U.S. DEPARTMENT OF ENERGY

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

Characterization Data Package for Containerized Sludge Samples Collected from Engineered Container SCS-CON-210

MS Fountain
SK Fiskum
DL Baldwin
SJ Bos
CA Burns
CD Carlson
DS Coffey
RC Daniel
CH Delegard
MK Edwards
LR Greenwood
D Neiner
BM Oliver
KN Pool
AJ Schmidt
RW Shimskey
SI Sinkov
LA Snow
CZ Soderquist
CJ Thompson
T Trang-Le
MW Urie

September 2013

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NATIONAL LABORATORY

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Richland, Washington 99352

Revision History

Revision Number	Description	Date
0	First issue	August 2011
1	Results for ASR 8878 and ASR 8919 were inserted and discussed in Sections 4.0 and 5.0 and primarily address results from undissolved solids analyses. Appendix D was updated to include both ASR reports. Two surveillance reports and one Occurrence Report were inserted into Table J.6 and Table J.4 (Appendix J), respectively. Minor editorial changes were incorporated throughout.	October 2011
2	According to the CH2MHill Plateau Remediation Company, Sludge Treatment Project (STP) Quality Assurance Project Plan/Sampling and Analysis Plan (QAPjP/SAP KBC-33786), Revision 1 of this characterization data package was validated and assessed by STP staff. The resulting STP validation report (PRC-STP-00560)[(a)] included detailed reviews and summaries of the final primary data (including those identified by the QAPjP/SAP documents) for the sludge in engineered container SCS-CON-210. As a consequence of the validation reviews of the laboratory data package, in addition to the baseline laboratory data qualifiers and data summaries provided in the validation report, a set of general corrections were also identified (e.g., typographical errors, general technical inconsistences, sample identification issues, etc.). While the STP validation report should be used as the primary source for STP sludge characterization and the corresponding STP Databook (HNF-SD-SNF-TI-015, Vol. 2, Rev. 24)[(b)] for design data, there are instances where certain detailed background information is only available in the Pacific Northwest National Laboratory (PNNL) report and data package. To assist future data users that have such background needs (e.g., for STP Phase 2), Revision 2 of the original report and data package (PNNL-20650) is being issued to correct for the general errors found during the STP validation that were subsequently confirmed by PNNL. Appendix J provides a detailed description of the changes for this revision. Figure 1 shows the hierarchy of SCS-CON-210 characterization data documentation and data authorization, and specifically how PNNL-20650 Revision 1 and Revision 2 reports are used relative to the STP-approved data.	September 2013

(a) Makenas BJ, RB Baker, JA Pottmeyer and TJ Welsh. 2013. *Data Validation and Assessment by the Sludge Treatment Project for Characterization Data from Engineered Container SCS-CON-210*, PRC-STP-00560, Revision 0, 0A, and 0B, CH2M Hill Plateau Remediation Company, Richland Washington.

(b) Johnson ME. 2013. *Spent Nuclear Fuel Project Technical Databook, Volume 2, Sludge*. HNF-SD-SNF-TI-015, Volume 2, Revision 24, CH2M Hill Plateau Remediation Company, Richland Washington.

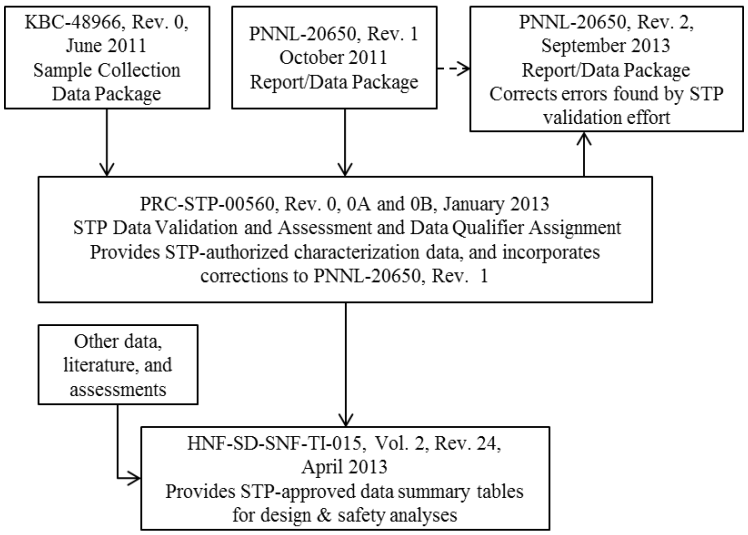


Figure 1. Hierarchy of SCS-CON-210 Characterization Data Documentation and Data Authorization

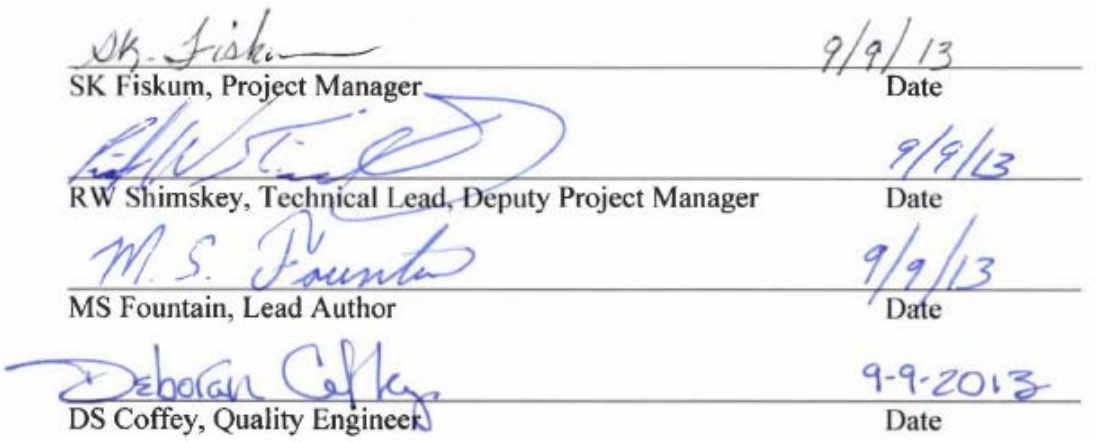
Certification Statement for Revsion 2

We certify that the data presented in PNNL-20650 Revision 2 describing the characterization analysis of K Basin containerized sludge samples collected from Engineered Containers SCS-CON-210 conform to the requirements in the following documents:

- Quality Assurance Project Plan/Sampling and Analysis Plan (QAPjP/SAP): *Quality Assurance Project Plan/Sampling and Analysis Plan for Sludge in the KW Engineered Containers*; KBC-33786, Rev. 2, December 2009
- DOE/RL-96-68 HASQARD, Revision 3, Volumes 1 and 4, *Hanford Analytical Services Quality Assurance Requirements Document, Administrative and Laboratory Technical Requirements*
- Analytical Support Operations (ASO) Quality Assurance Plan, ASO-QAP-001, Rev. 7
- Laboratory procedures and test instructions
- CH2M Hill Plateau Remediation, Contract 36402, Release 40, *Statement of Work, Technical Support for Planning, Sampling and Analyzing KW Container Sludges* (October 3, 2008)

Any deviations from procedures or test instructions are addressed in the completed test instructions (DVD attachment) and analysis report narratives (appendices) via references to Occurrence Reports. Throughout the project, Occurrence Reports were issued to inform participants and our client when analytical issues of concern were identified. When required, Occurrence Reports are elevated to Deficiency Reports to document conditions when data quality objective issues were not satisfied and procedure or plan deviations occurred or to Nonconformance Reports when an item did not meet specifications. A summary of the Occurrence Reports are summarized in Appendix J. There were no Deficiency Reports for this work activity and one Nonconformance Report was generated.

Multiple levels of data verification activities were performed as discussed in Appendix J.



Certification Statement

We certify that the data presented in this report for the characterization analysis of K Basin containerized sludge samples collected from Engineered Containers SCS-CON-210 conform to the requirements in the following documents:

- Quality Assurance Project Plan/Sampling and Analysis Plan (QAPjP/SAP): *Quality Assurance Project Plan/Sampling and Analysis Plan for Sludge in the KW Engineered Containers*; KBC-33786, Rev. 2, December 2009
- DOE/RL-96-68 HASQARD, Revision 3, Volumes 1 and 4, *Hanford Analytical Services Quality Assurance Requirements Document, Administrative and Laboratory Technical Requirements*
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C. D. Carlson, Project Manger	10/5/2011 Date
S. K. Fiskum, Technical Lead / Deputy Project Manager	10/3/11 Date
R. W. Shimskey, Technical Lead / Deputy Project Manager	10/3/11 Date
M. S. Fountain, Lead Author	10/3/11 Date
D. S. Coffey, Quality Engineer	10-03-2011 Date

Summary

This data package contains the K Basin sludge characterization results obtained by Pacific Northwest National Laboratory during processing and analysis of four sludge core samples collected from Engineered Container SCS-CON-210 in 2010 as requested by CH2M Hill Plateau Remediation Company. Sample processing requirements, analytes of interest, detection limits, and quality control sample requirements are defined in the KBC-33786, Rev. 2, *Quality Assurance Project Plan/Sampling and Analysis Plan for Sludge in the KW Engineered Containers* (Baker et al. 2009).

The core processing scope included reconstitution of a sludge core sample distributed among four to six 4-L polypropylene bottles into a single container. The reconstituted core sample was then mixed and subsampled to support a variety of characterization activities. Additional core sludge subsamples were combined to prepare two container composites. Only one container composite was fractionated by wet sieving through 2,000-µm and 500-µm mesh sieves. Each sieve fraction was sampled to support a suite of analyses. The unsieved container composite supported rheology testing.

The core composite analysis scope included sludge density determination, radioisotope analysis, and metals analysis, including the Waste Isolation Pilot Plant Hazardous Waste Facility Permit metals (with the exception of mercury). The container composite analysis included most of the core composite analysis scope plus particle size distribution, particle density, rheology, and crystalline phase identification.

A summary of the received samples, core sample reconstitution and subsampling activities, container composite preparation and subsampling activities, physical properties, and analytical results are presented. Supporting data and documentation are provided in the appendices. There were no cases of sample or data loss and all of the available samples and data are reported as required by the Quality Assurance Project Plan/Sampling and Analysis Plan; 100% completeness was attained.

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Acronyms and Abbreviations

AEA	alpha energy analysis
ALE	Arid Land Ecology (Reserve)
AOI	analyte of interest
ASO	Analytical Support Operations
ASTM	American Society for Testing and Materials
ASR	analytical service request
At%	atom percent
BTR	Buyer's Technical Representative
BS	blank spike
CCB	continuing calibration blank
CCV	continuing calibration verification
CEC	Consolidated Electrodynamics Corp.
CHPRC	CH2M Hill Plateau Remediation Company
COC	chain of custody
DI	deionized
DL	detection limit
DOE	Department of Energy
DQO	data quality objectives
DVD	digital versatile disc
EQL	estimated quantitation limit
GEA	gamma energy analysis
HASQARD	*Hanford Analytical Services Quality Assurance Requirements Documents*
HLRF	High Level Radiochemistry Facility
ICB	initial calibration blank
ICDD	International Centre for Diffraction Data
ICP-OES	inductively coupled plasma optical emission spectroscopy
ICV	initial calibration verification
ID	identification
IDL	instrument detection limit
IWTS	Integrated Water Treatment System
KBC	K-Basin Closure (Project)
KE	K-East (Basin)
KOP	knockout pot
KPA	kinetic phosphorescence analysis
KW	K-West (Basin)

LCC	large container composite, particles greater than 2,000 microns (or +2,000 μm) sieve fraction
LCS	laboratory control sample
LCTS	laboratory-created test sample
LRB	laboratory record book
LSC	liquid scintillation counter
MCC	middle container composite, particles between 2,000 and 500 microns (or -2,000/+500 μm) sieve fraction
MDA	minimum detectable activity
MDC	minimum detectible concentration
MDL	minimum detection limit
M&TE	measuring and test equipment
MS	matrix spike
NARA	National Archives and Records Administration
NBS	National Bureau of Standards
NIST	National Institute of Standards and Technology
NM	nuclear material or not measured, see context
NR	not recovered or not reported, see context
OIER	organic ion exchange resin
OTRS	one time request for shipment
PB	preparation blank
PDF	powder diffraction file (XRD analyses) or portable document format
PNNL	Pacific Northwest National Laboratory
PQL	process quantification limit
PS	post-digestion spike
PSD	particle size distribution
PUREX	Plutonium and Uranium Extraction
QA	quality assurance
QAP	quality assurance plan
QAPjP/SAP	Quality Assurance Project Plan/Sampling and Analysis Plan
QC	quality control
RPD	relative percent difference
RPL	Radiochemical Processing Laboratory
RSD	relative standard deviation
SAL	Shielded Analytical Laboratory
SCC	small container composite, particles less than 500 microns (or -500 μm) sieve fraction
SFO	Shielded Facility Operations

SNF	spent nuclear fuel
SPC	statistical process control
SRM	standard reference material
SSC	shielded sample container
STP	Sludge Treatment Project, CHPRC
SWB	standard waste box
TC	total carbon
TI	test instruction
TIC	total inorganic carbon
TIMS	thermal ionization mass spectrometry
TOC	total organic carbon
TRIM	Total Records Information Management
TS	total solids
UDS	undissolved solids
UHP	ultra-high purity
WIPP	Waste Isolation Pilot Plant
XRD	x-ray diffraction (spectrometry)

Definitions

Settled sludge	Sludge that has been fully suspended and then settled ~24 h from a relatively large fluidic headspace (i.e., batch settled). Typically, this batch settling leads to sample stratification, with the denser/larger particles at the bottom of the sample and the lighter/smaller particles at the top of the "settled sample". **Note 1:** based on testing performed with simulants, the "settled" density was found to be more reproducible than "dewatered" (or "as-sampled") density from one sampling session to the next. This trend was observed during actual waste testing. **Note 2:** in accordance with the Quality Assurance Project Plan/Sampling and Analysis Plan (QAPjP/SAP), analyte concentrations are to be reported on a settled sludge basis.
Dewatered sludge	Sludge that has been thickened by "batch settling" followed by mixing/ homogenizing the settled sludge and removing any resulting displaced interstitial water. Based on past experience with sludge, in the dewatered state, sludge possesses sufficient shear strength to suspend/uniformly disperse the dense uranium metal particles. Consequently, it is in this dewatered state that subsamples are collected from the parent sample. Once sampled, cover-water was added back to the dewatered sludge to avoid sample drying and maintain sludge integrity. **Note:** the "dewatered" state is an arbitrary measure since the water entrainment condition in the solids will range somewhere between a free-flowing condition with free-standing water on the surface (settled sludge) and mixed and centrifuged, where the maximum water is squeezed out of solids interstices. The time it takes to reach this state can be highly variable, as it is affected by the sludge composition and settling rates. In testing with simulants, the dewatered condition exhibited significantly more variability in density relative to settled sludge. This trend was observed during actual waste testing.
As-sampled sludge	The actual state of the sludge at the point of sampling; in most instances this is the "dewatered" sludge state, but in the case of sieved sample material retained on a sieve it is taken as "air-dried" (dried at ambient conditions in the hot cell).
Dry sludge	Sludge that has been dried to a constant mass at 105°C.
Slurry volume	The combined sludge volume (settled or dewatered) and fluid volume above the solids.
Estimated volume uncertainty	The uncertainty in measured volume is dependent on a plurality of factors. These include the spread in volume graduation marks on a container, the interpolation applied to the volume when the meniscus/level is between graduation marks, the inner diameter of the container, the evenness of the surface being measured, the amount of material that may be coating the upper interior container walls, and obscuration from other factors.
Opportunistic measurements	A variety of opportunistic data were collected (e.g., volume, mass, inductively coupled plasma optical emission spectrometry [ICP-OES] analytes, radioisotope analytes, etc.) with little or no additional cost to the project. The QAPjP/SAP did not specifically require these data be collected, and quality control (QC) sample acceptance limits requirements were not established for these analyses. The opportunistic data are presented to provide a broader understanding of the data set as a whole.

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(a) The report and appendices are also published on DVD.

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Section I: Electronic Version (Adobe PDF) of *Characterization Data Package for Containerized Sludge Samples Collected from Engineered Container SCS-CON-210*, PNNL-20650, Rev. 2, September 2013.

Section II: Standard Laboratory Operating and Analytical Procedures

Analysis Objective	Procedure Number	Rev.	Procedure Title
TI Implementation	RPL-OP-001	9	*Routine Research Operations*
Chemical Analysis Acid Digestion	PNL-ALO-129	0	*HNO_3-HCl Acid Extraction of Solids Using a Dry-Block Heater*
Chemical Analysis Fusion	PNL-ALO-115	1.1	*Solubilization of Metals from Solids Using a KOH-KNO_3 Fusion*
U Metal and ICP-OES Measurable Analytes	RPG-CMC-211	3	*Determination of Elemental Composition by Inductively Coupled Argon Plasma Optical Emission Spectrometry (lCP-OES)*
U/KPA	RPG-CMC-4014	1	*Uranium by Kinetic Phosphorescence Analysis*
U Metal and U Speciation Acid Digestion	RPG-CMC-107	1	*Sample Preparation for Determination of Uranium Metal Concentrations in Sludge*
U(IV) : U(VI) Ratio	RPG-CMC-255	0	*Sample Preparation and Analysis for Determining Uranium Oxide Oxidation States in K Basin Sludges*
Solution Density Determination	PNL-ALO-501	0	*Laboratory Procedure for Measurement of Physical and Rheological Properties of Solutions, Slurries, and Sludges*

Section II: Standard Laboratory Operating and Analytical Procedures (contd)

Analysis Objective	Procedure Number	Rev.	Procedure Title
TOC/TIC	RPG-CMC-385	2	*Carbon Measured in Solids, Sludge, and Liquid Matrices*
XRD	PNNL-RPG-268	4	*Solids Analysis by X-Ray Diffraction*
Particle Density	APEL-PIP-4	2	*Gas Pycnometry Method for Apparent Specific Gravity Determinations of Consolidated Solids*
Particle Size Distribution	RPL-COLLOID-01	1	*Particle Size Analysis Using Malvern MS2000*
Viscosity Shear Strength and Flow Curve	RPL-COLLOID-02	2	*Measurement of Physical and Rheological Properties of Solutions, Slurries, and Sludges*
U-Isotopic Analysis	RPG-CMC-4017	0	*Analysis of Environmental Water Samples for Actinides and Strontium-90*
	PNNL-98523-264	2	*Mass Spectrometer Isotopic Analysis (TIMS)*
^{238}Pu, $^{239+240}$Pu, ^{241}Am, and ^{237}Np—AEA	RPG-CMC-4017	0	*Analysis of Environmental Water Samples for Actinides and Strontium-90*
	RPG-CMC-496	0	*Coprecipitation Mounting of Actinides for Alpha Spectroscopy*
	RPG-CMC-422	2	*Solutions Analysis: Alpha Spectrometry*
Pu-Isotopic Analysis	RPG-CMC-455	0	*Separation of Uranium and Plutonium for Isotopic Analysis by Mass Spectrometry*
	PNNL-98523-264	2	*Mass Spectrometer Isotopic Analysis (TIMS)*
Gamma-emitters—GEA	RPG-CMC-450	1	*Gamma Energy Analysis (GEA) and Low-Energy Photon Spectrometry (LEPS)*
Gross Alpha and Beta	RPG-CMC-4001	1	*Source Preparation for Gross Alpha and Gross Beta Analyses*
	RPG-CMC-408	2	*Total Alpha and Beta Analysis*
AEA Screen	RPG-CMC-496	0	*Coprecipitation Mounting of Actinides for Alpha Spectroscopy*
	RPG-CMC-422	2	*Solutions Analysis: Alpha Spectrometry*
^{90}Sr	RPG-CMC-476	0	*Strontium Separation Using Eichrom Strontium Resin*
	RPG-CMC-474	1	*Measurement of Alpha and Beta Activity by Liquid Scintillation Spectrometry*
General	52578-ADM-001	0	*K Basin Requests for Analysis and Guidelines for Data Reporting and Technical, QE, and Spreadsheet Reviews*
	ADM-RSEG-BALANCES	0	*Balance Calibration Verification*
	SOP-SFO-512	0	*Receive K Basin Sludge via Standard Waste Box (SWB)*

Section III: Test Instructions, Laboratory Record Books, and Data Analysis Files

Folder ID	Contents Description
52578-TI001	K West Basin Sample Receipt and Preparation of Consolidated Core Composites: SCS-CON-210-A1 and SCS-CON-210-B3
52578-TI002	K West Basin Sample Receipt and Preparation of Consolidated Core Composites: SCS-CON-210-A4 and SCS-CON-210-B1
52578-TI008	Preparation of K West Basin Consolidated Container Composite for SCS-CON-210
52578-TI037	Particle Size Distribution Measurements for K West Basin Consolidated Container Composite SCS-CON-210, Using Minus 500 micron Particle Size Fraction Samples
52578-TI039	Rheological Measurements for K West Basin Consolidated Container Composite SCS-CON-210
52578-TI040	Processing of the Undissolved Solids from the K West Basin Core Samples Collected from SCS-CON-210 for Additional Analysis This folder also contains the data calculation spreadsheet underpinned by this test instruction: Sample Split Masses TI040 Rev. 1.xlsx.
ASR Data Reductions	Contains ASR 8798 Data Reduction.xlsx, ASR 8798 Data Reduction Rev1.xlsx, ASR 8798 Data Reduction Rev2.xlsx, ASR 8824 Data Reduction.xlsx, ASR 8824 Data Reduction Rev1.xlsx, and ASR 8824 Data Reduction Rev2.xlsx. Spreadsheets combine physical properties information with analytical data and produces summarized results. QE surveillance of ASR 8824 Data Reduction Rev1.xlsx and ASR 8798 Data Reduction Rev2.xlsx also included.
KW210 Bottle Volumes	Contains KW210_Bottle_Volumes.xls. Compares measurements of sludge volume of received core samples in 4-L polybottles to the measured volume of the reconstituted core after addition of each core sample. Individual core sample bottle information and reconstituted core settling behavior over time is also captured.
LRBs	Applicable pages of LRB 60605 that specifically relate to KW210 work.
TI001 Physical Properties	Contains KW210-A1 Rev2.xlsx and KW210-B3 Rev2.xlsx files. Spreadsheets calculate and summarize the physical properties and settling of the reconstituted core subsamples.
TI002 Physical Properties	Contains KW210-A4 Rev2.xlsx and KW210-B1 Rev2.xlsx files. Spreadsheets calculate and summarize the physical properties and settling of the reconstituted core subsamples.
TI008 Physical Properties	Contains KW210CC Rev. 1.xlsx Spreadsheet calculates and summarizes the physical properties and settling of the container composite samples.
TIMS Data Analysis	Contains the spreadsheet used to normalize the raw TIMS data to atom% for reporting, KW210 TIMS data July 19 2011.xlsx.

Section IV: Photographic Images

Folder ID	Contents Description
Color Charts	A color chart image was collected each day core reconstitution and subsampling work was conducted. Other work activities have a color chart view in the background of the image.
KW210 A1 Core	This folder contains 6 subfolders entitled: 4-L Polybottle Inspections, Core Reconstitution Stages, Core Subsample Settling, Reconstituted Core Inspections, Reconstituted Core Settling, and Reconstituted Core Subsampling.
KW210 A4 Core	This folder contains 6 subfolders entitled: 4-L Polybottle Inspections, Core Reconstitution Stages, Core Subsample Settling, Reconstituted Core Inspections, Reconstituted Core Settling, and Reconstituted Core Subsampling.
KW210 B1 Core	This folder contains 6 subfolders entitled: 4-L Polybottle Inspections, Core Reconstitution Stages, Core Subsample Settling, Reconstituted Core Inspections, Reconstituted Core Settling, and Reconstituted Core Subsampling.
KW210 B3 Core	This folder contains 6 subfolders entitled: 4-L Polybottle Inspections, Core Reconstitution Stages, Core Subsample Settling, Reconstituted Core Inspections, Reconstituted Core Settling, and Reconstituted Core Subsampling.
KW210 Container Composite	This folder contains 6 subfolders entitled: KW210-CC poke test, KW210-LCC, KW210-MCC, KW210-SCC poke test, Pre and Post comp, Settling test KW210-SCC
KW210 Particle Density Measurement	This folder contains the images collected from the particle density subsamples.
KW210 Dry Solids Inspections	The primary physical properties samples (K and L) for each core were dried at 105°C. Images documenting the inspection of the vials and dry materials are included.
KW210 UDS	This folder contains images of the undissolved solids (UDS) after removal from the hot cell from the core samples (subfolder: Cores) and the container composite samples (subfolder: Cont Comp).
KW210MK-ML Resin and Non-Resin	This folder contains images of the resin bead fraction and non-resin bead fraction after first combining samples TI008-MK and TI008-ML and then separting the resin beads from the combined UDS sample.
SAL 24hr Settle Tests	Archive and M samples from each core were remixed and 24-hr settling tests completed in SAL. Images are captured of slurry and sludge levels at the start and end of the settling tests.

1.0 Introduction

The Sludge Treatment Project (STP), managed for the U.S. Department of Energy (DOE) by CH2M Hill Plateau Remediation Company (CHRPC), is responsible for operating the K West (KW) Basin fuel storage pool and managing the sludge (and other materials) that it contains. The STP mission includes characterization, containment, safe removal, treatment, and transportation of all sludge (treated or untreated) from the KW Basin. Sludge from the floor and pits of the KW and K East (KE) Basins has been consolidated into five large engineered containers: SCS-CON-210, 220, 240, 250, and 260. Settler sludge was retrieved and transferred into Engineered Container SCS-CON-230.

Pacific Northwest National Laboratory (PNNL) was contracted by CHPRC to conduct detailed physical, chemical, and radioisotopic characterization on K Basin sludge samples collected by CHPRC in 2010 from Engineered Container SCS-CON-210. The characterization effort was required to establish

- the nuclear material (NM) accountability values for the KW Basin floor and pit sludge inventory and
- to provide specific characterization data to support the final process design to retrieve, treat, store, and package the sludge for disposal at the Waste Isolation Pilot Plant (WIPP).

All characterization testing was conducted at the PNNL Radiochemical Processing Laboratory (RPL). This summary report and data package presents the verified characterization results, as well as the supporting characterization documentation. These verified data will undergo further validation and assessment for use by the STP and others. The final/validated data for general use by the various end-users will be documented elsewhere (e.g., STP validation and assessment reports [“brown covers”], supporting documents [Plys and Schmidt 2006], and STP engineering databook [Schmidt 2010]).

This report is the third in a series of K Basin engineered container characterization reports and data packages. Fiskum et al. (2011a, 2011b) reported the results from Engineered Containers SCS-CON-220, 240, 250, and 260; Shimskey et al. (2011) reported the results from Engineered Container SCS-CON-230. Much of the previous reports’ structure, verbiage, and diagram descriptions are repeated in this report, allowing it to stand alone as a complete report and data package. The text and diagrams have been updated as appropriate.

The overall scope of the characterization effort supported by PNNL is provided in Figure 1.1 (see highlighted text in the red dotted box). In this case, PNNL is shown as receiving, processing, and analyzing samples, followed by project-driven data verification. Data validation is conducted separately, outside of the PNNL scope.

The sample handling and characterization requirements for this work are defined in KBC-33786, *Quality Assurance Project Plan/Sampling and Analysis Plan (QAPjP/SAP) for Sludge in the KW Engineered Containers*, Revision 2 (Baker et al. 2009). The work was performed by PNNL under CHPRC Contract 36402-40 and executed under PNNL Project Number 52578.

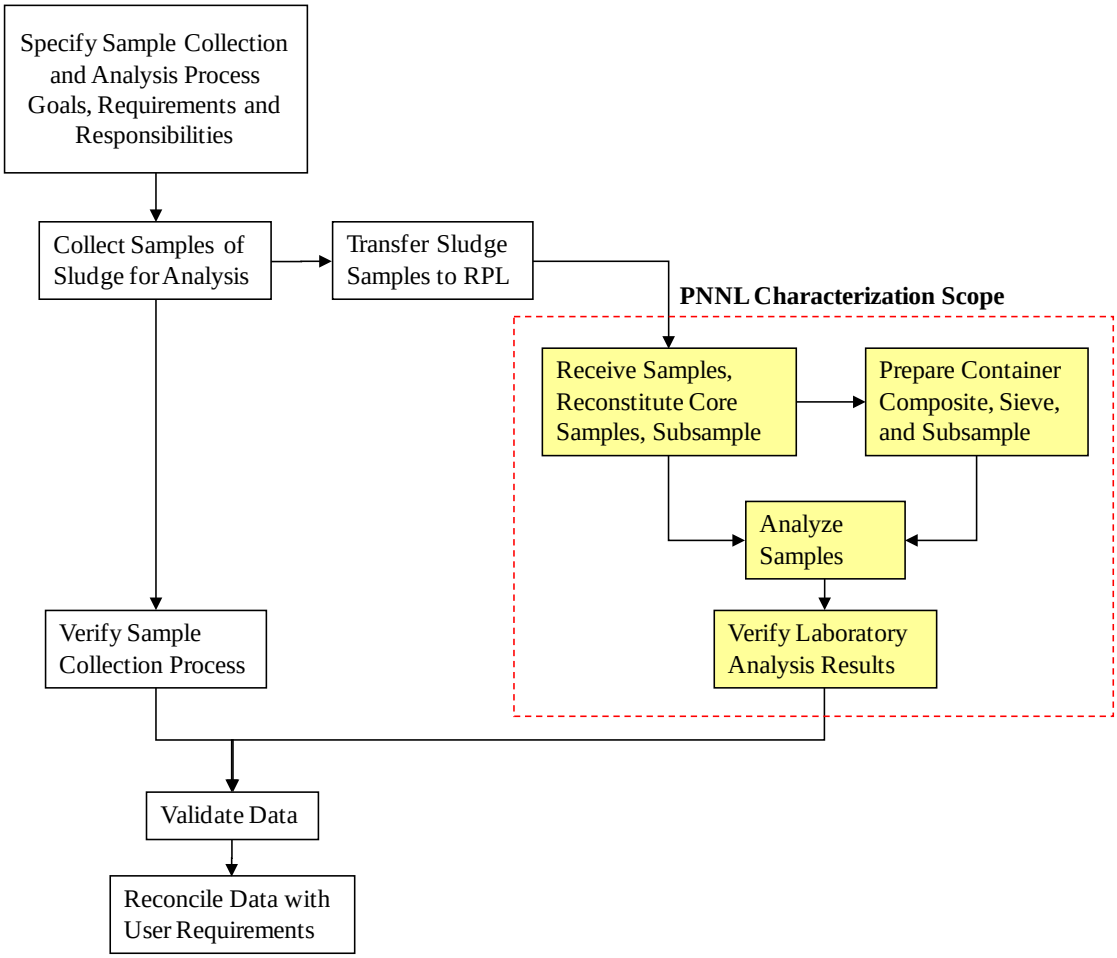


Figure 1.1. Project Scope with PNNL Characterization Scope Shown in the Red Dotted Box

1.1 Scope of Characterization

Four sludge core samples were collected from SCS-CON-210 (located at the KW Basin) in 4-L sample bottles. Each of the core samples was distributed among four to six sample bottles. The sample bottles were shipped to the RPL in a heavily shielded sample container (SSC) (i.e., lead-lined "Super Pig") within a standard Hanford waste box (SWB) with chain of custody (COC) documentation. Each shipment consisted of two to four sample bottles; nine shipments were received at RPL between November 18, 2010 and January 4, 2011.

Upon receipt at RPL, the sample bottles were transferred from the SWB into a shielded hot cell at the High Level Radiochemistry Facility (HLRF). After initial observations and volume measurements, the core samples were reconstituted by combining the sample materials from the appropriate sample bottles. Each core sample was then mixed and subsampled to support a variety of analytical activities. A portion of the core samples was combined to prepare a container composite sample. The container composite sample was size-fractionated by wet sieving through a 2,000-µm mesh sieve and a 500-µm mesh sieve to generate three distinct particle-size fractions. Each sieve fraction was sampled to support a suite of analyses. A second duplicate container composite was created but was not sieved; it was analyzed for shear strength.

Figure 1.2 and Figure 1.3 are generalized flow charts adapted from the QAPjP/SAP KBC-33786, Rev. 2 (not intended to show all operational steps) that summarize the characterization scope associated with the core samples and the container composite samples. Some additional sample analyses were conducted that were not explicitly addressed in the QAPjP/SAP. These analyses are discussed in Sections 4.0 and 5.0.

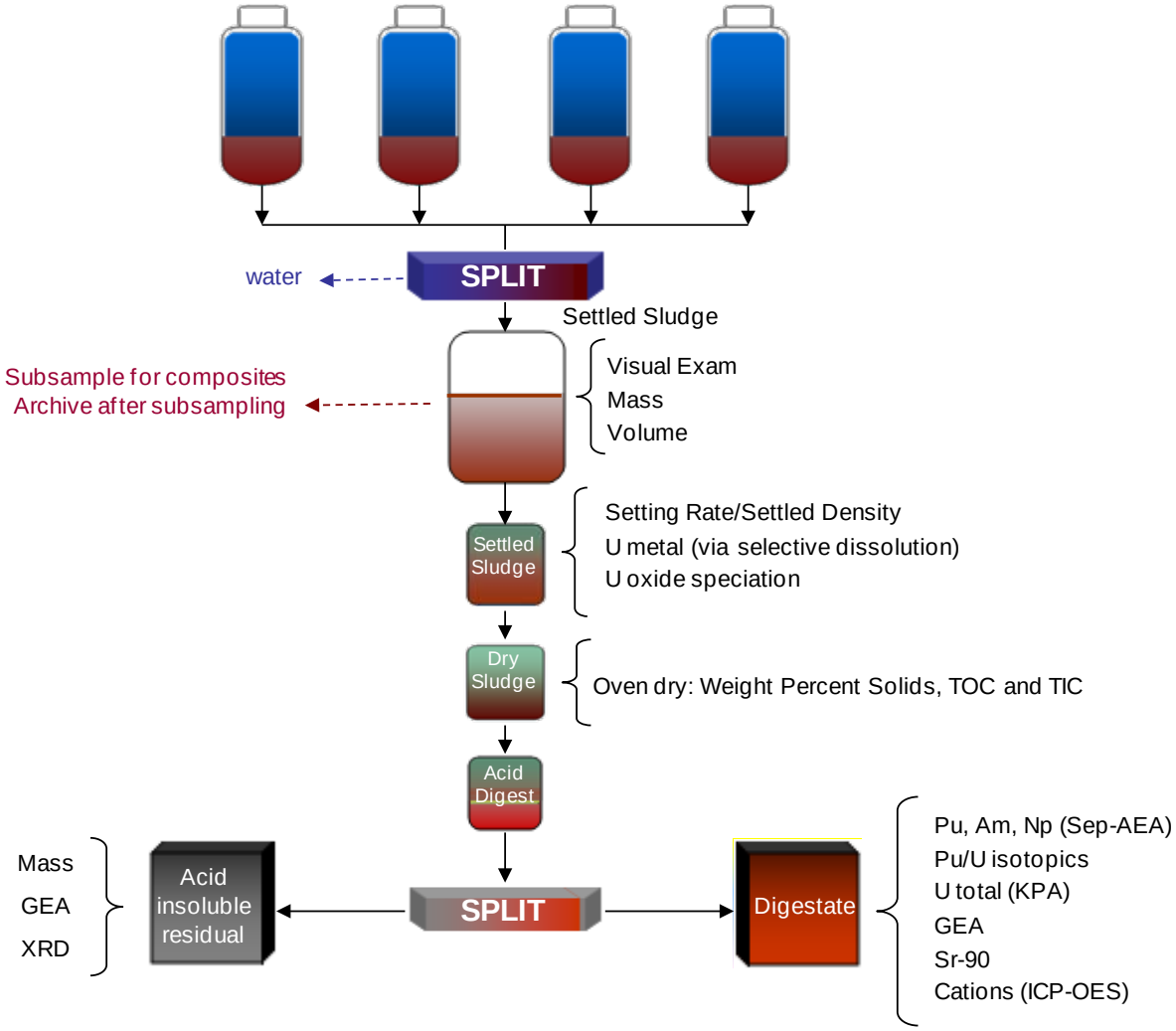
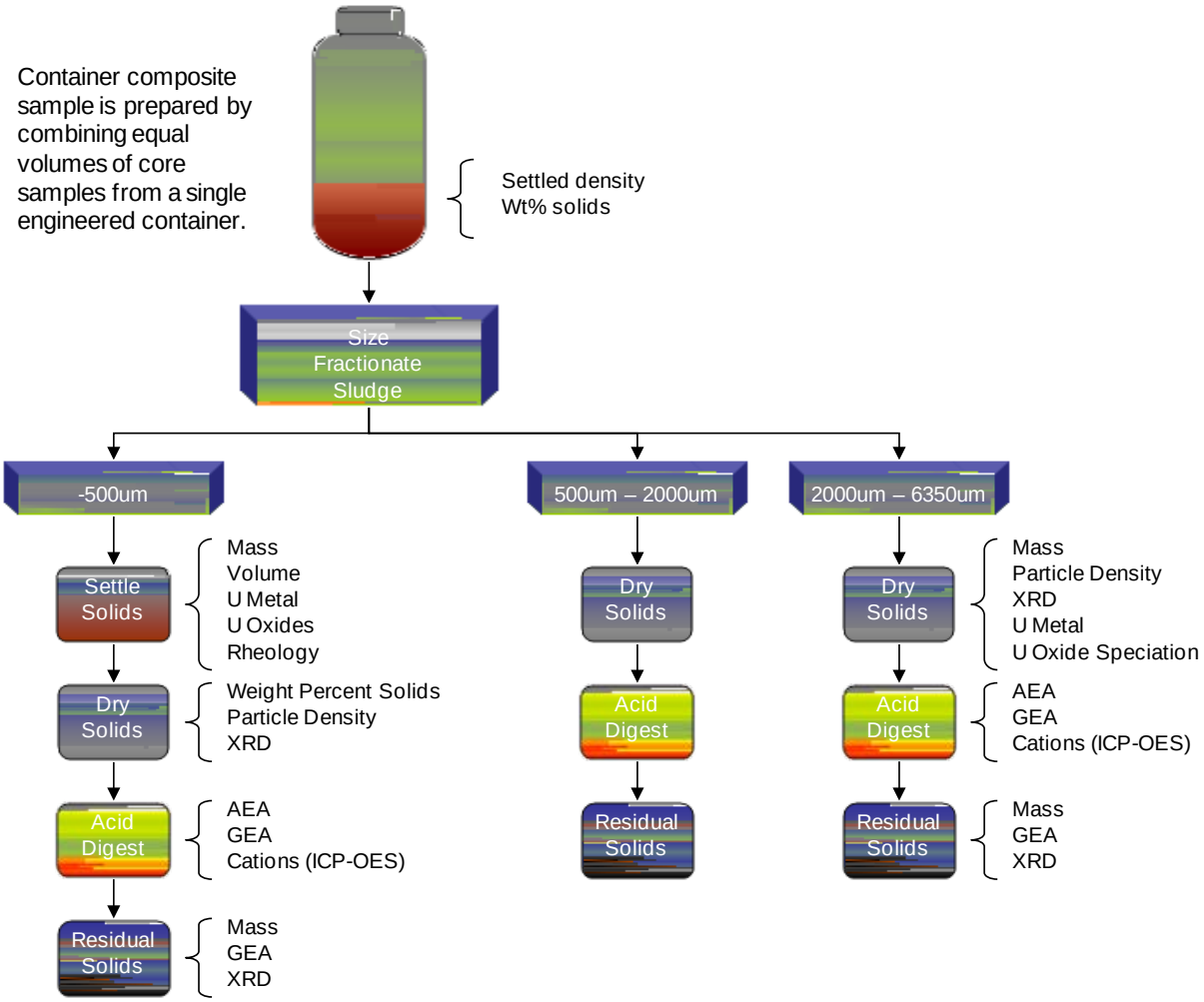


Figure 1.2. Generalized Core Sample Analysis Flowchart

The activities associated with sample consolidation, subsampling, and physical characterization were controlled and documented in detailed work documents (e.g., test instructions [TIs] or technical procedures). The sample handling and characterization requirements were complex, and before implementation, each TI underwent extensive internal reviews. Also, in accordance with the QAPjP/SAP and the Statement of Work (SOW) for Contract 36402-40, the TIs were reviewed and approved by the CHPRC Contract 36402-40 Buyer's Technical Representative (BTR) who was also the Manager of the CHPRC/STP Sampling and Characterization group. Approval and concurrence were documented by the BTR's signature on the TI.



- *−6,350 µm/+2,000 µm represent wet-sieved particles isolated on a 2,000-µm sieve, i.e., >2,000 µm.*
- *−2,000 µm/+500 µm represent wet-sieved particles isolated on a 500-µm sieve, i.e., <2,000 µm and >500 µm.*
- *−500 µm represent wet-sieved particles that passed through both sieves, i.e., <500 µm.*
- *Rheology measurements were conducted on unseived material instead of the −500-µm fraction (See Section 5.0)*
- *Scope of the +2,000-µm fraction analysis was limited by available mass to GEA and particle density.*

Figure 1.3. Generalized Container Sludge Composite Sample Analysis Flowchart

Analyses and associated quality control (QC) sample requirements in the QAPjP/SAP were provided to the analytical laboratory (referred to as the RPL Analytical Support Operations [ASO] laboratory) in the form of Special Instructions appended to the Analytical Service Request (ASR). Because the analytical requirements were complex, these Special Instructions were reviewed and approved both internally by PNNL staff and by the BTR to help make sure all requirements were properly conveyed to the ASO staff. As with the TIs, approval and concurrence were documented by signature on the Special Instructions.

All physical, chemical, and radioisotope analyses reported in this document were performed between November 2010 and August 2011.

1.2 Report and Data Package Structure

The contents and structure of the summary report and data package are described below.

Section 2.0. Sample Receipt and Processing

This section provides an overview of PNNL sample receipt, processing, subsampling, and characterization activities.

The physical and chemical characterization scope for the sludge samples is defined in the KBC-33786, Rev. 2 QAPjP/SAP (Baker et al. 2009). The approach taken by PNNL to execute the specific scope is detailed in Section 2.0 of this report. Section 2.0 documents sample receipt at the RPL and provides an overview of the sample characterization approach, including detailed descriptions of sample flow for the following activities:

- reconstitution of core samples by combining sludge material retrieved from the 4-L sample bottles
- sludge subsampling and subsample processing
- disposition of the water from the samples
- preparation of two container composites
- container composite sieving, subsampling, and sample processing.

In addition, test approaches and procedures used for physical and chemical (inclusive of radioisotopes) characterization are identified and described and quality control (QC) sample requirements and acceptance criteria for each analysis are provided.

Sample identification (ID) nomenclature used in this characterization effort included engineered container ID (SCS-CON-210), core IDs (e.g., A1), TI subsample IDs (e.g., TI001-A1-B), ASR numbers (e.g., 8798), and RPL analytical laboratory IDs (e.g., 11-0419). Section 2.0 includes sample ID cross reference tables to assist in the navigation of the document and appendices.

Section 3.0. Physical Property Characterization Samples: Sludge Densities and Conversion Factors to Dry and Wet Mass Bases

KBC-33786, Rev. 2 QAPjP/SAP identifies wet settled-sludge density as a key property that must be determined for core (and other) samples. Chemical/radioisotope analysis results were determined and reported by ASO on a dry-mass basis and, for U metal, an as-sampled mass basis. Section 3.0 provides results from density determinations, wt% solids measurements, and resulting conversion factors that allow the user to convert analyte concentrations from a dry mass to a settled sludge mass basis and from an as-sampled mass to a settled sludge mass basis. These factors can also be used to convert analyte concentration from a mass to a volume basis. Primary physical property samples were used for these measurements. Physical property data were also collected from the parent sample (the reconstituted core) and other core subsamples collected for additional processing (e.g., characterization subsamples and

container composite subsamples). These data were collected with the same rigor as the primary physical property sample data, and allow the data user to assess the general representativeness of the physical properties and subsamples.[(a)]

The primary physical property samples, typically 6 to 20 mL, were dried at 105ºC. Before discarding, the dried physical property samples were evaluated for apparent bulk volume and bulk morphology (i.e., powder vs. agglomerated monoliths) for insights into sludge behavior if it were allowed to dry out.[(b)] Section 3.0 includes a summary of these evaluations.

Section 4.0. Engineered Container SCS-CON-210 Core Samples

Four core samples were collected from Engineered Container SCS-CON-210 (i.e., floor and pit sludge originating from the KW Basin). Section 4.0 discusses the processing, reconstitution, observations (including photographs), and characterization results from each of these four core samples (see Figure 1.2). Consistent with KBC-33786, Rev. 2 QAPjP/SAP reporting requirements, characterization results are presented on a dry-solids mass basis and a settled-sludge mass basis. Also, for each type of analysis, a table is provided summarizing the process batch QC sample results for assessment of precision and accuracy (i.e., relative percent difference [RPD], laboratory control sample [LCS]/blank spike [BS] recoveries, matrix spike [MS] recoveries, and post-spike recoveries).

In accordance with the QAPjP/SAP, residual solids from the acid digestion preparation were evaluated by gamma energy analysis (GEA). Sufficient mass remained in these residual solids that further analyses were deemed necessary per direction from the BTR to better characterize the chemical composition of this material. Section 4.0 includes detailed discussion of the residual solids characterization.

A variety of data were collected on analytes not specifically listed in the QAPjP/SAP (e.g., 27 analytes measured by inductively coupled plasma-optical emission spectrometry [ICP-OES], radioisotope analytes [e.g., Cm].) with little or no additional cost to the project, which this report defines as opportunistic. The QAPjP/SAP did not specify QC sample acceptance limits requirements for these opportunistic analytes and the results may not be supported by the data quality evaluation rigor applied to the specified analytes of interest. Therefore, the quality of these opportunistic data may be considered indeterminate.

Section 5.0. SCS-CON-210 Container Composite Sample

Subsamples of each of the four core samples were collected and combined to create two container composite samples of equivalent composition. After measuring the physical properties, one of the container composites was sieved into three size fractions. Each sieve fraction was characterized in accordance with KBC-33786, Rev. 2 QAPjP/SAP, based on sample availability. In addition to chemical/ radiochemical analyses (ICP-OES, GEA, alpha energy analysis [AEA] screen), characterization included crystalline phase identification by x-ray diffraction (XRD) and particle density measurement. The

(a) Depending on the sample type, mass and/or volume measurements in the settled, as-sampled, and/or dried condition were collected. The uncertainties of these measurements were assessed and the propagated error of density and mass ratios were calculated.

(b) Consistent with the QAPjP/SAP, the characterization scope beyond the QAPjP/SAP was approved by the BTR in response to “in-field” observations/opportunities and unforeseen situations.

smallest sieve fraction (i.e., containing particles <500 µm) was characterized by particle-size distribution (PSD). The second container composite was not sieved and was used for shear strength testing. This section details the processing, observations, and characterization results from the container composite and the sieve fractions.

Section 6.0. Records

Records supporting the analytical results are maintained by the ASO records system, and copies of non-ASO records are provided in this report and data package. Section 6.0 details the types of ASO records that are stored and describes how they can be retrieved.

Section 7.0. References

Section 7.0 lists the references cited in this report.

Appendices

Copies of the COC records are provided in Appendix A. Appendices B through D provide all ASO-generated reports in accordance with each ASR request. Each appendix is specific to one ASR. Appendices E, F, G, H, and I provide additional sample characterization results and supporting data (thermal ionization mass spectrometry [TIMS], XRD, particle density, rheology, and PSD, respectively). Appendix J addresses the processes used to verify the data and provides a summary of Revision 2 corrections in this report.

1.3 Brief History/Overview of K Basin Sludge

Nuclear reactors were operated to produce weapons-grade plutonium from 1944 to 1990 at the U.S. Department of Energy's (DOE's) Hanford Site in Eastern Washington. The KE and KW reactors were built in the 1950s and operated into the early 1970s. Each reactor had a large water-filled concrete basin used for temporary storage of its irradiated fuel (approximately 125 feet by 67 feet and 21 feet deep, holding approximately 1.2 million gallons of water). One of the other newer reactors, N-Reactor, operated from 1964 through 1987. During N-Reactor operations from 1972 until 1983, fuel reprocessing at Hanford's Plutonium and Uranium Extraction (PUREX) facility was suspended because of safety and other concerns. With fuel processing suspended, spent nuclear fuel (SNF) accumulated to a point that exceeded the capacity of the N-Reactor storage basin, requiring utilization of the basins at the KE and KW Reactors for additional storage. Reprocessing at PUREX resumed from 1983 until 1989, but approximately 2,100 metric tons of Zircaloy-clad (with a small portion of aluminum-clad single pass), metallic uranium SNF remained in the K Basins when PUREX was permanently shut down in 1990. Some of the SNF was damaged when it was discharged from the N-Reactor. The damage was primarily to the cladding and occurred as a result of collisions among the fuel assemblies discharged and handled after irradiation at the reactors. Subsequent corrosion of the exposed uranium metal fuel resulted in the generation of radioactive sludge components. K Basin sludge is a mix of spent fuel products (including metallic uranium, zircaloy-2 cladding, plutonium, and fission and activation products), small fuel fragments, iron and aluminum oxides, concrete grit, sand, dirt, particulates of operational processes (e.g., ion exchange material), and material of biological origin (e.g., insects, plant matter). For the purpose of differentiating SNF and debris (e.g., nuts, bolts, wires) from the sludge, any material that will pass through a screen with 0.64 cm (0.25 in.) openings is defined by the STP as sludge.

The KE Basin was not cleaned out before being used to store N-Reactor fuel; the first N-Reactor fuel was received at KE Basin in 1975. Therefore, some fraction of the sludge material originated while the basin was used to support the KE Reactor operations. The basin pool was located inside a steel framed building, but the building was not air tight, and windblown dust and other environmental debris were able to enter the pool. The pool walls and floor were constructed of concrete (uncoated), and with time, degradation of the concrete (i.e., grit and small aggregates) contributed to the sludge volume.

Corrosion products from carbon steel and aluminum structures and components are significant sludge constituents (Johnson and Burke 1995). Material from water quality control operations, including sand (from sand filters) and organic and inorganic ion exchange materials (from the ion exchange systems), are known to be in the sludge. At the KE Basin, N-Reactor SNF was stored in open-top fuel storage canisters (constructed of aluminum and stainless steel), and 83% of these fuel canisters had open-screened bottoms. Significant quantities of fuel corrosion products were generated within these canisters. The open tops and screened-bottoms provided a pathway for this uranium-rich "canister sludge" (Makenas et al. 1997) to migrate to the basin floor. However, the fraction of this canister sludge that ended up on the KE basin floor (vs. the quantity that remained with the SNF through fuel cleaning operations) is not precisely known (Pearce and Klimper 2001). After SNF was removed from the KE Basin, sludge on the basin floors and pits was vacuumed into large containers in the KE Basin (2004/2005). Subsequently, this sludge was transferred via hose-in-hose and multiple pumps to the engineered containers (SCS-CON-240, 250, and 260) located in the KW Basin (sludge transfer from KE to KW was completed in 2007). Some composition variability is to be expected between the containers because precise control of which KE Basin sludge sources went into which of the three specific engineered containers was not a criterion for the transfer. Characterization of the core samples collected from Engineered Containers SCS-CON-240, 250, and 260 has been previously reported (Fiskum et al. 2011b).

At the KW Basin, before its use for N-Reactor fuel storage (1976), the basin pool was cleaned (i.e., any sludge from operation of the KW reactor was removed), and the concrete surface was coated. The N-Reactor fuel stored in the KW Basin was placed into closed fuel storage containers (each canister's lid was sealed with a Grafoil® gasket), and most fuel corrosion products were retained in the canisters during storage. As a result of the SNF pool management practices employed at the KW Basin, the quantity of sludge that accumulated in the KW Basin pit and floors was less than that in the KE Basin. Before commencement of fuel cleaning[(a)] and fuel transfer operations in the KW Basin, the small quantity of sludge on the basin floor was assumed to consist primarily of dust and sediment with a relatively low concentration of fuel corrosion products (fuel was in closed canisters). Similar to the KE Basin operations, sandfilters and ion exchange systems were used to maintain basin pool water quality, and materials from these operations (sand and possibly ion exchange material) are present in the sludge. After start-up of fuel cleaning and packaging operations in the KW Basin in 2000, some "fuel wash sludge," containing higher concentrations of uranium metal and fuel corrosion products (and likely ion exchange materials from KE Basin) was introduced to certain areas of the KW Floor. This fuel wash sludge is expected to make up the majority of the radionuclide inventory in the KW floor and pit sludge. Also, when recovering the fuel from the KW storage canisters, Grafoil® pieces and particles (from disintegration of the gasket material when lids were removed from the fuel storage canisters) were introduced to the KW Basin pool and floor. Sludge originating from the floor and pits in the KW Basin

(a) All fuel formerly stored in the KE Basin was transferred to the KW Basin where it was washed/cleaned, packaged, and removed (2000 to 2004).

has been consolidated into Engineered Containers SCS-CON-210 and 220. Characterization of the core samples collected from SCS-CON-220 has been previously reported (Fiskum et al. 2011b).

The particulate sludge generated from fuel packaging operations in the KW Basin was captured in equipment including Knock-Out Pots (KOPs), strainers and settler tubes that are part of the KW Integrated Water Treatment System (IWTS). Some of this fuel wash sludge also escaped to the KW Basin floor and pits. KOP sludge (which includes the associated strainers) is functionally represented by sludge that did not pass through ≤600[(a)] µm mesh screens. The sludge captured within the 10 settler tanks passed through these screens and is referred to as the Settler sludge. The Settler sludge was retrieved and transferred into Engineered Container SCS-CON-230. Characterization of the core samples collected from SCS-CON-230 has been previously reported (Shimskey et al. 2011).

As of November 2010, all sludge additions to SCS-CON-210 from the KW floor cleaning activities were completed. The objective of the sampling and analysis campaign is the characterization of the KW sludge after being consolidated in the Engineered Container SCS-CON-210.

Significant challenges exist in characterizing, handling, treating, and packaging the K Basin sludge:

- ***Presence of uranium metal***—Uranium metal is present in the sludge as determined by scientific investigations and observations of K Basin spent fuel handling activities. Uranium metal is pyrophoric and generates flammable hydrogen gas when reacted with anoxic water. While the sludge is safely stored in the water-filled basin, future handling and removal from the basin and transport must account for this issue during process steps.
- ***Remote handling***—Sludge is highly radioactive, and ion exchange material in the sludge has increased the quantity of ^{137}Cs retained within the sludge.
- ***Difficult to handle***—Sludge is a heterogeneous mixture of materials that consists primarily of very fine particles (<50 µm) but also contains up to ¼-in. pieces of debris and uranium metal particles. These materials range in density from about 1 (e.g., Grafoil®) to about 19 g/cm^3 (uranium metal). Experience has shown that mobilization and transport of sludge (within the basins) is a tedious and time-intensive undertaking. Due to the very fine particulate nature of much of the sludge, the re-capture of mobilized sludge is also challenging. High transfer velocities are needed to address the density and upper particle size of the uranium metal. Existing characterization data within a given sludge type are highly variable because of its heterogeneous nature and variable sources.

A number of major sludge characterization campaigns (Makenas et al. 1996, 1997, 1998, 1999; and Baker and Welsh 2001[(b)]) have been conducted in the past. These campaigns were conducted before sludge consolidation/containerization efforts (i.e., the samples were collected from original sludge locations in the basins). The goals of the past characterization efforts were to provide baseline characterization data and specific associated data to support the sludge disposition alternative being pursued at the time of sampling. Results from these past efforts are summarized in the data quality objectives (DQO) document (Westcott et. al 2009) and KBC-33786, Rev. 2 QAPjP/SAP (Baker et al. 2009).

(a) The sludge was screened with either a 500 or 600 µm screen; ≤600 µm is used for convenience.
(b) Baker and Welsh. 2001. "Summary of Initial Laboratory Data from the Consolidated and Single Pull Core Sludge Sampling Campaigns." Memo 01-SNF/RBB004, To: KL Pearce, from RB Baker and TL Welsh, Fluor Hanford, May 10, 2001.

Based on stakeholder decisions as documented in the DQO (Westcott et. al 2009), a new sampling and characterization analysis campaign, starting in 2009, was deemed necessary because:

1. Transfers of floor and pit sludge into containers may have affected the sludge since it was previously characterized.

2. The fraction of KE canister sludge that migrated to the KE floor vs. the fraction that followed the SNF through the fuel washing process is uncertain.

3. There has been no direct characterization of some types of sludge (including sludge from the KW Basin floor, pits, and settler tubes).

4. A new design concept to treat the K Basin sludges is being developed, and updated characterization information and special process testing[(a)] will allow refinement of design concepts and flowsheets.

The DQO document provides in-depth specific problem statements, background, and identified required characterization data for the campaign.

1.4 Sample Collection/Sample Plan

Sludge that originally resided on the KE Basin floors and pits is now consolidated in Engineered Containers SCS-CON-240, 250, and 260. Floor and pit sludge originating in the KW Basin is now consolidated into Engineered Containers SCS-CON-210 and 220. Sludge in the KW Settler tanks has been retrieved and is now consolidated in SCS-CON-230. Engineered containers are 11 ft 10 in. long, 5 ft wide, and 13 ft tall, and each contains between 1 and 8.1 m^3 of sludge. Engineered Container SCS-CON-210 contains about 4 m^3 of sludge.[(b)] Figure 1.4 shows a cut-away view of an engineered container. (Note: Further details and background on engineered containers are provided in the QAPjP/SAP [Baker et al. 2009].)

During sludge retrieval and consolidation into the engineered containers, sludge was introduced from two inlet distribution heads positioned above the sludge bed (Figure 1.4). With this mode of sludge introduction, it is expected that the sludge was generally deposited in a series of horizontal layers or strata. The composition of the sludge in a given layer of the KW engineered container is expected to vary based on the distance from the two inlet distribution heads (see Westcott 2009, DQO Section 8.0). Larger and denser particles likely settled closer to the inlet distribution heads. Lighter/finer particles are more likely to be uniformly distributed throughout the given layer.

While some mixing occurred during containerization, it is possible that some compositional identity (based on original sludge locations) was maintained. As such, there are potential layers in containers that may correspond to the location from which the sludge was collected and retrieved. Additionally, there may also be some compositional variation or stratification due to settling of sludge during the transfer

(a) Results from settling tests defined in the DQO and QAPjP/SAP for Engineered Containers SCS-CON-220, 240, 250, and 260 have been previously reported (Fiskum et al. 2010). Settler sludge settling tests have been conducted and reporting is pending as of August 2011. As of June 2011, settling tests have been deleted from processing scope; instead the samples of SCS-CON-210 sludge will be processed in a prototypic treat-and-transfer system with added flocculating agent (this process testing sequence includes settling steps).

