a Hidex SL300 liquid scintillation counter. Triplicate samples were prepared by adding approximately 0.3 mL of lysimeter effluent to 5 mL of Optiphase 'Hisafe' 3 liquid scintillation cocktail, purchased from Perkin Elmer, in a 7 mL polyethylene scintillation vial. The exact volume of effluent solution transferred to the LSC vial was determined gravimetrically. The counting efficiency of >99% was verified using the triple-to-double-coincidence-ratio counting protocol (Wanke et al., 2012) on the Hidex SL300.

An iodine selective electrode was calibrated using a set of iodide standards made by diluting a NIST traceable iodide stock solution purchased from High Purity Standards (Charleston, SC; Cat#IC-II-M 1000 ug/L Iodide stock solution). The calibration curve is shown in Figure 1. The top figure shows all of the calibration data. It can be seen that the calibration curve deviates from linearity below 10 ppm total iodide. Therefore, based on the range of the dataset, the calibration curve was truncated at 10 ppm as shown in the bottom of Figure 1. This calibration curve was used to determine the iodine concentrations in effluent waters discussed below. Samples were prepared for iodine analysis by pipetting 5 mL of sample into a 15mL centrifuge tube, adding 0.15 g of a reductant, sodium bisulfite ($NaHSO_3$), and adding 0.1 mL of concentrated hydrochloric acid (HCl). This step was to reduce all iodine to iodide which the probe measures. All measurements were low and only three were above the 0.01 parts per million (ppm) detection limit as discussed in the subsequent text. This is similar to measurement of iodine using alternate methods such as inductively coupled plasma mass spectrometry.

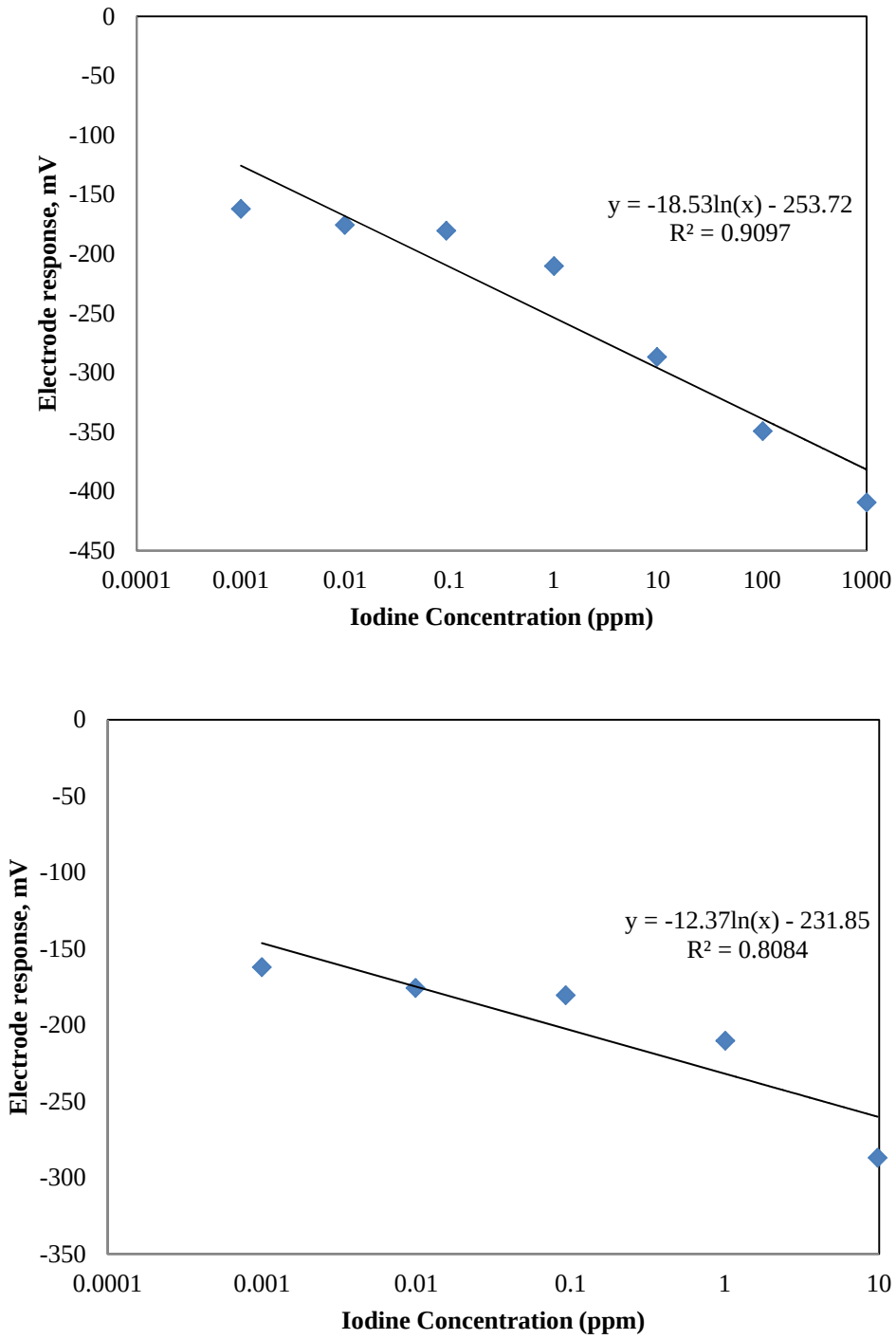

Figure 1: Iodine probe calibration curve using all calibration standards (top) and a truncated curve using only the measurements from the five lowest concentration standards. Standards were prepared using a dilution of a high-purity 1000 ug/L iodide standard.

Analysis of Gamma Emitting Radionuclides (^{60}Co, ^{137}Cs, ^{133}Ba, ^{152}Eu)

Samples containing gamma emitting radionuclides were analyzed using a high purity germanium detector (HPGe). Quarterly efficiency calibration of the germanium detector was performed using a NIST traceable ^{152}Eu stock solution. The calibration curve for the fourth quarter of the 2012 fiscal year (FY12Q4) sampling event is shown below (Figure 2). The calibration curves that were used for the remaining sampling events can be found in Appendix C. A standard geometry of 45mL of sample in a 50mL conical polypropylene centrifuge tube was used. The tube was placed in a plastic holder which was fitted to cover the detector and counted for 24 hours. A quarterly background radiation measurement was made by counting an empty sample tube. The contribution of the background was subtracted from each sample.

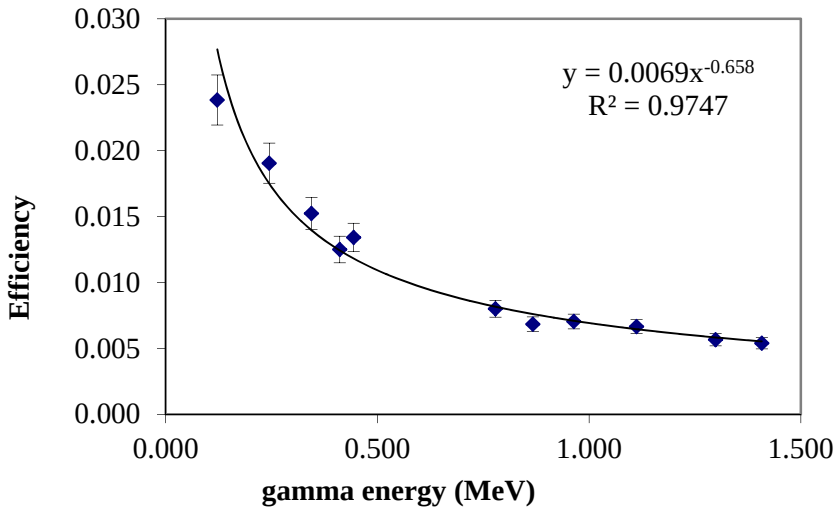

Figure 2: HPGe Efficiency curve determined from counting 45mL of a ^{152}Eu standard in a 50mL conical centrifuge tube.

Analysis of the actinides (^{237}Np and $^{239/240}$Pu)

Analysis of samples containing ^{237}Np and $^{239/240}$Pu was performed using inductively coupled plasma mass spectroscopy (ICP-MS). Approximately 10 mL of acidified sample was removed from the collection bottles and placed in a 15mL centrifuge tube and subsequently analyzed on the ICP-MS. ^{237}Np and ^{239}Pu standards were prepared by diluting NIST standard reference materials 4341 and 4334I for ^{237}Np and ^{239}Pu, respectively. The calibration curve for ^{237}Np (Figure 3) and ^{239}Pu (Figure 4) used for the FY13Q3 sampling interval is shown below. Sensitivity and intercept concentrations for ^{237}Np and ^{239}Pu calibration curves used for the analysis of the FY13Q3 samples can be found in Appendix D. The samples were run using ^{242}Pu as an internal standard.

