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K West Basin Sand Filter Backwash Sample Analysis

March 2016

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MR Smoot
DS Coffey
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U.S. DEPARTMENT OF ENERGY

Prepared for the U.S. Department of Energy
under Contract DE-AC05-76RL01830

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Pacific Northwest National Laboratory
Richland, Washington 99352

Executive Summary

A sand filter is used to help maintain water clarity at the K West Basin where highly radioactive sludge is stored. Eventually that sand filter will require disposal. The radionuclide content of the solids trapped in the sand filter will affect the selection of the sand filter disposal pathway. The Pacific Northwest National Laboratory (PNNL) was contracted by the K Basin Operations & Plateau Remediation Project (operations contractor CH2M Hill) to analyze the radionuclide content of the solids collected from the backwash of the K West Basin sand filter. The radionuclide composition in the sand filter backwash solids will be used by CH2M Hill to determine if the sand filter media and retained sludge solids will be designated as transuranic waste for disposal purposes or can be processed through less expensive means.

On October 19, 2015, K Basin Operations & Plateau Remediation Project staff backwashed the sand filter into the North Load-Out Pit (NLOP) and immediately collected sample slurry from a sampling tube positioned 24 in. above the NLOP floor. The 764 g sand filter backwash slurry sample, KW-105 SFBW-001, was submitted to PNNL for analysis on October 20, 2015. Solids from the slurry sample were consolidated into two samples (i.e., a primary and a duplicate sample) by centrifuging and measured for mass (0.82 g combined – wet centrifuged solids basis) and volume (0.80 mL combined). The solids were a dark brown/orange color, consistent with iron oxide/hydroxide. The solids were dried; the combined dry solids mass was 0.1113 g, corresponding to 0.0146 weight percent (wt%) solids in the original submitted sample slurry. The solids were acid-digested using nitric and hydrochloric acids. Insoluble solids developed upon dilution with 0.5 M HNO_3, corresponding to an average 6.5 wt% of the initial dry solids content.

The acid digestate and insoluble solids were analyzed separately by gamma spectrometry. Nominally, 7.7% of the ^{60}Co was present in the insoluble solids; less than 1% of other gamma-emitters (i.e., ^{137}Cs, $^{154/155}Eu$, and ^{241}Am) were present in the insoluble solids.

Aliquots of the acid digestate were analyzed directly using gamma energy analysis (GEA) and after separations for ^{238}Pu, $^{239+240}Pu$, ^{237}Np, and ^{241}Am radioisotopes using alpha energy analysis (AEA). The ^{90}Sr was measured by liquid scintillation counting (LSC) on the Sr-separated fraction. The plutonium isotopic distribution of the acid digestate was analyzed following Pu separations by thermal ionization mass spectrometry (TIMS). Table ES.1 summarizes the results for the primary and duplicate samples.

The $^{239+240}Pu$ concentration (µCi/g dry) relative to ^{90}Sr and to ^{137}Cs concentrations (µCi/g dry) was examined. The K West Basin sludge has a $^{239+240}Pu/^{90}Sr$ ranging from 0.1 to 1.2 and the $^{239+240}Pu/^{137}Cs$ ratio ranging from 0.10 to 0.47. In contrast, the sand filter backwash solids $^{239+240}Pu/^{90}Sr$ ratio was 10.6 and the $^{239+240}Pu/^{137}Cs$ ratio was 2.0. The ratio differences indicate a relative enhancement of the Pu concentration in the sand filter solids relative to the ^{137}Cs and ^{90}Sr sludge concentrations currently in the K West Basin. A dose-to-curie radioisotope evaluation of the sand filter waste form may need to consider this dissimilarity.

Table ES.1. Radionuclide Characterization in KW-105 SFBW-001 Acid Digestate (Dry Solids Mass Basis)

Analyte	68122-TI-001-F (Primary Sample)	68122-TI-001-G (Duplicate Sample)	Average	Relative Percent Difference
	μCi/g Dry Mass			
^{60}Co (GEA)	1.08E-1[(a)]	9.91E-2[(a)]	1.04E-1[(a)]	8.6
^{137}Cs (GEA)	1.02E+2	9.49E+1	9.85E+1	7.2
^{152}Eu (GEA)	3.88E-2	3.13E-2	3.51E-2	**21**[(b)]
^{154}Eu (GEA)	1.54E+0	1.46E+0	1.50E+0	5.3
^{155}Eu (GEA)	2.28E-1	2.55E-1	2.42E-1	11
^{238}Pu (AEA)	2.63E+01	2.61E+1	2.62E+1	0.8
$^{239+240}$Pu (AEA)	1.99E+2	2.00E+2	2.00E+2	0.4
^{241}Am (GEA)	9.64E+1	1.06E+2	1.01E+2	9.5
^{241}Am (AEA)	9.39E+1	8.69E+1	9.04E+1	7.7
^{237}Np (AEA)	1.80E-3	2.09E-3	1.94E-3	15
^{90}Sr (LSC)	1.88E+1	1.88E+1	1.88E+1	0.5
Plutonium Isotopic Fractionation, Weight % Basis				
^{238}Pu (AEA)	0.0639	0.0632	0.0635	1.1
^{239}Pu (TIMS)	86.33	86.33	86.33	0.002
^{239}Pu (TIMS)	12.98	12.98	12.98	0.04
^{241}Pu (TIMS)	0.3935	0.3915	0.3925	0.50
^{242}Pu (TIMS)	0.2363	0.2354	0.2359	0.38

The GEA analyte reference date is November 24, 2015; the AEA and ^{90}Sr measured by LSC reference date is December 15-30, 2015.

(a) An additional 7.0 and 8.5% of the ^{60}Co activity remained with the undissolved solids for the sample and duplicate, respectively.

(b) The RPD passed the mean difference test; see Appendix B.

Acknowledgments

The authors wish to acknowledge the work of Lori Darnell, Katharine Carson, Michael Cantaloub, Larry Greenwood, Chuck Soderquist, James Peterson, and Truc Trang-Le for their radioanalytical work inclusive of acid digestions, separations, counting, and reporting. The authors also thank Andy Schmidt, Cal Delegard, and Rick Shimskey for technical reviews of data calculations and final report. Contributions from Mike Parker, technical editor, are also greatly appreciated.

Acronyms and Abbreviations

AEA	alpha energy analysis
ASO	Analytical Support Operations
ASR	Analytical Service Request
BTR	Buyer's Technical Representative
CHPRC	CH2M Hill Plateau Remediation Company
GEA	gamma energy analysis
HASQARD	Hanford Analytical Services Quality Assurance Requirements Document
ID	identification
KBO & PR	K Basin Operations & Plateau Remediation Project
LSC	liquid scintillation counting
NBS	National Bureau of Standards
NLOP	North Load-Out Pit
PNNL	Pacific Northwest National Laboratory
QC	quality control
RPD	relative percent difference
RPL	Radiochemical Processing Laboratory
RPM	revolutions per minute
TI	test instruction
TIMS	thermal ionization mass spectrometry
TRU	transuranic
UDS	undissolved solids

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1.0 Introduction

A sand filter is used to help maintain water clarity at the K West Basin where highly radioactive sludge is stored. Eventually that sand filter will require disposal. The radionuclide content of the solids trapped in the sand filter will affect the selection of the sand filter disposal pathway. The Pacific Northwest National Laboratory (PNNL) was contracted by the K Basin Operations & Plateau Remediation Project (KBO & PR), operations contractor CH2M Hill, to analyze selected radionuclides in the centrifuged solids collected from the backwash of the K West Basin sand filter. The radionuclide composition in the sand filter backwash solids will be used by CH2M Hill to determine if the sand filter media and retained sludge solids will be designated as transuranic (TRU) waste for disposal purposes or can be processed through less expensive means.

1.1 Background

Containerized radioactive sludge material is currently present in the K West Basin located at the Hanford Site in southeast Washington. The sludge genesis and composition has been previously reported (Fiskum et al. 2011; Shimskey et al. 2013; Fountain et al. 2013; Makenas et al. 1997; Johnson 2014). The sludge is stored under ~15 to 20 ft of water in the K West Basin, which contains ~1.2 million gallons of water. The water in the K West Basin provides shielding from radiological dose of the highly radioactive sludge as well as general environmental protection by minimizing sludge dispersibility. The water in the K West Basin is continuously conditioned by passing through a recirculation cooling system to remove decay heat, passing through filters to remove particulate material, and passing through ion exchange media to remove dissolved radioactive species. The filtration systems in the K West Basin have been previously described by Kurta (1998). One of the filters in the filtration system is called the sand filter and it specifically traps entrained particulates from the K West Basin water and thus lower turbidity and improve clarity. It consists of a 7.5 in. depth of 1.4 mm support sand and a 32 in. depth of 0.3 mm filter sand in a 78 in. diameter vessel.

The K East Basin (now demolished) sand filter backwash was previously characterized for particle size and morphology.[1] The K East Basin sand filter backwash solids particle size distribution was found to range from 0.46 to 10.2 microns with a median value of 1.8 to 2.5 microns. The chemical composition was assumed to consist of iron hydroxides, aluminum hydroxides, uranium oxides and silica (Schmidt 2006). Radionuclide and chemical composition of the KE North Loadout Pit (also referred to as the Sand Filter Backwash Pit) has been described in Shelor et al. (2004) and Mellinger et al. (2004).[2]

Minimal characterization has been conducted on the K West Basin sand filter since the transfer of the K East Basin and K West Basin sludge into large engineered containers. However, the general chemical compositions and physical properties found in the K East Basin backwash are expected to be similar to those in the K West Basin sand filter.

[1] "*Measurement of Particle Size Distributions in 105-KE Basin Water Samples*," CH2M-0403713, transmitted from WS Calloway (CH2M Hill) to RM Jochen (FH) December 2, 2004, CH2M HILL Hanford Group, Inc., Richland, WA—as reported by Schmidt (2006).

[2] Schmidt AJ and RB Baker. 2004. "Revised Design and Safety Basis Value for Physical Properties, Radionuclide, and Chemical Composition of Sludge in the KE Basin North Load Pit, PNNL Letter Report 46497-RPT03, Rev 1. Transmitted to WW Rutherford (Flour Hanford) and JP Sloughter (Numatec Hanford Company) by KL Silvers on February 24, 2004, via PNNL transmittal letter 46497-L05.

1.2 Scope

KBO & PR requested the radiochemical characterization of ^{238}Pu, $^{239+240}Pu$, ^{241}Am, ^{237}Np, ^{90}Sr, and gamma emitter concentrations in the sand filter backwash centrifuged solids to better understand the sand filter radionuclide loading and better characterize the sand filter for waste-disposal purposes. By understanding the gamma-emitting isotope concentration relationship to the alpha-emitting isotope concentration, a dose-to-Curie estimate of total actinide loading may be determined. These data can also be used to help determine if the spent sand filter media with entrained solids will be categorized as TRU waste.

The backwash of the skimmer system sand filter into the North Load-Out Pit (NLOP) was conducted in accordance with procedure, OP-07-065W, *Backwash Sand Filter at 105-KW* by KBO & PR staff on October 19, 2015. Immediately (i.e., within 1 minute) after the backwash, a sample tube was lowered to 24 in. above the floor of the NLOP, the sample pump started, and the sample bottle filled per work package 1K-15-03261/0, 105-KW *Sample Backwash Pit After Sand Filter Backwash*. KBO & PR shipped the sand filter backwash sample, KW-105 SFBW-001, to PNNL on October 20, 2015.

This report summarizes the sample-handling processes used to consolidate the backwash solids and the concentrations of gamma-emitting and alpha-emitting radionuclides in the backwash solids conducted at PNNL under Project 68122. In addition, limited physical property testing of the wet centrifuged solids (wt% wet and dry solids and density of centrifuged solids) was conducted and is reported herein.

1.3 Quality Assurance

Elements of KBC-33786, Rev. 2, *Quality Assurance Project Plan/Sampling and Analysis Plan for Sludge in the KW Engineered Containers* (Baker et al. 2009) were applied to the sample preparation and analysis process and included PNNL commitment to the following:

1. continuation of PNNL on the CH2M Hill Plateau Remediation Company (CHPRC) evaluated supplier list for laboratory services
2. sample receipt and preparation for analysis
3. sample analysis and analysis of applicable quality assurance samples
4. disposal of residues and sample waste
5. data verification prior to delivery to the CHPRC
6. preparation of a data package for delivery to the project/buyer's technical representative (BTR).

The quality assurance program requirements defined Baker et al. (2009) invoke the Hanford Analytical Services Quality Assurance Requirements Document (HASQARD), Rev. 3, Volumes 1 and 4. All work was conducted to meet these requirements.

A test instruction (TI), 68122-TI-001, Rev. 0, *Sandfilter Backwash Solids Consolidation*, was prepared to delineate how initial sample handling and solids consolidation were to be conducted. The draft TI underwent technical, Quality Engineer, and project management reviews at PNNL. The CHPRC BTR also reviewed and approved the TI before implementation. The analytical procedures used in sample analysis were also reviewed and approved for use by the BTR before implementation. All reviewer issues and comments were resolved before issuing the TI. Following testing, the completed TI underwent technical review and Quality Engineer surveillance (SR 68122-2015-001, *Surveillance of Completed Test Instruction 68122-TI-001, Sandfilter Backwash Solids Consolidation*). Similarly, the analytical data

reports underwent technical reviews and Quality Engineer surveillance (ASO-2016-010, *QE Review of ASO ASR 9916 for Project 68122: K Basin Sandfilter Backwash Samples*). All issues were resolved before finalizing the TI and analytical reports.

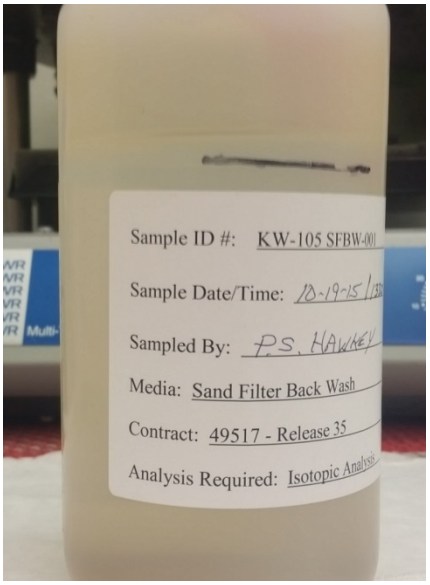
2.0 Sand Filter Backwash Sample Handling and Analysis

This section describes sample receipt, sample handling, solids consolidation, and analysis test procedures applied to the backwash filter sample, KW-105 SFBW-001.

2.1 Initial Receipt

One ~750 mL slurry sample, KW-105 SFBW-001, was received at PNNL's Radiochemical Processing Laboratory (RPL) on October 20, 2015 under chain of custody (see Appendix A, Attachment E). The sample was received in a 1 L polyethylene bottle. Flocculant solids were visible on the bottom of the container; however the fluid was lightly colored tan, indicative of a small particle size solid suspension. The sample contact dose rates were 1 mR/h gamma and 18 mR/h beta-gamma (closed and open window respectively) taken with an RO-20 portable ion chamber survey meter from the bottom of the container. Figure 2.1 provides images of the as-received container and the settled solids. The settled solids were a rust-brown color (consistent with iron oxides).

Upon receipt
(Note the light-tan aqueous phase)



After 24-h settling time
(Note the colorless aqueous phase)

Figure 2.1. As-Received Sample KW-105 SFBW-001 in a 1 L Polyethylene Bottle

2.2 Solids Consolidation

The entire sample was processed to consolidate all of the solids according to TI 68122-TI-001, *Sandfilter Backwash Solids Consolidation*. The completed TI is provided in Appendix A. One goal of this work was to determine the volume of centrifuged solids; therefore, the solids were to be consolidated into one or two (depending on total volume) volume-calibrated centrifuge tubes. Figure 2.2 illustrates the solids consolidation/processing strategy.

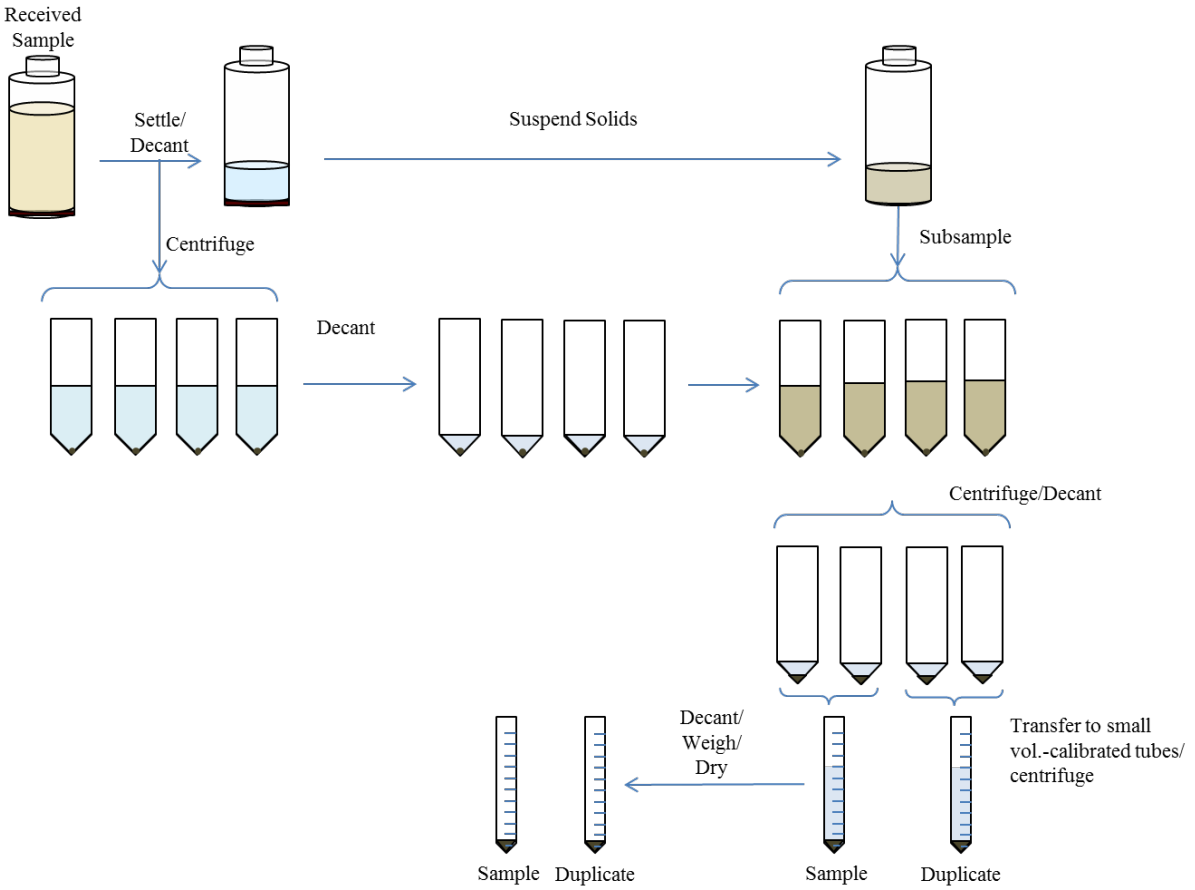


Figure 2.2. Solids Consolidation Strategy

The received sample container was weighed. The solids were allowed to settle overnight in the as-received container. After the 24-h settling period, the supernate was visually less turbid—i.e., the aqueous phase appeared colorless (see Figure 2.1). The aqueous phase was removed by siphoning, in 45 mL increments, using a polystyrene serological pipet, into each of four 50 mL polypropylene centrifuge tubes with plug-seal caps (Fisherbrand). Water was removed well above the settled solids level so as not to disturb the settled solids layer. The centrifuge tubes with water were then centrifuged at 1500 RPM for 20 minutes. Any solids collected at the bottom of a centrifuge tube remained undisturbed while decanting the aqueous phase from the centrifuge tube into a separate bottle (decantate). This process was repeated six times until the bulk of the water phase of sample KW-105 SFBW-001 had been processed. In all cases, the decantate was colorless and clear.

Next, the remaining sample in the as-received container (settled solids and water cover layer) was agitated by swirling to suspend the solids, which were then transferred into the four centrifuge tubes used to clarify the water. Additional aqueous phase sample water (from the decantate) was used to achieve a quantitative transfer from the sample bottle into the four centrifuge tubes. Efforts were made to distribute the solids equally between the four centrifuge tubes. Figure 2.3 shows the four centrifuge tubes with centrifuged sludge sample material (container identification (ID) numbers left to right 68122-TI-001-A through 68122-TI-001-D) and the clarified decanted water collected in a 1 L polycarbonate bottle (container ID 68122-TI-001-E). The transfer pipet used to transfer the water and then the slurried solids was lightly coated with yellow material, indicating that a small amount of solids adhered to the plastic (see Figure 2.4). This material was not recoverable. The emptied original sample container was weighed and the net sample mass received was calculated.

Figure 2.3. Centrifuge Tubes with Consolidated Sludge and Clarified Decanted Water

Figure 2.4. Transfer Pipet Used to Transfer the Solids Slurry

The contact dose rates for each of the four samples were measured with an RO-20 portable ion chamber survey meter and are provided in Table 2.1 under the "Initial" subheading. These dose rates are qualitative and were collected to obtain a sense of how well the radioactivity was distributed across the four containers. In general, the dose rates were similar, except for the last container (i.e., 68122-TI-001-D) which was appreciably lower.

Table 2.1. Contact Dose Rates of Collected Solids, mR/hour

Centrifuge Cone ID>>	68122-TI-001-A	68122-TI-001-B	68122-TI-001-C	68122-TI-001-D
		Initial		
Open window	5	5	5	2
Closed window	3	3.5	3.5	1
		Final, after Re-Split		
Open window	5.5	NA	NA	5
Closed window	<0.5	NA	NA	<0.5

NA = not applicable

Sufficient sample volume existed to create two samples (i.e., a primary and a duplicate sample) to support the solids analysis. Therefore, it was necessary to evenly split the sample activity (determined by dose rate) between the primary and duplicate samples. To this end, subsamples 68122-TI-001-A and 68122-TI-001-D, which had different dose rates, were combined and re-split; subsamples 68122-TI-001-B and 68122-TI-001-C appeared equivalent and were not further re-processed. The dose rates for 68122-TI-001-A and 68122-TI-001-D achieved after the re-splitting are shown in Table 2.1 under the subheading "Final, after Re-Split." The final dose rates indicate the subsamples had a higher combined dose rate than obtained with the initial measurement, which clearly cannot be the case. However, these measurements are not quantitative and significant changes can result by small changes in sample position in front of the detector, room background dose rate, and operator differences. Based on the near-equivalence of the final dose rate measurements, the re-combined and re-split subsamples 68122-TI-001-A and 68122-TI-001-D were considered equivalent to each other.

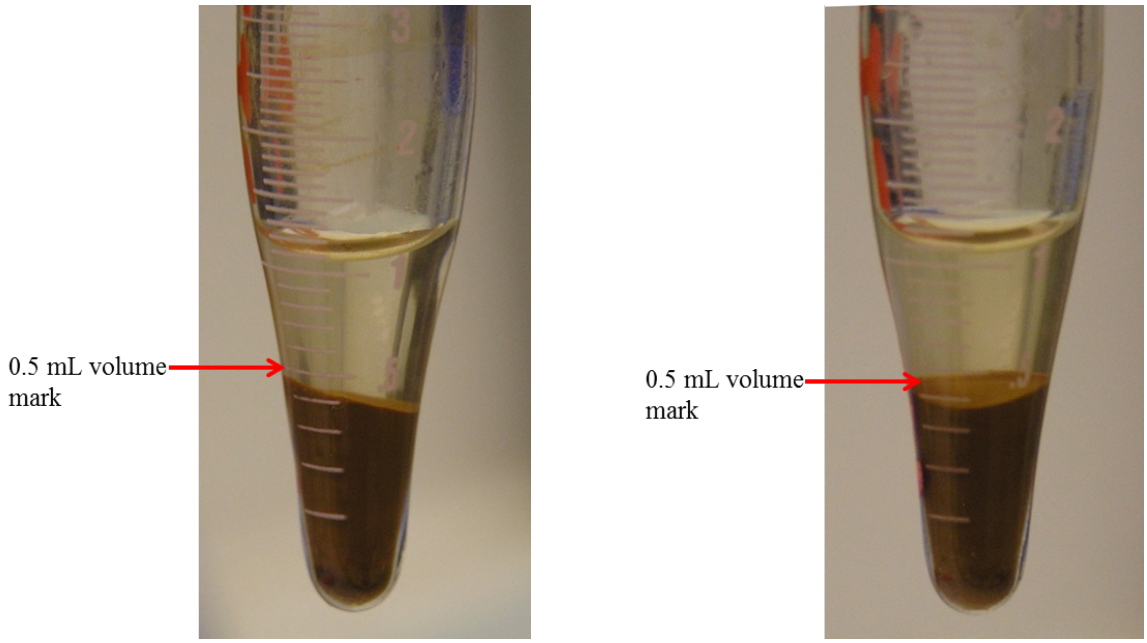
The solids from 68122-TI-001-A and 68122-TI-001-B were slurried, transferred, and combined into a tared, 10-mL glass, graduated, centrifuge cone (i.e., heavy-duty with screw cap closure, Kimble-Chase part number 45200-10) labeled 68122-TI-001-G. The solids from 68122-TI-001-C and 68122-TI-001-D were slurried, transferred, and combined into another tared, 10 mL glass, graduated, centrifuge cone labeled 68122-TI-001-F. These centrifuge tubes allowed for higher fidelity in volume measurement readings (to the nearest 0.1 mL) and could be placed in an oven to dry the solids for determination of dry solids mass. The consolidated solids were centrifuged at 1500 RPM for 30 minutes. Final centrifuged solids volumes were estimated to be 0.4 mL in each tube. Because the solids level was slanted and not even, it was difficult to discern exact solids volume. Images of the collected solids are shown in Figure 2.5; for a reference point, an arrow is positioned at the 0.5 mL volume graduation mark. The solids were dark brown/orange in color, consistent with iron oxides/hydroxides.

The overburden of water was removed until it was at the 0.5 mL volume point in each centrifuge tube. Each centrifuge tube and its contents were weighed and the total slurry and centrifuged solids mass was calculated according to Equation 2.1, where the water density was assumed to be 1 g/mL.

$$M_{CS}=G - T - W \qquad 2.1$$

where M_{CS} = Mass of wet centrifuged solids
G = Gross centrifuge cone mass
T = Centrifuge cone mass
W = Water mass (and volume above centrifuged solids)

The centrifuged solids density was calculated by dividing the solids mass by the solids volume.



68122-TI-001-F
(combined 68122-TI-001-C and 68122-TI-001-D)
ASR Sample ID 16-0032

68122-TI-001-G
(combined 68122-TI-001-A and 68122-TI-001-B)
ASR Sample ID 16-0032 (dup)

Figure 2.5. Centrifuged Solids KW-105 SFBW-001 Sample and Duplicate

2.3 Solids Digestion and Analysis Methods

The solids samples were submitted to the Analytical Support Operations (ASO) laboratory for drying and mass measurement, acid digestion, and radiochemical analysis under Analytical Service Request (ASR) 9916. Figure 2.5 and Appendix B cross reference sample IDs and ASR sample IDs. The ASR was submitted with special instructions (see Appendix B) that delineated sample handling and analysis processes and processes for handling and analysis of any observed undissolved solids (UDS).

The centrifuged solids were dried in the centrifuge cone at 105°C to constant mass. The net sample mass was calculated by subtracting the gross mass from the centrifuge cone tare mass. The solids were acid-digested using a combination of HNO_3 and HCl. The UDS were removed from the acid digestate using centrifugation and washed with dilute HNO_3 before drying to constant mass. The sample acid digestate and UDS wash were combined and brought to a known volume from which aliquots were collected for various radiochemical processing and isotopic analyses. The procedures applied to the sample and sample duplicate are provided in Table 2.2.

Table 2.2. ASO Analytical Procedures

Analyte	Separation	Mounting	Analysis / Counting
Sample and UDS dry mass	NA	NA	RPG-CMC-503, Rev. 0, *Determination of Physical Properties of Solutions, Sludges, Slurries, and Solids*
Acid digestion	RPG-CMC-129, Rev. 0, *HNO_3-HCl Acid Extraction of Solids Using a Dry-Block Heater*	NA	NA
GEA	NA	NA	RPG-CMC-450, Rev. 2 *Gamma Energy Analysis (GEA) and Low-Energy Photon Spectrometry (LEPS)*
^{238}Pu and $^{239+240}$Pu/AEA	RPG-CMC-4017, Rev. 0, *Analysis of Environmental Water Samples for Actinides and Strontium-90*	RPG-CMC-496, Rev.1, *Coprecipitation Mounting of Actinides for Alpha Spectroscopy*	RPG-CMC-422, Rev. 2 *Solutions Analysis: Alpha Spectrometry*
^{241}Am/AEA	RPG-CMC-4017, Rev. 0, *Analysis of Environmental Water Samples for Actinides and Strontium-90*	RPG-CMC-496, Rev. 1, *Coprecipitation Mounting of Actinides for Alpha Spectroscopy*	RPG-CMC-422, Rev. 2 *Solutions Analysis: Alpha Spectrometry*
^{237}Np/AEA	RPG-CMC-4017, Rev. 0, *Analysis of Environmental Water Samples for Actinides and Strontium-90*	RPG-CMC-496, Rev. 1, *Coprecipitation Mounting of Actinides for Alpha Spectroscopy*	RPG-CMC-422, Rev. 2 *Solutions Analysis: Alpha Spectrometry*
^{90}Sr	RPG-CMC-476, Rev. 0, *Strontium Separation using Eichrom Strontium Resin*	NA	RPG-CMC-474, Rev. 1, *Measurement of Alpha and Beta Activity by Liquid Scintillation Spectrometry*
Pu isotopic	RPG-CMC-455, Rev. 0, Separation of Uranium and Plutonium for Isotopic Analysis by Mass Spectroscopy	NA	RPL-TIMS-001, Rev. 0, *Thermal Ionization Mass Spectrometry (TIMS)*

Renormalization of the thermal ionization mass spectrometry (TIMS) analysis results was required. Despite care in the separation, a very small carry-over and/or contamination of U into the Pu fraction interfered with the analysis of ^{238}Pu (from the ^{238}U isobar; also see Appendix B). Therefore, the ^{238}Pu analysis by AEA provides more accurate results for this radioisotope. The correct contribution of ^{238}Pu with respect to ^{239}Pu was determined by the following steps:

- AEA provided relative amounts of ^{238}Pu and combined $^{239+240}$Pu in terms of activities (μCi) per gram of sludge. In AEA, ^{238}U interference does not contribute to the ^{238}Pu or to the $^{239+240}$Pu AEA peaks.
- The relative contributions of ^{239}Pu and ^{240}Pu to the $^{239+240}$Pu AEA peak were apportioned based on the ^{239}Pu and ^{240}Pu TIMS values and the specific activities of the individual ^{239}Pu and ^{240}Pu isotopes. This provided activities of the individual ^{239}Pu and ^{240}Pu isotopes per gram of sample.
- At that point, the activities of the ^{238}Pu and the ^{239}Pu per gram of sample were known.
- The atom percent (At%) of ^{238}Pu, with respect to the At% of ^{239}Pu reported by TIMS was calculated using Equation 2.2:

$$\text{At\% } {}^{238}\text{Pu} = \frac{\mu\text{Ci } {}^{238}\text{Pu/g sludge}}{\mu\text{Ci } {}^{239}\text{Pu/g sludge}} \times \frac{\text{Ci } {}^{239}\text{Pu/g } {}^{239}\text{Pu}}{\text{Ci } {}^{238}\text{Pu/g } {}^{238}\text{Pu}} \times \frac{\text{g } {}^{239}\text{Pu/mole } {}^{239}\text{Pu}}{\text{g } {}^{238}\text{Pu/mole } {}^{238}\text{Pu}} \times \text{At\% } {}^{239}\text{Pu} \qquad 2.2$$

in which the first term includes the specific activities of ^{238}Pu and ^{239}Pu in the sample, the second term includes the specific activities of ^{238}Pu and ^{239}Pu with respect to their isotopically pure element (from National Bureau of Standards [NBS] values), the third term includes the atomic weights of the ^{238}Pu and ^{239}Pu isotopes (from NBS values), and the fourth term is the At% of ^{239}Pu found by TIMS.

- The At% ^{238}Pu, with respect to the At% ^{239}Pu (TIMS) as determined by this method, was less than the ^{238}Pu value reported by TIMS. Therefore, the individual Pu isotope percentages by the above calculation for ^{238}Pu and the other Pu isotope percentages by TIMS, are summed and renormalized. This calculation approach provided the correct At% for all Pu isotopes.

3.0 Results

This section presents the results of the physical property and radionuclide analyses of the consolidated solids collected from backwash filter sample KW-105 SFBW-001.

3.1 Physical Properties

The calculated total slurry mass received was 764 g. The centrifuged solids mass, volume, and density and the dry centrifuged solids mass for the primary and duplicate sample are shown in Table 3.1. The centrifuged water and sludge volumes were read against a scale with 0.1 mL volume increments. The water left above the solids was brought to the 0.5 mL mark, minimizing the need to interpolate between 0.1 mL volume graduations. However, volume measures can only be reported to two significant figures at best. In both the sample and duplicate sample cases, the centrifuged solids densities are not much different than the density of water.

Table 3.1. Physical Properties of KW-105 SFBW-001 Centrifuged Solids

Sample ID	68122-TI-001-F 16-0032	68122-TI-001-G 16-0032 (dup)
Centrifuge cone tare (g)	20.6859	20.6253
Centrifuge cone gross mass with wet solids (g)	21.2217	21.1095
Centrifuged solids volume (mL)	0.40	0.40
Total slurry volume (mL)	0.50	0.50
Centrifuged solids mass (g)	0.44	0.38
Centrifuged solids density (g/mL)	1.1	0.96
Centrifuge cone gross mass with dry solids (g)	20.7404	20.6821
Dry centrifuged solids mass (g)	0.0545	0.0568
Mass loss on drying (%)	87	85

The wt% wet centrifuged solids in the as-received slurry sample was calculated from combining the wet centrifuged solids masses of the primary and duplicate samples and dividing by the total received sample mass. The wt% dry centrifuged solids was calculated by combining the total dry solids masses of the primary and duplicate samples and dividing by the total received sample mass. Results are summarized in Table 3.2.

Table 3.2. Weight Percent Solids in Sample KW-105 SFBW-001 Slurry

Total slurry mass (g)	764.2
Combined wet centrifuged solids mass (g)	0.82
Wet centrifuged solids (wt%)	0.11
Combined dry centrifuged solids mass (g)	0.1113
Dry centrifuged solids[(a)] (wt%)	0.0146

(a) The dry solids mass does not consider mass contribution from dissolved solids.

3.2 Acid Digestion

The acid digestion procedure appeared to dissolve all solids. Upon adding water to dilute the acid, solids precipitated. As shown in Figure 3.1 the consolidated solids were flocculant, similar to colloidal silica, and colored dark gray. The aqueous phase is intensely yellow, characteristic of dissolved iron in HCl.