(b) Interoffice Memorandum CHPRC-1004013, from RK Nissen to JF Thompson, November 17, 2010.

process. Therefore, sludge within the containers may be stratified because of two primary factors: 1) the original location where the sludge was retrieved and 2) variability in sludge particle size and density during settling. This potential stratification was taken into account while developing the sampling plan (Westcott 2009, Section 8.0).

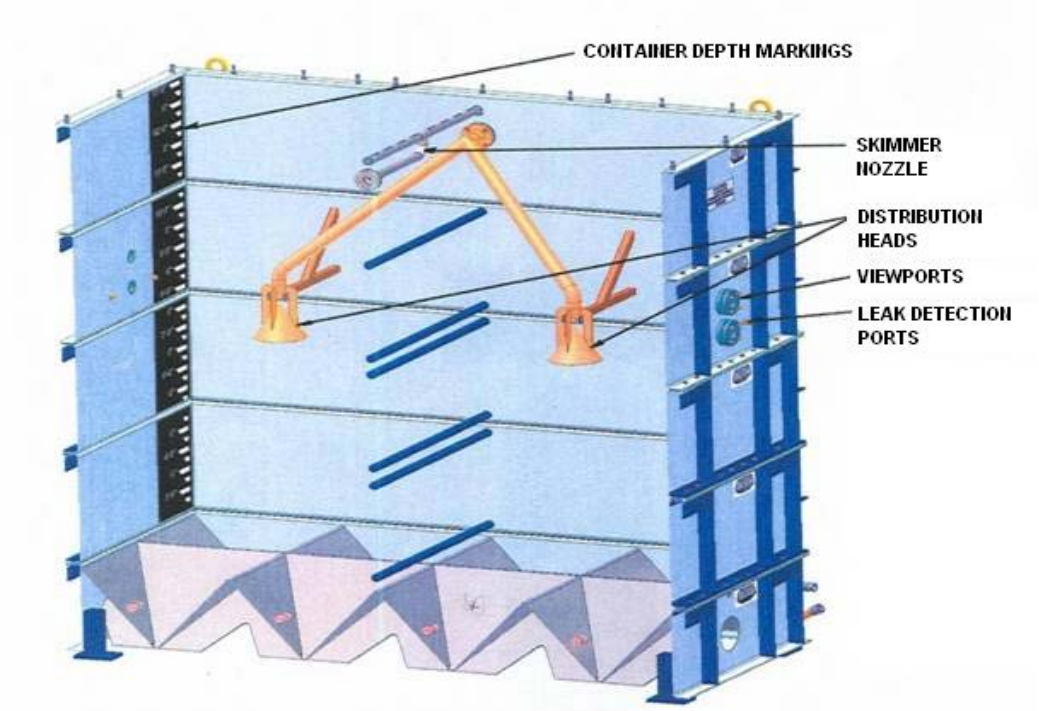
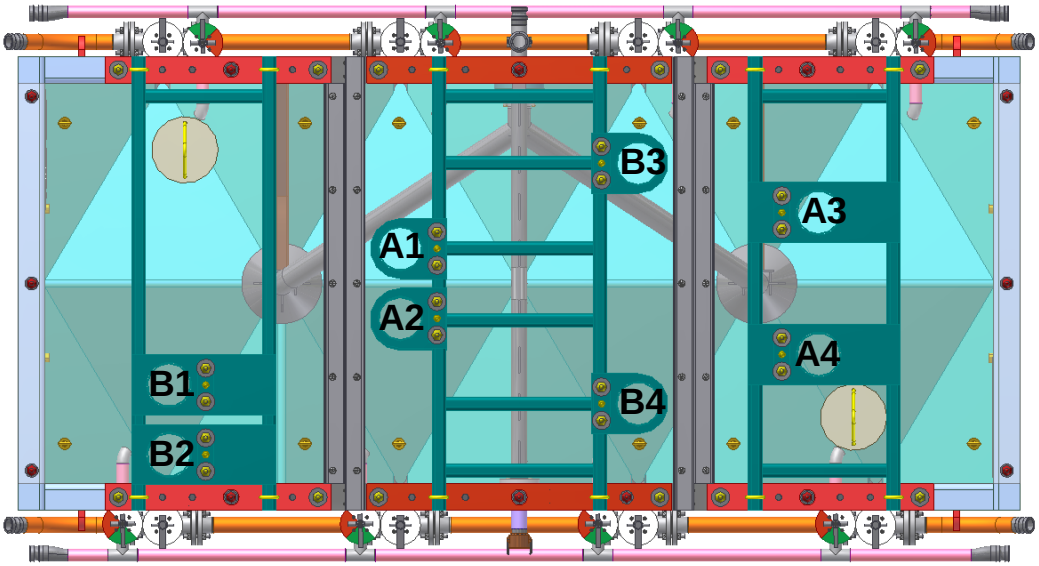


Figure 1.4. Cut-Away View of Engineered Containers SCS-CON-210 Through 260

As described in Baker et al. (2009), a probabilistic sample design was used, consistent with SW-846 and WIPP requirements for waste characterization. Full vertical cores of sludge were collected from each randomly selected sample location identified in each of the engineered containers. The sample locations available in each engineered container were constrained to the eight locations shown in Figure 1.5. The same eight locations are available for each of the engineered containers because all of the engineered containers are of the same design. Four of the sample locations are identified where sludge is more likely to contain larger and denser sludge particles (near distribution heads). These locations are identified with an "A" on Figure 1.5. Four of the sample locations are identified where sludge is less likely to contain larger and denser sludge particles (away from distribution heads). These locations are identified with a "B" on Figure 1.5. The "A" and "B" sample location identification has been maintained throughout the sampling and characterization campaign.

The DQO (Westcott 2009) specified that two representative vertical core samples (one "A" and one "B" sample) of the sludge in each of the three containers with KE-originating sludge (SCS-CON-240, 250, and 260) were required for supporting STP process design tasks. Four representative vertical core samples of the sludge in each of two containers with KW-originating sludge (SCS-CON-210 and 220) were required to provide the basis for determining the required NM accountability values and sludge characterization in support of STP process design tasks. Note, this KW-originating sludge was not sampled during the past characterization campaigns; therefore, more samples per container were collected.



Potential Core Sample Locations
A = Near Distribution Heads
B = Away from Distribution Heads

Figure 1.5. Sample Locations in Engineered Containers (top view looking down)

A design feature that is not apparent in Figure 1.5 is the clear polycarbonate cover bolted over the open top of the container to help control sludge overflow during filling and prevent the introduction of foreign material (dropped from above). K Basin operations require that these covers stay in place for process control of this material. Therefore, for sampling of the sludge, a series of holes were drilled through the polycarbonate covers. This drilling potentially introduced a small quantity of polycarbonate turnings to the sludge.

The sample-collection apparatus consists of an isolation tube (1 or 2 in. diameter) that is inserted through a hole in the top cover of the engineered container all the way down through the sludge to the bottom of the container (Baker et al. 2009, Section 3.1). For the sampling of SCS-CON-210, only 1-in. diameter isolation tubes were used. The sample system design incorporates a feature to provide a good seal between the container egg-crate bottom surface (Figure 1.4) and the end of the isolation tube. For sample collection, an extraction tube is slid inside the isolation tube, and sludge is suctioned 20 to 25 vertical feet into one of several sample polybottles configured in series. The sample polybottles are staged in a shielded cart located on the deck above the basin. Three heavy-walled 4-L sample polybottles are in the cart at one time (however, the third bottle is used only as a safety backup bottle and is not normally filled).

After samples were collected at the KW Basin into the 4-L sample polybottles, they were loaded into SSCs, which were then packaged into one or two SWBs. Each shipment consisted of two to four sample polybottles. Sample shipment was conducted in accordance with the shipping safety document (i.e., DOE 2009).

A separate data package documenting the sample collection and shipment activities conducted at the KW Basin has been prepared (Thompson and Ross 2011).

2.0 Sample Receipt and Processing

Four top-to-bottom core samples were collected from November to December 2010 from Engineered Container SCS-CON-210 located in the KW Basin. Each core sample was packaged in multiple 4-L polypropylene bottles (polybottles) and shipped in heavily shielded sample containers (i.e., lead-lined "Super Pigs") within a series of SWBs to the HLRF located at PNNL's RPL. Samples were received under COC (COC copies are available in Appendix A). Table 2.1 provides a summary of the polybottle ID, COC number, sampling date at the K Basin, sample receipt date at the HLRF, opportunistically measured gamma dose rates, and nominal measured volumes. Note that samples with the prefix "KW210" represent core samples from Engineered Container SCS-CON-210. The second alphanumeric designation (i.e., A1, A4, B1, and B3) represents the core ID (as shown in Figure 1.5). The final letter (e.g., -A, -B, -C) designates the sequential polybottle container of the core sample material. A single core sample was collected in four to six polybottles.

The gamma dose rates listed in Table 2.1 were opportunistically determined from each sludge sample container. Measurements were collected from the bottom of the container during the transfer process into the HLRF using a handheld extension dosimeter (Telescan XEXTI-0017) or standard dosimeter (Eberline RO-20). Every sample polybottle was enclosed in a plastic shipping bag. Because the sludge had settled to the container bottom, each dose rate should represent the highest rate for the sample.

The core sample volumes provided in Table 2.1 were determined following sludge consolidation processing into safety-coated, volume-graduated, 960-mL Qorpak jars; re-agitation, and both a ~24-hr and >112-hr settling period. Each sample volume was correlated to a height determined from a decal ruler affixed to the jar. The sludge height uncertainty was estimated to be ±0.2 cm, corresponding to a volume uncertainty of ±12 mL (specific to the 960-mL Qorpak jar).[(a)]

Sludge from individual cores was reconstituted, subsampled, and processed in accordance with directions given in detailed TIs. Sludge was subsampled from each reconstituted core sample to be used for preparation of two container composites. The container composite sludge processing and subsampling was also detailed in a TI. Before implementation, each TI underwent extensive internal reviews and was then submitted for review and approval by the BTR. Table 2.2 provides a cross-reference of core sample and container composite sample to TI identification.

Figure 2.1 depicts the project scope and its correlation to TIs. Sludge characterization results from other engineered containers have been previously determined and reported (Fiskum et al. 2011a, b, Shimskey et al. 2011) and the associated TIs are shaded in gray. The SCS-CON-210 sludge characterization TIs (current scope discussed herein) are not shaded. Completed TI packages are provided to the BTR as Portable Document Format (PDF) documents in digital format.[(b)] A TI package includes the completed and reviewed TI, reviewed calculation spreadsheets (inclusive of tabulated TI data, data calculations supporting the physical property testing, and opportunistic settling rate observations), photograph contact sheets, measuring and test equipment (MT&E) identification and calibration records, key communications between the project and BTR, a surveillance performed by the PNNL project Quality Engineer (QE), and occurrence reports (as applicable). The QE reviewed the TIs

(a) Although the linear dimension uncertainty of ±0.2 cm was constant, the volume uncertainty changes with changing jar shapes.

(b) DVD-52578-PNNL-20650 Rev. 2, Section III, September 2013.

Table 2.1. Samples Received from Engineered Containers SCS-CON-210

Client Sample ID	COC No.	Sample Date	Receipt Date	Dose Rate, R/h		Reconstituted Sample Volume, mL[(c)]		
				[(a)]	[(b)]	~24 hr Settled	>112 hr Settled	As-Sampled
KW210-A1-A[(d)]	6695	12/29/10	1/4/11	3	2	224 (23 hr)	212 (136 hr)	168
KW210-A1-B	6695	12/29/10	1/4/11	-	0.45			
KW210-A1-C	6697	12/30/10	1/4/11	-	0.045			
KW210-A1-D	6697	12/30/10	1/4/11	-	0.015			
KW210-A4-A[(d)]	6662	11/17/10	11/18/10	2	-	199 (23 hr)	193 (281 hr)	174
KW210-A4-B	6662	11/17/10	11/18/10	0.35	-			
KW210-A4-C	6664	11/18/10	12/2/10	4	-			
KW210-A4-D	6664	11/18/10	12/2/10	0.3	-			
KW210-A4-E	6668	11/19/10	12/6/10	0.06	-			
KW210-A4-F	6668	11/19/10	12/6/10	0.008	-			
KW210-B1-A[(d)]	6674	12/1/10	12/8/10	-	0.12	616 (24.5 hr)	560 (113 hr)	523
KW210-B1-B	6674	12/1/10	12/8/10	-	0.25			
KW210-B1-C	6678	12/7/10	12/14/10	-	0.27			
KW210-B1-D[(e)]	6678	12/7/10	12/14/10	-	0.07			
KW210-B1-F	6682	12/9/10	12/16/10	-	0.22			
KW210-B1-G	6682	12/9/10	12/16/10	-	0.08			
KW210-B3-A[(d)]	6686	12/15/10	12/27/10	1	-	585 (23 hr)	554 (136 hr)	485
KW210-B3-B	6686	12/15/10	12/27/10	0.5	-			
KW210-B3-C	6689	12/17/10	12/27/10	0.8	-			
KW210-B3-D[(f)]	6689	12/17/10	12/27/10	0.3	-			
KW210-B3-E	6692	12/27/10	12/29/10	0.7	-			
KW210-B3-F	6692	12/27/10	12/29/10	0.045	-			

(a)/(b) Dose rates were taken at contact and uncorrected from the bottom of the sample container while it was still encased in the plastic (shipping) bag. The dosimeter was a Telescan XEXTI-0017 (a) or the Eberline RO-20 (b). Historically, the Telescan XEXTI-0017 has provided slightly higer dose rates than the Eberline RO-20. This is likely due to the different radiation sensing area geometry. The Telescan has a cylindrical, 360 degree, sensing geometry and the Eberline has a flat, one-directional sensing area.

(c) The volumes are accurate to only two significant figures; the third significant figure is shown for information only. The settled volume represents the volume of the core sample after re-agitation and settling for ~24 hr. The >112 hr settling volume represents the core composites after a second re-agitation and settling for 112 hr or more. The as-sampled volume represents the core sample volume after excess water was removed and and sludge was mixed prior to subsampling.

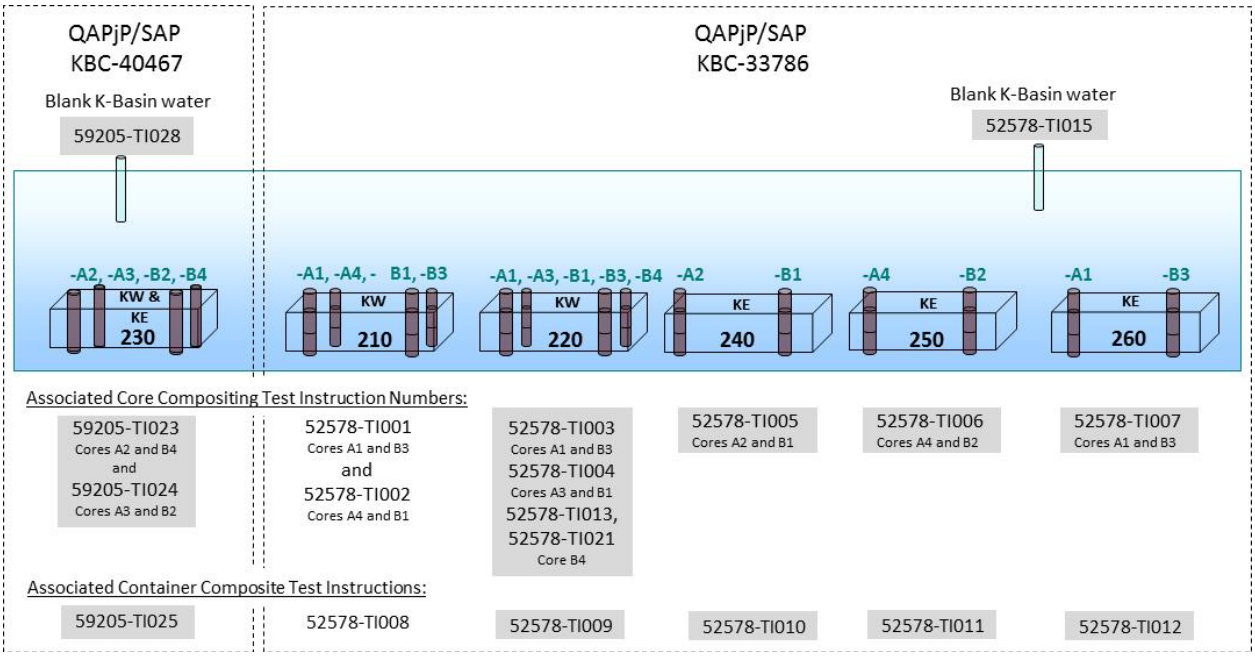
(d) One subsample collected from each reconstituted core sample was inadvertently given the client sample ID. The client sample IDs (e.g., KW210-A1-A, KW210-A4-A) designated in this table are different samples from the samples identically identified in Tables 2.3, 2.4, 2.5 and 2.6.

(e) Polybottle ID KW210-B1-E was not provided to PNNL because of a sampling equipment problem at KW Basin. A sample bottle was initially assigned this identification but found to contain no significant sludge material, hence it was not shipped from KW Basin to PNNL (Thompson and Ross 2011).

(f) Polybottle ID KW210-B3-D contained a flow deflector tube when received into HLRF (Thompson and Ross 2011).

Table 2.2. Cross-Reference of Core Sample ID to Test Instruction ID and Title

Core ID	TI ID	TI Title
KW210-A1 KW210-B3	52578-TI001	K West Basin Sample Receipt and Preparation of Consolidated Core Composites: SCS-CON-210-A1 and SCS-CON-210-B3
KW210-A4 KW210-B1	52578-TI002	K West Basin Sample Receipt and Preparation of Consolidated Core Composites: SCS-CON-210-A4 and SCS-CON-210-B1
Container Composites (KW210-CC-A & KW210-CC-N)	52578-TI008	Preparation of K West Basin Consolidated Container Composite for SCS-CON-210



Note: The K Basin indicators (i.e., KW and KE) refer to the source of the sludge in the containers. The three-digit numbers (e.g., "210") are abbreviations of the full engineered container designations (e.g., SCS-CON-210). The gray shading indicates scope that was previously completed and reported. The focus of this report is the SCS-CON-210 container material.

Figure 2.1. Depiction of K Basin Sludge Core and Equipment Blank Sampling Effort with Identification of Scope Associated with Specific Test Instructions

to evaluate completeness, that all entries were recorded properly, the errors were corrected appropriately and when needed, that justification was present to describe data corrections, that any issues affecting data quality were documented as Occurrence Reports, and that the attachments to the TI were at the stage to become a project record. Additionally, TIs were reviewed to assure that technical reviews were performed, issues were dispostioned, and that data were traceable to point of use, generally Excel spreadsheets, which also required technical review and comment/issue resolution.

2.1 Core Reconstitution and Subsampling Overview

Samples were transferred into the HLRF and allowed to settle for at least one day in the as-received 4-L polybottle. Sludge height measurements for the polybottles were collected; however, for most cases the sludge volumes were so low that meaningful volume measures could not be determined from the settled sludge height (see DVD-52578-PNNL-20650 Rev. 2, Section III, September 2013). Most of the clarified supernatant water (or liquid) was removed by vacuum-assisted siphoning. Figure 2.2 shows a schematic of the fluid removal system. Components (i.e., wand and rods) in the system were made of stainless steel, and Tygon® pressure tubing. Some water (~200 mL) was left with the solids. This concentrated sludge slurry was agitated, suspending solids in the remaining solution, and then poured into a 960-mL, wide-mouth, safety-coated, glass jar. A stainless steel canning jar funnel equipped for stability (welded pipe) and manipulator use (ring clamp) was positioned over the compositing jar to aid quantitative sludge transfer (see Figure 2.3). Figure 2.4 shows a flow diagram of the overall core reconsitituion process and targeted data collection.

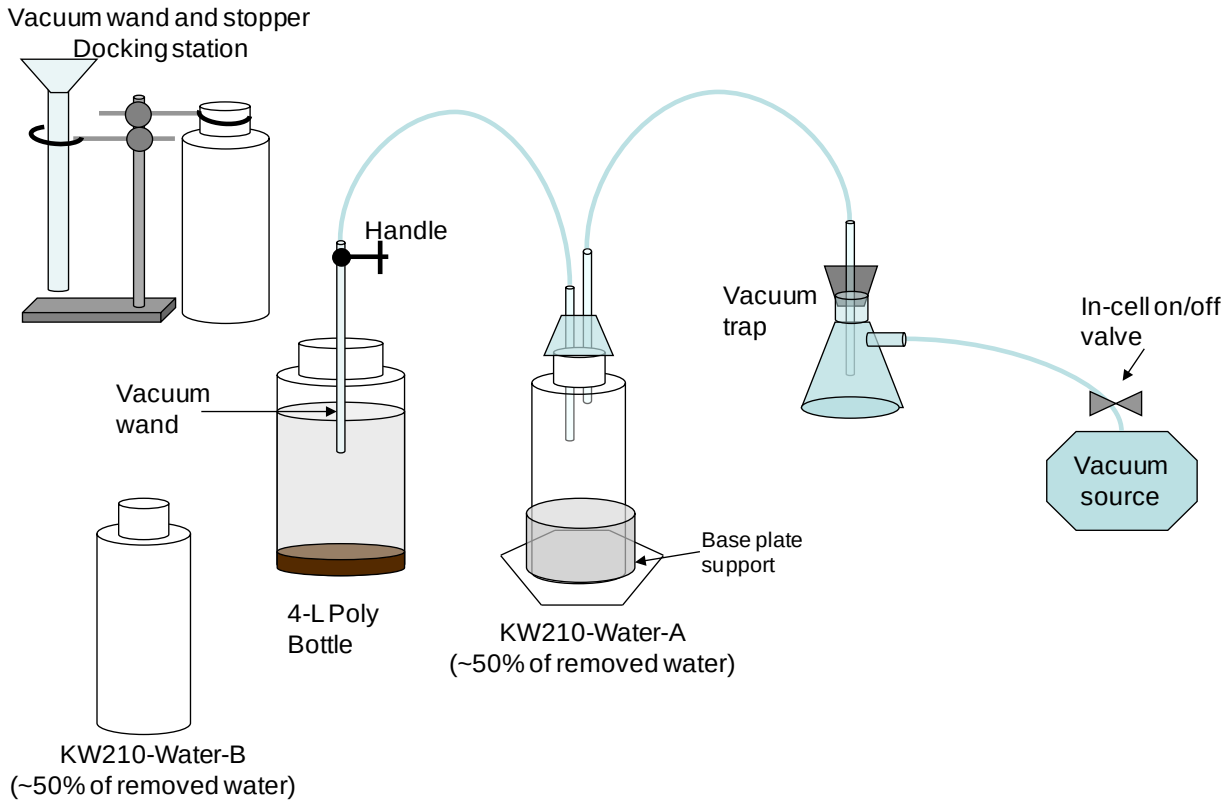


Figure 2.2. System Schematic for Fluid and Sludge Removal

Notes:

- *The pipe welded to the stainless steel funnel is provided for funnel stability in the bottle. The ring clamp on the pipe supported manipulator finger rings (opposite side in image).*
- *The jar was set into a special holder to minimize chances for tipping.*
- *An aluminum baking pan was used to provide a clean work surface for handling equipment.*
- *Part of the docking station is visible at the left.*

Figure 2.3. Prototypic Image of Sludge Core Reconstitution Step into the 960-mL Jar with Modified Funnel

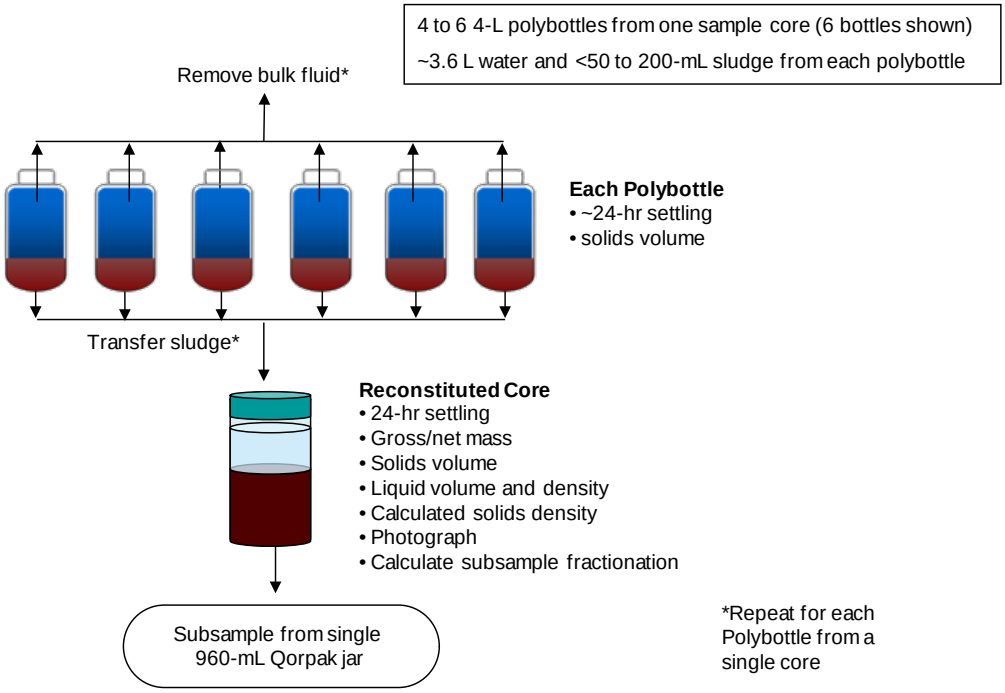
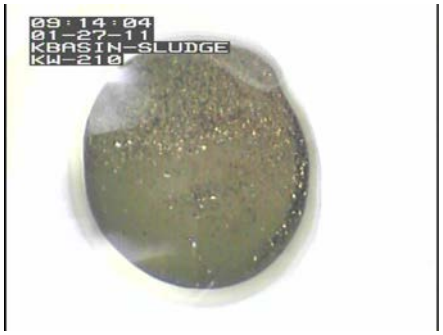


Figure 2.4. Schematic of a Core Sample Reconstitution

K Basin water was added to the residual solids in the polybottle for entrainment and removal by vacuum transfer. The entrained residual solids were collected in a separate glass bottle with a tapered neck; this design feature allowed for efficient solids transfer after collection and settling. The process was repeated until all solids were effectively removed from the 4-L polybottle (i.e., effectively, a quantitative transfer, as verified by visual inspection using an in-cell camera). Examples of full and emptied polybottles depicting the typically achieved quantitative transfers are shown in Figure 2.5.

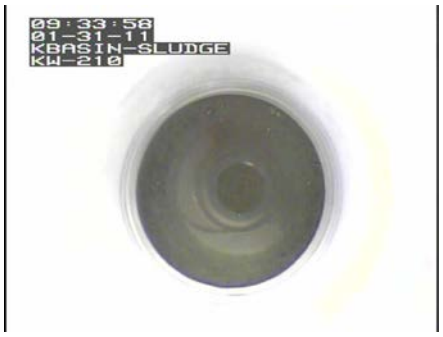
Bottle KW210-A1-B Prior to Bulk Transfer

Bottle KW210-A1-B After Final Rinse

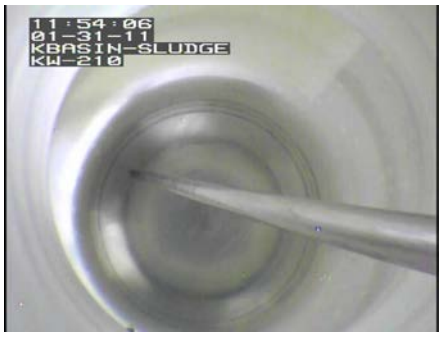


Bottle KW210-A4-C Prior to Bulk Transfer

Bottle KW210-A4-C After Final Rinse



Bottle KW210-B1-C Prior to Bulk Transfer



Bottle KW210-B1-C After Final Rinse

Bottle KW210-B3-C Prior to Bulk Transfer

Bottle KW210-B3-C After Final Rinse

Note: Images are looking down into the polybottle with in-cell camera.

Figure 2.5. Examples of Polybottles with Starting Concentrated Slurry and Emptied Polybottles Following Sludge Removal

The sludge removal and collection process was repeated for each core sample component, combining the sludge into a single jar and thus reconstituting the core sample (originally distributed across four to six 4-L polybottles) into a single jar (Figure 2.4). In all cases, the core sample volume was sufficiently small that the entire core sample could be reconstituted into a single 960-mL Qorpak jar and still allow sufficient headspace for efficient mixing. Once the core sample was reconstituted into the jar, the sludge was suspended by swirling and turning the jar. Then, the sludge was allowed to settle for ~24 hr. The 24-hr settled sludge volume was determined based on the sludge height. Opportunistic characterization data and photographs were collected on each core sample: settling behavior during the 24-hr settling period, the gross mass, total and settled sludge volumes, and fluid head volume were determined. The settled sludge mass and density were calculated from these parameters.

The reconstituted sludge samples were thickened before final mixing and subsampling by removing the fluid head from the sludge, mixing the wet sludge with a stainless steel spoon, allowing the sludge to settle, and removing the fluid head again (Figure 2.6). The process was repeated until the sludge appeared to have sufficient strength to suspend dense particles. Final dewatering was conducted by carefully tipping the sample jar to create a pool of fluid with enough depth to be retrieved with a transfer pipet. To accomplish this, an angle-vise, fitted with a custom-made mounting block, was used to hold the 960-mL jars at a given angle (Figure 2.7).[(a)] A transfer pipet was then used to retrieve the pooled water above the sludge.

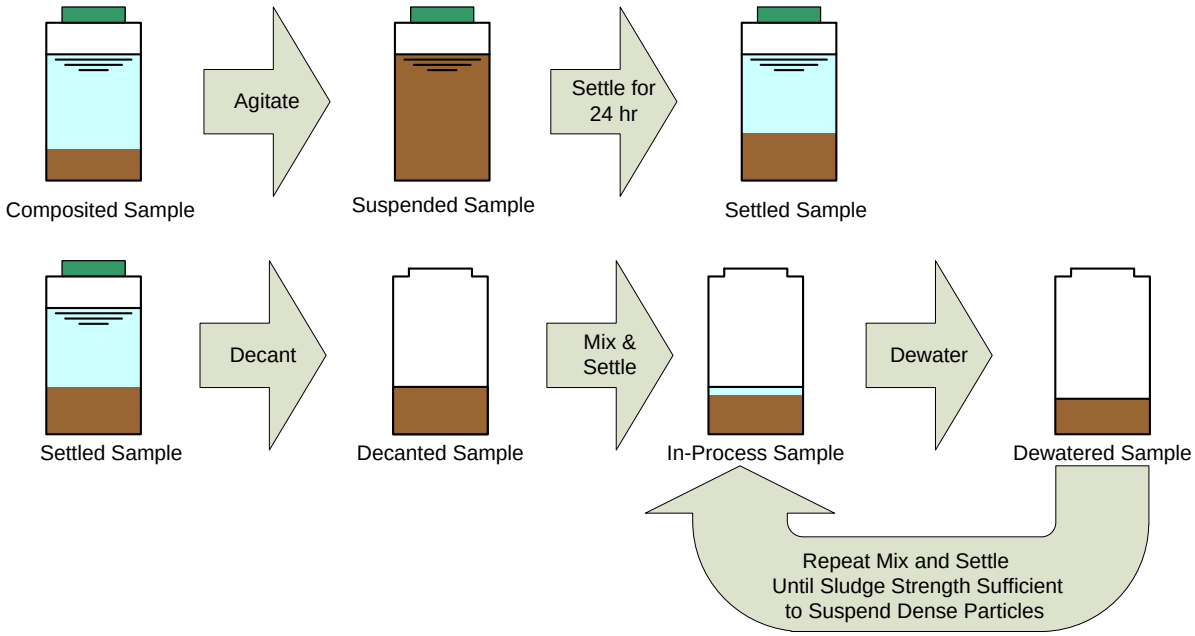


Figure 2.6. Sample Preparation for Subsampling

(a) The angle-vise was also used to hold the polybottle still for uncapping and for initial gross water removal.

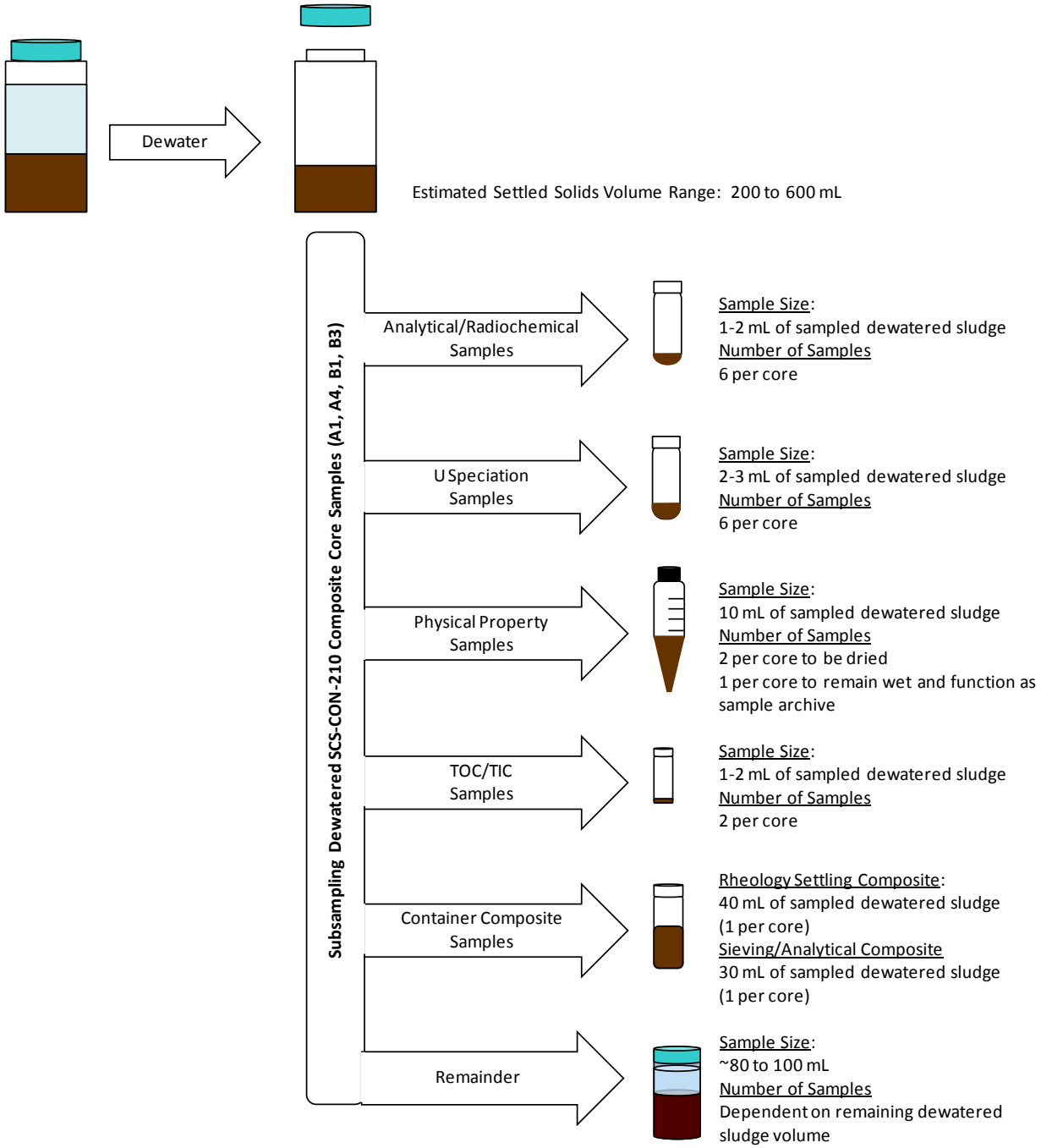
Vise is holding the compositing jar at angle for continued sludge dewatering activity.

Vise holding the 4-L polybottle still for uncapping and water removal.

Figure 2.7. Angle-Vise in Prototypic Uses

The sludge was thickened to provide more uniform sludge subsamples that would be less prone to particle segregation based on particle size or density. After achieving a thickened state, and before subsampling, the sludge was homogenized by mixing with a stainless steel spoon for 5 min. For subsampling, all subsample containers were staged, and aliquots of mixed/thickened sludge were transferred to the containers. Each subsampling operation was conducted in a single uninterrupted session at the hot cell window to minimize differences in the wt% solids being transferred into each subsample container.

Multiple subsamples were pulled from the mixed and thickened sludge to satisfy a multiplicity of analytical objectives (Figure 2.8). Additional characterization samples were collected in case of disagreement between the primary sample and duplicate, observed analytical problems, or required reanalysis. Test instructions 52578-TI001 and 52578-TI002 defined a specific subsampling strategy that required at least 140 mL of sludge be available per reconstituted core sample (as-sampled basis). The majority of this volume (80 mL) was to create a container composite. However the volumes of the "A" core sludge samples (i.e., A1 and A4) were about half of the anticipated volumes and a large population of particles were observed that would likely not pass through the 500-µm pore size sieve. Reducing the volume of <500-µm (referred to as "−500-µm") sieve fraction material would likely confound the ability to collect shear strength and flow curve measurements on this fraction (a situation previously observed on the SCS-CON-220 sample [Fiskum et al. 2011b] which precluded the ability to obtain reliable rheological data). Therefore the approach to the container composite creation and testing was modified with the BTR's concurrence. Two samples (30 mL and 40 mL) were collected, instead of one 80-mL sample, from each core sample to create two independent container composites. One container composite was destined to support the analytical characterization and the other was to support the rheological characterization (shear strength only, the flow curve analysis was cancelled per direction/concurrance of the BTR). The container composite created specifically for shear strength testing was assured to be of sufficient volume to support the test. Engineered Container SCS-CON-210 composite testing is described in Section 2.2. The original subsampling strategy was also modified to eliminate one contingency physical property subsample from each core sample.



Note: Subsamples collected to create the container composite were adjusted to 30 mL and 40 mL from each core sample. This was done to allow creation of an independent composite for rheology testing, removing the strictures of obtaining enough −500-μm sieve size material to support rheology as well as characterization.

Figure 2.8. Overall Processing and Subsampling Scheme for the SCS-CON-210 Core Samples

The "B" core sludge samples (i.e., B1 and B3) had more than sufficient volume to support all testing. The remaining "B" core sludge was distributed among three to four 120-mL Qorpak jars in readiness for long term storage tests, as directed by the BTR. The "A" core sludge samples (i.e., A1 and A4) were essentially consumed from subsampling.

Cross-references between core sample IDs, subsample IDs (from TIs), as-sampled (wet) mass, ASO sample IDs, and ASR IDs are provided in Table 2.3 through Table 2.6. These IDs will be referenced throughout this report. Note that the subsample collection date is provided in the table annotations.

Table 2.3. Cross-Reference of SCS-CON-210 A1 Core Sample (KW210-A1) to TI Subsample ID, Subsample Mass, RPL/ASO ID, ASR ID, and General Analysis Scope

Subsample ID	As-Sampled Mass (g)[a]	RPL/ASO ID	ASR	Analysis
KW210-A1-1	245.83	---	---	Parent core sample
KW210-A1-A[b]	62.61	---	---	Container composite[c]
TI001-A1-B	2.05	11-0419-129-S	8798	Chemical analysis
TI001-A1-C	2.28	11-0419-129-D	8798	Chemical analysis duplicate
TI001-A1-D	2.49	---	---	Chemical analysis reserve
TI001-A1-E	2.08	---	---	Chemical analysis reserve
TI001-A1-F	2.27	---	---	Chemical analysis reserve
TI001-A1-G	2.50	---	---	Chemical analysis reserve
TI001-A1-H	1.3503	11-0419-C-S	8798	TOC/TIC
TI001-A1-J	1.0210	11-0419-C-D	8798	TOC/TIC duplicate
TI001-A1-K	14.74	---	---	Primary physical properties
TI001-A1-L	18.50	---	---	Primary physical properties
TI001-A1-M	20.07	---	---	Analytical archive; supplemental physical properties
TI001-A1-N	44.62	---	---	Container composite[c]
TI001-A1-P	3.89	11-0419-107-S	8798	Uranium speciation
TI001-A1-Q	4.55	11-0419-107-D	8798	Uranium speciation duplicate
TI001-A1-R	4.09	---	---	Uranium speciation reserve
TI001-A1-S	4.79	---	---	Uranium speciation reserve
TI001-A1-T	4.14	---	---	Uranium speciation reserve
TI001-A1-U	4.79	---	---	Uranium speciation reserve
KW210-A1-Archive-A	16.76	---	---	Archive sample
KW210-A1-Archive-B	NM[d]	---	---	Archive sample
KW210-A1-Archive-C	NM[d]	---	---	Archive sample

(a) Subsampling date: February 22, 2011.

(b) Inadvertently one sample was given an ID identical to a client sample ID. The sample designated "KW210-A1-A" here is a different sample from the sample identically identified in Table 2.1.

(c) Two subsamples from each reconstituted core sample were collected to create two separate container composites according to TI 52578-TI008. Before compositing, the physical properties and settling behavior of these samples were evaluated.

(d) Samples were obtained through composite jar washes and as-sampled masses were not measured.

NM: Not Measured.

The net sample masses were calculated from data collected in TI 52578-TI001.

Gray shading indicates samples that were collected but not used.

Table 2.4. Cross-Reference of SCS-CON-210 A4 Core Sample (KW210-A4) to TI Subsample ID, Subsample Mass, RPL/ASO ID, ASR ID, and General Analysis Scope

Subsample ID	As-Sampled Mass (g)[a]	RPL/ASO ID	ASR	Analysis
KW210-A4-1	281.64	---	---	Parent core sample
KW210-A4-A[b]	74.51	---	---	Container composite[c]
TI002-A4-B	2.30	11-0420-129-S	8798	Chemical analysis
TI002-A4-C	2.61	11-0420-129-D	8798	Chemical analysis duplicate
TI002-A4-D	2.16	---	---	Chemical analysis reserve
TI002-A4-E	2.18	---	---	Chemical analysis reserve
TI002-A4-F	2.55	---	---	Chemical analysis reserve
TI002-A4-G	2.03	---	---	Chemical analysis reserve
TI002-A4-H	1.4289	11-0420-C-S	8798	TOC/TIC
TI002-A4-J	2.0583	11-0420-C-D	8798	TOC/TIC duplicate
TI002-A4-K	14.67	---	---	Primary physical properties
TI002-A4-L	16.76	---	---	Primary physical properties
TI002-A4-M	23.34	---	---	Analytical archive; supplemental physical properties
TI002-A4-N	49.38	---	---	Container composite[c]
TI002-A4-P	4.80	11-0420-107-S	8798	Uranium speciation
TI002-A4-Q	3.99	11-0420-107-D	8798	Uranium speciation duplicate
TI002-A4-R	4.37	11-0967-107-S	8824	Uranium speciation
TI002-A4-S	4.36	11-0967-107-D	8824	Uranium speciation duplicate
TI002-A4-T	4.30	---	---	Uranium speciation reserve
TI002-A4-U	4.02	---	---	Uranium speciation reserve
KW210-A4-Archive-A	37.22	---	---	Archive sample
KW210-A4-Archive-B	NM[e]	---	---	Archive sample
KW210-A4-Archive-D[d]	NM[e]	---	---	Archive sample

(a) Subsampling date: February 21, 2011.

(b) Inadvertently one sample was given an ID identical to a client sample ID. The sample designated "KW210-A4-A" here is a different sample from the sample identically identified in Table 2.1.

(c) Two subsamples from each reconstituted core sample were collected to create two separate container composites according to TI 52578-TI008. Before compositing, the physical properties and settling behavior of these samples were evaluated.

(d) KW210-A4-Archive-C was not created.

(e) Samples were obtained through composite jar washes and as-sampled masses were not measured.

NM: Not Measured.

The net sample masses were calculated from data collected in TI 52578-TI002.

Gray shading indicates samples that were collected but not used.

Table 2.5. Cross-Reference of SCS-CON-210 B1 Core Sample (KW210-B1) to TI Subsample ID, Subsample Mass, RPL/ASO ID, ASR ID, and General Analysis Scope

Subsample ID	As-Sampled Mass (g)[a]	RPL/ASO ID	ASR	Analysis
KW210-B1-1	612.10	---	---	Parent core sample
KW210-B1-A[b]	45.45	---	---	Container composite[c]
TI002-B1-B	1.84	11-0421-129-S	8798	Chemical analysis
TI002-B1-C	1.98	11-0421-129-D	8798	Chemical analysis duplicate
TI002-B1-D	2.15	11-0421-129-MS	8798	Chemical analysis matrix spike
TI002-B1-E	2.00	---	---	Chemical analysis reserve
TI002-B1-F	2.51	---	---	Chemical analysis reserve
TI002-B1-G	2.38	---	---	Chemical analysis reserve
TI002-B1-H	1.7868	11-0421-C-S	8798	TOC/TIC
TI002-B1-J	2.0942	11-0421-C-D	8798	TOC/TIC duplicate
TI002-B1-K	14.85	---	---	Primary physical properties
TI002-B1-L	12.51	---	---	Primary physical properties
TI002-B1-M	20.52	---	---	Analytical archive; supplemental physical properties
TI002-B1-N	37.28	---	---	Container composite[c]
TI002-B1-P	4.18	11-0421-107-S	8798	Uranium speciation
TI002-B1-Q	4.62	11-0421-107-D	8798	Uranium speciation duplicate
TI002-B1-R	3.97	11-0421-107-MS	8798	Uranium (metal-only) matrix spike
TI002-B1-S	4.41	---	---	Uranium speciation reserve
TI002-B1-T	4.15	---	---	Uranium speciation reserve
TI002-B1-U	4.08	---	---	Uranium speciation reserve
KW210-B1-Archive-1[d]	102.57	---	---	Archive sample
KW210-B1-Archive-2[d]	89.69	---	---	Archive sample
KW210-B1-Archive-3[d]	108.75	---	---	Archive sample
KW210-B1-Archive-4[d]	104.13	---	---	Archive sample
KW210-B1-Archive-5	NM[e]	---	---	Archive sample
KW210-B1-Archive-6	NM[e]	---	---	Archive sample

(a) Subsampling date: February 17, 2011.

(b) Inadvertently one sample was given an ID identical to a client sample ID. The sample designated "KW210-B1-A" here is a different sample from the sample identically identified in Table 2.1.

(c) Two subsamples from each reconstituted core sample were collected to create two separate container composites according to TI 52578-TI008. Before compositing, the physical properties and settling behavior of these samples were evaluated.

(d) Samples were aliquoted into 120-mL Qorpak jars to support the long-term storage testing or as directed by the BTR.

(e) Samples were obtained through composite jar washes and as-sampled masses were not measured.

NM: Not Measured.

The net sample masses were calculated from data collected in TI 52578-TI002.

Gray shading indicates samples that were collected but not used.

Table 2.6. Cross-Reference of SCS-CON-210 B3 Core Sample (KW210-B3) to TI Subsample ID, Subsample Mass, RPL/ASO ID, ASR ID, and General Analysis Scope

Subsample ID	As-Sampled Mass (g)[a]	RPL/ASO ID	ASR	Analysis
KW210-B3-1	602.56	---	---	Parent core sample
KW210-B3-A[b]	54.24	---	---	Container composite[c]
TI001-B3-B	2.26	11-0422-129-S	8798	Chemical analysis
TI001-B3-C	2.24	11-0422-129-D	8798	Chemical analysis duplicate
TI001-B3-D	2.64	---	---	Chemical analysis reserve
TI001-B3-E	2.09	---	---	Chemical analysis reserve
TI001-B3-F	2.23	---	---	Chemical analysis reserve
TI001-B3-G	2.48	---	---	Chemical analysis reserve
TI001-B3-H	1.8483	11-0422-C-S	8798	TOC/TIC
TI001-B3-J	1.5905	11-0422-C-D	8798	TOC/TIC duplicate
TI001-B3-K	12.58	---	---	Primary physical properties
TI001-B3-L	12.97	---	---	Primary physical properties
TI001-B3-M	20.57	---	---	Analytical archive; supplemental physical properties
TI001-B3-N	38.27	---	---	Container composite[c]
TI001-B3-P	4.33	11-0422-107-S	8798	Uranium speciation
TI001-B3-Q	3.93	11-0422-107-D	8798	Uranium speciation duplicate
TI001-B3-R	4.23	---	---	Uranium speciation reserve
TI001-B3-S	4.59	---	---	Uranium speciation reserve
TI001-B3-T	4.13	---	---	Uranium speciation reserve
TI001-B3-U	4.40	---	---	Uranium speciation reserve
KW210-B3-Archive-1[d]	92.84	---	---	Archive sample
KW210-B3-Archive-2[d]	94.11	---	---	Archive sample
KW210-B3-Archive-3[d]	96.70	---	---	Archive sample
KW210-B3-Archive-4	NM[e]	---	---	Archive sample

(a) Subsampling date: February 18, 2011.

(b) Inadvertently one sample was given an ID identical to a client sample ID. The sample designated "KW210-B3-A" here is a different sample from the sample identically identified in Table 2.1.

(c) Two subsamples from each reconstituted core sample were collected to create two separate container composites according to TI 52578-TI008. Before compositing, the physical properties and settling behavior of these samples were evaluated.

(d) Samples were aliquoted into 120-mL Qorpak jars to support the long-term storage testing or as directed by the BTR.

(e) Sample was obtained through composite jar washes and as-sampled masses were not measured.

NM: Not Measured.

The net sample masses were calculated from data collected in TI 52578-TI001.

Gray shading indicates samples that were collected but not used.

Samples processed with the PNL-ALO-129 acid digestion procedure, *HNO_3-HCl Acid Extraction of Solids Using a Dry-Block Heater,* for chemical and radioisotope analyses (e.g., TI001-A1-B) were further processed after much of the initial analytical scope was completed. Aliquots of the acid digestate were prepared for U and Pu isotopic measurement by TIMS once the total U and Pu concentrations were determined. The undissolved solids (UDS) were processed according to TI 52578-TI040[(a)] to obtain subsamples of 0.1 to 0.2 g for fusion and analysis. Additional aliquots of the UDS were processed according to TI 52578-TI040 to remove the bulk of the resin beads and subsequently the bead and non-bead fractions were weighed. One sample from each core that was depleted in resin beads was analyzed for crystalline structures by XRD. In all cases, the "daughter" samples were assigned modified IDs to delineate them from the parent samples. Table 2.7 cross-references the parent sample IDs to the daughter sample IDs for each of these analyses.

Table 2.7. Cross-reference of Core Sample IDs, TI Subsample IDs, and Original RPL/ASO IDs to TIMS and UDS Sample ID Assignments

			Daughter Sample Designation	
Core Sample ID	Subsample ID	Original RPL/ASO ID ASR 8798	TIMS U Preparation (Acid Digestate)	TIMS Pu Preparation (Acid Digestate)
KW210-A1	TI001-A1-B	11-0419-129-S	11-0419-129-S-U	11-0419-129-S-Pu
	TI001-A1-C	11-0419-129-D	11-0419-129-D-U	11-0419-129-D-Pu
KW210-A4	TI002-A4-B	11-0420-129-S	11-0420-129-S-U	11-0420-129-S-Pu
	TI002-A4-C	11-0420-129-D	11-0420-129-D-U	11-0420-129-D-Pu
KW210-B1	TI002-B1-B	11-0421-129-S	11-0421-129-S-U	11-0421-129-S-Pu
	TI002-B1-C	11-0421-129-D	11-0421-129-D-U	11-0421-129-D-Pu
KW210-B3	TI001-B3-B	11-0422-129-S	11-0422-129-S-U	11-0422-129-S-Pu
	TI001-B3-C	11-0422-129-D	11-0422-129-D-U	11-0422-129-D-Pu
			ASR 8878; RPL/ASO ID (UDS)	XRD (UDS)
KW210-A1	TI001-A1-B	11-0419-129-S	TI040-0419S-1b; 11-1279	--
	TI001-A1-C	11-0419-129-D	TI040-0419D-2b; 11-1280	TI040-419D-2x
KW210-A4	TI002-A4-B	11-0420-129-S	TI040-0420S-2c; 11-1281	--
	TI002-A4-C	11-0420-129-D	TI040-0420D-1c; 11-1282	TI040-420D-2x
			TI040-0420D-2c[(a)]; 11-1282D	--
KW210-B1	TI002-B1-B	11-0421-129-S	TI040-0421S-2b; 11-1283	--
	TI002-B1-C	11-0421-129-D	TI040-0421D-1b; 11-1284	TI040-421D-2x
KW210-B3	TI001-B3-B	11-0422-129-S	TI040-0422S-1c; 11-1285	--
	TI001-B3-C	11-0422-129-D	TI040-0422D-1c; 11-1286	TI040-422D-2x

(a) Processed as batch duplicate.

Note: The TIMS analysis was not conducted by the ASO; the ASO prepared purified Pu and U fraction samples for TIMS analysis.

(a) *Processing of the Undissolved Solids Samples from the K West Basin Core Samples Collected from SCS-CON-210 for Additional Analysis*, SK Fiskum, May 2011.

2.2 Container Compositing and Subsampling Overview

Container composite samples were intended to be created from the combination of equal settled sludge volumes from each core sample in accordance with the QAPjP/SAP. Subsamples collected for container composite preparation from each core sample were transferred from the HLRF to the Shielded Analytical Laboratory (SAL). Two independent container composites were prepared, one to support rheology testing and one to support sieving and characterization according to TI 52578-TI008.[(a)] Initial preparations of each container composite were identical. The "-A" samples (e.g., Table 2.3, KW210-A1-A) that had been collected from each core sample were combined to create a container composite (KW210-CC-A) in a 480-mL, safety-coated, Qorpak glass jar. Similarly, the "-N" samples (e.g., Table 2.3, TI001-A1-N), collected from each core sample, were combined to create a container composite (KW210-CC-N) in a 240-mL, safety-coated, Qorpak glass jar. An adhesive decal millimeter scale was affixed to each jar to facilitate determination of volume as a function of height. The contents were mixed by shaking the bottles and fully suspending the solids in a fluid head of deionized (DI) water. The solids were allowed to settle 24 hr. The settled solids volumes were tracked as a function of time (opportunistically) to create a settling curve. Physical-property information (i.e., settled solids volume, total slurry volume, and mass) were collected on each container composite.

Primary physical property subsamples were collected from each container composite sample. The headspace fluid was removed and set aside, and the sludge was mixed. The process was repeated until the sludge consistency had thickened enough to suspend dense particles. This thickening was performed to aid in providing more uniform sludge subsamples that would be less prone to particle segregation based on particle size or density. Two physical-property samples of thickened sludge were then collected from each container composite into 40-mL heavy-walled glass centrifuge tubes and then weighed. After subsampling, the reserved water was added back to the solids.

Preparation and processing of the KW210-CC-A container composite is summarized in Figure 2.9. The KW210-CC-A material was transferred directly to shear strength testing (see Section 2.9) without sieving. Preparation and processing of the KW210-CC-N container composite is summarized in Figure 2.10.

The KW210-CC-N container composite supporting analytical characterization was required to be fractionated into three sieve sizes: >2,000 μm, <2,000 and >500 μm, and <500 μm. Large-diameter sieves were used to process the wet sludge to best accommodate the large (~120 mL) sample volume. The sieves were 8-in. diameter × 2-in. deep and composed of stainless steel woven wire cloth (Advantech Manufacturing, New Berlin, WI and distributed by VWR). The sieves were manufactured in compliance with American Society for Testing and Materials (ASTM) Specification E-11. The sieve product summary is provided in Table 2.8.

(a) *Preparation of K West Basin Consolidated Container Composite for SCS-CON-210*. SK Fiskum, March 2011.