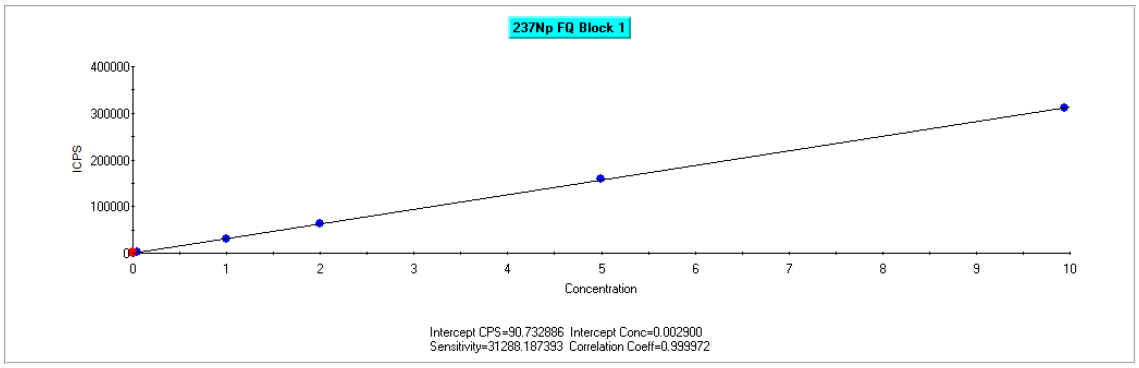

Figure 3: Screen capture of the ^{237}Np calibration curve using Thermo Plasma Lab software to control the data collection and analysis for the FY13Q3 sampling interval. R^2=0.99997, Intercept Conc. (Detection Limit) = 0.0029 ppb.

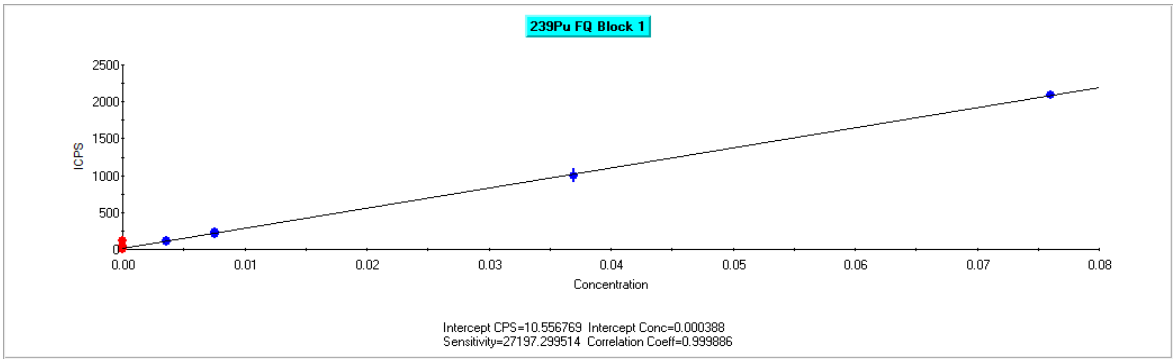

Figure 4: Screen capture of the ^{239}Pu calibration curve using Thermo Plasma Lab software to control the data collection and analysis for the FY13Q3 sampling interval. R^2=0.99989, Intercept Conc. (Detection Limit) = 0.0004 ppb.

Results and Discussion to Date

^{99}Tc and I

The measured activity concentration (Bq/L) of ^{99}Tc from the four sampling events is shown in Figure 5. There was a significant increase in activity measured in the FY13Q1 samples versus the FY12Q4 samples. There were two separate sampling events during the FY13Q2 sampling interval denoted by FY13Q2a and FY13Q2b. Lysimeter 20 samples from the FY13Q2 sampling event were not received. Sample collection for all lysimeters containing ^{99}Tc and iodine was stopped following the FY13Q2 sampling interval. Thus there is no data to report for these lysimeters during the FY13Q3 sampling interval. Figure 6 shows the cumulative fraction of the source that has been released from the respective lysimeter. Data for the control lysimeters is not included because measurements were below detectable limits (BLD). It is interesting to note the differences between the releases from the different sources. Lysimeters 7 and 8 contains a cementitious saltstone material without slag, and lysimeters 19 and 20 contain the same cementitious saltstone material with slag. The total release from the sources of lysimeters 7 and 8 is 52% and 57% respectively, compared to the source in lysimeters 19, which had a cumulative release of 27%. The cumulative release from lysimeter 20 was 10%, although it had one less

sampling event. Determining the release during the FY13Q2 sampling period for lysimeter 20 would have been beneficial to know in order to compare its relative release to lysimeter 19. Although the concentrations of ^{99}Tc in the effluent are similar (Figure 5), the cumulative fraction of ^{99}Tc released for lysimeter 19 was lower than that for lysimeters 7 and 8 (Figure 6) because the total volume of solution passing through lysimeter 19 was lower than lysimeters 7 and 8 (Appendix Table B3).

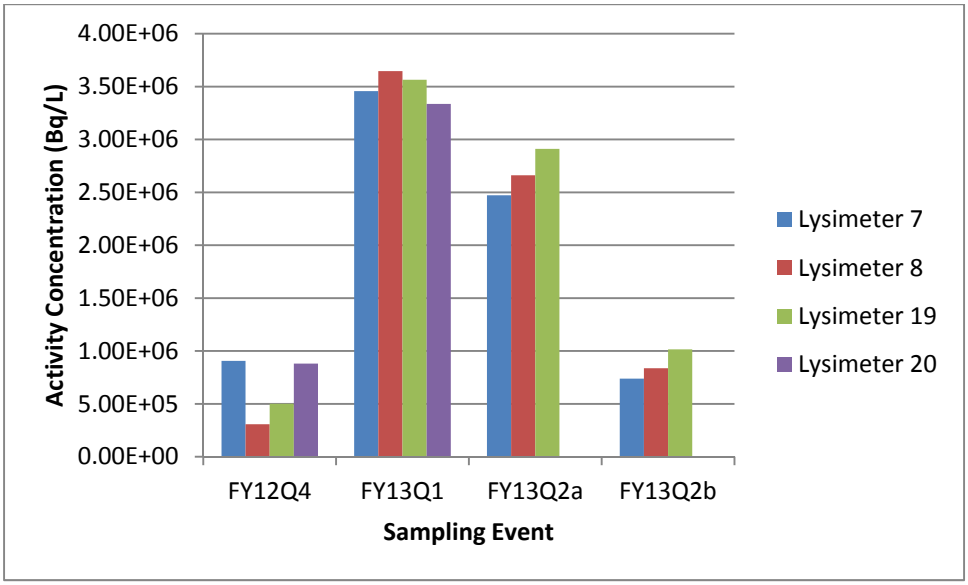

Figure 5: The activity concentrations of ^{99}Tc in lysimeters 7, 8, 19 and 20 for the FY12Q4, FY13Q1, FY13Q2a, and FY13Q2b sampling events. Activity concentrations are reported in Bq/L. (Note: *a* and *b* denote two separate sampling events in the fiscal quarter.)

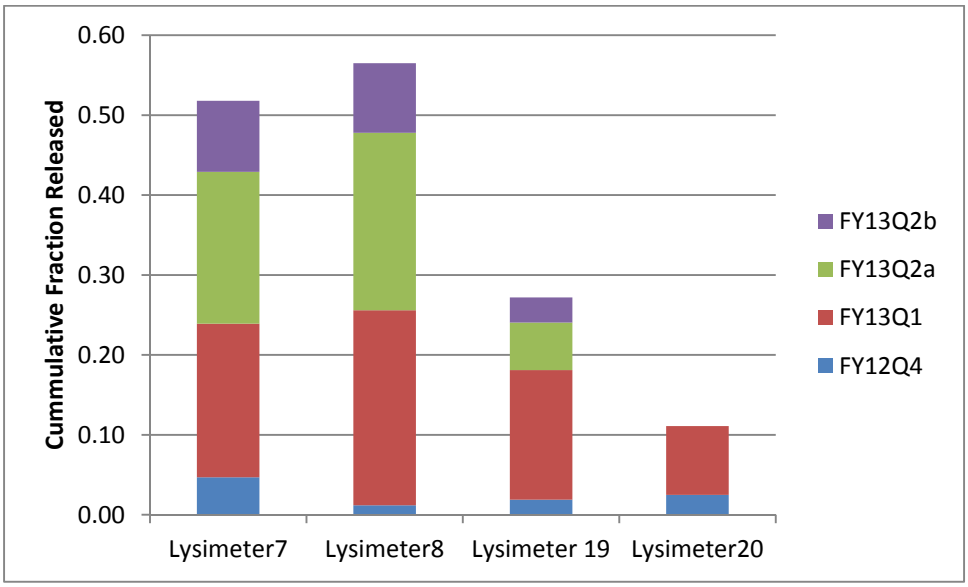

Figure 6: Cumulative fraction of ^{99}Tc relative to the source for lysimeters 7, 8, 19, and 20.