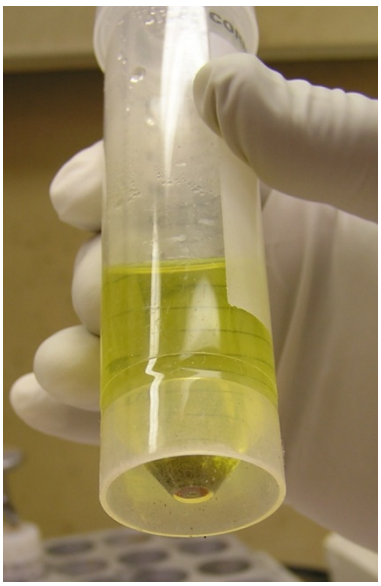
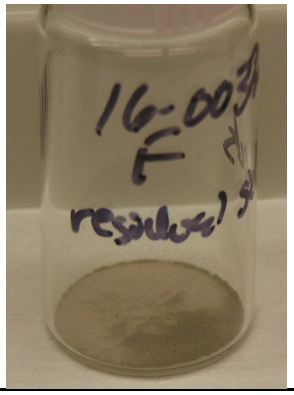
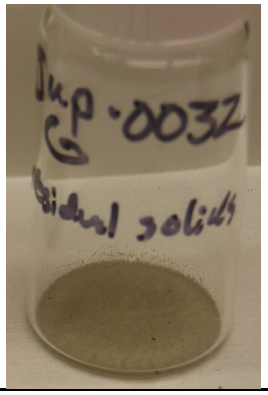


Figure 3.1. Post-Precipitated Solids from Sample 68122-TI-001-F

The solids were separated from the aqueous phase using centrifugation and decanting. The solids were washed several times with 0.5 M HNO_3 and dried. The dried solids masses and images are provided in Table 3.3. The average UDS represents ~6.5% of the total dry solids.

Table 3.3. Dry Undissolved Solids

Parameter	68122-TI-001-F 16-0032	68122-TI-001-G 16-0032 (dup)
Dry UDS mass (g)	0.0039	0.0039
UDS remaining (dry solids mass basis) (%)	6.7	6.4
Image		

3.3 Radionuclide Analysis

All radionuclide analyses were conducted on the acid digestate. Gamma energy analysis (GEA) was also performed on the UDS samples. Table 3.4 and Table 3.5 provide the results summaries for the measured radionuclides in the acid digestate reported on a dry mass basis (see the ASO reports in Appendix B for full discussion) and wet centrifuged solids mass basis, respectively.

Table 3.4. Radionuclide Summary Results for the Acid Digestate, Dry Centrifuged Solids Mass Basis

	68122-TI-001-F 16-0032	68122-TI-001-G 16-0032 (dup)	Average	
Analyte	μCi/g Dry Mass			RPD
^{60}Co (GEA)	1.08E-1[(a)]	9.91E-2[(a)]	1.04E-1[(a)]	8.6[(a)]
^{137}Cs (GEA)	1.02E+2	9.49E+1	9.85E+1	7.2
^{152}Eu (GEA)	3.88E-2	3.13E-2	3.51E-2	**21**[(b)]
^{154}Eu (GEA)	1.54E+0	1.46E+0	1.50E+0	5.3
^{155}Eu(GEA)	2.28E-1	2.55E-1	2.42E-1	11
^{238}Pu (AEA)	2.63E+01	2.61E+1	2.62E+1	0.8
$^{239+240}$Pu (AEA)	1.99E+2	2.00E+2	2.00E+2	0.4
^{241}Am (GEA)	9.64E+1	1.06E+2	1.01E+2	9.5
^{241}Am (AEA)	9.39E+1	8.69E+1	9.04E+1	7.7
^{237}Np (AEA)	1.80E-3	2.09E-3	1.94E-3	15
^{90}Sr (LSC)	1.88E+1	1.88E+1	1.88E+1	0.5

The GEA analyte reference date is November 24, 2015; the alpha energy analysis (AEA) and ^{90}Sr liquid scintillation counting (LSC) reference date is December 15-30, 2015.
ASR 9916, ASO sample 16-0032 and 16-0032 duplicate; see Appendix B.
Bolded values indicate the relative percent difference (RPD) exceeded acceptance criteria of 20%.
(a) An additional 7.0 and 8.5% of the ^{60}Co remained with the UDS for the sample and duplicate, respectively, see Table 3.7.
(b) The RPD passed the mean difference test; see Appendix B.

Table 3.5. Radionuclide Summary Results for the Acid Digestate, Wet Centrifuged Solids Mass Basis

	68122-TI-001-F 16-0032	68122-TI-001-G 16-0032 (dup)	Average	
Analyte	μCi/g Wet Centrifuged Solids			RPD
^{60}Co (GEA)	1.35E-2[(a)]	1.47E-2[(a)]	1.41E-2[(a)]	8.1[(a)]
^{137}Cs (GEA)	1.28E+1	1.40E+1	1.34E+1	9.5
^{152}Eu (GEA)	4.85E-3	4.63E-3	4.74E-3	4.7
^{154}Eu (GEA)	1.93E-1	2.16E-1	2.04E-1	11
^{155}Eu (GEA)	2.85E-2	3.77E-2	3.31E-2	**28**[(b)]
^{238}Pu (AEA)	3.29E+0	3.86E+0	3.57E+0	16
$^{239+240}$Pu (AEA)	2.49E+1	2.96E+1	2.73E+1	17
^{241}Am (GEA)	1.21E+1	1.57E+1	1.39E+1	**26**
^{241}Am (AEA)	1.17E+1	1.28E+1	1.23E+1	9.0
^{237}Np (AEA)	2.25E-4	3.08E-4	2.67E-4	**31**
^{90}Sr (LSC)	2.35E+0	2.79E+0	2.57E+0	17

The GEA analyte reference date is November 24, 2015; the AEA and ^{90}Sr reference date is December 15-30, 2015.
Bolded values indicate the RPD exceeded acceptance criteria of 20%.
(a) An additional 7.0 and 8.5 % of the ^{60}Co activity remained with the UDS for the sample and duplicate, respectively, see Table 3.7.
(b) The RPD passed the mean difference test; see Appendix B.

The sample averages and relative percent deviations (RPDs) are also provided in Table 3.4 and Table 3.5. RPDs that exceed 20% are in bold font to indicate where the data quality objective (RPD ≤20%) was exceeded. The dry solids net mass uncertainty was about 5%; the wet centrifuged solids mass uncertainty was 16 to 18%. The higher RPDs associated with the results on a wet centrifuged solids mass basis reflect the higher uncertainties associated with the centrifuged solids mass measurements.
The concentrations of gamma-emitting radionuclides in the UDS are provided in Table 3.6 along with the averages and RPDs. Only the ^{241}Am result in the UDS exceeded 20% RPD, indicating good overall precision.

Table 3.6. Radionuclide Summary Results for the UDS, Dry Solids Mass Basis

	68122-TI-001-F UDS 16-0032	68122-TI-001-G UDS 16-0032 (dup)	Average UDS	
Analyte	μCi/g dry mass			RPD
^{60}Co (GEA)	1.13E-1	1.34E-1	1.24E-1	17
^{137}Cs (GEA)	1.01E+1	1.07E+1	1.04E+1	5.8
^{152}Eu (GEA)	2.31E-3	2.06E-3	2.19E-3	11
^{154}Eu (GEA)	3.26E-2	3.41E-2	3.34E-2	4.5
^{155}Eu (GEA)	4.18E-3	5.03E-3	4.61E-3	18
^{241}Am (GEA)	1.25E+0	1.78E+0	1.52E+0	**35**

The GEA analyte reference date is November 24, 2015.
ASR 9916, ASO sample 16-0032 and 16-0032 duplicate; see Appendix B.
Bolded values indicate the RPD exceeded acceptance criteria of 20%.

The analyte activity fractionation to the UDS residue was evaluated for the measured gamma-emitters. The activity was corrected to total activity, as opposed to being evaluated on a per gram basis. Table 3.7 shows the total microCuries analyte in the acid digestate and UDS as well as the fraction remaining in the UDS. The total activity is heavily weighted to the acid digestate. Except for ^{60}Co present at ~8% in the UDS, analytes were present at much less than 1% in the UDS. It is assumed that the Sr, Pu, and Np are similarly fractionated to the acid digestate (chemistry following that of Am and Eu), and that the results shown in Table 3.4 accurately reflect the ^{90}Sr, Pu, and Np radionuclide concentrations in the initial centrifuged solids.

Table 3.7. Relative Radionuclide Content in the Acid Digestate and UDS

	68122-TI-001-F 16-0032			68122-TI-001-G 16-0032 (dup)		
Analyte	μCi in acid digest[(a)]	μCi in UDS[(b)]	Fraction in UDS	μCi in acid digest[(a)]	μCi in UDS[(b)]	Fraction in UDS
^{60}Co	5.89E-3	4.41E-4	7.0%	5.63E-3	5.23E-4	8.5%
^{137}Cs	5.56E+0	3.94E-2	0.70%	5.39E+0	4.17E-2	0.77%
^{152}Eu	2.11E-3	9.01E-6	0.42%	1.78E-3	8.03E-6	0.45%
^{154}Eu	8.39E-2	1.27E-4	0.15%	8.29E-2	1.33E-4	0.16%
^{155}Eu	1.24E-2	1.63E-5	0.13%	1.45E-2	1.96E-5	0.14%
^{241}Am (GEA)	5.25E+0	4.88E-3	0.09%	6.02E+0	6.94E-3	0.12%

(a) The radionuclide concentration (Table 3.4) was multiplied by the dry centrifuged solids sample mass (Table 3.1).
(b) The radionuclide concentration (Table 3.6) was multiplied by the UDS mass (Table 3.3).

Table 3.8 provides the normalized Pu isotopic mass ratio (see Section 2.3 for normalization discussion). The Pu isotopic fractions are similar to the fractions measured in the sludge samples collected from SCS-CON-220 (Fiskum et al. 2011) after decay-correction for ^{241}Pu.

Table 3.8. Plutonium Isotopic Analysis by TIMS and AEA for the Acid Digestate

Analyte	68122-TI-001-F 16-0032	68122-TI-001-G 16-0032 (dup)	Average	RPD
Atom Percent (Normalized)				
^{238}Pu (AEA)	0.0642	0.0635	0.0638	1.1
^{239}Pu	86.38	86.38	86.38	0.002
^{240}Pu	12.93	12.93	12.93	0.04
^{241}Pu	0.3904	0.3885	0.3895	0.50
^{242}Pu	0.2335	0.2326	0.2331	0.38
Weight Percent (Normalized)				
^{238}Pu (AEA)	0.0639	0.0632	0.0635	1.1
^{239}Pu	86.33	86.33	86.33	0.002
^{240}Pu	12.98	12.98	12.98	0.04
^{241}Pu	0.3935	0.3915	0.3925	0.50
^{242}Pu	0.2363	0.2354	0.2359	0.38

Reference date is January 27, 2016.

3.4 Radioisotopic Ratio Evaluation

The Pu is expected to be trapped as a fine particulate in the sand filter while the much more soluble Cs and Sr are expected to pass through the sand filter. Additionally, as basin water continues to be passed through the sand filter, Cs and Sr will be washed from the trapped particulates, further depleting their inventory in the particulates. The isotopic ratios of $^{239+240}$Pu to ^{90}Sr and $^{239+240}$Pu to ^{137}Cs were evaluated for the sand filter backwash sample in terms of µCi/g dry solids. The sample isotopic ratios were compared to the ratios from sludge in the large engineered containers (-500 µm sieve fraction where possible) as well as the suspended solids collected after a 30-minute settling time (i.e., SCS-CON-220, SCS-CON-240, SCS-CON-250, and SCS-CON-260) that had been decay-corrected to November 24, 2015 (sample KW-105 SFBW-001 reference date). The backwash sand filter $^{239+240}$Pu/^{90}Sr and $^{239+240}$Pu/^{137}Cs isotopic ratios were uniquely higher than all other K West Basin sludge samples as shown in Table 3.9. This indicates that the radiological content of the sand filter backwash solids is not well represented by the isotopic mix ratio of the sludge itself. Relative to the ^{137}Cs content, the Pu is 4 to 20× higher in the sand filter solids than in the K West Basin sludge source material. Relative to the ^{90}Sr content, the Pu is 10 to 100× higher in the sand filter solids than in the K West Basin sludge source material. The ratio differences indicate a relative enhancement of the Pu concentration in the sand filter solids relative to the ^{137}Cs and ^{90}Sr sludge concentrations currently in the K West Basin. A dose-to-curie radioisotope evaluation of the sand filter waste form may need to consider this dissimilarity.

Table 3.9. Isotopic Ratios in Backwash Sand Filter and Containerized Sludge

Description	Sample ID	$^{239+240}Pu/^{90}Sr$[a,d]	$^{239+240}Pu/^{137}Cs$[b,d]	Analytical Data Source Reference
Backwash sand filter sample	KW-105SFBW-001	10.6	2.0	Data in this report
KW210 container composite, -500 µm sieve fraction	Average of TI008-SA and TI008-SB	1.21	0.27	(Fountain et al. 2013)
KW220 container composite, -500 µm sieve fraction	TI009-SB-s	^{90}Sr not analyzed	0.23	(Fiskum et al. 2011)
KW230 Core A2	TI023-1C	0.14	0.41	(Shimskey et al. 2013)
KW230 Core A3	TI024-2C	0.10	0.13	(Shimskey et al. 2013)
KW230 Core B2	TI024-3C	0.14	0.38	(Shimskey et al. 2013)
KW230 Core B4	TI024-4C	0.12	0.22	(Shimskey et al. 2013)
KW240 container composite, -500 µm sieve fraction	TI010-SD	0.11[c]	0.12	(Fiskum et al. 2011)
KW250 container composite, -500 µm sieve fraction	TI011-SB	0.12[c]	0.10	(Fiskum et al. 2011)
KW260 container composite, -500 µm sieve fraction	TI012-SB	0.14[c]	0.16	(Fiskum et al. 2011)
KW240 suspended solids in settling test	SSK240-S	^{90}Sr not analyzed	0.37	(Fiskum et al. 2010)
KW250 suspended solids in settling test	SSK250-S	^{90}Sr not analyzed	0.25	(Fiskum et al. 2010)
KW260 suspended solids in settling test	SSK260-S	^{90}Sr not analyzed	0.46	(Fiskum et al. 2010)
KW220 suspended solids in settling test	SSK220A3-S	^{90}Sr not analyzed	0.47	(Fiskum et al. 2010)
KE NLOP suspended solids from top sample	KENLOP-DS2	1.26	0.61	Shelor et al. 2004
KE NLOP top 1/3 of core sample	KENLOP-AB2	2.77	0.48	Shelor et al. 2004

(a) µCi $^{239+240}Pu$ /g dry solids divided by µCi ^{90}Sr /g dry solids.
(b) µCi $^{239+240}Pu$ /g dry solids divided by µCi ^{137}Cs /g dry solids.
(c) ^{90}Sr was not analyzed in the -500 µm sieve fraction; the given ratio was calculated from the average A and B reconstituted core samples.
(d) Reference date is November 24, 2015.

Per Fiskum et al. (2011), the ^{90}Sr and ^{137}Cs concentrations were nearly equivalent in the K Basin water (3.36E-4 and 5.83E-4 µCi/g, respectively). The Cs is expected to be soluble in water and Sr will have some solubility in water. The higher Pu fractionation in the sand filter solids relative to Cs and Sr is expected based on solubility and functionality of the filter. However the extent of the divergence of the Pu/Cs and Pu/Sr ratios relative to other sludge types was higher than expected.

4.0 Conclusions

The sand filter backwash slurry sample, KW-105 SFBW-001, was successfully processed and analyzed for physical properties and radioisotopic concentrations. These properties will enable KBO & PR staff at CH2MHill to better evaluate the radionuclide composition of the sand filter located at the K West Basin.

- Sample receipt, handling, and analyses were executed consistent with the planning and no issues were encountered.
- The backwash solids physical characteristics were consistent with the majority composition of iron oxides/hydroxides: the color was orange/brown, the wet centrifuged solids density was close to 1 g/mL, and the mass loss on drying was about 86 wt%. However, mineral phase characterization was not conducted.
- Most of the radionuclide activity dissolved in the acid digestion, indicating that the acid digestate values were well-representative of the sand filter samples. The radionuclide activity fractions remaining in the UDS portion of the sample were 7.7% ^{60}Co and <1% ^{241}Am, $^{154/155}$Eu, and ^{137}Cs.
- The duplicate subsamples analytes ^{60}Co, ^{137}Cs, ^{152}Eu, ^{154}Eu, ^{155}Eu, ^{238}Pu, $^{239+240}$Pu, ^{237}Np, ^{241}Am, and ^{90}Sr were quantified on a dry mass basis and the primary and duplicate samples met quality control precision acceptance criteria of ≤20%.
- The Pu isotopic ratios, measured with TIMS and AEA, were consistent with sludge Pu isotopic ratios in SCS-CON-210 and SCS-CON-220 core samples.
- The relative fraction of Pu, when compared to ^{90}Sr and ^{137}Cs, was higher in the backwash sand filter sample than in the K West Basin sludge, indicating a physical enrichment of Pu in the sand filter solids.

5.0 References

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Appendix A

Completed Test Instruction 68122-TI-001, *Sandfilter Backwash Solids Consolidation*

Appendix A

Completed Test Instruction 68122-TI-001, *Sandfilter Backwash Solids Consolidation*

Appendix A provides the completed test instruction (TI) used to describe solids the consolidation approach, give step by step instructions, and record data and annotations. Attached to the TI are underpinning records of balance calibration and daily performance checks, pictorial summary, sample chain of custody provided by CH2M Hill, technical review, and data calculations.

The technical review is conducted by staff other than the analyst performing the work and who is knowledgeable of the area being reviewed. The technical review elements include data evaluation, method and quality control (QC) sample performance against requirements, compliance with technical and QC requirements, transcription accuracy into data analysis tables, calculation accuracy, consistency and reasonableness of the data.

Also included in Appendix A is the Quality Engineer's surveillance report, SR-68122-2015-005 that evaluates completeness, method implementation and QC sample performance against data quality requirements, compliance with technical and QC requirements, transcription accuracy, calculation accuracy, consistency and reasonableness of data, verification of technical review issue resolution, and as appropriate tracking of corrective actions and completion.

No Occurrence or Deficiency Reports were issued during the processing activity.

Appendix A Table of Contents

TEST INSTRUCTION

TITLE: Sandfilter Backwash Solids Consolidation

Unique Numerical Designation: 68122-TI-001

Revision Number: 0

Effective Date: Upon final signature

Controlling Procedure No: RPL-OP-001, current revision, *Routine Research Operations*

Test Instruction Approvals

Author: Sandra k Fiskum — SK Fiskum — Date: 8/24/15

Technical Reviewer: RW Shimskey — Date: 8/25/15

Project Manager: sandra k. Fiskum — SK Fiskum — Date: 8/24/15

CSM Approval: Chuck Soderquist — CZ Soderquist — Date: 8.24.15

Project Quality Engineer: Deborah Coffey — DS Coffey — Date: 8/24/15

Buyer's Technical Representative (BTR): GM Davis — Date: 8/27/15

Test Instruction Completion Review

Technical Reviewer: Sandra k. Fiskum — Date: 10/28/15

Printed Name: SANDRA Fiskum — Date: 10/28/15

Project Quality Engineer: Deborah Coffey — DS Coffey — Date: 10.29/2015

Project Specific Documents

- Baker RB, JL Westcott, TL Welsh, JA Pottmeyer, and AJ Schmidt. 2009. *Quality Assurance Project Plan/Sampling and Analysis Plan for Sludge in the KW Engineered Containers*. KBC-33786, Rev. 2, CH2M Hill Plateau Remediation, Company, Richland, Washington.
- Westcott JL, BJ Makenas, TL Welsh, JA Pottmeyer, and AJ Schmidt. 2009. Data Quality Objectives for Sampling and Analysis of K Basin Sludge (DQO), HNF-36985, Rev. 3, CH2M Hill Plateau Remediation, Company, Richland, Washington.
- Letter 52578-2015-L03 from SK Fiskum to JO Honeyman, *Cost Estimate for Analysis of the Sandfilter Backwash Solids*, May 27, 2015.
- CH2M Hill Plateau Remediation, Contract 49517, Release 35, Statement of Work, *Isotopic Analysis from PNNL* (July 7, 2015), Amendment 1.

Purpose

Provide characterization data to allow the client to characterize the sand filter media for disposal. The sand filter is located in the K West Basin.

Applicability

This test instruction (TI) applies to Radiochemical Processing Laboratory (RPL) staff performing work with the K-Basin sandfilter backwash sample obtained from the K West basin sandfilter system (Project Number 68122). It provides direction to staff on required material manipulations and data recording in order to meet the project technical and quality requirements.

Work with the bulk samples is anticipated to be performed in radiological fume hoods. However, if the sample dose rate is too high for hand manipulations (including use of extension tools), parts of this sample preparation activity will be moved to, and conducted in, the Shielded Facility Operations (SFO) Shielded Analytical Laboratory (SAL, hot cells). All work will be conducted under the direction of a Cognizant Scientist.

This TI does not address safety procedures; the safe-handling constraints are implemented through the governing procedure RPL-OP-001, *Routine Research Operations* for in-cell (if needed) and fume hood manipulations. Applicable SFO procedures for transfers in and out of the hot cells may be applied as needed.

Scope

The scope of this TI is to receive/unpackage the sample, consolidate the suspended and settled solids in the as-received 750-mL aqueous sample into two equivalent aliquots, and determine the centrifuged solids volume and mass.

The centrifuged solids will then be submitted to the Analytical Support Organization (ASO) for determination of dry sample mass, acid digestion, and radiochemical analysis of Cs-137 and other gamma emitters by gamma energy analysis, Pu-238, Pu-239+240, Np-237, and Am-241 by alpha energy analysis, and Sr-90 by beta analysis. An additional acid-digested aliquot will be purified for Pu isotopic analysis and analyzed thermal ionization mass spectrometry.

Quality Assurance (QA)

Elements of the *Quality Assurance Project Plan /Sampling and Analysis Plan for Sludge in the KW Engineered Containers*, KBC-33786, Rev. 2 December 2009 that apply to this sample preparation process include the following. Item (2) specifically applies to this TI; others apply to analysis, data reviews, and reporting. Pacific Northwest National Laboratory (PNNL) will perform the following:

1) Continue to be maintained on the CHPRC evaluated supplier list for laboratory services.
2) Receive samples at the laboratory and prepare the samples for analysis.
3) Analyze samples of sludge and associated applicable quality assurance samples.
4) Dispose of residues and sample waste.
5) Verify data prior to delivery to the customer.
6) Prepare data packages for delivery to the project/ Buyer's Technical Representative (BTR).

This document outlines PNNL's approach for handling and analyzing the backwash filter sample. The quality assurance program requirements defined in the Quality Assurance Project Plan (QAPjP)/Sampling and Analysis Plan (SAP) invoke Hanford Analytical Services Quality Assurance Requirements Document (HASQARD), Rev. 3, Volumes 1 and 4, and balance controls. The applicable quality control (QC) requirements that will be implemented for this work as defined in this test instruction are summarized in Table 1.

Table 1. QC Requirement and Implementation

QC Requirement	Implementation
Controlling sample identification	K Basin Operations and Central Plateau Remediation Project (KBO&PR) will apply a unique code to the sample. Sample splits from the backwash sample will be uniquely identified through this TI.
Container selection	Containers will be made of glass or plastic, consistent with the intended storage and follow-on processing.
Sample Preservation	Not applicable for current scope.
Sample chain of custody (COC)	COC is implemented by KBO&PR and provided with sample receipt at the RPL facility; internal COC is not required. Sample custody is maintained during the sample preparation phase. Sample security is maintained by limited access to the building and to laboratory areas. When the ASO laboratory receives samples, an Analytical Service Request (ASR) is generated.
Sample storage	It is anticipated that the entire sample will be split and acid digested. It is not anticipated that sample storage will be required.
Sample handling and transfer	Handling and transfers will be conducted in a manner to minimize potential for contamination (clean glassware and sampling devices), minimize loss (use secondary containment during process operations), and minimize tendency for sample to dry out (except where drying is required) where the wetting agent is deionized (DI) water.
Compositing and composite sub-sampling,	Mixing will be conducted on a best-effort basis. The sample will be thoroughly shaken to suspend finely divided solids as appropriate when sub-samples are pulled for centrifugation. Sub-samples will be pipetted out of the received sample container.

Table 1. QC Requirement and Implementation, continued

QC Requirement	Implementation
Holding time limits	A 6-month holding time has been designated for metals analysis by inductively coupled plasma optical emission spectrometry [ICP-OES]. Because radionuclide measurement is needed, in this case a hold time does not apply.
Waste disposition	Waste will be processed through the normal waste operations protocols and procedures.
Preparing required QC samples	Within the scope of work for this TI, QC sample requirements entail the creation of a sample duplicate for analysis.
Balance checks	Calibration of balances will be verified using one mass loading daily when used to assess accuracy. The balance performance results will be logged into this TI.

This TI will be approved by the BTR (QAPjP/SAP 3.4.3); the BTR's signature on the front page attests to the BTR approval.

Processing Summary

Part of the aqueous phase will be transferred to large-volume centrifuge containers and centrifuged to consolidate the fines; supernate will be decanted. The remaining sample will be mixed and the slurry transferred equally to the centrifuge containers with a transfer pipet. The slurry will be centrifuged to consolidate the solids and the supernate will be decanted. The client has requested to be present to observe the solids at this point.

After decanting supernate, the solids will be consolidated into one or two tared 15- or 40-mL glass, graduated, centrifuge cones (depending on solids quantity) and re-centrifuged. The centrifuged solids volume will be measured. The aqueous phase will be decanted and gross sample mass measured and net wet solids mass will be calculated. The sludge may be transferred into one centrifuge cone if the volume is not sufficient to measure—this option will be discussed with the BTR before proceeding and will likely hinge on which aspects are of greater importance: sludge density determination or duplicate analytical samples. A sample processing flow diagram is provided in Figure 1.

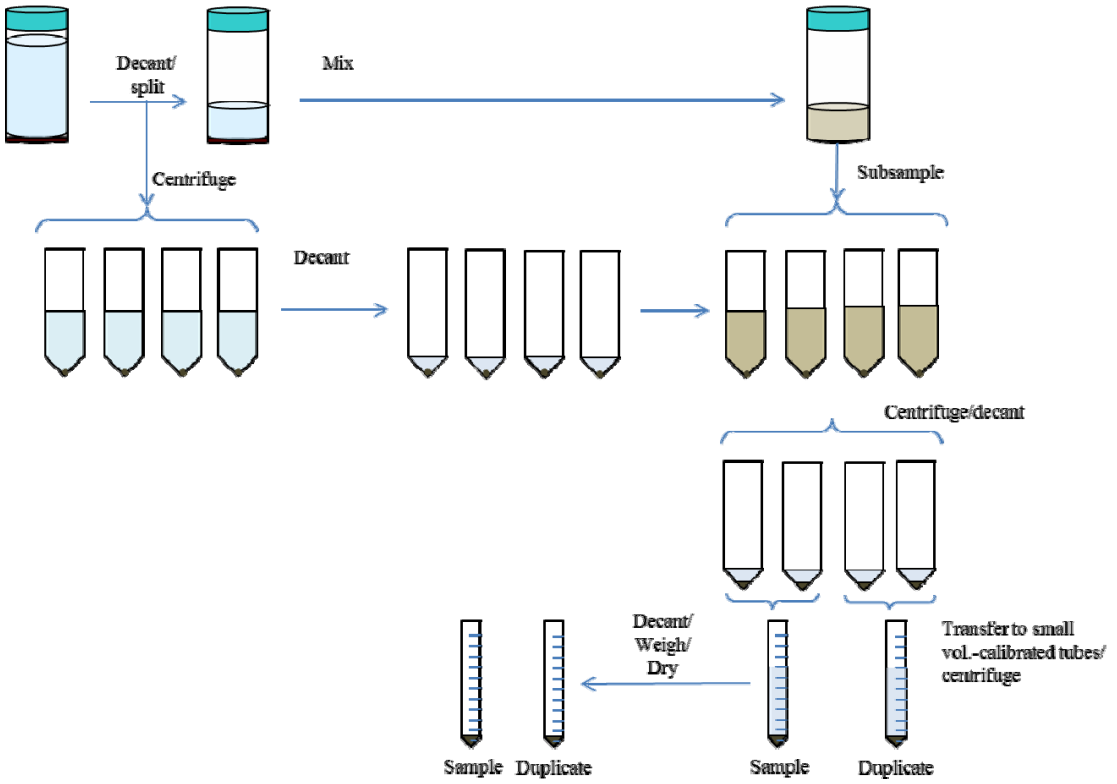

Figure 1. Sample Processing

The wet centrifuged solids will be transferred to the ASO laboratory for additional processing according to an ASR. ASO staff will dry the solids at 105°C to constant mass and the gross mass will be reported. The solids will be acid digested and the digestate will be analyzed for the radionuclides (except for gamma energy analysis, all analyses require radiochemical separation). Undigested residue will be weighed and measured for gamma emitters assuming the residue can be successfully transferred into a container suitable for gamma counting. Radionuclide measurement results will be provided on a dry mass basis.

Issues of Concern

Issues are of concern as defined below. The BTR will be kept informed as status progresses.

1) The relative uncertainty in the net sample mass increases as the sample mass decreases. Centrifuge tube mass measures will be collected on a four-place balance (0.0001 g readability with uncertainty of ~0.0006g). The uncertainty in the net sample mass will include the uncertainty in the tare weight and the gross sample masses according to Equation 1 where u is the total uncertainty and u_T is the uncertainty in the centrifuge container tare mass and u_G is the uncertainty in the centrifuge plus dry solids mass.

$$u = \sqrt{u_T^2 + u_G^2} \qquad \text{Eq. 1}$$

In this case the uncertainty in mass is expected to be ~0.0009 g. If the net sample mass is 0.01g, the mass uncertainty will represent 0.0009 g/0.01 g = 9%. This uncertainty will be added to the analytical uncertainty.

2) Once solids are consolidated, the solids dose rate may be too high to prepare in a fume hood. It is possible that part of the sample manipulations and the drying and acid digestion may have to occur in the hot cells.

Acronyms

ALARA	As low as reasonably achievable
ASO	Analytical Support Operations
ASR	Analytical Service Request
BPCL	Balance performance Check log (form)
BTR	Buyer's Technical Representative
COC	chain of custody
DI	deionized
DRR	Document review record (form)
HASQARD	Hanford Analytical Services Quality Assurance Requirements Document
ICP-OES	inductively coupled plasma optical emission spectrometry
ID	identification
KBO&PR	K Basin Operations & Plateau Remediation Project
LRB	laboratory record book (project-specific)
M&TE	measuring and test equipment
PNNL	Pacific Northwest National Laboratory
QA	quality assurance (program)
QAPjP	Quality Assurance Project Plan
QC	quality control (samples/activities)
RPL	Radiochemical Processing Laboratory
RPT	Radiological Protection Technologist
SAL	Shielded Analytical Laboratory
SAP	Sampling and Analysis Plan
SFO	Shield Facility Operations
TI	test instruction

M&TE List

Note:

A minimum of one balance performance check is to be performed daily when the balance is used. This entails the measure of one mid-range mass check, recording it on the balance performance check log (BPCL) form and comparing it to the acceptance criterion. The result will be used to verify that the balance is in working order with respect to accuracy.

If the balance is out of statistical process control, the Cognizant Scientist will provide guidance on how to proceed. The balance level may be verified, the balance re-zeroed, and then the control masses re-measured, or the balance may be re-calibrated by the user and the control masses re-measured. If the control masses are still out of the control limits, the statistical process control limits may be re-evaluated/re-established or other corrective action may be pursued (e.g., recalibrate balance; segregate, tag as out-of-service, and replace balance) with the concurrence of the Quality Engineer (QE).

List the balance information.

Balance 1: Make, model: Sartorius MSG234
Location: SAL
Capacity range: 0 to 240 g
Calibration ID: 40023610 Cal. Expiration Date: February 2016

Balance 2: Make, model: Sartorius R200D
Location: RPL Lab 525
Capacity range: 0 to 200 g
Calibration ID: 39080058 Cal. Expiration Date: August 2016

Balance 3: Make, model: Mettler AT400
Location: RPL Lab 305
Capacity range: 0 to 400 g
Calibration ID: N04143 Cal. Expiration Date: August 2016

√ Balance 4: Make, model: Mettler AT400 @ 10/23/15
Location: RPL Lab 525 Hood # 13
Capacity range: 0 to 400 g
Calibration ID: 1113061397 Cal. Expiration Date: August 2016

Cognizant Scientist Signature MK Fish, Date: 10/23/15

(M) 10/20/15 Balance 5: Make, model: Metler, SB16001

Location: RPL 1305 Bench top

Capacity range: 16160g

Calibration ID: 112520532 Cal. Expiration Date: 2/16

Cognizant Scientist Signature M. Fiske, Date: 10/23/15

Work Instructions

1.0 General Guidelines

The following general guidelines apply to all operations discussed in this TI.

1. TIs are controlled documents; TI steps are expected to be completed as written and TIs initialed and dated as work is performed. Skipping steps is not acceptable.
2. When a TI needs to be modified during the performance of work, modification needs to be discussed, before implementing, with the Project Manager (PM) and QE to determine what needs to be done to manage the work control process (this may include documenting modifications in notes, revising and re-issuing the TI, or issuing an addendum to the TI). Documentation for changes defined by the PM or QE needs to be added to the TI when received.
3. Concurrence for proposed changes or modifications also needs to be obtained from the K Basin client before the change or modification is implemented. Documentation for concurrence for changes needs to be added to the TI when received.
4. The PM or delegate is the point of contact for the request to modify TIs and to define and document how changes will be made. When TIs are revised during work, major revisions require the same level of review and approval as the initial TI to adequately evaluate the impact of changes on data and to evaluate those objectives and technical and quality requirements are adequately addressed and will be attained as expected. Minor or editorial changes require concurrence from the PM; the PM decides if the change is major or minor.
5. All TI and laboratory record book (LRB) entries need to be complete and legible. Initials need to be associated with a printed name, see Table 2 for printed name-initials cross-reference. Error corrections are to be made using a single line-out that is initialed and dated. If the reason for the correction is not obvious, an explanation may need to be added describing why the correction was made.
6. Initial and sign Table 2.
7. When finished with a Step, initial and date the item or step. This indicates that the step has been completed, by whom, and when.
8. Keep the sample in a sealed container as much as possible to prevent it from drying unless directed to do otherwise in this TI.
9. Minimize cross-contamination between samples and contamination of samples from outside sources. Use new or cleaned tools as directed in this TI or by the Cognizant Scientist.
10. Record any observations, problems, or information that might be needed later concerning samples and collection of representative sub-samples for analysis in space provided in this TI. Report any highly unusual/unexpected observation to the PM and QE, who will discuss the observation with the client. The Cognizant Scientist is authorized to stop work and re-plan to address anomalies based on evolving conditions during sample handling.

Table 2. Test Instruction User Identification

Initials	Printed Name	Signature	Position
MS	Margaret Smoot	M Smoot	Technician
SKF	Sandra Fiskum	Sandra K. Fiskum	Cognizant Scientist
	Rick Shimskey		Cognizant Scientist
		SKF 10/23/15	

Cognizant Scientist Signature S K. Fiskum, Date: 10/23/15

2.0 Sample Container Preparation

Date 10/13/15 Init MD 2.1 Select four 50-mL centrifuge containers with "leakproof" caps. Verify they hold water when tightened. Dry the centrifuge containers. (poly centrifuge tubes) MD 10/13/15

Date 10/14/15 Init MD 2.2 Label the centrifuge containers 68122-TI-001-A, -B, -C, and –D.

Date 9/14/15 Init MD 2.3 Prepare a 1-L poly bottle by labeling it as follows:

> **68122-TI-001-E**
> Backwash Filter Supernate
> Non-hazardous
> SK Fiskum

Date 10/15/15 Init MD 2.4 Tare the containers (use of the laboratory 305 balance is fine).

MD 2.4.1 Record the balance ID: N04143 (confirm balance is listed on the M&TE list or add the balance information to the M&TE list)

MD 2.4.2 Balance check weight ID: Fiona

MD 2.4.3 Balance check weight mass: 299.9996

MD 2.4.4 Verify that daily check was performed and acceptable (□) yes.

MD 2.4.5 Tare containers and record tare weights on Table 3.