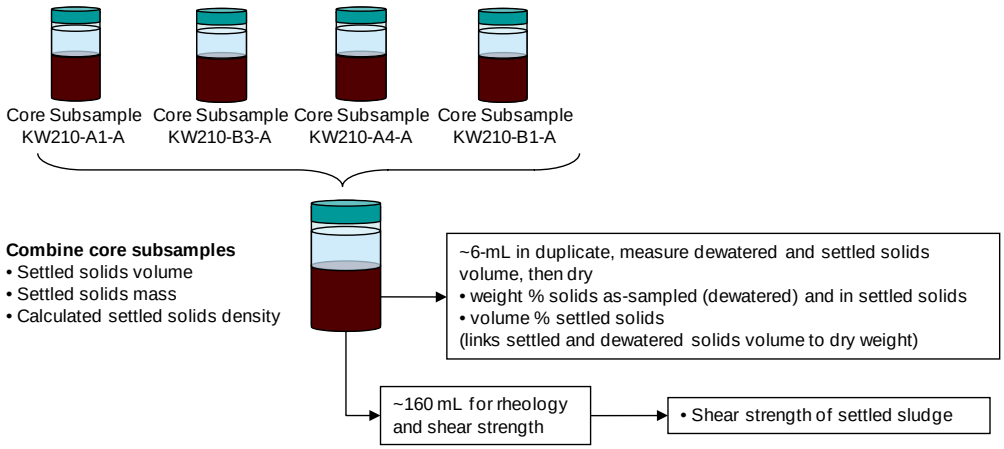
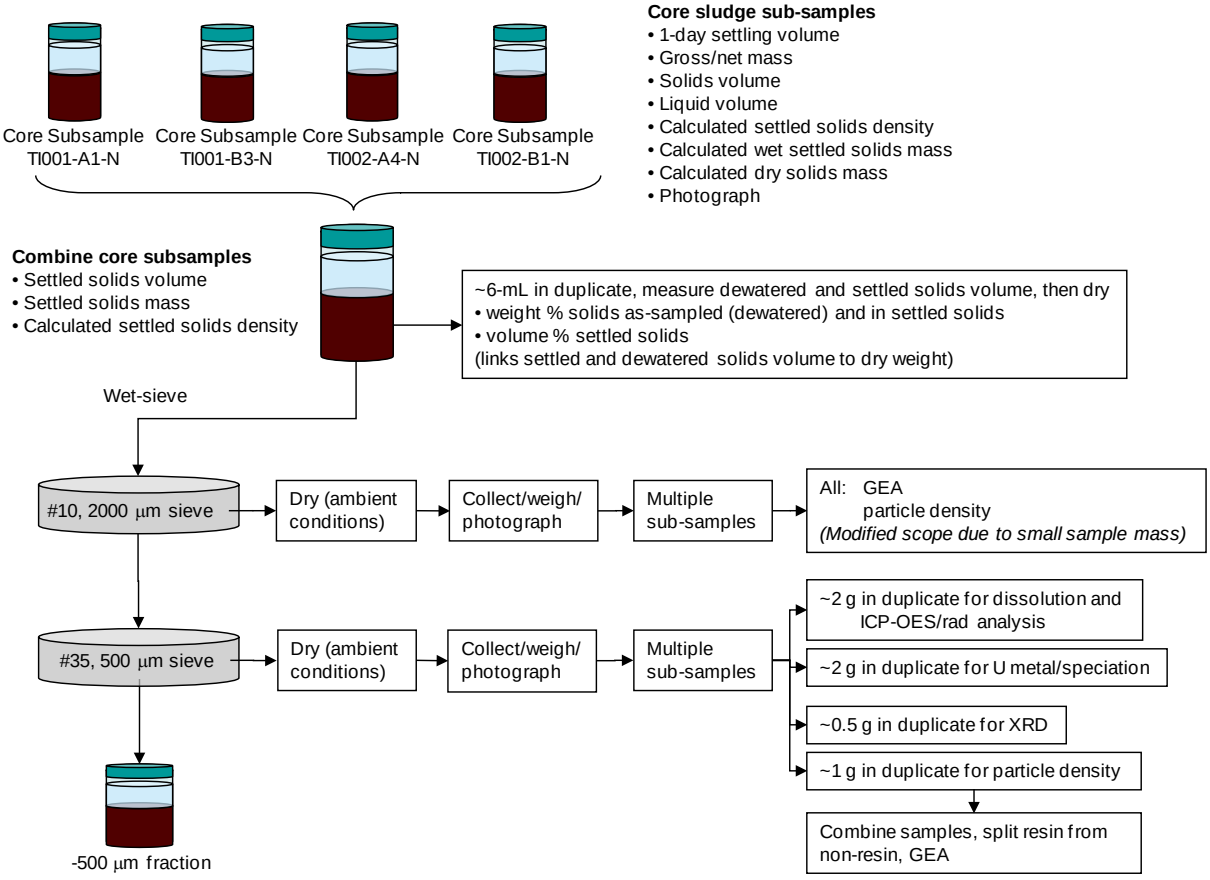


Figure 2.9. Overall Processing Schematic for the KW210-CC-A Container Composite Supporting Rheology



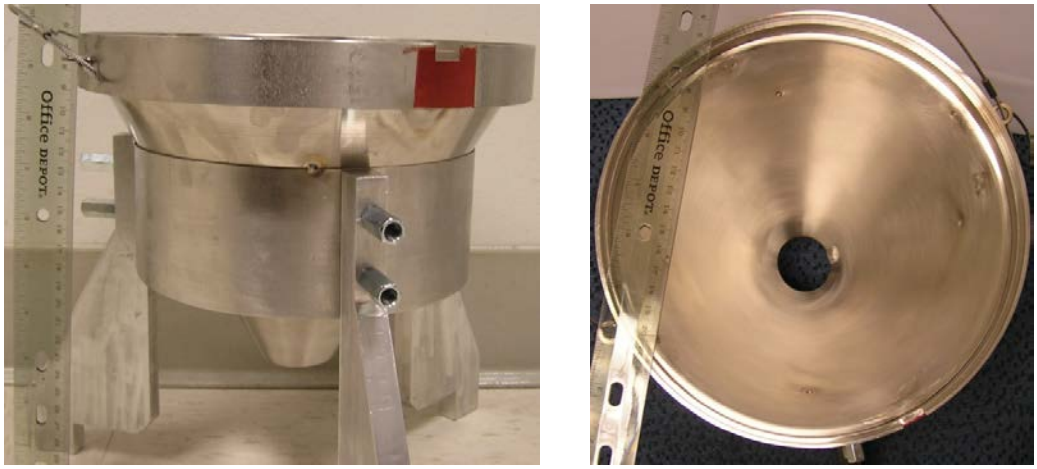
- *The >2,000-μm (referred to as "+2,000-μm") sieve fraction was too small to support the full analytical suite (targeted to equal the scope of the -2,000-μm/+500-μm sieve fraction; only GEA analysis followed by particle density measurement was conducted on the whole collected sample.*
- *Processing of the −500-μm container composite fines is defined in Figure 2.13.*

Figure 2.10. Initial Processing and Sieving Schematic of the KW210-CC-N Container Composite

Table 2.8. Sieve Parameters

US Series Sieve Designation	Standard Designation	Sieve Serial Number
#10	2.0 mm	10SS8F417114
#35	500 μm	35SS8F440521

The two sieves were stacked with the 2.0-mm mesh sieve on top of the 500-μm mesh sieve. These were placed onto a custom-made support stand welded to a stainless steel funnel (see Figure 2.11) such that the fines could be directed into a collection bottle after exiting the sieve. This configuration minimized the chance of losing solids. The sieves and sieve holder had been previously used on other K Basin sludge samples (Fiskum et al. 2011b). They were rinsed and wiped in-cell before use.



Side view Looking down

Figure 2.11. Fines-Collection Assembly Supporting Wet Sieving Activity

The solids were re-suspended and transferred onto the stacked sieve array. Water was used to facilitate quantitative transfer and to wash the solids through the sieve openings. The collection bottle was replaced as it was filled. Washing continued until solids visually appeared well-rinsed. The top sieve (2,000 μm) was removed. The solids continued to be washed on the 500-μm sieve until solids visually appeared well-rinsed, and no additional fines were falling into the collection bottle. The fines were collected in a rotation of four to five glass bottles; after the fines settled, the aqueous phase was retrieved and used in additional solids washing. The solids fractions collected on the sieve fabric were allowed to air dry overnight under ambient temperature conditions (typically 25 to 30°C) and air flow conditions. Solids were then retrieved from each sieve by inverting them over a duplicate sieve support assembly and then collecting the solids in a jar. The dry solids were weighed. The >2,000-μm (referred to as "+2,000-μm") fraction mass was small and the sample was not split but analyzed in its entirety. The −2,000-μm/+500-μm fraction was split using an inverted Y-connector attached to a funnel. Splits were repeated in a cascade fashion until appropriate analytical sizes were obtained (see example, Figure 2.12). This splitting effort was performed to produce representative subsamples.

The −500-μm wet sludge collected in the four to five bottles was composited into a single 480-mL glass jar. The combined solids were processed in a similar manner to the core composite material (see Figure 2.13). An opportunistic settling curve was collected on the bulk solids then multiple subsamples were pulled from the mixed and thickened sludge to satisfy a multiplicity of analytical objectives.

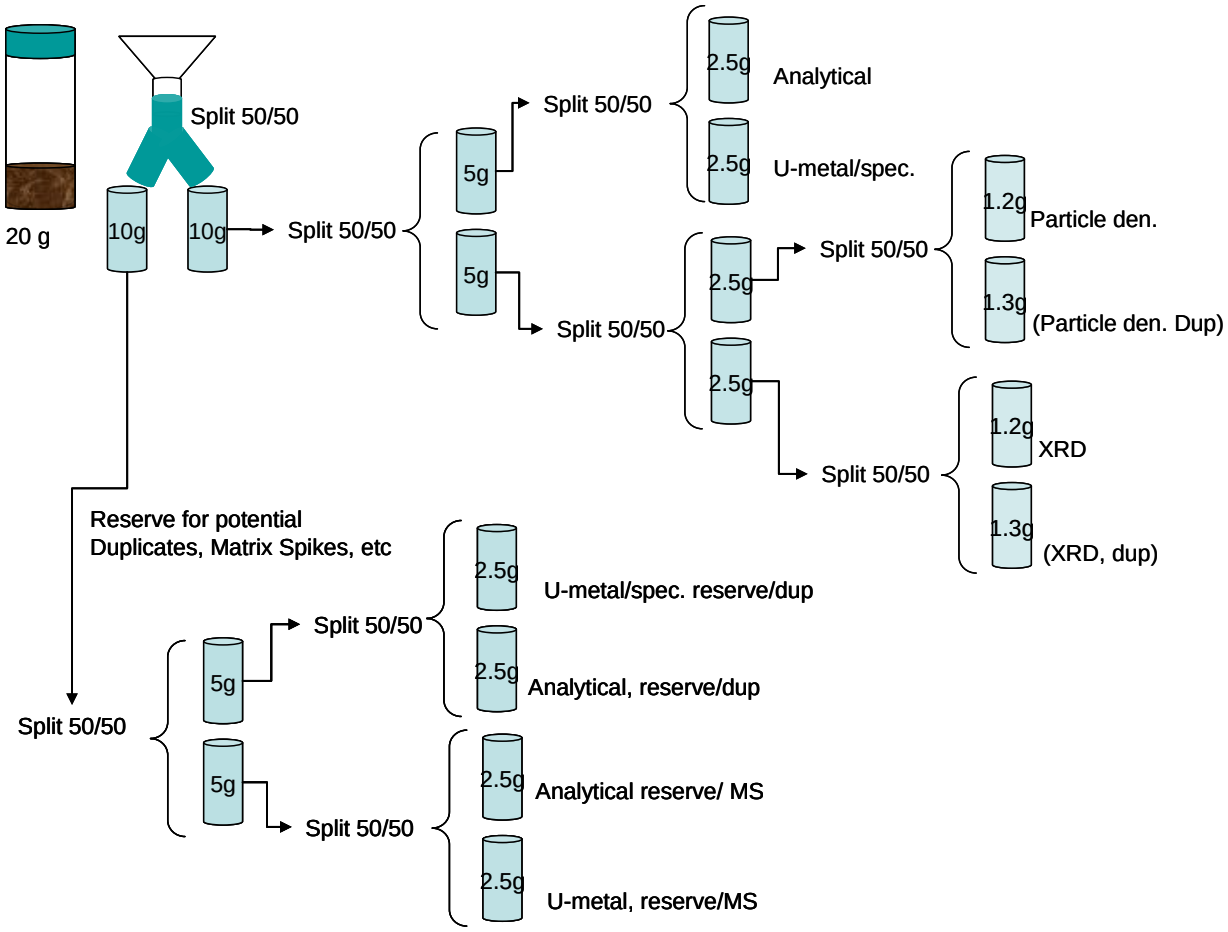


Figure 2.12. Prototypic Subsampling of the Dry Sludge Solids Collected on the 500-µm Mesh Sieve

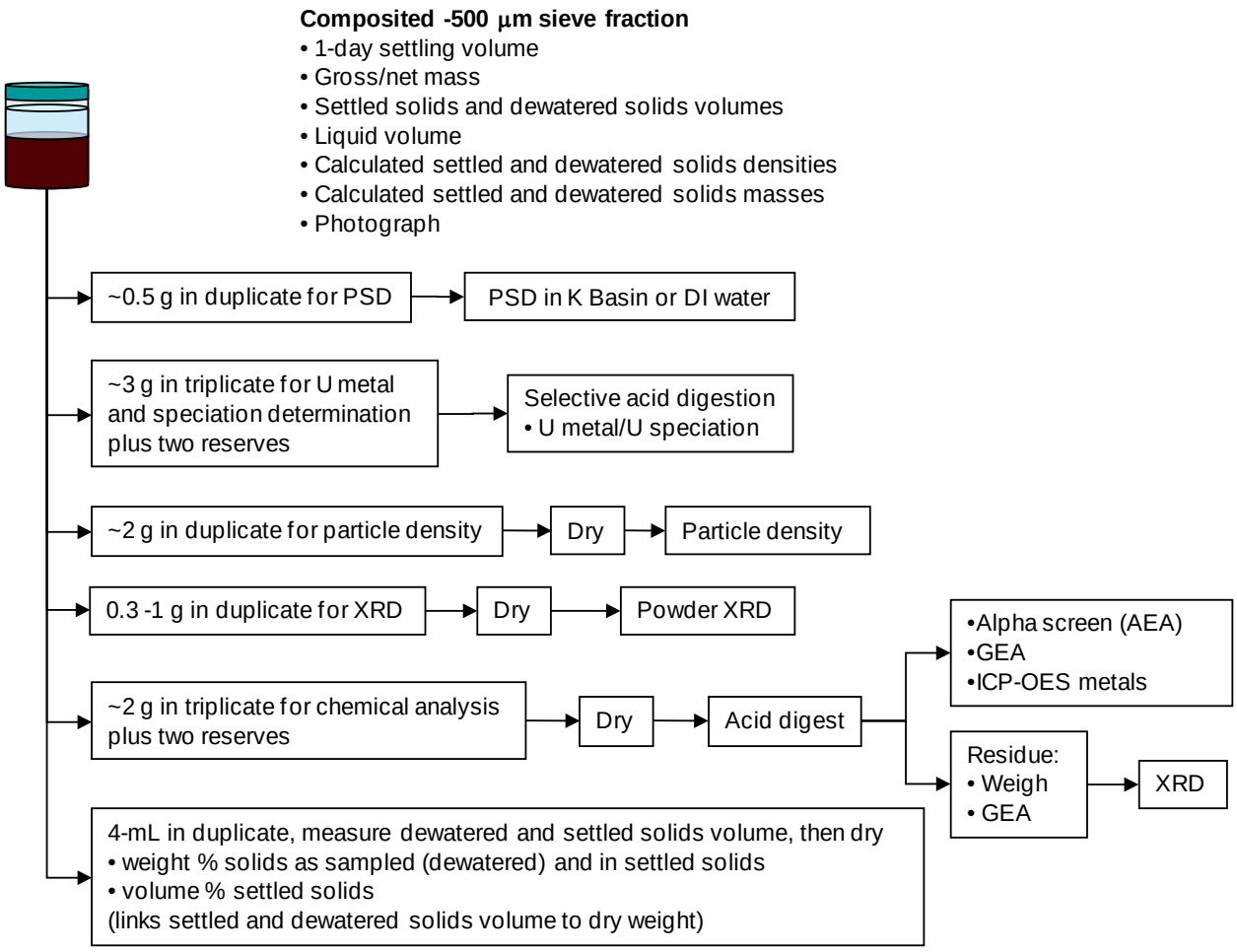


Figure 2.13. Processing Schematic of the KW210-CC-N Container Composite, −500-μm Fraction

A crosswalk among container composite IDs, subsample IDs (from TIs), as-sampled mass, sample split date, ASO sample IDs (RPL/ASO ID), ASR IDs, and analyses requested is provided in Table 2.9. More than the minimum required characterization samples were collected as a contingency in case of disagreement between duplicates, and/or when analytical problems were observed, and reanalysis was required.

The UDS collected following acid digestion were processed to support fusion analysis and XRD evaluation. These daughter samples are cross-referenced to the original material as shown in Table 2.10.

Table 2.9. Cross-Reference of SCS-CON-210 Container Composite Samples, TI Subsample ID, As-Sampled Mass, Split Date, RPL/ASO Subsample ID, and General Analysis Scope

Sieve Fraction	Subsample ID (TI)	As-Sampled Mass (g)[a]	Sample Split Date	RPL/ASO ID	Analysis
Composite	KW210-CC-AA	7.854	3/21/11	--	Primary physical properties
	KW210-CC-AB	9.391		--	Primary physical properties, duplicate
	KW210-CC-NA	7.572		--	Primary physical properties
	KW210-CC-NB	9.625		--	Primary physical properties, duplicate
+2,000 µm	TI008-LA[b]	0.7263	3/29/11		GEA and particle density
−2,000 µm / +500 µm	TI008-MA	1.5122	3/29/11	11-0792-129-S	Analytical
	TI008-MB	1.9603		11-0792-129-D	Analytical duplicate
	TI008-MC	1.3960		--	Analytical reserve
	TI008-MD	1.1887		--	Analytical reserve
	TI008-ME	2.5202		11-0792-107-S	U speciation
	TI008-MF	2.6176		11-0792-107-D	U speciation duplicate
	TI008-MG	2.4524		--	U metal matrix spike
	TI008-MH	NM[c]		--	XRD
	TI008-MJ	NM[c]		--	XRD duplicate
	TI008-MK	0.5231		--	Particle density
	TI008-ML	0.5074		--	Particle density duplicate
−500 µm	TI008-SA	1.9371	4/1/11	11-0793-129-S	Analytical
	TI008-SB	2.2705		11-0793-129-D	Analytical duplicate
	TI008-SC	1.9678		11-0793-129-MS	Analytical matrix spike
	TI008-SD	2.8118		11-0793-Archive	Analytical reserve
	TI008-SE	2.1878		11-0793-107-S	U speciation
	TI008-SF	3.5278		11-0793-107-D	U speciation duplicate
	TI008-SG	4.1801		11-0793-107-MS	U (metal) matrix spike
	TI008-SH	NM		--	XRD
	TI008-SJ	NM		--	XRD duplicate
	TI008-SK	0.6755		--	Particle density
	TI008-SL	0.3462		--	Particle density duplicate
	TI008-SM	4.9064		--	Primary physical properties
	TI008-SN	5.2429		--	Primary physical properties, duplicate
	TI008-SP	NM[c]		--	PSD
	TI008-SQ	NM[c]		--	PSD duplicate
	TI008-SR	3.5227		--	Analysis reserve
	TI008-SS	3.2927		--	Analysis reserve
	KW210-SCCP	21.47[d]		--	Post-sampled parent material

(a) The as-sampled mass basis for all large and medium sieve fraction samples (i.e. TI008-LA and TI008-M*, where * is A-L) is "air dried" and thus the sample may contain some residual water.
(b) TI008-LA was identified as TI008-L when submitted for particle density analysis.
(c) The PSD and XRD samples required a small aliquot of representative material; mass measurement was not required.
(d) Settled sludge mass basis. The as-sampled mass basis could not be determined.

Notes:

ASO samples were submitted under ASR 8824.

+2,000 µm = wet-sieved particles that were isolated on the 2,000-µm mesh sieve, i.e., >2,000 µm.

−2,000 µm/+500 µm = wet-sieved particles that passed through the 2,000-µm mesh sieve but collected on top of the 500-µm mesh sieve.

−500 µm = wet-sieved particles that passed through the 500-µm mesh sieve, i.e., <500 µm.

NM: Not Measured.

The net sample masses were calculated from data collected in TI 52578-TI008.

Gray shading indicates samples that were collected but not used.

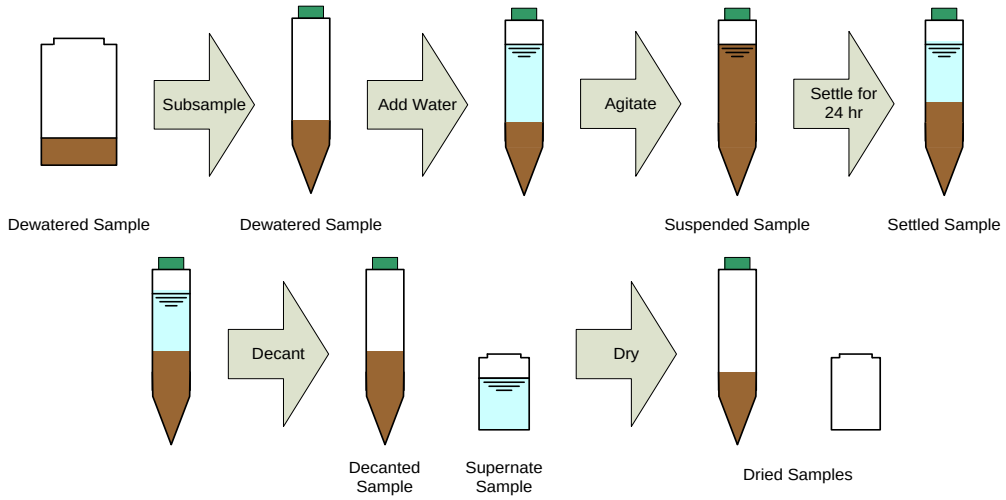
Table 2.10. Cross-reference of Container Composite Sample Description, TI Subsample IDs, and Original RPL/ASO IDs to UDS Sample ID Assignments

Container Sample Description	Subsample ID	Original RPL/ASO ID ASR 8824	UDS Daughter Sample Designation		
			ASR 8878 (RPL/ASO ID)	XRD	ASR 8919 (RPL/ASO ID)
−2,000 μm / +500 μm	TI008-MA	11-0792-129-S	TI040-0792S-1c (11-1289)		
	TI008-MB	11-0792-129-D	TI040-0792D-1c (11-1290)	TI040-792D-2x	
	TI008-MK & TI008-ML Combined				KW210 MK & ML Resin Beads (11-1555)
					KW210 MK & ML Residual Solids (11-1556)
−500 μm	TI008-SA	11-0793-129-S	TI040-0793S-1a (11-1287)		
	TI008-SB	11-0793-129-D	TI040-0793D-1b (11-1288)	TI040-793D-2x	
			TI040-0793D-2b[(a)] (11-1288D)		

(a) Processed as batch duplicate.

2.3 Physical Properties: Wet Sludge Densities and Mass Conversion Factors

Samples collected for primary physical property measurements were processed in accordance with the specific TI. Figure 2.14 depicts the overall primary physical property sample handling process. After measuring the as-sampled mass and volume, K Basin water was added to each sample. The ratio of fluid to as-sampled solids varied somewhat from sample to sample, but for the primary physical property characterization samples, the volume of fluid added was approximately equal to the volume of as-sampled sludge. Solids were then re-suspended by shaking (with manipulator hand and the aid of a vortex mixer) and allowed to settle undisturbed for up to 24 hr at which time constant volume (undisturbed) had been attained. Settling curves were generated opportunistically from the primary physical property samples and samples collected for the container composites. The settled solids volume and the total (settled solids plus the headspace water) volume were recorded or later determined from the video documentation, and the gross mass was measured. Most of the fluid headspace was removed (without disturbing the settled solids) and placed into a separate tared vial. The wet solids and removed fluid were dried to constant mass at 105°C.



- *Top row shows collection of a dewatered sample and how it is returned to a settled sludge state.*
- *Bottom row shows processing of a sample from a settled sludge state to a dry state.*

Figure 2.14. Sample Processing for Physical Property Measurements

The physical properties of the sludge (i.e., density, wt% solids) were assessed from duplicate "primary" subsamples collected from each core sample, container composite sample, and the −500-µm sieve fraction. Additional physical property data were collected from other samples where possible for sample-to-sample assessment of the general representativeness of the primary physical properties and subsamples.

- The primary physical property subsamples were used to determine 1) the as-sampled sludge mass, volume, and density, 2) the settled sludge mass, volume, and density after dispersion in water and settling for 24 hr, and 3) the wt% solids (i.e., from measurement of the dry sludge mass). These samples were the primary linkage source for all physical properties (i.e., used to convert from as-sampled to settled to dry mass basis) associated with the reconstituted core samples, the container composite samples, and −500-µm sieve size fraction of the container composite. Primary physical property samples were collected from each of the test materials in heavy-walled, 40-mL glass centrifuge tubes (Kimble-Kontes, Vineland, NJ, part number 45200-40). The lower tapered portion tube height is 5 cm and the total tube height (to the bottom of the neck) is 12 cm; the inside diameter increases from ~0.5 cm to ~2.5 cm. Volume measures of the as-sampled sludge were sometimes confounded by sludge hold-up on the sample vessel sides.
- The reconstituted core samples and the container composite samples in the parent glass Qorpak jars were evaluated for as-sampled sludge mass, volume, and density and settled sludge mass, volume, and density. In these cases, the ratio of fluid to settled sludge prior to mixing was not as carefully controlled and depended in part on available space in the sample jar. Volume measures of the as-sampled sludge were sometimes confounded by sludge hold-up on the sample vessel sides.
- Subsamples collected from the reconstituted core samples and the −500-µm sieve size fraction of the container composite sample were evaluated for as-sampled sludge mass, volume, and density and settled sludge mass, volume, and density. In these cases, the sample containers varied in material of construction (glass and plastic) and inside diameter. As with the parent samples, the ratio of fluid volume to settled sludge volume was not carefully controlled and depended in part on available space in the sample container. Volume measures of the as-sampled sludge were sometimes confounded by sludge hold-up on the sample vessel sides.

- For the core samples, chemical analysis subsamples were dried by the ASO, and the dried sludge mass was reported such that the g dry solids/g sampled sludge could be determined. These physical property data were generated using the same conditions and processes as the primary physical property samples.
- The density for all contact water was considered equivalent to DI water, and assumed to have a density of 1.000 ±0.005 g/cm^3.[(a)]

The data collected from samples of various sizes, showed that the physical properties were generally consistent within the range of sample sizes used in this characterization effort (i.e., several grams to several hundred grams). In the KW Basin, the sludge depth in the engineered containers varied from ~10 to 183 cm; in the laboratory, the sludge depth in sample jars ranged from <2.5 to ~10 cm. The settling rate and extent of sludge compaction or density may be influenced by sludge morphology, composition, concentration (sludge depth), and container dimension.

The collected data supported calculations of a variety of physical property attributes as defined in the equations below.

as-sampled sludge mass (M)
$$M = M_G - m_{CT} \quad (2.1)$$

dry sludge mass per as-sampled sludge mass
$$\frac{g_{dry}}{g_{as\text{-}sampled}} = \frac{m_{DS}}{M} \quad (2.2)$$

settled sludge mass (m_s)
$$m_S = m_G - m_{CT} - \left[(v_T - v_S) \times D_L\right] \quad (2.3)$$

dry sludge mass per settled sludge mass
$$\frac{g_{dry}}{g_{settled}} = \frac{m_{DS}}{m_S} \quad (2.4)$$

as-sampled sludge mass per settled sludge mass
$$\frac{g_{as\text{-}sampled}}{g_{settled}} = \frac{M}{m_S} \quad (2.5)$$

settled sludge density
$$D_S = \frac{m_S}{v_S} \quad (2.6)$$

dry sludge mass per settled sludge volume
$$\frac{g_{dry}}{mL_{settled}} = \frac{m_{DS}}{v_S} \quad (2.7)$$

where M_G = gross mass of as-sampled sludge plus container tare (g)
m_{CT} = tare mass of container (g)
m_G = mass of container tare + wet settled sludge + liquid above sludge (g)
v_T = total slurry volume (liquid plus settled sludge) (mL)
v_S = volume of settled sludge (mL)
D_L = liquid density (g/mL)
m_{DS} = mass of dried sludge (g).

Note: Dissolved solids in liquid were found to be insignificant.

(a) The water density was assigned 0.005 g/cm^3 uncertainty which was propagated into the overall uncertainties as appropriate.

Before disposal, and following drying to constant mass, the primary physical property samples were subjected to volume measurement and morphology assessments. The volume was measured directly without attempting to further settle solids. Before drying, interstitial spaces around settled particles were filled with water. After drying, interstitial spaces around solids were filled with air. Some retraction of the solids away from the vessel walls was observed; this increased void space was ignored. Morphology was determined in two stages: 1) the centrifuge tube was inverted to assess solids flow, and 2) the agglomerated material was prodded to assess resistance (this was a subjective measure through the constraint of a manipulator).

Images were intermittently collected with an in-cell video camera during all phases of the sample handling, subsampling, and sieving. Still images were collected from the video DVD.[(a)] To the extent practical, a MacBeth ColorChecker® Rendition Chart (MacBeth Color Chart) was included in the field-of-view in an effort to better define the sludge sample coloration. Figure 2.15 shows a scanned image of the MacBeth Color Chart.

Figure 2.15. MacBeth ColorChecker® Rendition Chart

2.4 Sample Characterization

All chemical, radiochemical, U speciation, and total organic carbon (TOC)/ total inorganic carbon (TIC) characterization samples were submitted to the ASO under various ASRs (as previously identified). Detailed Special Instructions were provided to the ASO, defining project-specific DQOs and requirements (see Appendices B–D). Each Special Instruction was reviewed internally and then provided to the BTR for review and concurrence before finalizing. The multiple reviews confirmed that the complexity of the required analyses was accurately conveyed to the ASO. Similarly, to assure that the client DQOs were properly delineated, the TIMS analysis requests were developed with a series of instructions that were first reviewed internally and then by the BTR before being issued for analysis.

Table 2.11 and Table 2.12 provide crosswalks of the analysis procedures, related QC sample acceptance criteria, and required duplicate frequency. Full copy procedures are provided as part of the

(a) Video DVDs will not be maintained as project records. Relevant still images are documented in the TIs and this report. The inventory of DVDs will be provided to STP CHPRC as non-record, information copies.

Table 2.11. Digestion, Chemical, and Physical Property Analytical Procedures and QC Sample Criteria

Analysis Objective	Procedure Number	Procedure Title	Duplicate (Precision)	Blank Spike (Accuracy)	Matrix Spike (Accuracy)	Calibration Verification	Duplicate[a] Frequency
TI implementation	RPL-OP-001	*Routine Research Operations*	NA	NA	NA	NA	NA
Acid digestion for chemical analysis	PNL-ALO-129	*HNO_3-HCl Acid Extraction of Solids Using a Dry-Block Heater*	NA	NA	NA	NA	100% Cores 1/Container Batch
Fusion for UDS dissolution	PNL-ALO-115	*Solubilization of Metals from Solids Using a KOH-KNO_3 Fusion*	NA	NA	NA	NA	1 / Batch[b]
ICP-OES for acid digestates and U metal analysis	RPG-CMC-211	*Determination of Elemental Composition by Inductively Coupled Argon Plasma Optical Emission Spectrometry (ICP-OES)*	± 20%/ (± 20% U metal >0.1 wt%)	± 20% (± 25% U metal)	± 20% (NA U metal)	LLS ± 25% ICV and CCV ± 10%[c]	100% Cores 1/Container Batch
U/KPA for total U	RPG-CMC-4014	*Uranium by Kinetic Phosphorescence Analysis*	± 20%	± 20%	± 25%	± 7.5%	100% Cores
Acid Digestion for U Speciation	RPG-CMC-107	*Sample Preparation for Determination Uranium Metal Concentrations in Sludge*	NA	NA	NA	NA	100% Cores 1/Container Batch
U(IV) : U(VI) Ratio	RPG-CMC-255	*Sample Preparation and Analysis for Determining Uranium Oxide Oxidation States in K Basin Sludges*	NA	± 25%	NA	NA	1/Container Batch
TOC/TIC analysis	RPG-CMC-385	*Carbon Measured in Solids, Sludge, and Liquid Matrices*	± 20%	± 25%	± 25%	NA	100% Cores
XRD preparation and analysis	PNNL-RPG-268	*Solids Analysis by X-Ray Diffraction*	NA	NA	NA	NA	NA
Particle density analysis	APEL-PIP-4	*Gas Pycnometry Method for Apparent Specific Gravity Determinations of Consolidated Solids*	± 5%	NA	NA	± 1%	1/Container Batch
Particle size distribution analysis	RPL-COLLOID-01	*Particle Size Analysis Using Malvern MS2000*	NA	NA	NA	NA	1/Container Batch
Shear strength analysis	RPL-COLLOID-02	*Measurement of Physical and Rheological Properties of Solutions, Slurries, and Sludges*	NA	NA	NA	NA	NA

(a) A laboratory duplicate sample is an aliquot of one of the original samples processed in duplicate through entire the preparative processes. Subsamples from each core were processed in duplicate; hence, 100% of the core samples were analyzed as true duplicates. Only one duplicate per batch was required for the container composite analysis.

(b) The fusion preparation (applied to undissolved solids) was not specified in the QAPjP/SAP; therefore, consistent with the PNNL procedure, a duplicate frequency of at least one per batch was used.

(c) Calibration verification criteria for the ICP-OES analysis was established in procedure RPG-CMC-211.

ICP-OES: inductively-coupled plasma optical emission spectrometry; KPA: kinetic phosphorescence analysis; LLS: low level standard; ICV: initial calibration standard; CCV: continuing calibration standard; TOC: total organic carbon; TIC: total inorganic carbon; XRD: x-ray diffraction; NA: not applicable.

Table 2.12. Radioisotopic Analytical Procedures and QC Sample Criteria

Analysis Objective	Procedure Number	Procedure Title	Duplicate (Precision)	Blank Spike (Accuracy)	Matrix Spike (Accuracy)	Calib. Verif./ Counter Control[a]	Duplicate[b] Frequency
U – Isotopic analysis	RPG-CMC-4017	*Analysis of Environmental Water Samples for Actinides and Strontium-90*	± 20%	NA	NA	± 2%[d]	100% Cores
	PNNL-98523-264	*Mass Spectrometer Isotopic Analysis (TIMS)*					
Pu – Isotopic analysis	RPG-CMC-455	*Separation of Uranium and Plutonium for Isotopic Analysis by Mass Spectrometry*	± 20%	NA	NA	± 6%[d]	100% Cores
	PNNL-98523-264	*Mass Spectrometer Isotopic Analysis (TIMS)*					
^{238}Pu, $^{239+240}$Pu —AEA	RPG-CMC-4017	*Analysis of Environmental Water Samples for Actinides and Strontium-90*	± 20%	± 20%	± 25%[c]	NA	100% Cores
	RPG-CMC-496	*Coprecipitation Mounting of Actinides for Alpha Spectroscopy*					
	RPG-CMC-422	*Solutions Analysis: Alpha Spectrometry*					
^{241}Am—AEA	RPG-CMC-4017	*Analysis of Environmental Water Samples for Actinides and Strontium-90*	± 20%	± 20%	± 25%[c]	NA	100% Cores
	RPG-CMC-496	*Coprecipitation Mounting of Actinides for Alpha Spectroscopy*					
	RPG-CMC-422	*Solutions Analysis: Alpha Spectrometry*					
Gamma-emitters —GEA	RPG-CMC-450	*Gamma Energy Analysis (GEA) and Low-Energy Photon Spectrometry (LEPS)*	± 20%	NA	NA	SPC	100% Cores 1/Container Batch
^{237}Np—AEA	RPG-CMC-4017	*Analysis of Environmental Water Samples for Actinides and Strontium-90*	± 20%	± 20%	± 25%[c]	NA	100% Cores
	RPG-CMC-496	*Coprecipitation Mounting of Actinides for Alpha Spectroscopy*					
	RPG-CMC-422	*Solutions Analysis: Alpha Spectrometry*					
AEA screen	RPG-CMC-496	*Coprecipitation Mounting of Actinides for Alpha Spectroscopy*	± 25%	± 20%	± 25%[c]	NA	1/Container Batch
	RPG-CMC-422	*Solutions Analysis: Alpha Spectrometry*					
^{90}Sr analysis	RPG-CMC-476	*Strontium Separation Using Eichrom Strontium Resin*	± 20%	± 25%	± 25%[c]	SPC	50% Cores
	RPG-CMC-474	*Measurement of Alpha and Beta Activity by Liquid Scintillation Spectrometry*					
Total alpha and total beta screen analysis	RPG-CMC-4001	*Source Preparation for Gross Alpha and Gross Beta Analysis*	NA	NA	NA	NA	NA
	RPG-CMC-408	*Total Alpha and Beta Analysis*					

(a) Counting instruments were checked daily with counter control samples to confirm that performance was within statistical process control (SPC).

(b) A laboratory duplicate sample is an aliquot of one of the original samples processed in duplicate through the entire preparative processes. Each core was processed in duplicate; hence, 100% of the core samples were analyzed as true duplicates. Only one duplicate per batch was required for the container composite analysis.

(c) In accordance with the ASO QA Plan, post- preparative blank and matrix spikes were performed for Pu-AEA, Am-AEA, Np-AEA, AEA screen, and ^{90}Sr.

(d) The isotopic analysis calibration verification standard criteria apply to ^{235}U, ^{238}U, ^{239}Pu, and ^{240}Pu isotopes and are based on the historical TIMS performance established through control charts. Control charts were not prepared for the other U and Pu isotopes. The Analysis Request approved by the BTR passed down the accuracy requirement for isotope concentrations exceeding 0.1 atom%. See Occurrence Report 52578-05-09-11 in Appendix E for details.

TIMS: thermal ionization mass spectrometry; AEA: alpha energy analysis; GEA: gamma energy analysis; LEPS: low energy photon spectroscopy; NA: not applicable.

complete data package. All reconstituted core samples were analyzed in duplicate. Duplicate frequency for the container composite samples was specified as one per analytical batch. The matrix spike (MS) was prepared from a triplicate sample that was spiked with the analytes of interest (AOIs). The laboratory control samples (LCSs) or blank spike (BS) samples were spiked as appropriate for the analysis. The LCS/BS and MS samples for radionuclides and U kinetic phosphorescence analysis (KPA) analyses were prepared post-digestion at the analytical workstation. In the case of TIC/TOC analysis, each duplicate sample was analyzed in small-mass triplicate sub-aliquots; the MS was created from another replicate sub-aliquot.

Project staff conducted physical property characterization activities outside of the ASO (XRD, particle density, PSD, and rheology) directly on samples. The particle density and XRD samples were submitted under analysis requests that incorporated relevant elements of the QAPjP/SAP data quality requirements. The PSD and rheology characterizations were conducted per client-approved TIs implementing the indicated operational procedure.

All laboratory procedures used are provided as part of the complete data package on DVD-52578-PNNL-20650 Rev. 2, Section II, September 2013.

2.5 Chemical Analysis

Chemical analyses (ICP-OES and radionuclide analysis) were conducted on the duplicate core subsamples and the container composite sieve fraction samples following acid digestion or fusions. The following sections provide brief descriptions. Cited procedures and reports found in Appendices B through D contain additional details for sample processing, QC sample processing, and instrument calibration. Analytes not listed as AOIs in the QAPjP/SAP were measured opportunistically as the procedures and detection systems allowed (with little or no additional cost to the project) and were not necessarily subjected to the same evaluation rigor applied to AOIs. Therefore, the quality of these opportunistic data is indeterminate.

2.5.1 Acid Digestion for ICP-OES and Radionuclides

Process QC samples prepared for the acid digestion included sample duplicates (all core and sieve fraction samples), a BS, and MS sample (the BS and MS sample were spiked with ICP-OES AOIs), a BS (spiked only with U to support KPA), and one preparation blank (PB). The samples processed for chemical analysis were dried to constant mass at 105°C. The dried residue was then acid-digested per PNL-ALO-129, Rev. 0, *HNO_3-HCl Acid Extraction of Solids Using a Dry-Block Heater,* modified to double the reagent acid volumes and concentrations (8 M each HNO_3 and HCl). The acid volumes used were proportioned to the mass of dried sample, with 8 mL of each acid for samples 1 g or less, 12 mL of each acid for samples between 1 and 1.5 g, and 16 mL of each acid for samples between 1.5 and 2 g.

The post-digested UDS were separated from the aqueous phase and thoroughly rinsed with acidic solution. The aqueous phase was brought to a 100-mL volume in a volumetric flask, and aliquots were distributed for ICP-OES and radiochemical analyses. The solution distributed to the workstations is termed the digestate. The UDS was dried to constant mass at 105°C. The UDS was removed from the SAL and counted by GEA. Sample digestion descriptions and mass measurements are provided in Appendices B and C.

The UDS samples were split into smaller sizes using the splitting technique previously identified (Figure 2.12) to support fusion processing. One sample split from each sample was fractionated to remove resin beads from the non-resin bead fraction. This was done on a best effort basis according to TI 52578-TI040[(a)]. Material was placed on folded paper; rolling particles (resin beads) were collected into a vial and the non-rolling fraction was collected in a separate vial. Each fraction was weighed to estimate the mass associated with the resin and non-resin material. One of the duplicate samples of non-resin material was evaluated by XRD (Table 2.7 and Table 2.10). Core and container composite UDS subsamples were selected and submitted for fusion processing and ICP-OES and AEA screen (Table 2.7 and Table 2.10).

2.5.2 Fusion

Selected UDS subsamples from ASR 8798 (Table 2.7) and ASR 8824 (Table 2.10) were processed according to ASR 8878 Special Instructions and were dissolved with the aid of fusion. Fusion was conducted in accordance with procedure PNL-ALO-115, Rev. 1.1, *Solubilization of Metals from Solids Using a KOH-KNO_3 Fusion* using a KOH/KNO_3 flux in a nickel crucible. The sample size for fusion processing ranged from 0.1 to 0.2 g, therefore large process dilution factors apply. The LCS used in fusion preparations was National Institute of Standards and Technology (NIST) standard reference material (SRM) 2710 Montana Soil. The fused material was dissolved and brought to a 100-mL volume with DI water. The solution was then analyzed by ICP-OES for Al, Ca, Fe, S, Si, Zr, and U, for gross alpha, and by AEA screen for $^{238}Pu/^{241}Am$ and $^{239+240}Pu$. Process QC for the fusion preparations included a sample duplicate, an LCS, and one PB.

2.5.3 ICP-OES

Analysis was conducted using a PerkinElmer Model 5300DV ICP-OES equipped to handle radioactive samples in accordance with procedure RPG-CMC-211, Rev. 3, *Determination of Elemental Composition by Inductively Coupled Argon Plasma Optical Emission Spectrometry (ICP-OES)*. Instrument calibration was conducted per the manufacturer-recommended procedure using multi-analyte custom standard solutions traceable to the NIST. Detailed calibration descriptions are located in the ICP-OES reports provided in Appendices B, C, and D.

Typically, the acid digestates were diluted 10× at the workstation to reduce the acid concentration and solution density at the instrument. However, the digestates also were run undiluted in an effort to meet the detection limits required for several elements (i.e., As, Sb, Se, Tl). The direct analysis run introduces potential matrix issues related to increased solution density and acidity. Samples prepared by fusion required larger dilution (~100×) to reduce the flux salt concentration. All reported results were corrected for the sample-specific process and dilution factors.

(a) *Processing of the Undissolved Solids Samples from the K West Basin Core Samples Collected from SCS-CON-210 for Additional Analysis*, SK Fiskum, May 2011.

Analytes are reported in two tiers. AOIs were specifically designated in the QAPjP/SAP for the core samples and the container composite samples and have been fully evaluated relative to QC sample performance. The AOIs for the fusion preparations were designated in the ASR (8878) with BTR concurrence. Other analytes were opportunistically measured but have not been fully evaluated for QC performance.

The ICP-OES report lists analyte concentrations that are less than the method detection limit (MDL) as "--"; the MDLs for these analytes were *estimated* by multiplying the instrument detection limit (IDL) of the analyte by the process factor or "multiplier". Sample results shown in brackets "[ ]" were less than the estimated quantitation limit (EQL) but greater than the MDL. Both the IDL and EQL are based on acidified water and/or fusion flux matrices (as applicable). Routine precision and bias is typically ±15% or less for samples in dilute, acidified water at analyte concentrations >EQL up to the established linear range. Bracketed values have potential uncertainties >15%. The maximum number of significant figures for all ICP-OES measurements is two. The third significant figure provided for analytes detected greater than the EQL is provided for additional information.

2.5.4 Alpha Energy Analysis

Radioisotopes measured by alpha spectrometry require radiochemical separations to provide a pure chemically separated solution containing the radioisotopes of interest. This allows the preparation of a mount that is free of residue that would otherwise attenuate the alpha emission, resulting in peak broadening and absorption. Separation also allows for elemental purification such that other alpha-emitting radionuclides with equivalent energies are stripped from the test fraction. A typical AEA system has an energy resolution of ~0.03 MeV. Table 2.13 shows the significant alpha emitters expected in the test samples, their corresponding primary alpha-emission energies, the primary interfering radioisotopes, and a method to resolve interference. Some isotopes have essentially equivalent energies and must be chemically separated to provide accurate analysis (e.g., ^{238}Pu and ^{241}Am). Other isotopes require radiochemical separation followed by TIMS to differentiate (e.g., ^{239}Pu and ^{240}Pu).

Table 2.13. Summary of Alpha Emitters and Corresponding Energies

Isotope	Alpha Energy, MeV[(a)]	Potential Interfering Isotope	Resolution to Interference
^{238}Pu	5.46, 5.50	^{241}Am	radiochemical separation
^{239}Pu	5.14, 5.16	^{240}Pu	TIMS
^{240}Pu	5.12, 5.17	^{239}Pu	TIMS
^{242}Pu tracer	4.86, 4.90	None	NA
^{237}Np	4.77, 4.79	^{234}U	radiochemical separation
^{234}U	4.72, 4.78	^{237}Np	radiochemical separation
^{235}U	4.32, 4.36, 4.40, 4.56, 4.60	^{236}U	TIMS
^{236}U	4.44, 4.49	^{235}U	TIMS
^{238}U	4.15, 4.20	None	NA
^{241}Am	5.44, 5.49	^{238}Pu	radiochemical separation
^{243}Am tracer	5.23, 5.28	None	NA

(a) Browne and Firestone 1986.

All alpha spectra were collected on Ortec silicon charged-particle detectors mounted in model 576A Dual Alpha Spectrometers operated with Canberra GENIE2000 Alpha Acquisition and Analysis software. Detectors were energy- and efficiency-calibrated with NIST-traceable standards.

2.5.4.1 Neptunium (Alpha Energy Analysis)

An aliquot of each core sample and duplicate acid digestate was passed through a strong-base anion exchange resin bed in a hydrochloric acid matrix in accordance with procedure RPG-CMC-4017, Rev. 0, *Analysis of Environmental Water Samples for Actinides and Strontium-90*. The Np fraction collected from the anion exchange column was processed through a second anion exchange separation to more completely remove interference from U. The ^{234}U (4.72 and 4.78 MeV alpha energies) and ^{237}Np (4.77 and 4.79 MeV alpha energies) cannot be resolved by alpha spectrometry. The purified Np fraction was then co-precipitated and mounted for alpha counting in accordance with procedure RPG-CMC-496, Rev. 0, *Coprecipitation Mounting of Actinides for Alpha Spectroscopy,* and counted by alpha spectrometry in accordance with procedure RPG-CMC-422, Rev. 2, *Solutions Analysis: Alpha Spectrometry*. Each alpha spectrum was assessed for ^{237}Np. An internal tracer was not used, and the results are reported uncorrected. Each alpha spectrum was also examined for the presence of ^{238}U to assess the degree of separation from the U and potential ^{234}U interference. A ^{237}Np BS and MS (spiked into the acid digestate at the workstation) were analyzed with each analytical batch. The recovery of the BS and MS provide information on batch performance. See the analytical report in Appendix B for additional detail.

2.5.4.2 Plutonium and Americium (Alpha Energy Analysis)

Core samples were analyzed for ^{238}Pu, $^{239+240}$Pu, and ^{241}Am concentrations. An initial gross alpha screen analysis was conducted on the acid digestate to optimize the test sample size and tracer addition (gross alpha results are included in the radiochemistry reports provided in Appendices B and C). The acid digestate aliquots were traced with ^{242}Pu and ^{243}Am; these were used as internal standards correcting for radiochemical yields and counter efficiencies. The BS and MS were also spiked (post-digestion) with ^{239}Pu and ^{241}Am. Each aliquot was then processed through a strong-base anion exchange resin bed in a hydrochloric acid matrix in accordance with procedure RPG-CMC-4017. The Pu and Am fractions were collected separately. The purified Pu and Am fractions were co-precipitated and mounted for alpha counting in accordance with procedure RPG-CMC-496 and counted by alpha spectrometry in accordance with procedure RPG-CMC-422.

The Pu alpha spectra were evaluated for ^{238}Pu, $^{239+240}$Pu, and tracer ^{242}Pu. Note that the ^{239}Pu and ^{240}Pu have nearly identical alpha energies and cannot be resolved; these isotope activities are reported as a sum. Each spectrum was also examined for the presence of ^{243}Am tracer to assess the degree of separation from Am. The U isotopes alpha emission energies do not interfere with those of Pu isotopes.

The Am alpha spectra were evaluated for ^{241}Am and tracer ^{243}Am. Each spectrum was also examined for the presence of $^{239+240}$Pu to assess the degree of separation from Pu. Alpha emission energies from U isotopes do not interfere with those of Am isotopes.

2.5.4.3 Alpha Screen (Alpha Energy Analysis)

Aliquots from acid digestion of container composite samples and fusion processing of UDS from both core samples and container composite samples were measured for alpha emitters from their AEA spectra. An initial gross alpha screen analysis was conducted on the acid digestate to optimize the test sample size (gross alpha results are included in the radiochemistry reports provided in Appendices C and D). The acid digestate aliquots were processed directly by co-precipitation with NdF_3 and mounted for alpha counting in accordance with procedure RPG-CMC-496. The mounts were counted by alpha spectrometry in accordance with procedure RPG-CMC-422. The alpha spectra were evaluated for $^{238}Pu/^{241}Am$ (unresolved spectral peaks), $^{239+240}Pu$, and $^{243+244}Cm$ (opportunistically) for spectral peaks. Spectral peaks from U isotopes were not present. From an activity concentration basis, ^{234}U and ^{238}U were at least 400× less than that of $^{238}Pu/^{241}Am$ and $^{239+240}Pu$ in the original core samples. Batch QC for the alpha screen included a process sample duplicate, process blank, BS, and MS. The BS and MS samples were post-digestion spiked with ^{239}Pu at the workstation. See Appendices C and D for additional information.

2.5.5 Gamma Energy Analysis

Core and container composite sample acid digestate aliquots (2 mL and/or 10 mL in glass liquid scintillation counter [LSC] vials) were counted directly on high-purity germanium detectors operated with Canberra GENIE2000 Gamma Analysis Software (VMS and PC versions). Analysis was conducted according to RPG-CMC-450, Rev. 1, *Gamma Energy Analysis (GEA) and Low-Energy Photon Spectrometry (LEPS)*. Because this was a direct analysis (i.e., no chemical separations were applied), no LCS, BS, or MS samples were required or prepared. Energy and efficiency calibrations were checked through statistical process control of the counter calibration verification standards. Sample count times were established such that the required MDLs for all AOIs could be reached. Interference from Compton scattering of high ^{137}Cs concentrations may have affected achievable detection limits of other (opportunistic) gamma-emitting isotopes at energies <661 keV.

Solids (e.g., UDS) were similarly placed in glass LSC vials for direct GEA measurement. Unlike liquid samples, their geometries could not be carefully controlled to match the calibration standards. The solids samples were counted at ~10 cm from the detector face to minimize the differences between the sample geometry and the calibration geometry. (Calibrations are performed using aqueous samples and reproducible geometries.) The UDS samples consisted of mostly resin beads (based on observation) and silica (based on XRD analyses, however, the exact sample mineralogical composition and associated density were not known). The small sample size (~1 g) minimized the absorption effects; therefore, density corrections were not applied. See Appendices B, C, and D for additional information.

2.5.6 Strontium-90 Analysis

An initial gross beta screen analysis was conducted on each core sample acid digestate to optimize the test sample size (results are included in the radiochemistry report provided in Appendix B). Aliquots of the acid digestate were processed through Sr Resin (Eichrom Technologies, Lisle, IL) to purify the Sr fraction in accordance with procedure RPG-CMC-476, Rev. 0, *Strontium Separation Using Eichrom Strontium Resin,* (tracer- and carrier-free). The purified Sr fraction was then analyzed by LSC in accordance with procedure RPG-CMC-474, Rev. 1, *Measurement of Alpha and Beta Activity by Liquid Scintillation Spectrometry*. See the ASO report in Appendix B for additional information on instrument calibration and control. The gross beta screen results were compared with the sum of beta emitters (^{137}Cs

and $^{90}Sr/^{90}Y$) for indication of agreement. BS and MS samples were prepared at the analytical workstation by spiking with a ^{90}Sr standard into the acid digestate.

2.5.7 Total Uranium (Kinetic Phosphorescence Analysis)

Aliquots of the core sample acid digestates were diluted (~10,000×) and analyzed directly by KPA (Chem Chek Instruments, Richland, WA) in accordance with procedure RPG-CMC-4014, Rev. 1, *Uranium by Kinetic Phosphorescence Analysis*. A hot cell BS prepared with only added U was provided with the samples. The MS sample (U spiked into the acid digestate) was prepared at the analytical workstation (post-digestion spike). Calibration verification standards were also analyzed during the analytical run to assess calibration stability. See the analytical report for additional detail in instrument calibration and calibration verification (Appendix B).

2.5.8 Uranium Isotopic Analysis

Aliquots of each core sample acid digestate were processed in accordance with procedure RPG-CMC-4017 to purify the U component. Subsample aliquot sizes (0.1 mL of the acid digestate) were determined after analyzing the digestate total U concentrations by ICP-OES. The PB (1 mL acid digestate) was also prepared for TIMS analysis.

A portion of the isolated U fraction was then mounted on a Re filament and analyzed by TIMS on a CEC (Consolidated Electrodynamics Corp.) 21-703 single stage mass spectrometer in accordance with procedure PNNL-98523-264, Rev. 2, *Mass Spectrometer Isotopic Analysis (TIMS)*. The calibration of the instrument was assessed with U standard reference material NIST U-030. Details of the sample analysis and instrument calibrations are located in Appendix E.

2.5.9 Plutonium Isotopic Analysis

Aliquots of each of the core sample acid digestates were processed in accordance with procedure RPG-CMC-455, Rev. 0, *Separation of Uranium and Plutonium for Isotopic Analysis by Mass Spectrometry* to isolate the Pu fraction using strong base anion exchanger. Subsample aliquot size (0.1 mL of undiluted digestate) was determined after analyzing the digestate Pu concentrations by AEA. The PB (1 mL acid digestate) was also prepared for TIMS analysis. No tracer was added because Pu atom ratios were needed.

A portion of the isolated Pu fraction was then mounted on a Re filament and analyzed by TIMS in accordance with procedure PNNL-98523-264. The calibration of the instrument was assessed with Pu standard reference material NIST Pu-946. Details of the sample analysis and instrument calibrations are located in Appendix E.

The ^{238}U is a major component of the sample matrix. Despite care in the separation, a very small carry-over of U into the Pu fraction is expected to interfere with the analysis of ^{238}Pu (from the ^{238}U isobar). Therefore, the ^{238}Pu analysis by AEA is expected to provide more accurate results for this radioisotope. The correct contribution of ^{238}Pu with respect to ^{239}Pu was determined by the following steps:

- The AEA provides relative amounts of ^{238}Pu and combined ^{239,240}Pu in terms of activities (μCi) per gram of sludge. In the AEA, the ^{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 are 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 provides activities of the individual ^{239}Pu and ^{240}Pu isotopes per gram of sludge.
- The activities of the ^{238}Pu and the ^{239}Pu per gram of sludge then are known.
- The atom percent (At%) of ^{238}Pu, with respect to the At% of ^{239}Pu reported by TIMS is calculated using the following equation:

$$\text{At\% } ^{238}\text{Pu} = \frac{\mu\text{Ci}\,^{238}\text{Pu/g sludge}}{\mu\text{Ci}\,^{239}\text{Pu/g sludge}} \times \frac{\text{Ci}\ ^{239}\text{Pu/g}\ ^{239}\text{Pu}}{\text{Ci}\ ^{238}\text{Pu/g}\ ^{238}\text{Pu}} \times \frac{\text{g}\ ^{239}\text{Pu/mole}\ ^{239}\text{Pu}}{\text{g}\ ^{238}\text{Pu/mole}\ ^{238}\text{Pu}} \times \text{At\% } ^{239}\text{Pu} \qquad (2.8)$$

  in which the first term includes the specific activities of ^{238}Pu and ^{239}Pu in the sludge, 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.
- Because the At% ^{238}Pu, with respect to the At% ^{239}Pu (TIMS), as determined by this method, is less than the ^{238}Pu value reported by TIMS, the individual plutonium isotope percentages, by the above calculation for ^{238}Pu and by TIMS for the other Pu isotopes, are summed and renormalized. This calculation approach provided the correct At% of all of the plutonium isotopes.

2.5.10 Uranium Speciation—Metal and Oxidation State Ratio

Uranium speciation was determined on the core and container composite samples following a step-wise acid dissolution, spectrophotometry, and U analysis by ICP-OES. The U speciation included analysis for the U oxidation state (U[IV] and U[VI]) distribution for the uranium oxides and analysis of the U metal concentration. Most of the samples taken for U speciation were not dried; a small amount of K Basin water had been added to the as-sampled wet solids shortly after the sample was taken to protect them from drying during the interim storage period before analysis. However, the container composite middle-sieve fraction samples (i.e., −2,000 μm/+500 μm) had been air-dried in the SAL.

The samples were processed in accordance with RPG-CMC-107, Rev. 1, *Sample Preparation for Determining Uranium Metal Concentrations in Sludge*. The ASO reports (Appendices B and C) provide details of sample digestion processing and analysis. The post-digested phosphoric acid solution was provided for spectrophotometric analysis to determine the distribution to the U(IV) and U(VI) oxidation states in accordance with procedure RPG-CMC-255, Rev. 0, *Sample Preparation and Analysis for Determining Uranium Oxide Oxidation States in K Basin Sludges*. The detection limit for both U(IV) and U(VI) is 0.001M. The post-digested nitric acid solution was provided for U analysis by ICP-OES in accordance with procedure RPG-CMC-211 to determine U metal concentration. The process quantitation limit (PQL) is 0.03 wt% U metal in the sludge matrix; the detection limit is 0.011 wt% U metal, both on a settled sludge basis. Unlike other analyte concentrations reported by the ASO (reported on a dry mass basis), the U metal results were reported on an as-sampled mass basis.

Batch QC samples included sample duplicates, one process blank, and one LCS. Each LCS contained ~0.5 g U_3O_8, 0.6 g Hanford sand, and ~0.01 g U metal (weighed to the nearest 0.00001 g). The LCS served to determine the method performance for U oxide oxidation state distribution [U_3O_8 is 33.3% U(IV)] and for U metal recovery. A MS sample was also analyzed but was not required by the QAPjP/SAP; it was included to provide additional information on method performance. The MS sample was prepared by adding ~0.01 g U metal to a triplicate sample of sludge.

Previous U oxidation state measurements of K Basin sludge have shown that a known interference in the sludge matrix is present and that U(IV) and U(VI) oxidation state results should be used with caution. Ferrous iron (Fe[II]), present as magnetite, has been observed in K Basin sludge (Fiskum et al. 2011a, b) and has been shown to chemically reduce U(VI) to U(IV) in Na_2SO_4/H_3PO_4 solutions. While the amount of Fe(II) is not known, the concentration of Fe in sludge samples is high enough (at 3 to 10 wt% on a dry basis) to cause concern. The U(VI) fractions can be considered lower bounding values and the U(IV) fractions the upper bounding values.

2.5.11 Total Organic Carbon and Total Inorganic Carbon

Samples collected for TOC and TIC measurements were distributed directly to the workstation for analysis in accordance with RPG-CMC-385, Rev. 2, *Carbon Measured in Solids, Sludge, and Liquid Matrices*. This method was revised specifically for K Basin work to accommodate measurements of organic carbon that potentially include organic ion exchange resin (OIER) and Grafoil® via sequential temperature combustion at 600 and 1,000ºC, respectively (Fiskum et al. 2011a). The combustion method was required to convert OIER to CO_2. (Oxidation of OIER in solution using the Ag[I] catalyzed persulfate oxidation method at ~95ºC was not effective.) Analytical sample size is generally limited so that the product CO_2 does not exceed 5,000 µg (as C). Special care was further required of the sludge samples because of their high activity and requisite drying, which resulted in a dispersible powder.

The TIC/TOC subsamples collected from each core sample were dried to constant weight at ~105°C such that the sample mass basis could be directly related to the settled sludge mass basis using the conversion factors from physical property samples. Small subsamples from each dried sample were transferred to quartz reaction vessels and weighed. Each subsample was then transferred to the tube furnace set at 600ºC and allowed to react with the carrier oxygen sweep gas. The gas was processed through barium chromate catalyst to ensure complete oxidation of carbon to CO_2. The CO_2 was absorbed in a solution and its reaction product titrated from an electrolytically generated titrant; the titrant was measured in terms of coulombs. All CO_2 collected from the 600ºC oxidation was attributed to TOC. Once the combustion at 600ºC was completed, the same analytical subsample was then subjected to the same oxidation process except at 1,000ºC, a process that was shown to oxidize carbonate compounds and Grafoil® to CO_2. All CO_2 collected from the 1,000ºC oxidation temperature was attributed to TIC.