The release of iodine was slightly above detection limits in the lysimeter 7 effluents from the FY12Q4, FY13Q1 and FY13Q2a sampling periods and below detection limits for all other effluents. Calculations of the fraction of iodine in the effluent for each quarter relative to the total amount in the lysimeter are also shown in Table 1. The iodine concentrations for all of the lysimeters containing ^{99}Tc and stable iodine are shown in Table 1. It appears that the fraction of iodine being released is decreasing but further sampling events would be useful to verify this.

Table 1: Iodine release data for lysimeters 7, 8, 19 and 20.

Sample ID	Millimoles (and mg) of iodine in source	Iodine Concentration in effluent (mM)*	Fraction of iodine in effluent relative to total iodine in source*
121004-7-S	0.07 (8.82)	7.25E-05	1.04E-03
130109-7-S	0.07 (8.82)	2.48E-05	3.54E-04
130212-7-S	0.07 (8.82)	2.88E-05	4.12E-04
130307-7-S	0.07 (8.82)	BLD	BLD
121004-8-S	0.07 (8.82)	BLD	BLD
130109-8-S	0.07 (8.82)	BLD	BLD
130212-8-S	0.07 (8.82)	BLD	BLD
130307-8-S	0.07 (8.82)	BLD	BLD
121004-19-S	0.07 (8.82)	BLD	BLD
130109-19-S	0.07 (8.82)	BLD	BLD
130212-19-S	0.07 (8.82)	BLD	BLD
130307-19-S	0.07 (8.82)	BLD	BLD
121004-20-S	0.07 (8.82)	DNR	
130109-20-S	0.07 (8.82)	DNR	

***BLD – Measurement was below detection limits.**

****DNR - Data not available because the sample was not received**

Gamma suite radionuclides - ^{60}Co, ^{137}Cs, ^{133}Ba, ^{152}Eu

Nine lysimeters (4-6, 16-18, and 26-28) contained sources consisting of the gamma emitting radionuclides ^{60}Co, ^{137}Cs, ^{133}Ba, and ^{152}Eu. Lysimeters 4-6 contained the nuclides within a saltstone matrix without additional blast furnace slag (BFS), 16-18 were contained within a saltstone matrix with BFS, and 26-28 were control lysimeters where the nuclides were added directly to a filter and placed in the lysimeters. Of those radionuclides, ^{60}Co was determined to have the highest releases. The cumulative activity (Bq) of ^{60}Co from each sampling interval is shown in Figure 7. The activities for each lysimeter are broken down by sampling event in Appendix C. Figure 8 shows the fraction of ^{60}Co relative to the source for each lysimeter. The concentration of ^{60}Co was greatest in the effluents of lysimeters 4, 5 and 6, which had cumulative releases of 6%, 58%, and 13%, respectively. Due to the release of over half of the source from lysimeter 5 and the relatively high releases from lysimeters 4 and 6, it will be necessary to model ^{60}Co transport to support this data. Examination of the source material of these lysimeters to look for a physical change which may have facilitated the high release from lysimeter 5 would be useful.

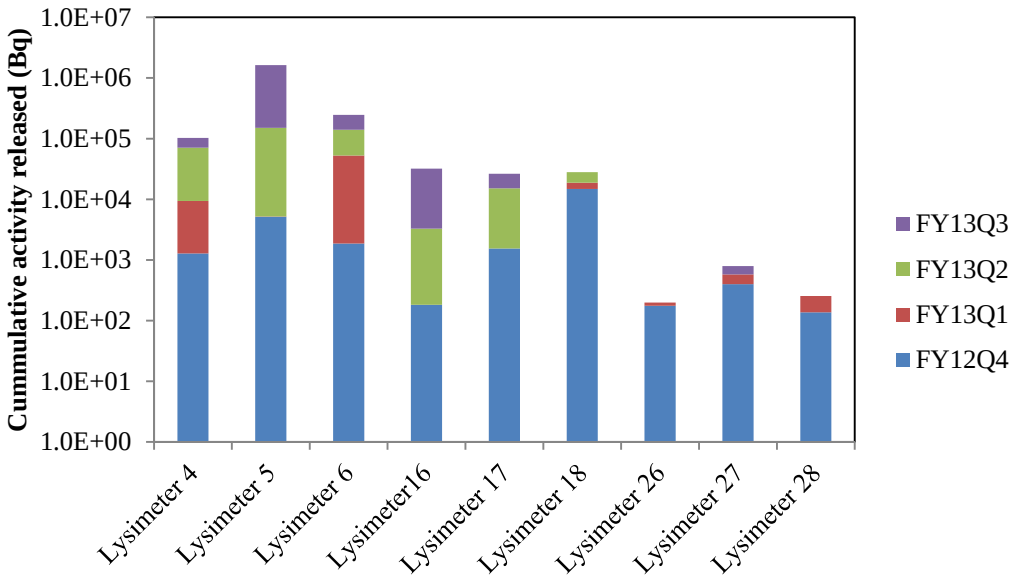

Figure 7: Cumulative ^{60}Co activity in the effluent of the lysimeters containing gamma emitting radionuclides.

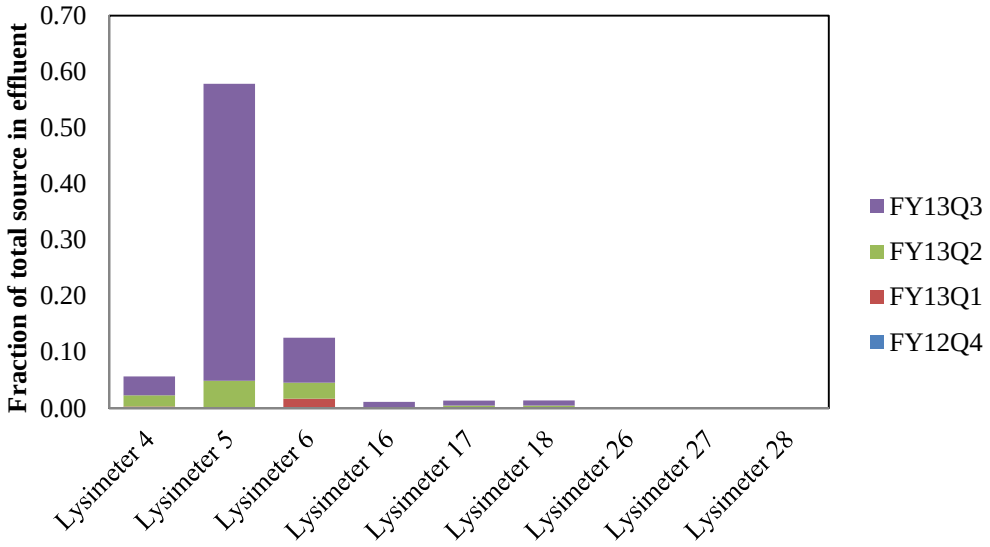

Figure 8: The cumulative fraction of ^{60}Co activity in the effluent relative to the source for the lysimeters containing gamma emitting radionuclides.

Actinides ($^{239/240}$Pu and ^{237}Np)

There was no measurable release of ^{239}Pu from the eighteen lysimeters containing plutonium during the first year of sample collection. This is consistent with the currently accepted model that plutonium has limited mobility in the subsurface primarily due to reduction of mobile Pu(V) to immobile Pu(IV) on mineral surfaces (Kaplan, D.I., et al., 2004). Four lysimeters contain ^{237}Np sources and there was no

measurable release during the first three sampling events. However, during the FY13Q3 sampling event, lysimeter 30 had a release of 146 Bq from the source. That release corresponds to a 0.2% release of the source. The ^{237}Np data from the FY13Q3 sampling event is summarized in Table 2. Lysimeter 30 contained neptunium added as the highly mobile Np(V) oxidation state. It is noteworthy that no mobility of neptunium was observed in lysimeters 31 and 32 since neptunium was added in the highly immobile Np(IV) state. A complete summary of the data for the lysimeters containing Np and Pu can be found in Appendix D.

Table 2: Summary of the ^{237}Np data from the FY13Q3 sampling event

Sample ID	Description	Activity of ^{237}Np in source (uCi)	Measured activity of ^{237}Np in effluent (Bq)	Fraction of ^{237}Np activity in the effluent relative to the source
130617-29-S	Np(V)nitrate	1.7 ^{237}Np	BLD	BLD
130617-30-S	Np(V)nitrate		146	0.002
130617-31-S	Np(IV)O_2	1.9 ^{237}Np	BLD	BLD
130617-32-S	Np(IV)O_2		BLD	BLD

Summary

In this work, the concentrations of radionuclides and stable iodine were measured in effluents from field lysimeters. These measurements are from the first year of a long-term, multi-year experiment. Highlights from these measurements are:

1. ^{99}Tc concentrations ranging from 1 x 10^5 to 3.5 x 10^6 Bq/L were measured in the lysimeter effluents. These values represent between 10% and 50% of the total ^{99}Tc contained within the source leaching out and transporting through the soil column. The lysimeters containing saltstone with no BFS exhibited the largest fraction released. (Appendix Table B3).
2. The concentrations of iodine in the effluents were very low or below the detection limit.
3. The concentrations of Np and Pu in the effluents were very low or below the detection limit with the notable exception that a small fraction of Np (0.002 of the source) was measured in lysimeter 30 during the FY13Q3 sample analysis.
4. ^{60}Co was the only gamma emitting radionuclide measured in the effluent. The highest concentrations were observed eluting from the lysimeters containing saltstone with no added BFS.