Table 3. Centrifuge Container Tare

Container ID	Tare weight (g)
68122-TI-001-A	13.6274
68122-TI-001-B	13.6040
68122-TI-001-C	13.5193
68122-TI-001-D	13.4829
68122-TI-001-E	171.8946

* See Notes pg 13 MD 10/15/15

Cognizant Scientist Signature SK. Fiskum, Date: 10/23/15

3.0 Sample Receipt and Unpackaging

The sample Viking shipping container needs to be opened in the presence of a Radiological Protection Technologist (RPT).

Date 10/20/15 Init (TM) 3.1 Look over the chain of custody (COC) documentation for general correctness. Sign the COC and provide the original to SK Fiskum (TM) 10/26/15
Sample ID: KW-105 SF BW-001 (taken from photo records)

Date 10/21/15 Init (TM) 3.2 Unpack the sample from the Viking container; ~~reserve the Viking container for return shipment to the client.~~ Container sent back to client per SK Fiskum (TM) 10/20/15 Returned on shipping truck after sample ID was verified with COC ID SK Fiskum. 10/21/15

(TM) 3.2.1 Record the contact dose rate

Open Window: 1Y & 18B,

Closed Window: <5X,

and the 30-cm dose rate: <5mr

Dosimeter type RO20

Check the RWP limits; is dose rate within limits? yes

Date 10/21/15 Init (TM) 3.3 Record observations of the sample. Check visually for presence of settled solids.
took photos 10/20/15 - (No mac Beth color chart)
- Supernate was slightly turbid upon (TM) 10/21/15 ~~receipt~~ receipt
- Supernate was visualy less turbid than on 10/20/15 (see photos captured on 10/21/15)

Date 10/21/15 Init (TM) 3.4 Take photos of the sample showing the sample identification (ID) and the water level, turbidity, and any potential solids. Include Mac Beth Color Checker chart (or equivalent) for comparison/reference.

Date 10/21/15 Init (TM) 3.5 Weigh the sample.

(TM) 3.5.1 Record the balance ID: 1125202532 (confirm balance is listed on the M&TE list or add the balance information to the M&TE list)

(TM) 3.5.2 Balance check weight ID: Good, Bad & the ugly

(TM) 3.5.3 Balance check weight mass: 13000.4

(TM) 3.5.4 Verify that daily check was performed and acceptable (✓) (TM) yes ✓

(TM) 3.5.5 Record gross sample mass: 887.0g including bags & tape (TM) 10/26/15
0 Bags (1 & 2)
Tape (1 & 2)

Cognizant Scientist Signature SK. fiskum, Date: 10/27/15

Notes:

10/15/15 → also weighed 68122-TI-001-F (20.6780g) & 68122-TI-001-G (20.6185), which are both 10mL glass centrifuge tubes. (N) 10/15/15
↳ labled with orange paint pen (N) 10/15/15 & allowed to dry in oven over an hr. also did ~~test~~ leak check on them. (N) 10/15/15

Balance check on 1125202532 OK ✓

Balance (N) 10/23/15
- mass of Bag 1: 14.7g
- mass of Bag 2: 14.7g
- mass of tape 1: 0.9g
- mass of tape 2: 0.9g

} used in step 3.5.5 - to safely transport Sample to a balance with appropriate capacity.

Balance check on 1125202532 OK ✓

(N) 10/23/15
- Mass of Bag 3: 14.7g
- Mass of Bag 4: 14.7g
- mass of tape 3: 1.0g
- mass of tape 4: 1.0g

} used in Step 4.14 for the same reasons as step 3.5.5.

Dose Rates taken after Steps done in Step 4.19 (N) 10/23/15
Dosimeter type: RO20

Sample ID:	68122-TI-001-A	68122-TI-001-D
Contact open window:	5.5	5
Contact close window:	<0.5	<0.5
30 cm:	<0.5	<0.5

Cognizant Scientist Signature MK Fisher, Date: 10/23/15

4.0 Solids Consolidation

NOTE: Lead brick sample holders and bricks should be used as needed to keep dose at as low as reasonably achievable (ALARA) levels.

Date 10/20/15 Init [initials] 4.1 Set the sample aside in a clean secondary containment and leave undisturbed overnight to allow solids to settle.

Date 10/21/15 Init [initials] 4.2 Photograph the settled solids. Contact the Cognizant Scientist or PM to evaluate the settled solids volume. Cognizant Scientist/PM Discussion: Same set used in Step 3.4. taken [initials] 10/21/15

Date 10/21/15 Init [initials] 4.3 Place the centrifuge containers into a support stand lined up from A to D and uncap.

Date 10/21/15 Init [initials] 4.4 Open the sample bottle/jar and decant, using a transfer pipet, the water starting from the top surface. Place a transfer pipet load of water into each successive centrifuge container (starting with A and ending with D). Once each container has one pipet volume of water, start the filling cycle again from A to D. Continue until each centrifuge container is filled to about 35 to 40 mL.

Date 10/21/15 Init [initials] 4.5 Cap the sample container.

Date 10/21/15 Init [initials] 4.6 ~~Cap the centrifuge containers and~~ centrifuge for 20 minutes at 1500 RPM. ~~Centrifuge containers have no caps~~ [initials] 10/21/15 typo [initials] 10/27/15

Date 10/21/15 Init [initials] 4.7 Return centrifuge containers to the rack and examine for centrifuged solids. Take photographs of the samples (show sample ID in the image). If the supernate is cloudy, contact the Cognizant Scientist before proceeding.

Date 10/21/15 Init [initials] 4.8 Decant the aqueous phase from each centrifuge container, using a transfer pipet and being careful not to disturb the solids. If solids are disturbed, the sample will need to be re-centrifuged. Collect the water in the 1-L polybottle, 68122-TI-001-E. Leave about 5 mL of water overburden in each centrifuge container. If no solids are present, the entire contents can be transferred. Annotate in Table 4 the volume processed and if suspended solids were observed for each centrifuge container.

Date 10/21/15 Init [initials] 4.9 Repeat Steps 4.3 through 4.8 until the parent sample volume has been reduced to about 120 mL. Annotate in Table 4 the volume processed and if suspended solids were observed for each centrifuge

[initials] 10/21/15

Cognizant Scientist Signature Sandra L. Fiske, Date: 10/27/15

container. (Note, six cycles are anticipated, additional rows provided in case additional centrifuge cycles are required.

Table 4. Centrifuging Step Log

	Centrifuge Container ID			
	68122-TI-001-A	68122-TI-001-B	68122-TI-001-C	68122-TI-001-D
1st Sampling Volume, supernate clarity	✓ 45mL Very Clear	45mL Very Clear	45mL Very Clear	45mL Very Clear
2nd Sampling Volume, supernate clarity	✓ 45mL Very Clear	45mL Very Clear	45mL Very Clear	45mL (NS 10/21/15) Very Clear
3rd Sampling Volume, supernate clarity	✓ 45mL Very Clear	45mL Very Clear	45mL Very Clear	45mL Very Clear
4th Sampling Volume, 5th supernate clarity	✓ Solids transfer 45mL Very clear Supernate	45mL	45mL	45mL
4th Sampling Volume, 6th supernate clarity	* Solids transfer & Rinse 35mL	very clear		
5th Sampling Volume, 4th supernate clarity	45mL Very Clear	45mL Very Clear	45mL very clear	45mL Very Clear
6th Sampling Volume, 7th supernate clarity				
			(NS 10/23/15)	

Missed this supernate transfer Data taken from photo record. (NS 10/23/15)

(NS 10/23/15) (NS 10/23/15)

* Note: Rinsed with liquid From 68122-TI001-E. (NS 10/21/15)

Date 10/21/15 Init (NS) 4.10 Mix the parent sample to suspend solids—this can be accomplished with the transfer pipet (rapid in and out motions) or by swirling.

Date 10/21/15 Init (NS) 4.11 Using the same technique described in Step 4.4, transfer the suspended solids into each of the four centrifuge containers. Re-suspend the parent sample solids as needed so that the solids are equally distributed across each centrifuge tube.

Cognizant Scientist Signature D K. Fisher, Date: 10/23/15

Date 10/21/15 Init MD 4.12 Add water from 68122-TI-001-E to the parent bottle as needed to quantitatively transfer the solids.

Date 10/21/15 Init MD 4.13 Cap the sample bottle and centrifuge containers.

Date 10/22/15 Init MD 4.14 Weigh the emptied sample bottle.

MD 4.14.1 Record the balance ID: 1125202532 (confirm balance is listed on the M&TE list or add the balance information to the M&TE list).

MD 4.14.2 Balance check weight ID: Good, Bad & Ugly

MD 4.14.3 Balance check weight mass: 13000.3

MD 4.14.4 Verify that daily check was performed and acceptable (✓) MD ✓ yes

MD 4.14.5 Record emptied container mass: 123.1 g with bags & tape
bags (3) tape (3) MD 10/26/15

Date 10/21/15 Init MD 4.15 Centrifuge the centrifuge containers for 30 minutes at 1500 RPM.

Date 10/21/15 Init MD 4.16 Return centrifuge containers to the rack and examine for centrifuged solids. Take photographs of the samples (show sample ID in the image). If the supernate is cloudy, contact the Cognizant Scientist before proceeding.

Date 10/21/15 Init MD 4.17 Decant the aqueous phase from each centrifuge container, using a transfer pipet and being careful not to disturb the solids. Collect the water in the 1-L polybottle, 68122-TI-001-E. Leave about 5 mL of water overburden in each centrifuge container.

Date 10/22/15 Init MD 4.18 Obtain a dose rate for each sample from the RPT and record in Table 5.

Dosimeter type R020

Cognizant Scientist Signature M. Fiske, Date: 10/27/15

Table 5. Centrifuge Container Dose Rates

Container ID	Dose Rates, mR/hr
68122-TI-001-A	Contact open window 5 Contact closed window 3 30 cm <0.5
68122-T-I001-B	Contact open window 5 Contact closed window 3.5 30 cm <0.5
68122-TI-001-C	Contact open window 5 Contact closed window 3.5 30 cm <0.5
68122-TI-001-D	Contact open window 2 Contact closed window 1 30 cm <0.5
68122-TI-001-E	Contact open window <0.5 Contact closed window <0.5 30 cm <0.5
Parent bottle	Contact open window <0.5 Contact closed window <0.5 30 cm <0.5

Date 10/23/15 Init [illegible] 4.19 HOLDPOINT—Share dose rate results with SK Fiskum, PM, to determine if continued work in fume hood is viable and if processing of all four solids subsamples or two solids subsamples will be conducted (this decision depends on the quantity of solids collected and variation in the dose rates). Client has requested to be present to observe the consolidated solids at this point.

Holdpoint Direction: B/C are ok, but Combine A/D & Re-Split. take another dose rate in an attempt to keep dose rate #'s as evenly Split as possible

See page 13 for New Dose rates taken after this step. (10/26/15)

PM signature/date SK. Fiskum 10/23/15

Cognizant Scientist Signature SK Fiskum, Date: 10/27/15

Date 10/15/15 Init MD 4.20 Determine the appropriate size and number of calibrated glass centrifuge tubes: 15-mL or 40-mL.

Cognizant Scientist/PM Discussion: 10 mL as provided by SK Fiskum MD 10/15/15

Date 10/15/15 Init MD 4.21 Prepare centrifuge tubes by labeling them as 68122-TI-001-~~S~~ G MD 10/23/15 and 68122-TI-001-~~Dup~~ F (duplicate is used if enough solids are available for duplicate processing). MD 10/23/15

Date 10/23/15 Init MD 4.22 Tare the centrifuge tubes using the laboratory ~~525 Sartorius balance~~ Mettler AT 400 hood #13 in lab 525 * this is the balace for Acid digests ~~digets~~. wanted to use same balace through out analysis MD 10/23/15

MD 4.22.1 Record the balance ID: 1113292667 (confirm balance is listed on the M&TE list or add the balance information to the M&TE list)

MD 4.22.2 Balance check weight ID: Sleepy

MD 4.22.3 Balance check weight mass: 300.0017g

MD 4.22.4 Verify that daily check was performed and acceptable (✓) MD ✓ yes

MD 4.22.5 Tare centrifuge tubes and record tare weights on Table 6.

Table 6. Centrifuge Container Tare

Container ID	Tare weight (g)
68122-TI-001-~~S~~ G MD 10/23/15	20.6253
68122-TI-001-~~Dup~~ F	20.6859

Date 10/23/15 Init MD 4.23 Stage the centrifuge tubes in a rack to maintain them upright and in secondary containment.

Date 10/23/15 Init MD 4.24 Slurry solids from 68122-TI-001-A and transfer to 68122-TI-001-~~S~~. G MD 10/23/15

Date 10/23/15 Init MD 4.25 Slurry solids from 68122-TI-001-B and transfer to 68122-TI-001-~~S~~. G MD 10/23/15

Date 10/23/15 Init MD 4.26 Slurry solids from 68122-TI-001-C and transfer to 68122-TI-001-~~Dup~~. F

Date 10/23/15 Init MD 4.27 Slurry solids from 68122-TI-001-D and transfer to 68122-TI-001-~~Dup~~. F MD 10/23/15

Cognizant Scientist Signature SK. Fiskum, Date: 10/27/15

Date 10/23/15 Init MD 4.28 Allow solids in each centrifuge tube to settle. If needed, decant water with a transfer pipet from the glass centrifuge tube back to the larger centrifuge container to slurry and remove residual solids.

Date 10/23/15 Init MD 4.29 The total volume in the centrifuge tubes needs to be equivalent to balance the centrifuge—adjust with the decant water in 68122-TI-001-E, as needed.

Date 10/23/15 Init MD 4.30 Cap the centrifuge tubes.

Date 10/23/15 Init MD 4.31 Centrifuge for 30 minutes at 1500 RPM.

Date 10/23/15 Init MD 4.32 Return centrifuge tubes to the rack and examine for centrifuged solids. Take photographs of the samples (show sample ID in the image). If the supernate is cloudy, contact the Cognizant Scientist before proceeding.

Date 10/23/15 Init MD 4.33 Decant the aqueous phase from each centrifuge tube, using a transfer pipet and being careful not to disturb the solids. If solids are disturbed, the sample will need to be centrifuged again. Collect the decant water in the ~~250-mL~~ 1L typo MD 10/23/15 polybottle, 68122-TI-001-E. Leave about 1 mL of water overburden in each centrifuge tube.

Date 10/23/15 Init MD 4.34 Record the centrifuged solids volume, total slurry volume, and measure the gross sample mass (same balance that the tare was taken).

MD 4.34.1 Record the balance ID: 1113292667 (confirm balance is listed on the M&TE list or add the balance information to the M&TE list)

MD 4.34.2 Balance check weight ID: Slurpy

MD 4.34.3 Balance check weight mass: 300.0017g

MD 4.34.4 Verify that daily check was performed and acceptable (✓) CK ✓ MD

MD 4.34.5 Record information in Table 7.

Table 7. Sample Volumes and Masses

MD 10/23/15

Parameter	68122-TI-001-S	68122-TI-001-Dup
Centrifuged solids volume , mL	0.4ml	0.4mL
Slurry volume, mL	0.5ml	0.5ml
Gross mass, g	21.1095	21.2217

Cognizant Scientist Signature AK. Fiskum, Date: 10/23/15

Did the same with c/D as well only with 2mL from bottle E each time.

MD 10/23/15 Did 2 rinses on AB using 1.5 mL of supernate from bottle E 1st rinse 1.5mL used in A then transferd to B & placed in glass centrifuge tube 2nd rinse repeat 1st rinse process with a new 1.5mL from bottle E,

Date 10/27/15 Init (NA) 4.35 Submit Samples to the ASO for drying, acid digestion, and analysis.
Record the ASR number: 99116

Notes:

Took photos of empty centrifuge tubes with ID's:

(NA) 10/28/15
68122-TI-001-A
68122-TI-001-B
68122-TI-001-C
68122-TI-001-D

also took a photo of the mac-beth color chart

MK-Acid 10/27/15

Cognizant Scientist Signature MK Fish, Date: 10/27/15

5.0 Closing Activities

Date 10/23/15 Init (AD)	5.1	Download images and create a contact sheet and attach to this TI. Attachment C
Date 10/28/15 Init JLT	5.2	Generate calculation worksheets (sludge mass and density after centrifuging) and attach a printed copy of reviewed calculations to this TI.
Date 10/23/15 Init (AD)	5.3	Collect relevant BPCL form copies and attach to this TI. Attachment A
Date 10/27/15 Init (AD)	5.4	Be sure balance Calibration Reports are attached to the TI. Attachment B
Date 10/23/15 Init (AD)	5.5	Attach any relevant Occurrence, Deficiency, or Nonconformance Reports generated during the work to this TI.
Date 10/27/15 Init (AD)	5.6	Submit completed TI for technical review. Attach completed Document Review Record (DRR) form by the technical reviewer to TI.
Date 10/28/15 Init JLT	5.7	Submit technically reviewed TI to the QE for review.

* Attachment D → email correspondence regarding this TI (AD) 10/23/15

* Attachment E → original Chain of ~~Co~~ Custody Docs included with the shipment of the Sample (AD) 10/26/15 (AD) 10/27/15

Cognizant Scientist Signature Mk. Fish, Date: 10/27/15

68122-TI-001 Attachment A Pg 1 of 3

✓ZnP 8/25/15 (ND) 10/23/15

Balance Performance Check Log

Pacific Northwest National Laboratory

Mettler AT400

Analytical Support Operations

Balance Information
Identificaiton (e.g. WD34567)
N04143
Location (e.g. Building/Room)
RPL / 305 Benchtop
Calibration Due Date
Aug-16

Check Weight (CW) Information	#1	CW Units	#2	CW Units	#3	CW Units	#4	CW Units
CW Identification	FIONA							
CW Assigned Mass (+Units):	299.9999	g		g		g		g
Method for Assigning Diff/Range[a]:	Option 1		Option 1		Option 1		Option 1	
CW Difference ± Mass (+Units):								
Or Range Low Mass (+Units) &:	299.9978	g		g		g		g
Range High Mass (+Units):	300.0020	g		g		g		g

(a) Enter "Option #1" or "Option #2" from Procedure PNL-ASO-052, Table 1. For factors >3, append factor in parentheses, e.g., Option #1 (4)

Date	Lab Staff Initials	Check Weight ID or #	Check Weight Measured Mass (+Units)	Meets Acceptance Criterion (Within Diff/Range)? (Y/N)	Comments/Corrective Action
8/24/2015	RG	Fiona	299.9999		Weights taken to establish acceptance criteria.
8/24/2015	RG	Fiona	299.9999		
8/24/2015	RG	Fiona	299.9999		
8/24/2015	RG	Fiona	299.9999		
8/24/2015	RG	Fiona	300.0000		
8/27/15	SK7	"	299.9999	yes	
9/2/15	ND	Fiona	300.0000	yes	
9/3/15	ND	Fiona	300.0000	yes	
9/4/15	ND	Fiona	300.0003	yes	
9/4/15	SK7	"	300.00011	yes	
9/24/15	SK7	"	300.0003	yes	
10/2/15	ND	"	300.0004	yes	
10/2/15	SK7	"	300.0006	yes	
10/5/15	HAC	"	300.0001	yes	
10/6/15	HAC	"	300.0010	yes	
10/9/15	SK7	"	300.0003	yes	
10/12/15	ND	"	299.9999	yes	
10/13/15	SK7	"	300.0003	yes	
10/14/15	SK7	"	300.0000	yes	
10/15/15	ND	"	299.99990	yes	
10/16/15	HAC	"	300.0000	yes	
10/19/15	HAC	"	300.0005	yes	
10/20/15	SK7	"	299.9995	yes	
10/21/15	SK7	"	299.9994	yes	
10/22/15	HAC	"	299.9985	yes	

> Complete all column entries.

> For corrections, place single line through incorrect entry, enter correction and initial/date.

> When full, forward to the ASO M&TE Records Custodian; replace with new Log.

> Following recalibration, lineout/ initial/date unused rows and forward to ASO M&TE Records Custodian; replace with new Log.

> To calibrate or recalibrate a balance contact the ASO M&TE Records Custodian.

> If additional check weights are used on balance, enter appropriate information on another sheet and append to this Log form.

Reviewer Signature _____ **Date** _____ **Date to ASO Records** _____

68122-TI001 Attachment A pg 2 of 3

Balance Performance Check Log

√ xmp 2/12/15

Radiochemical Processing Laboratory

Mettler SB16001

Balance Information
Identificaiton (e.g. WD34567)
1125202532
Location (e.g. Building/Room)
RPL/305 (benchtop)
Calibration Due Date
Feb-16

Check Weight (CW) Information	#1	CW Units	#2	CW Units	#3	CW Units	#4	CW Units
CW Identification	Good, Bad & Ugly							
CW Assigned Mass (+Units):	13000.0	g		g		g		g
Method for Assigning Diff/Range[a]:	Option 1		Option 1		Option 1		Option 1	
CW Difference ± Mass (+Units):								
Or Range Low Mass (+Units) &:	12998.8	g		g		g		g
Range High Mass (+Units):	13001.2	g		g		g		g

(a) Enter "Option #1" or "Option #2" from Procedure ADM-RSEG-BALANCES, Table 1. For factors >3, append factor in parentheses, e.g., Option #1 (4)

Date	Lab Staff Initials	Check Weight ID or #	Check Weight Measured Mass (+Units)	Meets Acceptance Criterion (Within Diff/Range)? (Y/N)	Comments/Corrective Action
2/11/2015	RG	Good, Bad & Ugly	13000.0		Weights taken to establish acceptance criteria.
2/11/2015	RG	Good, Bad & Ugly	13000.0		
2/11/2015	RG	Good, Bad & Ugly	13000.0		
2/11/2015	RG	Good, Bad & Ugly	13000.0		
2/11/2015	RG	Good, Bad & Ugly	13000.1		
9/14/15	DKT	"	13,000.3	yes	
10/9/15	DKT	"	13,000.5	yes	
10/12/15	HMC	"	13,000.3	yes	
10/13/15	HMC	"	13,000.4	yes	
10/16/15	[illegible]	"	13,000.3	yes	
10/21/15	[illegible]	"	13,000.4	yes	
10/22/15	HMC	"	13,000.3	yes	

> **Complete all column entries.**

> **For corrections, place single line through incorrect entry, enter correction and initial/date.**

> When full, forward to the Balance Calibration Coordinator and request a new log; replace with new Log.

> Following Vendor recalibration, lineout/ initial/date unused rows and forward to Balance Calibration Coordinator who will replace the BPCL with a new log.

> To calibrate or recalibrate a balance contact the Balance Calibration Coordinator.

> If additional check weights are used on balance, enter appropriate information on another sheet and append to this Log form.

Reviewer Signature _____ **Date** _____ **Date to ASO Records** _____

BPCL 2_11_15_Mettler SB16001_1125202532_RPL_305_Benchtop.xls

68122-TI-001
Attachment A
Pg 3 of 3 RG 10/23/15

✓ xnP 8/19/15

Balance Performance Check Log

Pacific Northwest National Laboratory

Mettler AT 400

Analytical Support Operations

Balance Information
Identificaiton (e.g. WD34567)
1113292667
Location (e.g. Building/Room)
RPL / 420 Fumehood #13
Calibration Due Date
Aug-16

Check Weight (CW) Information	#1	CW Units	#2	CW Units	#3	CW Units	#4	CW Units
CW Identification	SLEEPY							
CW Assigned Mass (+Units):	300.0001	g		g		g		g
Method for Assigning Diff/Range[(a)]:	Option 1		Option 1		Option 1		Option 1	
CW Difference ± Mass (+Units):								
Or Range Low Mass (+Units) &:	299.9980	g		g		g		g
Range High Mass (+Units):	300.0022	g		g		g		g

(a) Enter "Option #1" or "Option #2" from Procedure PNL-ASO-052, Table 1. For factors >3, append factor in parentheses, e.g., Option #1 (4)

Date	Lab Staff Initials	Check Weight ID or #	Check Weight Measured Mass (+Units)	Meets Acceptance Criterion (Within Diff/Range)? (Y/N)	Comments/Corrective Action
8/18/2015	RG	Sleepy	300.0005		Weights taken to establish acceptance criteria.
8/18/2015	RG	Sleepy	300.0001		
8/18/2015	RG	Sleepy	300.0000		
8/18/2015	RG	Sleepy	299.9999		
8/18/2015	RG	Sleepy	300.0000		
10/23/15	RG	Sleepy	300.0017	yes	

> **Complete all column entries.**

> **For corrections, place single line through incorrect entry, enter correction and initial/date.**

> When full, forward to the ASO M&TE Records Custodian; replace with new Log.

> Following recalibration, lineout/ initial/date unused rows and forward to ASO M&TE Records Custodian; replace with new Log.

> To calibrate or recalibrate a balance contact the ASO M&TE Records Custodian.

> If additional check weights are used on balance, enter appropriate information on another sheet and append to this Log form.

Reviewer Signature _____ **Date** _____ **Date to ASO Records** _____

BPCL 8_18_15_Mettler AT 400_1113292667_RPL_420_Fumehood.xls

68122-TI-001
Attachment B
Pg 1 of 3 10/26/15

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ACCREDITED
Calibration Services
Certificate Number 1550.01
Laboratory code 115953

Battelle Pacific N.W. Natl. Lab
902 Battelle Blvd.
Richland, WA 99354

Report Number: BATN03N04143150824

A2LA ACCREDITED
CERTIFICATE OF CALIBRATION WITH DATA

INSTRUMENT INFORMATION

Item	Make	Model	Serial Number	Customer ID	Location
Balance	Mettler	AT400	N04143	360-06-01-048	RPL 305
Units	**Readability**	**SOP**	**Cal Date**	**Last Cal Date**	**Cal Due Date**
g	0.0001	QC012	8/24/15	8/22/14	8/2016

FUNCTIONAL CHECKS

ECCENTRICITY
Test Wt: 200 Tol: 0.0004
As-Found: Pass: ☑ Fail: ☐
As-Left: Pass: ☑ Fail: ☐

LINEARITY
Test Wt: 100x4 Tol: 0.0005
As-Found: Pass: ☑ Fail: ☐
As-Left: Pass: ☑ Fail: ☐

STANDARD DEVIATION
Test Wt: 100 Tol: 0.0002

1. 100.0000	5. 100.0002	9. 100.0001
2. 100.0000	6. 100.0001	10. 100.0000
3. 100.0001	7. 99.9999	Result
4. 100.0001	8. 100.0000	0.00008

ENVIRONMENTAL CONDITIONS
☐ Good ☑ Fair ☐ Poor
Temperature: 23.9°C

A2LA ACCREDITED SECTION OF REPORT

Standard	As-Found	As-Left	Expanded Uncertainty
400	400.0000	399.9989	0.00025
300	300.0002	299.9995	0.00025
200	200.0005	200.0001	0.00020
100	100.0002	100.0000	0.00020
50	50.0002	50.0001	0.00020
1	1.0004	1.0002	0.00020

☑ Accepted ☐ Rejected / Per Clause(s): 17.5.b

AQSS Reviewer ds cuffy 9-21-15

CALIBRATION STANDARDS

Item	Make	Model	Serial Number	Cal Date	Cal Due Date	NIST ID
Weight Set	Rice Lake	30 kg-1mg	S751	12/2/14	12/2015	OR-13-314-C

Permanent Information Concerning this Equipment:

Comments/Info Concerning this Calibration:
8/15 PO #263294 As found / as left within tolerance.

Report prepared/reviewed by: [signature] **Date:** 9-1-15 **Technician:** R. Hintz

Signature: [signature]

The uncertainty is calculated according to the ISO Guide to the Expression of Uncertainty in Measurement and includes the uncertainty of standards used combined with the observed standard deviation and readability of the unit under test. The uncertainty is expanded with a k factor of 2 for an approximate 95% level of confidence. Instruments listed above were calibrated using standards traceable to the National Institute of Standards and Technology (NIST). Calibration data reflect results at the time and location of calibration. Calibration data should be reviewed to insure that the instrument is performing to its required accuracy. Calibrations comply with ISO/IEC 17025 and ANSI/Z540-1-1994 quality standards.

PT ID: BATN03

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68122-TI-001
Attachment B
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Certificate Number: 1550.01
Laboratory code: 115953

Battelle Pacific N.W. Natl. Lab
902 Battelle Blvd.
Richland, WA 99354

Report Number: BATN031125202532150211

A2LA ACCREDITED
CERTIFICATE OF CALIBRATION WITH DATA

INSTRUMENT INFORMATION

Item	Make	Model	Serial Number	Customer ID	Location
Balance	Mettler	SB16001	1125202532	1125202532	RPL 305
Units	**Readability**	**SOP**	**Cal Date**	**Last Cal Date**	**Cal Due Date**
g	0.1	QC012 10/14	2/11/15	7/14/14	2/2016

FUNCTIONAL CHECKS

ECCENTRICITY	LINEARITY	STANDARD DEVIATION	ENVIRONMENTAL CONDITIONS
Test Wt: 5000 Tol: 0.5	Test Wt: 5000x3 Tol: 0.3	Test Wt: 5000 Tol: 0.1	☐ Good ☑ Fair ☐ Poor
As-Found: Pass: ☑ Fail: ☐	As-Found: Pass: ☑ Fail: ☐	1. 5000.0 5. 5000.0 9. 5000.0	
As-Left: Pass: ☑ Fail: ☐	As-Left: Pass: ☑ Fail: ☐	2. 5000.0 6. 5000.0 10. 5000.0	
		3. 5000.0 7. 5000.0 Result	Temperature: 22.4°C
		4. 5000.0 8. 5000.0 0.00	

☑ Accepted ☐ Rejected / Per Clause(s): 1750
AQSS Reviewer
3-3-15

A2LA ACCREDITED SECTION OF REPORT

Standard	As-Found	As-Left	Expanded Uncertainty
15000	14999.8	15000.1	0.11
10000	9999.8	10000.0	0.11
5000	4999.9	5000.0	0.11
2000	2000.0	2000.0	0.11
1000	1000.0	1000.0	0.11
500	500.0	500.0	0.11

CALIBRATION STANDARDS

Item	Make	Model	Serial Number	Cal Date	Cal Due Date	NIST ID
Weight Set	Rice Lake	30 kg-1mg	S751	12/2/14	12/2015	OR-13-314-C

Permanent Information Concerning this Equipment:

Comments/Info Concerning this Calibration:
2/15 PO #251158 As found / as left within tolerance (manufacturer specifications).

Report prepared/reviewed by: M. Mercer **Date:** 2/19/15 **Technician:** R. Hintz

Signature:

The uncertainty is calculated according to the ISO Guide to the Expression of Uncertainty in Measurement and includes the uncertainty of standards used combined with the observed standard deviation and readability of the unit under test. The uncertainty is expanded with a k factor of 2 for an approximate 95% level of confidence. Instruments listed above were calibrated using standards traceable to the National Institute of Standards and Technology (NIST). Calibration data reflect results at the time and location of calibration. Calibration data should be reviewed to insure that the instrument is performing to its required accuracy. Calibrations comply with ISO/IEC 17025 and ANSI/Z540-1-1994 quality standards.

PT ID: BATN03

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68122-TI-001
Attachment B
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Calibration Services
Certificate Number: 1850.01
Laboratory code: 115853

Battelle Pacific N.W. Natl. Lab
902 Battelle Blvd.
Richland, WA 99354

Report Number: BATN031113292667150818

A2LA ACCREDITED
CERTIFICATE OF CALIBRATION WITH DATA

INSTRUMENT INFORMATION

Item	Make	Model	Serial Number	Customer ID	Location
Balance	Mettler	AT400	1113292667	360-06-01-037	RPL 420
Units	**Readability**	**SOP**	**Cal Date**	**Last Cal Date**	**Cal Due Date**
g	0.0001	QC012	8/18/15	8/20/14	8/2016

FUNCTIONAL CHECKS

ECCENTRICITY
Test Wt: 200 Tol: 0.0004
As-Found: Pass: ☑ Fail: ☐
As-Left: Pass: ☑ Fail: ☐

LINEARITY
Test Wt: 100x4 Tol: 0.0005
As-Found: Pass: ☑ Fail: ☐
As-Left: Pass: ☑ Fail: ☐

STANDARD DEVIATION
Test Wt: 50 Tol: 0.0002

1. 49.9999	5. 49.9997	9. 49.9998
2. 49.9999	6. 49.9995	10. 49.9995
3. 49.9999	7. 49.9994	**Result**
4. 49.9999	8. 49.9993	0.00023

ENVIRONMENTAL CONDITIONS
☐ Good ☑ Fair ☐ Poor
Temperature: 22.8°C

A2LA ACCREDITED SECTION OF REPORT

Standard	As-Found	As-Left	Expanded Uncertainty
400	399.9996	400.0003	0.00050
300	299.9995	300.0001	0.00050
200	199.9997	200.0001	0.00048
100	99.9998	100.0000	0.00048
50	49.9998	49.9999	0.00048
1	0.9998	0.9999	0.00048

☑ Accepted ☐ Rejected / Per Clause(s): 17.5.b

AQSS Reviewer ds cathey 9-21-15

CALIBRATION STANDARDS

Item	Make	Model	Serial Number	Cal Date	Cal Due Date	NIST ID
Weight Set	Rice Lake	30 kg-1mg	S751	12/2/14	12/2015	OR-13-314-C

Permanent Information Concerning this Equipment:

Comments/Info Concerning this Calibration:
8/15 PO #263294. As found / as left within tolerance.

Report prepared/reviewed by: JH Hintz **Date:** 9-1-15 **Technician:** R. Hintz

Signature:

The uncertainty is calculated according to the ISO Guide to the Expression of Uncertainty in Measurement and includes the uncertainty of standards used combined with the observed standard deviation and readability of the unit under test. The uncertainty is expanded with a k factor of 2 for an approximate 95% level of confidence. Instruments listed above were calibrated using standards traceable to the National Institute of Standards and Technology (NIST). Calibration data reflect results at the time and location of calibration. Calibration data should be reviewed to insure that the instrument is performing to its required accuracy. Calibrations comply with ISO/IEC 17025 and ANSI/Z540-1-1994 quality standards.

PT ID: BATN03

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Attachment C
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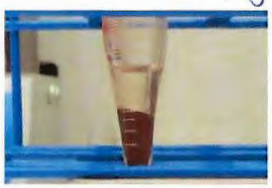
10/23/15
10/26/15

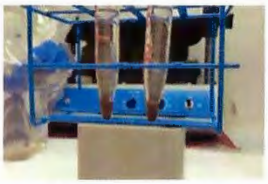

Color Chart 10-23-15.jpg


mpty tubes after step 4.28.1.jp
Empty tubes after Step 4.28.1.jpg
10/23/15

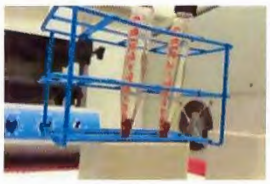

Empty tubes after step 4.28.jpg
10/23/15


final spin down F.jpg


Final spin down G.jpg


Initial spin down.1.jpg


Initial spin down.2.jpg


Initial spin down.jpg

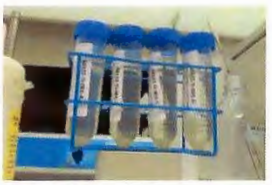

after 1st supernate spin.1.jpg


after 1st supernate spin.jpg


after 2nd supernate spin.1.jpg


after 2nd supernate spin.jpg


after 3rd supernate spin.1.jpg


after 3rd supernate spin.jpg


after 4th supernate spin.1.jpg


after 4th supernate spin.jpg

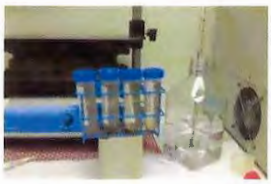

Color chart 10-21-15.jpg


ample condtion after 24hrs.1.jp
10/23/15


ample condtion after 24hrs.2.jp
Sample 10/23/15 JPG


ample condtion after 24hrs.3.jp
Sample 10/23/15 JPG


Sample condtion after 24hrs.jpg
10/23/15


Solids transfer rinse 1.1.jpg


Solids transfer rinse 1.jpg


Solids transfer rinse 2.jpg

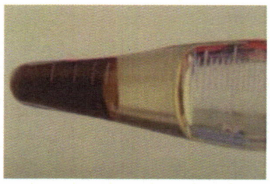

Solids transfer rinse 2.1.jpg


ived sample condition 10-20-1
As received 10-20-15.jpg
10/23/15
10/23/15

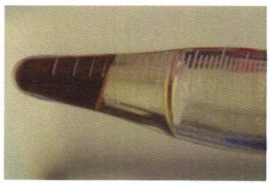

Shipping conditions 10-20-15.jpg
10/23/15


eived sample condition 10-20-
As received Sample Condition 10-20-15.jpg
10/23/15

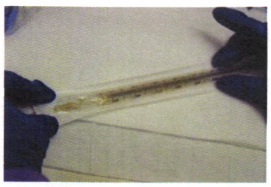
68122-TI-001
Attachment C
Pg 2 of 2



68122-TI-001-G solids.jpg



68122-TI-001-F solids.jpg



68122TI0-001 transfer tube.jpg

68122-TI-001
Attachment D
Pg 1 of 2
10/23/15

Fiskum, Sandra K

Subject: FW: sample observation

From: Johnson, Michael E (CHPRC)
Sent: Thursday, October 22, 2015 8:49 AM
To: Fiskum, Sandra K
Cc: Davis, George M (CHPRC)
Subject: RE: sample observation

Sandy,

I confirm that CHPRC observation is not required of the 105-KW sand filter backwash sample from the north load-out pit.