The Coulometrics coulometer is calibrated at the factory; instrument calibration is not performed as part of this procedure. However, a minimum of two initial calibration verification samples (ICVs) α-D glucose, and two initial blanks are analyzed before sample analysis, after analysis of every 15 samples, and at the end of the day. The results for all samples are corrected for the average contribution from the blanks and average recovery of the ICVs.

Method performance is evaluated from the LCS and MS sample each spiked with α-D-glucose and calcium carbonate. For the ASR 8798 analyses sequential temperature oxidation method performance

was further evaluated with a mixture of Purolite® NRW37 OIER and Grafoil® that was processed both before and after the sludge samples were processed. Triplicate measurements were made on small subsamples from each duplicate sludge sample providing a total of six measurements per core sample.

2.6 X-Ray Diffraction

Samples were prepared for XRD analysis in accordance with procedure PNNL-RPG-268, Rev. 4, *Solids Analysis by X-Ray Diffraction*. Samples TI008-SH and TI008-SJ were provided as slurries and required an initial drying step. Each vial containing slurry material was covered with filter paper such that water (vapor) could pass through, yet contain particulate. To enhance the drying process, each vial was placed in a bed of Drierite and evaporation continued at room temperature until complete (based on visual examination). In general, the process took several days. All other samples were provided as dry material and were used as received.

All the samples were mixed with amyl acetate to produce slurries, transferred into a boron carbide mortar, and ground to a fine powder. After grinding, a small fraction of each sample was pipetted onto individual Krylon-covered glass slides (one sample per slide) and allowed to dry. The dried samples were then covered with Kapton®. Selected sample preparations were mixed with the rutile (TiO_2) internal standard from SRM 674b.

Each sample was analyzed using a Rigaku Ultima IV diffractometer XRD unit equipped with a high speed position sensitive detector system D/teX and a copper X-ray tube (Cu Kα radiation, $\lambda = 1.5418$ Å). Data were collected in a step scan mode between 5 and 65° 2-theta with a step size of 0.02°, at a speed of 2°/min. The X-ray tube operating conditions were 40 kV and 44 mA.

Crystalline phase identification was conducted using JADE software, Version 9.0 (Materials Data Inc., Livermore, CA), search match routines with comparison to the International Centre for Diffraction Data (ICDD) database Powder Diffraction File (PDF)-2 (2009). The ICDD database included the Inorganic Crystal Structure Database maintained by Fachinformationszentrum, Karlsruhe, Germany. Phase identification incorporated chemistry restrictions based on the elements determined from chemical analysis. In this case, the search included the following elements: C, Na, K, Mg, Ca, B, Al, Sr, Si, O, H, Cd, Cr, Ba, Cu, Ni, Pb, Mn, Fe, Zn, Zr, Ti, and U. Selection of these elements addresses the evaluation of hydrate compounds, Al hydrates, U hydrides, U oxides, and Zr metal plus any other identifiable compounds.

2.7 Particle Density

Sample particle density was determined by gas pycnometry using an AccuPyc 1330 (Micromeritics, Norcross, GA) in accordance with procedure APEL-PIP-4, Rev 2, *Gas Pycnometry Method for Apparent Specific Gravity Determinations of Consolidated Solids*. This method measures the amount of displaced ultra-high purity (UHP) He gas in each sample as determined by pressure. The two pressures observed upon 1) filling the sample chamber and then 2) discharging it into a second empty chamber allowed computation of the sample solid phase volume using Boyle's Law. In this method, gas molecules rapidly fill the tiniest pores of the sample; thus, only the truly solid phase of the sample displaced the gas. Ultimately, the sample volume was assessed according to Equation 2.9 (Lowell 2004):

$$V_S = V_C + \left(\frac{V_R}{1 - \frac{P_1}{P_2}} \right) \quad (2.9)$$

where V_S = sample volume
V_C = volume of the empty sample chamber
V_R = reference volume
P_1 = first pressure measured (sample chamber only)
P_2 = second pressure measured (after expansion of the gas into the combined volumes of sample chamber and reference chamber).

Once the sample volume was determined, the density was calculated based on the sample mass introduced into the pycnometer. The pycnometer was calibrated per the manufacturer's instructions using a certified calibration standard and confirmed with a secondary certified standard at the beginning of each series of analyses.

Duplicate sample slurries were collected from the −500-μm sieve fraction of the container composite sample and required an initial drying step. The samples were dried at ambient conditions within the glovebox to provide proper containment of the highly radioactive materials. The +2,000-μm and the −2,000-μm/+500-μm sieve fraction samples were submitted for analysis already air-dried (in the SAL).

Analytical samples sizes were volume-limited to fit within the 1-cm^3 pycnometer sample cup. The subsampling was conducted such that the provided sample could be nearly quantitatively transferred to the analysis cup thus eliminating additional subsampling error. Each sample was placed in the cup, weighed, and analyzed five sequential times; the results were averaged and the density, with standard deviation of the average, was reported. Details of the sample analysis are provided in Appendix G.

2.8 Particle Size Distribution

PSD was determined on duplicate sample slurries collected from the −500-μm sieve fraction of the container composite sample in accordance with procedure RPL-COLLOID-01 Rev. 1, P*article Size Analysis Using Malvern MS2000*. Specific processing parameters were delineated in TI 52578-TI037, *Particle Size Distribution Measurements for K West Basin Consolidated Container Composite SCS-CON-210, Using Minus 500 Micron Particle Size Fraction Samples.*

A Mastersizer 2000 (Malvern Instruments Ltd., Worcestershire, UK) equipped with a Hydro μP dispersion unit was used to analyze the samples. The instrument software (Mastersizer 2000 Version 5.60, Malvern Instruments, Ltd., Copyright 2009) was used to calculate the PSD (i.e., fractional volume contribution versus particle diameter) from the laser diffraction patterns using Mie scattering theory (see instrument manual and Malvern product literature for additional information). Based on the equipment specifications, particles with diameters between 0.02 and 2,000 μm can be analyzed by the Malvern Mastersizer 2000; however, it is a challenge for the Hydro μP dispersion unit to suspend and detect greater than ~150 μm diameter components (and/or high density materials).

The QAPjP/SAP does not provide specific precision or accuracy requirements for PSD measurements, but defers to procedural controls. Therefore, per procedure, the Malvern analyzer was to be checked with size distribution standards once at the beginning of a series of analyses that may span several days not exceeding a calendar quarter (~90 days). The median value (D_{50}) of three standard measurements should deviate less than a target value of 10% from the certified value together with its standard deviation unless other criteria are provided by the manufacturer of the standard material. Before conducting particle-size measurements with sludge samples, the PSD of a particle size standard, Malvern Quality Audit Standard QAS3004, traceable to NIST was measured to verify instrument performance. The standard consisted of polydispersed silica particles with diameters falling primarily between 13 and 130 μm. The results from this measurement were compared to the standard's certificate of analysis provided by the supplier, and the acceptable performance of the size analyzer was confirmed before running sludge samples in accordance with procedure RPL-COLLOID-01.

An in-depth study (initial parameter evaluation) was conducted using K Basin simulants (Burns et al. 2011) and on actual K Basin sludge samples (Fiskum et al. 2011b). The parameters evaluated included: refractive index, particle adsorption index, shape factor (spherical/non-spherical), recirculation time, recirculator rpm (mixing/resuspension), and sonication (power and duration). From these studies, a standard set of operational and analysis parameters were developed and recommended for use when measuring other K Basin sludge samples to provide a consistent PSD measurement method for all samples. For the current study, the same instrument configuration and operational/analysis parameters were used for the −500-μm sieve fraction samples collected from the SCS-CON-210 container composites. Appendix I provides detailed information on the selected run parameters.

Sample slurries received from the SAL were subsampled using a transfer pipet. The slurry was mixed and solids suspended by rapid transfer in and out of the transfer pipet. Then, a sample was collected and transferred dropwise into the Hydro μP dispersion unit until obscuration was satisfactory. The sampled material was analyzed for PSD in accordance with Table 2.14 and then removed from the system.

Table 2.14. Condition Matrix for PSD Determinations

Recirculation Rate, RPM[(a)]	Recirculation Time, min[(a)]	Sonication[(a,b)] Power, Time
2,000	1	Pre-sonication
2,000	3	100 % / 120 sec
2,000	5	Post-sonication

(a) The two initial measurement conditions for recirculation rpm, recirculation time and sonication power and time are based on results of the parameter evaluation in TI 52578-TI017 (Fiskum et al. 2011b). A third measurement condition (at 5 min recirculation) was added to further evaluate the impacts of sonication on the measured PSD.

(b) The Hydro μP dispersion cell employed for particle size analysis is equipped with variable power sonicator with an ultrasound power of up to 20W (at an instrument sonicator setting of 100%).

Other Measures/Conditions

Number of PSD measurements/conditions: each measurement consists of three separate observations (with each observation consisting of a10 s red light measurement and a 10 s blue light measurement).

2.9 Rheology/Shear Strength Measurement

Rheology testing (shear strength) was conducted on the whole PSD (i.e., unseived) of the container composite (KW210-CC-A) in accordance with procedure RPL-COLLOID-02 Rev. 1, "*Measurement of Physical and Rheological Properties of Solutions Slurries and Sludges*". Specific processing parameters were delineated in TI 52578-TI039, *Rheological Measurements for K West Basin Consolidated Container Composite SCS-CON-210.*

The QAPjP/SAP does not provide specific accuracy or precision requirements for these tests, but defers to procedural controls. Viscometer performance checks were conducted before and after a series of measurements (series time window of up to 30 days) with NIST-traceable viscosity standards. For testing with SCS-CON-210 materials, a 96.0 cP viscosity standard purchased from Brookfield Engineering Laboratories was employed. The acceptance criterion of ±10% applies for this standard and translates to an acceptable viscosity range of 86.4 to 105.6 cP

Shear strength was measured on settled sludge samples. The measurements were performed in the SAL with a Haake RV20-M5 viscometer. The RV20-M5 viscometer is capable of evaluating fluid rheological properties using Couette flow devices or shear strength using shear vanes.

For shear strength measurements, two different shear vane tools were used: 1) a 6 × 6 mm (vane diameter by height) four-paddle vane that can measure strengths up to ~110,000 Pa and 2) a 16 × 16 mm (vane diameter by height) four-paddle vane that can measure strengths up to ~5,700 Pa. Vane tool selection is dependent on expected sample shear strength and sample container geometries. The 6 × 6 mm vane tool was employed for the initial shear strength characterization of the as-received material to accommodate the potential for higher-than-expected shear strengths. The 16 ×16 mm vane was used on all other shear strength characterizations.

RPL-COLLOID-02 outlines geometrical spacing requirements for vane tool and solids containers when testing for shear strength. For all of the test cases reported herein, the combination of container and sludge volume yielded a settled solids configuration that met these test geometry requirements.

2.10 Viscosity Measurement

Apparent viscosity was not determined on the SCS-CON-210 container composite. The unseived material contained a significant amount of large particles (>500 μm) which would not meet the minimum requirements for the geometry of the flow curve measurement apparatus in SAL. A minimum geometry gap of 3 times the largest particle diameter is required for flow curve measurements to ensure that the material does not get caught inside the geometry; a factor of 10 is desirable. The maximum gap available to us in SAL is the MV2 geometry, this geometry has a gap of 2.6 mm which does not permit the analysis of particles with one linear dimension greater than ~850 μm. The −500-μm sieve fraction material did not contain enough sample volume to support the flow curve measurement. This scope deletion was directed/concurred by the BTR.

3.0 Physical Property Characterization Samples: Sludge Densities and Conversion Factors to Dry and Wet Mass Bases

This section provides a summary of the results generated from the physical property characterization subsamples obtained from the core and container composite samples from Engineered Container SCS-CON-210. These include wet sludge densities (settled and as-sampled mass bases), weight percent (wt%) solids, and mass conversion factors as follows:

- dry mass: sampled sludge mass
- as-sampled sludge mass: settled sludge mass
- dry mass: settled sludge mass.

3.1 Processing Description

Two wet-density bases are given for each sample: settled density and as-sampled density. As-sampled density refers to the thickened state of sludge at the point at which subsamples were collected from the parent sample. Before subsampling was conducted, the sludge was batch-settled, excess water was removed, and the sludge was mixed and allowed to re-settle. This process of dewatering, mixing, and settling was continued until a thickened sludge state was obtained (i.e., a state at which the sludge possessed sufficient shear strength to suspend/uniformly disperse any dense uranium metal particles) (Figure 3.1). Attainment of the thickened state was a subjective measure based on visual observation of the rheology (i.e., sludge flow from the mixing spoon). Once sufficiently thickened, the sludge was mixed and then subsampled (i.e., as-sampled sludge). The mass and volume of the as-sampled sludge in the various subsample containers was measured. Processing details are provided in Section 2.3.

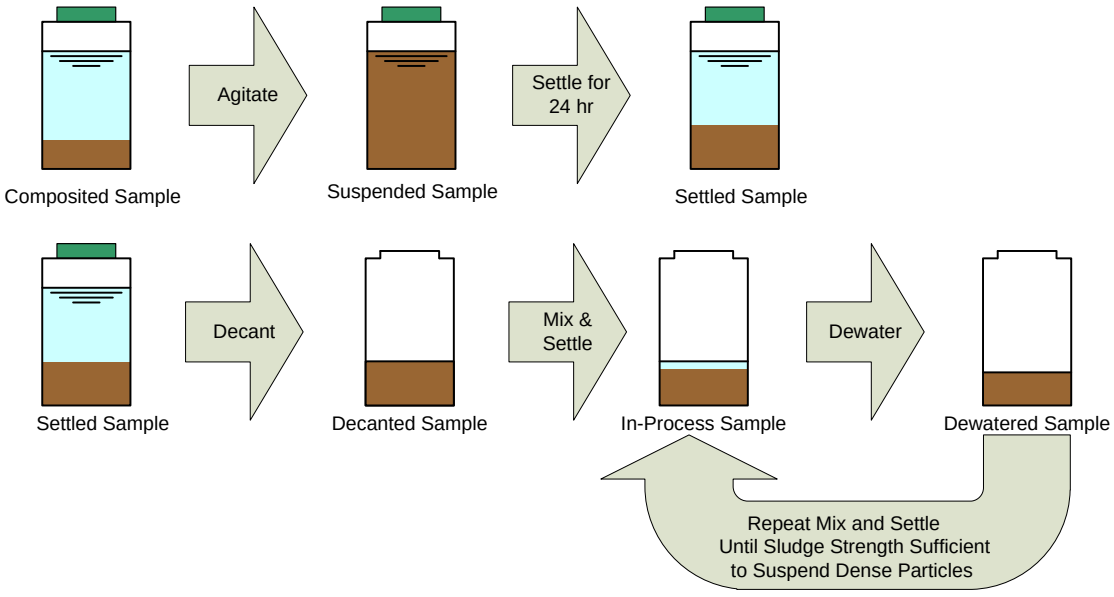
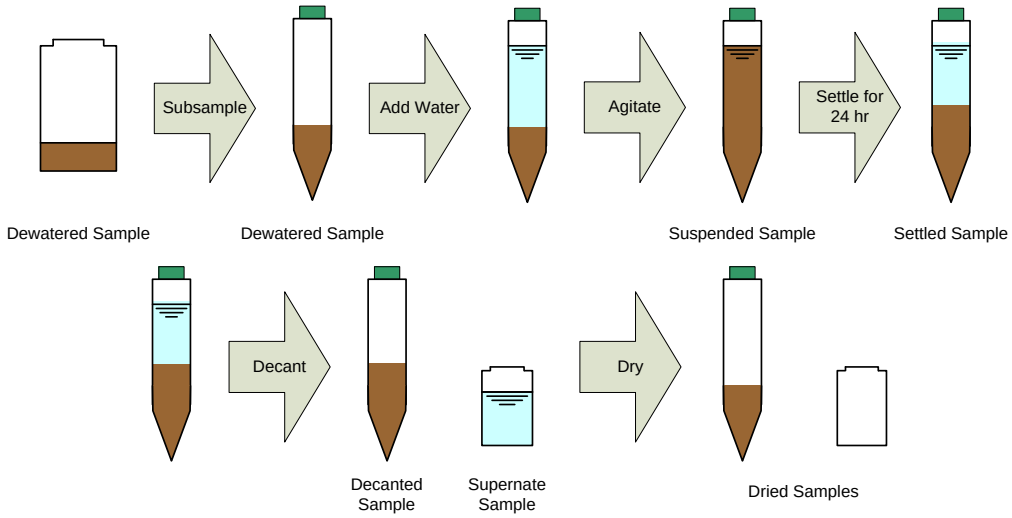


Figure 3.1. Sample Preparation for Subsampling

After the as-sampled sludge mass and volume were measured, select samples, including the physical property samples, were returned to a settled sludge state. K Basin water was added to the as-sampled sludge, and the water and sludge were mixed to create a suspension (typically 20 to 80 vol% settled solids and more narrowly ~50 vol% settled solids for the primary physical property subsamples), and then the suspension was allowed to settle undisturbed. The mass and volume of the settled sludge were measured to provide settled sludge densities in accordance with the QAPjP/SAP (see Section 2.3). Figure 3.2 depicts the sample manipulation processes. The settled sludge density values in Table 3.1 represent the undisturbed, settled sludge volume (~24-h).



- *Top row shows collection of a dewatered sample and how the dewatered sample is returned to a settled sludge state.*
- *Bottom row shows the processing of a sample from a settled sludge state to a dry state.*

Figure 3.2. Sample Processing for Physical Property Measurements

3.2 Physical Property Results

Table 3.1 provides the sludge densities and conversion factors for all samples from SCS-CON-210. The values are relevant for understanding analyte concentration data presented later in this report. Details on how the density and wt% solids measurements were performed are provided in Section 2.3. Opportunistic data obtained from the reconstituted core samples and other characterization subsamples are also presented in Table 3.1 for comparison purposes.

The average physical properties of the primary sample pairs that were specifically collected for physical property measurements are shown in **bold** text in Table 3.1 – along with related uncertainties. These average values were used to convert analyte concentration data from a dry solids mass basis to a wet settled sludge mass basis (chemical characterization results) or from an as-sampled sludge mass basis to a settled sludge mass basis (U metal results) in subsequent sections of this report.

The opportunistic data gathered from the core reconstitution and container composite vessels (large sample masses) and other analytical subsamples (small sample masses) are shown in *italics* as supplemental information. These data provide an estimate of the overall representativeness and reproducibility of the average values and demonstrate that the density values were generally consistent at a range of sample sizes.

Table 3.1. Sludge Densities and Mass Conversion Factors for Samples from SCS-CON-210

Core and Sample Identification	As-Sampled Sludge Mass,[a] g	Settled Density[a]		As-Sampled Density[a,b]		Mass Ratios[a]					
		g/cm^3	Relative % Uncertainty	g/cm^3	Relative % Uncertainty	g dry / g sampled	Relative % Uncertainty	g sampled / g settled	Relative % Uncertainty	g dry / g settled	Relative % Uncertainty
Core A1[c]											
TI001-A1-K	14.74	1.33	10.1%	1.40	9.5%	0.5671	0.5%	0.7649	7.4%	0.4338	7.4%
TI001-A1-L	18.50	1.31	8.4%	1.42	7.7%	0.5724	0.4%	0.8040	6.2%	0.4602	6.2%
KW210-A1 Average (K & L)	----	**1.32**	**6.6%**	**1.41**	**6.1%**	**0.5697**	**0.3%**	**0.7845**	**4.8%**	**0.4470**	**4.8%**
RPD	----	**1.06%**	----	**1.37%**	----	**0.93%**	----	**4.98%**	----	**5.91%**	----
KW210-A1-1	*245.83*	*1.38*	*8.2%*	*1.46*	*7.1%*	----	----	*0.8433*	*5.9%*	----	----
KW210-A1-A	*62.61*	*1.49*	*10.0%*	*1.57*	*8.8%*	----	----	*0.8686*	*6.9%*	----	----
TI001-A1-B	*2.05*	----	----	----	----	*0.5476*	*3.9%*	----	----	----	----
TI001-A1-C	*2.28*	----	----	----	----	*0.5605*	*3.5%*	----	----	----	----
TI001-A1-M	*20.07*	*1.40*	*5.7%*	*1.43*	*7.2%*	----	----	*0.7977*	*1.2%*	----	----
TI001-A1-N	*44.62*	*1.46*	*3.9%*	*1.49*	*3.3%*	----	----	*0.8491*	*2.7%*	----	----
KW210-A1-Archive-A	*16.76*	*1.23*	*40.1%*	NR[d]	----	----	----	*1.020*	*30.5%*	----	----
Core A4[c]											
TI002-A4-K	14.67	1.34	12.1%	1.47	5.0%	0.6272	3.5%	0.9106	8.8%	0.5711	8.8%
TI002-A4-L	16.76	1.44	10.8%	1.52	9.1%	0.6277	3.1%	0.8958	7.6%	0.5623	7.6%
KW210-A4 Average (K & L)	----	**1.39**	**8.1%**	**1.50**	**5.3%**	**0.6274**	**2.3%**	**0.9032**	**5.8%**	**0.5667**	**5.8%**
RPD	----	**6.92%**	----	**3.75%**	----	**0.08%**	----	**1.65%**	----	**1.56%**	----
KW210-A4-1	*281.64*	*1.60*	*8.3%*	*1.61*	*6.9%*	----	----	*0.9137*	*5.5%*	----	----
KW210-A4-A	*74.51*	*1.50*	*8.5%*	*1.49*	*7.0%*	----	----	*0.8754*	*5.9%*	----	----
TI002-A4-B	*2.30*	----	----	----	----	*0.6189*	*3.5%*	----	----	----	----
TI002-A4-C	*2.61*	----	----	----	----	*0.6117*	*3.1%*	----	----	----	----
TI002-A4-M	*23.34*	*1.54*	*5.7%*	*1.56*	*6.7%*	----	----	*0.8436*	*1.1%*	----	----
TI002-A4-N	*49.38*	NR[f]		*1.62*	*3.3%*	----	----	NR[e]		----	----
KW210-A4-Archive-A	*37.22*	*1.54*	*9.8%*	*1.72*	*16.2%*	----	----	*0.9068*	*9.1%*	----	----
Core B1											
TI002-B1-K	14.85	1.12	9.5%	NR[f]	----	0.4204	0.5%	0.7812	7.5%	0.3284	7.5%
TI002-B1-L	12.51	1.07	11.1%	1.14	9.1%	0.4197	0.6%	0.7809	8.9%	0.3277	8.9%
KW210-B1 Average (K & L)	----	**1.09**	**7.3%**	NA	NA	**0.4201**	**0.4%**	**0.7810**	**5.8%**	**0.3281**	**5.8%**
RPD	----	**4.62%**	----	NA	----	**0.17%**	----	**0.04%**	----	**0.21%**	----

Table 3.1. (contd)

Core and Sample Identification	As-Sampled Sludge Mass,[a] g	Settled Density[a]		As-Sampled Density[a,b]		Mass Ratios[a]					
		g/cm^3	Relative % Uncertainty	g/cm^3	Relative % Uncertainty	g dry / g sampled	Relative % Uncertainty	g sampled / g settled	Relative % Uncertainty	g dry / g settled	Relative % Uncertainty
KW210-B1-1	612.10	1.17	3.4%	1.17	2.3%	----	----	0.9322	2.6%	----	----
KW210-B1-A	45.45	1.15	11.1%	1.19	9.2%	----	----	0.7897	8.7%	----	----
TI002-B1-B	1.84	----	----	----	----	0.4063	4.4%	----	----	----	----
TI002-B1-C	1.98	----	----	----	----	0.4135	4.0%	----	----	----	----
TI002-B1-D	2.15	----	----	----	----	0.4111	3.7%	----	----	----	----
TI002-B1-M	20.52	1.21	4.7%	1.17	5.7%	----	----	0.7731	1.2%	----	----
TI002-B1-N	37.28	1.23	4.4%	1.17	3.1%	----	----	0.8656	3.3%	----	----
KW210-B1-Archive-1	102.57	1.13	5.6%	1.14	3.9%	----	----	0.9048	4.4%	----	----
KW210-B1-Archive-2	89.69	1.12	6.2%	1.12	4.4%	----	----	0.8754	4.9%	----	----
KW210-B1-Archive-3	108.75	1.16	5.4%	1.12	3.6%	----	----	0.9066	4.2%	----	----
KW210-B1-Archive-4	104.13	1.17	5.4%	1.16	3.9%	----	----	0.8649	4.2%	----	----
Core B3[c]											
TI001-B3-K	12.58	1.14	11.0%	1.26	5.0%	0.4818	0.6%	0.7610	8.6%	0.3667	8.6%
TI001-B3-L	12.97	1.24	10.8%	1.18	9.1%	0.4810	0.6%	0.7450	8.2%	0.3583	8.2%
KW210-B3 Average (K & L)	----	**1.19**	**7.7%**	**1.22**	**5.1%**	**0.4814**	**0.4%**	**0.7530**	**5.9%**	**0.3625**	**5.9%**
RPD	----	**8.71%**	----	**6.46%**	----	**0.17%**	----	**2.13%**	----	**2.30%**	----
KW210-B3-1	*602.56*	*1.25*	*3.3%*	*1.24*	*2.5%*	----	----	*0.8717*	*2.5%*	----	----
KW210-B3-A	*54.24*	*1.33*	*10.7%*	*1.25*	*8.1%*	----	----	*0.8446*	*7.9%*	----	----
TI001-B3-B	*2.26*	----	----	----	----	*0.4707*	*3.5%*	----	----	----	----
TI001-B3-C	*2.24*	----	----	----	----	*0.4697*	*3.6%*	----	----	----	----
TI001-B3-M	*20.57*	*1.25*	*4.8%*	*1.21*	*5.9%*	----	----	*0.7745*	*1.1%*	----	----
TI001-B3-N	*38.27*	*1.22*	*4.3%*	*1.25*	*3.3%*	----	----	*0.8708*	*3.2%*	----	----
KW210-B3-Archive-1	*92.84*	*1.24*	*6.3%*	*1.30*	*4.9%*	----	----	*0.8809*	*4.7%*	----	----
KW210-B3-Archive-2	*94.11*	*1.21*	*6.0%*	*1.28*	*4.8%*	----	----	*0.8627*	*4.6%*	----	----
KW210-B3-Archive-3	*96.70*	*1.24*	*6.0%*	*1.29*	*4.7%*	----	----	*0.8800*	*4.6%*	----	----
Container Composite											
KW210-CC-NA	7.5722	1.22	12%	1.16	7.7%	0.4773	0.039%	0.7410	11%	0.354	11%
KW210-CC-NB	9.6252	1.29	10%	1.20	6.3%	0.5196	0.029%	0.7795	9.0%	0.405	9.0%
KW210-CC-N Average (NA and NB)	----	**1.25**	**8.1%**	**1.18**	**4.9%**	**0.498**	**0.024%**	**0.760**	**7.0%**	**0.379**	**7.0%**
RPD	----	**5.6%**	----	**3.2%**	----	**8.5%**		**5.1%**		**14%**	----

Table 3.1. (contd)

Core and Sample Identification	As-Sampled Sludge Mass,[a] g	Settled Density[a]		As-Sampled Density[a,b]		Mass Ratios[a]					
		g/cm^3	Relative % Uncertainty	g/cm^3	Relative % Uncertainty	g dry / g sampled	Relative % Uncertainty	g sampled / g settled	Relative % Uncertainty	g dry / g settled	Relative % Uncertainty
KW210-CC-AA	7.8543	1.22	12%	1.12	7.1%	0.5037	0.036%	0.7556	11%	0.381	11%
KW210-CC-AB	9.3908	1.31	11%	1.17	6.3%	0.5208	0.029%	0.7953	9.4%	0.414	9.4%
KW210-CC-A Average (AA and AB)	----	**1.27**	**8.2%**	**1.15**	**4.7%**	**0.512**	**0.023%**	**0.775**	**7.1%**	**0.397**	**7.1%**
RPD	----	**7.0%**	----	**4.5%**	----	**3.3%**		**5.1%**		**8.5%**	----
KW210-CCA	*250.554*	*1.29*	*5.3%*	*1.25*	*4.0%*	NA	NA	*0.8828*	*3.9%*	NA	NA
KW210-CCN	*178.768*	*1.23*	*4.9%*	*1.23*	*3.5%*	NA	NA	*0.9402*	*3.7%*	NA	NA
Fines, −500 µm											
TI008-SM	4.9064	1.27	15%	1.23	7.5%	0.4700	0.061%	0.7555	11%	0.355	11%
TI008-SN	5.2429	1.30	13%	1.31	7.5%	0.4777	0.056%	0.7211	9.7%	0.344	9.7%
KW210-SCC Average (SM and SN)	----	**1.29**	**9.8%**	**1.27**	**5.3%**	**0.474**	**0.041%**	**0.738**	**7.3%**	**0.350**	**7.3%**
RPD	----	**1.9%**	----	**6.6%**	----	**1.6%**		**4.7%**		**3.0%**	----
KW210-SCC	*52.283*	*1.08*	*14%*	*1.38*	*13%*	NA	NA	*0.8525*	*12%*	NA	NA
TI008-SA	*1.9371*	----	----	----	----	*0.4581*	*0.16%*	----	----	----	----
TI008-SB	*2.2705*	----	----	----	----	*0.4791*	*0.13%*	----	----	----	----
TI008-SC	*1.9678*	----	----	----	----	*0.4573*	*0.16%*	----	----	----	----

(a) Individual mass measurements are presented in appropriate significant figures. Individual mass ratios and averages are presented to three and four significant digits in this table. Volume measurements (and thus density) are accurate to two significant figures. The third significant figure of the density values are shown for information only. The additional digits are provided for information and to help the reader re-create computed values without introducing the rounding error.

(b) Volume measurements of the dewatered or as-sampled sludge were challenging due to sludge smearing and uneven surfaces. Consequently, as-sampled sludge density values should be used with caution.

(c) Additional archive subsamples were collected by sequentially rinsing residual solids coating the interior composite containers with K Basin water for cores A1, A4, and B3. The settled sludge volumes were typically <5 mL and relative uncertainties of the settled density measurements exceeded 90% for cores A1 and A4. The sequential rinses were collected in different bottles and may not be representative of the reconstituted core without initial recombination.

(d) KW210-A1-Archive-A as-sampled sludge density was not reported because of a severely sloped sludge level and resulting uncertainty of the measured value.

(e) TI002-A4-N settled sludge density and g sampled/g settled conversion factor are not reported because the re-suspension of the as-sampled sludge was not successful. The phase ratio of suspending solution to sludge was small and there appeared to be an unknown compound in the liquid phase that clung to the side walls and cap of the sample vial after agitation. This resulted in a settled sludge volume and total slurry volume that was not credible. See OR 52578-08-19-11 in TI 52578-TI002 for details.

(f) TI002-B1-K as-sampled sludge density was not reported because the sludge level was severely sloped. This resulted in an as-sampled density greater than three times the standard deviation of the B1 subsamples.

Notes:

Refer to Definitions (page xiii) for descriptions of as-sampled, settled, and dried sludge conditions.

Calculations of averages, relative percent differences (RPDs), and relative uncertainty were performed using untruncated values (Microsoft Excel calculation spreadsheet). Input data and Excel spreadsheet calculations used to generate data for this table are provided in DVD-52578-PNNL-20650 Rev. 2, Section III, September 2013.

The italicized values are provided as supplemental, opportunistic data.

NR: Not Reported. NA: Not Applicable.

The relative uncertainty of each measurement was calculated using variance propagation. The relative uncertainties of the net as-sampled sludge masses ranged from 0.01% to 4.4% and are not shown in Table 3.1.[a, b] The density uncertainties were primarily driven by the volume uncertainty component. Volume measurements were made by visually reading the decal millimeter scale affixed to the sample jars (e.g., ±2 mm = 12 mL in the 960-mL jar) or from factory-calibrated and marked centrifuge tubes. Volume measurements of as-sampled sludge were sometimes affected by sludge smears on the inside walls and uneven sludge levels (i.e., the thickened as-sampled sludge was difficult to level). Smearing was not an issue for settled sludge and the sludge settled in a level manner. In all cases, the volume could only be measured to two significant figures.

For all cores, the 24-hr settled sludge volume was considerably different from the as-sampled sludge volume. The sludge mass difference between the as-sampled and settled states can be significant as it is driven primarily by the difference in sludge volumes. The average mass ratios of the as-sampled to settled sludge ranged from 0.74 to 0.90 (Table 3.1). However, the as-sampled and settled sludge densities were not much different.

The relative uncertainties for average values of the primary characterization pairs given in Table 3.1 were propagated in accordance with standard practices and ranged from 0.02 to 9.8%.[c] Another measure of precision, RPD, was also determined for each primary physical property characterization pair and ranged from 0.04 to 14%.

The mass of dry solids per gram of settled sludge (g dry/g settled) is provided as a ratio. Multiplying this value by 100 will provide the wt% dry solids in settled sludge, as requested in the QAPjP/SAP.

All sludge-sample aliquots were weighed in the wet dewatered or as-sampled form. Data initially normalized to the as-sampled mass (e.g., U metal) convert to a settled solids mass basis by multiplying by the conversion factor “g sampled/g settled” (Equation 3.1).

$$\frac{U_{metal}}{g_{sampled}} \times \frac{g_{sampled}}{g_{settled}} = \frac{U_{metal}}{g_{settled}} \qquad (3.1)$$

(a) Relative % Uncertainty = (absolute uncertainty / measured value) x 100.

(b) The relative uncertainty of the as-sampled sludge mass measurements varied as a function of sample mass, balance type, and balance tolerance control limits. For example, using the HLRF balance, a 2.05-g analytical sample (collected in the 40-mL Oak Ridge centrifuge tube) has a 3.9% relative uncertainty. A 14.74-g physical property sample (collected in the 40-mL glass centrifuge tubes) and weighed on the HLRF balance has a 0.54% relative uncertainty. Individual sample mass uncertainties are provided in the physical properties spreadsheets contained in DVD-52578-PNNL-20650 Rev. 2, Section III, September 2013.

(c) Relative uncertainties for average values were calculated as follows:

Step 1—Calculate the uncertainty of each measurement in absolute units (standard deviation).

Step 2—Calculate the variance associated with the mean function (X + Y)/2). The variance of this function is the constant squared [$(1/2)^2$ = ¼] times the sum of Var(X) and Var(Y) where Var(X) is the answer in step 1 for the first measurement squared, and Var(Y) is the answer in step 1 for the second measurement squared.

Step 3—Add the Var (X) and Var (Y), divide by 4, and take the square root of the answer.

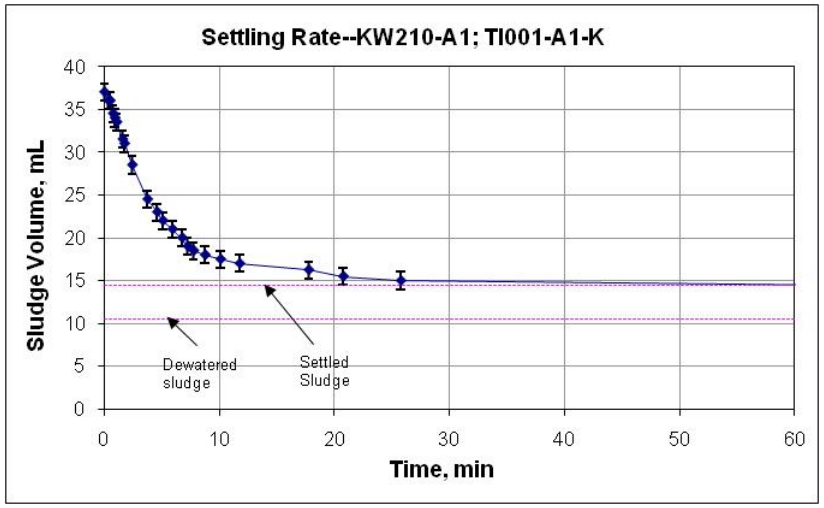
Step 4—Put the answer back into relative terms (RSD) by dividing the answer in step 3 by the overall average of the two measurements.

Data initially normalized to the dry sample mass (e.g., chemistry and radionuclide results) convert to the settled solids mass basis by multiplying by the conversion factor “g dry/g settled” (Equation 3.2).

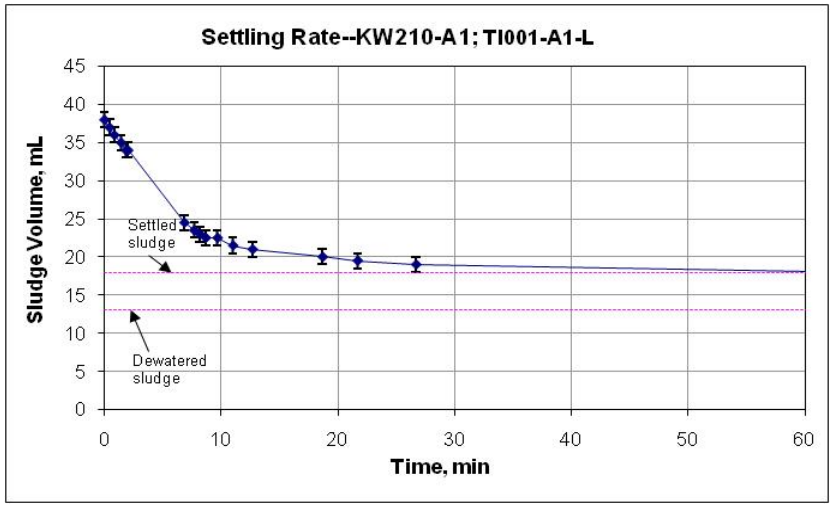
$$\frac{A_X}{g_{dry}} \times \frac{g_{dry}}{g_{settled}} = \frac{A_X}{g_{settled}} \quad (3.2)$$

All physical properties samples, reconstituted core samples, and container composite samples for SCS-CON-210 were re-agitated to acquire a settled sludge volume. Constant settled sludge volume was generally achieved within 1.5 hr of undisturbed settling after mixing/suspension. Plots of settled sludge volume as a function of time were opportunistically collected on the primary physical property sample pairs (Figure 3.3 through Figure 3.6) and many of the other collected samples (see DVD-52578-PNNL-20650 Rev. 2, Section III, September 2013). In all cases, the samples rapidly settled in the first ten minutes, then slowly approached the final measured settled volume within 1.5 hr. Because no changes in settled sludge volumes were seen after 12 hr, final settled sludge volumes were recorded prior to the 24-hr mark.

Settled sludge volumes were re-measured after transferring the samples from HLRF to SAL to assess compaction as a function of the sample handling. SCS-CON-210 material appeared to be heterogeneous in nature (i.e., with respect to particle morphology and size distributions). The previously characterized physical properties sludge subsamples collected from SCS-CON-220 containerized sludge core samples appeared to have similar heterogeneous characteristics and significantly compacted from handling activities (Fiskum et al. 2011b). Table 3.2 shows the change in settled sludge densities for the SCS-CON-210 primary physical properties samples and compares them to the as-sampled densities.

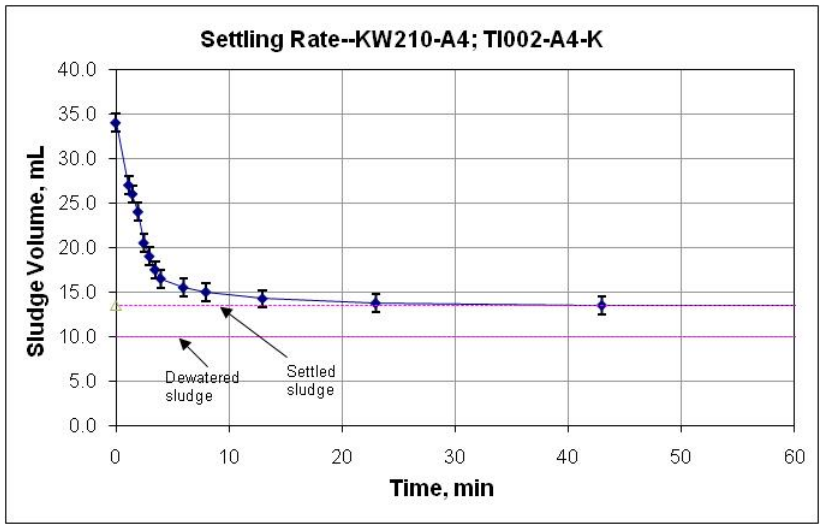


A. Settling Behavior of Subsample TI001-A1-K

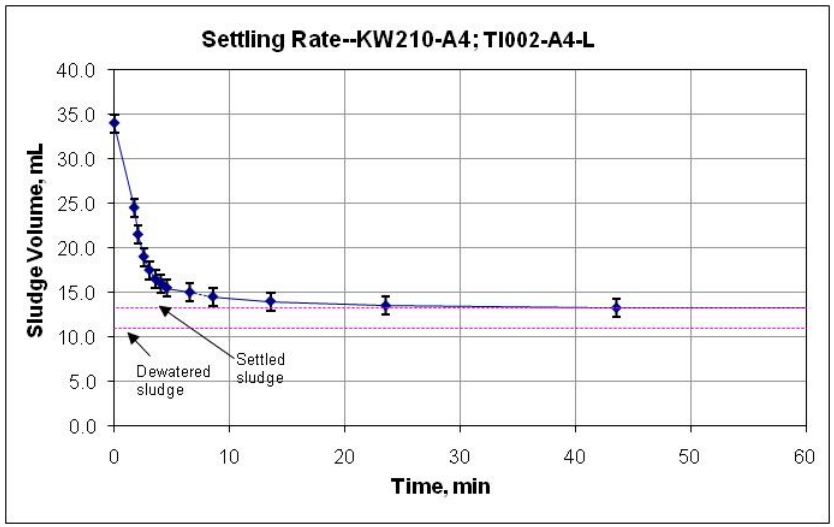


B. Settling Behavior of Subsample TI001-A1-L

Figure 3.3. Settling Behavior of Physical Property Samples Collected from the KW210-A1 Core

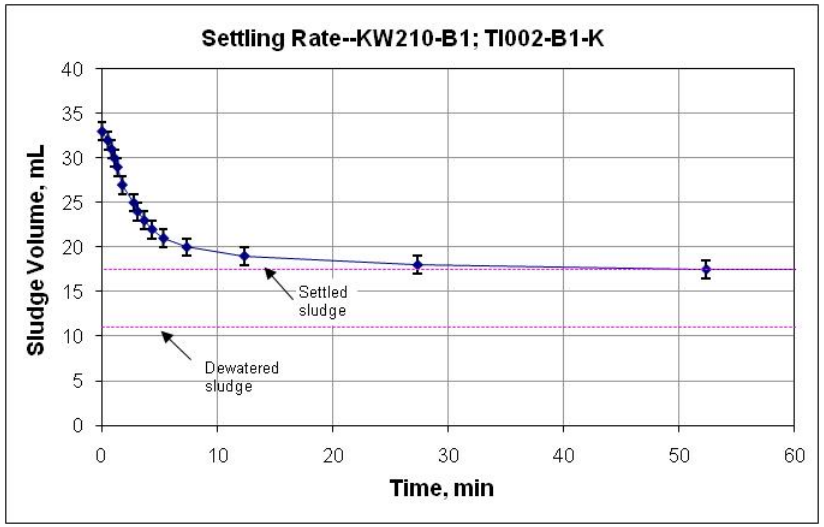


A. Settling Behavior of Subsample TI002-A4-K

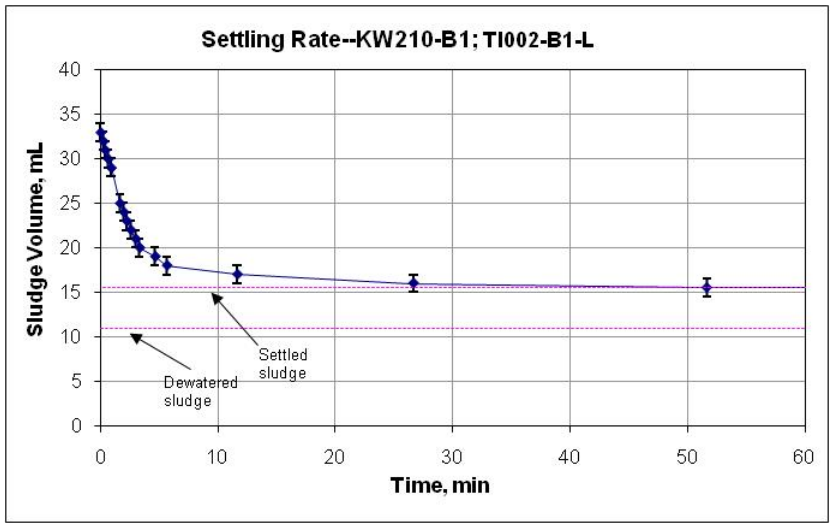


B. Settling Behavior of Subsample TI002-A4-L

Figure 3.4. Settling Behavior of Physical Property Samples Collected from the KW210-A4 Core

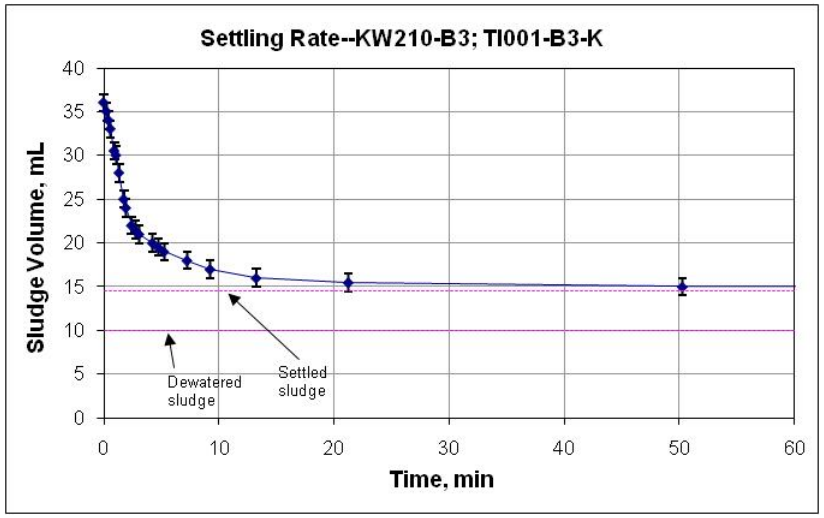


A. Settling Behavior of Subsample TI002-B1-K

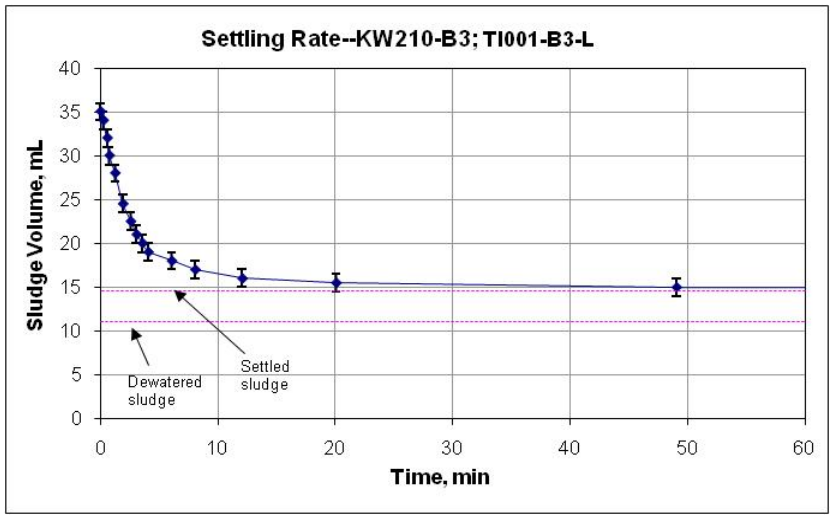


B. Settling Behavior of Subsample TI002-B1-L

Figure 3.5. Settling Behavior of Physical Property Samples Collected from the KW210-B1 Core



A. Settling Behavior of Subsample TI001-B3-K



B. Settling Behavior of Subsample TI001-B3-L

Figure 3.6. Settling Behavior of Physical Property Samples Collected from the KW210-B3 Core

Table 3.2. SCS-CON-210 Sludge Density Changes for Subsamples K and L with Movement

	Sludge Density							
	Core A1				Core A4			
	TI001-A1-K		TI001-A1-L		TI002-A4-K		TI002-A4-L	
Condition	g/cm^3	Uncertainty	g/cm^3	Uncertainty	g/cm^3	Uncertainty	g/cm^3	Uncertainty
24-h Settled	1.33	10.1%	1.31	8.4%	1.34	12.1%	1.44	10.8%
Moved to SAL[(a)]	1.37	11.5%	1.38	9.3%	1.43	12.3%	1.54	11.3%
As-sampled	1.40	9.5%	1.42	7.7%	1.47	5.0%	1.52	9.1%
	Core B1				Core B3			
	TI002-B1-K		TI002-B1-L		TI001-B3-K		TI001-B3-L	
	g/cm^3	Uncertainty	g/cm^3	Uncertainty	g/cm^3	Uncertainty	g/cm^3	Uncertainty
24-h Settled	1.12	9.5%	1.07	11.1%	1.14	11.0%	1.24	10.8%
Moved to SAL[(a)]	1.13	13.4%	1.11	11.6%	1.22	11.4%	1.22	16.7%
As-sampled	NR[(b)]	----	1.14	9.1%	1.26	5.0%	1.18	9.1%

(a) Containers were moved from the HLRF to the SAL

(b) TI002-B1-K as-sampled sludge density was not reported (NR) because the sludge level was severely sloped, confounding measurement of the sludge volume. This resulted in a calculated, as-sampled density greater than three times the standard deviation of the other B1 subsamples.

3.3 Volume Measurements and Morphology Assessments of Dried Physical Property Samples

Before discarding the 105°C-dried physical property characterization samples, they were opportunistically evaluated for apparent bulk volume and evidence of agglomeration. The simple examinations included prodding the agglomerate with a spatula to qualitatively assess strength. General observations included:

- Much of the dry material was free flowing and granular.
- Fines formed friable and relatively weak agglomerates. Fibers that were found mixed in the fines helped hold the agglomerates together. These fiber-reinforced agglomerates were broken apart with relatively little force using a 1-cm wide stainless steel spatula.
- No evidence of the formation of strong agglomerates was observed for the core subsamples.
- Strong agglomerates were observed on each of the −500-μm sieve fraction container composite samples.
- Generally, the dried sludge did not cling to the glass centrifuge tube walls.
- Sludge typically contracted ~18 to 51% from a settled to a dried volume basis.
- Significant quantities of OIER beads were observed in all samples; they visually appeared to be in a greater abundance in the “B” core samples.

Figures 3.7 through 3.10 show select images of SCS-CON-210 core primary physical properties samples post-drying, Figure 3.11 shows select images of SCS-CON-210 container composite and −500-μm sieved fraction samples post-drying. Table 3.3 summarizes observations collected from selected core and container composite samples. [Note: The inside diameter of the centrifuge tubes shown in Figures 3.7 through 3.11 increases from ~0.5 cm at the cone tip to ~2.5 cm at the top of the tapered cone region.]

a) TI001-A1-K initial look at dried solids levels, some material adhered to centrifuge tube wall.

b) TI001-A1-K prior to pouring out solids. Large agglomerate pieces on top of underlying granular particles.

c) TI001-A1-K vial after pouring out solids. Small removable heel and some material adhered to centrifuge tube wall at sludge/water interface.

d) TI001-A1-K agglomerate particle with 1-cm wide stainless spatula. Little effort required to break.

e) TI001-A1-K agglomerate broken into pieces with little force as indicated by material resting point in immediate sectioning area.

f) TI001-A1-K bulk materials image capturing general particle size, color, and morphology.

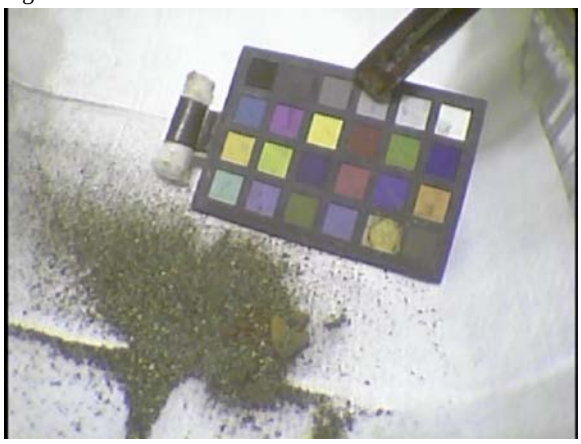
Figure 3.7. Select Post-Test Examination Images of TI001-A1-K (KW210-A1)

a) TI002-A4-K initial look at dried solids levels, some material adhered to centrifuge tube wall.

b) TI002-A4-K prior to pouring out solids. Large agglomerate pieces on top of underlying granular particles.

c) TI002-A4-K vial after pouring out solids. Small removable heel and some material adhered to centrifuge tube wall at sludge/water interface.

d) TI002-A4-K agglomerate particle with 1-cm wide stainless spatula. Little effort required to break.



e) TI002-A4-K bulk materials image capturing general particle size, color, and morphology.

Figure 3.8. Select Post-Test Examination Images of TI002-A4-K (KW210-A4)

a) TI002-B1-L initial look at dried solids levels, some material adhered to centrifuge tube wall.

b) TI002-B1-L prior to pouring out solids. Large agglomerate pieces on top of underlying granular particles.

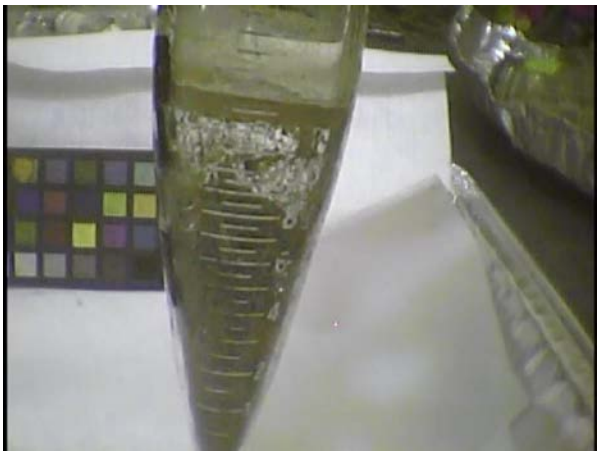
c) TI002-B1-L vial after pouring out solids. Small removable heel and some material adhered to centrifuge tube wall at sludge/water interface.

d) TI002-B1-L agglomerate particle with 1-cm wide stainless spatula. Little effort required to break.

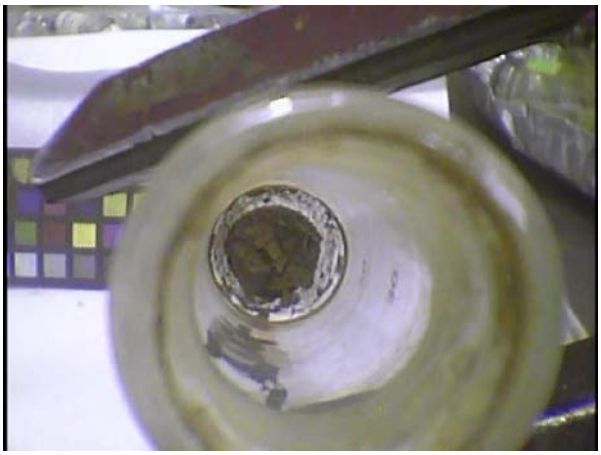
e) TI002-B1-L agglomerate broken into pieces with little force as indicated by material resting point in immediate sectioning area.

f) TI002-B1-L bulk materials image capturing general particle size, color, and morphology.

Figure 3.9. Select Post-Test Examination Images of TI002-B1-L (KW210-B1)



a) TI001-B3-K initial look at dried solids levels, some material adhered to centrifuge tube wall.



b) TI001-B3-K prior to pouring out solids. Large agglomerate pieces on top of underlying granular particles.

c) TI001-B3-K vial after pouring out solids. Small removable heel and some material adhered to centrifuge tube wall at sludge/water interface.

d) TI001-B3-K agglomerate particle with 1-cm wide stainless spatula. Little effort required to break.

e) TI001-B3-K agglomerate pieces held together by fiber. One piece held by tweezers, other piece suspended by fiber strand.

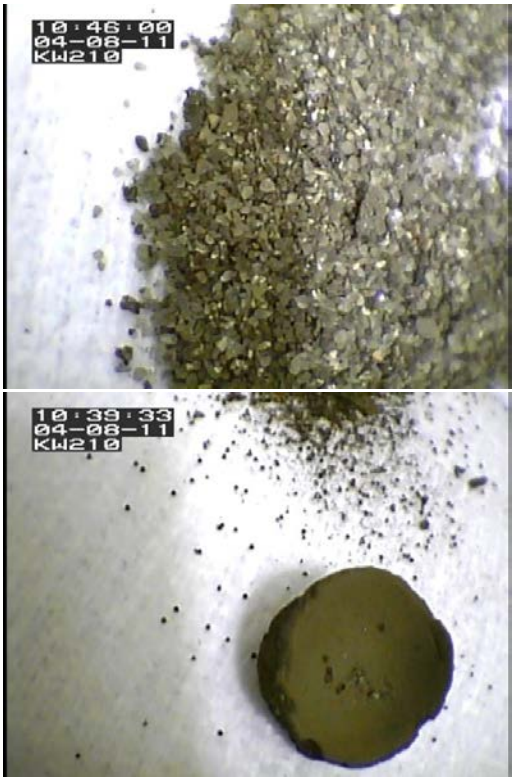
f) TI001-B3-K bulk materials image capturing general particle size, color, and morphology.

Figure 3.10. Select Dried Solids Examination Images of TI001-B3-K (KW210-B3)

a) TI008-NB, KW210 container composite (un-sieved).

b) TI008-NB poured from centrifuge tube.

c) TI008-SN (TI008-SM was identical), −500-µm sieve fraction. Note the monolithic structure between 2 and 3 mL mark.



d) TI008-SM, the monolithic structure remained cohesive after pouring from the glass centrifuge tube. The structure was hard, requiring significant effort to break.