References

Roberts, K.A., et al.,, “SRNL Radionuclide Field Lysimeter Experiment: Baseline Construction and Implementation” SRNL-STI-2012-00603, Savannah River National Laboratory, Aiken, SC, 2012.

Kaplan, D.I., et al., “Influence of oxidation states on plutonium mobility during long-term transport through an unsaturated subsurface environment”. Environmental Science & Technology, 2004. 38(19): p. 5053-5058.

Powell, B. A. and Arai., Y. “Test Plan Document Task 2: Lysimeter Leachate Chemistry” G-SOW-Z-00007, Rev. 1, Clemson University , Clemson, SC, 2013.

Wanke, C., Kossert, K., Nahle, O. J., “Investigations on TDCR measurements with the Hidex 300SL using a free parameter model”, Applied Radiation and Isotopes, 2012. 70: p. 2176-2183.

Appendix A: Example Datalog Sheets

Datasheet for Instrument Calibration Documentation for FY12Q4 Sampling Interval

Identification number for linking calibration data to sample measurements (Format: *Month-Year-Labbook Number-Labbook page*, where month and year are month and year of sample receipt from SRS).	11-12-1-2
Date of Sample Receipt from SRS	11/06/2012
Description of ^{152}Eu gamma spectroscopy standard (date of preparation, total activity, total volume, vessel)	11/14/12, 0.271 uCi/L, 45mL, 50mL conical centrifuge tube
Gamma spectrometer ID	HPGE6
Filename of gamma spectroscopy standard measurement	152Eu_std(HPGE6)_01112013
Filename of gamma spectroscopy background measurement	HPGE6_Background_111212
pH meter calibration slope and pH(0) value	98.1%, pH(0)=1.0233

Datasheet for Lysimeter 2 – FY12Q4

Lysimeter number	2
Sample ID	100412L2
Date of sample collection at SRS	10/04/2012
Date of sample receipt from SRS	11/06/2012
Date of sample being prepared for analysis by Clemson	11/11/2012
Date of analysis at Clemson	11/11/2012 (pH,DO), 11/16/2012 (HPGe)
ID Number for Instrument Calibration datalog sheet	11-12-1-2

Sample pH:	6.73
Sample Dissolved Oxygen Concentration (mg/L):	7.90
Mass of solution plus bottle (g)	653.11
Mass of solution subtracting average mass of collection bottles (g)	398.82

Estimated volume of solution removed for sample archiving (mL)	-
Archived sample ID:	-

Gamma detector ID used for analysis:	HPGe6
Gamma spectroscopy sample count filename:	45mL_SRS_100412L2_11162012.CNF

Appendix B: Summary of Data for Lysimeters containing ^{99}Tc and Stable Iodine

Table B1: Summarized Data for the lysimeters containing 99Tc and stable iodine from the FY12Q4 sampling interval

Sample Interval: FY12Q4 Date Sample taken @ SRS: 10/04/2012 Date Sample Analyzed/Prepared at Clemson University: 04/17/2013						Activity or Mass Concentration (Bq/L for ^{99}Tc or ppm for I)	
Lysimeter number	Constituent(s)	Sample ID	pH	DO	Vol. measured @ Clemson Univ. (mL)	^{99}Tc	Iodine
7	Cement Tc & I	121004-7-S	5.39	7.40	462.88	906202	0.0224
8	Cement Tc & I	121004-8-S	6.20	7.60	339.29	307423	BLD
19	Saltstone Tc & I	121004-19-S	5.15	7.70	337.15	500237	BLD
20	Saltstone Tc & I	121004-20-S	6.40	7.70	260.03	879884	BLD

Table B2: Summarized Data for the lysimeters containing 99Tc and stable iodine from the FY13Q1 sampling interval

Sample Interval: FY13Q1 Date Sample taken @ SRS: 01/09/2013 Date Sample Analyzed/Prepared at Clemson University: 04/17/2013						Activity or Mass Concentration (Bq/L for ^{99}Tc or ppm for I)	
Lysimeter number	Constituent(s)	Sample ID	pH	DO	Vol. measured @ Clemson Univ. (mL)	^{99}Tc	Iodine
7	Cement Tc & I	130109-7-S	4.70	7.70	498.93	3456660	0.0104
8	Cement Tc & I	130109-8-S	4.87	7.70	596.23	3646757	BLD
19	Saltstone Tc & I	130109-19-S	4.59	7.50	406.54	3564398	BLD
20	Saltstone Tc & I	130109-20-S	4.70	7.40	231.36	3335746	BLD

Table B3: Summarized Data for the lysimeters containing 99Tc and stable iodine from the FY13Q2 sampling interval

Sample Interval: FY13Q2* Date Sample taken @ SRS: 02/12/2013 and 03/07/2013 Date Sample Analyzed/Prepared at Clemson University: 05/09/2013						Activity or Mass Concentration (Bq/L for ^{99}Tc or ppm for I)	
Lysimeter number	Constituent(s)	Sample ID	pH	DO	Vol. measured @ Clemson Univ. (mL)	99Tc	Iodine
7	Cement Tc & I	130212-7-S	4.77	8.20	709.97	2472342	0.0091
7	Cement Tc & I	130307-7-S	4.89	8.10	1111.09	738894	BLD
8	Cement Tc & I	130212-8-S	4.95	8.40	771.17	2661447	BLD
8	Cement Tc & I	130307-8-S	4.98	8.30	959.33	836698	BLD
19	Saltstone Tc & I	130212-19-S	4.57	8.10	188.99	2910442	BLD
19	Saltstone Tc & I	130307-19-S	4.71	7.90	285.99	1015858	BLD

***Samples for lysimeter 20 from the FY13Q2 sampling interval were not received**

Appendix C: Summary of Data for Lysimeters Containing Gamma Suites

Table C1: Summarized data for the lysimeters containing gamma emitting radionuclides from the FY12Q4 sampling interval

Sample Interval: FY12Q4 Date Sample taken @ SRS: 10/04/2012 Date Sample Analyzed/Prepared at Clemson University: 11/11/2012*						Total Activity in Effluent (Bq)			
Lysimeter number	Constituent(s)	Sample ID	pH	DO	Vol. measured @ Clemson Univ. (mL)	^{137}Cs**	^{60}Co**	^{133}Ba**	^{152}Eu**
2	Cement Control	100412L2	6.73	7.90	398.82	BLD	BLD	BLD	BLD
3	Cement Control	100412L3	6.67	8.00	76.70	BLD	BLD	BLD	BLD
4	Cement Gamma Suite	100412L4	6.57	8.20	296.00	120(+/-)2.2	1281(+/-)25.5	3.0(+/-)4.4	BLD
5	Cement Gamma Suite	100412L5	7.01	8.00	338.81	BLD	5131(+/-)56.6	21.0(+/-)32.2	BLD
6	Cement Gamma Suite	100412L6	5.72	8.10	323.27	BLD	1861(+/-)120.3	BLD	77.0(+/-)21.0
14***	Saltstone Control	-	-	-	-	-	-	-	-
15	Saltstone Control	100412L15	6.21	8.00	412.71	BLD	BLD	BLD	BLD
16	Saltstone Gamma Suite	100412L16	7.27	8.00	326.78	BLD	182(+/-)41.1	BLD	BLD
17	Saltstone Gamma Suite	100412L17	5.02	8.00	391.00	82(+/-)1.5	1550(+/-)108	BLD	BLD
18	Saltstone Gamma Suite	100412L18	5.55	8.20	340.50	47(+/-)0.9	1377(+/-)81.1	BLD	14.0(+/-)24.0
25	Sediment Control	100412L25	6.54	7.90	414.16	BLD	BLD	BLD	BLD
26	Gamma Suite	100412L26	6.57	7.90	379.61	BLD	119(+/-)43.0	BLD	BLD
27	Gamma Suite	100412L27	6.61	7.80	373.06	BLD	181(+/-)135	3.0(+/-)4.8	BLD
28	Gamma Suite	100412L28	6.18	7.90	451.12	110(+/-)2.0	5.0(+/-)7.6	BLD	28.0(+/-)49.0

***Date of pH and DO measurements. Samples were counted between 11/16/2012 – 2/07/2013. **Error measurements for ^{137}Cs were calculated using counting statistics assuming an 80% confidence in the error associated with the efficiency calibration curve. The (+/-) for ^{60}Co, ^{133}Ba and ^{152}Eu is the standard deviation of the activity measurements determined from characteristic peaks for each isotope.**
*****-DNR(-): Data not available because the sample was not received**