Michael E. Johnson
Senior Technical Advisor
CH2MHILL Plateau Remediation Company
Office 509-372-3628
Cell 509-430-3291

From: Fiskum, Sandra K [mailto:sandy.fiskum@pnnl.gov]
Sent: Thursday, October 22, 2015 7:55 AM
To: Johnson, Michael E
Cc: Davis, Mike
Subject: RE: sample observation

The quantity of centrifuged solids in each of the 50-mL centrifuge tubes appears to be on the order of 0.3 to 0.5 mL.
I believe that we have enough total solids to conduct duplicate sample analysis as indicated in the test instruction (Figure 1)—that is ~0.6 to 1 mL centrifuged solids/sample.

I believe we can get reasonable volume and mass measures in our 10-mL glass centrifuge tubes.

I would like to proceed in completing the TI steps. Step 4.19 indicates client observation request. Your message below indicates that this is not necessary. Please confirm this for our records with an email response indicating that client observation is not required.

Thank you,
Sandy

From: Johnson, Michael E (CHPRC)
Sent: Thursday, October 22, 2015 5:22 AM

68122-TI-001
Attachment D
pg 2 of 2 (10/10/23)

To: Fiskum, Sandra K
Subject: RE: sample observation

Sandy,

I appreciate the photographs.

I don't need to see the samples in person.

Mike Davis is away on personal business and won't be back until November 2. He is periodically checking his email.
I think Mike Davis was more interested in getting a general tour of the lab.

Does it look like you'll have enough sample to perform the requested analyses?

Thank you,

Michael E. Johnson
Senior Technical Advisor
CH2MHILL Plateau Remediation Company
Office 509-372-3628
Cell 509-430-3291

From: Fiskum, Sandra K [mailto:sandy.fiskum@pnnl.gov]
Sent: Wednesday, October 21, 2015 2:01 PM
To: Davis, Mike; Johnson, Michael E
Subject: sample observation

Mike and John,

Some time back you had requested the opportunity to come and see the samples.

Attached is a picture of the initial spin-down processing of the samples (4 conical vial with brown solids at bottom; the jug of water to the right is the decanted water).

The samples are located in a Radiation Area. If you want to see them, I will need to request dosimeters for you. This could be arranged perhaps for Monday or Tuesday. Do you still want to see the samples or is the photograph sufficient?

Thank you,

Sandy Fiskum
Senior Research Scientist
Pacific Northwest National Laboratory
509-375-5677

CH2MHILL CHPRC	CHAIN OF CUSTODY/SAMPLE ANALYSIS REQUEST	COC # STP-15-0023	PAGE ___ of ___

COLLECTOR	COMPANY CONTACT	TELEPHONE NO.	PROJECT COORDINATOR	PRICE CODE / DATA TURNAROUND
P.S. HAWKEY	BILL M. KRUEGER	373-7976	BILL M KRUEGER	
SAMPLING LOCATION	**PROJECT DESIGNATION**		**SAF NO.**	**AIR QUALITY** ☐
105KW BASIN	105KW SAMPLING WATER		N/A	

ICE CHEST NO.	FIELD LOGBOOK NO.	ACTUAL SAMPLE DEPTH	COA	METHOD OF SHIPMENT
N/A		N/A		GOVERNMENT VEHICLE

SHIPPED TO	OFFSITE PROPERTY NO.	BILL OF LADING/AIR BILL NO.
PNNL LAB	N/A	N/A

MATRIX *	POSSIBLE SAMPLE HAZARDS/REMARKS	PRESERVATION	N/A
A=Air, DL=Drum Liquids, DS=Drum Solids, L=Liquid, O=Oil, S=Soil, SE=Sediment, T=Tissue, V=Vegetation, W=Water, WI=Wipe, X=Other	RADIOACTIVE	HOLDING TIME	
		TYPE OF CONTAINER	G/P
		NO. OF CONTAINER(S)	1
		VOLUME	1 L
	SPECIAL HANDLING AND/OR STORAGE: N/A	SAMPLE ANALYSIS	

SAMPLE NO.	MATRIX*	SAMPLE DATE	SAMPLE TIME
KW-105SFBW-001	WATER	10-19-15	1330

CHAIN OF POSSESSION	Sign/Print Names	SPECIAL INSTRUCTIONS
RELINQUISHED BY/REMOVED FROM: P.S. HAWKEY FOR Tally — DATE/TIME: 10-19-15 1330	RECEIVED BY/STORED IN: LOCKED IN ROOM #4 — DATE/TIME: 10-19-15 1415	Analysis Per 49517-Release 35
RELINQUISHED BY/REMOVED FROM: Room #4 Jim Bussche — DATE/TIME: 10/20/15 0745	RECEIVED BY/STORED IN: Jim Bussche — DATE/TIME: 10/20/15	
RELINQUISHED BY/REMOVED FROM: — DATE/TIME:	RECEIVED BY/STORED IN: — DATE/TIME: 10/20/15	
RELINQUISHED BY/REMOVED FROM: Jim Bussche — DATE/TIME: 10/20/15 0824	RECEIVED BY/STORED IN: Curt Morgan — DATE/TIME: 10-20-15 0824	
RELINQUISHED BY/REMOVED FROM: Curt Morgan — DATE/TIME: 10-20-15 1009	RECEIVED BY/STORED IN: MF Smoot — DATE/TIME: 10/20/2015 10:05	
RELINQUISHED BY/REMOVED FROM: — DATE/TIME:	RECEIVED BY/STORED IN: — DATE/TIME:	

LABORATORY SECTION	RECEIVED BY	TITLE	DATE/TIME
FINAL SAMPLE DISPOSITION	DISPOSAL METHOD	DISPOSED BY	DATE/TIME

A-6003-618 (REV 2)

6821.22-TI-001 Attachment E page 1 of 2 @ 10/20/15

68122-11-001
Attachment E ⓐ 10/26/15
Pg 2 of 2

RADIOACTIVE SHIPMENT RECORD

3. Page 1 of 1 | 4. Ship Prepaid | 5. Via Motor

1. SHIP FROM U.S. DEPT. OF ENERGY C/O
Company CHPRC
Address K Basin
City, State, Zip Hanford
Contact John Hasson
Phone 373-1431/430-7947

2. SHIP TO ☒ U.S. DEPT. OF ENERGY C/O
Company PNNL
Address 325 Building 300 Area North Door
City, State, Zip Hanford SITE
Attention Sandra Fiskum
Phone 1-509-375-5677

6. SHIPMENT AUTHORIZATION NUMBER RJ005
7. EMERGENCY CONTACT INFO
Telephone 373-3800
ERG #s 163
Charge Code N/A

8. HM UN ID Proper Shipping Name: PRI HAZ SUB HAZ
☐ UN2915 Radioactive Material, Type A package 7 N/A

9. No. Pkg.	Model Package	COC/Spec	Serial No.	Seal No.	Isotopes	C.S.I.	T.I.	Bq/Package	Gr. Wt. Kg.
1	Plastic Box	IHT-623	010042	0001	AM-241, PU-239, PU-240	N/A	.5	1.62e-3 TBq	14

10. Identify for Normal Form Only
Physical Form Liquid
Chemical Form Mixture

11.
☐ Highway Route Controlled Quantity
☐ Exclusive Use Shipment with instructions
☒ Placards Applied Rad 7
☐ Fissile Excepted, Grams
☒ UN ID Marking UN2915

12. LABELS APPLIED
Radioactive Yellow - II

13. ADDITIONAL INFO / LABELS / MARKINGS
PU-238, PU-241. BRM#16736

8. HM UN ID Proper Shipping Name: PRI HAZ SUB HAZ

9. No. Pkg.	Model Package	COC/Spec	Serial No.	Seal No.	Isotopes	C.S.I.	T.I.	Bq/Package	Gr. Wt. Kg.

10. Identify for Normal Form Only
Physical Form
Chemical Form

11.
☐ Highway Route Controlled Quantity
☐ Exclusive Use Shipment with instructions
☐ Placards Applied
☐ Fissile Excepted, Grams
☐ UN ID Marking

12. LABELS APPLIED

13. ADDITIONAL INFO / LABELS / MARKINGS

8. HM UN ID Proper Shipping Name: PRI HAZ SUB HAZ

9. No. Pkg.	Model Package	COC/Spec	Serial No.	Seal No.	Isotopes	C.S.I.	T.I.	Bq/Package	Gr. Wt. Kg.

10. Identify for Normal Form Only
Physical Form
Chemical Form

11.
☐ Highway Route Controlled Quantity
☐ Exclusive Use Shipment with instructions
☐ Placards Applied
☐ Fissile Excepted, Grams
☐ UN ID Marking

12. LABELS APPLIED

13. ADDITIONAL INFO / LABELS / MARKINGS

14. Shipment DE-Ci: 5.762e-6

Shipment Totals	C.S.I.	T.I.	Bq/Package	Gr. Wt. Kg.
	n/a	.5	1.62e-3 TBq	14

15. Surface Dose Rate of Package
☒ <0.005 or _____ mSv/hr
<0.5 or _____ mrem/hr (N+ß γ)

Dose Rate @ 1 Meter from Surface of Package
☒ <0.005 or _____ mSv/hr
<0.5 or _____ mrem/hr (N+ß γ)

Additional Data and Instructions (inc. Readings on Internal Packaging)

Smears of Outer Container
☐ <4.0 Bq (220 dpm) ß γ /cm²
☐ <0.4 Bq (22 dpm) α /cm²
☒ <Thl. 2-2 HNF 5172 Limits

TRUCK LOAD OR EXCLUSIVE USE
Surface ☒ <2 mSv/hr (200 mrem/hr)
@ 2 meters ☒ <0.1 mSv/hr (10 mrem/hr)
@ Cab or sleeper ☒ <0.02 mSv/hr (2 mrem/hr) (Using N+ß γ)

Bldg. 105KW | Survey No. L-150166B | Date 10-20-15

16. TRANSPORTER

Vehicle Number	DRIVER SIGNATURE	PRINT NAME
G43102H	Curt Magn	CURT MORGAN

17. RECEIVER

RECEIVER SIGNATURE	PRINT NAME	Date
Sandra Fiskum	Sandra Fiskum	10/20/15

18. This is to certify that the above-named materials are properly classified, described, packaged, marked and labeled, and are in proper condition for transportation according to the applicable regulations of the Department of Transportation.

Certifier's Signature	Print Name	On behalf of DOE-RL	Date	Organization
John Hasson	John Hasson		10-20-15	CHPRC Shipping

19. AUTHORIZATION FOR SHIPMENT

AIR TRANSPORT CERTIFICATION ☐ N/A

CARGO AIRCRAFT ☐ Cargo Aircraft Only Labels Applied

PASSENGER AIRCRAFT ☐ Ltd Qty ☐ Research/Medical Diagnosis ☐ <3 T.I. ☐ Human Medical Research

Pkg. Dimensions (cm)

20. OFFSITE AUTHORIZATION

Survey No.	Date Shipped	Routing	ETA

Approved for Shipment Offsite | Date

A-6003-214.1 (REV 4)

RPL K Basin Project ~~52578~~ 68122 SKF 10/28/15	Document Review Record
Title:	Completed 68122-TI-001 Sandfilter Backwash Solids Consolidation

Please return the completed form to:		Comments Due:
Organization/Department:	**Designated Reviewer:**	**Reviewer Signature/Date:** (Upon completion of review)
D9H63	SK Fiskum	Sandra K. Fiskum 10/27/15

It is optional, but helpful when comments are identified as to type or if the reviewer wishes to specify mandatory comments requiring resolution. In this case, the following conventions apply:

***Type:** **E** – Editorial, addresses word processing errors that do not adversely impact the integrity of the calculation.
O – Optional, comment resolution would provide clarification, but does not impact the integrity of the work or calculations
M – Mandatory, comment shall be resolved; reviewer identifies impact on the integrity of the work or calculations

Comment Number	Section/ Page	Type	Comment	Comment Resolution
1	Step 3.5.5 and 4.14.5	E	Identify if the gross mass includes the over-package of bags and tape.	Added note that the gross container mass includes the bags and tape used to safely removed the bottle from the CA fume hood and move to laboratory with a balance to accommodate the weight.
2	Step 4.19	E	Annotate where the new dose rates are for the re-mixed and split –A and –D samples are located.	Added note to indicate new dose rates are on page 13.
3	throughout	E	Minor editorial changes (spelling) as identified.	Spelling errors fixed as identified.
END				

Concur with comment resolution, review complete.	Comments Resolved By:
Sandra K. Fiskum 10/28/15	MK Smoot 10/28/15
Reviewer Signature Date	Author Signature Date

Title:	Calculations from 68122-TI-001, *Sandfilter Backwash Solids Consolidatian*
Filename:	68122-TI-001 Data Reduction.xlsx
Revision:	0
Date Prepared:	10/28/2015
Prepared By:	SK Fiskum
Purpose:	The purpose of this spreadsheet is to calculate the net mass and density of centrifuged solids sample splits from KW-105SFBW-001, K West Basin backwash from the sand filter. Data used in this file are taken from raw data recorded in 68122-TI-001.
Approach:	Solids received in a 750-mL volume as a dilute slurry were consolidated into 2 centrifuge tubes. The tubes were 10-mL glass with high fidelity (nearest 0.1 mL) of volume readability. Simple subtraction, summation, and division is used to calculate the total centrifuged solids mass and wt% centrifuged solids in the received slurry.

Parent Container ID	Container Type	Gross Mass with Packaging, g	Packaging Mass, g	Gross Sample Container Mass, g	Empty Mass with Packaging, g	Packaging Mass, g	Empty Sample Container Mass, g	Net Slurry Mass, g	Centifuged Solids, wt%
KW-105SFBW-001	1-L poly bottle	887.0	31.2	855.8	123.1	31.5	91.6	764.2	0.11%

Centrifuged sludge volume

Subsample Container ID	Container Type	Tare, g	Gross Mass, g	Sludge Volume, mL*	Total Slurry Volume, mL	Net Slurry Mass, g	Net Centrifuged Sludge Mass, g*	Centrifuged Sludge Density, g/mL*
68122-TI-001-F	10-mL glass cent tube	20.6859	21.2217	0.40	0.50	0.5358	0.44	1.09**
68122-TI-001-G	10-mL glass cent tube	20.6253	21.1095	0.40	0.50	0.4842	0.38	0.96

Assumption: the water density is 1 g/mL.
*The total sludge volume determination was slightly confounded by the slanted surface of sludge in the centrifuge tube.
**The third significant figure is shown for information only.

Spreadsheet Review Form

Spreadsheet Author Name:	SK Fiskum	Review Date:	10/27/2015
Date Prepared:	10/23/2015	Spreadsheet Subject:	Calculations from 68122-TI-001 Sandfilter Backwash Solids Consolidation
Reviewer Name:	RW Shimskey	File Name:	68122-TI-001 Data Reduction.xlsx
Reviewer Title:	Sr Research Engineer	Revision No.	0

Scope of Spreadsheet Review: (Check one or more of the following)

☐	General Validation Review: (General review and spot checks)	☐	Independent calculation check (With hand calculations or independent spreadsheet software)
☐	Review of updated spreadsheet/calc (Revised portion only)	☐	Other:
☑	100% Verification Review (Verification of all cells/calculations)		

REVIEW CHECK LIST

Spreadsheet/Calculation Identification

Spreadsheet Information	Yes	No	NA
Title:	x		
Revision Number:	x		
Date Prepared:	x		
Prepared by:	x		
General Statement of Purpose:	x		
General Description of Approach:	x		
Comments:			

Input Values	Yes	No	NA
Are input parameters correct (verified with source?)	x		
Are parameter units consistent?	x		
Are input values properly referenced?	x		
Are input uncertainties correct?	x		
Comments: Corrections with formatting and use of significant figures made.			

Assumptions	Yes	No	NA
Are assumptions clearly stated?			x
Are assumptions supported and justified?			x
Are assumptions reasonable?			x
Comments: There are no assumptions with this data set.			

Equations/Approach	Yes	No	NA
Are equation algorithms adequately defined?	x		
Are equations properly referenced?	x		
Are limitations of approach/equations identified?	x		
Are equations appropriate?	x		
Are units consistent?	x		
Comments: Equations are simple add/subtract, multiply and divide.			

Results/Conclusions	Yes	No	NA
Are formulas consistent in cells?	x		
Are calculations correct?	x		
Are conclusions consistent with results?			x
Are conclusions consistent with applicable limits?			x
Comments: There are no conclusions from this data set.			

Reviewer Sign/Date: 10/28/15

PROJECTS 68112– SURVEILLANCE REPORT

Number: SR-**68122-2015-005**

PLANNING

Topic/Issue to be evaluated: Surveillance of Completed Test Instruction 68122-TI-001, *Sandfilter Backwash Solids Consolidation*

This surveillance was undertaken to evaluate completed test instruction (TI) 68122-TI-001 *Sandfilter Backwash Solids Consolidation*. TI-001 was used between 13 October 2015 and 27 October 2015. Margaret Smoot performed the work, Sandy Fiskum performed the technical review of the completed TI and Rick Shimskey performed the technical review of the data reduction and reporting spreadsheet, 68122 TI-001 Data Reduction.xlsx, prepared by Sandy Fiskum.

Source of Requirement(s):

- Baker RB, JL Westcott, TL Welsh, JA Pottmeyer, and AJ Schmidt. 2009. *Quality Assurance Project Plan/Sampling and Analysis Plan for Sludge in the KW Engineered Containers*. KBC-33786, Rev. 2, CH2M Hill Plateau Remediation, Company, Richland, Washington.
- Westcott JL, BJ Makenas, TL Welsh, JA Pottmeyer, and AJ Schmidt. 2009. Data Quality Objectives for Sampling and Analysis of K Basin Sludge (DQO), HNF-36985, Rev. 3, CH2M Hill Plateau Remediation, Company, Richland, Washington.
- Letter 52578-2015-L03 from SK Fiskum to JO Honeyman, *Cost Estimate for Analysis of the Sandfilter Backwash Solids*, May 27, 2015.
- CH2M Hill Plateau Remediation, Contract 49517, Release 35, Statement of Work, *Isotopic Analysis from PNNL* (July 7, 2015), Amendment 1.

Other Applicable Documents:

ADM-RSEG-BALANCES, Rev. 1 (or latest revision), *Balance Calibration Verification*

Test Instruction 68122-TI-001, *Sandfilter Backwash Solids Consolidation*

PERFORMANCE

Date Initiated: 10/28/15	**Location:** RPL/525, 410, and 305
Date Completed: 10/29/15	**DRAFT Report Issued:** 10/29/15 **FINAL Report Issued:** 10/29/15
Contacts: Sandy Fiskum, Rick Shimskey, and Peg Smoot	**Org Code:** PE 137, D9H63
Surveillance Team: Deborah Coffey	

PURPOSE

The purpose of sample collection, processing and analysis is to provide characterization data to allow the client to characterize the sand filter media for disposal. The sand filter is located in the K West Basin.

The K Basin-Project QE was requested to review the completed TI for sample receipt and processing activities leading to submittal of the sample to the Analytical Support Operations (ASO) laboratory for analysis under ASR 9916. The sample was received by M Smoot on 10/20/15 at 10:05 a.m. from the 105KW Basin by CH2MHill CHPRC staff; the sample was collected on 10/19/15 at 1330 (1:30 pm). The sample ID documented on the Chain of Custody form is KW-105 SFBW-001.

APPROACH

Completed TI-001 was submitted for QE review on 10/28/15. There initially was no DRR form from the technical reviewer included in the submitted TI package showing that all technical reviewer comments were resolved; the technical reviewer did sign off on the cover page of the completed TI on 10/28/15. The technical reviewer of the data reduction and reporting spreadsheet, 68122 TI-001 Data Reduction.xlsx, prepared by Sandy Fiskum, Rick Shimskey, signed the Spreadsheet Review Form, which referenced a DRR form as the location of any spreadsheet comments, but that document was not attached to the completed TI. Both the technical reviewer's DRR form and the revised Spreadsheet Review Form were submitted on 10/29/15 to address concerns.

RESULTS

The submitted data package included the following:

- Technical Reviewer's DRR form – 1 pp.
- Completed TI-001, *Sandfilter Backwash Solids Consolidation* – 21 pp.

- Attachment A –
  BPCL forms for:
  Mettler AT400 (SN: N04143) RPL/305 benchtop
  Mettler SB16001 (SN: 1125202532) RPL/305 benchtop
  Mettler AT400 (SN: 11132992667) RPL/410 fumehood
- Attachment B –
  QCS Certificate of calibration for:
  Mettler AT400 (SN: N04143) RPL/305 benchtop. Cal Date: 8/24/15 Expires: 8/2016
  Mettler SB16001 (SN: 1125202532) RPL/305 benchtop. Cal Date: 2/11/15 Expires: 2/2016
  Mettler AT400 (SN: 11132992667) RPL/410 fumehood. Cal Date: 8/18/15 Expires: 8/2016

- Attachment C
  Photographs – 2pp.
- Attachment D-
  Email confirming that the client did not want/need to observe work in progress – 2 pp.
- Attachment E –
  Chain of Custody/Analysis Request form for the sample that was received by M Smoot on 10/20/15 at 10:05 a.m. from the 105KW Basin by CH2MHill CHPRC staff; the sample was collected on 10/19/15 at 1330 (1:30 pm). The sample ID documented on the Chain of Custody form is KW-105 SFBW-001. 1 pp.
  Radioactive Shipment Record dated 10/20/15 documenting dose rates and smears of the shipped container. 1 pp.
- Data reduction and reporting spreadsheet, 68122-TI-001 Data Reduction.xlsx, prepared by Sandy Fiskum and titled, Calculations from 68122-TI-001, *Sandfilter Backwash Solids Consolidation.*

M&TE Used:

Balance Identification:	**Used:**
Mettler AT400, serial number: N04143 (360-06-01-048), RPL/305	10/15/15
Mettler SB16001 (SN: 1125202532) RPL/305 benchtop	10/21/13; 10/22/15
Mettler AT400 (SN: 11132992667) RPL/410 fumehood	10/23/15

Dates and documentation of performance check measures on the noted dates were consistent with the BPCL forms.

Sources of data entered into the data reduction and reporting spreadsheet, 68122TI-001 Data Reduction.xlsx were consistent with TI entries and traceable.

Issues of Concern:

There were no issues of concern, findings or observations identified.

Findings are defined as a statement of fact relating to noncompliance with previously agreed-upon procedures, plans, codes, standards, specifications, or other forms of contractual or legal obligation. It should be understood that any lack of a finding in a specific area is not considered an indication that deficiencies do not exist.

Observations are defined as a weakness that if not corrected, could yield a departure from a requirement although the weakness is not necessarily a departure from requirements.

PROJECTS 68112– SURVEILLANCE REPORT

Number: SR-**68122-2015-005** Page 4 of 4

There were no findings or observations. There were no issues identified that would result in a Project 68122 Occurrence, Deficiency, or Nonconformance Report and no required corrective actions. It appears that the TI objectives were met.

CORRECTIVE ACTION

[X] None Required. [ ] Complete [ ] Follow- up Corrective Action:

Surveillance Performed By:

Deborah Coffey, Quality Engineer Date: 10-29-2015

DISTRIBUTION: Deborah Coffey, Sandy Fiskum, Andy Schmidt, Rick Shimskey and Peg Smoot

Appendix B

ASR 9916 and Results

Appendix B

ASR 9916 and Results

Appendix B contains the signed Analytical Services Request (ASR) 9916 and Special Instructions generated for the Analytical Support Organization (ASO) analysis of the backwash sand filter solids sample and duplicate. The Special Instructions delineate sample-specific information, required detection limits, quality control (QC) sample requirements, and reporting requirements.

Analytical summary reports generated by the ASO in response this ASR are also provided in Appendix B. The analytical reports provide sample results associated uncertainties, and a discussion of sample processing, QC results, and data limitations.

Appendix B Table of Contents

ASO Analytical Service Request Form

Analytical Service Request (ASR)

(Information on this COVER PAGE is applicable to all samples submitted under this ASR)

Requestor --- Complete all fields on this COVER PAGE, unless specified as optional or ASR is a revision

Requestor:
Signature: Sandra K Fiskum
Print Name: SANDRA FISKUM
Phone: 375-5677 MSIN: P7-25

Project Number: 68122
Work Package: N51383

Matrix Type Information

- ♦ **Liquids:** ☐ Aqueous ☐ Organic ☐ Multi-phase
- ♦ **Solids:** ☐ Soil ☑ Sludge ☐ Sediment ☐ Glass ☐ Filter ☐ Metal ☐ Smear ☐ Organic ☐ Other
- ♦ **Other:** ☐ Solid/Liquid Mixture, Slurry ☐ Gas ☐ Biological Specimen

(If sample matrices vary, specify on Request Page)

QA/Special Requirements

- ♦ **QA Plan:**
  - ☒ ASO-QAP-001 (Equivalent to HASQARD)
  - ☐ Additional QA Requirements, List Document Below:
    Reference Doc Number: _____
- ♦ **Field COC Submitted?** ☒ No ☐ Yes
- ♦ **Lab COC Required?** ☒ No ☐ Yes
- ♦ **Sample/Container Inspection Documentation Required?** ☒ No ☐ Yes
- ♦ **Hold Time:** ☒ No ☐ *Yes*
  If *Yes*, Contact ASO Lead before submitting Samples
  ☐ Use SW 846 (PNL-ASO-071, identify analytes/methods where holding times apply)
  ☐ Other? Specify: _____
- ♦ **Special Storage Requirements:** ☒ None ☐ Refrigerate ☐ Other, Specify: _____
- ♦ **Data Requires ASO Quality Engineer Review?** ☐ No ☒ Yes

Disposal Information

- ♦ **Disposition of Virgin Samples:**
  Virgin samples are returned to requestor unless archiving provisions are made with receiving group!
  If archiving, provide:
  Archiving Reference Doc: _____
- ♦ **Disposition of Treated Samples:** ☒ Dispose ☐ Return

Data Reporting Information

- ♦ **Is Work Associated with a Fee-Based Milestone?** ☒ No ☐ Yes
  If yes, milestone due date: _____
- ♦ **Preliminary Results Requested, As Available?** ☐ No ☐ Yes
- ♦ **Data Reporting Level**
  ☐ ASO-QAP-001 (Equivalent to HASQARD).
  ☐ Minimum data report.
  ☒ Project Specific Requirements: Contact ASO Lead or List Reference Document: see attached
- ♦ **Requested Analytical Work Completion Date:** _____
  (Note: Priority rate charge for < 10 business day turn-around time)
- ♦ **Negotiated Commitment Date:** 12/15/15
  (*To be completed by ASO Lead*)

Waste Designation Information

- ♦ **ASO Sample Information Check List Attached?** ☐ No ☑ Yes
  If no, Reference Doc Attached: _____
  or, Previous ASR Number: _____
  or, Previous RPL Number: _____

Does the Waste Designation Documentation Indicate Presence of PCBs?
☒ **No** ☐ **Yes**

Send Report To: SK Fiskum **MSIN** _____
MSIN _____

Additional or Special Instructions _____

Receiving and Login Information (*to be completed by ASO staff*)

Date Delivered: 10/27/15
Delivered By (optional): M. Smoot
Time Delivered (optional): _____
Group ID (optional): _____
CMC Waste Sample? ☒ No ☐ Yes

Received By: L. Darnell
ASR Number: 9916 Rev.: 00
RPL Numbers: 16-0032 (first and last)

ASO Work Accepted By: KN Pool **Signature/Date:** Kal N Pool 10/27/15

Analytical Services Request (ASR)

(REQUEST PAGE ---- Information Specific to Individual Samples)

ASO Staff Use Only **RPL Number**	Client Sample ID	Sample Description (& Matrix if varies)	Analyses Requested	ASO Staff Use Only **Test**	ASO Staff Use Only **Library**
		Provide Analytes of Interest and Required Detection limits - ☐ **Below** ☐ **Attached**			
16-0032	**68122-TI-001-F**	**16-0032 Sample (Sample consists of wet solids collected in a centrifuge cone)**	1) Dry solids to constant Mass 2) Digest - 129 (Prep Lab) a) GEA b) Pu - Alpha AEA c) Np - Alpha AEA d) Am - Alpha AEA e) Sr-90 f) TIMS Prep for Pu isotopics 3) Residual Soilids - Dry to Constant Mass a) GEA		
16-0032 (dup)	**68122-TI-001-G**	**16-0032 Duplicate (Sample consists of wet solids collected in a centrifuge cone)**			
	Note: This ASR has very specific special instructions that should be read and understood before performing work on these samples. If there any questions on what you should be doing, contact the ASO Lead and or ASO QE before proceeding.				

ASO Sample Information Checklist (SICL) Form

Sample Location, Owner and Hazard Description Information
To be completed by the sample custodian relinquishing the sample(s) and based on best available information.

ASO Customer Information:

Company: PNNL
Project #: 68122
Point of Contact (name. telephone#): SK Fiskum, 375-5677

Comments:
Sample is consolidated solids from the K East sand filter backwash

Sample Description (medium, collection location, known contaminants, purpose of sample collection):

Sample Collection Date: 10/19/15 Sample Collection Time: 13:30
Sample collected at the K East Basin.

Is the sample known to be radioactive? ☒ Yes ☐ No

Comments *(list known isotopes)*:
Cs-137, Sr-90, others to be determined by ASO.

Is the sample known to contain or have come in contact with PCBs? ☐ Yes ☒ No
List any hazardous sample constituents known to be present:

Constituent/Chemical	Concentration	Comment
unknown		

Are any other comments applicable to sample receipt, storage, handling, or disposition?

Wait for SK Fiskum to authorize disposal of acid digests.

Checklist Prepared By:
SK Fiskum Sandra K Fiskum 10/27/15
Printed Name Signature Date

Special Instructions for K-Basin Sandfilter Backwash Samples

Cognizant Scientist: SK. Fiskum Date: 10/27/15
SK Fiskum

Project Manager: SK. Fiskum Date: 10/27/15
SK Fiskum

Project Quality Engineer: Deborah Coffey Date: 10-27-15
DS Coffey

ASO Lead: Kurt N. Pool Date: 10/27/15
KN Pool

STP Concurrence: Michael E. Johnson Date: 10/27/2015
for GM Davis

Special Instructions for K-Basin Sandfilter Backwash Samples

Cognizant Scientist:
SK Fiskum, 375-5677

A sample was collected by CHPRC staff from the sandfilter backwash processing at the K West (KW) Basin. The sample was received by the project (68122) staff from CHPRC under chain of custody at the RPL on 10/20/15. The material was received in the RPL/525 laboratory and logged into the Radioactive Material Tracking (RMT) system as RMT #86040.

The 68122 project staff consolidated the solids from this sample into two equal fractions, each at 0.4 mL with a small overburden of backwash water in 10-mL glass centrifuge tubes. These duplicate samples are presented to the Analytical Support Operations (ASO) laboratory identified as a sample and duplicate for analysis. The sample IDs and container tare masses are shown in Table 1. There is no other sample material available so it is very important to handle the samples carefully.

Table 1. Cross-Reference of Sample ID, RPL ID, Centrifuge Cone Tare

Sample ID	RPL ID	Container Tare, g	Sample Type
68122-TI-001-F	16-0032	20.6859	Sample
68122-TI-001-G	16-0032	20.6253	Sample duplicate
Container tares were collected from balance 113061397, lab 525 hood 13 on 10/23/15.			

The requested sample processing and analysis schematic is shown in Figure 1. Work is to be performed under the ASO QA Plan. All analysis work is to be charged to N51383.

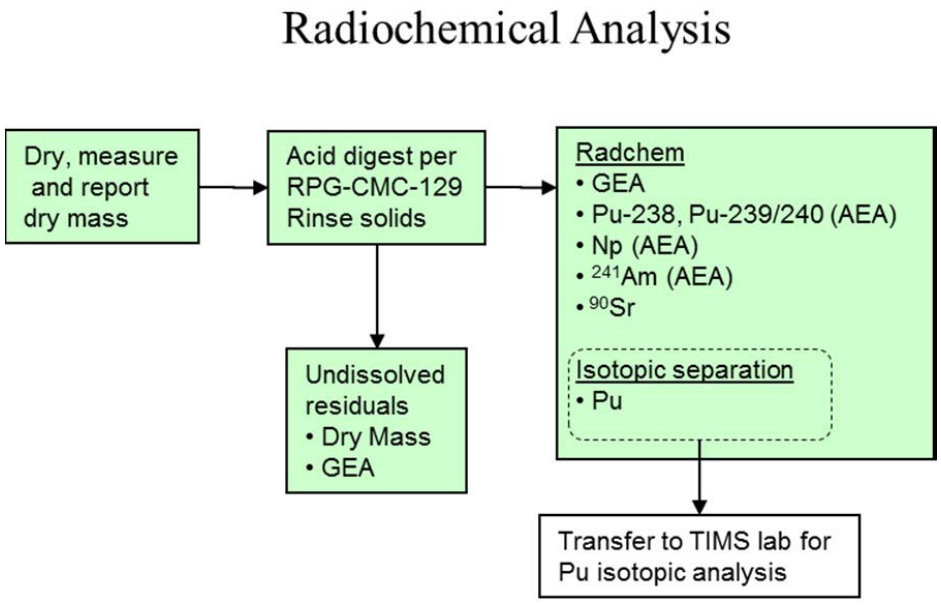


Figure 1. Analysis Schematic for K Basin Backwash Solids

Analytes of interest and required detection limits are provided in Table 3; quality control (QC) sample acceptance criteria are provided in Table 4.

Acid Digestion

The analytical samples are provided to ASO in tared centrifuge tubes. Analytical samples need to be dried to constant mass at 105°C using procedure, RPG-CMC-503, Rev. 0, *Determination of Physical Properties of Solutions, Sludges, Slurries and Solids*. Determine **and report** the net dry sample mass.

Acid digest the dried solids according to RPG-CMC-129, Rev. 0, *HNO_3-HCl Acid Extraction of Solids Using a Dry-Block Heater*. Report observations associated with the dissolution processing, including the undissolved solids mass.

Residual solids remaining after acid digestions are not expected. If present, they are of interest and shall not be lost (refer to Figure 1). Remove the acid digestate as per procedure; this may entail centrifuging and decanting. Rinse as appropriate, collecting rinse solutions with the acid digestion solution.