Figure 3.11. Select Dried Solids Examination Images of SCS-CON-210 Container Composite Samples

Table 3.3. Dried Physical Property Sample Observations

Core and Sample Identification	Settled Solids Volume, mL	Dried Solids Volume, mL	Volume Reduction (%)	Observations
Core A1				
TI001-A1-K	12	8.5	29	Very light, flaky, easily removed from glass centrifuge tube wall. Powdery. Breaks in clumps. At sludge/water interface, dried material on glass difficult to remove. Small heel remained in centrifuge tube (vial) after pour.
TI001-A1-L	14.5	9	38	Poured well and granular. Flaky. Large particulates and fines. Large chunks with porosity. Small heel remained in centrifuge tube after pour.
Core A4				
TI002-A4-K	10.5	8.5	19	Material loosely adhered to centrifuge tube walls. Large agglomerate formed on top of the bed of fines. Material composed of fines, granules, agglomerates, and some flakes. Long, hair-like fibers holding several large agglomerates together.
TI002-A4-L	11	9	18	Material composed of fines, granules, agglomerates, and some flakes. Agglomerates broke apart with little force. Small heel remained in centrifuge tube after pour.
Core B1				
TI002-B1-K	11.5	8	30	Poured well and little solids adhered to centrifuge tube. Large agglomerate particles. More force required to break large agglomerates. Material composed of fines, granules, large agglomerates, and some flakes.
TI002-B1-L	13.25	6.5	51	Same as TI002-B1-K. In addition, a long, hair-like fiber holding several large agglomerates together was observed. Small heel remained in centrifuge tube after pour.
Core B3				
TI001-B3-K	12.5	7	44	Granular, fines, and some large agglomerates. Poured well and little solids adhered to centrifuge tube. More difficult to break large agglomerates than A1 material. Long, hair-like fiber holding several large agglomerates together.
TI001-B3-L	13	7	46	Large agglomerates formed on top of dry solids. Many fines. Material did not adhere to centrifuge tube. Large agglomerates on top of fines, embedded fibers, and porous. Large chunks difficult to break. Small heel remained in centrifuge tube after pour.
Container Composite				
KW210-CC-AA	8.5	4.5	47	Granular particles poured easily with only minor adherence to the glass walls.
KW210-CC-AB	9.0	5.7	37	Granular particles that poured easily. A large whitish chunk was observed and it was brittle.
KW210-CC-NA	8.4	4.5	46	Granular particles poured easily with only minor adherence to the glass walls.
KW210-CC-NB	9.6	5.7	41	Granular particles poured easily with only minor adherence to the glass walls.
Fines, −500 µm				
TI008-SM	5.1	2.5	51	A distinct formation was observed as a cohesive hockey-puck shape on top of granular material (~20% of the material volume). All items poured out of the centrifuge tube easily. The puck-shaped monolith was hard and required effort to crack. The rest of the material was granular in appearance.
TI008-SN	5.6	2.9	48	Same as TI008-SM.

4.0 Engineered Container SCS-CON-210 Core Samples

Four core samples were taken from Engineered Container SCS-CON-210. This section discusses the processing and characterization results from each core sample. Refer to Section 2.0 for discussions of sample receipt and preparation processes.

4.1 Core Sample Observations

As discussed in Section 2.3, color representation relative to the MacBeth ColorChecker® Rendition Chart (MacBeth Color Chart or equivalent) was required per the QAPjP/SAP. Example images of the MacBeth Color Chart taken with the in-cell camera in the HLRF (where polybottle receipt and core reconstitution activities occurred) are shown in Figure 4.1. As in previous compositing campaigns (Fiskum et al. 2011b), most green and blue shades reproduced to some degree, but red hues appeared muted or missing. These color reproduction issues need to be considered when viewing digital images captured during processing activities for the SCS-CON-210 core samples. In addition, varying lighting conditions in the hot cell tended to cause shades of sludge layers between video stills to differ. For this reason, attempts were made to capture images with the MacBeth Color Chart present.

The in-cell video camera failed on February 7, 2011 during an overnight composite jar settling test for core A1 and had to be replaced. No progressive degradation was observed leading up to the failure, which has been directly attributed to extended camera exposure to the high-activity samples and high dose rates in cell. Figure 4.2 compares video stills of the MacBeth Color Chart before and after the camera was replaced. Comparison of the video stills shows no detectable color loss. The camera failure occurred after all core compositing was completed and prior to any subsampling activities.

All SCS-CON-210 core samples required only one jar for sludge collection because the total volume collected from each core sample was less than 650 mL. Material in each of the four reconstituted core samples was visually heterogeneous, varied in color, and contained observable stratification layers. No unusually large particulate was visible in the core sample (see Section 5.0 for sieved material images).

Dewatering the reconstituted core samples took between one and three days (as described in Section 2.1); they were subsampled once the material consistency exhibited sufficient strength to suspend dense particles. KW210-B1 and B3 cores required four dewatering steps prior to the final five-minute core mixing step just before the start of subsampling. KW210-A1 and A4 cores required three dewatering steps and two dewatering steps, respectively. The desired consistency for “A” cores was achieved more quickly and with less effort than the “B” cores.

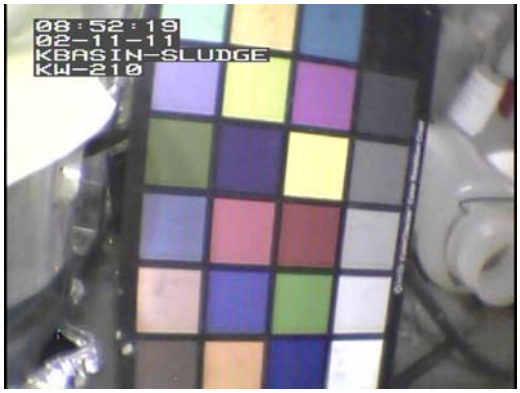
Subsamples H and J were collected first into clean vials and delivered directly to analytical workstations for subsequent analytical work (i.e., TIC/TOC). Analytical samples were collected next into 40-mL Oak Ridge digestion centrifuge tubes, followed by collection of physical property and container composite samples. When sufficient sludge volume remained in the reconstituted core sample jar, archive samples were collected (using the sample spoon) into 120-mL Qorpak jars. In addition, residual solids were rinsed from the reconstituted core sample jars and collected as archive samples to minimize sample loss.

MacBeth Checker Color Chart Standard

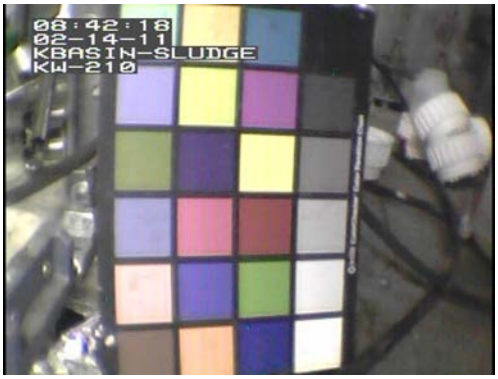
Note: Image is aligned to match 01/28/11 in-cell video still.

HLRF Color Chart: 01/28/11

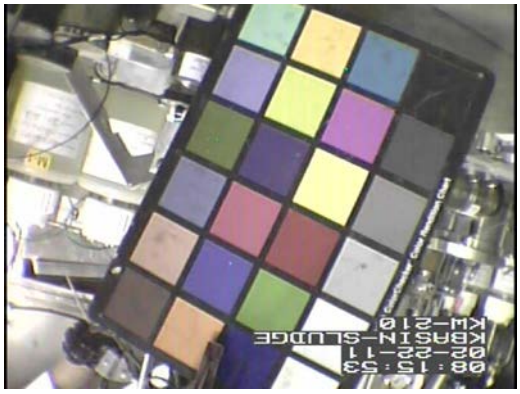
HLRF Color Chart: 02/03/11



HLRF Color Chart: 02/11/11



HLRF Color Chart: 02/14/11



HLRF Color Chart: 02/22/11

Figure 4.1. MacBeth Color Checker Chart Images in HLRF During Reconstitution and Subsampling of the SCS-CON-210 Core Samples

Images capturing the KW210-A1 core processing, from the reconstituted core to the examination of the dried primary physical properties samples, are shown in Figure 4.3. The images provided in Figure 4.3 are representative of the other three cores in terms of processing. Similar images for all four cores are provided in data package DVD-52578-PNNL-20650 Rev. 2, Section IV, September 2013.

HLRF Color Chart Image on 02/07/11

HLRF Color Chart Image on 02/10/11

Figure 4.2. Color Chart Images Before and After Camera Failure

KW210-A1-A Initial 4-L Polybottle, Core Sample

KW210-A1 Reconstituted Core Sample, Prior to Mixing

KW210-A1 Reconstituted Core Sample, Mixed and Settled

KW210-A1 Reconstituted Core Sample, Dewatered

Figure 4.3. Images of Reconstituted Core KW210-A1, Associated Subsamples, and a Dried Solids Examination

Subsampling Analytical

KW210-A1-A Sludge Level As-Sampled

As-Sampled TI001-A1-K

KW210-A1 Subsample Settling Test

Rewatered and Settled TI001-A1-K

TI001-A1-K Dried Solids Inspection ("Poke Test")

Figure 4.3. (contd)

Figure 4.4 provides sludge profile pictures of each of the four cores for a visual comparison of sludge morphology. The settled A1 and A4 core samples had similar material distributions with three distinct layers; first layer (bottom) a mixture of fines and agglomerates, second layer (middle) of primary granular agglomerates, and third (top) layer of grayish fines. The settled B1 and B3 core samples were also similar in appearance and settling behavior, but visually different from "A" cores. The "B" core samples had more dispersed fines distributed among a high number of resin beads.

KW210-A1

KW210-A4

KW210-B1

KW210-B3

Figure 4.4. Sludge Profile Images for All Four Reconstituted Core Samples After Initial Remix and Settle Test

Select images of unique observations made for the reconstituted cores are provided in Figures 4.5 through 4.10. The images document noteworthy and unique physical observations such as:

- material adhering to Teflon™-coated, magnetic spinbar in Oak Ridge tube
- solids materials and residues drying to jar walls
- scum layer and suspended particles at air/liquid interface
- gel-like material on mouth of 4-L core sample polybottles
- resin beads on top of fines prior to mixing
- vertical fluid channeling and solids mounds formation at sludge surface
- sludge surface mound formation and solids-ejecting plume captured in time lapsed photos.

Teflon-coated spinbar magnets were introduced into the Oak Ridge tubes for U metal analysis subsamples P and Q. Two example images, provided in Figure 4.5, captured the magnetic material adhering to the Teflon-coated magnets in samples TI002-B1-P and TI001-A1-Q. These images were captured after the subsamples were remixed and settled. All four cores produced this observation.

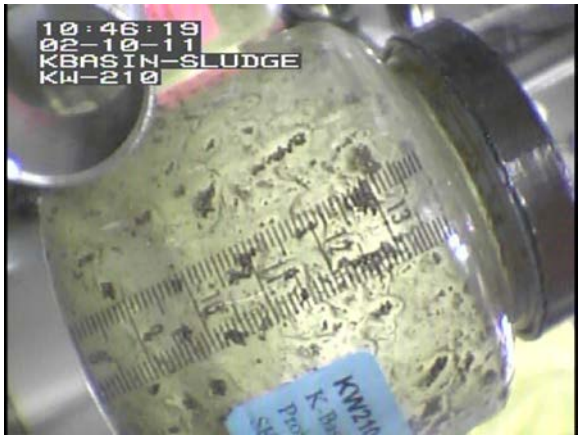
TI002-B1-P

TI001-A1-Q

Figure 4.5. Images of Magnetic Material Adhering to Telfon-Coated Spinbar Magnet in U Metal Subsamples

Solids residues and particles were observed to adhere to the jar walls for all four cores. After mixing and settling, Qorpak jar walls also became coated with what appeared to be a residue with many agglomerated solids distributed on the walls. Figure 4.6 provides select images for cores A1 and A4.

KW210-A1 Reconstituted Core, Surface Scum, Suspended Particles at Surface, and Dried Residue on Qorpak Jar Wall



KW210-A4 Dried Solids Adhered to Qorpak Jar After Remixing and Settling Sludge

Figure 4.6. Images of Dried Residues and Particles on Qorpak Jar Walls

All four cores exhibited similar scum layer formation at the air/liquid interface in the reconstituted core sample jars. This observation was also made during subsample settling tests. Figure 4.7 provides two select images capturing the scum layer. The scum frequently retained large particulates at the surface, apparently by surface tension, and often released the large particulates to the sludge layer below when the liquid surface was lightly disturbed.

KW210-B1 Reconstituted Core (Bottom Up View), Surface Scum and Suspended Particles at Liquid/Air Interface

KW210-A4 Reconstituted Core (Bottom Up View), Surface Scum and Suspended Particles at Liquid/Air Interface

Figure 4.7. Images of Scum Layer and Suspended Particles at Air/Liquid Interface

The observed scum material may be related to the gel-like material observed on the mouth of some 4-L polybottles while reconstituting the core samples. Sample images of this gel-like material are provided in Figure 4.8. This material was observed while bulk transferring the core sample to the composite jar. As with all four cores, the gel-like material was combined with the bulk sludge sample after rinsing the residual solids and vacuum transferring from the polybottle and transferring to the Qorpak composite jar. All 4-L polybottles were completely clean after the sludge transfer.

KW210-A1-A Gel-Like Material Adhesion to Polybottle Neck

KW210-A1-B Gel-Like Material Adhesion to Polybottle Neck

Figure 4.8. Images of Gel-Like Material on Mouth of 4-L Core Sample Polybottles

A notable observation on the behavior of the "A" cores was the compression of the sludge and movement of the fines as water and resin beads were displaced. Figure 4.9 presents two images of the KW210-A4 reconstituted core prior to mixing and settling. The black particles in the images appear to be resin beads suspended or mounded above the bulk fines level. When this core was mixed and settled this behavior was not observed again.

KW210-A4 Particles on Top of Fines Prior to Mixing

KW210-A4 Particles on Top of Fines Prior to Mixing

Figure 4.9. Images of Resin Beads on Top of Fines Prior to Mixing

Vertical fluid channeling and the formation of solids mounds at the sludge surface were observed uniquely in the "B" cores. After mixing the reconstituted core samples and allowing them to settle, vertical channels and voids appeared in the sludge (see Figure 4.10). If the jar was disturbed, these channels would frequently link to the sludge surface and fluid flow was observed that resulted in mound formations. The sediment mounds were commonly created at the jar walls, but also distributed on the sludge surface. The cause of this behavior is believed to be interstitial water trapped below a sludge fines layer cap. When a fissure was created or broadened by the jar disturbance, the fluid head pressure drove the material to the sludge/water interface. Fines and smaller granular particles were carried to the surface. This analogous behavior is described by the geologic term "clastic dike." Figure 4.11 shows the development of the "clastic dike" habit in KW210-B3.

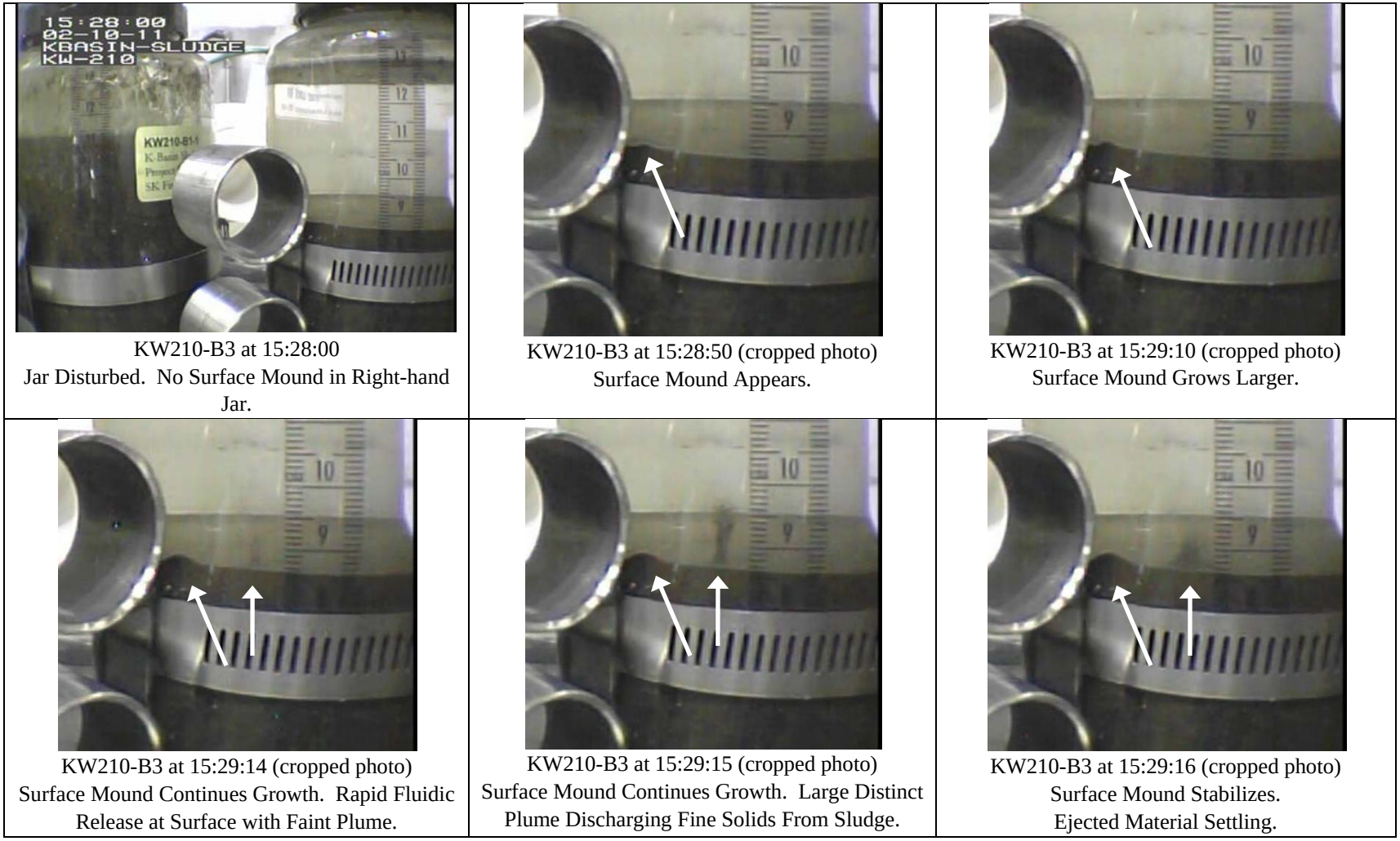
KW210-B1 Vertical Fluid Channels and Mounds

KW210-B1 Vertical Fluid Channels

KW210-B3 Surface Mounds

KW210-B3-Archive-1
Vertical Fluid Channels and Mounds

Figure 4.10. Images of Vertical Fluid Channels at Jar Walls and Solids Mounds at Sludge Surface for B1 and B3 Cores



KW210-B3 at 15:28:00
Jar Disturbed. No Surface Mound in Right-hand Jar.

KW210-B3 at 15:28:50 (cropped photo)
Surface Mound Appears.

KW210-B3 at 15:29:10 (cropped photo)
Surface Mound Grows Larger.

KW210-B3 at 15:29:14 (cropped photo)
Surface Mound Continues Growth. Rapid Fluidic Release at Surface with Faint Plume.

KW210-B3 at 15:29:15 (cropped photo)
Surface Mound Continues Growth. Large Distinct Plume Discharging Fine Solids From Sludge.

KW210-B3 at 15:29:16 (cropped photo)
Surface Mound Stabilizes.
Ejected Material Settling.

Figure 4.11. Time-Lapse Images of Fluid Ejecting Solids From the Sludge Surface and Mound Formation at the Wall in KW210-B3 Reconstituted Core Sample

4.2 Holding Time Limits

As stated in the data quality objectives document (Westcott et al. 2009), characterization of the reconstituted core samples may be used to assign hazardous waste codes (WIPP permit metals) in accordance with the WIPP Hazardous Waste Permit. To this end, a six-month holding time limit, defined from sample collection date to the metals analysis date, was applied to the samples. The samples were typically collected over a period of several days; ICP-OES analyses were conducted in a two-day span. The effective holding time was calculated from the most conservative standpoint (first sample date and last analysis date). The attained holding times for each core sample are provided in Table 4.1. Each core sample was processed within the six-month limit as defined above.

Table 4.1. Sample Holding Times Supporting WIPP Hazardous Waste Permit Metals Analysis

Core Sample ID	RPL/ASO ID[(a)]	Sample Date[(b)] (T_0)	End of 6-Month Holding Time	Acid Digestion Date	ICP-OES Analysis Date	Effective Holding Time, Months[(c)]
KW210-A4	11-0420	11/17/10	5/17/11	3/7/11	3/15/11-3/16/11	4.0
KW210-B1	11-0421	12/1/10	6/1/11			3.5
KW210-B3	11-0422	12/15/10	6/15/11			3.0
KW210-A1	11-0419	12/29/10	6/29/11			2.6

(a) RPL/ASO ID extensions included -129-S and -129-D.

(b) Sample date is conservatively applied to the date material was first collected from the isolation tube.

(c) The holding time was calculated from the most conservative application of dates, i.e., the first sample date and the last ICP-OES analysis date. For calculation purposes, a month was assumed to be 30 days.

4.3 Acid Dissolution for Chemical and Radiochemical Analysis

The reconstituted core samples were processed as one analytical batch (in accordance with ASR 8798) as described in Section 2.5.1. The pre-digestion dry mass, settled sludge mass, and UDS dry mass following the acid digestion procedure are shown in Table 4.2. The wt% of UDS (residual solids) was calculated on both the dried sample mass basis and the settled sludge mass basis. The wt% UDS varied between samples, ranging from 64 to 88 wt% on a dry sample mass basis, and from 27 to 40 wt% wet settled solids sample mass basis.

All samples exceeded the decision criterion of 5 wt% UDS (settled sludge mass) and thus triggered the need for GEA and XRD analyses (see Table 3-8 of the QAPjP/SAP [KBC-33786, Rev. 2]). The UDS samples were subsampled to support the KOH/KNO_3 fusion (PNL-ALO-115) dissolutions to measure ICP-OES analytes and alpha emitting isotopes by AEA in accordance with ASR 8878. The UDS composition is discussed in Section 4.8.

Table 4.2. SCS-CON-210 Chemical Analysis Characterization Samples for Acid Dissolution, Net UDS Masses, and UDS Weight Fraction

Container Core ID	Sample ID	RPL/ASO ID	Sample Pre-digest Dry Mass[a], g_{dry}	Average Conversion Factor[b], $g_{dry}/g_{settled}$	Pre-digest Settled Sample Mass[c], $g_{settled}$	UDS Post Digest Dry Mass[a], $g_{residual}$	Weight Fraction UDS (Residual)		
							Dry Mass Basis[d], $g_{residual} / g_{dry}$	RPD or RSD	Settled Mass Basis[e], $g_{residual} / g_{settled}$
KW210-A1	TI001-A1-B	11-0419-129-S	1.1233	0.4470	2.51	0.7262	0.6465	1.43%	0.289
	TI001-A1-C	11-0419-129-D	1.2773	0.4470	2.86	0.8140	0.6373		0.285
KW210-A4	TI002-A4-B	11-0420-129-S	1.4258	0.5667	2.52	1.0004	0.7016	0.09%	0.398
	TI002-A4-C	11-0420-129-D	1.5994	0.5667	2.82	1.1212	0.7010		0.397
KW210-B1	TI002-B1-B	11-0421-129-S	0.7459	0.3281	2.27	0.6192	0.8301	2.95%	0.272
	TI002-B1-C	11-0421-129-D	0.8192	0.3281	2.50	0.7206	0.8796		0.289
	TI002-B1-D	11-0421-129-MS	0.8818	0.3281	2.69	0.7621	0.8643		0.284
KW210-B3	TI001-B3-B	11-0422-129-S	1.0626	0.3625	2.93	0.9163	0.8623	0.98%	0.313
	TI001-B3-C	11-0422-129-D	1.0515	0.3625	2.90	0.9157	0.8709		0.316

(a) Mass measurements were made using four-place analytical balances. Results are found in analytical report "Mass Measurements" under ASR 8798 (see Appendix B).

(b) Conversion factors for $g_{dry}/g_{settled}$ are calculated from the average values of the primary physical property samples from each core as reported in Table 3.1. Individual mass ratios and averages are presented to four significant figures in this table. However, the accuracies of the mass ratios are only valid to the third significant digit; the fourth digit is provided for information and to help the reader re-create computed values without introducing the rounding error.

(c) The pre-digested settled sample mass is calculated from dividing the pre-digest dry mass by the average conversion factor ($g_{dry}/g_{settled}$) for the core. Data are presented to three significant figures to better represent the estimated accuracy.

(d) The weight fraction for UDS on a dry mass basis is calculated by dividing the dry mass of the UDS by the dry mass of the pre-digested solids. Data are presented to the appropriate significant figures.

(e) The weight fraction for UDS on a settled mass basis is calculated by multiplying the weight fraction for UDS on a dry mass basis by the average conversion factor ($g_{dry}/g_{settled}$) for the core. Data are presented to three significant figures to better represent the estimated accuracy.

Calculations of data presented above were performed using un-truncated values using a Microsoft Excel calculation spreadsheet. Calculation spreadsheets in Microsoft Excel are provided in DVD-52578-PNNL-20650 Rev. 2, Section III, September 2013.

4.4 Radiochemistry

Table 4.3 through Table 4.5 show the radioisotope analytical results for samples analyzed under ASR 8798. Results are presented on a dry mass basis (Table 4.3) with a companion summary of the QC sample results (precision and post-digestion BS and MS recoveries). The PB results are shown with the companion samples. The PB represents a blank prepared during the acid dissolution step and serves as an indicator of potential sources of contamination. The concentration basis of the PB was normalized to the average sample mass. Table 4.4 indicates the specific sample used for the MS evaluation. The QAPjP/SAP delineated a precision of ±20% between sample and duplicate results and accuracy to be within ±20 to ±25% as respectively measured from the BS and MS recoveries (see Table 2.12). All of the QAPjP/SAP requirements for precision (RPD) and accuracy (BS and MS percent recoveries) for the analytes listed in Table 4.4 were met. Results are also expressed in terms of wet settled sludge mass basis. The factors used to convert dry sludge mass to wet settled sludge mass were calculated from the average physical properties (Section 3.0) and are summarized in Table 4.5. The conversion to the wet settled mass basis was calculated according to Equation 3.2. The reference dates for the reported radioisotopes are provided in the corresponding data table. Analytical reports for ASR 8798 are found in Appendix B.

Table 4.6 through Table 4.9 provide the TIMS analytical results of the isotopic ratios for U and Pu. Results are presented in terms of At% and wt% in Table 4.6. As discussed in Section 2.5.9, Pu isotopic results were normalized to the ^{238}Pu AEA results. The QC sample results (RPD, results from the analysis of standard calibration verification check samples) are presented in Table 4.7 and discussed in detail in the sections below. The wt% results were converted to units of μg/g and μCi/g on a dry mass and settled mass basis in Table 4.8 and Table 4.9 respectively. Supporting documents for TIMS analytical results are found in Appendix E.

Specific radiochemistry results discussions are provided in the following sections.

4.4.1 Neptunium (Alpha Energy Analysis)

The alpha spectra were assessed for ^{237}Np and ^{238}U. Low levels of ^{238}U were observed in all of the samples. Three sample preparations contained ^{238}U equivalent to ~9% of the ^{237}Np activity detected in the sample (TI001-A1-C, TI002-B1-B, TI001-B3-B). The results from these samples were higher than their associated duplicate sample. The ^{234}U activity is ~1.4× the ^{238}U activity for this sample set (based on the isotopic distribution). Therefore a 9% ^{238}U impurity would be expected to increase the ^{237}Np alpha emission signal by an additional ~13% from the ^{234}U component (^{237}Np and ^{234}U have equivalent alpha emission energies).

The ^{237}Np results were nearly equivalent to the requested detection limit of 1E-3 μCi/g. The RPDs for each duplicate pair ranged from 2 to 82%; duplicate sample results from the A1 and B1 cores exceeded the ±20% acceptance criterion. Relative 1-σ uncertainties for each measurement ranged from 10 to 29% (actual uncertainties assigned to each sample are provided in Appendix B). These uncertainties represent the total propagated error associated with processing and counting operations, and include weighing errors, volume uncertainties, and counting error; they do not incorporate sub-sampling uncertainty (i.e., duplicate precision). The higher uncertainties were dominated by counting error, indicating that the ^{237}Np activity was nearing the detection limit.

Table 4.3. Radionuclide Results in Acid Digestate, Dry Sludge Mass Basis (ASR 8798)

Container:	SCS-CON-210									
Core:	A1			A4		B1			B3	
Sample ID:	PB	TI001-A1-B	TI001-A1-C	TI002-A4-B	TI002-A4-C	TI002-B1-B	TI002-B1-C	TI002-B1-D[(b)]	TI001-B3-B	TI001-B3-C
RPL/ASO ID >	11-0419-129-PB	11-0419-129-S	11-0419-129-D	11-0420-129-S	11-0420-129-D	11-0421-129-S	11-0421-129-D	11-0421-129-MS	11-0422-129-S	11-0422-129-D
AEA	μCi/g, Reference Dates: ^{241}Am April 11-12, 2011; $^{238, 239+240}$Pu April 8-12, 2011; ^{237}Np April 14-19, 2011 (reference dates same as counting dates)									
^{237}Np	<2E-6	1.74E-3	2.16E-3[(a)]	1.30E-3	1.19E-3	1.78E-3[(a)]	7.49E-4	--	1.14E-3[(a)]	1.12E-3
^{238}Pu	<3E-6	2.27E+0	2.56E+0	2.30E+0	2.01E+0	1.59E+0	1.65E+0	--	1.85E+0	1.77E+0
$^{239+240}$Pu	<3E-6	1.78E+1	1.92E+1	1.66E+1	1.47E+1	1.23E+1	1.26E+1	--	1.36E+1	1.26E+1
^{241}Am	5.67E-6	1.82E+1	1.97E+1	1.71E+1	1.43E+1	1.51E+1	1.48E+1	--	1.54E+1	1.47E+1
AEA	μg/g									
^{237}Np	<3E-3	2.47E+0	3.07E+0	1.85E+0	1.70E+0	2.54E+0	1.06E+0	--	1.63E+0	1.59E+0
^{241}Am	1.65E-6	5.31E+0	5.75E+0	4.98E+0	4.16E+0	4.39E+0	4.32E+0	--	4.49E+0	4.28E+0
Total U	μg/g									
U/KPA	0.02	98,000	108,000	87,900	77,100	55,500	48,800	--	56,200	53,100
U/ICP-OES	<11	93,800	103,000	82,800	73,600	51,900	45,600	--	55,700	51,000
GEA	μCi/g, Reference Date: January 11, 2011									
^{60}Co	<4E-5	2.30E-1	1.41E-1	1.34E-1	1.21E-1	7.34E-2	7.31E-2	6.73E-2	6.86E-2	6.39E-2
^{134}Cs	<4E-5	<3E-3	<6E-3	<3E-3	<6E-3	<4E-3	<3E-3	<5E-3	<5E-3	<5E-3
^{137}Cs	2.19E-4	3.68E+2	3.93E+2	3.38E+2	3.81E+2	1.43E+2	1.68E+2	1.50E+2	1.13E+2	1.02E+2
^{152}Eu	<2E-4	<5E-3	<8E-3	<5E-3	<9E-3	<4E-3	<7E-3	<1E-2	<1E-2	<1E-2
^{154}Eu	<1E-4	7.30E-1	7.59E-1	5.84E-1	5.41E-1	4.71E-1	5.16E-1	4.76E-1	5.28E-1	5.14E-1
^{155}Eu	<2E-4	1.18E-1	1.26E-1	<6E-2	<1E-1	8.71E-2	1.20E-1	<7E-2	8.04E-2	7.35E-2
^{241}Am	<3E-4	1.96E+1	2.13E+1	1.64E+1	1.39E+1	1.41E+1	1.68E+1	1.38E+1	1.48E+1	1.39E+1
Beta/LSC	μCi/g, Reference Date: April 1, 2011 (reference date same as separation date)									
^{90}Sr	<1E-4	3.44E+2	3.37E+2	2.71E+2	2.72E+2	1.39E+2	1.55E+2	--	1.44E+2	1.34E+2

(a) The sample Np fraction contained a slight quantity of U; ^{234}U has an equivalent alpha emission energy as ^{237}Np and would cause slight high bias. See Section 4.4.1.

(b) Sample TI002-B1-D was the batch MS sample that was spiked with analytes of interest for ICP-OES. From a GEA perspective, it can be used as a confirmatory sample for KW210 Core B1.

Relative uncertainties were typically between 2 and 6% except for ^{237}Np where 10 to 29% uncertainties were reported. Analytical results including 1-sigma uncertainties from ASR 8798 are found in Appendix B.

RPD calculations reported in Table 4.4 for KW210 Core B1 are based on samples TI002-B1-B and TI002-B1-C.

Shaded analyte results indicate the QAPjP/SAP precision criterion of ±20% was exceeded.

Table 4.4. Radionuclide QC Sample Results in Acid Digestate (ASR 8798)

Analyte	Container SCS-CON-210 Core Samples				LCS/BS	MS	MS
	A1	A4	B1[(a)]	B3	Recovery,	Recovery,	Sample
	RPD (%)	RPD (%)	RPD (%)	RPD (%)	%	%	ID
AEA							
^{237}Np	22	9	82	2	102	103	TI001-B3-B
^{238}Pu	12	13	3	5	--	--	--
$^{239+240}$Pu	7	12	2	7	100	101	TI001-B3-B
^{241}Am	8	18	2	5	102	97	TI001-B3-B
Total U							
U/KPA	10	13	13	6	103	103	TI001-B3-C
U/ICP-OES	9.6	12	13	8.9	100	NR	--
GEA							
^{60}Co	48	10	0.4	7	--	--	--
^{134}Cs	---	---	---	---	--	--	--
^{137}Cs	7	12	16	10	--	--	--
^{152}Eu	---	---	---	---	--	--	--
^{154}Eu	4	8	9	3	--	--	--
^{155}Eu	7	---	32	9	--	--	--
^{241}Am	8	17	17	6	--	--	--
Beta/LSC							
^{90}Sr	2	0.5	11	8	99	98	TI001-B3-B

(a) Sample TI002-B1-D was the batch MS sample spiked with analytes of interest for ICP-OES. From a GEA perspective, it can be used as a confirmatory sample for KW210 Core B1. Relative percent deviation (RPD) calculations for the KW210 Core B1 are based on samples TI002-B1-B and TI002-B1-C.

NR: not recovered; spike was too low relative to the uranium present in the sample matrix.

Dashed lines indicate the property was not applicable. The RPD could not be determined because the analyte concentration was less than the detection limit; the spike recovery did not apply.

Analytical results and uncertainties from ASR 8798 samples are found in Appendix B.

Shaded analyte results indicate the QAPjP/SAP precision criterion of ±20% was exceeded.

Table 4.5. Radionuclide Results in Acid Digestate, Wet Settled Sludge Mass Basis (ASR 8798)

Container:	SCS-CON-210									
Core:	A1			A4		B1			B3	
Sample ID:	PB	TI001-A1-B	TI001-A1-C	TI002-A4-B	TI002-A4-C	TI002-B1-B	TI002-B1-C	TI002-B1-D[b]	TI001-B3-B	TI001-B3-C
RPL/ASO ID >	11-0419-129-PB	11-0419-129-S	11-0419-129-D	11-0420-129-S	11-0420-129-D	11-0421-129-S	11-0421-129-D	11-0421-129-MS	11-0422-129-S	11-0422-129-D
Conversion Factor[a]	0.426	0.447	0.447	0.567	0.567	0.328	0.328	0.328	0.363	0.363
AEA	μCi/g, Reference Date: ^{241}Am April 11-12, 2011; $^{238, 239+240}$Pu April 8-12, 2011; ^{237}Np April 14-19, 2011 (reference dates same as counting dates)									
^{237}Np	9.84E-7	7.77E-4	9.66E-4[c]	7.39E-4	6.76E-4	5.85E-4[c]	2.46E-4	--	4.15E-4[c]	4.05E-4
^{238}Pu	1.44E-6	1.02E+0	1.15E+0	1.30E+0	1.14E+0	5.22E-1	5.40E-1	--	6.72E-1	6.42E-1
$^{239+240}$Pu	1.38E-6	7.97E+0	8.56E+0	9.40E+0	8.34E+0	4.02E+0	4.12E+0	--	4.92E+0	4.58E+0
^{241}Am	2.42E-6	8.15E+0	8.82E+0	9.68E+0	8.08E+0	4.95E+0	4.86E+0	--	5.59E+0	5.32E+0
AEA	μg/g									
^{237}Np	1.40E-3	1.11E+0	1.37E+0	1.05E+0	9.62E-1	8.32E-1	3.49E-1	--	5.89E-1	5.75E-1
^{241}Am	7.04E-7	2.37E+0	2.57E+0	2.82E+0	2.36E+0	1.44E+0	1.42E+0	--	1.63E+0	1.55E+0
Total U	μg/g									
U/KPA	0	43,800	48,300	49,800	43,700	18,200	16,000	--	20,400	19,200
U/ICP-OES	<4.5	41,900	46,000	46,900	41,700	17,000	15,000	--	20,200	18,500
GEA	μCi/g, Reference Date: January 11, 2011									
^{60}Co	<2E-5	1.03E-1	6.30E-2	7.59E-2	6.86E-2	2.41E-2	2.40E-2	2.21E-2	2.49E-2	2.32E-2
^{134}Cs	<2E-5	<1E-3	<3E-3	<2E-3	<3E-3	<1E-3	<1E-3	<2E-3	<2E-3	<2E-3
^{137}Cs	9.33E-5	1.64E+2	1.76E+2	1.92E+2	2.16E+2	4.69E+1	5.51E+1	4.92E+1	4.10E+1	3.70E+1
^{152}Eu	<9E-5	<2E-3	<4E-3	<3E-3	<5E-3	<1E-3	<2E-3	<4E-3	<4E-3	<4E-3
^{154}Eu	<4E-5	3.26E-1	3.39E-1	3.31E-1	3.07E-1	1.55E-1	1.69E-1	1.56E-1	1.91E-1	1.86E-1
^{155}Eu	<9E-5	5.27E-2	5.63E-2	<3E-2	<6E-2	2.86E-2	3.94E-2	<2E-2	2.91E-2	2.66E-2
^{241}Am	<1E-4	8.76E+0	9.52E+0	9.29E+0	7.88E+0	4.63E+0	5.51E+0	4.53E+0	5.37E+0	5.04E+0
Beta/LSC	μCi/g, Reference Date: April 1, 2011 (reference date same as separation date)									
^{90}Sr	6.21E-5	1.54E+2	1.51E+2	1.53E+2	1.54E+2	4.57E+1	5.08E+1	--	5.22E+1	4.84E+1

(a) The conversion factor, $g_{dry}/g_{settled}$, was determined from the primary physical property samples (see Table 3.1).

(b) Sample TI002-B1-D was the batch MS sample spiked with analyte of interest for ICP-OES. From a GEA perspective, it can be used as a confirmatory sample for KW210 Core B1.

(c) The sample Np fraction contained a slight quantity of U; ^{234}U has an equivalent alpha emission energy as ^{237}Np and would cause slight high bias. See Section 4.4.1.

RPD calculations reported in Table 4.4 for KW210 Core B1 are based on samples TI002-B1-B and TI002-B1-C.

Shaded analyte results indicate the QAPjP/SAP precision criterion of ±20% was exceeded.

Table 4.6. Uranium and Plutonium TIMS Isotopic Ratios in Acid Digestate

Container:	SCS-CON-210							
Core:	A1		A4		B1		B3	
Sample ID:	TI001-A1-B	TI001-A1-C	TI002-A4-B	TI002-A4-C	TI002-B1-B	TI002-B1-C	TI001-B3-B	TI001-B3-C
RPL/ ASO ID >	11-0419-129-S	11-0419-129-D	11-0420-129-S	11-0420-129-D	11-0421-129-S	11-0421-129-D	11-0422-129-S	11-0422-129-D
Isotopic U	TIMS Data, Reference Date: May 3-9, 2011							
Analyte	Atom%							
^{233}U	0	0	0	0	0	0	0	0
^{234}U(a)	0.008	0.007	0.007	0.007	0.007	0.007	0.008	0.011
^{235}U	0.711	0.709	0.702	0.705	0.706	0.704	0.710	0.714
^{236}U(a)	0.073	0.076	0.072	0.074	0.071	0.072	0.071	0.077
^{238}U	99.208	99.208	99.220	99.214	99.216	99.217	99.211	99.198
Analyte	Mass%							
^{233}U	0	0	0	0	0	0	0	0
^{234}U(a)	0.0078	0.0066	0.0067	0.0073	0.0065	0.0069	0.0076	0.0107
^{235}U	0.702	0.700	0.693	0.696	0.697	0.695	0.701	0.705
^{236}U(a)	0.073	0.075	0.071	0.073	0.071	0.072	0.070	0.077
^{238}U	99.217	99.218	99.229	99.224	99.226	99.227	99.221	99.208
Isotopic Pu	TIMS Data, Reference Date: May 18 and 25, 2011							
Analyte	Atom% (Normalized)							
^{238}Pu	0.0619	0.0651	0.0678	0.0667	0.0630	0.0639	0.0663	0.0685
^{239}Pu	86.115	86.250	85.926	86.203	86.431	86.235	86.251	86.016
^{240}Pu	12.918	12.920	13.219	12.995	12.794	12.982	12.918	13.183
^{241}Pu	0.674	0.516	0.533	0.508	0.486	0.485	0.535	0.522
^{242}Pu	0.231	0.250	0.254	0.228	0.227	0.234	0.230	0.209
Analyte	Mass% (Normalized)							
^{238}Pu	0.0617	0.0648	0.0675	0.0664	0.0627	0.0635	0.0660	0.0682
^{239}Pu	86.061	86.197	85.872	86.150	86.378	86.182	86.198	85.963
^{240}Pu	12.964	12.966	13.266	13.041	12.839	13.029	12.964	13.230
^{241}Pu	0.680	0.520	0.537	0.512	0.490	0.489	0.539	0.526
^{242}Pu	0.233	0.253	0.257	0.231	0.230	0.237	0.232	0.212

(a) Most of the measured ^{234}U and ^{236}U ratios of NIST standard U-030 exceeded the allowed ±2% difference from the certified values for the five performance checks of the system (see Appendix E for the individual results). The 1-σ uncertainties of the ^{234}U and ^{236}U values were large (much greater than 2%, relative) and generally bracketed the ±2% criterion range. The Analysis Request approved by the BTR only passed down the requirement for isotopes where the concentration exceeded an atom% of 0.1 (i.e., specifically applicable to ^{235}U and ^{238}U). See Occurrence Report 52578-05-09-11 in Appendix E for details.

The samples prepared by the ASO for U TIMS analysis included suffix "-U"; the samples provided for Pu TIMS analysis had a suffix "-Pu". These are not shown in the sample ID at the top for brevity and clarity.

Shaded values indicate RPD exceeded 20%; however, the ±20% QAPjP/SAP criterion only applies to isotopes where the atomic abundance is greater than 1%.

Additional significant figures (obtained from the supporting analytical data) are shown for additional information. See TIMS report in Appendix E. Calculation spreadsheets in Microsoft Excel are provided in DVD-52578-PNNL-20650 Rev. 2, Section III, September 2013.

Table 4.7. Uranium and Plutonium TIMS QC Sample Results in Acid Digestate

Container:	SCS-CON-210[(a)]				Calibration Verification Standard
Core:	A1	A4	B1	B3	% Difference of Pooled Average from Certificate Value
Analyte	RPD (%)	RPD (%)	RPD (%)	RPD (%)	SRM U-030
^{233}U	---	---	---	---	---
^{234}U	17	8.4	5.6	33	0.61[(b)]
^{235}U	0.26	0.43	0.33	0.58	-0.88
^{236}U	3.1	2.7	1.2	8.3	-2.66[(b)]
^{238}U	0.001	0.006	0.001	0.013	0.03
Normalized					SRM Pu-946
^{238}Pu	5.0	1.7	1.4	3.3	0.29
^{239}Pu	0.16	0.32	0.23	0.27	0.05
^{240}Pu	0.013	1.7	1.5	2.0	-0.35
^{241}Pu	27	4.7	0.22	2.5	0.69
^{242}Pu	8.0	11	2.9	9.2	-0.36

(a) The RPD values were calculated from the unrounded sample and the duplicate atom% values (TIMS values obtained from the TIMS calculation spreadsheet [Appendix E]). The Excel calculation spreadsheet file is provided in Appendix E (ASR 8798 Data Reduction Rev2.xlsx). Excel files can also be found in DVD-52578-PNNL-20650 Rev. 2, Section III, September 2013. Shaded blocks indicate that the RPD of the duplicate pair exceeded ±20%. However, the isotopic abundances of ^{240}Pu, ^{241}Pu, ^{234}U and ^{236}U were <1 atom%; therefore the ±20% RPD precision criterion was not applicable.

(b) Most of the measured ^{234}U and ^{236}U ratios of NIST standard U-030 exceeded the allowed ±2% difference from the certified values for the five performance checks of the system (see Appendix E for the individual results). The 1-σ uncertainties of the ^{234}U and ^{236}U values were large (much greater than 2%, relative) and generally bracketed the ±2% criterion range. The Analysis Request approved by the BTR only passed down the requirement for isotopes where the concentration exceeded an atom% of 0.1 (i.e., specifically applicable to ^{235}U and ^{238}U). See Occurrence Report 52578-05-09-11 in Appendix E for details.

SRM: Standard Reference Material.

Table 4.8. Uranium and Plutonium Isotope Mass and Activity Distributions in Acid Digestate, Dry Sludge Mass Basis

Container:	SCS-CON-210							
Core:	A1		A4		B1		B3	
Sample ID:	TI001-A1-B	TI001-A1-C	TI002-A4-B	TI002-A4-C	TI002-B1-B	TI002-B1-C	TI001-B3-B	TI001-B3-C
RPL/ ASO ID >	11-0419-129-S	11-0419-129-D	11-0420-129-S	11-0420-129-D	11-0421-129-S	11-0421-129-D	11-0422-129-S	11-0422-129-D
Isotopic U	TIMS Data, Reference Date: May 3-9, 2011							
Analyte	µg/g							
^{233}U	0E+0	0E+0	0E+0	0E+0	0E+0	0E+0	0E+0	0E+0
^{234}U[(a)]	7.6E+0	7.1E+0	5.9E+0	5.6E+0	3.6E+0	3.4E+0	4.3E+0	5.7E+0
^{235}U	6.88E+2	7.56E+2	6.09E+2	5.36E+2	3.87E+2	3.39E+2	3.94E+2	3.74E+2
^{236}U[(a)]	7.1E+1	8.1E+1	6.3E+1	5.7E+1	3.9E+1	3.5E+1	4.0E+1	4.1E+1
^{238}U	9.723E+4	1.072E+5	8.722E+4	7.650E+4	5.507E+4	4.842E+4	5.576E+4	5.268E+4
Analyte	µCi/g							
^{233}U	0E+0	0E+0	0E+0	0E+0	0E+0	0E+0	0E+0	0E+0
^{234}U[(a)]	4.7E-2	4.4E-2	3.7E-2	3.5E-2	2.3E-2	2.1E-2	2.7E-2	3.5E-2
^{235}U	1.49E-3	1.63E-3	1.32E-3	1.16E-3	8.36E-4	7.33E-4	8.51E-4	8.09E-4
^{236}U[(a)]	4.6E-3	5.2E-3	4.1E-3	3.7E-3	2.5E-3	2.3E-3	2.6E-3	2.6E-3
^{238}U	3.268E-2	3.602E-2	2.932E-2	2.571E-2	1.851E-2	1.628E-2	1.874E-2	1.771E-2
Isotopic Pu	TIMS Data, Reference Date: May 18 and 25, 2011							
Analyte	µg/g (Normalized)							
^{238}Pu	1.33E-1	1.50E-1	1.34E-1	1.18E-1	9.29E-2	9.61E-2	1.08E-1	1.03E-1
^{239}Pu	1.854E+2	1.992E+2	1.708E+2	1.528E+2	1.281E+2	1.303E+2	1.413E+2	1.303E+2
^{240}Pu	2.793E+1	2.996E+1	2.639E+1	2.312E+1	1.904E+1	1.970E+1	2.125E+1	2.006E+1
^{241}Pu	1.46E+0	1.20E+0	1.07E+0	9.08E-1	7.26E-1	7.39E-1	8.84E-1	7.98E-1
^{242}Pu	5.03E-1	5.84E-1	5.11E-1	4.09E-1	3.41E-1	3.58E-1	3.81E-1	3.21E-1
Analyte	µCi/g (Normalized)							
^{238}Pu	2.27E+0	2.56E+0	2.30E+0	2.01E+0	1.59E+0	1.65E+0	1.85E+0	1.77E+0
^{239}Pu	1.150E+1	1.235E+1	1.060E+1	9.475E+0	7.946E+0	8.085E+0	8.763E+0	8.084E+0
^{240}Pu	6.336E+0	6.797E+0	5.988E+0	5.246E+0	4.320E+0	4.471E+0	4.821E+0	4.551E+0
^{241}Pu	1.52E+2	1.25E+2	1.11E+2	9.42E+1	7.54E+1	7.67E+1	9.18E+1	8.28E+1
^{242}Pu	1.99E-3	2.31E-3	2.02E-3	1.62E-3	1.35E-3	1.42E-3	1.51E-3	1.27E-3

(a) Most of the measured ^{234}U and ^{236}U ratios of NIST standard U-030 exceeded the allowed ±2% difference from the certified values for the five performance checks of the system (see Appendix E for the individual results). The 1-σ uncertainties of the ^{234}U and ^{236}U values were large (much greater than 2%, relative) and generally bracketed the ±2% criterion range. The Analysis Request approved by the BTR only passed down the requirement for isotopes where the concentration exceeded an atom% of 0.1 (i.e., specifically applicable to ^{235}U and ^{238}U). See Occurrence Report 52578-05-09-11 in Appendix E for details.

Shaded values indicate RPD exceeded 20%; however, the ±20% QAPjP/SAP criterion only applies to isotopes where the atom abundance is greater than 1%.

See TIMS report in Appendix E. Calculation spreadsheets in Microsoft Excel are provided in DVD-52578-PNNL-20650 Rev. 2, Section III, September 2013.

Table 4.9. Uranium and Plutonium Isotope Mass and Activity Distributions in Acid Digestate, Wet Settled Sludge Mass Basis

Container:	SCS-CON-210							
Core:	A1		A4		B1		B3	
Sample ID:	TI001-A1-B	TI001-A1-C	TI002-A4-B	TI002-A4-C	TI002-B1-B	TI002-B1-C	TI001-B3-B	TI001-B3-C
RPL/ ASO ID >	11-0419-129-S	11-0419-129-D	11-0420-129-S	11-0420-129-D	11-0421-129-S	11-0421-129-D	11-0422-129-S	11-0422-129-D
Isotopic U	TIMS Data, Reference Date: May 3-9, 2011							
Analyte	µg/g							
^{233}U	0E+0	0E+0	0E+0	0E+0	0E+0	0E+0	0E+0	0E+0
^{234}U[(a)]	3.4E+0	3.2E+0	3.3E+0	3.2E+0	1.2E+0	1.1E+0	1.6E+0	2.1E+0
^{235}U	3.08E+2	3.38E+2	3.45E+2	3.04E+2	1.27E+2	1.11E+2	1.43E+2	1.36E+2
^{236}U[(a)]	3.2E+1	3.6E+1	3.6E+1	3.2E+1	1.3E+1	1.1E+1	1.4E+1	1.5E+1
^{238}U	4.35E+4	4.79E+4	4.94E+4	4.34E+4	1.81E+4	1.59E+4	2.02E+4	1.91E+4
Analyte	µCi/g							
^{233}U	0E+0	0E+0	0E+0	0E+0	0E+0	0E+0	0E+0	0E+0
^{234}U[(a)]	2.1E-2	2.0E-2	2.1E-2	2.0E-2	7.4E-3	6.9E-3	9.7E-3	1.3E-2
^{235}U	6.65E-4	7.31E-4	7.46E-4	6.57E-4	2.74E-4	2.40E-4	3.09E-4	2.93E-4
^{236}U[(a)]	2.1E-3	2.3E-3	2.3E-3	2.1E-3	8.3E-4	7.4E-4	9.3E-4	9.5E-4
^{238}U	1.46E-2	1.61E-2	1.66E-2	1.46E-2	6.07E-3	5.34E-3	6.79E-3	6.42E-3
Isotopic Pu	TIMS Data, Reference Date: May 18 and 25, 2011							
Analyte	µg/g (Normalized)							
^{238}Pu	5.94E-2	6.69E-2	7.61E-2	6.67E-2	3.05E-2	3.15E-2	3.92E-2	3.75E-2
^{239}Pu	8.29E+1	8.90E+1	9.68E+1	8.66E+1	4.20E+1	4.28E+1	5.12E+1	4.72E+1
^{240}Pu	1.25E+1	1.34E+1	1.50E+1	1.31E+1	6.25E+0	6.46E+0	7.70E+0	7.27E+0
^{241}Pu	6.54E-1	5.37E-1	6.05E-1	5.15E-1	2.38E-1	2.42E-1	3.20E-1	2.89E-1
^{242}Pu	2.25E-1	2.61E-1	2.90E-1	2.32E-1	1.12E-1	1.17E-1	1.38E-1	1.16E-1
Analyte	µCi/g (Normalized)							
^{238}Pu	1.02E+0	1.15E+0	1.30E+0	1.14E+0	5.22E-1	5.40E-1	6.72E-1	6.42E-1
^{239}Pu	5.14E+0	5.52E+0	6.00E+0	5.37E+0	2.61E+0	2.65E+0	3.18E+0	2.93E+0
^{240}Pu	2.83E+0	3.04E+0	3.39E+0	2.97E+0	1.42E+0	1.47E+0	1.75E+0	1.65E+0
^{241}Pu	6.79E+1	5.57E+1	6.28E+1	5.34E+1	2.47E+1	2.52E+1	3.33E+1	3.00E+1
^{242}Pu	8.89E-4	1.03E-3	1.15E-3	9.17E-4	4.42E-4	4.64E-4	5.46E-4	4.61E-4

(a) Most of the measured ^{234}U and ^{236}U ratios of NIST standard U-030 exceeded the allowed ±2% difference from the certified values for the five performance checks of the system (see Appendix E for the individual results). The 1-σ uncertainties of the ^{234}U and ^{236}U values were large (much greater than 2%, relative) and generally bracketed the ±2% criterion range. The Analysis Request approved by the BTR only passed down the requirement for isotopes where the concentration exceeded an atom% of 0.1 (i.e., specifically applicable to ^{235}U and ^{238}U). See Occurrence Report 52578-05-09-11 in Appendix E for details.

Shaded values indicate RPD exceeded 20%; however, the ±20% QAPjP/SAP criterion only applies to isotopes where the atomic concentration is greater than 1%.

See TIMS report in Appendix E. Calculation spreadsheets in Microsoft Excel are provided in DVD-52578-PNNL-20650 Rev. 2, Section III, September 2013.

The accuracy, expressed as post-digestion BS and MS recoveries (102 and 103%), was within the acceptance limits identified in the QAPjP/SAP (BS: 80 to 120%; MS: 75 to 125%). The hot cell acid digestion PB showed no detectable ^{237}Np activity (<2E-6) and indicated that contamination from hot cell processing activities was unlikely.

4.4.2 Plutonium (Alpha Energy Analysis)

The duplicate pair results for ^{238}Pu and $^{239+240}$Pu showed RPDs ranging between 3 and 12% for ^{238}Pu and 2 to 12% for $^{239+240}$Pu – each meeting the QAPjP/SAP requirements. Relative 1-σ uncertainties ranged from 3 to 6% for ^{238}Pu measurements from 2 to 3% for $^{239+240}$Pu measurements (actual uncertainties assigned to each sample and analyte are provided in Appendix B). These uncertainties represent the total propagated error associated with processing and count operations and include weighing errors, volume uncertainties, and counting error; they do not incorporate sub-sampling uncertainty. The accuracy, expressed as post-digestion BS and MS recoveries of the ^{239}Pu analytical spike (100 and 101%), was within the acceptance limits identified in the QAPjP/SAP. The hot cell acid digestion PB resulted in no detectable activity for ^{238}Pu or $^{239+240}$Pu. A ^{242}Pu and ^{243}Am tracer was added to every sample before the radiochemical separations. There was no evidence of ^{243}Am in the alpha spectra, indicating that the ^{238}Pu analyte peak was not confounded by the presence of ^{241}Am. Radiochemical yields based on recoveries of the ^{242}Pu internal standard ranged from 94 to 108%.

4.4.3 Americium (Alpha Energy Analysis)

The duplicate pair results for ^{241}Am by AEA showed RPDs ranging from 2 to 18%. Relative 1-σ measurement uncertainties (inclusive of all radiochemistry sources of laboratory errors—volumetric, mass, and counting uncertainties) for ^{241}Am were 2% (see ASO analysis report in Appendix B). The accuracy, expressed as post-digestion BS and MS recoveries of the ^{241}Am analytical spike (102 and 97%, respectively), was within the acceptance limits identified in the QAPjP/SAP. The hot cell acid digestion PB showed detectable activity for ^{241}Am. However, the blank activity concentration was six orders of magnitude less than the observed lowest sample activity concentration. A ^{242}Pu and ^{243}Am tracer was added to every sample before the radiochemical separations. The alpha spectra were free of $^{239+240}$Pu indicating that ^{238}Pu was not present and thus not confounding the ^{241}Am peak. Radiochemical yields based on recoveries of ^{243}Am internal standard ranged from 92 to 114%.

The ^{241}Am activity concentrations from GEA (where no radiochemical separations were applied) were also compared to the concentrations determined by AEA (where chemical separations were applied). The ratio of AEA/GEA results ranged from 0.88 to 1.07 with an average ratio of 1.00 with a standard deviation of 0.07. The close agreement between the two techniques provides another measure of the overall measurement accuracy for this analyte.

4.4.4 Gamma Energy Analysis

Five of the seven gamma-emitting radionuclides of interest were detected in the sample aliquots: ^{60}Co, ^{137}Cs, ^{154}Eu, and ^{241}Am were detected in all samples; ^{155}Eu was detected in three sample duplicate pairs (not detected in KW210-A4) in quantities below the requested detection limit. Neither ^{134}Cs nor ^{152}Eu were detected, and their minimum detectible concentrations (MDCs) are reported. In most cases, the RPDs were within ±20%; RPDs exceeding the QAPjP/SAP criteria include ^{60}Co in KW210-A1 (RPD

of 48%) and ^{155}Eu in KW210-B1 (RPD 32%). Relative 1-σ measurement uncertainties (inclusive of all radiochemistry lab errors—volumetric, mass, and counting uncertainties) for ^{60}Co, ^{137}Cs, ^{154}Eu and ^{241}Am ranged from 2 to 5% (with the exception of TI002-B1-C where 20% 1-σ error was reported for ^{241}Am). The 1-σ error for ^{155}Eu results ranged from 7 to 19%. Specific uncertainties assigned to each sample are provided in Appendix B. The hot cell PB resulted in no detectable analyte activity except for ^{137}Cs, which was five orders of magnitude less than the lowest activity in the samples. The GEA reference date is January 11, 2011.

4.4.5 Strontium-90 Analysis

The duplicate pair results for ^{90}Sr showed RPDs ranging from 0.5 to 11%, meeting the QAPjP/SAP criterion. The reported 1-σ uncertainty (inclusive of all sources of radiochemistry laboratory error—volumetric, mass, and counting uncertainties) for each ^{90}Sr sample concentration was ±2%. The accuracy, expressed as BS and MS recoveries of the ^{90}Sr analytical spike (99 and 98%, respectively), was within the acceptance limits identified in the QAPjP/SAP. No ^{90}Sr activity was detected in the hot cell PB, indicating cross-contamination was not likely to have occurred. (The radiochemistry report is provided in Appendix B.) The reference date for the reported ^{90}Sr concentration is the separation date (April 1, 2011).