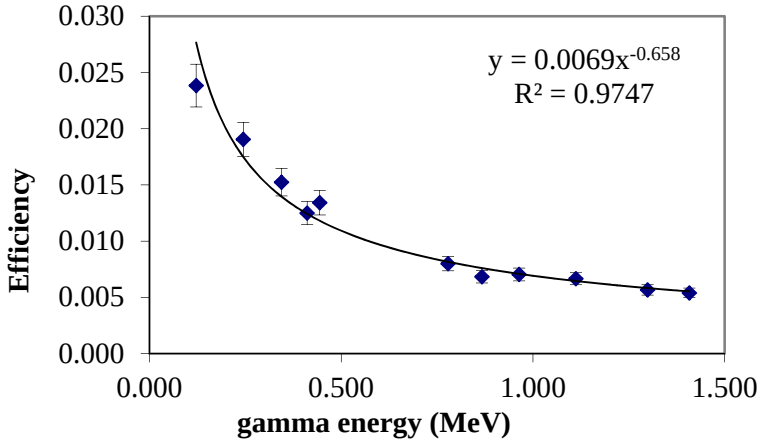

Figure C1: HPGe6 Efficiency curve used for analysis of samples from FY12Q4 sampling interval determined from counting 45mL of a ^{152}Eu standard in a 50mL conical centrifuge tube

Table C2: Summarized data for the lysimeters containing gamma emitting radionuclides from the FY13Q1 sampling interval

Sample Interval: FY13Q1 Date Sample taken @ SRS: 01/09/2013 Date Sample Analyzed/Prepared at Clemson University: 04/02/2013*						Total Activity in Effluent (Bq)			
Lysimeter number	Constituent(s)	Sample ID	pH	DO	Vol. measured @ Clemson Univ. (mL)	^{137}Cs**	^{60}Co**	^{133}Ba**	^{152}Eu**
2	Cement Control	130109-2-S	4.95	7.40	519.96	BLD	BLD	BLD	BLD
3	Cement Control	130109-3-S	5.57	7.40	295.95	BLD	BLD	BLD	BLD
4	Cement Gamma Suite	130109-4-S	4.95	7.70	410.53	2503(+/-)46.05	8102(+/-)101.1	16(+/-)23	BLD
5	Cement Gamma Suite	130109-5-S	5.78	7.80	513.08	458(+/-)9.00	84.0(+/-)119	21(+/-)21	BLD
6	Cement Gamma Suite	130109-6-S	4.73	7.60	533.85	379(+/-)6.98	50894(+/-)1037.0	76(+/-)29	BLD
14	Saltstone Control	130109-14-S***	-	-	22.22	-	-	-	-
15	Saltstone Control	130109-15-S	4.78	7.30	568.6	BLD	BLD	BLD	BLD
16	Saltstone Gamma Suite	130109-16-S	4.53	7.30	444.35	BLD	BLD	BLD	BLD
17	Saltstone Gamma Suite	130109-17-S	4.61	7.60	571.54	75(+/-)1.3	BLD	9.0(+/-)16	BLD
18	Saltstone Gamma Suite	130109-18-S	4.72	7.60	520.83	BLD	3972(+/-)1354	BLD	133(+/-)230
25	Sediment Control	130109-25-S	5.51	7.50	180.25	BLD	BLD	BLD	BLD
26	Gamma Suite	130109-26-S	5.26	7.50	325.00	BLD	22.0(+/-)30.7	102(+/-)117	11(+/-)20
27	Gamma Suite	130109-27-S	5.50	7.70	451.44	BLD	177(+/-)73.2	14(+/-)24	BLD
28	Gamma Suite	130109-28-S	5.32	7.80	510.07	41(+/-)0.7	118(+/-)62.6	11(+/-)13	33(+/-)41

***Date of pH and DO measurements. Samples were counted between 5/03/2013 – 5/18/2013.**

****Error measurements (+/-) for ^{137}Cs were calculated using counting statistics assuming an 80% confidence in the error associated with the efficiency calibration curve. The (+/-) for ^{60}Co, ^{133}Ba and ^{152}Eu is the standard deviation of the activity measurements determined from characteristic peaks for each isotope.**

*****Insufficient volume for measurement. Therefore, data are not reported.**

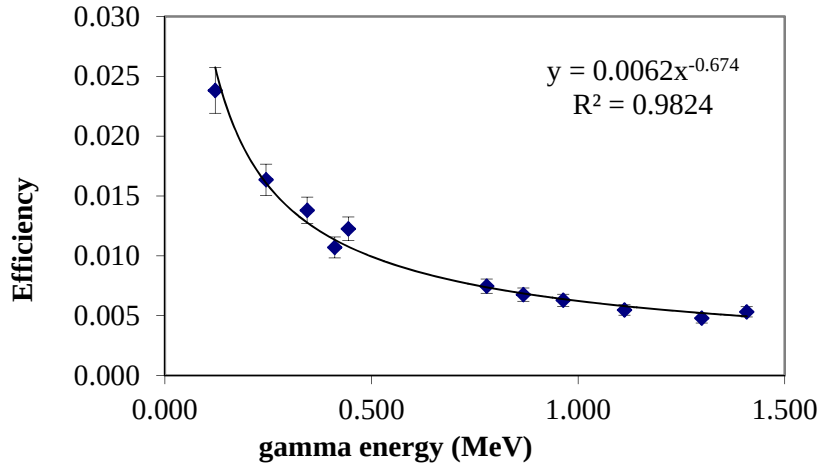

Figure C2: HPGe6 Efficiency curve used for analysis of samples from FY13Q1 sampling interval determined from counting 45mL of a ^{152}Eu standard in a 50mL conical centrifuge tube.

Table C3: Summarized data for the lysimeters containing gamma emitting radionuclides from the FY13Q2 sampling interval

Sample Interval: FY13Q2 Date Sample taken @ SRS: 03/07/2013 Date Sample Analyzed/Prepared at Clemson University: 05/07/2013*						Total Activity in Effluent (Bq)			
Lysimeter number	Constituent(s)	Sample ID	pH	DO	Vol. measured @ Clemson Univ. (mL)	^{137}Cs	^{60}Co	^{133}Ba	^{152}Eu
2	Cement Control	130307-2-S	5.19	7.80	1773.21	BLD	BLD	BLD	BLD
3	Cement Control	130307-3-S	4.88	8.10	1371.43	BLD	BLD	BLD	BLD
4	Cement Gamma Suite	130307-4-S	4.90	7.80	1467.93	BLD	61765(+/-)2280.3	BLD	BLD
5	Cement Gamma Suite	130307-5-S	5.25	7.90	1721.21	BLD	145782(+/-)2335.4	1.9(+/-)3.2	35(+/-)61
6	Cement Gamma Suite	130307-6-S	4.94	7.70	1601.21	BLD	87046(+/-)5245.5	BLD	BLD
14	Saltstone Control	130307-14-S	4.74	8.00	1069.04	BLD	BLD	BLD	BLD
15	Saltstone Control	130307-15-S	5.18	7.80	1517.73	BLD	BLD	BLD	BLD
16	Saltstone Gamma Suite	130307-16-S	4.88	8.10	1366.82	BLD	3091(+/-)747.9	BLD	BLD
17	Saltstone Gamma Suite	130307-17-S	5.04	8.10	1680.71	BLD	13607(+/-)16216	25(+/-)44	BLD
18	Saltstone Gamma Suite	130307-18-S	5.13	7.90	1654.71	BLD	9216(+/-)511.7	206(+/-)278	BLD
25	Sediment Control	130307-25-S	5.80	7.50	309.60	BLD	BLD	BLD	BLD
26	Gamma Suite	130307-26-S	5.18	7.70	1156.38	BLD	BLD	BLD	BLD
27	Gamma Suite	130307-27-S	5.22	7.90	1490.40	BLD	BLD	BLD	BLD
28	Gamma Suite	130307-28-S	5.27	8.00	1360.09	BLD	BLD	BLD	46(+/-)80

***Date of pH and DO measurements. Samples were counted between 5/21/2013 – 6/25/2013. **Error measurements (+/-) for ^{137}Cs were calculated using counting statistics assuming an 80% confidence in the error associated with the efficiency calibration curve. The (+/-) for ^{60}Co, ^{133}Ba and ^{152}Eu is the standard deviation of the activity measurements determined from characteristic peaks for each isotope.**

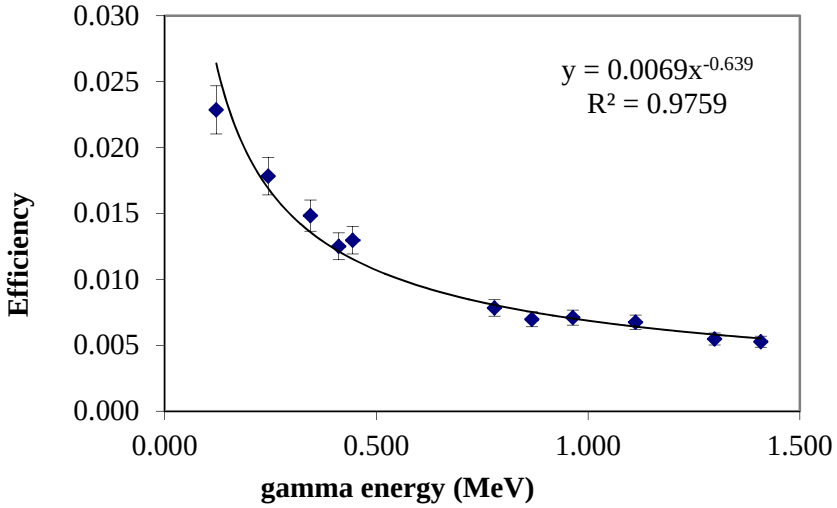

Figure C3: HPGe6 Efficiency curve used for analysis of samples from FY13Q2 and FY13Q3 sampling intervals determined from counting 45mL of a ^{152}Eu standard in a 50mL conical centrifuge tube.