After residual solids are rinsed and isolated, dry the solids (in the digestion tube) to constant mass at 105°C. Calculate the net residual solids mass along with the starting material dry solids mass and report to SK Fiskum. Additional processing of residual solids will depend on the mass relative to the starting (wet/dewatered) material. Additional processing may include GEA.

Acid Digestion Reporting Units

Report the dry sample mass before digestion as g_{dry}, and report the dried residuals solids mass after digestion and rinsing as $g_{residual}$.

Radiochemistry

Preparations and Analysis

Acid digestate aliquots shall be directly aliquoted for GEA analysis. Acid digestate aliquots shall be aliquoted for separations, mounting, and/or analysis. Operations will be conducted according to the specific radiochemistry procedures delineated in Table 2. No procedure or procedure revision substitutions are to be made without prior written authorization from SK Fiskum or CD Carlson to requests submitted by the ASO Lead.

Table 2. Authorized Radiochemistry Procedures for K Basin Sludge Analysis

Analyte	Separation	Mounting	Counting/Analysis
GEA	NA	NA	RPG-CMC-450, Rev. 2 *Gamma Energy Analysis (GEA) and Low-Energy Photon Spectrometry (LEPS)*
^{238}Pu and $^{239+240}$Pu/AEA	RPG-CMC-4017, Rev. 0, *Analysis of Environmental Water Samples for Actinides and Strontium-90*	RPG-CMC-496, Rev.1, *Coprecipitation Mounting of Actinides for Alpha Spectroscopy*	RPG-CMC-422, Rev. 2 *Solutions Analysis: Alpha Spectrometry*

Analyte	Separation	Mounting	Counting/Analysis
^{241}Am/AEA	RPG-CMC-4017, Rev. 0, *Analysis of Environmental Water Samples for Actinides and Strontium-90*	RPG-CMC-496, Rev. 1, *Coprecipitation Mounting of Actinides for Alpha Spectroscopy*	RPG-CMC-422, Rev. 2 *Solutions Analysis: Alpha Spectrometry*
^{237}Np/AEA	RPG-CMC-4017, Rev. 0, *Analysis of Environmental Water Samples for Actinides and Strontium-90*	RPG-CMC-496, Rev. 1, *Coprecipitation Mounting of Actinides for Alpha Spectroscopy*	RPG-CMC-422, Rev. 2 *Solutions Analysis: Alpha Spectrometry*
^{90}Sr	RPG-CMC-476, Rev. 0, *Strontium Separation using Eichrom Strontium Resin*	NA	RPG-CMC-474, Rev. 1, *Measurement of Alpha and Beta Activity by Liquid Scintillation Spectrometry*
Pu isotopic	RPG-CMC-455, Rev. 0, *Separation of Uranium and Plutonium for Isotopic Analysis by Mass Spectroscopy*	NA	Deliver to Pu Isotopic Analysis Workstation RPL-TIMS-001, Rev. 0, *Thermal Ionization Mass Spectrometry (TIMS)*

The separations conducted to support Pu isotopic analysis need to provide purified fractions with sufficient analyte concentrations to create a measureable signal by TIMS. Therefore, the preparation shall not be considered complete until satisfactory isotopic results are obtained. It is understood that the process/preparation blank (PB) may not have sufficient analyte concentration to meet this objective. There are no laboratory control samples (LCS) or matrix spike (MS) samples required to be processed through separations processes to support isotopic analysis by TIMS.

Radiochemistry Reporting Units

Report solids sample analyte concentrations as $\mu Ci/g_{dry}$ where g_{dry} is the initial dry sample mass determined prior to the start of the PNL-ALO-129 acid digestion.

If needed, report the residual solids GEA analytes as $\mu Ci/g_{residual}$, where $g_{residual}$ is the mass of residual solids that did not dissolve during acid digestion. The precision criteria defined in Table 4 do not apply to the undissolved solids.

Quality Control

All work is required to be conducted to the requirements of the Hanford Analytical Services Quality Assurance Requirements Document (HASQARD, Rev. 3). Performing work to the requirements of the ASO QA Plan (ASO-QAP-001, Rev. 9 or most current revision) will satisfy the HASQARD requirements. Where there is a discrepancy between the data quality objectives (DQOs) in the ASO QA Plan and these Special Instructions, the Special Instructions takes precedence as there are specific K Basin project requirements.

Preparative quality control (QC) sample analysis is to include a preparation blank (PB) sample, a sample duplicate (provided), an LCS or blank spike (BS) sample and a sample MS (a separate sample is provided for spiking by the ASO) as indicated in Table 4. The BS sample for radioisotopes will be generated after acid digestion on a sample split, i.e., post-digestion spikes. The MS for radioisotopes will be generated after acid digestion on a sample split, i.e., post-digestion spikes.

The duplicate, LCS/BS, and MS sample QC acceptance criteria are provided in Table 4. The following section discusses actions to take in the event of QC sample failures.

Note: The ASO staff must inform/report QC sample failures promptly to the ASO Lead who will in turn inform the Cognizant Scientist/Project Manager with copy to the K Basin Project QE (Deborah Coffey), thus enabling Pacific Northwest National Laboratory (PNNL) to fulfill its commitment to the client for prompt notification of such failures. The impact of any QC sample failure on data quality or project schedule (e.g., re-runs) will be assessed in conjunction with the client.

1. The preparation blank (PB) analyte concentration shall be less than the estimated quantitation limit (EQL) or the minimum detectable activity (MDA) of the associated sample. When the PB concentration is equal to or exceeds the EQL or MDA, then the PB concentration shall not exceed 5% of the measured concentration present in the sample.
2. If and when it has been determined that the data are not useable, then the 68122 Project Manager (PM) must be notified promptly.
3. If and when the MS sample fails to meet the acceptance criteria, the results shall be investigated for potential sources of error by comparing the results of the MS to the BS. When the sources of error cannot be identified, the failure of the MS will be attributed to a sample matrix effect and any resulting limitations on the data shall be included in the report.

Reporting

The analytical data report shall be prepared in accordance with procedure PNL-ASO-058, Rev. 1, *ASO Data Reporting*, Section 5.3, Comprehensive Data Report. Please be sure to include action taken with respect to any identified unexpected results and discrepancies.

The Comprehensive Data Report contains three main parts; a data report **cover page**, a **narrative**, and the **data summary**. These elements are identified below; red text indicates project-driven scope. Black text is taken directly from the ASO procedure: PNL-ASO-058, Rev. 1

Data Report Cover Page Contents:

- Header identifying name/address of laboratory
- Customer Name:
- Project #/WP #(s):
- ASR #(s): (ASR forms included date and time of sample receipt)
- Total # Samples:
- Report Date:
- Report Revision Date: when applicable
- Filename(s): where data files can be accessed
- Dates of Sample Processing and Analysis:
- Procedure(s): Number, revision, and title (both sample processing and analysis)
- M&TE used: (manufacturer/model) including instrument software (name, version and date), and serial number or property number) as applicable
- Reference Date (when applicable):
- Customer Sample ID and ASO/RPL ID Sample Numbers:
- Report Review and Approval:

_____ _____

Preparer Date

(Printed name and signature of responsible individual)

Technical Reviewer Date
(Printed name and signature of technical reviewer)

A case narrative describing the analysis, limitations on the analysis results, and QA/QC issues

Narrative Contents:

- Sample Description and Analyses
- Sample dissolution/preparation dates
- Sample Preparation (prior to receipt)
- Method-specific Sample preparation, Separation, Mounting and Counting or Analysis Methods as applicable
- Analysis dates
- Deviations from the written procedure, if any
- Reporting Basis/Units
- QC Criteria/Results
- Limitations of the Data
  - Interferences/Resolution
  - Uncertainty
- Comments

Data Summary Contents:

- Customer ID
- ASO/RPL Sample ID
- Analytes of Interest as per ASR form; opportunistic analyses as applicable with reporting units
- IDL, MDL, EQL or MDA as applicable
- Results and uncertainties
- QC results as applicable
- Reference date as applicable
- Additional information as needed – COC forms, Special Instructions, SOW

The following elements may be included in the final report or be traceable to the test results (usually by annotation on bench sheets) and be maintained as ASO records:

- identification of standards used
- identification of M&TE used
- records of daily check weight tracking
- signature and date of person who performed the test and recorded the data
- analytical bench sheets
- hand calculation review documentation.

Technical reviewers are required to be staff other than the analyst performing the work and knowledgeable of the area being reviewed. The technical review shall be completed and all reviewer issues resolved before reporting final results. Technical reviewers must receive a complete data package including narratives for review. A technical review consists of the following elements:

- evaluation of method and QC performance

- evaluation of compliance with technical (including procedural) and QC requirements, as defined in the ASR
- verification of transcription accuracy
- check for correctness of calculations
- assuring the correct reporting units (and reference dates) are used
- evaluation of overall consistency and reasonableness of data
- evidence of implementation of appropriate corrective action, when necessary.

Analytical results shall be reported both in hard copy **and electronically** (including excel data files to support accurate data transcription into project records). Appropriately marked "Preliminary" data reports and electronic files shall be provided by the ASO Lead as soon as practical after completion of analysis. The final ASR data report shall be provided no later than the commitment date on the ASR.

Table 3. Method Detection Limits for Solids

Analyte	Solids	Analysis Method
	$\mu Ci/g_{(dry)}$	
^{134}Cs	2.0E-01	GEA (Reference date = date and time of the first sample analysis measured by GEA.)
^{137}Cs	1.5E-02	
^{60}Co	1.0E-02	
^{152}Eu	1.5E+00	
^{154}Eu	1.5E+00	
^{155}Eu	2.0E+00	
^{241}Am	1.5E+01	
^{241}Am	1.0E-03	Separation and AEA
^{238}Pu	1.0E-03	
$^{239+240}Pu$	1.0E-03	
^{237}Np	1.0E-03	
^{90}Sr	1.0E+00	Separation and proportional counting
Note: The detection limits (DL) shown in this table are based on dry mass, whereas the DL in the Quality Assurance Project Plan/Sampling and Analysis Plan (QAPjP/SAP, KBC-33786, Rev. 2) are based on settled solids (wet) mass basis. These DL values are therefore, tighter than those in the QAPjP/SAP.		

Table 4. Analytical Quality Control Acceptance Criteria

Analyte	Analytical Technique	LCS or BS % Recovery[(a)]	Matrix Spike % Recovery[(b)]	Duplicate RPD[(c)]
		Post-digestion spike		
Pu isotopes (spike material is ^{239}Pu)	AEA	80 - 120%	75 - 125%	≤20%
^{241}Am	AEA	80 - 120%	75 - 125%	≤20%
^{237}Np	AEA	80 - 120%	75 - 125%	≤20%
^{90}Sr	Proportional counting	75 - 125%	75 - 125%	≤20%
As identified in Table 3	GEA	Counter control source is used [(d)]	N/A	≤20%

N/A – not applicable

Footnotes:

(a) LCS = Laboratory Control Standard; BS = Blank Spike. A laboratory control sample (LCS) or blank spike (BS) samples are used to monitor the effectiveness of the sample preparation process and are good indicators of method accuracy. Ideally, the LCS is a material similar to the sample being processed, containing the analytes of interest (e.g., standard reference material). An LCS, if available, shall be prepared with each batch of samples processed at the same time. When an appropriate LCS is not available, a BS shall be used in lieu of the LCS. A BS is distilled or deionized water or another suitable matrix spiked with the analytes of interest. It may not be possible to prepare a single BS that contains all analytes of interest (e.g., chemical incompatibility). In such cases, an agreement with the client shall be made to identify the analytes of interest used to prepare the BS, and more than one BS may be used. The BS and AS results are expressed as percent recovery; i.e., the amount measured, divided by the known concentration, multiplied by 100.

(b) MS = matrix spike; AS = Analytical Spike. For some methods, the sample accuracy is expressed as the percent recovery of a matrix spike (MS) or analytical spike (AS) sample. Post-digestion spikes and analytical spikes are also included under these acceptance criteria. The spiked sample result is expressed as percent recovery; i.e., the amount measured less the amount in the sample, divided by the spike added, times 100. One MS (or post spike or analytical spike [AS]) is performed per analytical batch. Samples are batched with similar matrices. For ICP analytes, the accuracy can also be determined based on use of serial dilutions. In cases where the ICP-OES MS concentration is low relative to the sample analyte concentration (resulting in meaningless recovery calculations), matrix effects will be evaluated from a post-digestion spike.

(c) RPD = Relative Percent Difference between the samples. Sample precision is estimated by analyzing replicates taken separately through preparation and analysis.

(d) Per RPG-CMC-450, Rev. 2, a counter control source is checked daily and must be within ±3 sigma or ±3%, whichever is greater, of the control value.

References

68122-TI-001, Rev. 0, *Sandfilter Backwash Solids Consolidation*

ASO-QAP-001, Rev. 9 or current revision, *Analytical Support Operations Quality Assurance Plan*

PNL-ASO-052, Rev. 2, *Balance Performance Checks*

PNL-ASO-058, Rev. 1, *ASO Data Reporting*

RPG-CMC-129, Rev. 0, *HNO_3-HCl Acid Extraction of Solids Using a Dry-Block Heater*

RPG-CMC-422, Rev. 2, *Solutions Analysis: Alpha Spectrometry*

RPG-CMC-450, Rev. 2, *Gamma Energy Analysis (GEA) and Low-Energy Photon Spectrometry (LEPS)*

RPG-CMC-474, Rev. 1, *Measurement of Alpha and Beta Activity by Liquid Scintillation Spectrometry*

RPG-CMC-476, Rev. 0, *Strontium Separation using Eichrom Strontium Resin*

RPG-CMC-496, Rev. 1, *Coprecipitation Mounting of Actinides for Alpha Spectroscopy*

RPG-CMC-4017, Rev. 0, *Analysis of Environmental Water Samples for Actinides and Strontium-90*

Hanford Analytical Services Quality Assurance Requirements Document (HASQARD), 2007, DOE/RL-96-68, Revision 3, Department of Energy Richland Office, Richland Washington.

Hanford Analytical Services Quality Assurance Requirements Document (HASQARD), September 2014, DOE/RL-96-68, Revision 4, Department of Energy Richland Office, Richland Washington.

QAPjP/SAP, KBC-33786, Rev. 2. 2009. *Quality Assurance Project Plan/Sampling and Analysis Plan for Sludge in the KW Engineered Containers.*

Battelle PNNL/RPL/ASO Mass Measurement Analysis Report
P.O. Box 999, 902 Battelle Blvd., Richland, Washington 99352

Mass Measurements

Project / WP#:	68122/N51383
ASR#:	9916
Client:	S. Fiskum
Total # of Samples:	2

Client Sample ID	RPL Number
68122-TI-001-F	16-0032
68122-TI-001-G	16-0032 (dup)

Analysis Type:	Mass measurements using analytical balances
Sample Processing	☐ None ☒ Digested as per PNL-ALO-129, Rev. 0, *HNO_3-HCl Acid Extraction of Solids Using a Dry Block Heater, 4-20-11 (J. Chenault)* ☐ Fusion as per PNL-ALO-115, *Solubilization of Metals from Solids Using a KOH-KNO_3 Fusion* ☐ Other:
Pre-dilution Prior to Radiochemical Processing?	☒ No ☐ Yes -- *example 2 mL to 100 mL; 50x dilution*
Analysis Procedures:	RPG-CMC-503, Rev. 0, *Determination of Physical Properties of Solutions, Sludges, Slurries and Solids* PNL-ASO-052, Rev. 1, *Balance Performance Checks*
Analysis Date or Date Range:	11/13/15 thru 12/3/15
Technician/Analyst:	L. Darnell and K. Carson
Analysis Data (File):	ASR 9916 dried solids memo.doc
CMC Project 98620 File:	File Plan 5871: T 68122 9916: Sample preparation and analysis records;
M&TE Number(s):	360-06-01-037 – Lab 525

Preparer / 1/29/16 Date

Lori L. Darnell / 1-29-16
Reviewer Date

MEMORANDUM

Pacific Northwest NATIONAL LABORATORY | Battelle The Business of Innovation

Date:	**December 21, 2015**	Project No.:	**68122**
To:	**Sandy Fiskum**		
From:	**Karl Pool**	Internal Distribution:	**File/LB**
Subject:	**Initial dried solids and residual dried solids masses for ASR 9916**		

Prior to ASO sample digestion, the slurry samples for ASR 9916 were dried to constant weight in pre-tared 15 mL glass centrifuge tubes in lab 525 using a drying oven. The dry masses of the solids prior to digestion are presented in the Table 1. The dried solids were then acid digested using ASO procedure PNL-ALO-129, "HNO_3-HCl Acid Extraction of Solids Using a Dry-Block Heater". The mixed acid digestion did not completely dissolve the solids. An additional 5 mL of concentrated nitric acid was added to try and dissolve the residual. At the completion of the acid digestion, significant residual solids remained that were brown and flocculent. The acid digestions were then centrifuged , the liquid removed and solids rinsed several times with 0.5M HNO_3 The rinses were collected and combined with the initial acid digestate. The solids were then transferred to a glass vial for drying. The liquid fraction was left standing for several days before bringing the solution to final volume (100 mL). Prior to dilution to final volume, the solutions contained a slight wispy fine precipitate that appeared to dissolve upon final dilution. The diluted samples were left to stand overnight to verify no further precipitate developed.

The residual solids were dried in the lab 525 drying oven. The mass of the residual is very small in comparison to the vial mass the solids are dried in. The reported residual solid weights are the average of the two dry weights obtained on different days. The masses are included in Table 1.

Table 1 – Dried solids masses

RPL ID	Client ID	Initial dried solids mass prior to digestion (grams)	Final dried solids mass remaining after digestion (grams)
16-0032	**68122-TI-001-F**	**0.0545**	**0.0039**
16-0032 (dup)	**68122-TI-001-G**	**0.0568**	**0.0039**

E54-1900-001 (8/98)

Battelle PNNL/RPL/ASO Radiochemistry Analysis Report
P.O. Box 999, 902 Battelle Blvd., Richland, Washington 99352

Gamma Energy Analysis (GEA)

Project / WP#: 68122/N51383
ASR#: 9916.00
Client: SK Fiskum
Total Samples: 2

RPL ID	Client Sample ID
16-0032	68122-TI-001-F
16-0032 DUP	68122-TI-001-G

Analysis Type:	GEA- for all positively measured or non-detected isotopes
Sample Processing Prior to Radiochemical Processing/Analysis	☐ None ☒ Digested as per RPG-CMC-129, Rev. 0 *HNO_3-HCl Acid Extraction of Solids Using a Dry Block Heater*, Analyst: LP Darnell (11/18/15) & KJ Carson (11/24/15) ☐ Fusion as per RPG-CMC-115, *Solubilization of Metals from Solids Using a KOH-KNO_3 Fusion* ☐ Other: Preparation may have also involved attaining a GEA geometry that is compatible with the calibration geometry.
Analysis Procedure:	RPG-CMC-450, Rev. 2 *Gamma Energy Analysis (GEA) and Low-Energy Photon Spectrometry (LEPS)*
Reference Date:	November 24, 2015
Analysis Date or Date Range:	November 24, 2015 and December 4, 2015
Technician/Analyst:	KJ Carson, T Trang-Le, L. Darnell
Rad Chem Electronic Data File:	*RPG-RC\16-0032 Fiskum.xlsx*
ASO Project 98620 File:	*File Plan 5871, T4.4 Technical (Radiochemistry), Gamma Calibration, daily checks, and maintenance records; and T3 standard certificates and preparation. Also, balance calibration and performance check records.*
M&TE Number(s):	*Detectors C, G, M and N (M & TE unique identifiers in attached supporting documents)*

T Trang-Le / 1/4/16
Prepare **Date**

J R Greenwood / 1/4/16
Reviewer **Date**

SAMPLE RESULTS

Activities for all gamma emitters detected in these samples are presented in an attached Excel spreadsheet for ASR 9916.00. All sludge data are reported in units of $\mu Ci/g_{dry}$ and all residual solids are reported as $\mu Ci/g_{residual}$ with estimates of the total propagated uncertainty reported at the 1-sigma level. The K Basin ASR specifies that the residual solids GEA analytes are to be reported as $\mu Ci/g_{residual,}$ where g residual is the mass of dried residual solids that did not dissolve during acid digestion.

ASO Project File, ASR-9916 has been created for this report including all appropriate supporting records which may include the Pipette Performance Check Sheet, Standard Certificates, Laboratory Bench Sheets and Gamma Energy Analysis printouts. Detector calibration records, control charts and balance calibration records can be found in the ASO Records.

Sample Preparation, Separation, Mounting and Counting Methods

K-Basin back wash filter samples were prepared in lab 525 under Analytical Service Request (ASR) 9916 for radioanalytical analyses.

ASO received two samples (sample and duplicate) for the project, each sample in a 15 mL glass centrifuge cone. The solids were dried to constant weight, then quantitatively transferred to a plastic digestion tube where the dried solids were leached with hot dilute nitric and hydrochloric acids per procedure RPG-CMC-129. The leachate solution was used for radioanalytical analyses, only GEA data are included in this report. At the completion of the acid digestion, residual unsolubilized material remained. The residual solids were transferred to a clean 22 mL glass scintillation vial, dried to constant weight and submitted for GEA analyses. The residual solids do have measurable activity and those results are included in this report.

Quality control (QC) samples prepared include duplicates of all samples as well as a Reagent blank. The quality control (QC) sample results for each of the isotopes measured above background have been evaluated and are discussed below. A summary of the GEA analysis results, including QC sample performance, is given in the attached Data Report.

The following is a list of QC sample results relative to both the K Basin data quality objectives and the ASO Quality Assurance (QA) Plan (ASO-QAP-001, Rev. 9); in many cases the DQOs are the same. In cases where there are differences in the DQOs, the K Basin DQO takes precedence for data reporting.

QUALITY CONTROL RESULTS

Tracer:

Tracers are not used for ASO GEA methods.

Process Blank (PB):

The ASO-QAP-001 requires the activities of the gamma emitting isotopes of interest measured in the PB to be within the acceptance criteria of less than 5% of the sample isotope concentration or less than sample minimum detectable activity (MDA) – (See comments) For isotopes of interest that were not detected in the PB and samples, the PB minimum detectable concentration (MDC) was less than the MDC measured in the samples. The isotopes of interest that were detected in the samples above the MDC are all greater than 5% of the activity level present in the PB.

Required Detection Limits

The ASR required detection limits were met for all analytes for both the digestate and residual solid samples. The GEA detection limit requirements are shown on the summary data sheets.

Blank Spike (BS)/Laboratory Control Sample (LCS)/ Matrix Spike (MS):

There are no BS, LCS or MS samples analyzed for ASO GEA analyses. Instrument performance is assessed by the analyses of daily control counts and weekly background counts, as discussed below.

Duplicate Relative Percent Difference (RPD):

Duplicate results are required to agree within <20% relative percent difference, taking into consideration the statistical uncertainties in the data. The statistical uncertainties in the data are used to calculate the 2-sigma uncertainty (2s% value listed on the report). RPD values below this 2-sigma value are acceptable since the duplicate results agree within the measured 1-sigma uncertainties. Duplicate results for the sample ranged from 5% to 21%. Eu-152 has a duplicate RPD of 21.4% which exceeds the acceptance criteria of <20% RPD. However, this is acceptable since the RPD is less than the 2-sigma value of 34.0%. Residual solids duplicate RPD range from 5% to 36%. The RPD for Sb-125 is 36.3%; however, the 2-sigma value is 42.9%. Am-241 has a duplicate RPD of 34.5% which exceeds the acceptance criteria of <20% RPD. In this case the 2-sigma value is 5.1% indicating that the Am-241 duplicate measurements exceed the measured 2-sigma statistical difference.

Replicate Relative Standard Deviation (RSD):

Replicate results are required to agree within <20% relative percent difference or within 2-sigma standard deviation taking into account the uncertainties for each measurement.

Instrument Calibration and Quality Control

Gamma detectors are calibrated using multi-isotope standards that are NIST-traceable and prepared in the identical counting geometry to samples for all detectors. Counter control sources containing Am-241, Cs-137 and Co-60 are then analyzed daily before the use of each detector. There is a K Basin DQO that is referenced to ASO procedure RPG-CMC-450, that requires that a counter control source is checked daily and must be within ±3 sigma or ±3% of the control value, whichever is greater. Gamma counting was not performed unless the control counts were within the required limits. Background counts are performed on all gamma detectors at least weekly for either an overnight or weekend count. The most recent background is subtracted from all sample counts.

Assumption and Limitations of the Data

The digestion was performed exactly as stated in procedure RPG-CMC-129. Following the digestion process, each sample had a visible quantity of residual solids. The residual solids have measureable activity. The GEA results for the residual solids are included in this report.

Interferences/Resolution

None.

Uncertainty

For gamma counting, the uncertainty in the counting data, photon abundance and the nuclear half-life are included in the calculation of the total uncertainty along with a systematic uncertainty for sample preparation. The Canberra Genie software includes both random and systematic uncertainties in the calculation of the total uncertainties which are listed on the report. We conservatively estimate that 2% is the lowest uncertainty possible for our GEA measurements taking into account systematic uncertainties in gamma calibration standards.

Solid residues were placed in glass LSC vials. Unlike liquid samples, their geometries could not be carefully controlled to match the calibration standards. The solids samples were counted at about ~6 cm from the detector face to minimize the differences between the sample geometry and the calibration geometry. Calibrations are performed using water samples whereas the samples consisted of unknown undissolved solids (The exact sample mineralogical composition and associated density were not known). The small sample size (< 1 g) minimized the gamma absorption effects; therefore, density corrections were not applied.

Comments

1. The results account for all dilution factors resulting from sample processing.

2. Minimum Detectable Activity (MDA) and Minimum Detectable Concentration (MDC). Sample results are compared to the process blank results to evaluate if the blank contains 5% or more of the analyte of interest. When samples undergo digestion and/or dilution (process factors), the MDA must be adjusted for the process factors to accurately compare activity levels in the blank to activity levels in the samples. Process blank results have been adjusted for digestion and/or process factors for evaluation of the 5% criteria.

3. An occurrence report documents DQO related issue(s) with this report: RPD's outside the <20% requirement, OR-98620-12-23-15

Attachment: Data Report Sample Results for ASR 9916.

Pacific Northwest National Laboratory
PO Box 999, Richland, WA
Radiochemical Sciences and Engineering Group

filename: 16-0032 Fiskum.xlsx
12/7/2015

Client: S. Fiskum
ASR: 9916

Project: 68122
WP: N51383

Prepared by: T Trang-le 1/4/16

Concur: L.R. Greenwood 1/4/16

Procedures: RPG-CMC-450, Rev. 2 Gamma Energy Analysis (GEA) and Low-Energy Photon Spectrometry (LEPS)
Count date: November 24-December 4, 2015
M & TE: C, G,M,N

Measured Activities, µCi per g_{dry} ± 1s uncertainty

Sample ID	RPL ID	Mn-54	Co-60	Sb-125	Cs-134	Cs-137	Eu-152	Eu-154	Eu-155	Am-241
68122-TI-001-F	16-0032	<6.E-3	**1.08E-1 ± 2%**	<1.E-1	<7.E-3	**1.02E+2 ± 2%**	**3.88E-2 ± 8%**	**1.54E+0 ± 2%**	**2.28E-1 ± 3%**	**9.64E+1 ± 4%**
68122-TI-001-G	16-0032DUP	<4.E-3	**9.91E-2 ± 2%**	<8.E-2	<5.E-3	**9.49E+1 ± 2%**	**3.13E-2 ± 16%**	**1.46E+0 ± 2%**	**2.55E-1 ± 9%**	**1.06E+2 ± 2%**
RPD		-	8.6%	-	-	7.2%	**21.4%**	5.3%	11.2%	9.5%
2s%*							**34.0%**			
Blank	BLK-0032	<3.E-4	<3.E-4	<8.E-4	<3.E-4	<3.E-4	<7.E-4	<5.E-4	<1.E-3	<3.E-3

Residual
Measured Activities, µCi per gram ± 1s uncertainty

Sample ID	RPL ID	Mn-54	Co-60	Sb-125	Cs-134	Cs-137	Eu-152	Eu-154	Eu-155	Am-241
68122-TI-001-F	16-0032	**8.28E-5 ± 33%**	**1.13E-1 ± 2%**	**3.18E-3 ± 15%**	<3.E-4	**1.01E+1 ± 2%**	**2.31E-3 ± 15%**	**3.26E-2 ± 2%**	**4.18E-3 ± 48%**	**1.25E+0 ± 2%**
68122-TI-001-G	16-0032DUP	<3.E-4	**1.34E-1 ± 2%**	**4.59E-3 ± 15%**	<3.E-4	**1.07E+1 ± 2%**	**2.06E-3 ± 14%**	**3.41E-2 ± 2%**	**5.03E-3 ± 7%**	**1.78E+0 ± 2%**
RPD		-	17.0%	**36.3%**	-	6.2%	11.4%	4.6%	18.4%	**34.5%**
2s%*				**42.9%**						**5.1%**

*2-sigma variation of the duplicate measurements taking into account the counting uncertainties. The RPD value is statitically acceptable if it is below this value.

Radiochemistry M&TE List

ID	Number of Detectors	Property #	Model or Serial #	Location, in Bldg. 325	Procedures using the M&TE	PM Contract?
Dual Alpha Beta Gas Proportional Counters						
Oxford LB4100	16	WD13066	L8285-O	425	Total alpha, total beta, Sr/Y90, Tc99	Y
Alpha Counters						
Ludlum	10	multiple	multiple	425	Total Alpha	N
AEA (Ortec)*	32	multiple	multiple	425	AEA (total alpha, Pu, Am/Cm,Np)	N
Liquid Scintillation						
Perkin Elmer 3100TR	1	WD48466	DG08061340	425	H-3, C-14, Ni-63, Se-79, Pu-241, Sr-90	Y
Packard 2550	1	WD06783	401664	425	H-3, C-14, Ni-63, Se-79, Pu-241, Sr-90	Y
Gamma						
HpGe*	7	see list	see list	425	GEA with 2 Ortec Nomad portables	N
LEPS (x-rays)*	2	see list	see list	425	X-ray (Fe-55, Ni-59, Nb-93m)	N
Uranium						
Chemcheck	1	WC47898	KPA11R	525	Uranium KPA	N
***Spectral Analysis System**						
~~Canberra - VAX 3000~~	~~1~~	~~WD12890~~	~~PE42AJB~~	~~425~~	~~AEA, GEA~~	~~N~~
~~Canberra - VAX 3100~~	~~1~~	~~WC38624~~	~~KA235W0225~~	~~425~~	~~AEA, GEA (Backup VAX)~~	~~N~~

List of Gamma Detectors

Name	Property Tag	Vendor	Model	Serial #	Type	Eff. %	CMC?
C	WD25157	EG&G Ortec	GEM70200P	33TP40378A	Ge	70	Y
D	WD06582	Princeton	IGC7022SD	2542	Ge	70	Y
E	WD12833	EG&G Ortec	GEM100210	34P40535A	Ge	100	Y
G	WD35719	Canberra	GC4020	9923007	Ge	40	Y
H	PT00941	Tennelec	CPVS3040195	6028	Ge	40	Y
I	WD12076	EG&G Ortec	GLP3638510P	33TE187	LEPS	X-RAY	Y
J	WD12075	EG&G Ortec	GLP3638510P	33TE190	LEPS	X-RAY	Y
K	WD06581	Princeton	NIGC6022SD	3612	P-Ge	60	Y
L	WD36413	EG&G Ortec	GEM20P-Plus	44TP21884A	Ge	20	Y
M	WD73130	Canberra	GC10021	9722	Ge	100	Y
N	WD73129	Canberra	GC10021	9716	Ge	100	Y
~~P~~	~~WD59508~~	~~EG&G Ortec~~	~~GEM30210~~	~~24P80V~~	~~Ge~~	~~30~~	~~Y~~
T	WD81868	EG&G Ortec	GEM20180	35TP21056A	Ge	20	Y

Name	Property Tag	Vendor	Model	Serial #	Type	Eff. %	CMC?
~~W1~~	~~PT27133~~	~~Canberra~~	~~GC4018~~	~~9919~~	~~Ge~~	~~40~~	~~Y~~
F	PT16418	Canberra	GC10021	10182	Ge	100	Y
Q	PT16419	Canberra	GC10021	10188	Ge	100	Y
R	PT16420	Canberra	GC10021	10181	Ge	100	Y
S	PT16430	Canberra	GC10021	10101	Ge	100	Y
U	PT16429	Canberra	GC10021	10178	Ge	100	Y
V	PT16428	Canberra	GC10021	10150	Ge	100	Y
W	PT16394	Canberra	GC10021	10129	Ge	100	Y
X	PT16402	Canberra	GC10021	10104	Ge	100	Y
Y	PT16401	Canberra	GC10021	10145	Ge	100	Y
Z		Canberra	GC10021		Ge	100	Y
LEPS1	WB61769	Ortec	101351600S	20M102	Ge	10	
LEPS2	WD48418	Canberra	GL2015R	6068240	Ge	15	Y

Battelle PNNL/RPL/ASO Radiochemistry Analysis Report
P.O. Box 999, 902 Battelle Blvd., Richland, Washington 99352

Plutonium 238, 239+240 Analysis
Revision 1

Project / WP#:	68122/N51383
ASR#:	9916
Client:	SK Fiskum
Total # of Samples:	2

RPL ID	Client Sample ID
16-0032	68122-TI-001-F
16-0032 DUP	68122-TI-001-G

Analysis Type:	AEA – Pu-238, Pu-239+240
Sample Processing Prior to Radiochemical Processing/Analysis	☐ None ☐ Digested as per PNL-ALO-106, Rev. 1, *Acid Digestion of Waters, Soils, and Sludges for Subsequent Radiochemical Sample Analyses* ☐ Fusion as per RPG-CMC-115 Rev. 0, *Solubilization of Metals from Solids Using a KOH-KNO_3 Fusion* ☒ Digested as per RPG-CMC-129, Rev.0, *HNO_3-HCL Acid extraction of Solids Using a Dry-Block Heater*
Pre-dilution Prior to Radiochemical Processing?	☒ No ☐ Yes -- *example 2 mL to 100 mL; 50x dilution*
Total Alpha Preparation Procedure:	RPG-CMC-4001, Rev. 1, *Source Preparation For Gross Alpha and Gross Beta analyses.*
Technician/Analyst:	LP Darnell, 01/06/2016
Spike Standard ID's	R-485-b-14 (Pu-239)
Analysis Procedure	RPG-CMC-408, Rev. 2, *Total Alpha and Beta Analysis*
Technician/Analyst:	T Trang-Le, 01/08/2016
Plutonium Separation Procedure:	RPG-CMC-4017, Rev. 0, *Analysis of Environmental Water Samples for Actinides and Strontium-90*
Technician/Analyst:	LP Darnell, 12/16/2015
Co-Precipitation Procedure:	RPG-CMC-496, Rev. 1, *Coprecipitation Mounting of Actinides for Alpha Spectroscopy*
Technician/Analyst:	LP Darnell 12/15/2015
Spike and Tracer Standard ID's:	R-485-b-14(Pu-239), R-671-a-6 (Pu-242 tracer)
Analysis Procedure:	RPG-CMC-422, Rev. 2, *Solutions Analysis: Alpha Spectrometry*
Reference Date:	Same as analyses dates
Analysis Date or Date Range:	12/15/2015
Technician/Analyst:	T. Trang-Le
Analysis Data (File):	RPG-RC\PNL\Projects\Backup files\Backup 16\16-0032 Fiskum.xlsx
CMC Project 98620 File:	File Plan 5871: T 68122: Sample preparation and analysis records; T-4.4 Alpha Detector calibration, calibration verification checks, and maintenance records; and T3 Standard certificates and preparation. Also balance calibration and instrument performance checks.
M&TE Number(s):	Ortec AEA counters – 32 counters – See attached M&TE list

Preparer / 2/2/16 Date

T Trang-Le Reviewer / 2/2/2016 Date

Revision 1 – Report revised to report the acid digestion blank data instead of the lab prepared blank data.