4.4.6 Total Uranium (Kinetic Phosphorescence Analysis)

The measured precision of the duplicate pairs ranged from 6 to 13% RPD and met the QAPjP/SAP requirement of ±20% RPD. The reported measurement uncertainty for each U sample concentration was ±4% (1-σ). The BS, prepared as part of the sample digestion activity, recovered 103%, meeting the QAPjP/SAP requirement. Recovery of the post-digestion MS was also 103%. The calibration check sample results ranged from 102 to 103% of true value, which was within the ±7.5% limits defined in the QAPjP/SAP.

The KPA U results demonstrated agreement with the ICP-OES U results. The U KPA/U ICP-OES ratio averaged 1.05 with a standard deviation of 0.02. The close agreement between the two techniques provides another measure of the overall accuracy for this analyte.

4.4.7 Uranium Isotopic Analysis

Table 4.6 shows the results of the TIMS uranium isotopic analysis conducted on May 3-9, 2011. All U isotopes of interest were detected except ^{233}U. The duplicate pairs resulted in atom% precision measures of 6 to 33% for ^{234}U (measured values had high uncertainties 13 to 45%), ≤1% RPD for ^{235}U, ≤8% RPD for ^{236}U, and ≤0.01% ^{238}U (specific measurement uncertainties for each isotope are provided in Appendix E). The TIMS accuracy was assessed with one standard reference material (SRM) U-030. The QAPjP/SAP-delineated accuracy for U TIMS was ±2%. Measures of the major U isotopes, ^{235}U and ^{238}U, from all five analysis runs easily met this requirement (see Appendix E). However, the measured concentrations of for ^{234}U and ^{236}U exceeded ±2% during some of the performance checks of the system. The high spread of the SRM ^{234}U and ^{236}U atom% values was not unexpected considering their isotopic concentrations are low at 0.019 and 0.020 atom%, respectively. This issue is discussed in detail in Occurrence Report 52578-05-09-11 located in Appendix E with the TIMS final report. The relative

difference of the certified values from the pooled averages for this analysis set were less than 3% (-0.88% for ^{235}U and 0.03% for ^{238}U) as shown in Table 4.7.

The wt% of each U isotope in each core subsample was determined by multiplying the atom% (Table 4.6) by the atomic mass of the specific U isotope and then dividing by the atomic weight of U (238.03). The U isotope concentration (µg/g) was determined by multiplying the wt% of each U isotope by the U KPA concentration in Table 4.3. The U isotope concentration (µCi/g) was determined by multiplying the mass concentration of each isotope by the specific activity (Ci/g) of the isotope.

4.4.8 Plutonium Isotopic Analysis

Plutonium TIMS analysis was conducted May 18 and 25, 2011. All Pu isotopes of interest were detected. The TIMS results shown in Table 4.6, Table 4.8, and Table 4.9 were normalized to ^{238}Pu determined from Pu AEA analysis (see calculation defined in Section 2.5.9). The normalization was considered necessary because there was a significant probability that U could contaminate the Pu fraction confounding the ^{238}Pu with isobaric ^{238}U.

All duplicate pairs met the QAPjP/SAP precision criteria of ±20% RPD (applicable to isotopes with atom abundance is greater than 1%, i.e., ^{239}Pu and ^{240}Pu). The minor isotopes of the duplicate pairs also were within ±20% RPD with one exception; ^{241}Pu in KW210-A1 resulted in 27% RPD. Accuracy for Pu isotopic distribution analysis by TIMS was assessed with SRM Pu-946; all measured isotopes for each of the nine standard runs were measured within ±6% of the certified value allowed by the QAPjP/SAP. The relative difference of the certified and decay-corrected values from the pooled averages for this analysis set were less than 1% (0.05% for ^{239}Pu and 0.35% for ^{240}Pu). These values are shown in Table 4.7 and further details are provided in the TIMS final report located in Appendix E.

The wt% and isotope concentrations for each Pu isotope were calculated analogously to the U concentrations after normalizing to the ^{238}Pu (AEA) concentration (see Sections 2.5.9 and 4.4.7).

4.5 ICP-OES Analytes

The ICP-OES-measured AOI concentrations for ASR 8798 are shown in Table 4.10. Table 4.11 presents opportunistically measured analytes. The analytes are reported on a dry mass basis in both tables. The ICP-OES measurements have a general overall uncertainty of ±15% or better where analytes are greater than estimated quantitation limit (EQL) and in dilute, acidified water. Bracketed values indicate that concentrations were measured at less than the EQL but greater than the method detection limit; uncertainty for these values will likely be higher than 15%. The less-than concentrations were determined by multiplying the IDL by the sample-specific process factor; process factor components include the sample size and final volume of digestate.

Overall QC results for the AOIs are summarized in Table 4.12. Additional details (also the opportunistic analytes) are provided in the ICP-OES analytical report for ASR 8798 located in Appendix B. The RPD was calculated only where both duplicate pair values exceeded the EQL. A majority of the RPD measurements met the QAPjP/SAP criterion of ±20%. However, several RPD failures (highlighted with gray shading in the tables) were found in three of the core samples.

Accuracy was assessed through the LCS/BS and MS recoveries (where the spike was added before digestion), as well as from the post-digestion spikes (PSs) and serial dilution. All LCS/BS recoveries met the acceptance criterion of 80 to 120%. While a majority of the MS recoveries also met the acceptance criteria as well, four analytes did not: Sb (45%), Tl (30%), Zn (74%), and Zr (244%). The MS recoveries for Sb and Tl were significantly lower than had been obtained previously (Fiskum et al. 2011a, b, and Shimskey et al. 2011).[(a)] The low MS recoveries point to significant matrix-related signal depression issues with respect to these two analytes. The high Zr MS recovery indicates inhomogeneity in the subsamples for this analyte; the duplicate pair associated with the MS sample had a high Zr RPD of 135%, also indicative that the subsamples collected for analysis varied in Zr concentration. In selected cases (for analytes Al, Ca, Fe, and U), MS recoveries were not able to be calculated and thus not reported (NR). This occurred when the sample analyte concentration greatly exceeded the spiked amount. All PS recovery results met the acceptance criterion (80 to 120%); a further indication that a matrix effect exists. The serial dilution compared analyte concentrations above EQL after an additional 5× dilution at the workstation and is an additional measure of accuracy. The calculated percent differences were well below the 10% criterion established within the ASO procedure for those analytes where MS recoveries were not possible.

Detection limit requirements defined in the QAPjP/SAP were met for all AOIs with the exception of Se in the KW210-B1 duplicate pair. The duplicate pair (nominal MDL = 22 μg/g on a dry mass basis) exceeded the targeted 20 μg/g limit delineated in the ASR. However, the nominal detection limit was 7 μg/g on a wet settled sludge mass basis, which met the objective of the QAPjP/SAP (KBC-33786, Rev.2; Table 3-8). The PB showed analyte concentrations above the MDL but less than EQL for Ba, Cr, Na, B, Bi, K, P, Sn, and Sr.

Table 4.13 and Table 4.14 show the AOIs and opportunistic analytes, respectively, on a wet settled sludge mass basis. As in Table 4.10 through Table 4.11, analyte concentrations are shaded where RPDs exceeded ±20%. However, DQO requirements were not applicable for opportunistic analytes.

(a) The historically low Sb, Se, and Tl MS recoveries in the K Basin sludge sample matrices were communicated to the BTR in PNNL correspondence 52578-2011-L06. The issues concerning 100% attainment of the various QAPjP/SAP requirements on all QC samples were discussed in detail in PNNL correspondence 59205-2011-L04 (K Basin Settler sludge). Because the same issues apply to the KW210 core sample results, the letters are provided as part of this data package in Appendix B.

Table 4.10. ICP-OES Analytes of Interest in Acid Digestate, Dry Sludge Mass Basis (ASR 8798)

Container:	SCS-CON-210								
Core:		A1		A4		B1		B3	
Sample ID:	PB	TI001-A1-B	TI001-A1-C	TI002-A4-B	TI002-A4-C	TI002-B1-B	TI002-B1-C	TI001-B3-B	TI001-B3-C
RPL/ASO ID >	11-0419-129-PB	11-0419-129-S	11-0419-129-D	11-0420-129-S	11-0420-129-D	11-0421-129-S	11-0421-129-D	11-0422-129-S	11-0422-129-D
Analyte	(µg/g)	(µg/g)	(µg/g)	(µg/g)	(µg/g)	(µg/g)	(µg/g)	(µg/g)	(µg/g)
Ag	<1.7	[5.5]	[3.8]	[2.8]	[2.8]	[4.2]	[3.5]	[2.0]	[3.9]
Al	<4.0	32,500	32,600	20,700	22,400	15,600	15,500	12,400	12,100
As	<8.4	89.2	98.4	72.3	72.7	[46]	[41]	[54]	[50]
Ba	[0.24]	192	199	135	143	75.4	78.0	66.8	62.4
Be	<0.059	6.99	10.2	12.2	6.70	3.03	18.6	2.76	2.51
Ca	<1.9	7,640	6,750	6,690	7,740	7,410	6,590	8,350	7,510
Cd	<1.3	35.9	33.5	29.8	34.6	65.5	57.4	48.8	48.8
Cr	[3.4]	354	277	569	337	287	274	225	218
Fe	<4.0	86,800	88,600	96,400	93,700	35,100	27,500	31,800	27,800
Mg	<0.54	1140	1010	730	943	582	648	588	582
Na	[14]	708	655	1,420	1650	1,710	1,610	1,460	1,340
Ni	<2.3	185	128	334	206	223	178	157	156
Pb	<18	345	345	238	264	319	322	313	287
Sb[(a)]	<1.9	[8.6]	[8.0]	[9.4]	[8.5]	<2.9	[4.6]	[5.1]	[3.9]
Se	<15	<15	<13	<12	<11	<23	<21	<16	<16
Tl[(a)]	<7.6	[9.2]	[8.7]	[6.6]	[6.8]	<12	<10	<8.1	<8.2
U	<11	93,800	103,000	82,800	73,600	51,900	45,600	55,700	51,000
V	<1.0	[15]	[15]	[13]	14.2	[9.8]	[11]	[9.8]	[8.4]
Zn[(a)]	<4.9	469	398	291	383	394	243	211	200
Zr[(a)]	<0.74	334	369	373	148	77.6	400	91.8	97.2

(a) The batch MS recovery exceeded the QAPjP/SAP criteria, see Table 4.12.

Shaded analyte results indicate the QAPjP/SAP precision criterion of ±20% was exceeded.

Results shown in brackets indicate that the analyte concentrations were greater than the MDL but less than the EQL.

Less-than values represent the instrument detection limit multiplied by the process factors.

Table 4.11. ICP-OES Opportunistic Analytes in Acid Digestate, Dry Sludge Mass Basis (ASR 8798)

Container:	SCS-CON-210								
Core:	A1			A4		B1		B3	
Sample ID:	PB	TI001-A1-B	TI001-A1-C	TI002-A4-B	TI002-A4-C	TI002-B1-B	TI002-B1-C	TI001-B3-B	TI001-B3-C
RPL/ASO ID >	11-0419-129-PB	11-0419-129-S	11-0419-129-D	11-0420-129-S	11-0420-129-D	11-0421-129-S	11-0421-129-D	11-0422-129-S	11-0422-129-D
Analyte	(µg/g)	(µg/g)	(µg/g)	(µg/g)	(µg/g)	(µg/g)	(µg/g)	(µg/g)	(µg/g)
B	[39]	52.9	[38]	[26]	[25]	[52]	[47]	[31]	[26]
Bi	[59]	[120]	[110]	[100]	[86]	[140]	[150]	[120]	[100]
Ce	<7.6	79.2	74.9	63.1	55.9	[32]	[45]	[48]	[53]
Co	<2.2	[4.6]	[3.3]	[6.3]	[2.4]	[5.8]	[6.0]	[4.7]	[2.9]
Cu	<1.4	1,680	1,070	510	487	1,190	1,170	822	807
Dy	<1.1	<1.1	<0.94	<0.84	<0.75	<1.6	<1.5	[1.7]	<1.1
Eu	<0.24	[2.3]	2.34	2.00	2.02	[1.3]	[1.7]	[0.86]	[1.1]
K	619	1,240	1,230	967	912	1,340	1,120	657	696
La	<1.1	24.5	24.7	20.1	20.9	19.8	18.9	21.9	17.9
Li	<0.40	[3.2]	3.67	[2.1]	[2.4]	<0.62	<0.56	[0.85]	[0.70]
Mn	<0.19	343	291	313	297	222	227	214	193
Mo	<6.2	[43]	[44]	[42]	[35]	[27]	[30]	[27]	[27]
Nd	<2.4	86.9	92.6	54.0	50.6	[35]	41.6	57.6	45.7
P	[47]	[430]	[370]	[210]	[240]	[450]	[470]	[470]	[480]
Pd	<4.2	<4.3	<3.8	<3.4	<3.0	<6.4	<5.9	<4.5	<4.6
Rh	<7.7	<7.8	<6.9	<6.2	<5.5	<12	<11	<8.3	<8.4
Ru	<3.9	[19]	[21]	[13]	[18]	[13]	[12]	[18]	[14]
S	<140	7,060	6,130	6,140	7,120	9,720	8,930	9,470	8,870
Si	<11	749	678	592	610	697	656	492	482
Sn	[35]	[88]	[91]	[67]	[56]	286	[160]	[71]	[58]
Sr	[0.041]	63.8	57.8	43.2	49.8	27.3	26.2	23.7	21.6
Ta	<17	<17	<15	<13	<12	<25	<23	<18	<18
Te	<14	<14	[16]	<11	<10	<21	<20	<15	<15
Th	<4.4	<4.5	<3.9	<3.5	<3.1	<6.7	<6.1	<4.7	<4.8
Ti	<0.46	464	358	382	374	197	205	138	127
W	<13	<13	<12	<11	<9.4	<20	<18	<14	<14
Y	<0.22	7.32	6.86	5.43	5.66	[4.4]	[4.7]	5.08	[4.5]

Results shown in brackets indicate that the analyte concentrations were greater than the MDL but less than the EQL. Opportunistic analytes are analytes not identified within the (QAPjP/SAP) and are shown for information-only; QAPjP/SAP QC sample requirements do not apply to these analytes. Less-than values represent the instrument detection limit multiplied by the process factors.

Shaded analyte results indicate the sample and duplicate sample RPD exceeded ±20%.

Table 4.12. ICP-OES QC Sample Results for Analytes of Interest in Acid Digestate (ASR 8798)

Container:	SCS-CON-210[a]				LCS/BS[b]	MS[b]	Post Spike	Serial
Core:	A1	A4	B1	B3	Recovery,	Recovery,	Recovery,	Dilution,[c]
Analyte	RPD	RPD	RPD	RPD	%	%	%	% Diff
Ag	--	--	--	--	96	100	99	--
Al	0.4	7.6	0.8	2.1	101	NR	102	1.6
As	9.9	0.6	--	--	85	90	97	--
Ba	3.8	5.5	3.3	6.9	97	104	101	3.1
Be	37.7	58.5	144.1	9.3	98	105	103	--
Ca	12.4	14.6	11.6	10.6	99	NR	101	1.8
Cd	7.0	15.0	13.2	0.1	100	90	104	--
Cr	24.5	51.0	4.6	3.0	95	102	101	0.2
Fe	2.1	2.9	24.4	13.7	105	NR	99	2.1
Mg	12.1	25.4	10.8	1.1	102	118	101	8.3
Na	7.8	14.9	6.4	8.5	103	119	100	0.3
Ni	36.5	47.3	22.0	0.8	101	105	105	2.0
Pb	0.0	10.2	0.8	8.7	100	105	100	--
Sb	--	--	--	--	89	45	95	--
Se	--	--	--	--	89	96	99	--
Tl	--	--	--	--	84	30	92	--
U	9.6	11.7	13.0	8.9	100	NR	98	1.7
V	--	--	--	--	94	99	99	--
Zn	16.3	27.3	47.3	5.3	93	74	99	6.8
Zr	9.8	86.4	135.0	5.7	104	244	104	3.5

(a) Shaded RPD values indicate the QAPjP/SAP precision criterion of ±20% was exceeded. A dashed line indicates that the analyte concentration was below EQL and RPD could not be determined.

(b) The LCS and MS targeted recoveries were 80 to 120%. Shaded MS recoveries indicate QAPjP/SAP accuracy criterion of ±20% (80 to 120% analyte recovery) was exceeded.

(c) The dashed line indicates the analyte in the diluted sample was <EQL and the percent difference could not be determined.

NR: Not Recovered; analyte concentration in the sample far exceeded the added spike amount.

Table 4.13. ICP-OES Analytes of Interest in Acid Digestate, Wet Settled Sludge Mass Basis (ASR 8798)

Container:	SCS-CON-210								
Core:	A1		A4		B1		B3	
Sample ID:	PB	TI001-A1-B	TI001-A1-C	TI002-A4-B	TI002-A4-C	TI002-B1-B	TI002-B1-C	TI001-B3-B	TI001-B3-C
RPL/ASO ID >	11-0419-129-PB	11-0419-129-S	11-0419-129-D	11-0420-129-S	11-0420-129-D	11-0421-129-S	11-0421-129-D	11-0422-129-S	11-0422-129-D
Analyte	(µg/g)	(µg/g)	(µg/g)	(µg/g)	(µg/g)	(µg/g)	(µg/g)	(µg/g)	(µg/g)
Ag	<0.71	[2.5]	[1.7]	[1.6]	[1.6]	[1.4]	[1.1]	[0.73]	[1.4]
Al	<1.7	14,500	14,600	11,700	12,700	5,120	5,090	4,500	4,390
As	<3.6	39.9	44.0	41.0	41.2	[15]	[13]	[20]	[18]
Ba	[0.10]	85.8	89.0	76.5	81.0	24.7	25.6	24.2	22.6
Be	<0.025	3.12	4.56	6.91	3.80	0.994	6.10	1.00	0.910
Ca	<0.82	3,420	3,020	3,790	4,390	2,430	2,160	3,030	2,720
Cd	<0.56	16.0	15.0	16.9	19.6	21.5	18.8	17.7	17.7
Cr	[1.4]	158	124	322	191	94.2	89.9	81.6	79.0
Fe	<1.7	38,800	39,600	54,600	53,100	11,500	9,020	11,500	10,100
Mg	<0.23	510	451	414	534	191	213	213	211
Na	[6.0]	316	293	805	935	561	528	529	486
Ni	<1.0	82.7	57.2	189	117	73.2	58.4	56.9	56.6
Pb	<7.9	154	154	135	150	105	106	113	104
Sb[(a)]	<0.82	[3.8]	[3.6]	[5.3]	[4.8]	<1.0	[1.5]	[1.8]	[1.4]
Se	<6.4	<6.8	<5.9	<6.8	<6.0	<7.5	<6.8	<5.8	<5.9
Tl[(a)]	<3.2	[4.1]	[3.9]	[3.7]	[3.9]	<3.8	<3.4	<2.9	<3.0
U	<4.5	41,900	46,000	46,900	41,700	17,000	15,000	20,200	18,500
V	<0.41	[6.7]	[6.7]	[7.4]	8.05	[3.2]	[3.6]	[3.6]	[3.0]
Zn[(a)]	<2.1	210	178	165	217	129	79.7	76.5	72.5
Zr[(a)]	<0.31	149	165	211	83.9	25.5	131	33.3	35.2

(a) The batch MS recovery exceeded the QAPjP/SAP criteria, see Table 4.12.

Shaded analyte results indicate the QAPjP/SAP precision criterion of ±20% was exceeded.

Results shown in brackets indicate that the analyte concentrations were greater than the MDL but less than the EQL. Less-than values represent the instrument detection limit multiplied by the process factors.

Table 4.14. ICP-OES Opportunistic Analytes in Acid Digestate, Wet Settled Sludge Mass Basis (ASR 8798)

Container:	SCS-CON-210								
Core:	A1			A4		B1		B3	
Sample ID:	PB	TI001-A1-B	TI001-A1-C	TI002-A4-B	TI002-A4-C	TI002-B1-B	TI002-B1-C	TI001-B3-B	TI001-B3-C
RPL/ASO ID >	11-0419-129-PB	11-0419-129-S	11-0419-129-D	11-0420-129-S	11-0420-129-D	11-0421-129-S	11-0421-129-D	11-0422-129-S	11-0422-129-D
Analyte	(µg/g)	(µg/g)	(µg/g)	(µg/g)	(µg/g)	(µg/g)	(µg/g)	(µg/g)	(µg/g)
B	[17]	23.6	[17]	[15]	[14]	[17]	[15]	[11]	[9.4]
Bi	[25]	[54]	[49]	[57]	[49]	[46]	[49]	[44]	[36]
Ce	<3.3	35.4	33.5	35.8	31.7	[10]	[15]	[17]	[19]
Co	<0.94	[2.1]	[1.5]	[3.6]	[1.4]	[1.9]	[2.0]	[1.7]	[1.1]
Cu	<0.60	751	478	289	276	390	384	298	293
Dy	<0.45	<0.48	<0.42	<0.48	<0.43	<0.53	<0.48	[0.62]	<0.41
Eu	<0.10	[1.0]	1.05	1.13	1.14	[0.43]	[0.56]	[0.31]	[0.40]
K	264	554	550	548	517	440	367	238	252
La	<0.45	11.0	11.0	11.4	11.8	6.50	6.20	7.94	6.49
Li	<0.17	[1.4]	1.64	[1.2]	[1.4]	<0.20	<0.18	[0.31]	[0.25]
Mn	<0.082	153	130	177	168	72.8	74.5	77.6	70.0
Mo	<2.6	[19]	[20]	[24]	[20]	[8.9]	[9.8]	[9.8]	[9.8]
Nd	<1.0	38.8	41.4	30.6	28.7	[11]	13.6	20.9	16.6
P	[20]	[190]	[170]	[120]	[140]	[150]	[150]	[170]	[170]
Pd	<1.8	<1.9	<1.7	<1.9	<1.7	<2.1	<1.9	<1.6	<1.7
Rh	<3.3	<3.5	<3.1	<3.5	<3.1	<3.9	<3.5	<3.0	<3.0
Ru	<1.6	[8.5]	[9.4]	[7.4]	[10]	[4.3]	[3.9]	[6.5]	[5.1]
S	<60	3,160	2,740	3,480	4,030	3,190	2,930	3,430	3,220
Si	<4.9	335	303	335	346	229	215	178	175
Sn	[15]	[39]	[41]	[38]	[32]	93.8	[52]	[26]	[21]
Sr	[0.017]	28.5	25.8	24.5	28.2	8.96	8.60	8.59	7.83
Ta	<7.1	<7.6	<6.6	<7.6	<6.7	<8.4	<7.6	<6.5	<6.6
Te	<6.0	<6.4	[7.2]	<6.4	<5.7	<7.0	<6.4	<5.5	<5.5
Th	<1.9	<2.0	<1.7	<2.0	<1.8	<2.2	<2.0	<1.7	<1.7
Ti	<0.19	207	160	216	212	64.6	67.3	50.0	46.0
W	<5.6	<6.0	<5.2	<6.0	<5.3	<6.6	<6.0	<5.1	<5.2
Y	<0.094	3.27	3.07	3.08	3.21	[1.4]	[1.5]	1.84	[1.6]

Results shown in brackets indicate that the analyte concentrations were greater than the MDL but less than the EQL.

Opportunistic analytes are analytes not identified within the QAPjP/SAP and are shown for information only; QAPjP/SAP QC sample requirements did not apply to these analytes. Shaded analyte results indicate the sample and duplicate sample RPD exceeded ±20%.

Less-than values represent the instrument detection limit multiplied by the process factors.

4.6 TOC and TIC Analysis

Table 3-4a and Figure 3-4 of the QAPjP/SAP specified the analysis for TOC and indicated that the samples are to be analyzed on a wet (settled sludge mass basis); Table 3-8 indicated TOC/TC[(a)] be performed. As discussed in the following paragraphs, and in accordance with the BTR-approved ASR 8798 Special Instructions, the samples were dried at 105°C to constant weight before TOC and TIC analyses were performed.

Grafoil® and OIER were found to be present in some sludge samples based on observations of the sludge make-up during previous containerized sludge characterization activities (Fiskum et al. 2011b). Consequently, the BTR provided additional direction to include the analysis of Grafoil® and the carbon contribution from OIER in K Basin sludge samples. These observations and requirements drove the need to use sequential temperature combustion processing (600 and 1,000°C) as previously described (Fiskum et al. 2011a) to oxidize the carbon in these materials (procedure RPG-CMC-385, Rev. 2). Using this method, the TOC results from OIER Purolite NRW37 samples were shown to recover 77.7 to 107.8% (average of 90±8 %), and the TIC from Grafoil® were shown to recover 66.6 to 111% (average of 103±8 %) (Fiskum et al. 2011a). The OIER material is considered an upper limit in difficulty for organic compound combustion; therefore, any other organic species present will also be effectively combusted at 600°C. Similar rationale applies to the Grafoil® combustion in that all inorganic carbon forms will oxidize at 1,000°C (e.g., calcium carbonate). Therefore, results are not exclusive to these materials in the sample, but will be inclusive of them.

Small, representative subsamples collected for TOC and TIC analyses from the dewatered parent material (i.e., core samples) were transferred to 30-mL glass vials and removed from the hot cell. The transferred subsample masses varied sample to sample and needed to be <1 g (for ALARA purposes). Each subsample contained significant headspace and no additional water was added to maintain them in a wetted condition prior to analysis. The TOC and TIC analyses were performed at the workstation several months after collection (June 2011).

The subsamples collected from the parent material were dried at 105°C to constant mass. This resulted in a normalized mass basis for each subsample and allowed the dry-mass/wet-settled mass conversion factor (Table 3.1) to be applied such that the TOC and TIC concentrations could be reported on a wet-settled sludge mass basis. (Note that an attempt to subsample wetted sludge at the analytical workstation would be confounded because the water content, and thus the mass basis, would be different from known conditions such as dried, as-sampled, and as-settled sample material.) Three small replicate subsamples were collected from the dried sample material for sequential TOC and TIC analyses. The small replicate subsamples were collected on a best-effort basis to be as representative as possible of the supplied material. The replicate subsample masses ranged from 3.5 to 64.8 mg. The analytical samples were driven to small sizes by the nature of the material (i.e., high organic carbon content, high dose rate, high actinide concentrations, and small PSD leading to highly dispersible sample characteristics) and the processing location (i.e., fume hood) where the carbon analyzer was located. Many of the samples analyzed for the K Basin project have high carbon content (typically from OIER and Grafoil®), so small sample sizes are required to prevent overwhelming the detector system with product CO_2 (upper working limit is generally limited to ~5,000 μg C).

(a) The TC is the sum of TIC and TOC.

It is understood that the sample collection, storage, and drying process could result in loss of volatile organic compounds, if present. Any component that volatilizes around 105°C will be lost in this method; typically this condition applies to organic solvents (benzene boiling point is 80°C and octane ~125°C [Weast and Astle 1979]). Organic salts tend to volatilize or decompose at higher temperatures (for example, the melting point of sodium formate is 253°C, the melting point of glucose is 146°C [Weast and Astle 1979]).

Table 4.15 and Table 4.16 summarize the TOC and TIC concentrations, respectively, in terms of μg carbon per g dry sample for each replicate subsample, along with the associated dry mass sample size. The EQL (considered 5× the MDL) was exceeded for all reported sample results except for the TIC on the duplicate B1 core subsamples and one of the B3 core subsamples. The precision of the three replicate samples from each analysis is shown in the data tables. Although significant variability existed in the "A" core sample TOC results, the RPD between the weighted average of the sample and duplicate (each were measured in triplicate) met the QAPjP/SAP precision criterion for each core.

The TIC results showed a lot more variability within the replicate sample set and the weighted average of the duplicate pairs than was observed with the TOC results. All TIC results were close to the MDL, a consequence of the small sample aliquot taken for analysis to accommodate the high TOC content. Table 4.17 summarizes the TOC and TIC concentrations on a wet settled mass basis from the weighted average sample results and the conversion factor (g dry/g settled) found in Table 3.1.

Each sample was processed on a different day because of the long duration of an analysis run. A LCS was processed each day samples were processed; two samples were prepared with analytical spike α-D-glucose and calcium carbonate (normal spike materials to assess system performance). The TOC LCS (a-D-glucose) recovered between 93 and 115%; the TIC LCS (calcium carbonate) recovered between 80 and 97%. One analytical spike sample recovery (132% TOC on sample 11-419-C-S) exceeded the acceptance limit[(a)]; all others met acceptance criteria (see Appendix B).

Additionally, staff were directed to prepare a laboratory-created test sample (LCTS) consisting of combined Purolite NRW37 and Grafoil® (RPL/ASO ID 11-0423-C-S and -D). The LCTS was processed at the beginning and at the conclusion of the sludge sample processing. Recoveries of the LCTS TOC and TIC were assessed and are summarized in Table 4.18. All TOC and TIC recoveries were within ±10% of the known C concentration indicating system performance was acceptable.

(a) The high analytical spike recovery was most likely an artifact of the subsampling processing and associated TOC variability between sub-samples.

Table 4.15. TOC of SCS-CON-210 Core Samples, Dry Sludge Mass Basis (ASR 8798)

Core:	KW210-A1					
TI ID:	TI001-A1-H			TI001-A1-J		
RPL/ASO Sample ID:	11-0419-C-S			11-0419-C-D		
	Sample size, g	TOC, µg/g	TOC, µg	Sample size, g	TOC, µg/g	TOC, µg
Replicate 1	0.0326	131,000	4,271	0.0184	130,000	2,392
Replicate 2	0.0165	178,000	2,937	0.0241	193,000	4,651
Replicate 3	0.0058	104,000	603	0.0097	97,400	945
RSD		27%			35%	
Total	0.0549		7,811	0.0522		7,988
Weighted Average (total µg C/ mass sum g)		142,300			153,000	
RPD of Weighted Averages			7.2%			
Core:	KW210-A4					
TI ID	TI002-A4-H			TI002-A4-J		
RPL/ASO Sample ID:	11-0420-C-S			11-0420-C-D		
	Sample size, g	TOC, µg/g	TOC, µg	Sample size, g	TOC, µg/g	TOC, µg
Replicate 1	0.0189	40,900	773	0.0200	51,500	1,030
Replicate 2	0.0340	72,300	2,458	0.0648	93,900	6,085
Replicate 3	0.0577	94,700	5,464	0.0362	118,000	4,272
RSD		39%			38%	
Total	0.1106		8,695	0.1210		11,386
Weighted Average (total µg C/ mass sum g)		78,620			94,100	
RPD of Weighted Averages			18%			
Core:	KW210-B1					
TI ID	TI002-B1-H			TI002-B1-J		
RPL/ASO Sample ID:	11-0421-C-S			11-0421-C-D		
	Sample size, g	TOC, µg/g	TOC, µg	Sample size, g	TOC, µg/g	TOC, µg
Replicate 1	0.0132	518,000	6,838	0.0046	439,000	2,019
Replicate 2	0.0035	516,000	1,806	0.0101	505,000	5,101
Replicate 3	0.0188	510,000	9,588	0.0148	499,000	7,385
RSD		0.8%			7.6%	
Total	0.0355		18,232	0.0295		14,505
Weighted Average (total µg C/ mass sum g)		513,600			491,700	
RPD of Weighted Averages			4.4%			
Core:	KW210-B3					
TI ID	TI001-B3-H			TI001-B3-J		
RPL/ASO Sample ID:	11-0422-C-S			11-0422-C-D		
	Sample size, g	TOC, µg/g	TOC, µg	Sample size, g	TOC, µg/g	TOC, µg
Replicate 1	0.0096	356,000	3,418	0.0106	353,000	3,742
Replicate 2	0.0261	366,000	9,553	0.0219	378,000	8,278
Replicate 3	0.0053	464,000	2,459	0.0141	371,000	5,231
RSD		15%			4%	
Total	0.041		15,429	0.0466		17,251
Weighted Average (total µg C/ mass sum g)		376,330			370,200	
RPD of Weighted Averages			1.6%			

Table 4.16. TIC of SCS-CON-210 Core Samples, Dry Sludge Mass Basis (ASR 8798)

Core:	KW210-A1					
TI ID:	TI001-A1-H			TI001-A1-J		
RPL/ASO Sample ID:	11-0419-C-S			11-0419-C-D		
	Sample size, g	TIC, µg/g	TIC, µg	Sample size, g	TIC, µg/g	TIC, µg
Replicate 1	0.0326	7,880	257	0.0184	644	11.8
Replicate 2	0.0165	3,190	52.6	0.0241	2,090	50.4
Replicate 3	0.0058	[1,240]	[7.2]	0.0097	[507]	[4.9]
RSD		83%			81%	
Total	0.0549		317	0.0522		67
Weighted Average (total µg C/ mass sum g)		5770			1290	
RPD of Weighted Averages			127%			
Core:	KW210-A4					
TI ID	TI002-A4-H			TI002-A4-J		
RPL/ASO Sample ID:	11-0420-C-S			11-0420-C-D		
	Sample size, g	TIC, µg/g	TIC, µg	Sample size, g	TIC, µg/g	TIC, µg
Replicate 1	0.0189	2,360	44.6	0.0200	[590]	[12]
Replicate 2	0.0340	3,550	121	0.0648	491	31.8
Replicate 3	0.0577	[163]	[9.4]	0.0362	707	25.6
RSD		85%			18%	
Total	0.1106		175	0.1210		69
Weighted Average (total µg C/ mass sum g)		1580			570	
RPD of Weighted Averages			94%			
Core:	KW210-B1					
TI ID	TI002-B1-H			TI002-B1-J		
RPL/ASO Sample ID:	11-0421-C-S			11-0421-C-D		
	Sample size, g	TIC, µg/g	TIC, µg	Sample size, g	TIC, µg/g	TIC, µg
Replicate 1	0.0132	<660	<8.7	0.0046	<3,600	<17
Replicate 2	0.0035	[2,790]	[9.8]	0.0101	[1,800]	[18]
Replicate 3	0.0188	<460	<8.6	0.0148	<1,100	<16
RSD		NA			NA	
Total	0.0355		[9.8 to 27]	0.0295		[18 to 51]
Weighted Average (total µg C/ mass sum g)		<760			<1,700	
RPD of Weighted Averages			NA			
Core:	KW210-B3					
TI ID	TI001-B3-H			TI001-B3-J		
RPL/ASO Sample ID:	11-0422-C-S			11-0422-C-D		
	Sample size, g	TIC, mg/g	TIC, mg	Sample size, g	TIC, mg/g	TIC, mg
Replicate 1	0.0096	[800]	[7.7]	0.0106	5,180	54.9
Replicate 2	0.0261	865	23	0.0219	3,300	72.3
Replicate 3	0.0053	<840	<4.5	0.0141	2,470	34.8
RSD		7.8%[(a)]			38%	
Total	0.0410		[31 to 35]	0.0466		162
Weighted Average (total µg C/ mass sum g)		[800]			3,480	
RPD of Weighted Averages			NA			

(a) The precision (RPD) was calculated from the two measurements above the MDL.

Table 4.17. TOC and TIC of SCS-CON-210 Core Samples, Wet Settled Sludge Mass Basis (ASR 8798)

	KW210-A1		KW210-A4	
TI ID >	TI001-A1-H	TI001-A1-J	TI002-A4-H	TI002-A4-J
RPL/ASO ID >	11-0419-C-S	11-0419-C-D	11-0420-C-S	11-0420-C-D
TOC	63,600	68,400	44,600	53,300
TIC	2,580	577	895	323
	KW210-B1		KW210-B3	
TI ID >	TI002-B1-H	TI002-B1-J	TI001-B3-H	TI001-B3-J
RPL/ASO ID >	11-0421-C-S	11-0421-C-D	11-0422-C-S	11-0422-C-D
TOC	169,000	161,000	136,000	134,000
TIC	<250[(a)]	<560[(a)]	[290][(b)]	1,260

(a) Two of the three replicate TIC analysis results were <MDL and one result was <EQL; the reported less-than value provides a bounding assessment of the TIC concentration in the sample.

(b) One of the three replicate TIC analysis results was <MDL, one result was <EQL, and one result was >EQL; the brackets indicate the sample result has significant uncertainty.

Shading indicates RPD of the sample and duplicate exceeded ±20%.

Calculations of data presented above were performed using untruncated values in Table 3.1 to convert weighted average results in Table 4.15 and Table 4.16 from as-dried mass basis to a wet as-settled sludge mass basis. Calculation spreadsheets using Microsoft Excel are provided in data package DVD-52578-PNNL-20650 Rev. 2, Section III, September 2013.

Table 4.18. TOC and TIC QC Summary Table for the LCTS, Dry Sludge Mass Basis (ASR 8798)

	Purolite NRW37at 600°C (TOC)			Grafoil® at 1,000°C (TIC)		
	Sample size, g	Result, µgC/g	Recovery, %	Sample size, g	Result, µgC/g	Recovery, %
Before Series						
11-0423-C-S	0.0029	612,000	91.0	0.0046	1,060,000	106.9
11-0423-C-D	0.0053	636,000	94.7	0.0044	1,050,000	105.6
After Series						
11-0423-C-S	0.0044	726,000	108	0.0080	953,000	95.7
11-0423-C-D	0.0047	687,000	102	0.0044	981,000	98.6

4.7 Uranium Speciation Results

The sample digestion (RPG-CMC-107) for all core samples (ASR 8798) was completed on March 2, 2011, while the sample digestion for KW210-A4 re-analysis (ASR 8824) was completed on April 13, 2011. The PB samples were prepared with 0.6 g of Hanford sand from the Arid Land Ecology Reserve (ALE); LCS samples were prepared using the ALE sand with measured quantities of U_3O_8 powder and U metal beads. Reagent blank samples were also analyzed for U. Duplicate samples from each core were prepared for evaluation of method precision. A third core subsample was spiked with U metal and processed as the batch MS sample (the MS sample was supplemental to the QAPjP/SAP-defined requirements). Table 4.19 and Table 4.20 summarize the U metal and oxidation state results for each core sample.

Table 4.19. Uranium Metal in SCS-CON-210 Core Samples (ASR 8798 and 8824)

Sample Description	Sample ID	RPL/ASO ID	U in 7th rinse (µg/mL)	U Metal, As-Sampled (wt%)	U Metal Settled (wt%)[a]	U Metal Recovery (%)	RPD (%)
ASR 8798							
LCS	LCS	11-0419-107-LCS	0.405	---	---	102	---
Hanford Sand	PB 2	11-0419-107-B2	0.023	<0.0006	---	---	---
KW210-A1	TI001-A1-P	11-0419-107-S	0.362	0.0685	0.0537	---	NA
	TI001-A1-Q	11-0419-107-D	0.546	0.116	0.0910	---	
KW210-A4	TI002-A4-P	11-0420-107-S	0.814	1.19	1.07	---	54
	TI002-A4-Q	11-0420-107-D	0.583	0.685	0.619	---	
KW210-B1	TI002-B1-P	11-0421-107-S	0.080	<0.011	<0.0086	---	NA
	TI002-B1-Q	11-0421-107-D	0.186	0.0191	0.0149	---	
Matrix spike	TI002-B1-R	11-0421-107-MS	0.092	0.0102[b]	---	99	
KW210-B3	TI001-B3-P	11-0422-107-S	0.519	0.0176	0.0133	---	NA
	TI001-B3-Q	11-0422-107-D	0.335	0.0130	0.0098	---	
ASR 8824							
LCS	LCS	11-0792-107-LCS	0.146	---	---	103	---
Hanford Sand	PB 2	11-0792-107-B2	<0.012	0.00065	---	---	---
KW210-A4	TI002-A4-R	11-0967-107-S	0.475	0.825	0.745	---	4
	TI002-A4-S	11-0967-107-D	0.937	0.795	0.718	---	

(a) The conversion factors ($g_{sampled}/g_{settled}$) are provided in Table 3.1.

(b) The MS sample from ASR 8798 was taken from the B1 core (TI002-B1-R). The U metal concentration of MS sample before the U metal spike was added is back-calculated to be 0.0102 wt% on an as-sampled mass basis. This is lower than the method detection limit of 0.011 wt%.

Shaded blocks indicate that the RPD of the duplicate pair exceeded ±20%.

NA: Not Applicable. The RPD calculation of duplicates is not performed when uranium concentration is <0.1 wt% of the as-sampled sludge.

Analytical results from ASR 8798 are found in Appendix B and the results for ASR 8824 are found in Appendix C.

Table 4.20. Uranium Oxidation State Measurements in SCS-CON-210 Core Samples (ASR 8798 and 8824)

Sample Description	Sample ID	RPL/ASO ID	U(IV) (mol%)	U(VI) (mol%)	RPD U(IV) (%)	RPD U(VI) (%)	Quality Checks
ASR 8798							
LCS	LCS	11-0419-107-LCS	33.7	66.3	---	---	Pass[a]
KW210-A1	TI001-A1-P	11-0419-107-S	88.4	11.6	1.7	14	Pass[b]
	TI001-A1-Q	11-0419-107-D	90.0	10.0	---	---	
KW210-A4	TI002-A4-P	11-0420-107-S	>94.1	<5.9[c]	---	---	NA
	TI002-A4-Q	11-0420-107-D	>91.2	<8.8[c]	---	---	
KW210-B1	TI002-B1-P	11-0421-107-S	>87.4	<12.6[c]	---	---	
	TI002-B1-Q	11-0421-107-D	>88.4	<11.6[c]	---	---	NA
Matrix spike	TI002-B1-R	11-0421-107-MS	>85.9	<14.1[c]	---	---	
KW210-B3	TI001-B3-P	11-0422-107-S	>89.5	<10.5[c]	---	---	NA
	TI001-B3-Q	11-0422-107-D	>90.5	<9.5[c]	---	---	
ASR 8824							
LCS	LCS	11-0792-107-LCS	34.2	65.8	---	---	Pass[a]
KW210-A4	TI002-A4-R	11-0967-107-S	>91.8	<8.2	---	---	NA
	TI002-A4-S	11-0967-107-D	>92.1	<7.9	---	---	

(a) The LCS requirement per procedure is ±5% (absolute) of the nominal from the standard to pass. The standard value for U(IV) is 33.3%, allowing for the LCS to fall between 28.3% and 38.3%. The standard value for U(VI) is 66.7%, allowing the LCS to fall between 61.7% and 71.7%.

(b) The QAPjP/SAP requires that the sample and all duplicates shall agree within ±5% of the average when the U concentration of each oxidation state is greater than 0.001M.

(c) The U(VI) concentration measured by spectrophotometry were <0.001 M.

The U oxidation state ratios for K Basin sludge samples should be used with caution. Ferrous iron (Fe[II]) present as magnetite has been observed in K Basin sludge, and has been shown to reduce U(VI) to U(IV) in Na_2SO_4/H_3PO_4 solutions. The U(VI) can be considered a lower bounding case and the U(IV) fraction the upper bounding case.

NA: Not Applicable. The RPD calculation of duplicate samples is not performed when the U concentration of one oxidation state is below detection.

Analytical results from ASR 8798 are found in Appendix B and the results for ASR 8824 are found in Appendix C.

Table 4.19 contains the U metal concentration results and QC sample results for assessment of precision (RPD) and accuracy (LCS, MS sample, PB samples). Because the A4 core duplicate pair sample results varied significantly (RPD = 54%), two reserve A4 core subsamples were processed for U metal according to ASR 8824. In the second analysis, the precision met acceptance criterion (4% RPD) and the duplicate results were between the results of the duplicate pair of the initial analyses. The uncertainty for U metal concentrations >0.03 wt% is estimated to be ±25% (2-σ) or less.

The U measured in the solids 7th rinse solution (before nitric acid dissolution of U metal) ranged from 0.08 to 0.81 µg/mL, well below the 100-µg/mL acceptance threshold, indicating the measured U metal was not confounded by carryover of non-metallic U. The PBs contained no or minute amounts of U (≤0.00065 wt%) indicating that results were not confounded by contamination during processing. The U metal LCS for both batches recovered 102 and 103%, respectively. The U metal MS sample was not

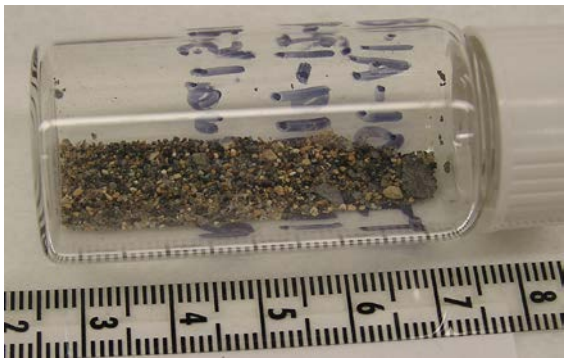
required by the QAPjP/SAP and was processed for additional information; sample TI002-B1-R (ASR 8798) was spiked with a U metal bead that recovered 99%. The quantity of U metal in the sample before the U metal bead was added was back-calculated / estimated by subtracting the quantity of the known U metal spike. Its result was between the results of the duplicate pair but below the 0.011 wt% MDL.

The A4 core samples contained ~4.2 to 4.7 wt% total U on a settled mass basis (ICP-OES, Table 4.13). With the A4 core showing an average of 0.79 wt% U metal on a settled mass basis (Table 4.19) as determined by results from all four samples, ~18% of the total U exists as U metal (0.79 wt%/4.5 wt%).

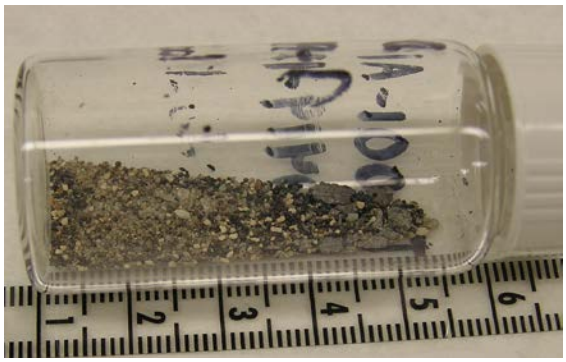
Table 4.20 summarizes the U oxidation state measurements performed along with the QC samples. The Hanford sand blanks and the reagent blanks from both batches showed the U(IV) and U(VI) concentrations to be less than 0.001M. The LCS measurements were also within ±5% of the theoretical oxidation state ratio for U_3O_8 (33.3% U[IV], 66.7% U[VI]), and therefore within the ±25% allowed deviation in the QAPjP/SAP. All the core samples were found to be overwhelmingly (>86%) in the U(IV) oxidation state. This is supported by the XRD measurements of the −500-μm sieve fraction where crystalline U phases were present as UO_2 and U_4O_9 (see Section 5.4). However, these U(IV) and U(VI) oxidation state ratios should be used with caution. Ferrous iron (Fe[II]), present as magnetite, has been observed in K Basin sludge (Fiskum et al. 2011b), and has been shown to reduce U(VI) to U(IV) in Na_2SO_4/H_3PO_4 solutions (Fiskum et al. 2011a). While the amount of Fe(II) is not known, the concentration of Fe in sludge samples is significant enough (0.9 to 5 wt% on a settled sludge mass basis) to cause concern (the possibility of the presence of magnetite is indicated by the qualitative observations of magnetic material in the subject samples as shown in Figure 4.5). The U(VI) can be considered to be the lower bounding case and the U(IV) fraction the upper bounding case.

4.8 Undissolved Solids

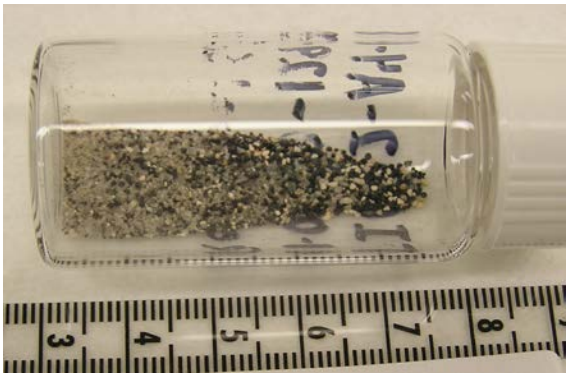
As discussed in Section 4.3, the UDS (or residual solids) fractions of the analytical core samples following the acid digestion (PNL-ALO-129) were well over 5 wt% (wet settled mass basis). Per the QAPjP/SAP, UDS >5 wt% require GEA and XRD analysis. Figure 4.12 and Figure 4.13 show photographs of the residual solids after being removed from the hot cells. The material contains a variety of particle types: resin beads, sand, and gray platy pieces. The acid digestion procedure (PNL-ALO-129) is not expected to dissolve OIER or sand (quartz).



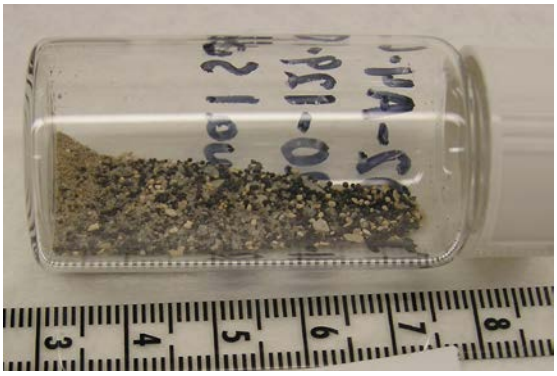
TI001-A1-B



TI001-A1-C



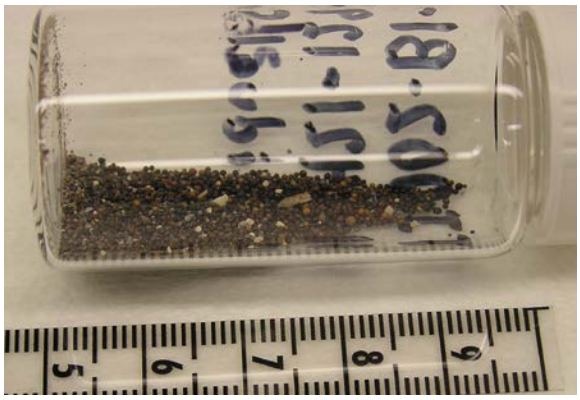
TI002-A4-B



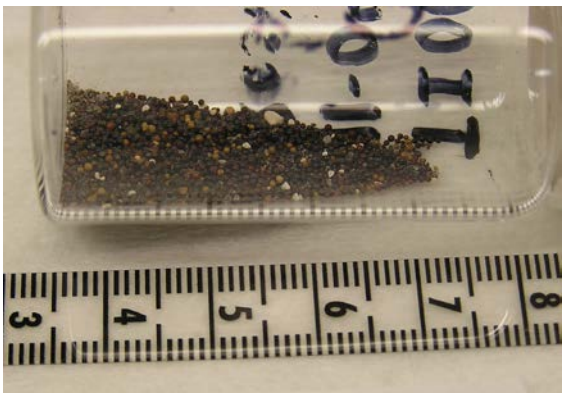
TI002-A4-C

Note: The ruler scale is a centimeter scale with millimeter subdivisions.

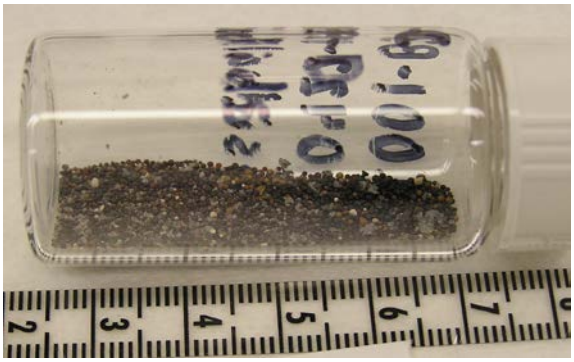
Figure 4.12. Photographs of UDS from the KW210-A Core Subsamples (Core A1 on top; Core A4 on bottom)



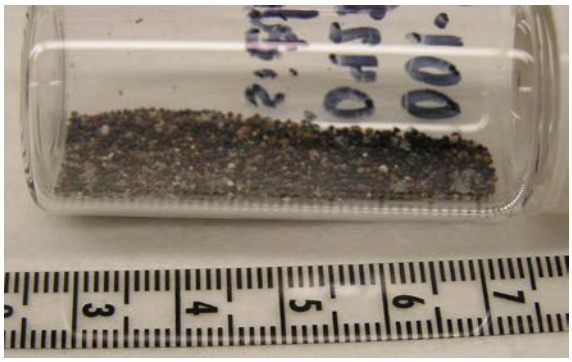
TI002-B1-B



TI002-B1-C



TI001-B3-B



TI001-B3-C

Note: The ruler scale is a centimeter scale with millimeter subdivisions.

Figure 4.13. Photographs of UDS from the KW210-B Core Subsamples (Core B1 on top; Core B3 on bottom)

The UDS were measured directly by GEA according to ASR 8798 and Special Instructions to assess fractionation of ^{137}Cs, ^{154}Eu, and ^{241}Am between digestate and solids. Table 4.21 shows the analyte concentrations. The UDS ^{134}Cs, ^{152}Eu, and ^{155}Eu concentrations were near or below the MDL and were not further evaluated. Table 4.21 also shows the fractionation of detected analytes between the aqueous digestates and UDS. Analyte fractionation to the UDS was calculated according to Equation 4.1.

$$X_{UDS} = \frac{(C_{UDS} \times g_{UDS})}{[(C_{UDS} \times g_{UDS}) + (C_S \times g_S)]} \quad (4.1)$$

where X_{UDS} = fraction of analyte not dissolved from acid digestion
C_s = concentration of analyte in the acid digestate
C_{UDS} = concentration of analyte in UDS fraction (post-acid digestion)
g_{UDS} = mass of UDS (post-acid digestion)
g_S = mass of dry solids in aliquot before digestion (pre-digest dry mass).

Table 4.21. GEA Analysis of UDS and Analyte Fractionation to the UDS (ASR 8798)

Container:	SCS-CON-210								
Core:	A1		A4		B1			B3	
Sample ID:	TI001-A1-B	TI001-A1-C	TI002-A4-B	TI002-A4-C	TI002-B1-B	TI002-B1-C	TI002-B1-D[(a)]	TI001-B3-B	TI001-B3-C
RPL/ASO ID >	11-0419-129-S	11-0419-129-D	11-0420-129-S	11-0420-129-D	11-0421-129-S	11-0421-129-D	11-0421-129-MS	11-0422-129-S	11-0422-129-D
UDS Mass, g	0.7262	0.8140	1.0004	1.1212	0.6192	0.7206	0.7621	0.9163	0.9157
As-Measured μCi/g in UDS, Reference Date: January 11, 2011									
^{60}Co	4.51E-4	5.29E-4	1.93E-4	3.76E-4	4.51E-4	7.18E-4	1.04E-3	4.09E-4	4.42E-4
^{125}Sb	2.52E-3	1.94E-3	<8.E-4	1.53E-3	4.04E-3	4.69E-3	3.01E-3	3.41E-3	3.22E-3
^{126}Sn	8.14E-5	<3.E-4	<2.E-4	3.25E-5	4.96E-4	6.00E-4	<2.E-4	3.73E-4	3.98E-4
^{134}Cs	1.23E-4	<1.E-4	<2.E-5	<5.E-5	<5.E-5	<3.E-5	<9.E-5	<2.E-5	<2.E-5
^{137}Cs	2.05E+0	2.18E+0	1.59E+0	2.77E+0	8.67E-1	8.03E-1	2.23E+0	6.91E-1	5.73E-1
^{152}Eu	<1.E-4	<1.E-4	<5.E-5	<9.E-5	<2.E-4	<7.E-5	<3.E-4	<6.E-5	7.97E-5
^{154}Eu	2.43E-3	1.75E-3	2.22E-3	2.07E-3	2.30E-3	3.24E-3	3.65E-3	3.70E-3	3.39E-3
^{155}Eu	<1.E-3	<9.E-4	<5.E-4	7.79E-4	<5E-4	3.21E-4	<3.E-3	5.87E-4	6.80E-4
^{241}Am	6.62E-2	4.80E-2	6.98E-2	5.37E-2	8.72E-2	1.31E-1	1.48E-1	1.22E-1	1.14E-1
Fractionation, Relative Amount of Analyte Remaining in the UDS									
^{60}Co	0.13%	0.24%	0.10%	0.22%	0.51%	0.86%	1.32%	0.51%	0.60%
^{137}Cs	0.36%	0.35%	0.33%	0.51%	0.50%	0.42%	1.27%	0.52%	0.49%
^{154}Eu	0.21%	0.15%	0.27%	0.27%	0.40%	0.55%	0.66%	0.60%	0.57%
^{241}Am	0.22%	0.14%	0.30%	0.27%	0.51%	0.68%	0.92%	0.71%	0.71%

(a) Sample TI002-B1-D was prepared as a MS for ICP-OES analysis only; the GEA results are provided for additional information.

Calculation spreadsheets using Microsoft Excel are provided in data package DVD-52578-PNNL-20650 Rev. 2, Section III, September 2013. Analytical results and the 1-σ uncertainty from ASR 8798 are found in Appendix B.

Overall, the GEA measurements showed that the UDS in the sample and duplicate sample contained less than 1% of the total activity when compared to the dissolved fraction of the parent samples. Each analyte (^{60}Co, ^{137}Cs, ^{154}Eu, and ^{241}Am) averaged 0.4 % fractionation to the UDS. The ICP-OES MS sample (TI002-B1-D) results are shown for additional information; the ^{60}Co and ^{137}Cs analyte fractionation to the UDS slightly exceeded 1% in this sample.

Figure 4.14 compares the GEA-measured analyte fractionation (Table 4.21) in the UDS. The chart demonstrates that the initial analytical digestion succeeded in dissolving the significant fraction of the ^{137}Cs, ^{154}Eu, and ^{241}Am and that slightly higher fractions of ^{154}Eu and ^{241}Am were retained in the "B" cores UDS samples relative to the "A" core UDS samples. The slightly higher fractions of ^{137}Cs and ^{241}Am in sample TI002-B1-D may result from slight differences in the acid digestate chemistry associated with the addition of the ICP-OES MS analytes. The altered acid digestate matrix may have slightly altered the OIER affinity for specific (cationic and anionic) analytes. This behavior is discussed in more detail for the remainder of this section.