Table C4: Summarized data for the lysimeters containing gamma emitting radionuclides from the FY13Q3 sampling interval

Sample Interval: FY13Q3 Date Sample taken @ SRS: 06/11/2013 and 06/17/2013 Date Sample Analyzed/Prepared at Clemson University: 07/01/2013*						Total Activity in Effluent (Bq)			
Lysimeter number	Constituent(s)	Sample ID	pH	DO	Vol. measured @ Clemson Univ. (mL)	^{137}Cs**	^{60}Co**	^{133}Ba**	^{152}Eu**
2	Cement Control	130617-2-S	5.22	8.50	2041.21	BLD	BLD	BLD	BLD
3	Cement Control	130617-3-S	5.15	8.00	2040.21	BLD	BLD	BLD	BLD
4	Cement Gamma Suite	130617-4-S	3.94	8.00	1632.71	BLD	31738(+/-)376.1	BLD	BLD
5	Cement Gamma Suite	130617-5-S	4.03	8.01	2050.21	BLD	1474574(+/-)14443.97	539 (+/-)933	2003(+/-)2203
6	Cement Gamma Suite	130617-6-S	3.65	8.10	1254.21	BLD	107035(+/-)148.2	BLD	BLD
14	Saltstone Control	130611-14-S	4.82	8.10	2001.21	BLD	BLD	BLD	BLD
15	Saltstone Control	130611-15-S	5.53	8.30	1994.71	BLD	BLD	BLD	BLD
16	Saltstone Gamma Suite	130611-16-S	4.97	8.30	1994.21	BLD	28669(+/-)2598	39(+/-)49	BLD
17	Saltstone Gamma Suite	130611-17-S	5.48	8.10	1995.71	BLD	11270(+/-)13439	22(+/-)38	BLD
18	Saltstone Gamma Suite	130613-18-S	4.82	8.20	347.92	BLD	743 (+/-) 11	BLD	BLD
25	Sediment Control	130613-25-S	4.78	8.30	1425.71	BLD	BLD	BLD	BLD
26	Gamma Suite	130613-26-S	4.65	8.30	1189.71	2334(+/-)42.3	BLD	BLD	77(+/-)134
27	Gamma Suite	130613-27-S	4.68	8.30	2043.71	BLD	213(+/-)302	7(+/-)12	178(+/-)154
28	Gamma Suite	130613-28-S	4.62	8.30	2013.71	BLD	BLD	BLD	BLD

***Date of pH and DO measurements. Samples were counted between 7/21/2013 – 8/17/2013.**

****Error measurements (+/-) for ^{137}Cs were calculated using counting statistics assuming an 80% confidence in the error associated with the efficiency calibration curve. The (+/-) for ^{60}Co, ^{133}Ba and ^{152}Eu is the standard deviation of the activity measurements determined from characteristic peaks for each isotope.**

Appendix D: Summary of data for the lysimeters containing actinides (^{237}Np and $^{239/240}$Pu)

Table D1: ICP-MS sensitivity and intercept concentration (detection limit) for ^{239}Pu and ^{237}Np from the calibration data used for analysis of samples from the FY12Q4, FY13Q1, FY13Q2, and FY13Q3 sampling intervals.

Date of Run	Sensitivity, ^{237}Np (detector ion counts per ppb ^{237}Np)	Sensitivity, ^{239}Pu (detector ion counts per ppb ^{239}Pu)	Intercept Concentration, ^{237}Np	Intercept Concentration, ^{239}Pu
11/15/2012	28501	-	0.04475	-
04/18/2013	6525.4	207307	7.7214	0.00628
05/21/2013	25856	21508	0.00572	0.00216
07/08/2013	31288	27197	0.00290	0.0004

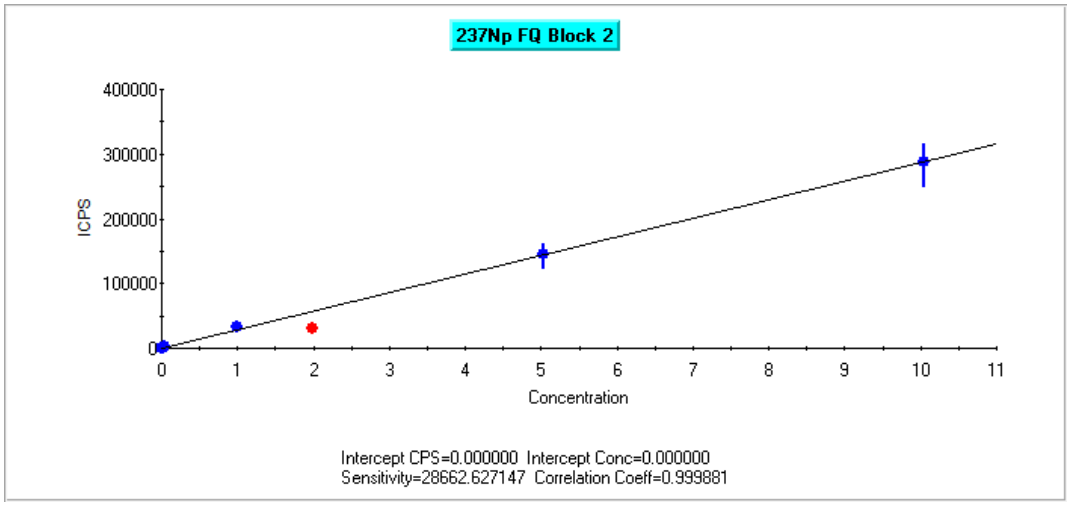

Figure D1: Screen capture of the ^{237}Np calibration curve using Thermo Plasma Lab software to control the data collection and analysis for the FY12Q4 sampling interval. R^2=0.99988, Intercept Conc. (Detection Limit) = 0.00000 ppb.

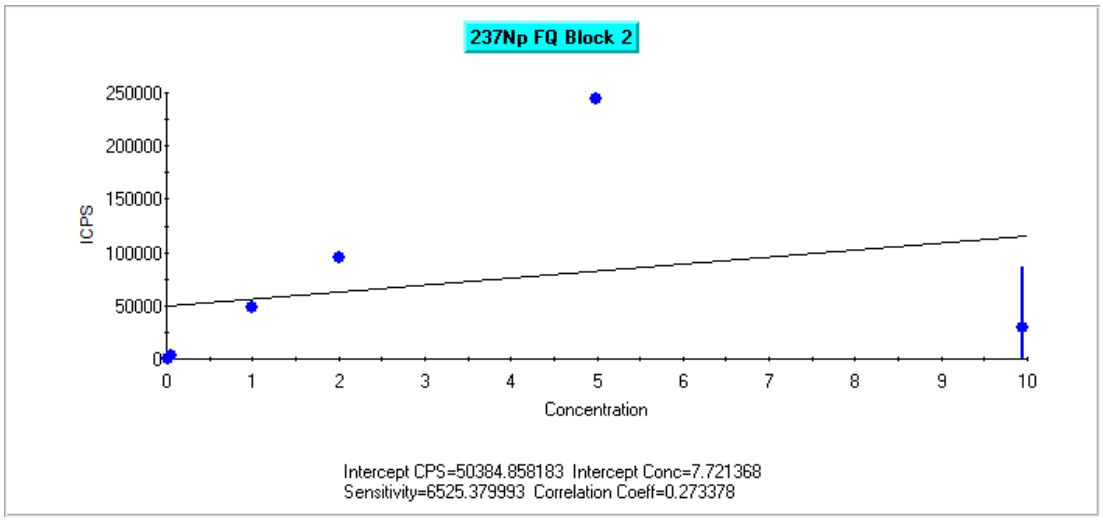

Figure D2: Screen capture of the ^{237}Np calibration curve using Thermo Plasma Lab software to control the data collection and analysis for the FY13Q1 sampling interval. R^2=0.27338, Intercept Conc. (Detection Limit) = 7.7214 ppb.