Sample Results

See attached data report, Sample Results for ASR 9916. All data are reported in units of µCi per g_{dry} with a 1-σ uncertainty.

Sample Preparation, Separation, Mounting and Counting Methods

Two wet solids samples submitted under Analytical Service Request (ASR) 9916 were analyzed for analyzed for plutonium by Alpha Spectrometry. Prior to acid digestion, the wet solids were dried to constant weight. The dried solids samples were then acid digested using procedure RPG-CMC-129, "HNO_3-HCl Acid Extraction of Solids Using a Dry-Block Heater". All the samples were prepared in laboratory 420. The leachate solution was used for radioanalytical analyses; only Pu-AEA data are included in this report.

Following the digestion process, the Pu was separated from the leachate by anion exchange using procedure RPG-CMC-4017. The separated Pu fraction was then mounted for alpha spectrometry by co-precipitation using procedure RPG-CMC-496, and then counted using alpha spectrometry using procedure RPC-CMC-422. The samples were counted on December 15, 2015; no decay corrections were made.

Although not formally requested by the ASR, gross alpha and beta analyses were performed on each sample and duplicate to obtain information to estimate aliquot sizes for other analyses and for checking the internal consistency of the alpha isotopic data. The gross alpha results are included in the data tables. Gross alpha activity were measured by evaporating small aliquots of leachate onto counting planchets per procedure RPG-CMC-4001 and counting per procedure RPG-CMC-408.

QUALITY CONTROL RESULTS

Quality control (QC) samples prepared in laboratory 420 include a process blank (PB) and sample duplicates. Additional QC samples were prepared prior to alpha counting including a laboratory blank, a reagent blank spike (RS, Pu-239), and addition of Pu-239 standard to an aliquot of the sample digestate selected as the matrix spike (MS).

The QC sample results for Pu-AEA have been evaluated and are discussed below. A summary of the Pu-AEA analysis results, including QC sample performance, is given in the attached data report.

Tracer:

The Pu-242 tracer is added to every sample after appropriate dilution and prior to plutonium separations. The use of a Pu-242 tracer corrects for radiochemical yield and mathematically removes the detector counting efficiency from the results calculations. Tracer recovery is required to be high enough to provide acceptable counting statistics. The Pu-242 tracer counting statistics were acceptable for all samples. The tracer recoveries ranged from 95% to 118%.

Laboratory Preparation Blank (PB):

The detection limit achieved in the laboratory preparation blank for Pu-238 is 2.37E-4 µCi per g_{dry} and for Pu-239+240 is 9.59E-4 µCi per g_{dry}. The activity level of the plutonium alpha emitters present in the blank is well below 5% of the activity present in the sample, meeting the acceptance criteria of less than 5% of the sample activity or less than the sample MDA.

Blank Spike (BS)/Reagent Spike (RS):

The BS recovery of 104% (Pu-239) meets the acceptance criteria of 80% to 120% recovery.

Matrix Spike (MS):

The MS recovery of 101% (Pu-239) meets the acceptance criterion of 75% to 125% recovery. Note: The MS sample was prepared after digestion, by adding a known quantity of Pu-239 standard to a diluted aliquot of the digestate. Sample number 16-0032 (68122-TI-001-F) was selected as the matrix spike sample.

Laboratory Duplicate - Relative Percent Difference (RPD):

For sample 68122-TI-001-F/G, the Pu-238 sample and duplicate RPD of 1% within the acceptance limit of ≤ 20% RPD for. The RPD for Pu-239+240 is 0.4%, within the acceptance limit of ≤ 20% RPD.

Instrument Quality Control

Alpha counters receive initial calibration with NIST traceable sources to determine the counter efficiency. When internal tracers are not used, the counter efficiency is used in calculation of final results.

Detector backgrounds are determined every 4 weeks or after the last analytical run, whichever is longer. Detector background counts are subtracted from all subsequent sample counts. A process blank is analyzed with each analytical batch to evaluate for contamination in the sample preparation process.

Assumption and Limitations of the Data

None

Attachment: Data Report -- Sample Results for ASR 9916.

Battelle PNNL/RPL/ASO Radiochemistry Analysis Report
P.O. Box 999, 902 Battelle Blvd., Richland, Washington 99352

Neptunium 237 Analysis
Revision 1

Project / WP#:	68122/N51383
ASR#:	9916
Client:	SK Fiskum
Total # of Samples:	2

Sample IDs	RPL Number
68122-TI-001-F	16-0032
68122-TI-001-G	16-0032 DUP

Analysis Type:	AEA – Np-237
Sample Processing Prior to Radiochemical Processing/Analysis	☐ None ☐ Digested as per RPG-CMC-106, Rev. 1, *Acid Digestion of Waters, Soils, and Sludges for Subsequent Radiochemical Sample Analyses* ☐ Fusion as per RPG-CMC-115, *Solubilization of Metals from Solids Using a KOH-KNO_3 Fusion* ☒ Digested as per RPG-CMC-129, Rev.0, *HNO_3-HCL Acid extraction of Solids Using a Dry-Block Heater* Other:
Pre-dilution Prior to Radiochemical Processing?	☒ No ☐ Yes -- *example 2 mL to 100 mL; 50x dilution*
Total Alpha and Beta Preparation Procedure:	RPG-CMC-4001, Rev. 1, *Source Preparation For Gross Alpha and Gross Beta analyses.*
Technician/Analyst:	LP Darnell, 01/06/2016
Spike Standard ID's	R-485-b-14 (Pu-239), R-676-b-2 (Sr-90)
Analysis Procedure	RPG-CMC-408, Rev. 2, *Total Alpha and Beta Analysis*
Technician/Analyst:	T. Trang-Le, 01/08/2016
Neptunium Separation Procedure:	RPG-CMC-4017, Rev. 0, *Analysis of Environmental Water Samples for Actinides and Strontium- 90*
Technician/Analyst:	LP Darnell, 12/28/2015
Spike Standard ID's:	RS-219-int-2-4 (Np-237)
Co-Precipitation Procedure:	RPG-CMC-496, Rev. 1, *Coprecipitation Mounting of Actinides for Alpha Spectroscopy*
Technician/Analyst:	LP Darnell, 12/28/2015
Analysis Procedure:	RPG-CMC-422, Rev. 2, *Solutions Analysis: Alpha Spectrometry*
Reference Date:	Same as counting dates
Analysis Date or Date Range:	12/30/2015
Technician/Analyst:	T. Trang-Le
Analysis Data (File):	RPG-RC\PNL\Projects\Backup files\Backup 16\16-0032 Fiskum.xlsx
CMC Project 98620 File:	File Plan 5871: T 68122: Sample preparation and analysis records; T-4.4 Alpha Detector calibration, calibration verification checks, and maintenance records; and T3 Standard certificates and preparation. Also balance calibration and instrument performance checks.
M&TE Number(s):	Ortec AEA counters – 32 counters – See attached M&TE list

Preparer / Date: 2/2/16

Reviewer / Date: T Trang-Le / 2/2/2016

Battelle PNNL/RPL/ASO Radiochemistry Analysis Report

Revision 1 – Report revised to clarify the matrix spike QC discussion.

Sample Results

See attached data report, Sample Results for ASR 9916. All data are reported in units of uCi per g_{dry} with a 1-σ uncertainty unless noted otherwise (see comments).

Sample Preparation, Separation, Mounting and Counting Methods
Two wet solids samples submitted under Analytical Service Request (ASR) 9916 were analyzed for analyzed for neptunium by Alpha Spectrometry. Prior to acid digestion, the wet solids were dried to constant weight. The dried solids samples were then acid digested using procedure RPG-CMC-129, "HNO_3-HCl Acid Extraction of Solids Using a Dry-Block Heater". All the samples were prepared in laboratory 420. The leachate solution was used for radioanalytical analyses; only Np-AEA is reported in this report.

Following the digestion process, the Np was separated from the sludge leachate using anion exchange chromatography using procedure RPG-CMC-4017. The separated Np fraction was mounted for alpha spectrometry by co-precipitation using procedure RPG-CMC-496, and then counted by alpha spectrometry using procedure RPC-CMC-422.

Although not formally requested by the ASR, gross alpha and gross beta analyses were performed on each sample to obtain information to estimate aliquot sizes for other analyses and for checking the internal consistency of the Np alpha isotopic data. The gross alpha and gross beta results are included in the data tables as supplemental information only. Gross alpha and gross beta activity were measured by evaporating small aliquots of leachate onto counting planchets per procedure RPG-CMC-4001 and counting per procedure RPG-CMC-408.

QUALITY CONTROL RESULTS

Quality control (QC) samples include an acid digestion blank, sample duplicate. Additional QC samples were prepared prior to separations; these include a laboratory separation blank, a reagent blank spike (BS), and a matrix spike (MS) made by adding Np-237 standard to a diluted sample.

Tracer:

Tracer is not used for analyses of Np.

Laboratory Preparation Blank (PB) and Laboratory separations blank (LB):

The Np-237 activity measured in the PB is required to be within the acceptance criteria of less than sample minimum detectable activity (MDA) or less than 5% of the sample isotope concentration. The Np-237 PB is <MDA; thus meeting the acceptance criteria. There are no acceptance criteria for LB.

Blank Spike (BS) – reagent spike (RS):

The RS recovery of 93% meets the acceptance criteria of 80% to 120% recovery.

Matrix Spike (MS):

The MS recovery of 96% meets the acceptance criterion of 75% to 125% recovery. Note: the MS sample was prepared "after" digestion, by adding a known Np-237 standard quantity to an aliquot of the leachate. Sample number 16-0032 (68122-TI-001-F) was selected as the matrix spike sample.

Duplicate -- Relative Percent Difference (RPD):

Duplicate results are required to agree within ≤20% RPD. Duplicate results were 15% RPD. The Np-237 activity detected in the samples all have 1-σ counting error of 10% or greater. 1-σ counting error at these levels indicates the activity measured in the samples is nearing the minimum detectable activity for the samples.

Instrument Quality Control

Alpha counters undergo calibration annually to determine the counter's efficiency over the normal calibration range of 3 to 6 MeV. The vendor software determines a constant detector efficiency for this energy range. Np samples are counted and results calculated using the established detector efficiency.

Detector backgrounds are determined every 4 weeks or after the last analytical run, whichever is longer. Detector background counts are subtracted from all subsequent sample counts. A process blank is analyzed with each analytical batch to evaluate for contamination in the sample preparation process.

Assumption and Limitations of the Data

Undissolved residue remained after the completion of the acid digestion/leach of all sludge samples.

Comments

1. The results have been corrected for all dilution factors resulting from sample processing.
2. Post-Digestion Spike (PS) - A spike made after the initial sample preparation (e.g., fusion, digestion, or leach) is considered a PS. When extremely radioactive samples are analyzed, most of the radioanalytical spikes are made after the sample preparation (to avoid excessive consumption of spike and avoid creating unnecessary waste) and are post-digestion spikes. The MS prepared with this batch of sample is considered a PS, since the Np-237 spike was not added prior to the digestion process.
3. The 1-sigma uncertainty represents the total propagated error associated with processing and counting operations and include; weighing errors, volume uncertainties, and counting error.
4. The sample results are compared to the process blank to evaluate if the blank contains 5% or more of the measured isotope; the process blank result has been adjusted for all processing factors for evaluation of the 5% criterion.
5. The Laboratory Blank (LB) is prepared using laboratory reagents and provides data on the cleanliness of the radiochemistry preparation/separation processes. LB results are not normalized to processing or dilution factors associated with the samples.

6. The sample results are compared to the process blank to evaluate if the blank contains 5% or more of the measured isotope; the process blank result has been adjusted for all processing factors for evaluation of the 5% criterion.

Attachment: Data Report -- Sample Results for ASR 9916.

Battelle PNNL/RPL/ASO Radiochemistry Analysis Report
P.O. Box 999, 902 Battelle Blvd., Richland, Washington 99352

Am-241 Analysis
Revision 2

Project / WP#:	68122/N51383
ASR#:	9916
Client:	SK Fiskum
Total # of Samples:	2

RPL ID	Client Sample ID
16-0032	68122-TI-001-F
16-0032 DUP	68122-TI-001-G

Analysis Type:	AEA – Am-241
Sample Processing Prior to Radiochemical Processing/Analysis	☐ None ☐ Digested as per RPG-CMC-106, Rev. 1, *Acid Digestion of Waters, Soils, and Sludges for Subsequent Radiochemical Sample Analyses* ☐ Fusion as per RPG-CMC-115 Rev. 0, *Solubilization of Metals from Solids Using a KOH-KNO_3 Fusion* ☒ Digested as per RPG-CMC-129, Rev.0, *HNO_3-HCL Acid extraction of Solids Using a Dry-Block Heater* Other:
Pre-dilution Prior to Radiochemical Processing?	☒ No ☐ Yes -- *example 2 mL to 100 mL; 50x dilution*
Total Alpha and Beta Preparation Procedure:	RPG-CMC-4001, Rev. 1, *Source Preparation For Gross Alpha and Gross Beta analyses.*
Technician/Analyst:	LP Darnell, 01/06/2016
Spike Standard ID's	R-485-b-14 (Pu-239), R-676-b-2 (Sr-90)
Analysis Procedure	RPG-CMC-408, Rev. 2, *Total Alpha and Beta Analysis*
Technician/Analyst:	TL Trang-Le, 01/08/2016
Americium Separation Procedure:	RPG-CMC-4017, Rev. 0, *Analysis of Environmental Water Sample for Actinides and Sr-90*
Technician/Analyst:	LP Darnell, 12/14/2015
Spike and Tracer Standard ID's:	R-542-a-7 (Am-241), R-628-a-6 (Am-243 tracer)
Co-Precipitation Procedure:	RPG-CMC-496, Rev. 1, *Coprecipitation Mounting of Actinides for Alpha Spectroscopy*
Technician/Analyst:	LP Darnell 12/16/2015
Analysis Procedure:	RPG-CMC-422, Rev. 2, *Solutions Analysis: Alpha Spectrometry*
Reference Date:	Same as analyses dates
Analysis Date or Date Range:	12/16/2015
Technician/Analyst:	T. Trang-Le
Analysis Data (File):	RPG-RC\PNL\Projects\Backup files\Backup16\16-0032 Fiskum.xlsx
CMC Project 98620 File:	File Plan 5871: T 68122: Sample preparation and analysis records; T-4.4 Alpha Detector calibration, calibration verification checks, and maintenance records; and T3 Standard certificates and preparation. Also balance calibration and instrument performance checks.
M&TE Number(s):	Ortec AEA counters – 32 counters – See attached M&TE list

Preparer / Date: [signature] / 3/2/16

Reviewer / Date: T Trang-Le / 3/2/16

Battelle PNNL/RPL/ASO Radiochemistry Analysis Report

Revision 1 – Report revised to clarify application of MDA value to the acid digestion blank.

Revision 2 – Report revised to correct the Americium separation procedure reference on the cover page

Sample Results

See attached data report, Sample Results for ASR 9916. All data are reported in units of µCi per g_{dry} with a 1-σ uncertainty (see comments).

Sample Preparation, Separation, Mounting and Counting Methods

Two wet solids samples submitted under Analytical Service Request (ASR) 9916 were analyzed for analyzed for americium by Alpha Spectrometry. Prior to acid digestion, the wet solids were dried to constant weight. The dried solids samples were then acid digested using procedure RPG-CMC-129, "HNO_3-HCl Acid Extraction of Solids Using a Dry-Block Heater". All the samples were prepared in laboratory 420. The leachate solution was used for radioanalytical analyses; only Am-AEA data are included in this report.

Following the digestion process, the Am was separated from the leachate by anion exchange using procedure RPG-CMC-4017. The separated Am fraction was then mounted for alpha spectrometry by co-precipitation using procedure RPG-CMC-496, and then counted using alpha spectrometry using procedure RPC-CMC-422. The samples were counted on December 16, 2015; no decay corrections were made.

Although not formally requested by the ASR, gross alpha and gross beta analyses were performed on each sample to obtain information to estimate aliquot sizes for other analyses and for checking the internal consistency of the Am alpha isotopic data. The gross alpha and gross beta results are included in the data tables as supplemental information only. Gross alpha and gross beta activity were measured by evaporating small aliquots of leachate onto counting planchets per procedure RPG-CMC-4001 and counting per procedure RPG-CMC-408.

QUALITY CONTROL RESULTS

Quality control (QC) samples prepared in laboratory 420 include a process blank (PB) and sample duplicates. Additional QC samples were prepared prior to alpha counting including a laboratory blank, a reagent blank spike (BS, Am-241), and addition of Am-241 standard to an aliquot of the sample digestate selected as the matrix spike (MS).

The QC sample results for Am-AEA have been evaluated and are discussed below. A summary of the Alpha-AEA analysis results, including QC sample performance, is given in the attached data report.

Tracer:

The Am-243 tracer is added to every sample after appropriate dilution and prior to americium separations. The Am-243 tracer corrects for radiochemical yield and mathematically removes the detector counting efficiency from the results calculations. Tracer recovery is required to be high enough to provide acceptable counting statistics. The Am-243 tracer counting statistics were acceptable for all samples. The tracer recoveries ranged from 98% to 117%.

Laboratory Preparation Blank (PB):

The detection limit achieved in the acid digestion preparation blank for Am-241 is 1.81E-4 ± 15% µCi per g_{dry} (Note: The minimum detectable activity for the acid digestion preparation blank is 2.9E-5µCi per g_{dry}). The Am-241 activity measured in the PB are required to be within the acceptance criteria of less than 5% of the sample isotope concentration or less than sample minimum detectable activity (MDA) – see comments. The PB Am-241 activity is significantly less than 5% of any sample activity. There are no acceptance criteria for LB

Blank Spike (BS)/Reagent Spike (RS):

The RS recovery of 100% (Am-241) meets the acceptance criteria of 80% to 120% recovery.

Matrix Spike (MS):

The MS recovery of 101% (Am-241) meets the acceptance criterion of 75% to 125% recovery. Note: the MS sample was prepared after digestion, by adding a known quantity of Am-241 standard to a diluted aliquot of the digestate. Sample number 16-0032 (68122-TI-001-F) was selected as the matrix spike sample.

Laboratory Duplicate - Relative Percent Difference (RPD):

The Am-241 sample and duplicate RPD of 8% less than ≤ 20% RPD for sample 68122-TI-001-F/G.

Instrument Quality Control

Alpha counters receive initial calibration with NIST traceable sources to determine the counter efficiency. When internal tracers are not used, the counter efficiency is used in calculation of final results.

Detector backgrounds are determined every 4 weeks or after the last analytical run, whichever is longer. Detector background counts are subtracted from all subsequent sample counts. A process blank is analyzed with each analytical batch to evaluate for contamination in the sample preparation process.

Assumption and Limitations of the Data

None

Attachment: Data Report -- Sample Results for ASR 9916.

Battelle PNNL/RPL/ASO Radiochemistry Analysis Report
P.O. Box 999, 902 Battelle Blvd., Richland, Washington 99352

Sr-90 by Liquid Scintillation Spectrometry
Revision 1

Project / WP#:	68122/N51383
ASR#:	9916
Client:	SK Fiskum
Total # of Samples:	2

RPL ID	Client Sample ID
16-0032	68122-TI-001-F
16-0032 DUP	68122-TI-001-G

Analysis Type:	Sr-90
Sample Processing Prior to Radiochemical Processing/Analysis	☐ None ☒ Digested as per RPG-CMC-129, Rev. 0, *HNO_3-HCl Acid Extraction of Solids Using a Dry Block Heater.* ☐ Fusion as per PNL-ALO-115, *Solubilization of Metals from Solids Using a KOH-KNO_3 Fusion* ☐ Other:
Pre-dilution Prior to Radiochemical Processing?	☒ No ☐ Yes
Total Alpha and Beta Preparation Procedure:	RPG-CMC-4001, Rev. 1, *Source Preparation For Gross Alpha and Gross Beta Analyses.*
Technician/Analyst:	LP Darnell, 01/06/2016
Spike Standard ID's	R-485-b-14 (Pu-239), R-676-B-2 (Sr-90)
Analysis Procedure	RPG-CMC-408, Rev. 2, *Total Alpha and Beta Analysis*
Technician/Analyst:	T. Trang-Le, 01/08/2016
Separation Procedure:	RPG-CMC-476, Rev. 0, *Strontium Separation Using Eichrom Strontium Resin*
Spike Standard ID:	R-569-a-10 (Sr-90)
Separation Date:	12/7/15 @ 13:45 p.m.
Technician/Analyst:	L. Darnell
Analysis Procedure:	RPG-CMC-474, Rev. 1, *Measurement of Alpha and Beta Activity by Liquid Scintillation Spectrometry*
Reference Date:	12/30/2015
Analysis Date or Date Range:	12/30/15 (first count), 1/4/16 (second count)
Technician/Analyst:	T. Trang-Le
Rad Chem Electronic Data File:	RPG-RC\PNL\Projects\Backup files\Backup 16\16-0032 Fiskum.xlsx
ASO Project 98620 File:	File Plan 5871: T 68122 9916: Sample preparation and analysis records; T-4.4 LSC 3100 calibration, daily checks, and maintenance records; and T3 standard certificates and preparation. Also, balance calibration and performance check records.
M&TE Number(s):	Perkin Elmer Tri-Carb 3100, Serial # DG08061340, RPL 425, Tri-Carb 2700TR software version 1.04 dated 9/99.

Preparer	Date	Reviewer	Date
[signature]	2/2/16	T Trang-Le	2/2/2016

Battelle PNNL/RPL/ASO Radiochemistry Analysis Report

Revision 1 – Report revised to correct the sample result uncertainty and provide the correct reference for the sample preparation location.

SAMPLE RESULTS

See attached data report, Sample Results for ASR 9916. All sample data are reported in $\mu Ci/g_{dry}$ with a 1-σ uncertainty (see Comments).

Sample preparation, separation, mounting, and counting

Two wet solids samples submitted under Analytical Service Request (ASR) 9916 were analyzed for Sr-90 by chemical separation and liquid scintillation counting. Prior to acid digestion, the wet solids were dried to constant weight. The dried solids samples were then acid digested using procedure RPG-CMC-129, "HNO_3-HCl Acid Extraction of Solids Using a Dry-Block Heater". All the samples were prepared in laboratory 420. The leachate solution was used for radioanalytical analyses; only Sr-90 data are included in this report.

Although not formally requested by the ASR, gross alpha and gross beta analyses were performed on each sample to obtain information to estimate aliquot sizes for other analyses and for checking the internal consistency of the Sr-90 data. The gross alpha and gross beta results are included in the data tables as supplemental information only. Gross alpha and gross beta activity were measured by evaporating small aliquots of leachate onto counting planchets per procedure RPG-CMC-4001 and counting per procedure RPG-CMC-408.

QUALITY CONTROL RESULTS

Radioanalytical quality control (QC) samples prepared in samples prepared in laboratory 420 and include a process blank (PB), and sample duplicate. No Sr-90 MS was prepared at time of digestion. Additional laboratory QC samples were prepared prior to separations; these include a laboratory separation blank, a reagent blank spike (BS), and a matrix spike (i.e., addition of Sr-90 standard to an aliquot of one of the samples).

Instrument Calibration Control

Laboratory Preparation Blank and Laboratory separations blank (LB):

The Sr-90 activity measured in the PB is required to be within the acceptance criteria of less than sample minimum detectable activity (MDA) or less than 5% of the sample isotope concentration. The Sr-90 PB is <MDA; thus meeting the acceptance criteria. There are no acceptance criteria for LB (see Comments).

Blank Spike (BS) – reagent spike:

The BS recovery of 96% meets the procedure acceptance criteria of 80% to 120% recovery.

Matrix Spike (MS):

The MS recovery of 98% meets the acceptance criterion of 75% to 125% recovery. Note: the MS sample was prepared "after" digestion (see comments), by adding a known Sr-90 standard quantity to an aliquot of 16-0032 (68122-TI-001-F).

Duplicate -- Relative Percent Difference (RPD):

Duplicate results are required to agree within ≤20% RPD. The ASO QAP further specifies that the two results need to be >5 times the MDA or have individual uncertainties <20%. Duplicate result was 0.5% RPD; thus meeting the ≤20% requirement.

Instrument Quality Control

The liquid scintillation counter is calibrated for tritium and C-14 using quenched standard sets that are purchased from the vendor. Daily control counts are then performed using a tritium, C-14, and a background count sample. The instrument software assesses the performance of the control counts and provides control charts to ensure the continuing calibration of the instrument. If the daily performance check fails, then the instrument is not used. Preventative maintenance and repairs are performed by the vendor under our service contract. The counting efficiency for Sr-90 is assumed to be 100%; therefore no specific Sr-90 calibration is performed. The LSC system calibration and performance is verified by assessing the recovery of a reagent spike and a matrix spike that are included in every batch of samples. A preparation blank (i.e., digestion blank) and a laboratory separations blank are also included with every batch of samples; the instrument background is subtracted from all results and the preparation and separation blanks are used to assess sample contamination during sample processing steps.

Assumption and Limitations of the Data

The 1-σ uncertainty reported for each Sr-90 result has been set at 2%. Although the calculated uncertainty values are less than 2% for all samples, the radiochemistry convention is to not report calculated uncertainties less than 2%, but to provide a more realistic estimate of the uncertainty in view of systematic uncertainties that are not fully accounted for in the uncertainty calculations.

Comments

1. The results have been corrected for all dilution factors resulting from sample processing.
2. Post-Digestion Spike (PS) - A spike made after the initial sample preparation (e.g., fusion, digestion, or leach) is considered a PS. When extremely radioactive samples are analyzed, most of the radio-analytical spikes are made after the sample preparation (to avoid excessive consumption of spike and avoid creating unnecessary waste) and are post-digestion spikes. The MS prepared with this batch of sample is considered a PS, since the Sr-90 spike was not added prior to the digestion process.
3. Radiochemistry Electronic Systems File "RPG-RC\PNL\Projects\Backup files\Backup 16\16-0032 Fiskum.xlsx" has been created for this report. Supporting records such as Pipette Performance Verification forms, Laboratory Bench Record, Laboratory Sample Preparation Bench Sheet, Standards Certifications and preparation records, and balance calibration and performance check records are maintained per RS&E Group ASO File Plan 5871.
4. Sample results are compared to the process blank results to evaluate if the blank contains 5% or more of the measured isotope; the process blank results have been adjusted for all processing factors for the evaluation of the 5% criterion.
5. The stated 1-σ uncertainty represents the total propagated error associated with processing and counting operations and includes weighing errors, volume uncertainties, and counting error.

Attachment: Data Report -- Sample Results for ASR 9916

Battelle PNNL/RPL/ASO Radiochemistry Analysis Report
P.O. Box 999, 902 Battelle Blvd., Richland, Washington 99352

Gross Alpha and Beta
Revision 1

Project / WP#:	68122/N51383
ASR#:	9916
Client:	SK Fiskum
Total # of Samples:	2

RPL ID	Client Sample ID
16-0032	68122-TI-001-F
16-0032 DUP	68122-TI-001-G

Analysis Type:	Gross Alpha
Sample Processing Prior to Radiochemical Processing/Analysis	☐ None ☒ Digested as per RPG-CMC-129, Rev. 0 *HNO_3-HCl Acid Extraction of Solids Using a Dry- Block Heater* ☐ Fusion as per RPG-CMC-115, Rev.0 *Solubilization of Metals from Solids Using a KOH-KNO_3 Fusion* ☐ Other:
Pre-dilution Prior to Radiochemical Processing?	☒ No ☐ Yes -- *example 2 mL to 100 mL; 50x dilution*
Radio Chemical Preparation Procedure:	RPG-CMC-4001, Rev. 1, *Source Preparation for Gross Alpha and Gross Beta Analysis*
Technician/Analyst:	LP Darnell, 01/06/2016
Spike Standard ID's:	R-485-b-14 (Pu-239), R-676-B-2 (Sr-90)
Analysis Procedure:	RPG-CMC-408, Rev. 2, *Total Alpha and Beta Analysis*
Reference Date:	01/08/2016
Analysis Date(s) or Date Range:	01/08/2016
Technician/Analyst:	T. Trang-Le
Analysis Data (File):	RPG-RC\PNL\Projects\Backup files\Backup 16\16-0032 Fiskum.xlsx
CMC Project 98620 File:	File Plan 5871: T 68122: Sample preparation and analysis records; T-4.4 Alpha Detector calibration, calibration verification checks, and maintenance records; and T3 Standard certificates and preparation. Also balance calibration and instrument performance checks.
M&TE Number(s):	LB 4100 gas proportional counter – See attached M&TE list

Preparer / Date: 2/2/16

Reviewer / Date: T Trang-Le / 2/2/2016

Battelle PNNL/RPL/ASO Radiochemistry Analysis Report

Revision 1 – Report revised to add reference date for sample counting and correct the QC sample discussion to include the preparation of laboratory blank.

Sample Results

See attached data report, sample results for ASR 9916. All data are reported in units of µCi per g_{dry} solids with a 1-σ uncertainty (see comments).

Sample Preparation, Separation, Mounting and Counting Methods

Two wet solids samples submitted under Analytical Service Request (ASR) 9916 were analyzed for gross alpha/gross beta. Prior to acid digestion, the wet solids were dried to constant weight. The dried solids samples were then acid digested using procedure RPG-CMC-129, "HNO_3-HCl Acid Extraction of Solids Using a Dry-Block Heater". All the samples were prepared in laboratory 420. Aliquots of the acid digestions were diluted ~100x and mounted for gross alpha/gross beta counting using procedure RPG-CMC-4001, then counted using alpha/beta gas proportional counters per procedure RPG-CMC-408.

QUALITY CONTROL RESULTS

Quality control (QC) samples prepared in laboratory 420 include a laboratory duplicate sample and a preparation blank. Additional QC samples were prepared prior to alpha and beta counting including a laboratory blank, a reagent blank spike (RS, Pu-239 and Sr-90), and addition of Pu-239 and Sr-90 standard to a diluted aliquot of the sample selected as the matrix spike (MS).

A summary of the Gross Alpha and Gross Beta analysis results, including QC sample performance, is given in the attached data report.

Tracer:

Tracer is not used for this analysis.

Laboratory Preparation Blank (PB) and Laboratory blank (LB):

The gross alpha activity measured in the PB is required to be within the acceptance criteria of less than 5% of the sample isotope concentration or less than sample minimum detectable activity (MDA). Each sample counting disc was counted for 100 minutes. The PB and LB alpha and beta activity are less than the MDA

Blank Spike (BS) – Reagent Spike (RS):

The RS (Pu-239) recovery of 87% and (Sr-90) recovery of 97% meets the acceptance criteria of 80% to 120% recovery.

Matrix Spike (MS):

The MS (Pu-239) recovery of 128% exceeds the acceptance criterion of 75% to 125% recovery. The MS (Sr-90) recovery of 113% meets the acceptance criterion of 75% to 125%. Note: the MS sample was prepared by adding a known Pu-239 and Sr-90 standard quantity to an aliquot of the digestate. Sample number 16-0032 (68122-TI-001-F) was selected as the matrix spike sample.

Duplicate -- Relative Percent Difference (RPD):

Sample and duplicate sample result RPD is 6% for the gross alpha and 5% for the gross beta. Both RPD results are within the acceptance criterion of $\leq 20\%$.

Instrument Quality Control

LB4100 alpha and Beta counters undergo initial calibration to determine the detector efficiency. The established efficiency for each detector is used in the final calculation of the sample gross alpha and beta activity. Continuing calibration verification checks are performed on the detectors once per day as the system is used. Detector backgrounds are obtained once per day or as the system is used or per batch.

Assumption and Limitations of the Data

The gross alpha/beta analyses was run twice to confirm the gross beta results. The sum of the individual beta emitters is only 2/3 of the gross beta result. The alpha activity in the samples is about twice that of the beta. The gross beta results are biased high due to the high alpha activity.

Attachment: Data Report -- Sample Results for ASR 9916.

Pacific Northwest National Laboratory
PO Box 999, Richland, WA
Radiochemical Sciences and Engineering Group

filename 16-0032 Fiskum.xlsx
2/2/2016
Rev1

Client: S. Fiskum
ASR: 9916

Project: 68122
WP: N51383

Prepared by: T Trang-Le 2/2/2016

Concur: C. Soderquist 2-2-2016

Procedures: RPG-CMC-4001 Rev1 Source Preparation For Gross Alpha and Gross Beta Analysis
RPG-CMC-408 Rev2 Total Alpha and Beta Analysis
RPG-CMC-474 Rev1 Measurement of Alpha and Beta Activity by Liquid Scintillation Spectrometry
RPG-CMC-496 Rev0 Coprecipitation Mounting of Actinides for Alpha Spectroscopy
RPG-CMC-422 Rev2 Solutions Analysis: Alpha Spectrometry
RPG-CMC-476, Rev. 0, Strontium Separation Using Eichrom Strontium Resin

Count date: December 15-January 8, 2016

M & TE: LB4100 Gas Proportional , LSC3110, Alpha spectrometry counting system

Measured Activities, µCi per g_{dry} ± 1s uncertainty

Sample ID	RPL ID	Gross alpha		Gross beta		Sr-90		Np-237		Pu-238		Pu-239+240		Am-241	
68122-TI-001-F	16-0032	**3.58E+2**	**± 3%**	**2.13E+2**	**± 3%**	**1.88E+1**	**± 2%**	**1.80E-3**	**± 16%**	**2.63E+1**	**± 4%**	**1.99E+2**	**± 2%**	**9.39E+1**	**± 3%**
68122-TI-001-G	16-0032DUP	**3.35E+2**	**± 3%**	**2.02E+2**	**± 3%**	**1.88E+1**	**± 2%**	**2.09E-3**	**± 14%**	**2.61E+1**	**± 4%**	**2.00E+2**	**± 2%**	**8.69E+1**	**± 3%**
	RPD	6%		5%		0.5%		15%		1%		0.4%		8%	
	BLK-0032-129	<3.E-1		<6.E-1		<6.E-3		<4.E-5		2.37E-4	± 13%	9.59E-4	± 7%	1.81E-4	± 15%
	Lab blank	<1.E-7		<3.E-7		<3.E-6		<4.E-8		1.20E-7	± 14%	5.07E-7	± 7%	4.76E-7	± 7%
	Reagent spike	87%		97%		96%		93%		--		104%		100%	
	Matrix spike	128%		113%		98%		96%		--		101%		101%	

This sample has about twice as much alpha activity as beta activity. The gross beta results are biased high by the alpha activity. The sum of the individual beta emitters is only about 67% of the measured gross beta activity.
Rev.1 is issued to correct the sample result uncertainty.

PNNL/Radiochemistry Processing Laboratory
P.O. Box 999, 902 Battelle Blvd., Richland, Washington 99352

PLUTONIUM ISOTOPIC ANALYSIS BY TIMS

Project / WP#: 68122/ / N51383

ASR#: 9916

Client: *SK Fiskum*

Total Samples: 2

Client ID	RPL Number	TIMS ID
68122-TI-001-F	16-0032	16-0032 F
68122-TI-001-F	16-0032	16-0032 F #2
68122-TI-001-G	16-0032 (dup)	16-0032 G
68122-TI-001-G	16-0032 (dup)	16-0032 G #2

Analysis Type:	Plutonium Isotopic Analysis by TIMS
Sample Processing Prior to Radiochemical Processing/Analysis	Digested as per RPG-CMC-129, Rev.0, *HNO3-HCL Acid Extraction of Solids Using a Dry-Block Heater* Chemical separation via RPG-CMC-455, Rev. 0, *Separation of Uranium and Plutonium for Isotopic Analysis by Mass Spectrometry*
Analysis Procedure:	RPL-TIMS-001, Rev. 0, *Thermal Ionization Mass Spectrometry (TIMS)*
Analysis Date /Reference Date:	January 27 to February 2, 2016
Technician/Analyst:	J.M. Peterson
Report Date:	February 4, 2016
Instrument Electronic Data File:	RPG-IA\Triton\ASR 9916 Sandy Pu
Data Reduction and Reporting Spreadsheet:	RPG-IA\Triton\ASR-9916\Data PSR 9916 Sandy.xlsx
Project 9916 File:	RPL 301/ASO Records/ASR 9916
M&TE Number(s):	TIMS System: Serial Number: 00831

Prepared by: / 2-18-16 **Date** **Technical Reviewer:** C Soderquist / 2-22-2016 **Date**

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Sample Preparation Methods

Plutonium isotopic analysis samples were received in Teflon holders with the material at dryness. The samples were dissolved in a minimum amount of 0.5M HNO_3 (approximately 10-50 uL) at the time of filament loading.