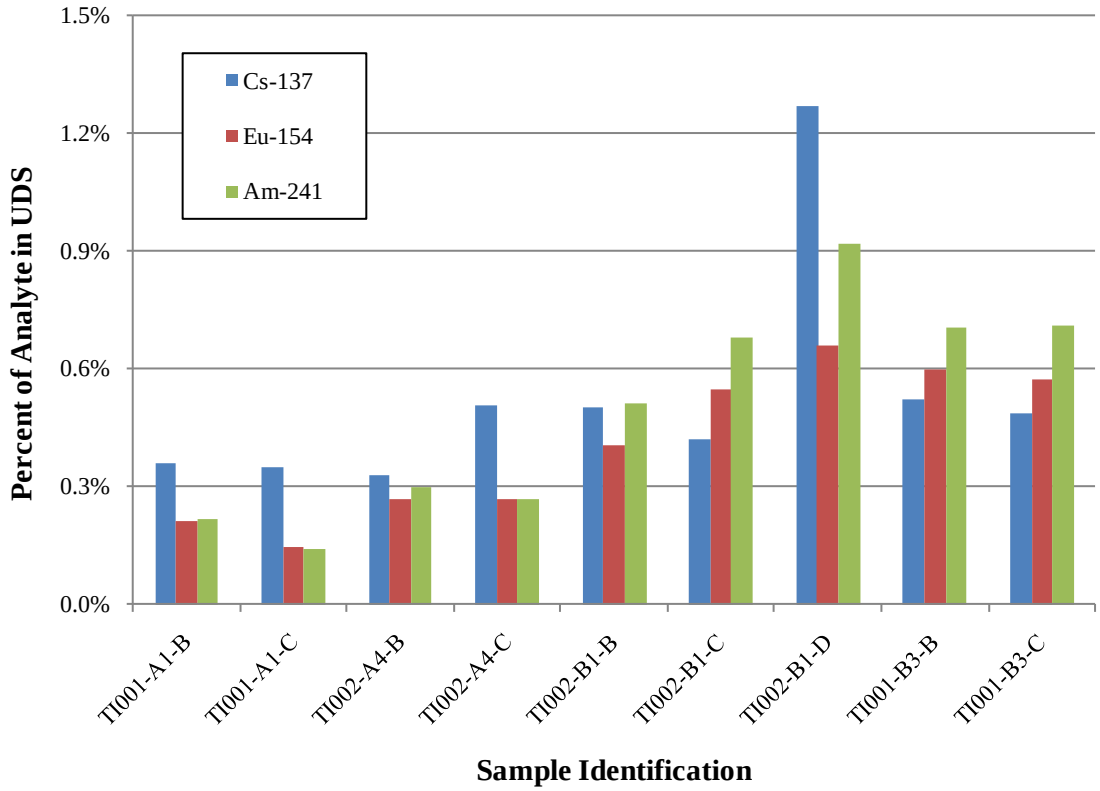


Figure 4.14. Percent of Analyte Remaining in the UDS Measured by GEA Following Acid Digestion for SCS-CON-210 Reconstituted Core Samples

In addition to XRD analysis, the BTR directed fusion dissolution of the UDS followed by ICP-OES, gross alpha, and AEA screen. To prepare for these analyses, the UDS were subsampled using the Y-splitter in a cascade (see Figure 2.12) to create small subsamples appropriate for fusion and samples appropriate for XRD.

The sample splits collected for XRD were phase-separated by panning off the rolling resin beads into a vial separate from the non-rolling/non-resin material. The rolling particles were the spherical-shaped

OIER. The intention of removing the OIER was two-fold: reduce the confounding effects of an amorphous background in the powder diffraction pattern and obtain mass fraction data from the resin and non-resin components splits. Both the OIER and the non-OIER fractions were weighed. The split was not perfect; some cross-contamination between material types was observed. However general trends in the approximate mass distribution were recorded. The approximate OIER mass fractions in the UDS samples are provided in Table 4.22. The calculated OIER mass fraction in the dry sample (before acid digestion) was calculated according to Equation 4.2 and is also provided in Table 4.22. This calculation approach assumes no resin mass was lost during the acid digestion processing.

$$\frac{M_{UDS} \times \left[\frac{m_{OIER}}{m_{UDS}} \right]}{M_S} = SF_{OIER} \quad (4.2)$$

Where M_{UDS} = Mass of UDS in the analytical sample
m_{OIER} = Mass of OIER in the fractionated subsample
m_{UDS} = Mass of UDS in the fractionated subsample (OIER plus non-OIER)
M_S = Mass of dry parent sample before acid digestion
SF_{OIER} = Mass fraction of the OIER in the dry parent sample before acid digestion

Table 4.22. Dry Mass Fraction of OIER in the Analytical Core Samples

Core Sample	RPL/ASO ID	OIER in UDS[(a)] wt %	OIER in UDS[(a)] RPD	Calculated OIER in Dry Parent Sample[(a)] wt %	Calculated OIER in Dry Parent Sample[(a)] RPD	Calculated OIER Based on Measured TOC RPL/ASO ID	Calculated OIER Based on Measured TOC wt%
A1	11-419-129-S	33.7		22		11-0419-C-S	21
	11-419-129-D	31.8	5.9	20	7.4	11-0419-C-D	23
A4	11-420-129-S	26.3		18		11-0420-C-S	12
	11-420-129-D	24.7	6.0	17	6.1	11-0420-C-D	14
B1	11-421-129-S	87.1		72		11-0421-C-S	76
	11-421-129-D	84.9	2.5	75	3.3	11-0421-C-D	73
B3	11-422-129-S	56.4		49		11-0422-C-S	56
	11-422-129-D	57.0	1.0	50	2.0	11-0422-C-D	55

(a) Mass fractions should be used with caution because the fractionation of OIER from non-OIER was not perfect. See text for discussion.

The OIER mass fraction results were compared to the TOC concentrations measured using furnace combustion. The weight percent of OIER was calculated from the TOC results according to Equation 4.3:

$$\frac{g_{TOC}}{g_{sample}} \times \frac{g_{Purolite}}{0.672\, g_{TOC}} = \text{wt\% OIER} \quad (4.3)$$

where: g_{TOC}/g_{sample} = ASO-reported TOC concentration
$g_{Purolite}/0.672\ g_{TOC}$ = Reciprocal of the TOC content in Purolite NRW37 ion exchange resin

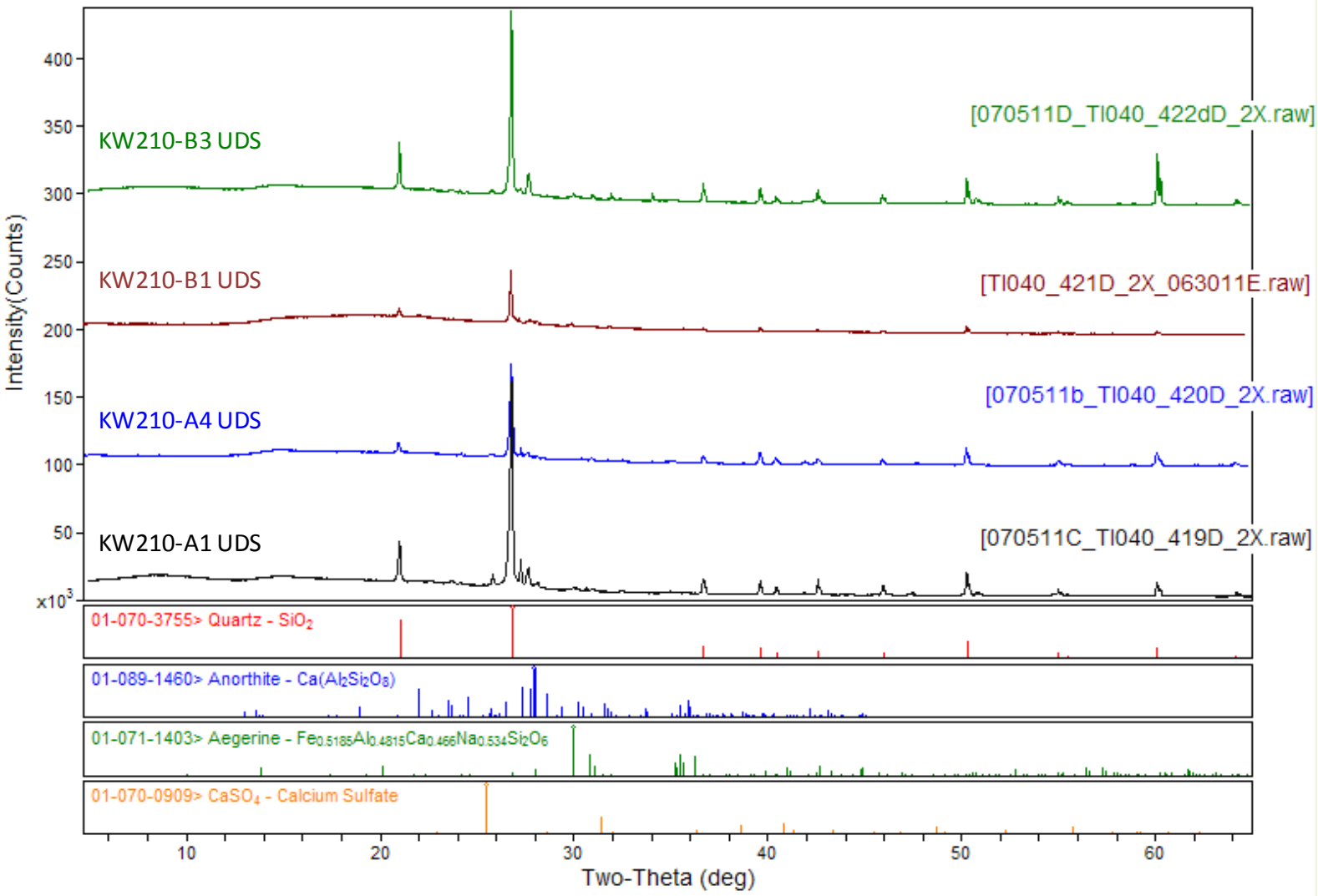
The calculation assumes all TOC is from OIER and consistent with the Purolite NRW37 carbon content of 67.2 wt% (Fiskum et al. 2011a). The result summary is provided in Table 4.22. Generally the wt% OIER measured from separation of the resin from the non-resin material agreed well with the wt% OIER calculated from TOC. The sample sizes for the direct mass measures ranged from 0.23 to 0.53 g and were much greater than the sample sizes for TOC, which ranged from 0.0035 to 0.065 g.

One non-OIER solid sample from each core was submitted for XRD analysis. Figure 4.15 presents a summary of the powder diffractions for all samples along with the stick-figure standard crystalline phases. Table 4.23 summarizes the crystalline phases identified in the UDS along with the sample progenitor IDs, ICDD card numbers, and relative intensities. The UDS samples primarily consisted of quartz and anorthite as major crystalline phases. Some of these samples also contained as minor phases aegerine and calcium sulfate. No other crystalline components were identified; all reflections were identified. The UDS did not contain oxyhydroxides, hydrates and hydroxides, aluminum oxides, oxyhydroxides and hydrates, or zirconium metal and zirconia (from cladding materials) as crystalline phases.

The UDS subsample splits (containing OIER) from all eight core subsamples were further split into small representative aliquots applicable for KOH/KNO_3 fusion (0.1 to 0.2 g). After fusion the digestates were split. One split was analyzed by ICP-OES for AOIs Al, Ca, Fe, S, Si, U, and Zr; the other split was analyzed for gross alpha and AEA screen for $^{239+240}Pu$ measurement. The fusion and analyses were conducted according to ASR 8878 and Special Instructions (Appendix D).[(a)] This analysis was supplemental scope to that defined in the QAPjP/SAP. All process QC samples met the ASR-delineated requirements. The LCS was prepared using NIST SRM 2710 Montana Soil and recovery values ranged from 95 to 107% for Al, Ca, Fe, and Si. The core sample/sample duplicate analyte results were within 18% RPD, and analytes in the PB were <MDL. See Appendix D for details and measurement uncertainties.

Table 4.24 provides the AOI results and their relative mass and activity fractionations to the UDS (results of ICP-OES opportunistic analytes and gross alpha can be found in Appendix D). Analyte fractionation to the UDS was calculated according to Equation 4.1 where C_{UDS} was determined from the analysis of the fused sample. The pre-digest dry mass (g_S) and UDS mass (g_{UDS}) values are reported in Table 4.2 and the concentrations of analytes in the acid digestate (C_S) are reported in Table 4.10. Figure 4.16 compares Fe, U, and Pu fractionations in the UDS for each core sample. The chart vividly shows the large step change in retention for these analytes in the “B” core sample UDS, which also had the highest calculated dry wt% OIER as shown in Table 4.22.

(a) The UDS associated with the container composite −2,000-µm /+500-µm and −500-µm sieve fraction samples were processed as part of ASR 8878. Core sample 11-0420-129-D and container composite SCC sample 11-793-129-D were split and run as batch duplicates. All process QC samples were shared between the core and container composite samples.



Note: Patterns were not background-subtracted.

Figure 4.15. Raw X-Ray Powder Diffraction Patterns for the KW210 UDS Samples

Table 4.23. Summary of Phases Found via X-Ray Powder Diffraction on UDS Following Acid Digestion and Bulk OIER Removal from SCS-CON-210 Core Samples

XRD Sample ID	*RPL/ASO Parent ID* (TI Parent ID, Core ID)	ICDD Card Number	Relative Intensity[(a)]	Formula	Common Phase Name
TI040-419D-2x	*11-0419-129-D* (TI001-A1-C, KW210-A1)	01-070-3755	Major	SiO_2	Quartz
		01-089-1460	Major	$Ca(Al_2Si_2O_8)$	Anorthite
TI040-420D-2x	*11-0420-129-D* (TI002-A4-C, KW210-A4)	01-070-3755	Major	SiO_2	Quartz
		01-089-1460	Major	$Ca(Al_2Si_2O_8)$	Anorthite
		01-071-1403	Minor	$Ca_{0.466}Fe_{0.5185}Al_{0.815}Na_{0.534}O_6Si_2$	Aegerine
		01-070-0909	Minor	$CaSO_4$	Calcium sulfate
TI040-421D-2x	*11-0421-129-D* (TI002-B1-C, KW210-B1)	01-070-3755	Major	SiO_2	Quartz
		01-089-1460	Major	$Ca(Al_2Si_2O_8)$	Anorthite
TI040-422D-2x	*11-0422-129-D* (TI001-B3-C, KW210-B3)	01-070-3755	Major	SiO_2	Quartz
		01-089-1460	Major	$Ca(Al_2Si_2O_8)$	Anorthite
		01-071-1403	Minor	$Ca_{0.466}Fe_{0.5185}Al_{0.815}Na_{0.534}O_6Si_2$	Aegerine

(a) Crystalline components above 10 wt% are classified as major phases, between 1-10 wt% as minor phases, and under 1 wt% as possible phases.

Table 4.24. Analytes in UDS Fusion, Dry UDS Mass Basis (ASR 8878)

Container:		SCS-CON-210								
Core:		A1		A4			B1		B3	
Parent Sample ID:		TI001-A1-B	TI001-A1-C	TI002-A4-B	TI002-A4-C		TI002-B1-B	TI002-B1-C	TI001-B3-B	TI001-B3-C
Parent Sample ASO/RPL ID:		11-0419-129-S	11-0419-129-D	11-0420-129-S	11-0420-129-D		11-0421-129-S	11-0421-129-D	11-0422-129-S	11-0422-129-D
Daughter Sample ID:		TI040-0419S-1b	TI040-0419D-2b	TI040-0420S-2c	TI040-0420D-1c	TI040-0420D-2c	TI040-0421S-2b	TI040-0421D-1b	TI040-0422S-1c	TI040-0422D-1c
RPL/ASO ID >	BLK-1279	11-1279-115-S	11-1280-115-S	11-1281-115-S	11-1282-115-S	11-1282-115-D	11-1283-115-S	11-1284-115-S	11-1285-115-S	11-1286-115-S
Analyte		(µg/g) or (µCi/g)								
Al	<1,400	20,100	16,400	22,200	[18,000]	21,800	[2,800]	[1,900]	[8,300]	[9,200]
Ca	<1,500	[3,400]	[2,100]	[2,700]	[3,300]	[2,900]	<1,400	<1,300	<1,800	<1,600
Fe	<570	16,500	16,000	7,080	11,500	9,650	33,700	34,000	27,100	22,800
S	<480	16,400	13,900	16,700	15,900	16,900	25,800	14,300	19,000	14,800
Si	<1,600	276,000	268,000	350,000	316,000	355,000	38,200	22,100	127,000	161,000
U	<31	7,840	7,370	1,920	3,310	2,970	11,000	16,000	15,200	12,800
Zr	<38	694	579	623	847	<49	<35	<32	<44	<40
$^{239+240}$Pu	6.83E-5	4.77E-1	6.82E-1	1.70E-1	2.79E-1	2.30E-1	3.36E+0	3.41E+0	2.97E+0	2.53E+0
Relative Amount of Analyte in the UDS										
Al	--	29%	24%	43%	[36%]	41%	[13%]	[10%]	[37%]	[40%]
Ca	--	[22%]	[17%]	[22%]	[23%]	[21%]	NA	NA	NA	NA
Fe	--	11%	10%	5%	8%	7%	44%	52%	42%	42%
S	--	60%	59%	66%	61%	62%	69%	58%	63%	59%
Si	--	NA	NA	NA	NA	NA	NA	NA	NA	NA
U	--	5%	4%	2%	3%	3%	15%	24%	19%	18%
Zr	--	57%	50%	54%	80%	NA	NA	NA	NA	NA
$^{239+240}$Pu	--	1.7%	2.2%	0.71%	1.3%	1.1%	19%	19%	16%	15%

Analyte results shown in brackets indicate that the analyte concentrations were greater than the MDL but less than the EQL. Less-than values represent the instrument detection limit multiplied by the process factors. See Appendix D for measurement uncertainties.

Percent values in brackets indicate one of the input parameters was <EQL but >MDL.

NA: Not Applicable; an input factor was <MDL or, in the case of Si, the analyte was not expected to dissolve in the PNL-ALO-129 acid digestion.

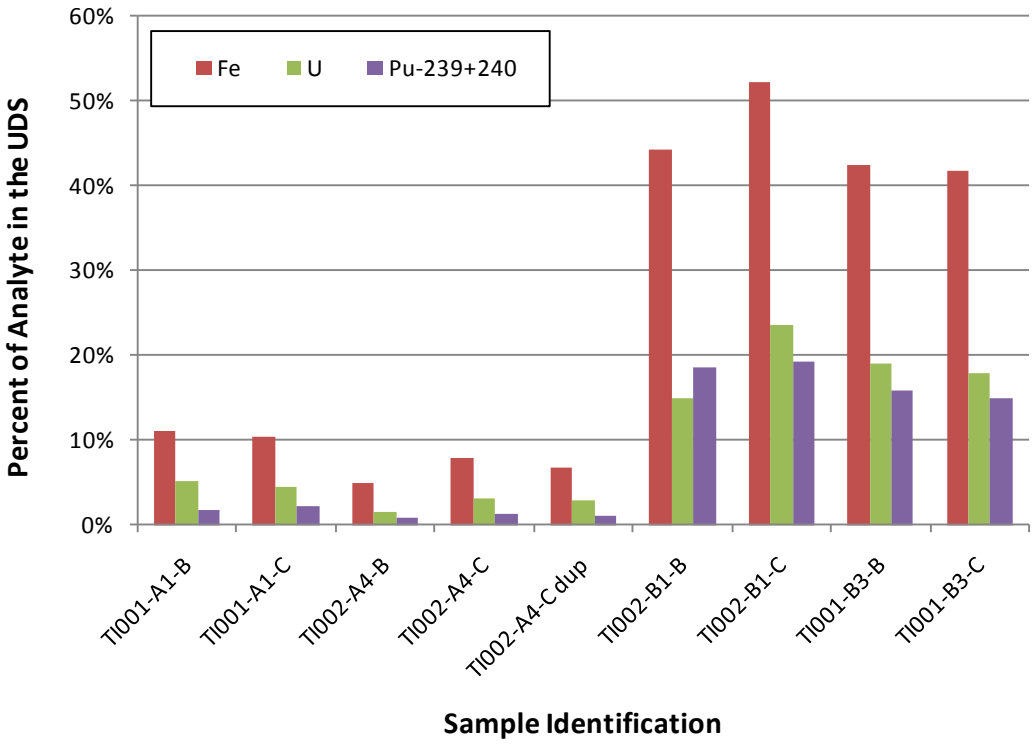
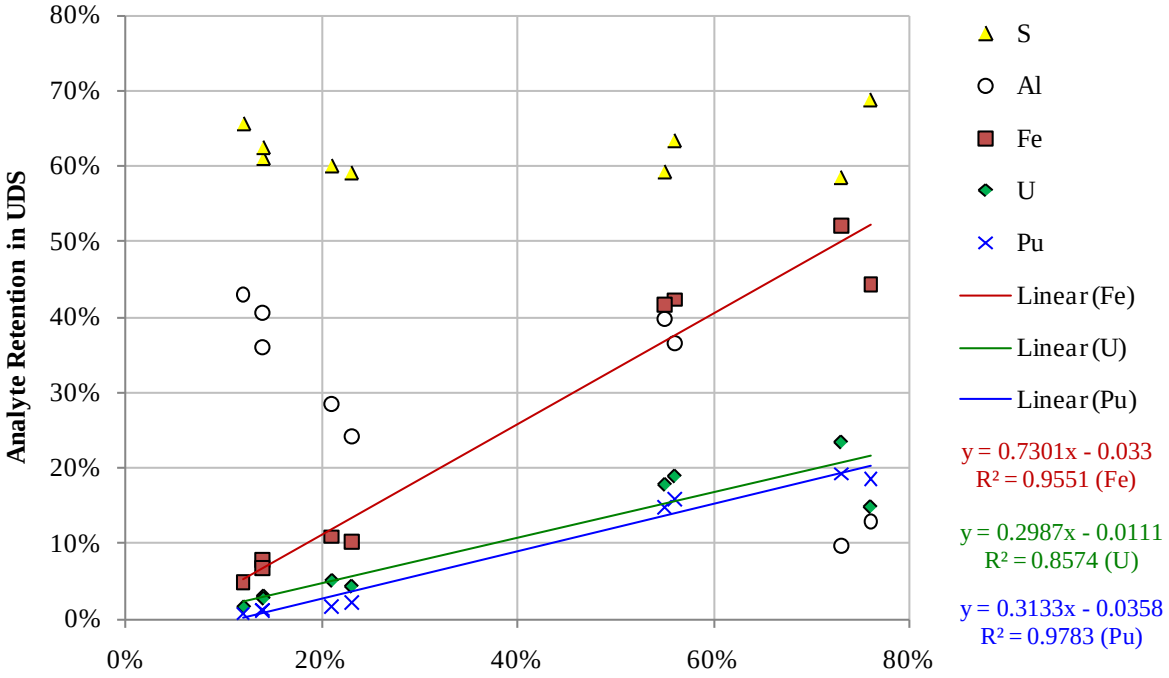


Figure 4.16. Percent of Analyte Remaining in the UDS Measured by ICP-OES and AEA Screen in the SCS-CON-210 Reconstituted Core Samples

Figure 4.17 graphically presents the Al, Fe, S, U and Pu retention in the UDS as a function of the OIER content in the dry parent sample (before subjection to PNL-ALO-129 acid digestion, see Table 4.22). The UDS contained ~50 to 60% of the total S content; no partitioning trend was observed as a function of OIER fraction in the sample. The S is postulated to come from the sulfonic acid functional group in the cation resin where only a percentage of the S was amendable to acid dissolution. Aluminum did not show a relationship to the OIER content; it appears to be a "spectator" species with respect to OIER content and exchange. The UDS aluminum is probably associated with anorthite and aegerine (as identified by XRD, see Table 4.23). Iron is present in aegerine, which was found as a minor crystalline phase in A4 and B3 core samples. However, a direct relationship appears to exist between Fe in the UDS and OIER content. Uranium phases in the UDS were not identified by XRD; however, up to 24% of the U content was found in the “B” core sample UDS. The Pu was similarly higher in the “B” core sample UDS, which contained up to 19% of the Pu content.

The direct relationship between the OIER content in the parent sample material to Fe, U, and Pu retention in the UDS suggests that analyte anionic or cationic species exchanged onto the OIER during the PNL-ALO-129 acid digestion and/or solids rinse steps. Similarly, analytes exchanging onto OIER were reported previously with increases in Fe and $^{239+240}$Pu concentrations by factors of 17 and 290, respectively, when a K Basin OIER (NRW-37) was exposed to a 6 M HNO_3 dissolver solution at 95°C for 7 hr (Schmidt et al 1999). In this same 1999 study, the U concentration increased by only a factor of 1.07. In contrast, the present work (where 8 M HNO_3 combined with equal volume of 8 M HCl was used) U retention in UDS tracked well with the $^{239+240}$Pu retention and Fe retention was greater than Pu retention (see Figure 4.17). Acid type and concentration are known to affect the affinity of specific (cationic and anionic) analytes onto OIER (Coleman 1965). The fraction of Fe, U, and $^{239+240}$Pu in the

OIER presented in Table 4.24 likely does not represent the analyte fractions in the sludge OIER currently contained in SCS-CON-210 because these analytes were not dissolved *in-situ* and the contacting medium is essentially pH 7 water (not acidic). Additional analyses of undigested OIER would be required to confirm this assessment.



- *Pu, U, and Fe increased in the UDS as OIER content increased in the parent analytical sample.*
- *Al and S in the UDS showed no relationship to increasing OIER content.*
- *Linear trend lines are provided for illustrative purposes and do not attempt to provide a model fit.*

Figure 4.17. Relationship of Al, Fe, S, U, and Pu to OIER in the Parent Analytical Subsample

The relative mass fractions of Pu, U, and Fe in the UDS versus the acid digestate were compared. This required that the Pu, U, and Fe concentrations in the UDS be re-normalized to the initial dry mass basis of each core sample and duplicate. The re-normalization was conducted according to Equation 4.4

$$Ci = Ci_{UDS} \times \left(\frac{g_{residual}}{g_{dry}} \right) \quad (4.4)$$

where Ci = Concentration of UDS analyte *i* in the original dry analytical sample ($\mu Ci/g_{dry}$)

Ci_{UDS} = Concentration of analyte *i* in the UDS ($\mu Ci/g_{UDS}$)

$\frac{g_{residual}}{g_{dry}}$ = UDS weight fraction, see Table 4.2.

Figure 4.18 compares the core sample Pu, U, and Fe total analyte concentrations in each sample (combined red and blue bars) relative to the analyte concentration that dissolved in the PNL-ALO-129 acid digestion (blue bars) and the normalized analyte concentration that was associated with the UDS (red bars). The concentration is presented on a dry mass basis. The total Pu concentrations did not differ greatly between core samples. The total U and Fe concentrations were higher in the "A" cores than the "B" cores.

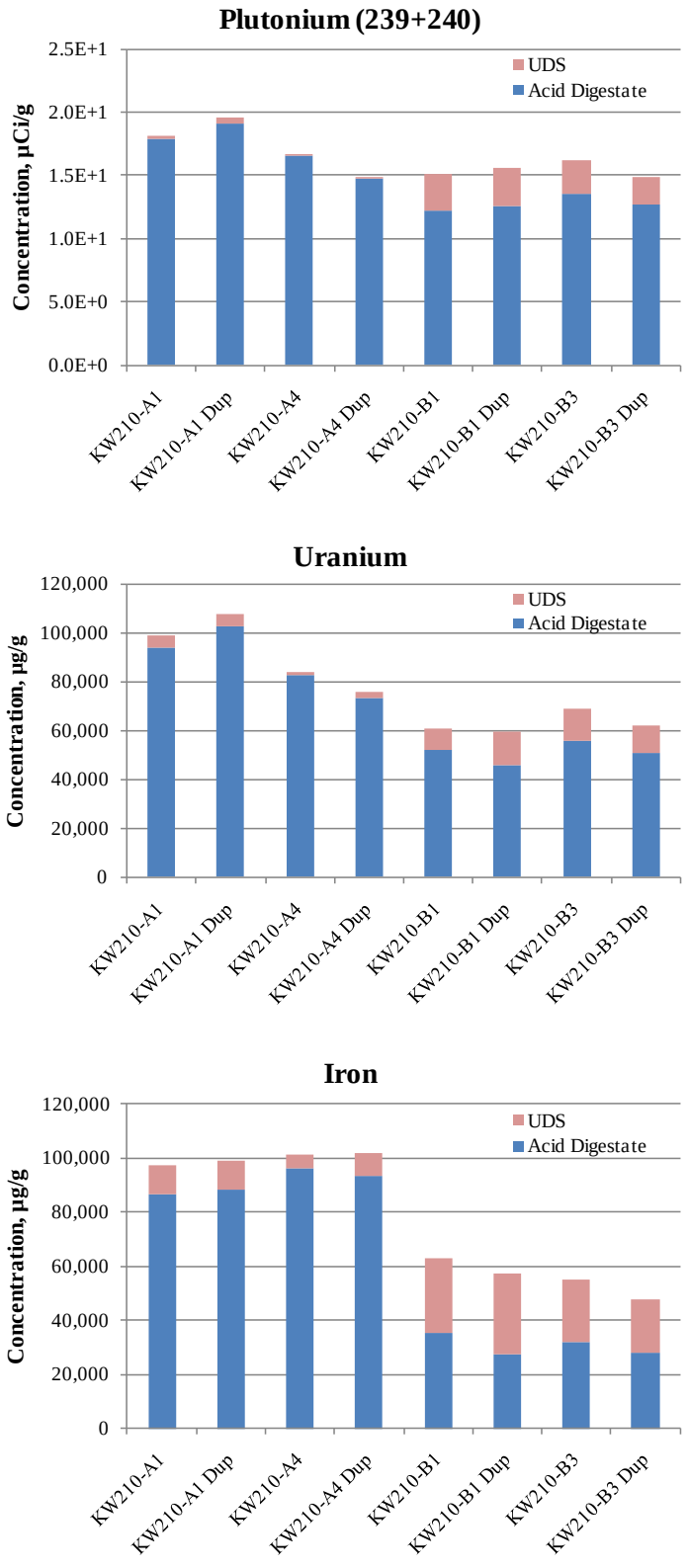


Figure 4.18. Total $^{239+240}$Pu, U, and Fe Concentrations and Partitions Between Acid Digestate and UDS Phases for Each Core Subsample

5.0 SCS-CON-210 Container Composite Samples

The four reconstituted core samples from Engineered Container SCS-CON-210 were subsampled to support the creation of two independent container composite samples of similar composition. After measuring gross physical properties, the container composite samples were subsampled to collect primary physical property samples. One of the container composite samples was then wet-sieved into three size fractions. Each sieve fraction was characterized to the extent possible, given limitations on sample size. The other container composite was used to measure shear strength. This section details the processing and characterization results from each container composite and sieve fraction. Refer to Section 2.2 for a detailed discussion of the container composite preparation processes.

5.1 Container Composite Preparations and Observations

The goal defined in the QAPjP/SAP (KBC-33786, Rev. 2) was to combine equal volumes (within ±10% per TI direction) of settled sludge from each core sample to prepare the SCS-CON-210 container composites. The settled sludge volume after a 24-hr settling time was used as the comparative volume basis. (It was noted that additional sludge settling occurred after subsamples were transferred to the SAL with three to four weeks of additional settling time. The volume compaction associated with sample movement and extended settling time ranged from ~6 to 22%.)

Table 5.1 and Table 5.2 summarize the core sample masses (as-sampled) and settled sludge volumes used to create the two container composites. The relative fractions of each core sample in the container composites on both a settled sludge volume basis and a dried sludge mass basis are also provided. After combination of the component core subsamples, the 24-hr settled sludge volumes and the as-sampled sludge masses were measured on each container composite. The measured 24-hr settled volumes and dewatered sludge masses of each container composite show reasonable agreement with the summed settled volumes and masses of the component subsamples.

Table 5.1. SCS-CON-210 Core Subsamples for Container Composite KW210-CC-A

Description	Constituent Core ID	TI Sample Tracking ID	As-Sampled Mass, g[(a)]	Settled Volume, mL[(a)]	Settled Volume Fraction, %	Core Dry Mass[(b)] Fraction, %
Component Subsamples	KW210-A1	KW210-A1-A[(c)]	62.61	48	24	28.0
	KW210-A4	KW210-A4-A[(c)]	74.51	57	28	36.6
	KW210-B1	KW210-B1-A[(c)]	45.45	50	25	15.0
	KW210-B3	KW210-B3-A[(c)]	54.24	48	24	20.5
	---	Sum	236.81	203		
Container Composite	---	KW210-CC-A	250.55[(d)]	220[(d)]	---	---

(a) The masses and 24-hr settled sludge volumes are from component core composite subsamples before combining them to create the container composite.

(b) The dry mass was calculated from the as-sampled mass and the conversion factors $g_{dry}/g_{sampled}$ (See Table 3.1).

(c) Inadvertently core subsample IDs were identical to client sample IDs. These samples are different from the samples identically identified in Table 2.1.

(d) Measured volume and mass after combining the core subsamples.

Table 5.2. SCS-CON-210 Core Subsamples for Container Composite KW210-CC-N

Description	Constituent Core ID	TI Sample Tracking ID	As-Sampled Mass, g[(a)]	Settled Volume, mL[(a)]	Settled Volume Fraction, %	Core Dry Mass[(b)] Fraction, %
Component Subsamples	KW210-A1	TI001-A1-N	44.62	36	26	28.1
	KW210-A4	TI002-A4-N	49.38	29[(c)]	21	34.2
	KW210-B1	TI002-B1-N	37.28	35	26	17.3
	KW210-B3	TI001-B3-N	38.27	36	26	20.4
	--	Sum	169.55	136		
Container Composite	--	KW210-CC-N	178.77[(d)]	155[(d)]	---	---

(a) The masses and 24-hr settled sludge volumes are from component core composite subsamples before combining them to create the container composite.

(b) The dry mass was calculated from the as-sampled mass and the conversion factors $g_{dry}/g_{sampled}$ (See Table 3.1).

(c) The settled volume is suspected to be too low because the as-sampled volume was recorded at 30.5 mL. Using the primary physical property sample results, the settled volume should be closer to 39 mL:

$$49.38\,g_{sampled} \times \left(\frac{1\,g_{settled}}{0.9032\,g_{sampled}}\right) \times \left(\frac{1mL_{settled}}{1.39\,g_{settled}}\right) = 39\ mL$$

(d) Measured volume and mass after combining the core subsamples.

Ideally the settled sludge volume contributions from the component core subsamples would be 25% ± 2.5%. Only one core sample was outside of this range: TI002-A4-N at 21% volume contribution. The volume basis for this sample is suspected to be significantly too low; applying correction factors from physical property samples to the as-sampled mass results in an estimated 39-mL volume for TI002-A4-N.[(a)] If the volume of TI002-A4-N is corrected to 39 mL, the relative volume contribution increases to 27% with the other cores contributing 24 to 25 vol%; this better matches the KW210-CC-A composition.

On a dry mass basis, the relative component core subsample contributions showed large variation. The A4 core dry mass contribution to the container composites was the highest followed by A1 core. Both "B" core samples contributed substantially less dry mass to the container composites than the "A" cores. These dry mass variations were consistently reflected in each of the container composites. Figure 5.1 shows graphically how the volume percent settled sludge and mass percent dry sludge for the two container composites compare. The relative dry mass contribution of the KW210-A4 core subsamples showed close agreement (34.2 vs 36.6%) whereas the relative settled volume contribution showed significant discrepancy (21 vs 28%).

A variety of images were collected while processing the container composite in the SAL. Color representation relative to the MacBeth ColorChecker® Rendition Chart (MacBeth Color Chart) was required per the QAPjP/SAP. Figure 5.2 shows the MacBeth Color Chart taken with the in-cell camera during the KW210-CC processing interval with direct comparison to the truer color image (out-of-cell scanned image). As indicated in Section 4.0, the red hues were largely washed out of the in-cell pictures.

(a) The observations for this sample and anomalous results are documented in Occurrence Report 52578-8-19-11, located with TI 52578-TI002 (DVD-52578-PNNL-20650 Rev. 2, Section III, September 2013).

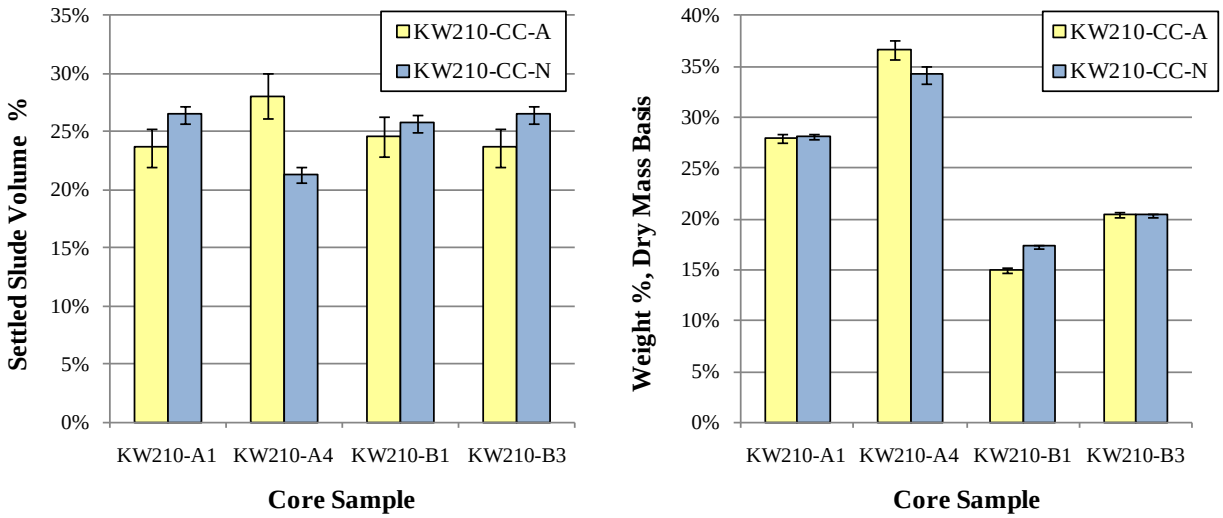


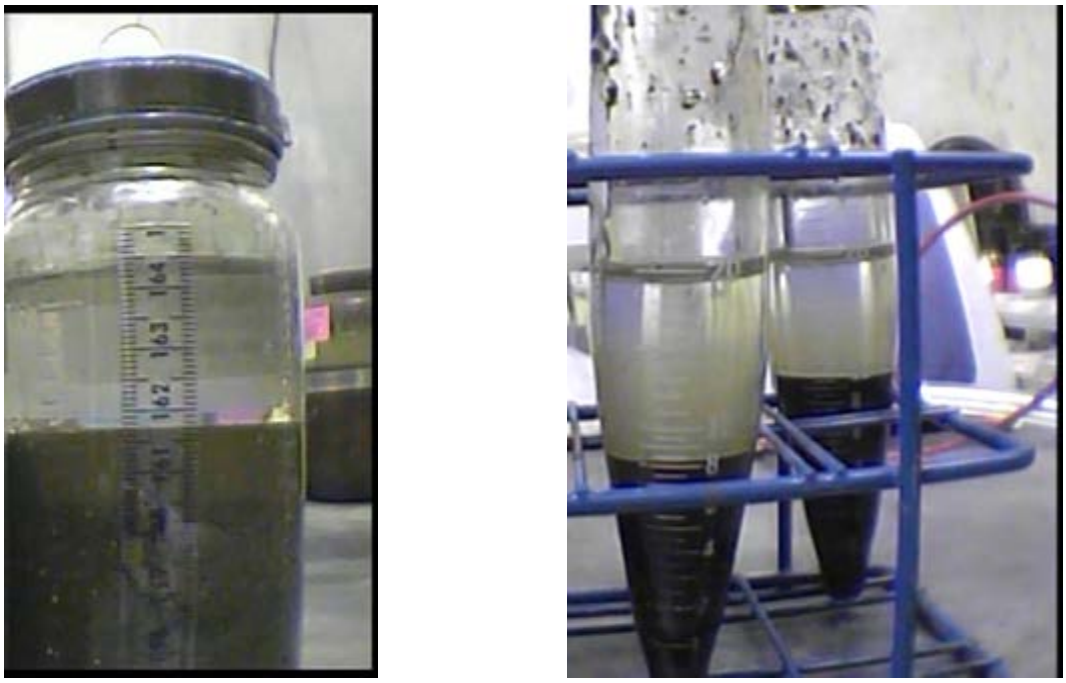
Figure 5.1. Relative Settled Sludge Volume and Dry Mass Contributions of Core Samples to the Container Composite Samples with the Propagated 1-σ Error

Figure 5.2. MacBeth Color Chart, In-Cell Image (Left), and Scanned Image (Right)

Images of the container composite samples are shown in Figure 5.3 and Figure 5.4. The parent material is shown in the 480-mL (for KW210-CC-A) and 240-mL (for KW210-CC-N) Qorpak jars; the physical property samples are shown in the 40-mL centrifuge tubes. The images were collected after the sludge in the jars was mixed and allowed 24-hr settling time. The volume of settled sludge was approximately equal to the mobilizing fluid (water), and good mixing and batch settling were obtained. Gas bubbles associated with this material were not apparent.

Parent Material (KW210-CC-A) Physical Property Samples

Figure 5.3. KW210-CC-A After Mixing and Settling



Parent Material (KW210-CC-N) Physical Property Samples

Figure 5.4. KW210-CC-N After Mixing and Settling

5.2 Rheology

Shear strength measurements were performed on only one KW210 unsieved container composite sample: KW210-CC-A. Appendix H contains the detailed rheology report. A total of four shear strength measurements were performed: one measurement of the as-received settled solids strength, two measurements of the settled solids strength after transfer to a small container and approximately 48 hr after solids resuspension, and one opportunistic measurement at an immersion depth of 26 mm (to evaluate if any significant differences in shear strength resulted as function of depth).

The as-received sample, with a settling time of approximately 11 weeks, was measured using a 6 × 6 mm vane tool to avoid exceeding the maximum operating torque of the RV20-M5 measuring system. This is consistent with October 11, 2010 guidance from CHPRC directing PNNL to use a 6 × 6 mm vane tool for initial shear strength measurements. The initial strength of the sample was established to be within the working stress range of the 16 × 16 mm vane and all subsequent measurements were made with this larger vane tool. The sample was transferred to a smaller container so that the 16×16 mm vane tool could be used, as there was insufficient settled solids height to use this vane in the original container. Table 5.3 provides a summary of all shear strength measurements. Table 5.3 also presents the average shear strength for sample KW210-CC-A, which was determined from the first and second settle-and-mix operations. For each test, the vane was immersed into the center of the jar at the indicated depth. The uncertainty for shear strength measurement average was calculated as twice the standard error of mean[(a)] of the two ~48 hr settled solids shear strengths. This provides an estimate of the lower bound of 95% confidence limits for the mean. Shear strength variation with vane depth had been previously observed with granular, heterogeneous simulant samples (Burns et al. 2011). Therefore, an opportunistic measurement at an immersion depth of 26 mm (which is greater than the standard immersion depth of one vane height) was taken to probe changes in shear strength with immersion depth.

Table 5.3. Shear Strength Summary for KW210-CC-A (all measurements were taken at ambient cell temperature [~29°C])

Sample ID	Measurement	Settling Time (hr)	Immersion Depth (mm)	Shear Strength (Pa)
KW210-CC-A [Note: Sample was not sieved.]	As-Received[(a)]	1,824	6	~281
	1st Mix	50	16	63[(b)]
	2nd Mix	47	16	58[(b)]
	2nd Mix	47	26	113
	Average			60 ± 5[(c)]

(a) Initial, as-received, shear strength measurement using a 6 × 6 mm vane. Subsequent measurements were taken with a 16 × 16 mm vane after the sample was transferred to a smaller container.

(b) Used in average calculation.

(c) Reported uncertainty is twice the standard error of the mean (which approximated the lower bound of the 95% confidence limits).

(a) The standard error of a mean is calculated as the sample standard deviation divided by the square root of the sample size, N (N = 2 for the shear strength measurements reported herein).

It should be noted that the as-received measurement indicated a shear strength of ~281 Pa; however, given that this measurement was subject to significant noise and that the measured value represents only ~0.2% full-scale reading with the 6 × 6 mm vane, this measurement is subject to large uncertainty.

Vane depth dependence was observed for the KW210 container composite sample used in this study under the mixing/settling regime employed. Typically, such increases in shear strength with depth are indicative of stratification of solids or lithostatic effects.

After mixing and settling for ~47 to 50 hr, the shear strength of the material was reduced considerably from the as-received condition. Subsequent mixing and settling operations yielded settled materials with similar shear strength; thus, the initial mixing operation appeared to have fully disrupted any sample structure.

For comparison against the results of actual KW Basin unseived sludge fraction shear strength, the shear strength results of the KW Container sludge simulant (KW-B, <500-μm fraction) are provided in Table 5.4. The KW Container sludge simulant results show shear strength ranging from 800 to 2,400 Pa (Burns et al. 2011), with an implied design/safety basis value for size-fractionated samples containing particles <250 μm of ~500 Pa (Plys and Schmidt 2006). The shear strength results for sample KW210-CC-A are bounded by those listed in Table 5.4. Similar to KW210-CC-A, the KW-B simulant evidenced shear strength variation with immersion depth, with a high measurement of ~6,000 Pa. Variation of the KW-B shear strength with immersion depth was attributed to "particle settling and continued dewatering of the simulant" (Burns et al. 2011). Similar mechanisms may contribute to the vertical stress variations observed in KW210-CC-A.

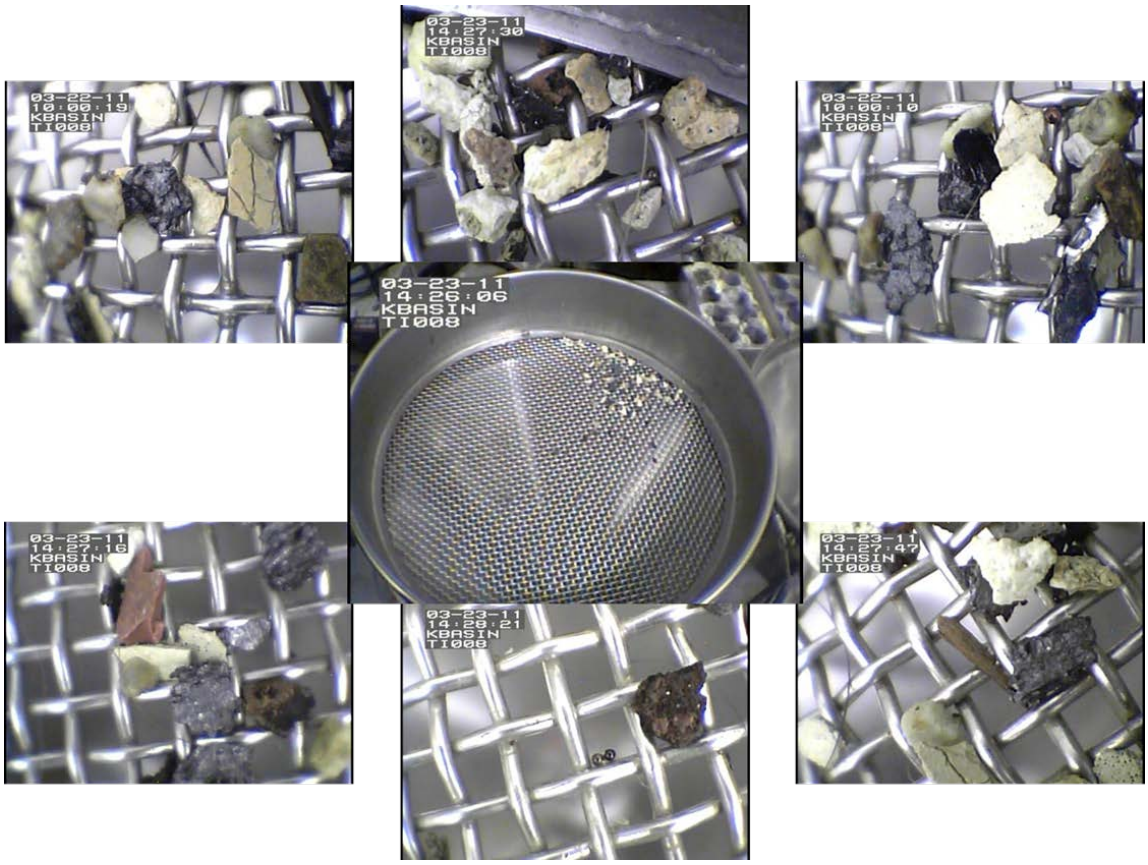
Table 5.4. K West Basin Simulant (−500-μm Sieve Fraction) Shear Strength Summary and Implied Safety Basis

Basis	Shear Strength [Pa]	Reference
Measurements of KW-B (<500-μm) Simulant	800 to 2,400	Burns et al. 2011
Implied Design/Safety; based on samples containing only particles <250 μm	500	Plys and Schmidt 2006, Table C-2

5.3 Sieve Fractions

The KW210-CC-N container composite material was sieved through the 2,000-μm and 500-μm mesh sieve assembly. The materials that collected on the 2,000-μm mesh sieve were designated large container composite (LCC) or +2,000 μm; materials that collected on the 500-μm mesh sieve were designated middle container composite (MCC) or −2,000 μm/+500 μm; and materials that passed through all sieves were designated small container composite (SCC) or −500-μm.

Images of LCC material are shown in Figure 5.5 and Figure 5.6. Some of the LCC particles were reflective and plate-shaped, which is consistent in appearance with Grafoil® particles. The LCC fraction also included light and dark rock-like materials, resin beads, and thread-like material. Although most particles appeared to exist in shades of gray, some particles appeared red to yellow in coloration.



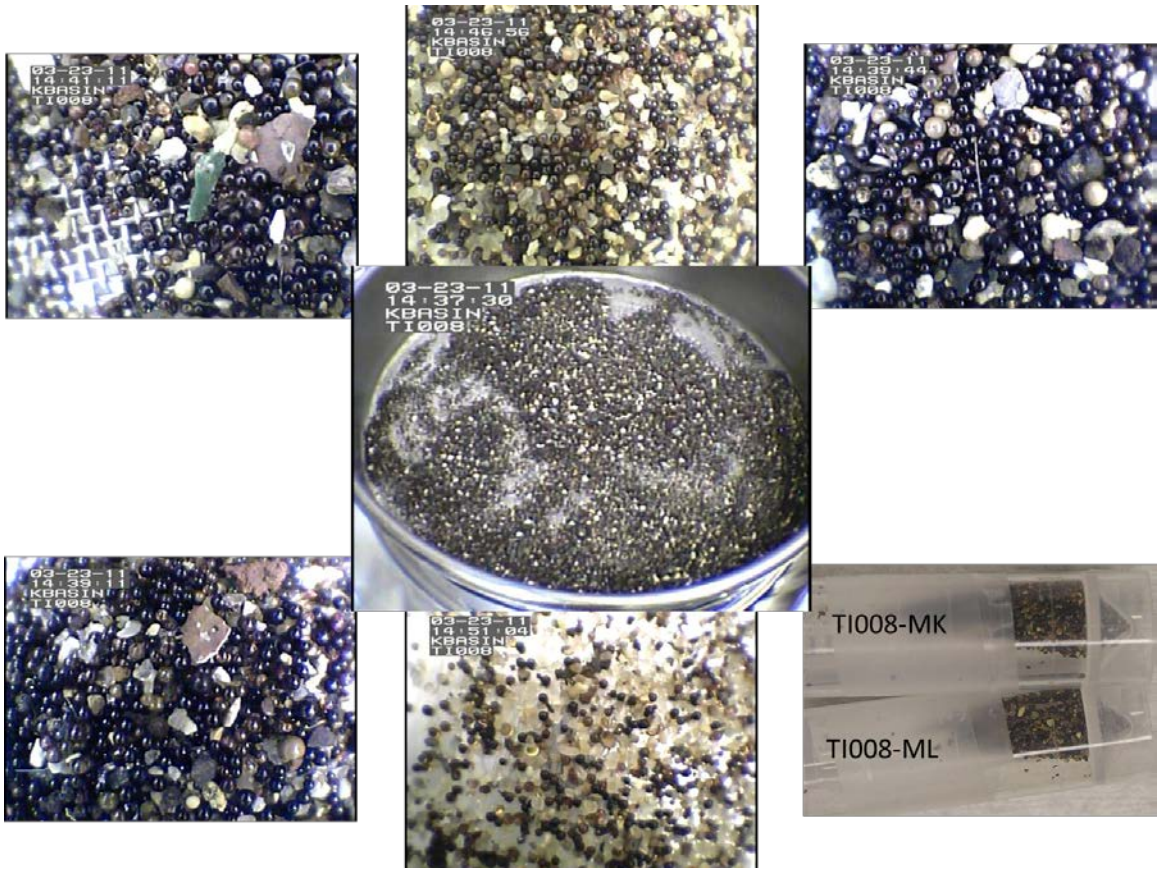
Note: There was a wide variety of particle morphologies; some particles appeared to have reflective surfaces.

Figure 5.5. KW210-LCC Solids Captured on the 2,000-µm Mesh Sieve

Note the reflective surfaces, the thread-like material, and the resin beads. The upper photograph was taken outside of the hot cell, so the colors better represent the "true" colors (sample is in a 20-mL glass scintillation vial).

Figure 5.6. KW210-LCC Transferred to Jar

The MCC consisted mostly of resin beads, almost transparent irregularly shaped particles, and a few colored (green and red) particles (see Figure 5.7).



Note: The lower right shows aliquots of the sample that were removed from the hot cell for particle density measurement (TI008-ML and -MK in 5-mL plastic vials).

Figure 5.7. KW210-MCC Solids Captured on the 500-μm Sieve

Figure 5.8 shows the SCC fraction after 23-hr and 142-hr settling. It appeared to be homogenously muddy-brown. No apparent stratification was observed.

Note: The sludge layer is the dark brown material; clarified water is above. The total (water plus sludge) volume was ~214 mL. The 23-hr settled sludge volume was ~57 mL (left); the 142-hr settled sludge volume was ~44 mL (right).

Figure 5.8. KW210-SCC After Mixing and Settling

Table 5.5 summarizes the mass distributions on a dry mass basis for each sieve fraction. The calculated dry masses of the initial composite and the −500-μm fraction were determined from the total settled solids volume and the physical property characterization samples (see Section 3.0). The dry masses of the wet sludge therefore corresponded to higher uncertainty. The dry mass fraction of the MCC fraction was calculated by multiplying the net fraction mass by the calculated wt% solids (90.4%, which was determined from drying the duplicate ASO samples [TI008-MA and TI008-MB] at 105°C to constant mass). The mass fractionation was calculated from the sample fraction dry mass divided by the sum of the dry fraction masses (77 g). The calculated input dry fraction mass (65 ± 6 g [1-σ]) was lower than the calculated dry mass sum of fractions (77 ± 3 g [1-σ]). The difference could be associated with experimental error or the resin beads in the MCC fraction may contain additional water.

Table 5.5. Dry Mass Distribution of KW210-CC-N Container Composite Sieve Fractions

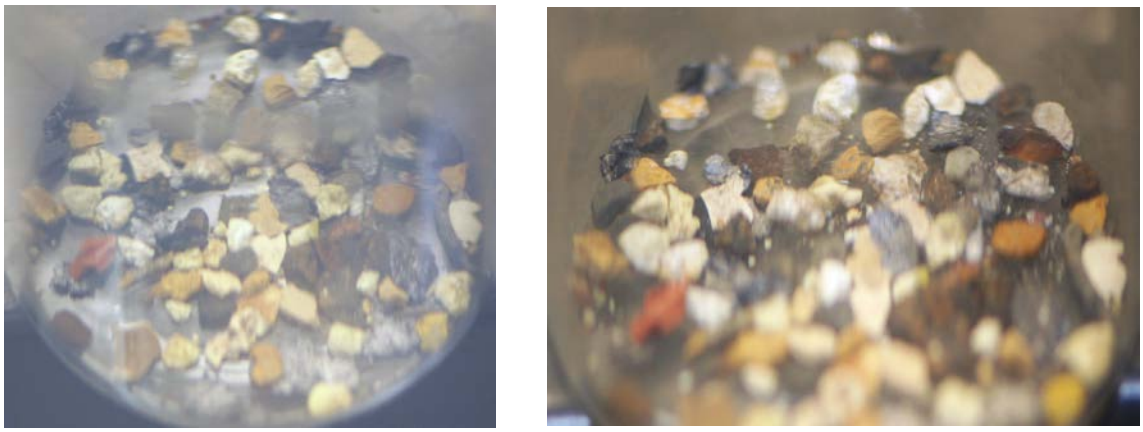
	KW210-CC		KW210-LCC	KW210-MCC	KW210-SCC
Parameter	Calculated Initial Composite[(a)]	Sum of Dry Fractions	+2,000 μm	−2,000 μm/+500 μm	−500 μm
Mass (g)	65 ± 6	77 ± 3	0.7263	55.2	21.5
Dry mass fraction	--	1.00	0.0094	0.71	0.28

(a) Mass of fraction of composite that was sieved. A portion of the composite had been subsampled for physical property measurements; the calculated initial composite mass accounts for this subsampling activity.

The MCC fraction contained approximately 2.5 times the mass of the SCC fraction. There was just sufficient volume and mass of the SCC fraction to accommodate all targeted subsample collections. The LCC fraction constituted <1 wt% of the container composite. The small mass collected for the LCC fraction limited the targeted analysis suite to direct GEA followed by particle density—no dissolution processing.

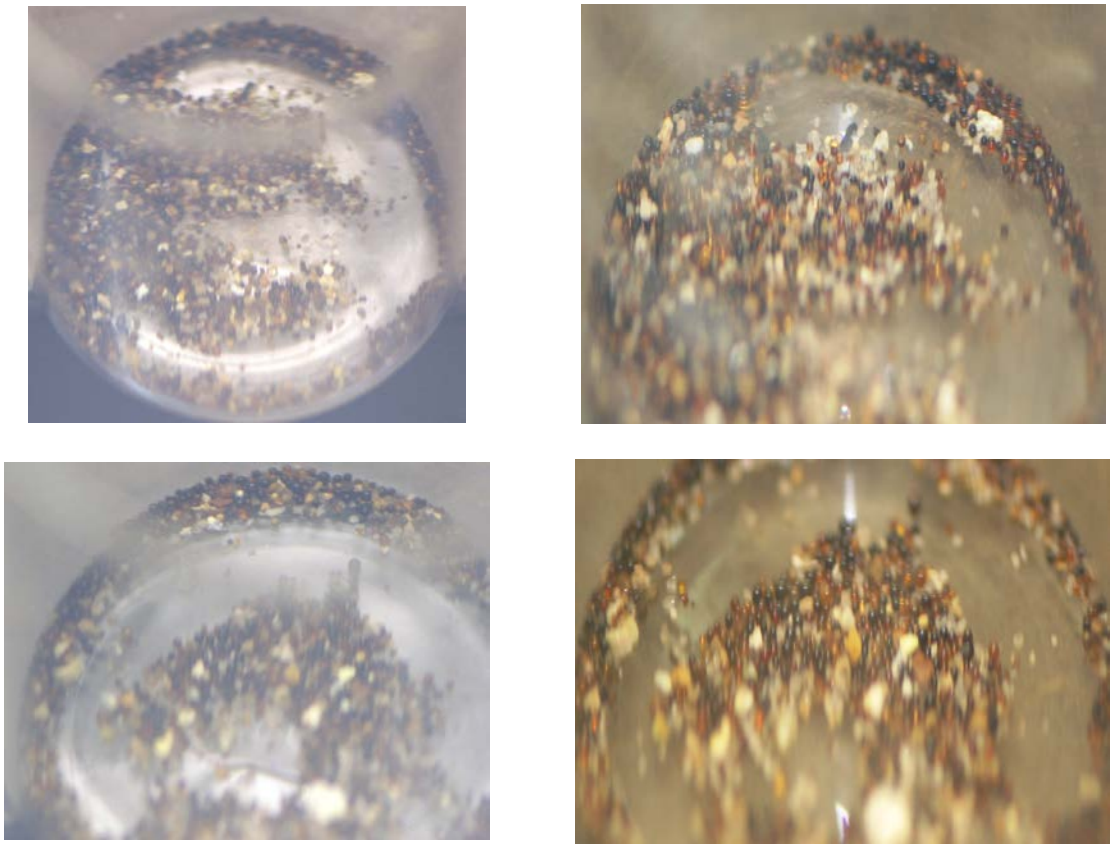
5.4 Particle Densities and Bulk Volume

Five samples were submitted for analysis, one LCC, two MCC, and two SCC fraction samples. The full particle density analysis report is provided in Appendix G. Figure 5.9 through Figure 5.11 present images of the sample materials. The TI008-L, TI008-MK, and TI008-ML samples had particles of varying shapes, sizes, and colors. The TI008-MK and TI008-ML samples had visible resin beads and appeared to have a significant charge, as evidenced by the particles clinging to the plastic container walls (see Figure 5.12). The TI008-SK and TI008-SL samples appeared as a dark brown powder.