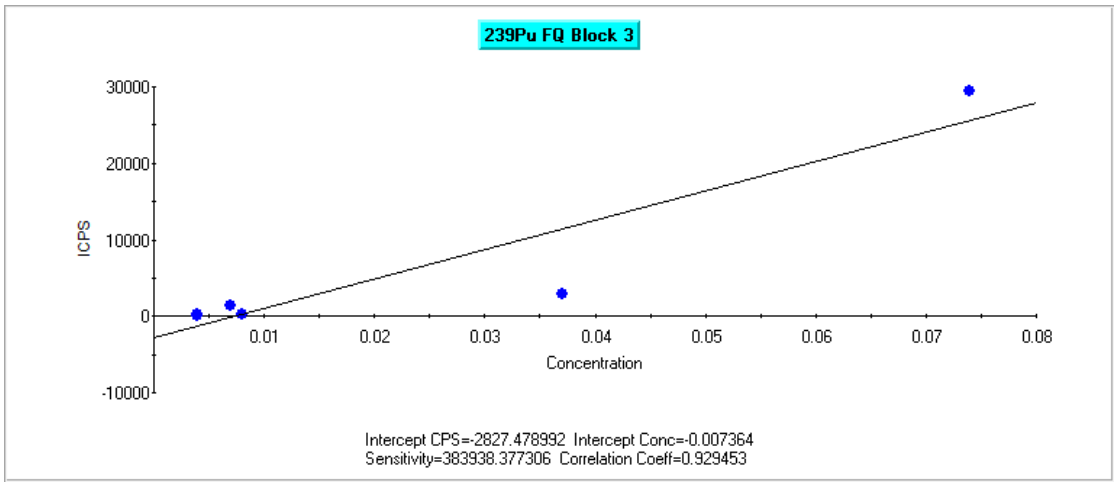

Figure D3: Screen capture of the ^{239}Pu calibration curve using Thermo Plasma Lab software to control the data collection and analysis for the FY13Q1 sampling interval. R^2=0.92945, Intercept Conc. (Detection Limit) = 0.00736 ppb.

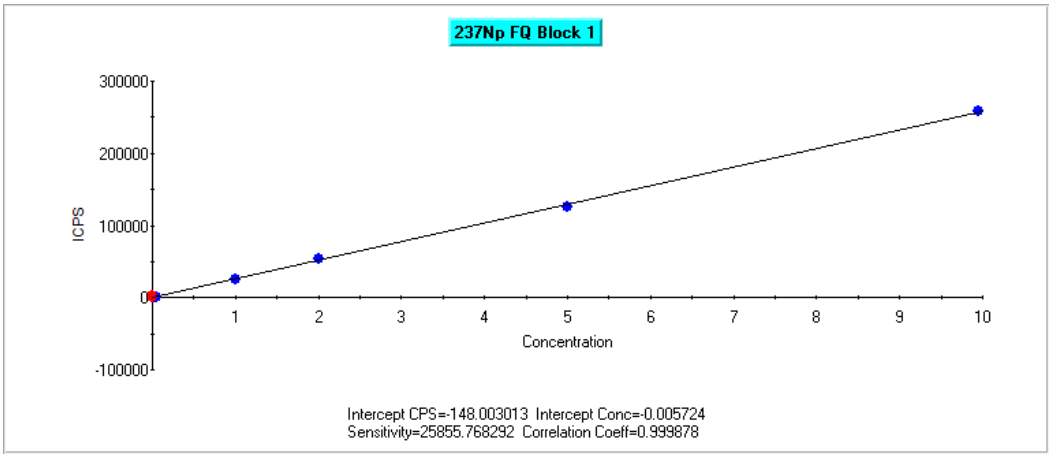

Figure D4: Screen capture of the ^{237}Np calibration curve using Thermo Plasma Lab software to control the data collection and analysis for the FY13Q2 sampling interval. R^2=0.99988, Intercept Conc. (Detection Limit) = 0.00572 ppb.

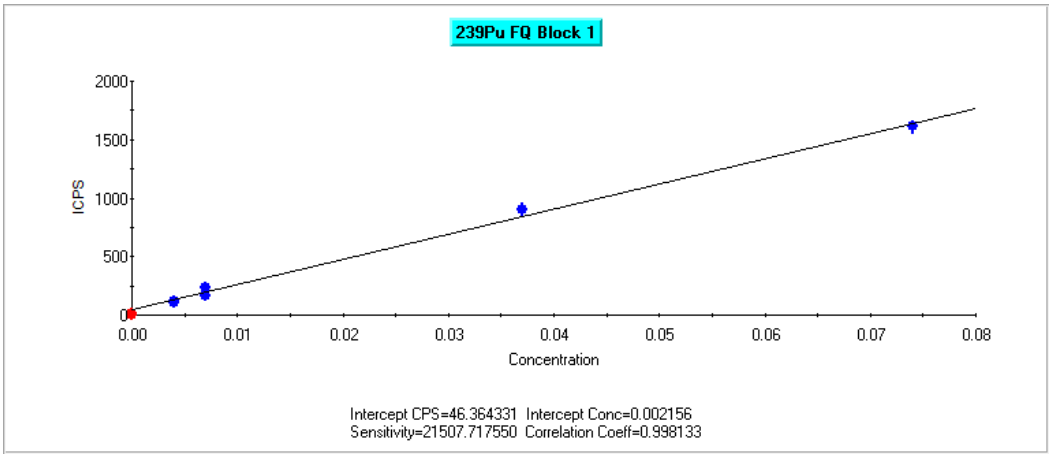

Figure D5: Screen capture of the ^{239}Pu calibration curve using Thermo Plasma Lab software to control the data collection and analysis for the FY13Q2 sampling interval. R^2=0.99813, Intercept Conc. (Detection Limit) = 0.00216 ppb.

Table D2: Summarized data for lysimeters containing ^{237}Np and ^{239}Pu from the FY12Q4 sampling interval.

Sample Interval: FY12Q4 Date Sample taken @ SRS: 10/04/2012 Date Sample Analyzed/Prepared at Clemson University: 11/11/2012						Activity (Bq)	
Lysimeter number	Constituent (s)	Sample ID	pH	DO	Vol. measured @ Clemson Univ. (mL)	^{237}Np	^{239}Pu
9	Pu(IV)-oxalate, grass	100412L9	5.42	8.10	210.09	-	BLD
10	Pu(IV)-oxalate, grass	100412L10	5.65	8.00	199.95	-	BLD
11	Pu(IV)-oxalate, grass	100412L11	6.54	8.00	575.87	-	BLD
12	Grass Control	100412L12	6.73	7.80	702.73	-	BLD
21	$Pu(V)NH_4(CO_3)$/OM	100412L21	6.48	8.10	668.55	-	BLD
22	$Pu(V)NH_4(CO_3)$/OM	100412L22	7.05	7.90	71.42	-	BLD
23	$Pu(V)NH_4(CO_3)$/OM	100412L23	5.53	7.90	920.19	-	BLD
24	Instrumental Control	100412L24	5.74	8.00	822.35	-	BLD
29	Np(V)nitrate	100412L29	5.12	7.80	460.10	BLD	-
30	Np(V)nitrate	100412L30	6.57	7.80	571.55	BLD	-
31	$Np(IV)O_2$	100412L31	6.33	7.70	632.47	BLD	-
32	$Np(IV)O_2$	100412L32	5.67	8.00	490.45	BLD	-
33	Pu(III)oxalate	100412L33	5.88	8.10	590.76	-	BLD
34	Pu(III)oxalate	100412L34	6.33	7.90	246.97	-	BLD
35	Pu(III)oxalate	100412L35	6.39	8.00	599.08	-	BLD
37	Instrumental Control	100412L37	5.37	7.90	843.41	-	BLD
38	Pu(IV)oxalate	100412L38	6.02	8.10	588.05	-	BLD
39	Pu(IV)oxalate	100412L39	6.18	8.10	572.58	-	BLD
40	Pu(IV)oxalate	100412L40	5.41	8.00	723.42	-	BLD
41	$Pu(V)NH_4(CO_3)$	100412L41	5.65	7.70	405.94	-	BLD
42	$Pu(V)NH_4(CO_3)$	100412L42	5.44	8.00	506.63	-	BLD
43	$Pu(V)NH_4(CO_3)$	100412L43	5.54	7.80	638.09	-	BLD
44	Pu Colloids	100412L44	5.86	7.90	651.61	-	BLD
45	Pu Colloids	100412L45	5.29	7.90	601.53	-	BLD
46	Pu Colloids	100412L46	5.40	8.00	617.24	-	BLD

-NA (not applicable)

Table D3: Summarized data for lysimeters containing ^{237}Np and ^{239}Pu from the FY13Q1 sampling interval.