Standards and samples were prepared by dropping 1-5 uL of the prepared solution onto a rhenium filament and lightly heating until dryness (~1 amp of current), the filament was then heated to 2 amps for 10 seconds to fix the sample.

Filaments used for all samples had been degassed for up to 3 hours at 5 amps and then stored in a desiccator until use. All filaments were handled with clean tools to minimize any cross contamination.

All samples submitted for Thermal Ionization Mass Spectrometry (TIMS) analysis were delivered in duplicate with a process blank from sample preparation activities using the procedures, RPG-CMC-129, Rev.0, *HNO3-HCL Acid Extraction of Solids Using a Dry-Block Heater* and RPG-CMC-455, Rev, 0, *Separation of Uranium and Plutonium for Isotopic Analysis by Mass Spectrometry.*

Client ID	RPL Number	TIMS ID
68122-TI-001-F	16-0032	16-0032 F
68122-TI-001-F	16-0032	dup-0032 F #2
68122-TI-001-G	16-0032 (dup)	16-0032 G
68122-TI-001-G	16-0032 (dup)	dup-0032 G #2
---	----	Blk-0032

The TIMS laboratory prepared all submitted samples and one sample in replicate (same solution, different filament);), this was identified as 16-0032 F Rep. Three filaments of SRM-946 and SRM-947 were prepared for the analysis, as well as a blank filament and reagent blank (0.5M HNO_3). Filaments can be analyzed multiple times if need be, this occurs with standards primarily.

All samples were loaded into the TIMS source on January 26, 2016 and pumped down overnight The instrument source pumped down to a pressure of $< <5x10^{-7}$ torr. The ion getter pressure for all analyses was $5x10^{-8}$ torr (or less).

Sample Analysis Method

Plutonium samples and standards were analyzed on a single secondary electron multiplier (SEM) detector targeting a ^{239}Pu signal between 100,000 counts per second (cps) and 500,000 cps. This was achieved by heating an ionizing filament between 4500 mA and 5500 mA and an evaporation filament between 1200 mA to 2000 mA and finding the combination of the two that achieve the greatest signal without evaporating the sample too quickly. The ^{239}Pu peak was

centered in the magnet and the mass calibration updated daily prior to analysis. The signal was then focused and tuned via a series of lenses to optimize the signal strength. Each isotope signal beam was integrated for three seconds with a three second dead time between each scan. Each run of the samples are the sum of forty scans (less scans that are used for detector yield factor). In addition to the target plutonium isotopes, the ^{235}U peak was scanned; this isotope can be used to indicate or correct for contamination in the ^{238}Pu analysis by ^{238}U.

RESULTS AND UNCERTIANTY

The results of the samples analysis is in the table below. The uncertainty of the analysis is provided as the standard deviation (in At% and Wt%) of the pooled measurements made by the instrument (~36 averaged measurement).

	^{239}Pu		^{238}Pu		^{240}Pu		^{241}Pu		^{242}Pu	
	At%	StDev	At%	StDev	At%	StDev	At%	StDev	At%	StDev
16-0032 F	86.32	0.04	0.1456	0.0157	12.92	0.05	0.3890	0.0059	0.2341	0.0059
16-0032 F #2	85.73	0.33	0.8223	0.3877	12.83	0.07	0.3886	0.0065	0.2310	0.0051
dup-0032 G	85.72	0.22	0.8323	0.2710	12.83	0.06	0.3853	0.0044	0.2304	0.0049
dup-0032 G #2	86.05	0.13	0.4359	0.1321	12.89	0.03	0.3872	0.0051	0.2322	0.0049
blk-0032	0.2547	0.1026	99.54	0.2217	0.0489	0.0842	0.0676	0.0750	0.0863	0.1310
	^{239}Pu		^{238}Pu		^{240}Pu		^{241}Pu		^{242}Pu	
	Wt%	StDev	Wt%	StDev	Wt%	StDev	Wt%	StDev	Wt%	StDev
16-0032 F	86.26	0.04	0.1449	0.0157	12.96	0.05	0.3920	0.0060	0.2369	0.0060
16-0032 F #2	85.68	0.33	0.8184	0.3859	12.88	0.07	0.3917	0.0066	0.2338	0.0052
dup-0032 G	85.68	0.22	0.8284	0.2698	12.87	0.06	0.3884	0.0044	0.2332	0.0050
dup-0032 G #2	86.00	0.13	0.4338	0.1314	12.94	0.03	0.3902	0.0052	0.2350	0.0049
blk-0032	0.2557	0.1030	99.54	0.2242	0.0493	0.0849	0.0685	0.0759	0.0877	0.1332

QUALITY CONTROL RESULTS

Laboratory Control Sample (LCS)/Mass Bias Standard (MBS)

Two isotopic standards were run at the opening, closing, and after every 5 unique samples; a mass bias standard (MBS), SRM-946 and a laboratory control sample (LCS), SRM-947. Based on the results of the SRM-946 standard and an acceptance criterion of ± 5% of the decay calculated certified value, it was determined that no mass bias correction factor is needed for these samples or standards. All standards were found to be within the control chart limits established for the method and covered in a memorandum, *Control chart limits for SRM-946 and SRM-947*, delivered and approved January 22, 2016.

SRM-946 Standards	^{239}Pu (% Recovery)	^{238}Pu (% Recovery)	^{240}Pu (% Recovery)	^{241}Pu (% Recovery)	^{242}Pu (% Recovery)
946-STD-02	100.0%	101.0%	99.89%	98.16%	100.4%
946-STD-03	100.0%	101.2%	100.1%	99.71%	101.2%
946-STD-03	100.0%	104.3%	99.67%	99.20%	101.8%

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SRM-946 Control Limits	^{239}Pu (At%)	^{238}Pu (At%)	^{240}Pu (At%)	^{241}Pu (At%)	^{242}Pu (At%)
Upper	86.31	0.1990	12.54	0.6370	0.5981
Lower	86.06	0.1698	12.47	0.4344	0.5838
SRM-947 Control Limits	^{239}Pu (At%)	^{238}Pu (At%)	^{240}Pu (At%)	^{241}Pu (At%)	^{242}Pu (At%)
Upper	79.06	0.2958	19.04	0.7699	1.239
Lower	78.76	0.1587	18.93	0.4515	1.215

SRM-946 Standards	^{239}Pu (At%)	^{238}Pu (At%)	^{240}Pu (At%)	^{241}Pu (At%)	^{242}Pu (At%)
946-STD-02	86.24	0.1832	12.49	0.4891	0.5883
946-STD-03	86.20	0.1835	12.52	0.4968	0.5929
946-STD-03	86.25	0.1892	12.47	0.4943	0.5967
SRM-947 Standards	^{239}Pu (Wt%)	^{238}Pu (Wt%)	^{240}Pu (Wt%)	^{241}Pu (Wt%)	^{242}Pu (Wt%)
947 STD-01	78.94	0.2280	19.03	0.5779	1.231
947 STD-03	78.92	0.2427	19.02	0.5785	1.235
947 STD-03	78.89	0.2360	18.99	0.6595	1.223

The control charting is provided in terms of atom percent (At%) and the client requested that the results be reported in wt%. The control chart, limits established in At% are comparable to wt%, standards that pass the control charting acceptance criteria for wt% will also pass when those limits were established in At%.

Filament Blank(FB):

The background counts per second of the degassed filaments used were assessed by analyzing a blank rhenium filament with no reagent or sample added. The results were acceptable for ^{239}Pu, ^{240}Pu, ^{241}Pu, and ^{242}Pu with counts < 0.1% of the sample's intensity (a standard with the lowest cps analyzed was used for this comparison). The ^{238}Pu and ^{235}U fail this analysis due to environmental contamination of uranium in the fume hoods. Their isotopic contributions are still quite small; 0.2% for ^{238}Pu and 1.5% for ^{235}U and are probably negligible when compared to the diluent and the process chemistry.

Sample ID	^{239}Pu (cps)	^{238}Pu (cps)	^{240}Pu (cps)	^{241}Pu (cps)	^{242}Pu (cps)	^{235}U (cps)
Filament Blank	0.4	2	0.2	0.4	0.2	0.3
946-STD-01	350978	743	50997	2002	2414	23
Percent of intensity	0.0001%	0.2%	0.0004%	0.02%	0.01%	**1.5%**

Diluent Blank (DB):

The samples are prepared in a 0.5M HNO_3 solution. The diluent blank is an evaluation of the background (counts per second) background contamination contribution from the preparation fume hood and reagents. The results were acceptable for ^{239}Pu, ^{240}Pu, ^{241}Pu, and ^{242}Pu with

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counts < 0.1% of the samples' intensity. The ^{238}Pu and ^{235}U fail this analysis due to uranium contamination in the diluent acid. The ^{238}Pu and ^{235}U contributions are 14% for ^{238}Pu and 4% for ^{235}U, but these are not the most significant source of background contamination.

Sample ID	^{239}Pu (cps)	^{238}Pu (cps)	^{240}Pu (cps)	^{241}Pu (cps)	^{242}Pu (cps)	^{235}U (cps)
Diluent Blank	1.3	106	0.3	0.3	0.2	0.9
946-STD-01	350978	743	50997	2002	2414	23
Percent of intensity	0.0004%	**14%**	0.001%	0.01%	0.01%	**4%**

Process Blank (PB):

The separation process for the plutonium is described in procedure RPG-CMC-455 and creates a process blank. This sample provides an indication of the contamination contributed by the separation process.

If the process blank results are compared to the diluent and the filament blank results and the acceptance criteria defined in RPL-TIMS-001, then the results are acceptable for ^{239}Pu, ^{240}Pu, ^{241}Pu, and ^{242}Pu with counts <0.1% of the samples intensity. The ^{238}Pu and ^{235}U both fail the acceptance criteria due to contamination of uranium from the process chemistry. The separation process is the most significant source of unwanted uranium in the analytical samples.

Sample ID	^{239}Pu (cps)	^{238}Pu (cps)	^{240}Pu (cps)	^{241}Pu (cps)	^{242}Pu (cps)	^{235}U (cps)
blk-0032	0.6	269	0.1	0.2	0.2	2.8
946-STD-01	350978	743	50997	2002	2414	23
Percent of intensity	0.0002%	**36%**	0.0003%	0.01%	0.01%	**13%**

Duplicate Sample Relative Percent Difference (RPD):

Both of the samples were provided in duplicate; and these were created during the analytical separation process using procedure RPG-CMC-455. All of the isotopes for the sample and duplicate were within < 1% RPD, based on wt%, except for ^{238}Pu. The ^{238}Pu varied dramatically in the samples resulting in RPD's of 140% for 16-0032 F and 63% for dup-0032-G. This is clearly due to contamination by ^{238}U, this is very difficult to avoid when working with such small amounts of sample, in this case nanogram (or less) amounts of material

Sample ID	^{239}Pu (At%)	^{238}Pu (At%)	^{240}Pu (At%)	^{241}Pu (At%)	^{242}Pu (At%)
16-0032 F	86.32	0.1456	12.92	0.3890	0.2341
16-0032 F #2	85.73	0.8223	12.83	0.3886	0.2310
RPD	0.68%	**140%**	0.66%	0.09%	1.34%
dup-0032 G	85.72	0.8323	12.83	0.3853	0.2304
dup-0032 G #2	86.05	0.4359	12.89	0.3872	0.2322
RPD	0.38%	**63%**	0.49%	0.47%	0.76%

Per client request, these results are presented as wt% Plutonium.

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Replicate Sample Relative Standard Deviation (RSD):

A replicate sample of 16-0032 F was prepared in the TIMS laboratory (same sample, different filament). All of the isotopes for the sample and duplicate were within <1% RSD, based on wt%, except for ^{238}Pu. The ^{238}Pu was measured with a RSD of 107.9 percent. It is noteworthy that this replicate result is less variable than the aggregate error of ^{238}Pu for all the samples received but is still quite high. This is likely due to contamination by ^{238}U in the environment

Sample ID	^{239}Pu (Wt%)	^{238}Pu (Wt%)	^{240}Pu (Wt%)	^{241}Pu (Wt%)	^{242}Pu (Wt%)
16-0032 F	86.26	0.1449	12.96	0.3920	0.2369
16-0032 F #2	85.68	0.8184	12.88	0.3917	0.2338
16-0032 F Rep	86.29	0.1300	12.96	0.3911	0.2368
Average	86.08	0.3644	12.93	0.3916	0.2358
%RSD	0.3993	**107.9**	0.3712	0.1122	0.7532

Instrument Calibration

The mass spectrometer is mass calibrated from ^{23}Na to ^{242}Pu. The instrument is peak centered on ^{239}Pu prior to each analysis. This peak centering updates the mass calibration table each time it is scanned. In addition to peak centering the signal output of the sample is tuned and maximized via a series of lenses. The SEM detector background and yield are assessed on a regular basis, and they typically do not need updating unless significant changes are observed in standard ratios.

Interferences/Resolution

It is known that the ^{241}Pu standard contains the decay product, ^{241}Am, which will skew the analysis of ^{241}Pu. Based on the records provided for the standards the decay can be calculated but these standards have also been separated previously, the dates of which are not fully recorded, this causes errors in the ^{241}Pu calculation. It is believed that the error in the standard ^{241}Pu is not due to any instrument bias and strictly due to the accounting for ^{241}Am in the standards, thus the control limits for ^{241}Pu are larger in range than the other isotopes. The separation performed on the samples prior to TIMS analysis not only separated Pu from U but also removed any ^{241}Am from the Pu fraction. This removes any significant contamination of ^{241}Am in the ^{241}Pu of the samples.

The MBS is used to determine if mass corrections are needed for the standards, the LCS is intended to be the quality check. The LCS required no mass bias, thus no bias factor was applied to the samples. The results of the LCS samples were within the control chart limits specified.

The plutonium results for ^{238}Pu have very high error associated with them. This is likely due to ^{238}U contamination. The ^{235}U is analyzed during this analysis as a means to identify the error in the ^{238}Pu as possibly being associated with uranium. We lack the isotopic ratio of the uranium and thus cannot correct for the uranium contamination but can identify its presence. Additionally the blank analyses conducted provide additional basis for the uranium contamination.

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It was noted that the ^{238}Pu samples showed higher standard deviation in the analysis for samples with ^{238}Pu <0.19 wt%. This can be interpreted in two ways, one the ^{238}U is a minor component and is evaporating off of the filament to a point of depletion, leaving only ^{238}Pu or two, the opposite effect that the Pu is being evaporated leaving the Uranium. The most likely is that the uranium is evaporating off the filament leaving the ^{238}Pu, as all plutonium isotopes will burn off in a similar way and no such variability is observed in the other plutonium isotopes.

All analyses make use of the attached Retarding Potential Quadrupole Lens (RPQ). The RPQ helps in filtering out large peak tailing from impacting smaller peaks. The RPQ is an energy filter that discriminates the dispersed ions that are not in the primary beam, when a large isotopic signal is neighboring a much smaller signal (i.e., ^{239}Pu in ^{238}Pu), the peak tailing of ^{239}Pu can impact the results of the ^{238}Pu. The use of the RPQ makes this potential impact 20 parts per billion and thus peak tailing is not an issue in this analysis.

Attachment: Data Report, Data Summary, Decay Calculations, Certificate of analysis SRM-946 and SRM-947

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Results and QC summary: ASR 9916

Results and Error analysis

	^{239}Pu		^{238}Pu		^{240}Pu		^{241}Pu		^{242}Pu	
	At%	StDev	At%	StDev	At%	StDev	At%	StDev	At%	StDev
16-0082 F	86.32	0.04	0.1456	0.0157	12.92	0.05	0.3880	0.0039	0.2941	0.0059
16-0082 F #2	85.73	0.33	0.8223	0.3677	12.83	0.07	0.3886	0.0095	0.2910	0.0051
dup-0082 G	85.72	0.22	0.8823	0.2710	12.88	0.05	0.3853	0.0044	0.2904	0.0048
dup-0082 G #2	86.05	0.13	0.4359	0.1321	12.89	0.03	0.3872	0.0051	0.2922	0.0049
blk-0082	0.2547	0.1026	99.54	0.2217	0.0489	0.0842	0.0576	0.0750	0.0863	0.1310
	^{239}Pu		^{238}Pu		^{240}Pu		^{241}Pu		^{242}Pu	
	Wt%	StDev	Wt%	StDev	Wt%	StDev	Wt%	StDev	Wt%	StDev
16-0082 F	86.26	0.04	0.1449	0.0157	12.96	0.05	0.3920	0.0040	0.2969	0.0060
16-0082 F #2	85.68	0.33	0.8184	0.3669	12.88	0.07	0.3917	0.0095	0.2938	0.0052
dup-0082 G	85.68	0.22	0.8284	0.2688	12.87	0.05	0.3884	0.0044	0.2932	0.0050
dup-0082 G #2	86.00	0.13	0.4338	0.1314	12.94	0.03	0.3902	0.0052	0.2950	0.0049
blk-0082	0.2557	0.1030	99.54	0.2242	0.0488	0.0849	0.0585	0.0759	0.0877	0.1332

Blanks

Sample ID	^{239}Pu (cps)	^{238}Pu (cps)	^{240}Pu (cps)	^{241}Pu (cps)	^{242}Pu (cps)	^{235}U (cps)
Filament Blank	0.4	2	0.2	0.4	0.2	0.3
946-STD-01	350978	743	50897	2002	2414	23
Percent of intensity	0.0001%	0.2%	0.0004%	0.02%	0.01%	1.5%
Sample ID	^{239}Pu (cps)	^{238}Pu (cps)	^{240}Pu (cps)	^{241}Pu (cps)	^{242}Pu (cps)	^{235}U (cps)
Diluent Blank	1.3	106	0.3	0.3	0.2	0.9
946-STD-01	350978	743	50897	2002	2414	23
Percent of intensity	0.0004%	14%	0.001%	0.01%	0.01%	4%
Sample ID	^{239}Pu (cps)	^{238}Pu (cps)	^{240}Pu (cps)	^{241}Pu (cps)	^{242}Pu (cps)	^{235}U (cps)
blk-0082	0.6	269	0.1	0.2	0.2	2.8
946-STD-01	350978	743	50897	2002	2414	23
Percent of intensity	0.0002%	36%	0.0003%	0.01%	0.01%	13%

RPD Analysis

Sample ID	^{239}Pu (At%)	^{238}Pu (At%)	^{240}Pu (At%)	^{241}Pu (At%)	^{242}Pu (At%)
16-0082 F	86.32	0.1456	12.92	0.3880	0.2941
16-0082 F #2	85.73	0.8223	12.83	0.3886	0.2910
RPD	0.68%	140%	0.68%	0.03%	1.34%
dup-0082 G	85.72	0.8823	12.88	0.3853	0.2904
dup-0082 G #2	86.05	0.4359	12.89	0.3872	0.2922
RPD	0.38%	68%	0.43%	0.47%	0.78%
Sample ID	^{239}Pu (Wt%)	^{238}Pu (Wt%)	^{240}Pu (Wt%)	^{241}Pu (Wt%)	^{242}Pu (Wt%)
16-0082 F	86.26	0.1449	12.95	0.3920	0.2969
16-0082 F #2	85.68	0.8184	12.88	0.3917	0.2938
RPD	0.7%	140%	0.7%	0.1%	1.1%
dup-0082 G	85.68	0.8284	12.87	0.3884	0.2932
dup-0082 G #2	86.00	0.4338	12.94	0.3902	0.2950
RPD	0.4%	68%	0.5%	0.5%	0.6%

Duplicate analysis

Sample ID	^{239}Pu (Wt%)	^{238}Pu (Wt%)	^{240}Pu (Wt%)	^{241}Pu (Wt%)	^{242}Pu (Wt%)
16-0082 F	86.26	0.1449	12.95	0.3920	0.2969
16-0082 F #2	85.68	0.8184	12.88	0.3917	0.2938
16-0082 F Rep	86.29	0.1300	12.95	0.3911	0.2968
Average	86.08	0.3644	12.93	0.3916	0.2958
%RSD	0.3998	107.9	0.3712	0.1122	0.7532

Standards and LCS analysis

Name	^{239}Pu (At%)	^{238}Pu (At%)	^{240}Pu (At%)	^{241}Pu (At%)	^{242}Pu (At%)
SRM 949	86.23	0.19	12.50	0.49	0.59
RSD	0.0003	0.02	0.002	0.01	0.01
Certified Value	86.23	0.1814	12.51	0.4882	0.5860
Recovery	100.0%	102.1%	99.9%	99.0%	101.1%
Control Limits	^{239}Pu (At%)	^{238}Pu (At%)	^{240}Pu (At%)	^{241}Pu (At%)	^{242}Pu (At%)
Upper	86.31	0.1980	12.54	0.6070	0.5881
Lower	86.06	0.1698	12.47	0.4344	0.5838
Name	^{239}Pu (At%)	^{238}Pu (At%)	^{240}Pu (At%)	^{241}Pu (At%)	^{242}Pu (At%)
SRM 947	78.92	0.24	19.01	0.61	1.23
RSD	0.0003	0.03	0.001	0.03	0.01
Certified Value	78.93	0.2180	19.05	0.5681	1.231
Recovery	100.0%	108.1%	99.8%	106.4%	99.9%
Control Limits	^{239}Pu (At%)	^{238}Pu (At%)	^{240}Pu (At%)	^{241}Pu (At%)	^{242}Pu (At%)
Upper	79.05	0.30	19.04	0.77	1.24
Lower	78.76	0.16	18.98	0.45	1.21
Pooled RSD	0.0004	0.0388	0.0028	0.0488	0.0097

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	^{239}Pu (At%)	^{238}Pu (At%)	^{240}Pu (At%)	^{241}Pu (At%)	^{242}Pu (At%)	^{239}Pu (Wt%)	^{238}Pu (Wt%)	^{240}Pu (Wt%)	^{241}Pu (Wt%)	^{242}Pu (Wt%)	^{235}U (CPS)
Position 1	13.31	52.67	6.77	10.43	16.81	13.32	52.62	6.78	10.68	16.59	0.3316
Fillament Blank	^{239}Pu (At%)	^{238}Pu (At%)	^{240}Pu (At%)	^{241}Pu (At%)	^{242}Pu (At%)	^{239}Pu (Wt%)	^{238}Pu (Wt%)	^{240}Pu (Wt%)	^{241}Pu (Wt%)	^{242}Pu (Wt%)	^{235}U (CPS)
Position 12	78.94	0.2280	19.03	0.5779	1.231	78.86	0.227	19.088	0.582	1.245	22.5
SRM-947	^{239}Pu (At%)	^{238}Pu (At%)	^{240}Pu (At%)	^{241}Pu (At%)	^{242}Pu (At%)	^{239}Pu (Wt%)	^{238}Pu (Wt%)	^{240}Pu (Wt%)	^{241}Pu (Wt%)	^{242}Pu (Wt%)	^{235}U (CPS)
Position 3	86.24	0.1832	12.49	0.4891	0.5883	86.19	0.1823	12.5394	0.4928	0.5953	1.065
SRM-946	^{239}Pu (At%)	^{238}Pu (At%)	^{240}Pu (At%)	^{241}Pu (At%)	^{242}Pu (At%)	^{239}Pu (Wt%)	^{238}Pu (Wt%)	^{240}Pu (Wt%)	^{241}Pu (Wt%)	^{242}Pu (Wt%)	^{235}U (CPS)
Position 5	86.32	0.1456	12.92	0.3890	0.2341	86.26	0.1449	12.96	0.3920	0.2369	0.8082
16-0032 F	36 (40)	36 (40)	36 (40)	36 (40)	36 (40)	36 (40)	36 (40)	36 (40)	36 (40)	36 (40)	40 (40)
Scans accepted (Scans run)	0.0445	0.0157	0.0483	0.0059	0.0059	0.0446	0.0157	0.0484	0.0060	0.0060	0.4886
Stdev	^{239}Pu (At%)	^{238}Pu (At%)	^{240}Pu (At%)	^{241}Pu (At%)	^{242}Pu (At%)	^{239}Pu (Wt%)	^{238}Pu (Wt%)	^{240}Pu (Wt%)	^{241}Pu (Wt%)	^{242}Pu (Wt%)	^{235}U (CPS)
Position 6	85.73	0.8223	12.83	0.3886	0.2310	85.68	0.8184	12.88	0.3917	0.2338	10.39
16-0032 F #2	36 (40)	36 (40)	36 (40)	36 (40)	36 (40)	36 (40)	36 (40)	36 (40)	36 (40)	36 (40)	40 (40)
Scans accepted (Scans run)	0.3312	0.3877	0.0710	0.0065	0.0051	0.3294	0.3859	0.0711	0.0066	0.0052	7.09
Stdev	^{239}Pu (At%)	^{238}Pu (At%)	^{240}Pu (At%)	^{241}Pu (At%)	^{242}Pu (At%)	^{239}Pu (Wt%)	^{238}Pu (Wt%)	^{240}Pu (Wt%)	^{241}Pu (Wt%)	^{242}Pu (Wt%)	^{235}U (CPS)
Position 7	85.72	0.8323	12.83	0.3853	0.2304	85.68	0.8284	12.87	0.3884	0.2332	13.03
dup-0032 G	36 (40)	36 (40)	36 (40)	36 (40)	36 (40)	36 (40)	36 (40)	36 (40)	36 (40)	36 (40)	40 (40)
Scans accepted (Scans run)	0.2228	0.2710	0.0553	0.0044	0.0049	0.2216	0.2698	0.0554	0.0044	0.0050	5.187
Stdev	^{239}Pu (At%)	^{238}Pu (At%)	^{240}Pu (At%)	^{241}Pu (At%)	^{242}Pu (At%)	^{239}Pu (Wt%)	^{238}Pu (Wt%)	^{240}Pu (Wt%)	^{241}Pu (Wt%)	^{242}Pu (Wt%)	^{235}U (CPS)
Position 8	86.05	0.4359	12.89	0.3872	0.2322	86.00	0.4338	12.94	0.3902	0.2350	6.782
dup-0032 G #2	36 (40)	36 (40)	36 (40)	36 (40)	36 (40)	36 (40)	36 (40)	36 (40)	36 (40)	36 (40)	40 (40)
Scans accepted (Scans run)	0.1264	0.1321	0.0303	0.0051	0.0049	0.1258	0.1314	0.0304	0.0052	0.0049	2.879
Stdev	^{239}Pu (At%)	^{238}Pu (At%)	^{240}Pu (At%)	^{241}Pu (At%)	^{242}Pu (At%)	^{239}Pu (Wt%)	^{238}Pu (Wt%)	^{240}Pu (Wt%)	^{241}Pu (Wt%)	^{242}Pu (Wt%)	^{235}U (CPS)
Position 4	86.20	0.1835	12.52	0.4968	0.5929	86.15	0.1826	12.57	0.5006	0.6000	151.5
SRM-946	^{239}Pu (At%)	^{238}Pu (At%)	^{240}Pu (At%)	^{241}Pu (At%)	^{242}Pu (At%)	^{239}Pu (Wt%)	^{238}Pu (Wt%)	^{240}Pu (Wt%)	^{241}Pu (Wt%)	^{242}Pu (Wt%)	^{235}U (CPS)
Position 14	78.92	0.2427	19.02	0.5785	1.235	78.84	0.2414	19.08	0.5827	1.250	59.33
SRM-947	^{239}Pu (At%)	^{238}Pu (At%)	^{240}Pu (At%)	^{241}Pu (At%)	^{242}Pu (At%)	^{239}Pu (Wt%)	^{238}Pu (Wt%)	^{240}Pu (Wt%)	^{241}Pu (Wt%)	^{242}Pu (Wt%)	^{235}U (CPS)
Position 10	86.34	0.1306	12.91	0.3881	0.2340	86.29	0.1300	12.96	0.3911	0.2368	0.5177
16-0032 F Rep	36 (40)	36 (40)	36 (40)	36 (40)	36 (40)	36 (40)	36 (40)	36 (40)	36 (40)	36 (40)	40 (40)
Scans accepted (Scans run)	0.0522	0.0118	0.0502	0.0090	0.0078	0.0523	0.0117	0.0503	0.0091	0.0079	0.4645
Stdev	^{239}Pu (At%)	^{238}Pu (At%)	^{240}Pu (At%)	^{241}Pu (At%)	^{242}Pu (At%)	^{239}Pu (Wt%)	^{238}Pu (Wt%)	^{240}Pu (Wt%)	^{241}Pu (Wt%)	^{242}Pu (Wt%)	^{235}U (CPS)
Position 11	1.169	98.10	0.3049	0.2562	0.1676	1.174	98.09	0.3074	0.2594	0.1704	0.8876
Diluent Blank	36 (40)	36 (40)	36 (40)	36 (40)	36 (40)	36 (40)	36 (40)	36 (40)	36 (40)	36 (40)	40 (40)
Scans accepted (Scans run)	0.6358	0.9612	0.2508	0.2338	0.2623	0.6383	0.9683	0.2528	0.2367	0.2667	0.5918
Stdev	^{239}Pu (At%)	^{238}Pu (At%)	^{240}Pu (At%)	^{241}Pu (At%)	^{242}Pu (At%)	^{239}Pu (Wt%)	^{238}Pu (Wt%)	^{240}Pu (Wt%)	^{241}Pu (Wt%)	^{242}Pu (Wt%)	^{235}U (CPS)
Position 9	99.54	0.2547	0.0489	0.0676	0.0863	0.2557	99.5388	0.0493	0.0685	0.0877	2.8440
blk-0032	36 (40)	36 (40)	36 (40)	36 (40)	36 (40)	36 (40)	36 (40)	36 (40)	36 (40)	36 (40)	40 (40)
Scans accepted (Scans run)	0.1026	0.2217	0.0842	0.0750	0.1310	0.1030	0.2242	0.0849	0.0759	0.1332	1.4800
Stdev	^{239}Pu (At%)	^{238}Pu (At%)	^{240}Pu (At%)	^{241}Pu (At%)	^{242}Pu (At%)	^{239}Pu (Wt%)	^{238}Pu (Wt%)	^{240}Pu (Wt%)	^{241}Pu (Wt%)	^{242}Pu (Wt%)	^{235}U (CPS)
Position 4	86.25	0.1892	12.47	0.4943	0.5967	86.20	0.1883	12.51	0.4981	0.6038	0.1414
SRM-946	^{239}Pu (At%)	^{238}Pu (At%)	^{240}Pu (At%)	^{241}Pu (At%)	^{242}Pu (At%)	^{239}Pu (Wt%)	^{238}Pu (Wt%)	^{240}Pu (Wt%)	^{241}Pu (Wt%)	^{242}Pu (Wt%)	^{235}U (CPS)
Position 14	78.89	0.2360	18.99	0.6595	1.2230	78.82	0.2348	19.05	0.6644	1.237	132.8
SRM-947											

Title:	Radiochemical data reduction
Filename:	ASR 9916 Data Reduction.xlsx
Revision:	0
Date Prepared:	1/21/2016
Prepared By:	SK Fiskum
Purpose:	The purpose of this spreadsheet is to calculate the activity concentration as a wet settled solids, total slurry concentration from the reported dry solids concentrations.
Approach:	Centrifuged solids mass and volume data are copied from 68122-TI-001 Data Reduction Final. The dry wt % solids was added from the ASO report for ASR 9916. The GEA, alpha-emitter, and Sr-90 data reported by ASO was converted to total uCi in the acid digest and the UDS. The total uCi content in the dissolved fraction was calculated (dissolved uCi/total uCi) for each analyte. More than 99% of the activity dissolved except for Co-60 where 91 to 93% of the Co-60 dissolved. The dissolved isotope activity was converted to the centrifuged solids mass basis by dividing the total uCi by the wet centrifuged solids mass. A similar calculation was conducted to evaluate the activity concentration as a function of wet centrifuged solids volume. Finally, the isotopic ratios of Pu was compared to Sr-90 and Cs-137 to see if there was a different fractionation in the sand filter solids relative to the K Basin sludge. In this case, the sand filter solids relative Pu content was significantly higher than that of the sludge and suspended solids from the settling tests.

Grav shaded values imported from 68122-TI-001.

Parent Container ID	Container Type	Gross Mass with Packaging, g	Packaging Mass, g	Gross Sample Container Mass, g	Empty Mass with Packaging, g	Packaging Mass, g	Empty Sample Container Mass, g	Net Slurry Mass, g	Centrifuged Solids, wt%	Dry Solids, wt%
KW-105SFBW-001	1-L poly bottle	887.0	31.2	855.8	123.1	31.5	91.6	764.2	0.11%	0.0146%

Centrifuged sludge volume

Subsample Container ID	Container Type	Tare, g	Gross Mass, g	Sludge Volume, mL*	Total Slurry Volume, mL	Net Slurry Mass, g	Net Centrifuged Sludge Mass, g*	Centrifuged Sludge Density, g/mL*
68122-TI-001-F	10-mL glass cent tube	20.6859	21.2217	0.40	0.50	0.5358	0.44	1.09**
68122-TI-001-G	10-mL glass cent tube	20.6253	21.1095	0.40	0.50	0.4842	0.38	0.96
Sum				0.80			0.82	

Assumption: the water density is 1 g/mL.
*The total sludge volume determination was slightly confounded by the slanted surface of sludge in the centrifuge tube.
**The third significant figure is shown for information only.

ASR 9916 dry solids mass data

		Dry solids mass, g	UDS (post acid digestion), g	wt% UDS	Mass loss on drying
68122-TI-001-F	16-0032	0.0545	0.0039	6.7%	87%
68122-TI-001-G	16-0032 dup	0.0568	0.0039	6.4%	85%
Sum		0.1113	0.0078	6.5%	

Uncertainty estimation.

Sample ID		68122-TI-001-F			68122-TI-001-G		
Backwash Sand Filter Sample		Wet centrifuged solids			Wet centrifuged solids		
Sample information		Sample			Duplicate		
Container information		10-mL glass cent tube			10-mL glass cent tube		
	Units	Value	Uncertainty		Value	Uncertainty	
Container tare mass	g	**20.6859**	0.0021	0.010%	**20.6253**	0.0021	0.010%
Vial + cent solids + aq	g	**21.2217**	0.0021	0.010%	**21.1095**	0.0021	0.010%
Cent sludge volume	mL	**0.40**	0.05	12.5%	**0.40**	0.05	12.5%
Slurry (total) solids volume	mL	**0.50**	0.05	10.0%	**0.50**	0.05	10.0%
Liquid density	g/mL	1.0	0.005	0.5%	1.0	0.005	0.5%
Liquid volume	mL	0.10	0.07	70.7%	0.1	0.07	70.7%
Liquid mass	g	0.10	0.07	70.7%	0.1	0.07	70.7%
Cent sludge mass	g	0.44	0.07	**16.2%**	0.38	0.1	**18.4%**
Cent sludge density	g/mL	1.09	0.22	20.5%	0.96	0.21	22.3%

Balances	ASO	Project	
Balance ID	1113292667	N04143	
Balance range	299.9980	299.9978	lower control bound
	300.0022	300.0020	upper control bound
	0.0042	0.0042	Range interval
	0.0021	0.0021	Half range inerval (balance uncertainty)

Sample ID		16-0032			16-0032 dup		
Backwash Sand Filter Sample		Dry solids			Dry Solids		
Sample information		Sample			Duplicate		
Container information		10-mL glass cent tube			10-mL glass cent tube		
	Units	Value	Uncertainty		Value	Uncertainty	
Container tare mass	g	**20.6859**	0.0021	0.01%	**20.6253**	0.0021	0.01%
Dry solids mass	g	**20.7404**	0.0021	0.01%	**20.6821**	0.0021	0.01%
Cent sludge mass	g	0.0545	0.0030	**5.4%**	0.0568	0.0030	**5.2%**

ASR 9916 Reported Data

	Dissolved solids, dry mass basis				Undissolved solids (UDS)			
	68122-TI-001-F	68122-TI-001-G			68122-TI-001-F	68122-TI-001-G		
GEA analyte	16-0032 uCi/g dry	16-0032dup uCi/g dry	Average uCi/g dry	RPD	16-0032 uCi/g dry	16-0032dup uCi/g dry	Average uCi/g dry	
Mn-54	<6.E-3	<4.E-3			8.28E-5	<3.E-4		
Co-60	1.08E-1	9.91E-2	1.04E-1	8.6%	1.13E-1	1.34E-1	1.24E-1	17%
Sb-125	<1.E-1	<8.E-2			3.18E-3	4.59E-3		
Cs-134	<7.E-3	<5.E-3			<3.E-4	<3.E-4		
Cs-137	1.02E+2	9.49E+1	9.85E+1	7.2%	1.01E+1	1.07E+1	1.04E+1	5.8%
Eu-152	3.88E-2	3.13E-2	3.51E-2	21%	2.31E-3	2.06E-3	2.19E-3	11%
Eu-154	1.54E+0	1.46E+0	1.50E+0	5.3%	3.26E-2	3.41E-2	3.34E-2	4.5%
Eu-155	2.28E-1	2.55E-1	2.42E-1	11%	4.18E-3	5.03E-3	4.61E-3	18%
Am-241	9.64E+1	1.06E+2	1.01E+2	9.5%	1.25E+0	1.78E+0	1.52E+0	35%
Dry Mass basis, g >>	**0.0545**	**0.0568**			**0.0039**	**0.0039**		

Multiply uCi/g concentration by the sample mass.

									Dissolved fraction See note a. 68122-TI-001-F	See note a. 68122-TI-001-G	**UDS fraction** See note b. 68122-TI-001-F	See note b. 68122-TI-001-G	
	total uCi	total uCi			total uCi	total uCi			16-0032	16-0032dup	16-0032	16-0032dup	Average
Mn-54	<3.E-4	<2.E-4			3.23E-7	<1.E-6							
Co-60	5.89E-3	5.63E-3	5.76E-3	4.5%	4.41E-4	5.23E-4	4.82E-4	17%	93.0%	91.5%	7.0%	8.5%	7.7%
Sb-125	<5.E-3	<5.E-3			1.24E-5	1.79E-5							
Cs-134	<4.E-4	<3.E-4			<1.E-6	<1.E-6							
Cs-137	5.56E+0	5.39E+0	5.47E+0	3.1%	3.94E-2	4.17E-2	4.06E-2	5.8%	99.3%	99.2%	0.70%	0.77%	0.7%
Eu-152	2.11E-3	1.78E-3	1.95E-3	17%	9.01E-6	8.03E-6	8.52E-6	11%	99.6%	99.6%	0.42%	0.45%	0.4%
Eu-154	8.39E-2	8.29E-2	8.34E-2	1.2%	1.27E-4	1.33E-4	1.30E-4	4.5%	99.8%	99.8%	0.15%	0.16%	0.2%
Eu-155	1.24E-2	1.45E-2	1.35E-2	15%	1.63E-5	1.96E-5	1.80E-5	18%	99.9%	99.9%	0.13%	0.14%	0.1%
Am-241	5.25E+0	6.02E+0	5.64E+0	14%	4.88E-3	6.94E-3	5.91E-3	35%	99.9%	99.9%	0.09%	0.12%	0.1%

Note a) total uCi in solution / sum of solution plus undissolved uCi.
Note b) total uCi in UDS / sum of solution plus UDS in uCi.

Calculate the analyte concentration on a wet settled solids mass basis
Divide the total uCi by the new mass basis of wet settled solids

	Dissolved solids, wet mass basis				
Wet Mass basis, g >>	**0.44**	**0.38**	Average		
	16-0032 uCi/g wet	16-0032dup uCi/g wet	uCi/g wet		
Mn-54	<8.E-4	<6.E-4			
Co-60	1.45E-2	1.60E-2	1.53E-2	9.8%	(includes 7% in UDS)
Sb-125	<1.E-2	<1.E-2			
Cs-134	<9.E-4	<7.E-4			
Cs-137	1.28E+1	1.40E+1	1.34E+1	9.5%	
Eu-152	4.85E-3	4.63E-3	4.74E-3	4.7%	
Eu-154	1.93E-1	2.16E-1	2.04E-1	11.4%	
Eu-155	2.85E-2	3.77E-2	3.31E-2	28%	
Am-241	1.21E+1	1.57E+1	1.39E+1	26%	

Calculate the analyte concentration on a wet settled solids volume basis
Divide the total uCi by the volume basis of wet settled solids

	Dissolved solids, wet volume basis				
Volume basis, mL >>	**0.40**	**0.40**	Average		
	16-0032 uCi/mL wet	16-0032dup uCi/mL wet	uCi/g wet		
Mn-54	<8.E-4	<6.E-4			
Co-60	1.58E-2	1.54E-2	1.56E-2	2.8%	(includes 7% in UDS)
Sb-125	<1.E-2	<1.E-2			
Cs-134	<1.E-3	<7.E-4			
Cs-137	1.39E+1	1.35E+1	1.37E+1	3.1%	
Eu-152	5.29E-3	4.44E-3	4.87E-3	17%	
Eu-154	2.10E-1	2.07E-1	2.09E-1	1.2%	
Eu-155	3.11E-2	3.62E-2	3.36E-2	15%	
Am-241	1.31E+1	1.51E+1	1.41E+1	14%	

Shading indicates the RPD is >20%.

ASR 9916 Reported Data

	Dissolved solids, dry mass basis			
	68122-TI-001-F	68122-TI-001-G		
	16-0032	16-0032dup	Average	RPD
Analyte	uCi/g dry	uCi/g dry	uCi/g dry	
Sr-90	1.88E+1	1.88E+1	1.88E+1	0.5%
Pu-238	2.63E+1	2.61E+1	2.62E+1	0.8%
Pu-239+240	1.99E+2	2.00E+2	2.00E+2	0.4%
Np-237	1.80E-3	2.09E-3	1.94E-3	15%
Am-241 (AEA)	9.39E+1	8.69E+1	9.04E+1	7.7%
Am-241 (GEA)	9.64E+1	1.06E+2	1.01E+2	9.5%
Dry Mass basis, g >>	**0.0545**	**0.0568**		

Multiply by the sample mass

	total uCi	total uCi	Average uCi/g dry	RPD
Sr-90	1.02E+0	1.07E+0	1.05E+0	4.6%
Pu-238	1.43E+0	1.48E+0	1.46E+0	3.4%
Pu-239+240	1.09E+1	1.14E+1	1.11E+1	4.5%
Np-237	9.81E-5	1.18E-4	1.08E-4	19%
Am-241 (AEA)	5.12E+0	4.94E+0	5.03E+0	3.6%
Am-241 (GEA)	5.25E+0	6.02E+0	5.64E+0	14%

Calculate the analyte concentration on a wet settled solids mass basis
Divide the total uCi by the new mass basis of wet settled solids

	Dissolved solids, wet mass basis			
Wet Mass basis, g >>	**0.44**	**0.38**		
	16-0032	16-0032dup	Average	
	uCi/g wet	uCi/g wet	uCi/g wet	RPD
Sr-90	2.35E+0	2.79E+0	2.57E+0	17%
Pu-238	3.29E+0	3.86E+0	3.57E+0	16%
Pu-239+240	2.49E+1	2.96E+1	2.73E+1	17%
Np-237	2.25E-4	3.08E-4	2.67E-4	31%
Am-241 (AEA	1.17E+1	1.28E+1	1.23E+1	9.0%
Am-241 (GEA)	1.21E+1	1.57E+1	1.39E+1	26%

Shading indicates the RPD is >20%.

Calculate the analyte concentration on a wet settled solids volume basis
Divide the total uCi by the volume basis of wet settled solids

	Dissolved solids, wet volume basis			
Volume basis, mL >>	**0.40**	**0.40**		
	16-0032	16-0032dup	Average	
	uCi/mL wet	uCi/mL wet	uCi/g wet	RPD
	2.56E+0	2.68E+0	2.62E+0	5%
	3.58E+0	3.71E+0	3.64E+0	3%
	2.72E+1	2.84E+1	2.78E+1	5%
	2.45E-4	2.96E-4	2.71E-4	19%
	1.28E+1	1.23E+1	1.26E+1	4%
	1.31E+1	1.51E+1	1.41E+1	14%

File: ASR 9916 Data Reduction.xlsx
Worksheet: AEA and Sr-90 data

Purpose:
Evaluate the isotope fractionation from various studies and compare with observed fractionation in the Sand Filter Backwash solids sample.

	Ref. date: 24-Nov-15				PNNL-20650, R2, Table 5.23			Decay correction			PNNL-19035. R1, Table 7.13	Decay correction
	Dissolved solids, dry mass basis			Constants for decay correction	KW210 SCC						KW220-SCC	
	68122-TI-001-F	68122-TI-001-G		Decay calculation: $a = aoExp(-ln2*\Delta T/t_{1/2})$	Ref. date: 25-May-11			ΔT = 4.50 years			GEA Ref. date: 13-Jul-09	ΔT = GEA 6.37 years
					Container composite, -500 micron sieve fraction						Pu Ref. date: 16-Nov-09	ΔT = Pu 6.02 years
Analyte	16-0032 uCi/g dry	16-0032dup uCi/g dry	Average	$t_{1/2}$, years	TI008-SA uCi/g dry	TI008-SB uCi/g dry	Average uCi/g dry	TI008-SA uCi/g dry	TI008-SB uCi/g dry	Average uCi/g dry	TI009-SB-s uCi/g dry	TI009-SB-s uCi/g dry
Sr-90	1.88E+01	1.88E+01	1.88E+01	28.5	45.6	47.2	46.4	40.9	42.3	41.6		
Pu-238	2.63E+01	2.61E+01	2.62E+01									
Pu-239+240	1.99E+02	2.00E+02	2.00E+02	24,000	50.3	50.5	50.4	50.3	50.5	50.4	70.7	70.7
Np-237	1.80E-03	2.09E-03	1.94E-03									
Am-241 (AEA)	9.39E+01	8.69E+01	9.04E+01	432.7								
Am-241 (GEA)	9.64E+1	1.06E+2	1.01E+02	432.7	39.4	36.5	37.95	39.1	36.2	37.7	73.1	72.4
Co-60	1.08E-1	9.91E-2	1.04E-01									
Cs-137	1.02E+2	9.49E+1	9.85E+01	30	208	203	205.5	187.5	182.9	185.2	357.0	308.2
Eu-152	3.88E-2	3.13E-2	3.51E-02									
Eu-154	1.54E+0	1.46E+0	1.50E+00									
Eu-155	2.28E-1	2.55E-1	2.42E-01									
Pu239+240/Sr-90	10.63	10.62	10.63					1.23	1.19	1.21		Sr-90 not analyzed
Pu239+240/Cs-137	1.96	2.11	2.03					0.27	0.28	0.27		0.23
Sand filter ratio / sample ratio (Sr-90)								8.6	8.9	8.8		
Sand filter ratio / sample ratio (Cs-137)								7.6	7.4	7.5		8.9

$t_{1/2}$ (half life in years) was taken from Browne and Firestone, Table of Radioactive Isotopes, John Wiley and Sons, New York, 1986.

The Pu-239+240 decay correction calculation was based on the Pu-239 half life only. These samples contained more Pu-239 than Pu-240.
The Pu-240 half-life is 6563 years; replacing the Pu half-life to 6369 years does not change the decay-corrected Pu activity concentration.

Conclusion: The isotopic ratio observed in the backwash solids do not look like that of the containerized sludge material. The Pu-239+240 is enriched relative to Cs-137 and Sr-90 in the sand filter backwash solids

	PNNL-20470 R1, Table 4.3				Decay correction				PNNL-19035 R1, Table 4.3											
	KW230 core samples														Decay correction					
	Ref. date: 11-Jan-11				Δ T = 4.87 years				Ref. date: 13-Jul-09						Δ T = 6.37 years					
	Core A2	Core A3	Core B2	Core B4	Core A2	Core A3	Core B2	Core B4	KW240-A2	KW240-B1	KW250-A4	KW250-B2	KW260-A1	KW260-B3	KW240-A2	KW240-B1	KW250-A4	KW250-B2	KW260-A1	KW260-B3
	TI023-1C	TI024-2C	TI024-3C	TI024-4C	TI023-1C	TI024-2C	TI024-3C	TI024-4C	TI005-1A	TI005-2A	TI006-2A	TI006-1A	TI007-1A	TI007-2A	TI005-1A	TI005-2A	TI006-2A	TI006-1A	TI007-1A	TI007-2A
Analyte	uCi/g dry	uCi/g dry	uCi/g dry	uCi/g dry	uCi/g dry	uCi/g dry	uCi/g dry	uCi/g dry	uCi/g dry	uCi/g dry	uCi/g dry	uCi/g dry	uCi/g dry	uCi/g dry	uCi/g dry	uCi/g dry	uCi/g dry	uCi/g dry	uCi/g dry	uCi/g dry
Sr-90	1090	834	1080	838	968	741	959	744	93.4	80.8	96.7	86.9	98.3	90.4	80.0	69.2	82.8	74.4	84.2	77.4
Pu-238																				
Pu-239+240	131	73.7	133	92.3	131	73.7	133	92.3	8.61	7.06	8.71	9.92	10.6	11.7	8.61	7.06	8.71	9.92	10.6	11.7
Np-237																				
Am-241 (AEA)	134	79.9	129	94.7	133	79.3	128	94.0												
Am-241 (GEA)	129	74.7	125	93.6	128	74.1	124	92.9	10.2	7.41	8.46	10.2	10.8	11.1	10.1	7.3	8.4	10.1	10.7	11.0
Co-60																				
Cs-137	356	615	390	471	318	550	349	421	94.4	77.8	111	70.4	93.4	95.8*	81.5	67.2	95.8	60.8	80.6	82.7
Eu-152																				
Eu-154																				
Eu-155																				
Pu239+240/Sr-90					0.14	0.10	0.14	0.12							0.11	0.10	0.11	0.13	0.13	0.15
Pu239+240/Cs-137					0.41	0.13	0.38	0.22							0.11	0.11	0.09	0.16	0.13	0.14
Sand filter ratio / sample ratio (Sr-90)					78.6	106.9	76.7	85.7							98.8	104.2	101.1	79.8	84.4	70.4
Sand filter ratio / sample ratio (Cs-137)					4.9	15.2	5.3	9.3							19.2	19.3	22.4	12.5	15.5	14.4

*The Cs concentration is the average of TI007-2A, TI007-2C and TI007-2C

ids.

	PNNL-19035 R1, Table 5.20, 5.21, 5.22						PNNL-19213, Table 4.15									
	KW240	KW250	KW260	Decay correction			KW240	KW250	KW260	KW220A3	Decay correction					
	Ref. date: 13-Jul-09			Δ T = 6.37 years			Ref. date: 13-Jul-09				Δ T = 6.37 years					
	Container composite, -500 micron sieve fractions						Filtered solids from settling test suspension, dry mass basis									
Analyte	TI010-SD uCi/g dry	TI011-SB uCi/g dry	TI012-SB uCi/g dry	TI010-SD uCi/g dry	TI011-SB uCi/g dry	TI012-SB uCi/g dry	SSK240-S uCi/g slurry	SSK250-S uCi/g slurry	SSK260-S uCi/g slurry	SSKW220A3-S uCi/g slurry	SSK240-S uCi/g slurry	SSK250-S uCi/g slurry	SSK260-S uCi/g slurry	SSKW220A3-S uCi/g slurry		
Sr-90																
Pu-238																
Pu-239+240	8.73	8.17	12.0	8.73	8.17	12.0	3.12E+1	2.15E+1	3.89E+1	1.70E+2	3.12E+1	2.15E+1	3.89E+1	1.70E+2		
Np-237																
Am-241 (AEA)																
Am-241 (GEA)	9.61	8.37	13.8	9.54	8.30	13.7	2.88E+1	2.29E+1	4.15E+1	1.46E+2	2.85E+1	2.27E+1	4.11E+1	1.45E+2		
Co-60																
Cs-137	83.8	89.4	84.9	74.9	79.9	75.9	9.83E+1	9.97E+1	9.77E+1	4.20E+2	8.49E+1	8.61E+1	8.43E+1	3.63E+2		
Eu-152																
Eu-154																
Eu-155																
															Max	Min
Pu239+240/Sr-90				Sr-90 not analyzed							Sr-90 not analyzed				1.23	0.10
Pu239+240/Cs-137				0.12	0.10	0.16					0.37	0.25	0.46	0.47	0.47	0.09
Sand filter ratio / sample ratio (Sr-90)																
Sand filter ratio / sample ratio (Cs-137)				17.4	19.9	12.9					5.5	8.1	4.4	4.3		

Spreadsheet Review Form

Spreadsheet Author Name:	SK Fiskum	Review Date:	2/9/2016
Date Prepared:	1/21/2016	Spreadsheet Subject:	Sand filter back wash solids radiochemical characterization
Reviewer Name:	Rick Shimskey	File Name:	ASR 9916 Data Reduction.xlsx
Reviewer Title:	Research Engineer	Revision No.	0

Scope of Spreadsheet Review: (Check one or more of the following)

☐	General Validation Review: (General review and spot checks)	☐	Independent calculation check (With hand calculations or independent spreadsheet software)
☐	Review of updated spreadsheet/calc (Revised portion only)	☐	Other:
☑	100% Verification Review (Verification of all cells/calculations)		

REVIEW CHECK LIST

Spreadsheet/Calculation Identification

Spreadsheet Information	Yes	No	NA
Title:	x		
Revision Number:	x		
Date Prepared:	x		
Prepared by:	x		
General Statement of Purpose:	x		
General Description of Approach:	x		
Comments:			

Input Values	Yes	No	NA
Are input parameters correct (verified with source?)	x		
Are parameter units consistent?	x		
Are input values properly referenced?	x		
Are input uncertainties correct?	x		
Comments:			

Assumptions	Yes	No	NA
Are assumptions clearly stated?			x
Are assumptions supported and justified?			x
Are assumptions reasonable?			x
Comments:			

Equations/Approach	Yes	No	NA
Are equation algorithms adequately defined?	x		
Are equations properly referenced?	x		
Are limitations of approach/equations identified?	x		
Are equations appropriate?	x		
Are units consistent?	x		
Comments: Equations are simple add/subtract, multiply and divide.			

Results/Conclusions	Yes	No	NA
Are formulas consistent in cells?	x		
Are calculations correct?	x		
Are conclusions consistent with results?			x
Are conclusions consistent with applicable limits?			x
Comments:			

Reviewer Sign/Date: 2/9/16

Filename: ASR 9916 Pu isotopic correction Rev. 1.xlsx
Tab: ASR 9916 Pu isotopic correction Rev. 1.xlsx

The samples were each prepared in duplicate for TIMS analysis.
This spreadsheet tab averages each sample pair.
It also calculates the fraction of Pu-239 relative to the sum of Pu-239 and Pu-240
The average values are rolled up into the next tab, Radionuc. Calc.

	Pu-239	Pu-238	Pu-240	Pu-241	Pu-242
	Atom %				
16-0032 F	86.32	0.1456	12.92	0.3890	0.2341
16-0032 F#2	85.73	0.8223	12.83	0.3886	0.2310
dup-0032 G	85.72	0.8323	12.83	0.3853	0.2304
dup-0032 G #2	86.05	0.4359	12.89	0.3872	0.2322
Average of the replicate preparations					
16-0032 F	86.03	0.4840	12.88	0.3888	0.2326
16-0032 G	85.89	0.6341	12.86	0.3863	0.2313

Atom fraction ^{239}Pu in ^{239,240}Pu
0.869818
0.869766

	Weight %				
16-0032 F	86.26	0.1449	12.96	0.3920	0.2369
16-0032 F#2	85.68	0.8184	12.88	0.3917	0.2338
dup-0032 G	85.68	0.8284	12.87	0.3884	0.2332
dup-0032 G #2	86.00	0.4338	12.94	0.3902	0.2350
Average of the replicate preparations					
16-0032 F	85.97	0.4817	12.92	0.3919	0.2354
16-0032 G	85.84	0.6311	12.91	0.3893	0.2341

Weight fraction ^{239}Pu in ^{239,240}Pu
0.86935
0.86931

Filename: ASR 9916 Pu isotopic correction Rev. 1.xlsx
Tab: ASR 9916 Pu isotopic correction Rev. 1.xlsx

Title: Radionuc. Calc.
Purpose: Convert Pu, Np, and Am analytical data to specific gravimetric and activity bases.
K Basin Backwash Sand Filter Radionuclide Calculations

Date: 02 February 2016
Prep'd. by SK Fiskum
Rev'd. by: CH Delegard
Signature:

Cells for data entry

Comments
Entry Date: 2/4/2016
Filename: ASR 9916 Pu isotopic correction.xlsx
Reporting units are in terms of µg/g dry mass basis.
Microsoft Excel 2003 SP3

Sand filter backwash, Sample KW-105 SFBW-001		
Sample ID	68122-TI-001-F	68122-TI-001-G
ASO ID	16-0032	16-0032 dup
Analysis Results		
TIMS	**Atom%**	**Atom%**
^{238}Pu	0.484	0.634
^{239}Pu	86.03	85.89
^{240}Pu	12.88	12.86
^{241}Pu	0.389	0.386
^{242}Pu	0.233	0.231
AEA	**µCi/g**	**µCi/g**
^{238}Pu	2.63E+1	2.61E+1
$^{239+240}Pu$	1.99E+2	2.00E+2
^{241}Am	9.39E+1	8.69E+1
^{237}Np	1.80E-3	2.09E-3
GEA	**µCi/g**	**µCi/g**
^{60}Co	1.08E-1	9.91E-2
^{134}Cs	<7.E-3	<5.E-3
^{137}Cs	1.02E+2	9.49E+1
^{152}Eu	3.88E-2	3.13E-2
^{154}Eu	1.54E+0	1.46E+0
^{155}Eu	2.28E-1	2.55E-1
^{241}Am	9.64E+1	1.06E+2

Core wash, Sample KW-105 SFBW-001		
Sample ID	68122-TI-001-F	68122-TI-001-G
ASO ID	16-0032	16-0032 dup
Atom fraction ^{239}Pu in $^{239,240}Pu$	0.86982	0.86977
Mass fraction ^{239}Pu in $^{239,240}Pu$	0.86934	0.86929
^{239}Pu, µCi/g sludge	1.287E+02	1.292E+02
^{240}Pu, µCi/g sludge	7.074E+01	7.103E+01
At% $^{238}Pu_{AEA}$, w/ resp. to $^{239}Pu_{TIMS}$	0.0639	0.0631
^{238}Pu, atom%, normalized	0.0642	0.0635
^{239}Pu, atom%, normalized	86.3832	86.3811
^{240}Pu, atom%, normalized	12.9286	12.9343
^{241}Pu, atom%, normalized	0.3904	0.3885
^{242}Pu, atom%, normalized	0.2335	0.2326
Atomic weight Pu	239.20	239.20
^{238}Pu, wt% Pu	0.0639	0.0632
^{239}Pu, wt% Pu	86.3313	86.3292
^{240}Pu, wt% Pu	12.9750	12.9807
^{241}Pu, wt% Pu	0.3935	0.3915
^{242}Pu, wt% Pu	0.2363	0.2354

Average	RPD
0.0638	1.1
86.38	0.002
12.93	0.04
0.3895	0.50
0.2331	0.38
Average	RPD
0.0635	1.1
86.33	0.002
12.98	0.04
0.3925	0.50
0.2359	0.38

Atom fraction ^{239}Pu in $^{239,240}Pu$

0.86982	0.86977 (From TIMS Report)
1.00000	1.00000 Ratio TIMS report/spreadsheet calc

Mass fraction ^{239}Pu in $^{239,240}Pu$

0.86935	0.86931 (From TIMS Report)
1.000007	1.000021 Ratio TIMS report/spreadsheet calc

Slight difference in the mass fraction calculation in the 5th significant figure.This difference is too small to report (0.0007% and 0.0021%)

		Core wash, Sample KW-105 SFBW-001	
	Sample ID	68122-TI-001-F	68122-TI-001-G
	ASO ID	16-0032	16-0032 dup
Isotopic Conc., Weight Basis, µg/g, as reported	Total Pu, ^{238}Pu basis	2.403E+3	2.412E+3
	Total Pu, ^{239,240}Pu basis	2.403E+3	2.412E+3
	^{238}Pu	1.535E+0	1.524E+0
	^{239}Pu	2.075E+3	2.082E+3
	^{240}Pu	3.118E+2	3.131E+2
	^{241}Pu	9.455E+0	9.443E+0
	^{242}Pu	5.679E+0	5.678E+0
	^{237}Np	2.559E+0	2.965E+0
	^{241}Am, AEA	2.736E+1	2.533E+1
	^{241}Am, GEA	2.809E+1	3.089E+1

Isotopic Conc., Activity Basis, µCi/g, as reported	^{238}Pu	2.629E+1	2.609E+1
	^{239}Pu	1.287E+2	1.292E+2
	^{240}Pu	7.074E+1	7.103E+1
	^{241}Pu	9.813E+2	9.800E+2
	^{242}Pu	2.247E-2	2.247E-2
	^{237}Np	1.800E-3	2.086E-3
	^{241}Am, AEA	9.387E+1	8.691E+1
	^{241}Am, GEA	9.640E+1	1.060E+2

		Core wash, Sample KW-105 SFBW-001	
	Sample ID	68122-TI-001-F	68122-TI-001-G
	ASO ID	16-0032	16-0032 dup
Cooling Time, y	By ^{241}Am AEA	28.0	26.9
	By ^{241}Am GEA	28.4	29.9

Isotopic Data				
Isotope	Atomic Mass	$t_{½}$, y	Spec. Act., Ci/g	λ, y^{-1}
^{237}Np	237.0481734	2144000	7.034E-04	3.233E-07
^{238}Pu	238.0495599	87.7	1.712E+01	7.904E-03
^{239}Pu	239.0521634	24110	6.203E-02	2.875E-05
^{240}Pu	240.0538135	6564	2.269E-01	1.056E-04
^{241}Pu	241.0568515	14.290	1.038E+02	4.851E-02
^{242}Pu	242.0587426	373300	3.956E-03	1.857E-06
^{241}Am	241.0568291	432.2	3.431E+00	1.604E-03

The atomic mass data are from Brookhaven National Laboratory, National Nuclear Data Center. Found at:
http://www.nndc.bnl.gov/masses/mass.mas03

The radioactive half-lives are those that Safeguards uses for NMMSS (Nuclear Materials Management & Safeguards System) purposes. From Brookhaven National Laboratory, National Nuclear Data Center, Nuclear Wallet Cards, 6th edition, January 2000. Found at:
http://www.nndc.bnl.gov/wallet/wcdoe.html

Filename: ASR 9916 Pu isotopic correction Rev. 1.xlsx
Tab: ASR 9916 Pu isotopic correction Rev. 1.xlsx

Title: compare to SCS-CON
Purpose Evaluate the isotope fractionation from various studies and compare with observed fractionation in the Sand Filter Backwash solids sample.

Ref. date: 25-Jan-16

Dissolved solids, mass basis

Analyte	68122-TI-001-F 16-0032 uCi/g dry	68122-TI-001-G 16-0032dup uCi/g dry
Pu-238		
Pu-239		
Pu-240		
Pu-241		
Pu-242		
Pu-238	0.0639	0.0632
Pu-239	86.3313	86.3292
Pu-240	12.9750	12.9807
Pu-241	0.3935	0.3915
Pu-242	0.2363	0.2354
Pu-238		
Pu-239		
Pu-240		
Pu-241		
Pu-242		

Constants for decay correction

Decay calculation: $a = aoExp(-ln2*\Delta T/t_{1/2})$

$t_{1/2}$, years	PNNL-20650, R2, Table 4.6 KW210 Representative core samples TI002-A4-B wt%	TI001-B3-B wt%	TI002-B1-E wt%	Decay correction Ref. date: 25-May-11 Δ T = 4.67 years TI002-A4-B wt%	TI001-B3-B wt%	TI002-B1-E wt%
87.7	0.0675	0.0660	0.0627	0.0651	0.0636	0.0604
24110	85.872	86.198	86.378	85.8605	86.1864	86.3664
6564	13.266	12.964	12.839	13.2595	12.9576	12.8327
14.290	0.537	0.539	0.490	0.4281	0.4297	0.3907
373300	0.257	0.232	0.230	0.2570	0.2320	0.2300
sum	99.9995	99.9990	99.9997	99.8701	99.8694	99.8802
			Renormalized	0.0651	0.0637	0.0605
				85.9721	86.2992	86.4700
				13.2767	12.9746	12.8481
				0.4287	0.4303	0.3911
				0.2573	0.2323	0.2303

PNNL-19035. R1, Table 6.6 KW220 Core samples	Decay correction Ref. date: 19-Nov-09 Δ T 6.18 years					
TI003-1D-s wt%	TI003-2D-s wt%	TI004-1D-d wt%	TI003-1D-s wt%	TI003-2D-s wt%	TI004-1D-d wt%
0.0683	0.0663	0.0670	0.0650	0.0631	0.0638
86.0572	86.0973	86.1575	86.0419	86.0820	86.1422
13.0949	13.0522	13.0042	13.0864	13.0437	12.9957
0.5377	0.5418	0.5368	0.3984	0.4014	0.3977
0.2419	0.2423	0.2345	0.2419	0.2423	0.2345
100.0000	99.9999	100.0000	99.8336	99.8325	99.8339
			0.0652	0.0632	0.0639
			86.1853	86.2264	86.2855
			13.1082	13.0656	13.0173
			0.3991	0.4021	0.3984
			0.2423	0.2427	0.2349

Ratio: Sandfilter sample result to SCS-CON sample result
Shading reflects ratios that exceeded +/- 5%

0.9809	1.0032	1.0561	
1.0042	1.0004	0.9984	0.9807 1.0102 0.9997
0.9773	1.0000	1.0099	1.0017 1.0012 1.0005
0.9178	0.9144	1.0059	0.9898 0.9931 0.9967
0.9183	1.0173	1.0262	0.9860 0.9785 0.9876
			0.9753 0.9737 1.0061

Filename: ASR 9916 Pu isotopic correction Rev. 1.xlsx
Tab: ASR 9916 Pu isotopic correction Rev. 1.xlsx

Title: compare to SCS-CON
Purpose Evaluate the isotope fr

Ref. date: 25-Jan-16

Dissolved solids, mass basis

Analyte	68122-TI-001-F 16-0032 uCi/g dry	68122-TI-001-G 16-0032dup uCi/g dry
Pu-238	0.0639	0.0632
Pu-239	86.3313	86.3292
Pu-240	12.9750	12.9807
Pu-241	0.3935	0.3915
Pu-242	0.2363	0.2354

PNNL-20470. R1, Table 4.6 Decay correction

KW230

Ref. date: 22-Feb-11 Δ T 4.92 years
Representative core samples

TI024-2C wt%	TI024-3C wt%	TI024-4C wt%	TI024-2C wt%	TI024-3C wt%	TI024-4C wt%
0.071	0.070	0.067	0.0683	0.0673	0.0644
84.8820	85.4870	85.361	84.8700	85.4749	85.3489
14.0680	13.452	13.585	14.0607	13.4450	13.5779
0.6440	0.676	0.656	0.5072	0.5324	0.5167
0.3350	0.3140	0.331	0.3350	0.3140	0.3310
100.0000	99.9990	100.0000	99.8412	99.8336	99.8390
			0.0684	0.0674	0.0645
			85.0050	85.6173	85.4866
			14.0831	13.4674	13.5998
			0.5080	0.5333	0.5175
			0.3355	0.3145	0.3315

PNNL-19035. R1, Table 4.6 Decay correction

KW240, 250, 260

Ref. date: 1-Sep-09 Δ T 6.40 years
Representative core samples

TI005-1A wt%	TI006-1A wt%	TI007-1A wt%	TI005-1A wt%	TI006-1A wt%	TI007-1A wt%
0.070	0.070	0.07	0.0665	0.0665	0.0665
85.5100	86.1000	85.22	85.4943	86.0842	85.2043
13.5100	13.04	13.84	13.5009	13.0312	13.8307
0.6100	0.54	0.60	0.4472	0.3959	0.4399
0.3000	0.2600	0.27	0.3000	0.2600	0.2700
100.0000	100.0100	100.0000	99.8089	99.8378	99.8114
			0.0667	0.0667	0.0667
			85.6579	86.2240	85.3653
			13.5267	13.0524	13.8568
			0.4481	0.3966	0.4407
			0.3006	0.2604	0.2705

Ratio: Sandfilter sample result to SCS-CON sample result
Shading reflects ratios that exceeded +/- 5%

Pu-238			0.9341	0.9474	0.9898	0.9583	0.9585	0.9583
Pu-239			1.0156	1.0083	1.0099	1.0079	1.0012	1.0113
Pu-240			0.9213	0.9634	0.9541	0.9592	0.9941	0.9364
Pu-241			0.7745	0.7378	0.7603	0.8781	0.9922	0.8927
Pu-242			0.7043	0.7513	0.7128	0.7862	0.9074	0.8736

Filename: ASR 9916 Pu isotopic correction Rev. 1.xlsx
Tab: ASR 9916 Pu isotopic correction Rev. 1.xlsx

Spreadsheet Review Form

Spreadsheet Author Name:	SK Fiskum	Review Date:	2/10/2016
Date Prepared:	2/2/2016	Spreadsheet Subject:	"Radionuc. Calc." tab
Reviewer Name:	Cal Delegard	File Name:	ASR 9916 Pu isotopic correction.xlsx
Reviewer Title:	chemist	Revision No.	1

Scope of Spreadsheet Review: (Check one or more of the following)

☐	General Validation Review: (General review and spot checks)	☐	Independent calculation check (With hand calculations or independent spreadsheet software)
☐	Review of updated spreadsheet/calc (Revised portion only)	☐	Other: Point-by-point 100% review of TIMS data input (TIMS calculation spreadsheet) to "Radionuc. Calc." tab.
☒	100% Verification Review (Verification of all cells/calculations)		

REVIEW CHECK LIST

Spreadsheet/Calculation Identification

Spreadsheet Information	Yes	No	NA
Title:	x		
Revision Number:	x		
Date Prepared:	x		
Prepared by:	x		
General Statement of Purpose:	x		
General Description of Approach:	x		
Comments:			

Input Values	Yes	No	NA
Are input parameters correct (verified with source?)	x		
Are parameter units consistent?	x		
Are input values properly referenced?			x
Are input uncertainties correct?			x
Comments: Pu and Am isotopic input values to "Radionuc. Calc." tab verified from TIMS calculation spreadsheet.			

Assumptions	Yes	No	NA
Are assumptions clearly stated?			x
Are assumptions supported and justified?			x
Are assumptions reasonable?			x
Comments:			

Equations/Approach	Yes	No	NA
Are equation algorithms adequately defined?			x
Are equations properly referenced?			x
Are limitations of approach/equations			x
Are equations appropriate?			x
Are units consistent?	x		
Comments:			

Results/Conclusions	Yes	No	NA
Are formulas consistent in cells?	x		
Are calculations correct?	x		
Are conclusions consistent with results?			x
Are conclusions consistent with applicable limits?			x
Comments:			

Reviewer Sign/Date: 23 Feb 2016

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