Sample Interval: FY13Q1 Date Sample taken @ SRS: 01/09/2013 Date Sample Analyzed/Prepared at Clemson University: 04/02/2013						Activity (Bq)	
Lysimeter number	Constituent (s)	Sample ID	pH	DO	Vol. measured @ Clemson Univ. (mL)	^{237}Np	^{239}Pu
9	Pu(IV)-oxalate, grass	130109-9-S	5.39	7.40	333.90	-	BLD
10	Pu(IV)-oxalate, grass	130109-10-S	5.49	7.50	112.25	-	BLD
11	Pu(IV)-oxalate, grass	130109-11-S	5.58	7.50	456.76	-	BLD
12	Grass Control	130109-12-S	5.67	7.40	563.03	-	BLD
21	$Pu(V)NH_4(CO_3)$/OM	130109-21-S	4.78	7.60	332.39	-	BLD
22	$Pu(V)NH_4(CO_3)$/OM	130109-22-S*	-	-	-	-	BLD
23	$Pu(V)NH_4(CO_3)$/OM	130109-23-S	4.58	7.40	823.24	-	BLD
24	Instrumental Control	130109-24-S	5.16	7.40	782.23	-	BLD
29	Np(V)nitrate	130109-29-S	4.95	7.70	533.09	BLD	-
30	Np(V)nitrate	130109-30-S	5.21	7.60	545.36	BLD	-
31	$Np(IV)O_2$	130109-31-S	5.19	7.50	381.33	BLD	-
32	$Np(IV)O_2$	130109-32-S	5.35	7.70	517.43	BLD	-
33	Pu(III)oxalate	130109-33-S	5.20	7.80	548.50	-	BLD
34	Pu(III)oxalate	130109-34-S	5.49	7.30	191.91	-	BLD
35	Pu(III)oxalate	130109-35-S	5.11	7.50	545.21	-	BLD
37	Instrumental Control	130109-37-S	4.97	7.40	784.24	-	BLD
38	Pu(IV)oxalate	130109-38-S	5.15	7.50	574.60	-	BLD
39	Pu(IV)oxalate	130109-39-S	5.18	7.40	628.23	-	BLD
40	Pu(IV)oxalate	130109-40-S	4.98	7.40	518.32	-	BLD
41	$Pu(V)NH_4(CO_3)$	130109-41-S	4.95	7.40	570.69	-	BLD
42	$Pu(V)NH_4(CO_3)$	130109-42-S	5.61	7.50	432.99	-	BLD
43	$Pu(V)NH_4(CO_3)$	130109-43-S	5.49	7.50	168.25	-	BLD
44	Pu Colloids	130109-44-S	5.07	7.70	561.99	-	BLD
45	Pu Colloids	130109-45-S	5.15	7.60	484.81	-	BLD
46	Pu Colloids	130109-46-S	5.20	7.80	505.70	-	BLD

-NA (not applicable)

*Insufficient volume for measurement

Table D4: Summarized data for lysimeters containing ^{237}Np and ^{239}Pu from the FY13Q2 sampling interval.

Sample Interval: FY13Q2 Date Sample taken @ SRS: 03/07/2013 Date Sample Analyzed/Prepared at Clemson University: 05/07/2013						Activity (Bq)	
Lysimeter number	Constituent (s)	Sample ID	pH	DO	Vol. measured @ Clemson Univ. (mL)	^{237}Np	^{239}Pu
9	Pu(IV)-oxalate, grass	130307-9-S	5.33	7.60	1485.37	-	BLD
10	Pu(IV)-oxalate, grass	130307-10-S	5.34	7.70	1601.21	-	BLD
11	Pu(IV)-oxalate, grass	130307-11-S	5.53	7.60	1268.03	-	BLD
12	Grass Control	130307-12-S	5.24	7.60	1811.71	-	BLD
21	$Pu(V)NH_4(CO_3)$/OM	130307-21-S	4.78	7.70	1012.18	-	BLD
22	$Pu(V)NH_4(CO_3)$/OM	130307-22-S*	-	-	59.64	-	BLD
23	$Pu(V)NH_4(CO_3)$/OM	130307-23-S	5.11	7.40	1738.71	-	BLD
24	Instrumental Control	130307-24-S	5.42	7.50	1260.04	-	BLD
29	Np(V)nitrate	130307-29-S	5.14	8.20	1746.71	BLD	-
30	Np(V)nitrate	130307-30-S	5.68	8.10	1678.71	BLD	-
31	$Np(IV)O_2$	130307-31-S	5.20	7.70	867.94	BLD	-
32	$Np(IV)O_2$	130307-32-S	5.28	7.80	1587.21	BLD	-
33	Pu(III)oxalate	130307-33-S	5.39	8.10	1214.23	-	BLD
34	Pu(III)oxalate	130307-34-S	5.22	7.90	434.21	-	BLD
35	Pu(III)oxalate	130307-35-S	5.17	7.70	1484.13	-	BLD
37	Instrumental Control	130307-37-S	5.10	7.50	1838.21	-	BLD
38	Pu(IV)oxalate	130307-38-S	5.21	7.70	1729.71	-	BLD
39	Pu(IV)oxalate	130307-39-S	5.10	7.60	1628.21	-	BLD
40	Pu(IV)oxalate	130307-40-S	5.12	7.70	1593.21	-	BLD
41	$Pu(V)NH_4(CO_3)$	130307-41-S	5.04	7.90	1737.71	-	BLD
42	$Pu(V)NH_4(CO_3)$	130307-42-S	5.63	7.60	1172.48	-	BLD
43	$Pu(V)NH_4(CO_3)$	130307-42-S	5.41	7.60	826.60	-	BLD
44	Pu Colloids	130307-44-S	5.27	7.90	1510.30	-	BLD
45	Pu Colloids	130307-45-S	5.28	7.60	1377.93	-	BLD
46	Pu Colloids	130307-46-S	5.23	7.90	1568.21	-	BLD

-NA (not applicable)

*Insufficient volume for measurement

Table D4: Summarized data for lysimeters containing ^{237}Np and ^{239}Pu from the FY13Q3sampling interval.

Sample Interval: FY13Q3 Date Sample taken @ SRS: 05/14/2013, 06/11/2013 and 06/13/2013 Date Sample Analyzed/Prepared at Clemson University: 07/01/2013						Activity (Bq)	
Lysimeter number	Constituent (s)	Sample ID	pH	DO	Vol. measured @ Clemson Univ. (mL)	^{237}Np	^{239}Pu
9	Pu(IV)-oxalate, grass	130613-9-S	4.75	8.30	1729.21	-	BLD
10	Pu(IV)-oxalate, grass	130613-10-S	4.67	8.10	365.71	-	BLD
11	Pu(IV)-oxalate, grass	130613-11-S	4.76	8.20	1826.71	-	BLD
12	Grass Control	130613-12-S	4.70	8.20	2045.71	-	BLD
21	$Pu(V)NH_4(CO_3)$/OM	130613-21-S	5.40	8.30	332.98	-	-
22*	$Pu(V)NH_4(CO_3)$/OM	-	-	-	-	-	-
23	$Pu(V)NH_4(CO_3)$/OM	130613-23-S	5.0	8.20	511.52	-	-
24*	Instrumental Control	-	-	-	-	-	-
29	Np(V)nitrate	130613-29-S	4.61	8.20	2038.21	BLD	-
30	Np(V)nitrate	130617-30-S	4.69	8.00	2033.71	146	-
31	$Np(IV)O_2$	130613-31-S	4.72	8.20	1818.71	BLD	-
32	$Np(IV)O_2$	130613-32-S	4.93	8.20	2001.21	BLD	-
33	Pu(III)oxalate	130613-33-S	4.66	8.20	2040.21	-	BLD
34	Pu(III)oxalate	130613-34-S	4.90	8.20	413.21	-	BLD
35	Pu(III)oxalate	130613-35-S	4.73	8.10	1651.71	-	BLD
37	Instrumental Control	130613-37-S	4.84	8.20	2011.21	-	BLD
38	Pu(IV)oxalate	130613-38-S	4.70	8.10	2011.71	-	BLD
39	Pu(IV)oxalate	130613-39-S	4.69	8.00	2025.21	-	BLD
40	Pu(IV)oxalate	130613-40-S	4.59	8.30	2008.71	-	BLD
41	$Pu(V)NH_4(CO_3)$	130613-41-S	4.67	8.20	1810.21	-	BLD
42	$Pu(V)NH_4(CO_3)$	130514-42-S	5.10	8.30	960.21	-	BLD
43	$Pu(V)NH_4(CO_3)$	130514-43-S	5.02	7.90	405.21	-	BLD
44	Pu Colloids	130514-44-S	5.14	8.30	842.21	-	BLD
45	Pu Colloids	130514-45-S	4.01	8.30	802.71	-	BLD
46	Pu Colloids	130514-46-S	4.91	8.10	906.21	-	BLD

*Data not available because the sample was not received