Note: Focus issues are an artifact of the glovebox window.

Figure 5.9. Images of TI008-L, +2,000-µm Sieve Fraction of the KW210-CC-N Container Composite



Note: Focus issues are an artifact of the glovebox window.

Figure 5.10. Images of TI008-MK (top row) and TI008-ML (bottom row), −2,000-µm /+500-µm Sieve Fraction of the KW210-CC-N Container Composite

Figure 5.11. Images of TI008-SK and TI008-SL in the Gas Pycnometry Analysis Cup, −500-µm Sieve Fraction of the KW210-CC-N Container Composite

Figure 5.12. Sample Vials Containing As-Received TI008-MK and TI008-ML After Placement in the Glovebox

The sample volumes were small enough to allow most if not all material transfer into a tared 1-cm^3 pycnometer sample cup for analysis. The pycnometer cup holding the sample was placed into the sample chamber and analyzed five sequential times. The results were averaged and the density with standard deviation was recorded. Table 5.6 presents the density results in g/cm^3 along with %RSD for the five replicate sample runs. The averaged sample result and the averaged duplicate sample result were used to determine the RPD. Data are provided in Appendix G.

Table 5.6. Particle Density Results

Sieve Fraction	Sample ID	Physical Description	Air-Dried Sample Mass (g)[(a)]	Mass Analyzed (g)[(b)]	Average Density, g/cm^3	Sample % RSD	% RPD
+2,000 µm	TI008-L[(c)]	Particles of varying shapes, sizes and colors	0.7588	0.6780	2.60	0.15	NA
−2,000 µm/ +500 µm	TI008-MK	Particles of varying shapes, sizes and colors. Resin beads evident.	0.5231	0.4142	1.62	0.04	0
	TI008-ML		0.5074	0.3420	1.62	0.03	
−500 µm	TI008-SK	Dark brown powder	0.6755	0.6355	2.57	0.05	3
	TI008-SL		0.3462	0.2962	2.49	0.07	

(a) The tare masses of the vials were recorded in TI 52578-TI008. The samples were air-dried in the glovebox and a gross weight was obtained. Mass of the subdivision is the difference between the gross and tare masses.

(b) Mass of the sample removed from vial and placed in analysis sample cup.

(c) Sample TI008-L was labeled TI008-LA when analyzed by GEA per ASR 8824.

NA: Not Applicable.

The QAPjP/SAP delineated a particle density precision of ±5%. Over 50% of each subsample was analyzed, minimizing possible variations from subsampling at the workstation. Because the LCC sample was not split for analysis, the RPD could not be calculated. The duplicate sample precision for the −2,000-µm/+500-µm (TI008-MK and TI008-LK) and −500-µm (TI008-SK and TI008-SK) sieve fractions met the QAPjP/SAP criterion.

Table 5.7 summarizes the bulk dry density, dry mass, bulk dry volume, and absolute dry volume for the LCC and MCC sieve fractions (as applicable). These measures were determined opportunistically and acceptance criteria do not apply. The bulk density should be used with caution because it includes void space. The dry mass was taken from Table 5.5 and is shown again for information.

Table 5.7. Approximate Bulk Density and Volume of Dried Solids

Sample ID	Dry Bulk Density, g/mL (D_B)	Dry Mass, g (M)	Bulk Volume, mL (V_B)	Absolute Volume, mL (V_A)
KW210-LCC	--	0.7263	--	0.279
KW210-MCC	1.0	55.18	55	34.1

The bulk density value should be used with caution since this measure includes the void fraction.

Dry masses were copied from Table 5.5 and are supplied here for reference.

The variable symbols (D_B, M, V_B, and V_A) are referenced in Equations 5.1 and 5.2. The absolute density (D_A) was determined by gas pycnometry and is provided in Table 5.6.

The bulk volume refers to the total volume of solids (inclusive of void space) in the sieve fraction and was calculated according to Equation 5.1.

$$V_B = \frac{M}{D_B} \tag{5.1}$$

where V_B is the bulk volume, M is the total mass of sieve fraction, and D_B is the bulk density (determined from a sample aliquot). The bulk volume of the LCC solids was not determined because of the difficulty of measuring a small volume of large granular materials in a vial. The MCC bulk density was determined opportunistically from the volume of a sample split measured in a 50-mL, volume-calibrated centrifuge tube and its corresponding net mass. In this case, the MCC solids were poured into the centrifuge tube—no other method was employed to further pack the particles in the tube.

The absolute volumes of the MCC and LCC samples were calculated from the total sieve fraction mass, and the particle density (determined by gas pycnometry) according to Equation 5.2.

$$V_A = \frac{M}{D_A} \tag{5.2}$$

where V_A is the absolute volume, M is the total mass of sieve fraction, and D_A is the absolute density determined by gas pycnometry. The absolute volume represents the total particle volume of the sieve fraction free of void space. The absolute volume for the MCC fraction was ~62% of the bulk volume.

Table 5.8 compares the 24-hr settled sludge volumes of the initial container composite before sieving to the SCC fraction. The SCC fraction represented 42 vol% of the total container composite. The sum of combined wet settled SCC sludge volume and the absolute volumes of the MCC and LCC fractions is

also provided in Table 5.8. The summed volumes represented only 67% of the total container composite. The drying process of the MCC and LCC fractions could have resulted in a significant contraction of resin beads and other components and/or the OIER volume measured from gas pycnometry did not include the resin void spaces (pore spaces). The observations for the KW210 container composite material are consistent with those reported for the KW220 container composite (Fiskum et al. 2011b).

Table 5.8. Volume Comparisons of the KW210-CC-N Container Composite to the SCC and Combined Fractions

Initial Settled Wet Sludge Composite Volume, mL[(a)]	Wet SCC Volume Fraction		Combined Wet SCC and Dry MCC and LCC Volume Fractions	
	Settled Wet Sludge, mL[(a)]	% of Composite	Total, mL[(b)]	% of Composite
137	57	42%	91.3	67%

(a) Measured after ~24 hr of settling. The volume uncertainty was estimated to be ±5 mL.

(b) The volume sum combined the wet settled SCC volume plus the absolute dry volumes of the LCC and MCC fractions.

5.5 Crystal Structure

Two sample aliquots from each of the MCC and the SCC solids were characterized for crystalline phases. The full XRD analysis reports with XRD patterns can be found in Appendix F.

Table 5.9 summarizes the crystalline phases identified in the two subsamples collected for analysis from each of the SCC solids (−500 μm). These solids were dominated by quartz (SiO_2) and uranium oxide (UO_2 and U_4O_9), present as major phases. Unassigned peaks are also shown. In all cases the unassigned peaks were very low intensity and exhibited broadening over ~0.3° 2-theta with a non-descript peak center.

Table 5.9. Crystalline Phases Identified in KW210-SCC (−500-μm Fraction)

Phase Present	Chemical Formula	Relative Intensities (Rietveld Analysis)	
		TI008-SH	TI008-SJ
Quartz	SiO_2	Major	Major
Uraninite	UO_2	Major	Major
Uranium oxide	U_4O_9	Major	Major
Unassigned peaks		23.6°, 34.5°, 35.6°[(b)]	24°,[(a)] 35°, 35.8°,[(b)] 37°, 38°, 51.5°

(a) The peak at 24° 2-theta was found only on the subsample that included the internal TiO_2 tracer. It was consistent with the peak located at 23.6° 2-theta in TI008-SH.

(b) The peaks at 35.6 and 35.8° 2-theta for TI008-SH and TI008-SJ, respectively, represent the same peak. See text for discussion of peak center identification.

Table 5.10 summarizes the crystalline phases identified in the two subsamples collected for analysis from each of the MCC solids (−2,000 μm/+500 μm). Only quartz was identified in the MCC fraction.

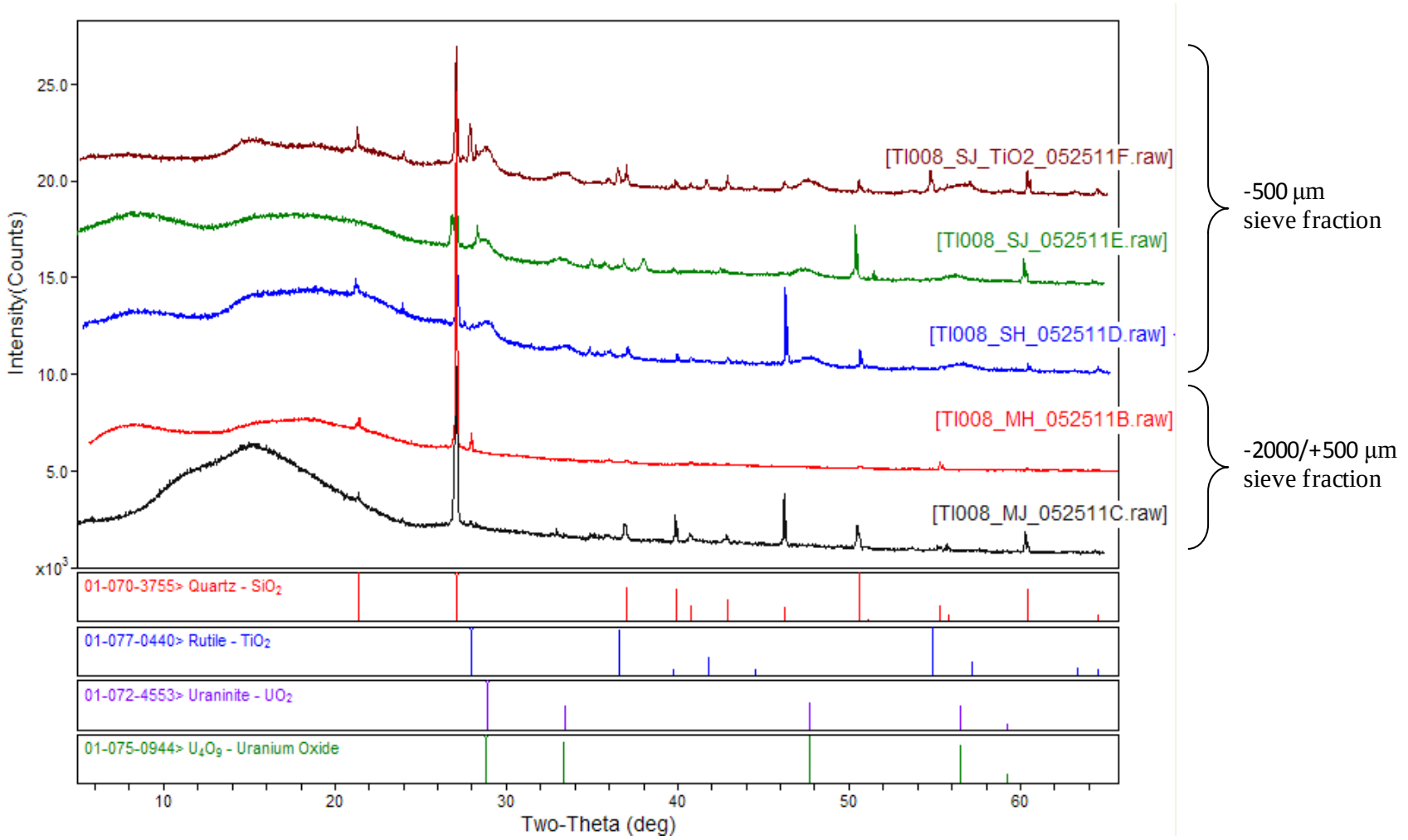
Table 5.10. Crystalline Phases Identified in KW210-MCC (−2,000-μm/+500-μm Fraction)

		Peak Intensities (Rietveld Analysis)	
Phase Present	Chemical Formula	TI008-MH	TI008-MJ
Quartz	SiO_2	Major	Major
Unassigned peaks		27.3°	33°, 35°-36°[(a)]

(a) A broad peak was present between 35° to 36° 2-theta.

Quartz is not expected to dissolve in the acid digestion and thus Si would not be substantially present in the acid digestate measured by ICP-OES. There was no evidence from the SCC or MCC diffraction patterns for the presence of Zr metal, Al hydrates, or U hydroxides.

Figure 5.13 shows the X-ray powder diffraction patterns for each of the MCC and SCC samples. All samples resulted in a raised, low-angle background between approximately 8 and 25° 2-theta. The backgrounds for replicate SCC samples (TI008-SH, -SJ, and –SJ with TiO_2) appeared similar in shape and intensity. The low-angle background shape and relative intensity for sample TI008-MJ was substantially different from the duplicate sample, TI008-MH. The differences may be caused by one or both of the following phenomena: 1) less TI008-MJ sample mass was loaded on the slide relative to the sample mass loading for TI008-MH; 2) more amorphous component was present in sample TI008-MJ relative to that of sample TI008-MH. The most intense peaks of TI008-MH and TI008-MJ were equalized using the system software. Raising the sample peak height also proportionately raises all other diffraction pattern features, including the background.



Note: Sample TI008-SJ was run with and without TiO_2 internal standard (first and second diffraction patterns from top, respectively).

Figure 5.13. XRD Powder Diffraction Patterns for the KW210-MCC (−2,000-µm/+500-µm) and KW210-SCC (−500-µm) Samples

5.6 Particle Size Distribution

Two subsamples collected from KW210-SCC (−500-µm sieve fraction) were evaluated for PSD, TI008-SP (primary) and TI008-SQ (duplicate). Appendix I of this report contains the detailed particle size analysis report. The parameters used for sample analysis are given in Table 5.11.

Table 5.11. Default Optical Properties Used for PSD Analysis of KW210-SCC Samples

Property	Values Employed
Refractive Index	2.42
Absorption Index	1
Particle Shape	Irregular
Model	General Purpose (Broad Distribution)
These properties were assessed through post-measurement reanalysis of KE KW240 (TI010-SP) data collected during the initial parameter evaluation study (Fiskum et al. 2011b). Application of these parameters to the KW sludge (KW210) was considered to be appropriate.	

PSD characterizations consisted of an average of three individual PSD measurements for two sub-aliquots (collected at the workstation) per sample. Data obtained for all sub-aliquots were averaged to obtain one PSD for each measuring condition per sample, i.e. at 1 min recirculation (unsonicated), 100% sonication (20 watts), and post sonication.

Table 5.12 provides the matrix of conditions for both of the PSD samples analyzed. The initial measurement conditions at 1 and 3 min recirculation times are based on results obtained from a parameter evaluation study (Fiskum et al. 2011b, TI 52578-TI017). An additional data measurement set was collected (a post-sonicated measurement after 5 min of total recirculation) to further evaluate the effect of sonication on the measured PSD.

Table 5.12. Test Condition Matrix for PSD Determinations

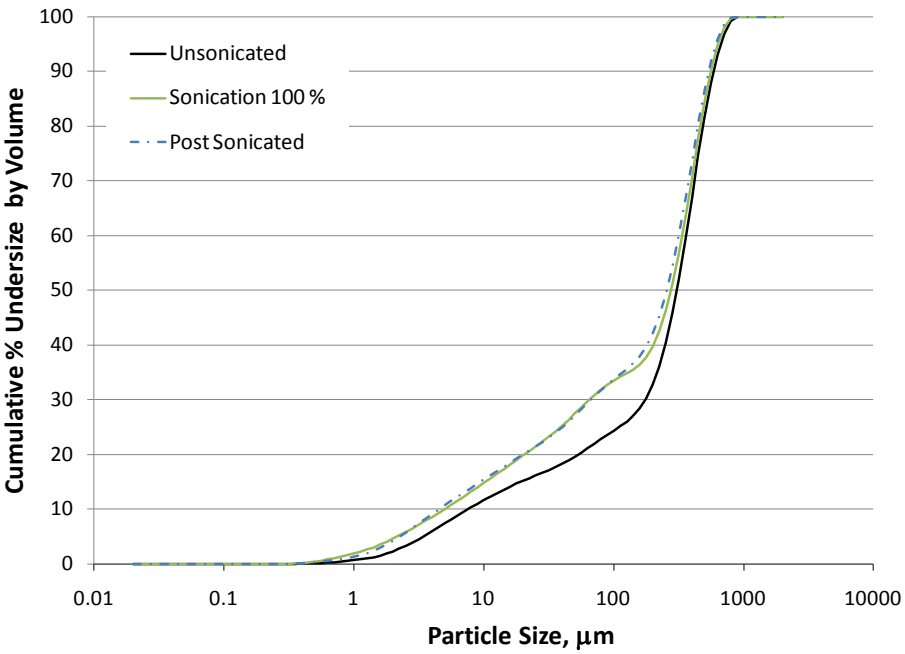
Recirculation Rate, RPM	Recirculation Time, min	Sonication Power, Time
2,000	1	Pre-sonication
2,000	3	120 sec/100 %
2,000	5	Post-sonication

The cumulative % undersize data for the 1-min recirculation time and the post-sonicated results are given in Table 5.13; these data have been plotted along with the volume contribution % PSD and are shown in Figure 5.14 and Figure 5.15. The 100% sonication data (see Appendix I) have also been included on Figure 5.14 and Figure 5.15. Post-sonicated data were collected as a precautionary measure, given the sample dilutions required to obtain a suitable sample obscuration. Dilute samples sometimes form bubbles and or agglomerate as a result of sonication. Neither was observed for either sample analyzed. The data obtained at 1-min recirculation time are more representative of the suspended particles present during settling of actual sludge material while the post-sonicated data better represents the true PSD of the fully suspended sample particles. Based on the similarity and consistency between the sample and duplicate, the PSDs from the samples were averaged.

Table 5.13. Cumulative Percent Undersize at 1-min Recirculation Time and Post-Sonication

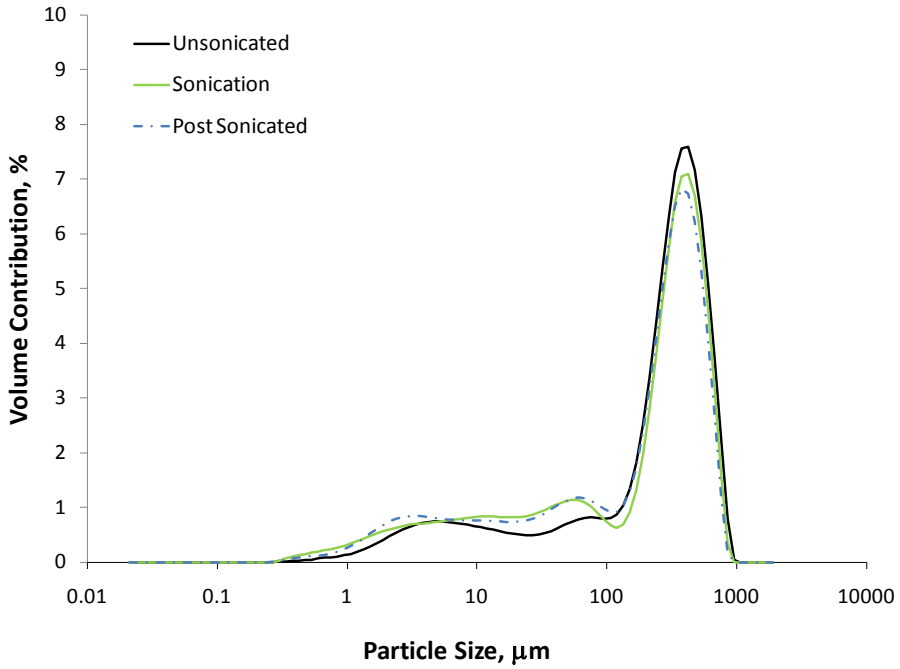
	KW210 −500-µm Size Fraction Average[a] Cumulative Volume % Less Than	
Size, µm	1-min Recirculation, Unsonicated	Post-Sonication
0.05	0	0
0.5	0.14	0.48
1	0.68	1.90
2.5	3.28	5.83
5	7.41	10.1
10	11.7	14.8
20	15.2	19.8
40	18.3	25.3
60	20.8	29.2
80	22.8	31.9
100	24.4	33.5
125	26.0	34.8
150	27.7	35.9
175	29.9	37.5
200	32.8	39.7
300	49.1	54.0
400	67.1	70.6
500	81.4	84.0
600	90.9	92.9
700	96.5	97.7
800	99.2	99.7
1000	100	100

(a) Based on the average of 12 PSD measurements generated from two aliquots of sample TI008-SP (primary) and two aliquots of TI008-SQ (duplicate).



Note: The PSD is based on the average of 12 measurements generated from four aliquots, two aliquots from sample TI008-SP (primary) and two aliquots from TI008-SQ (duplicate)

Figure 5.14. Average Cumulative Volume % Undersize Plot of KW210-SCC (TI008-SP and TI008-SQ, −500-µm Size Fraction)



Note: The PSD is based on the average of 12 measurements generated from four aliquots, two aliquots from sample TI008-SP (primary) and two aliquots from TI008-SQ (duplicate)

Figure 5.15. Average Volume % PSD Plot of KW210-SCC (TI008-SP and TI008-SQ, −500-µm Size Fraction)

A major peak in the PSD was observed between 100 and 1000 µm, apex at ~400 µm. Resin beads were observed in the −500-µm sieve fraction sample (see Figure 5.17 of the UDS). Product specification for the mixed bed resin Purolite NRW37 indicates that it has a wet PSD between 425 and 1200 µm and <2% is <425 µm. The ~400 µm peak observed in Figure 5.15 may in part be represented by the small portion of resin beads that passed through the 500-µm mesh sieve.

For all sample aliquots, grinding and knocking sounds could be heard inside the dispersion unit, indicative of large particles caught-up in, and knocking on, internal parts of the dispersion unit. This would suggest that there were particles present in both samples provided for analysis with at least one linear dimension >500 µm. Sieving through a 500-µm mesh sieve does not always eliminate particles with one dimension >500 µm. If large particles are held up in the dispersion components, they may not flow into the observation cell and be measured by the instrument. Additionally, hold-up of large particles may block the flow of smaller particles, preventing their observation. Hence the PSDs provided in Table 5.13, Figure 5.14, and Figure 5.15 may not provide an accurate representation of the sample (<150 µm) with the respect to the larger size fraction. The grinding and knocking sounds were not heard once the instrument was flushed, cleaned, and filled with K Basin water for the background measurement, which suggests that the large particles did not accumulate in the system and affect subsequent measurements.

Table 5.14 summarizes selected percentiles for comparing the KW210-SCC samples with the settler sludge simulant and the KW container sludge simulant (Burns et al. 2011). The actual sludge has larger particles present when compared with the K Basin simulants. This is observed in the larger particle size for the d(50) and d(90) for the KW210-SCC samples analyzed. Table 5.14 provides a brief overview of the particle sizes present, in particular the tails of the PSD (upper and lower limits).

Table 5.14. Summary Table of Selected Percentile Values for KW210-SCC (−500-µm Size Fraction) and K Basin Simulants

	KW210-SCC, µm		K Basin Simulants, µm[(a)]		
Percentile	1-min Recirculation, Unsonicated	100% Sonication	Settler Simulant ST-A	KW Container Simulant KW-B (<500 µm)	KE Container Simulant KE-PNNL-500 (<500 µm)
d(0.01)	1.24	0.68	-	-	-
d(0.05)	3.42	2.15	-	-	-
d(0.10)	7.52	4.96	-	3	0.7
d(0.20)	53.4	20.5	5	-	-
d(0.50)	305	276	14	25	8
d(0.90)	588	562	334	345	71
d(0.95)	666	636	-	-	-
d(0.99)	787	750	-	-	-

(a) Burns et al. 2011. Simulant samples PSD results are provided in a sonicated state.

5.7 Uranium Speciation

The MCC and SCC fractions were subsampled for U speciation according to 52578-TI008. The sample collected on the 2,000-μm mesh sieve (i.e., the LCC fraction) was not analyzed for U speciation because the sample mass was too small to split for a variety of analyses. Subsamples were processed according to ASR 8824 Special Instructions using selective acid digestion (procedure PNL-ALO-107) that provided separate aliquots for analysis: U metal and U(IV)/U(VI) ratio. All samples were processed (digestion, U metal analysis by ICP-OES, and U(IV)/U(VI) ratio by spectrometry) in one analytical batch.

Samples provided for analysis, along with cross-reference information (from Section 2.0) are identified in Table 5.15 along with the U speciation results: metal (on wt% dry mass and wt% settled sludge mass bases) and U(IV) and U(VI) oxidation state distribution. For reference, the wt% U total is shown on a dry mass basis as determined from ICP-OES analysis (refer to Table 5.20). Uranium metal was measurable in all samples. The uncertainty for U metal concentrations >0.03 wt% is estimated to be ±25% (2-σ) or less.

Table 5.15. Uranium Speciation in KW210-CC Sieve Fractions MCC and SCC: Metal Concentration and Oxide Fractionation (ASR 8824)

Sieve Fraction ID:		MCC		SCC[a]	
Sieve Fraction:		−2,000 μm /+500 μm		−500 μm	
Sample ID:	Preparation Blank	TI008-ME	TI008-MF	TI008-SE	TI008-SF
RPL/ASO ID:	11-0792-PB	11-0792-107-S	11-0792-107-D	11-0793-107-S	11-0793-107-D
U Metal, As-sampled Basis					
U metal, wt%	<0.011	0.426[b]	0.170[b]	0.0674	0.0426
U Metal, Dry Mass Basis					
U metal, wt%	<0.023	0.471	0.188	0.142[d]	0.0899[d]
RPD		86		NA	
U Metal, Wet Settled Sludge Mass Basis					
U metal, wt%	<0.0081	0.236[c]	0.0940[c]	0.0498[d]	0.0315[d]
U Oxide Fractionation, %[e]					
U(IV)	NA	>80	>80	80.3	86.1
U(VI)	NA	<20	<20	19.7	13.9
U total (ICP-OES), Dry Mass Basis from Chemical Characterization Samples					
Sample ID:	Preparation Blank	TI008-MA and -MB (Avg.)		TI008-SA and -SB (Avg.)	
U Total, wt%	<0.0011	2.77		16.6	

(a) The wt% uranium metal was back-calculated for the MS sample (TI008-SG) by correcting for the amount of U metal spike added to the sample. The resulting 0.241 wt% U metal (dry mass basis) in the MS sample was higher than that of the sample and duplicate concentrations.

(b) The as-sampled mass basis for medium sieve fraction samples (i.e., TI008-ME and TI008-MF) is air dried and thus the sample may contain some residual water.

(c) The factor used to convert the middle sieve fraction from a dry mass basis to a wet settled sludge mass basis was 0.5 ($g_{dry}/g_{settled}$). It is an approximation based on observations of other K Basin sludge materials.

(d) The factor used to convert from an as-sampled mass basis to a dry mass basis was 0.474 ($g_{dry}/g_{sampled}$), and to a wet settled sludge mass basis was 0.738 ($g_{sampled}/g_{settled}$); see Section 3.0.

(e) The U(IV) and U(VI) oxidation state ratios should be used with caution. Fe(II) has been shown to reduce U(VI) to U(IV); the amount of Fe(II) is not known in these samples. The U(VI) fraction can be considered a lower bounding case and the U(IV) fraction can be considered an upper bounding case (Fiskum et al. 2011b, Appendix J.)

Gray shading indicates that the target RPD (±20% when the U metal concentration is >0.1 wt%) was exceeded.
The analytical reports for these samples can be found in Appendix C.
NA: Not Applicable. The U metal concentration in one of the subsamples was <0.1wt%, therefore the RPD calculation does not apply; the U(IV) and U(VI) concentrations measured in the preparation blank were <0.001 M.

The KW210-CC sieve fraction samples were primarily U(IV). No U(VI) phases were identified by XRD in either the SCC or MCC fractions of the container composite; U(IV) phases were identified in the SCC fraction. Iron (II)-bearing phases (such as magnetite and iron oxide [Fe_3O_4]) were not specifically identified by XRD in these fractions; however, Figure 4.5 shows that during the sample aliquoting process, an appreciable quantity of particles were attracted to the Teflon coated magnetic stir bars. It is possible that the U oxidation state ratios could be confounded by reduction of U(VI) with the matrix-related Fe(II) (Fiskum et al. 2011b, Appendix J). Thus, the reported oxidation state fractions should be used with caution. The difference between the sample and duplicate oxidation state ratios is 5.8%.

The QAPjP/SAP required ±20% RPD when the U metal content was >0.1 wt% wet settled mass basis. Subsamples from both the MCC and SCC fractions were processed in duplicate. The MCC sample and duplicate RPD (86%) exceeded the acceptance criterion (U metal concentration 0.471 and 0.188 wt% dry mass basis). The variability was higher than the MCC sample/sample duplicate variability determined from ICP-OES (see Section 5.8). One of the duplicate SCC sample U metal concentrations was <0.1 wt%, therefore the duplicate precision calculation was not applicable.

Table 5.16 summarizes the U speciation QC sample results. The QAPjP/SAP required a measure of accuracy (75 to 125% recovery) for a LCS (U metal and U_3O_8 added to Hanford sand matrix). The U metal LCS recovery was 103%. The U oxidation state fractionation of the LCS sample was well within the acceptance criterion. Opportunistically, a replicate SCC (TI008-SG) sample was processed as an MS sample. It recovered 123% of the added U metal. Assuming that the amount of U metal added in the MS was recovered at 100%, the U metal concentration in the TI008-SG sample was back-calculated to be 0.241wt% on a dry mass basis. Thus, the average U metal concentration in the three analyzed KW210-SCC samples was 0.158 wt% ± 0.077% (48% RSD), at one σ, dry mass basis. U metal distribution heterogeneity is shown in these three separate analyses. The U measured in the solids 7th rinse solution (before nitric acid dissolution of U metal) ranged from 0.04 to 1.5 μg/mL, well below the 100-μg/mL acceptance threshold, indicating the measured U metal was not confounded by carryover of non-metallic U. Detailed results and a discussion of the U metal and U oxidation state analyses can be found in Appendix C.

Table 5.16. Uranium Speciation QC Sample Results Summary

	Process Batch QC Sample Results
U Metal	
LCS Recovery	103%
MS Recovery (Opportunistic) on SCC Sample (TI008-SG)	123%
U Oxidation State LCS Recovery	
U(IV) (Accepted 33.3±5%)	Measured 34.2%
U(VI) (Accepted 66.7±5%)	Measured 65.8%

5.8 Chemical and Radiochemical Analysis

Chemical and radiochemical analyses were conducted according to ASR 8824. Subsamples from the middle and small particle size sieve fractions were processed by acid digestion (PNL-ALO-129 procedure) in preparation for analysis: GEA, AEA screen, and ICP-OES. Because a small amount of

sample was collected on the 2,000-µm mesh sieve, the sample was measured directly by GEA and for particle density. Samples provided for chemical analysis, along with cross-reference information (from Section 2.0) are identified in Table 5.17. The ASO analytical reports for these samples can be found in Appendix C.

Table 5.17. ASR 8824 Batch Processing by PNL-ALO-129 Acid Digestion

Sieve Fraction	Fraction Code	Sample ID	RPL/ASO ID	Analysis
+2,000 µm	LCC	TI008-LA[(a)]	11-0791-S[(a)]	Direct GEA only
−2,000 µm/+500 µm	MCC	TI008-MA	11-0792-129-S	Radiochemistry, ICP-OES metals
		TI008-MB	11-0792-129-D	
−500 µm	SCC	TI008-SA	11-0793-129-S	Radiochemistry, ICP-OES metals
		TI008-SB	11-0793-129-D	
		TI008-SC	11-0793-129-MS	

(a) There was insufficient mass to conduct the acid digestion and other analyses. Sample was only analyzed directly by GEA and for particle density. No acid digestion was performed. This sample is identified as TI008-L for particle density analysis.

The holding time applicable to ICP-OES AOIs was calculated for the preparation batch samples. The sample date was defined as the date associated with the first of the core sample material that was removed from SCS-CON-210. The end of the holding time was defined as the date of the ICP-OES analysis for the sieve fractions of the container composite. In all cases, the 6-month holding time requirement was met (Table 5.18).

Table 5.18. KW210-CC Sieve Fraction Sample Holding Times Supporting WIPP Hazardous Waste Permit Metals Analysis

Core Sample ID	First Sample Date	Acid Digestion Date	ICP-OES Analysis Date	Effective Holding Time, Months[(a)]
KW210-A1	12/29/2010	April 20, 2011	April 26, 2011	3.93
KW210-A4	11/17/2010			5.33
KW210-B1	12/1/2010			4.87
KW210-B3	12/15/2010			4.40

(a) The effective holding time was calculated from the first core sample collection date to the last sample analysis date (e.g., November 17, 2010 to April 26, 2011).

The pre-digestion dry masses, as-settled sludge masses (SCC fractions), and UDS dry masses following the acid digestion procedure are shown in Table 5.19. The UDS masses were also calculated relative to a wet settled solids mass basis for the SCC solids only. The applied conversion factors, $g_{dry}/g_{settled}$, are also shown, as applicable. The UDS of the SCC and MCC fractions exceeded the 5 wt% threshold of the settled solids mass, thus triggering the need for GEA and XRD analyses. Additional analyses (dissolution using fusion and ICP-OES and AEA screen) of the UDS were requested by the BTR.

Table 5.19. Chemical Analysis Characterization Samples for Acid Dissolution, Net Residue Masses, and UDS Weight Fractions (ASR 8824)

Sieve Fraction	Sample ID	RPL/ASO ID	Pre-digest dry mass, g_{dry}	Conversion factor,[(a)] $g_{dry}/g_{settled}$	Pre-digest settled sample mass,[(a)] $g_{settled}$	UDS post-digest dry mass, $g_{residual}$	Weight Fraction UDS		
							Dry mass basis, $g_{residual} / g_{dry}$	RPD or RSD	Settled mass basis,[(a)] $g_{residual} / g_{settled}$
MCC	TI008-MA	11-0792-129-S	1.3641	--	--	1.1750	0.8614	0.70	--
	TI008-MB	11-0792-129-D	1.7754	--	--	1.5186	0.8554		--
SCC	TI008-SA	11-0793-129-S	0.8873	0.350	2.537	0.4087	0.4606	3.0	0.161
	TI008-SB	11-0793-129-D	1.0879	0.350	3.111	0.5009	0.4604		0.161
	TI008-SC	11-0793-129-MS	0.8999	0.350	2.573	0.3934	0.4372		0.153

(a) Conversion factors, pre-digestion wet settled masses, and the weight fraction of UDS on a settled mass basis for the MCC samples were not determined as part of the sieving process/procedure.

Note: The LCC sample (TI008-LA) was not acid-digested.

5.8.1 ICP-OES Analysis

Table 5.20 and Table 5.21 summarize the ICP-OES AOI results and opportunistic analyte results, respectively. The reporting mass basis is relative to the dry solids mass submitted to the acid digestion step. Also reported are the process batch QC sample results. All samples were analyzed in one process batch. The RPDs were calculated from the MCC sample and duplicate as well as the SCC sample and duplicate. A SCC sample (TI008-SC) was used as the process batch MS sample.

The ICP-OES measurements have been shown to have a general overall uncertainty of ±15%. Bracketed values indicate that concentrations were measured at less than the EQL but greater than the MDL; uncertainty for these values will likely be higher than 15%. The less-than concentrations were determined by multiplying the instrument detection limit by the sample-specific process factor; process factor components include the sample mass, final volume of digestate, and any dilutions. All samples were diluted 10× at the ICP-OES analytical workstation. Results are based on this additional 10× dilution with the exception of As, Sb, Se, and Tl where results are based on the undiluted digestate.

The RPDs for all analytes from the SCC sample and duplicate met the acceptance criterion of ±20%. The MCC sample and duplicate analytes Ba, Cr, Fe, Ni, Pb, and Zr exceeded the ±20% RPD criterion. All AOIs in the LCS met the accuracy acceptance criterion of ±20% (80 to 120% recovery). The MS sample met the ±20% accuracy acceptance criterion (80 to 120% analyte recovery) for all AOIs except Ca, Na, and Tl. The MS sample recoveries for Al, Fe, and U are listed as not recovered (NR) because the spiked quantity was less than 25% of the sample analyte concentration. Sources of error for these analytes are evaluated from the post-spike recoveries and the serial dilution (5× dilution of sample, where 10% difference is acceptable per the analysis procedure). Detection-limit requirements defined in the QAPjP/SAP were met for all AOIs, and/or the analyte was present in sufficient concentration that the detection limit was not challenged.

The mass balance was calculated to evaluate the overall consistency of the analytical data and to understand which analytes gravitate to the MCC versus SCC fractions. Table 5.22 shows the calculated AOI masses in the input container composite sample relative to the average analyte masses in the MCC and SCC fractions. The estimated input analyte mass (A_{input}) is based on the measured analyte concentrations (C) from each core sample multiplied by its dry mass fractionation (F) in the container composite and divided by the mass of the container composite (65 g, dry basis) according to Equation 5.3. See Table 4.10 for core sample concentrations on dry mass basis and Table 5.2 for the dry mass fractionation.

$$A_{input} = \frac{C_{A1} \times F_{A1} + C_{A4} \times F_{A4} + C_{B1} \times F_{B1} + C_{B3} \times F_{B3}}{65\,g} \quad (5.3)$$

The analyte masses of the MCC and SCC fractions were calculated by multiplying the average analyte concentration (Table 5.20) by the recovered mass in the sieve fraction (dry mass basis) (see Table 5.5). The ratio (sum/input) represents the summed analyte masses from the SCC and MCC fractions divided by the input mass. Ratios between 0.75 and 1.25 were considered to show reasonable and consistent mass balances. Most ratios were within these bounds (with the exception of Ag, Ba, Be, Pb, Sb, V, and Zr) indicating the mass balance was reasonably consistent.

Table 5.20. KW210-CC Sieve Fractions ICP-OES AOI Results, Dry Mass Basis (ASR 8824)

Sieve Fraction >	NA	MCC	MCC duplicate	SCC	SCC duplicate	Process Batch QC Results					
TI Sample ID >		TI008-MA	TI008-MB	TI008-SA	TI008-SB				TI008-SC		
RPL/ASO ID >	PB	11-0792-129-S	11-0792-129-D	11-0793-129-S	11-0793-129-D	MCC & MCC dup	SCC & SCC dup	LCS[b]	11-0793-129-MS[b]	11-0793-S Post Spike	11-0792-S Serial Dilution
(Analyte)	(µg/g)[a]	(µg/g)[a,b]	(µg/g)[a,b]	(µg/g)[a]	(µg/g)[a]	RPD[b]	RPD	% Recovery	% Recovery	% Recovery	% Difference
Ag	<1.5	[7.8]	<1.1	46.7	45.2	--	3.3	101	102	103	--
Al	<3.6	17,200	15,300	36,200	34,000	11.4	6.4	103	NR	NR	0.5
As	<7.5	[35]	[31]	182	167	--	8.1	89	90	100	--
Ba	[0.097]	141	316	81.4	82.0	76.7	0.7	102	101	101	1.0
Be	<0.052	6.44	6.62	15.9	15.2	2.8	4.7	103	102	99	4.7
Ca	<1.7	9,220	9,800	2,080	1,770	6.0	16.3	102	162	101	0.2
Cd	<1.2	36.6	38.1	51.5	45.5	3.9	12.4	106	107	92	---
Cr	<1.6	137	209	714	671	41.7	6.3	98	104	104	2.7
Fe	[3.8]	37,100	49,500	135,000	130,000	28.7	3.7	104	NR	NR	1.2
Mg	<0.48	675	808	1,090	981	17.9	10.5	103	119	98	2.0
Na	71.5	1,450	1,570	449	384	8.0	15.6	103	123	98	4.6
Ni	<2.0	99.2	149	214	194	40.2	9.8	106	107	106	2.6
Pb	<16	175	260	925	936	39.1	1.2	108	107	114	--
Sb	<1.7	[5.8]	[4.1]	[15]	[17]	--	--	89	87	96	--
Se	<13	<12	<9.6	<19	<16	--	--	90	91	98	--
Tl	<6.7	<6.3	<4.8	[15]	[12]	--	--	86	78	89	--
U	<9.4	30,300	25,000	172,000	160,000	18.9	7.3	102	NR	102	0.7
V	<0.86	[14]	16.6	58.2	56.0	--	3.9	103	103	97	--
Zn	[6.7]	250	240	505	474	4.1	6.4	100	103	103	4.5
Zr	<0.66	573	94.9	432	363	143.2	17.3	107	NR	102	1.0

(a) Results shown in brackets indicate that the analyte concentrations were >MDL but <EQL, and measurement uncertainties were >15%. Less-than values represent the instrument detection limit multiplied by the dilution process factors.

(b) Gray shading indicates that the target QC criterion was exceeded. The targeted RPD was ±20% and the LCS and MS targeted recoveries were 80% to 120%.

NR: Not Recovered. This indicates the spike concentration was less than 25% of the sample concentration.

Dashed lines (--) indicate the measurement calculation was not applicable.

Table 5.21. KW210-CC Sieve Fractions ICP-OES Opportunistic Analyte Results, Dry Mass Basis (ASR 8824)

Sieve Fraction >	NA	MCC	MCC duplicate	SCC	SCC duplicate	Process Batch QC Results					
TI Sample ID >		TI008-MA	TI008-MB	TI008-SA	TI008-SB				TI008-SC	11-0793	11-0792
RPL/ASO ID >	PB	11-0792-129-S	11-0792-129-D	11-0793-129-S	11-0793-129-D	MCC & MCC dup	SCC & SCC dup	LCS	11-0793-129-MS	Post Spike	Serial Dilution
(Analyte)	(µg/g)[a]	(µg/g)[a,b]	(µg/g)[a,b]	(µg/g)[a]	(µg/g)[a]	RPD[b]	RPD	% Recovery	% Recovery	% Recovery	% Difference
B	[22]	[24]	[17]	[36]	[36]	--	--	104	105	101	--
Bi	[59]	[63]	[43]	[120]	[98]	--	--	--	--	92	--
Ce	<6.8	[25]	[17]	155	136	--	12.8	101	98	105	--
Co	<2.0	[4.2]	[3.3]	[5.4]	[3.9]	--	--	--	--	96	--
Cu	<1.3	423	403	1,920	1,820	4.7	5.5	110	NR	NR	0.5
Dy	<0.94	<0.88	<0.68	<1.4	<1.1	--	--	--	--	101	--
Eu	<0.21	[0.25]	[0.58]	4.09	4.41	--	7.6	--	--	102	--
K	[120]	461	479	541	470	3.7	14.0	105	109	99	--
La	<0.94	13.0	13.5	43.1	40.0	4.1	7.6	99	101	101	--
Li	5.39	5.83	5.25	14.1	11.8	10.5	17.6	113	108	105	--
Mn	<0.17	250	305	279	253	19.8	10.0	107	113	108	1.8
Mo	<5.5	[20]	[17]	[68]	[64]	--	--	105	109	105	--
Nd	<2.1	24.6	[13]	146	135	--	7.3	97	101	103	--
P	<40	[120]	[150]	819	734	--	10.9	--	118	96	--
Pd	<3.8	[4.6]	[3.1]	[6.9]	[6.8]	--	--	--	--	99	--
Rh	[7.8]	<6.5	<5.0	<9.9	<8.1	--	--	--	--	98	--
Ru	<3.4	<3.2	<2.5	[37]	[31]	--	--	--	--	101	--
S	<125	9,130	9,230	2,260	1,940	1.1	15.5	--	99	102	0.6
Si	[13]	583	506	1,220	1,280	14.1	4.6	108	0[b]	98	3.4
Sn	<11	[22]	[29]	[61]	[68]	--	--	--	--	85	--
Sr	[0.025]	51.3	54.9	15.9	14.1	6.7	12.1	103	128	102	0.3
Ta	<15	<14	<11	<21	<17	--	--	--	--	98	--
Te	<13	<12	[13]	[52]	[26]	--	--	--	--	101	--
Th	<3.9	[34]	[26]	205	180	--	13.0	100	101	102	--
Ti	<0.41	221	394	453	400	56.1	12.5	106	107	112	2.1
W	<12	<11	<8.4	<17	[26]	--	--	--	--	98	--
Y	<0.20	[2.4]	2.97	11.3	10.7	--	5.7	--	--	98	--

(a) Results shown in brackets indicate that the analyte concentrations were >MDL but <EQL, and measurement uncertainties were >15%. Less-than values represent the instrument detection limit multiplied by the dilution process factors.

(b) Gray shading indicates that the RPD exceeded ±20% and uncharacteristic failure of the Si MS recovery.

NR: Not Recovered. This indicates the spike concentration was less than 25% of the sample concentration.

Dashed lines (--) indicate the measurement calculation was not applicable.

Table 5.22. Acid Soluble Analyte Distribution Between MCC (−2,000-µm /+500-µm) and SCC (−500-µm) Sieve Fractions Relative to the Calculated Input Composition

(Analyte)	Calculated KW210-CC-N (µg)	KW210-MCC (µg)	KW210-SCC (µg)	Ratio Sum/Input
Ag	[230]	[240]	986	[5.3]
Al	1,420,000	897,000	753,000	1.16
As	[4500]	[1800]	3,740	[1.2]
Ba	8,410	12,600	1,750	1.71
Be	526	360	334	1.32
Ca	477,000	525,000	41,400	1.19
Cd	2,700	2,060	1,040	1.15
Cr	22,000	9,550	14,900	1.11
Fe	4,480,000	2,390,000	2,840,000	1.17
Mg	53,100	40,900	22,300	1.19
Na	84,100	83,300	8,930	1.10
Ni	13,200	6,850	4,380	0.85
Pb	19,500	12,000	20,000	1.64
Sb	[450]	[270]	[340]	[1.4]
Se	<970	<610	<370	--
Tl	<550	<310	[290]	--
U	4,810,000	1,530,000	3,560,000	1.06
V	[820]	[840]	1220	[2.5]
Zn	21,800	13,500	10,500	1.10
Zr	16,200	18,400	8,530	1.66

Shaded ratios indicate mass balance divergence from the range of 0.75 to 1.25.

Bracketed mass values indicate the ICP-OES result was <EQL. Bracketed ratios indicate the ratio is calculated with a value that was <EQL.

Examples of analyte distribution from the data provided in Table 5.22 are as follows. Aluminum, beryllium, iron, and zinc are approximately evenly distributed between the MCC and SCC sieve fractions. Barium, calcium, and sodium are primarily in the MCC fraction. Cadmium, magnesium, nickel and zirconium split between fractions but were more heavily associated with the MCC fraction. Chromium, lead and uranium split between fractions but are more heavily associated with the SCC fraction. Silver appears to be heavily associated with the SCC fraction and measured 4 times higher than the calculated input. Note that these mass distributions only represent the acid-soluble materials. Some of these elements are likely also present in the UDS (see Section 5.8.3).

5.8.2 Radiochemistry Analysis

The radiochemistry analysis of the container composite acid digestates was limited to GEA and AEA screen.[(a)] The GEA and AEA screen results from each sieve fraction are shown in Table 5.23. All results are reported on a dry mass basis. The PB results are also provided for direct comparison to the sample results.

(a) The AEA screen measures $^{239+240}$Pu and the combined ^{238}Pu plus ^{241}Am. Curium isotopes are also measured and reported opportunistically.

Table 5.23. KW210-CC Sieve Fraction Radionuclide Analysis, Dry Mass Basis (ASR 8824)

Sieve Fraction >	NA	LCC	MCC	MCC Duplicate	SCC	SCC Duplicate	QC Sample Results			
TI Sample ID >		TI008-LA	TI008-MA	TI008-MB	TI008-SA	TI008-SB	MCC & MCC dup	SCC & SCC dup	BS recovery	MS recovery
RPL/ASO ID >	PB	11-0791	11-0792-129-S	11-0792-129-D	11-0793-129-S	11-0793-129-D	RPD	RPD	%	%
AEA Screen	µCi/g, Reference Date May 25, 2011									
$^{239+240}$Pu	4.43E-5	NA	2.17E+0	1.62E+0	4.56E+1	4.72E+1	29%	3%	100%	96%
^{238}Pu+^{241}Am	6.47E-5	NA	3.61E+0	2.56E+0	5.03E+1	5.05E+1	34%	1%	--	--
$^{243+244}$Cm[(a)]	1.36E-5	NA	5.03E-3	2.96E-3	<4.E-2	5.98E-2	52%	--	--	--
GEA	µCi/g, Reference Date January 11, 2011									
^{60}Co	<1.E-5	1.48E-1	9.10E-2	6.69E-2	2.07E-1	1.94E-1	31%	6%	--	--
^{134}Cs	<1.E-5	<6.E-4	<9.E-3	<1.E-2	<8.E-3	<8.E-3	--	--	--	--
^{137}Cs	3.98E-4	6.25E+1	2.96E+2	3.44E+2	2.08E+2	2.03E+2	15%	2%	--	--
^{152}Eu	<4.E-5	<6.E-4	<7.E-3	<7.E-3	<1.E-2	<1.E-2	--	--	--	--
^{154}Eu	<3.E-5	5.64E-2	1.52E-1	1.17E-1	1.52E+0	1.46E+0	26%	4%	--	--
^{155}Eu	<5.E-5	<9.E-3	<1.E-1	<1.E-1	2.55E-1	2.36E-1	--	8%	--	--
^{241}Am	<2.E-4	1.04E+0	2.45E+0	2.26E+0	3.94E+1	3.65E+1	8%	8%	--	--

(a) $^{243+244}$Cm is an opportunistic measure, and QC sample requirements do not apply.

The analyte concentration 1-σ uncertainties varied from 2% to 32%; see Appendix C for the full analytical report.

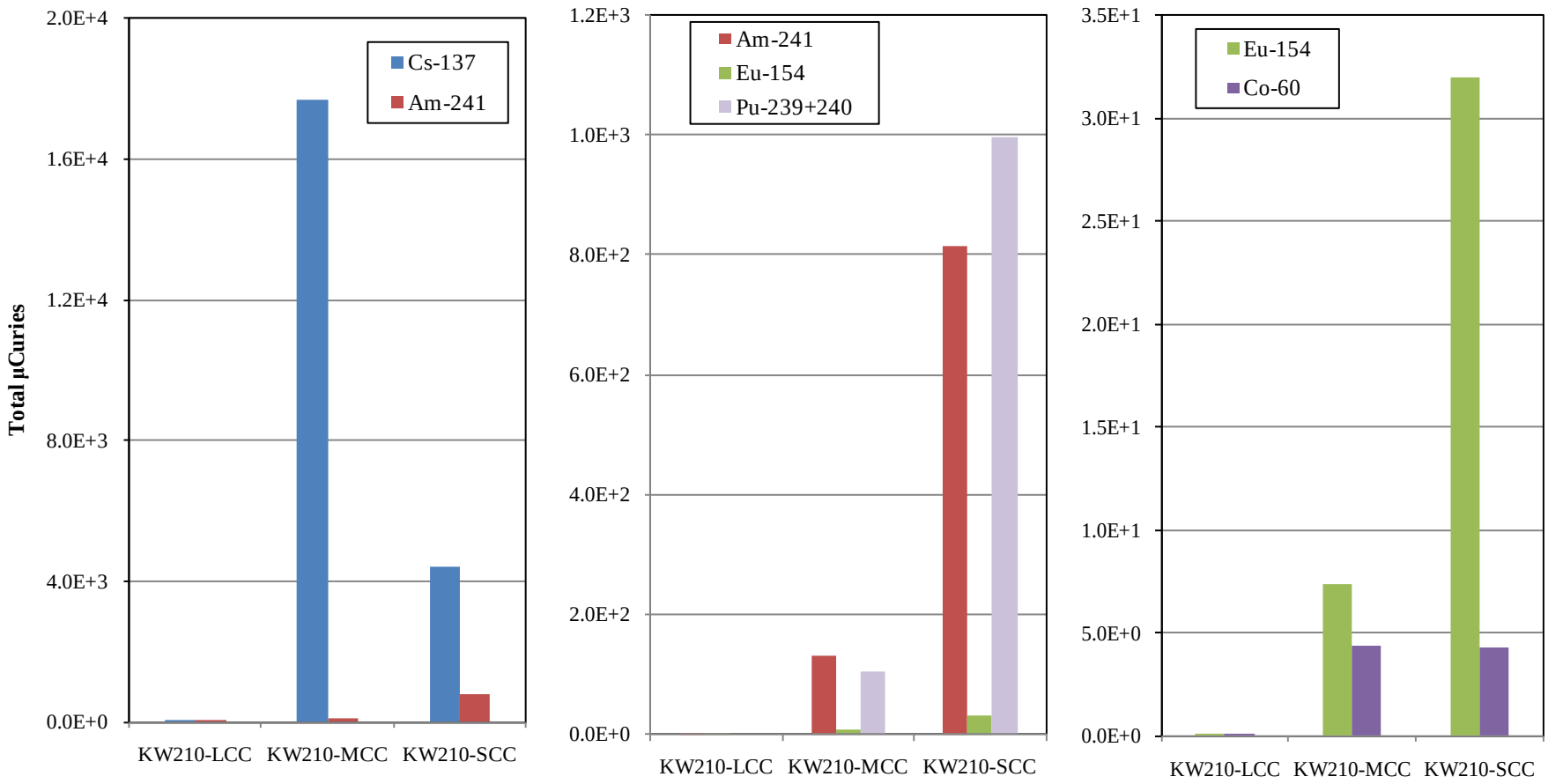
NA: Not Analyzed.

Dashed lines "--" indicates parameter does not apply.

Gray shading indicates that the precision criterion (±20% RPD for GEA and ±25% for AEA) was exceeded.

QC was evaluated from the PB concentration relative to the sample concentration, precision, and in the case of the AEA screen, post-MS and post-BS recoveries of ^{239}Pu. The PB results are shown in Table 5.23. In all cases, the analyte concentrations in the PB were a small fraction (<0.5%) of the sample analyte concentrations. The AOI RPDs calculated from the SCC sieve fraction duplicate samples met the ±20% acceptance criteria for GEA and ±25% acceptance criteria for the AEA screen. The RPDs for the MCC sieve fraction ranged from 8 to 52% demonstrating higher variability commensurate with the greater variability in particle size and type (see Figure 5.7). The AEA screen BS and MS ^{239}Pu recoveries (100 and 96%, respectively) met the 75 to 125% recovery acceptance criteria.

Figure 5.16 compares the total activity measured from each sieve fraction for various analytes. Comparisons are based on the dissolved analytes from the MCC and SCC sieve fractions and the gamma-emitted only analytes from the LCC sieve fraction. Total activity was calculated by multiplying the average reported concentration (µCi/g) by the total dry mass of the sieve fraction. The gamma emitters were measured from all sieve fractions ^{60}Co, ^{154}Eu, ^{137}Cs, and ^{241}Am. The $^{239+240}$Pu activity was not measured in the KW210-LCC sample. The ^{154}Eu, $^{239+240}$Pu, and ^{241}Am activities were concentrated in the SCC sieve fraction. The ^{137}Cs activity was concentrated in the MCC sieve fraction. The ^{60}Co content appeared to be equally split between the MCC and SCC sieve fractions.



- *$^{239+240}$Pu was not measured in the KW210-LCC fraction per direction of the BTR; there was insufficient LCC sample mass to accommodate multiple analyses.*
- *Any analyte that binds to the ion exchange resin beads during the acid digestion process would not be accounted for in this comparison.*

Figure 5.16. Analyte Content in KW210-CC-N Sieve Fractions

The MCC sieve fraction was substantially comprised of resin beads and the SCC fraction was comprised of a small fraction of resin beads (see Section 5.8.3). Any analyte that exchanges onto the resin beads during the acid digestion process would not be accounted for in this comparison. Analyte exchange onto ion exchange materials in nitric acid digestions was previously reported (Schmidt et al. 1999) and is further discussed in Section 5.8.3.

5.8.3 Undissolved Solids

The solids that did not dissolve during the PNL-ALO-129 acid digestion were measured by GEA to assess the efficacy of the acid digestion and associated extraction of radionuclides from the solids phase. The GEA results are presented, along with the fractionation remaining with the UDS, in Table 5.24. Maximums of 0.71% of the ^{137}Cs and 0.59% of the ^{241}Am were found in the UDS.

Table 5.24. Radionuclide Results in KW210-CC-N Sieve Fraction UDS, Dry Mass Basis (ASR 8824)

	KW210-MCC		KW210-SCC	
Sieve Fraction:	−2,000 μm / +500 μm		−500 μm	
Sample ID:	TI008-MA	TI008-MB	TI008-SA	TI008-SB
RPL/ASO ID:	11-0792-129-S	11-0792-129-D	11-0793-129-S	11-0793-129-D
UDS mass, g:	1.1750	1.5186	0.4087	0.5009
Analyte	(μCi/g)	(μCi/g)	(μCi/g)	(μCi/g)
As-Measured μCi/g in the UDS, Reference Date: January 11, 2011				
^{60}Co	4.08E-4	2.89E-4	1.12E-3	7.98E-4
^{137}Cs	1.19E+0	2.89E+0	5.83E-1	6.13E-1
^{154}Eu	9.22E-4	1.77E-3	1.30E-3	1.22E-3
^{241}Am	1.68E-2	1.46E-2	3.65E-2	3.30E-2
Relative Amount of Analyte in the UDS				
^{60}Co	0.38%	0.37%	0.25%	0.19%
^{137}Cs	0.35%	0.71%	0.13%	0.14%
^{154}Eu	0.52%	1.3%	0.039%	0.038%
^{241}Am	0.59%	0.55%	0.043%	0.042%

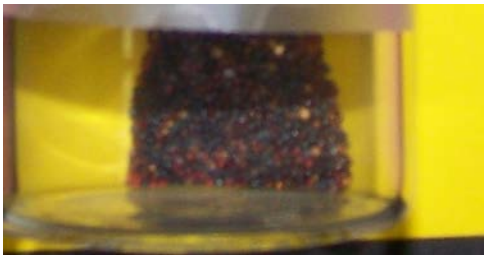
All analyte concentrations are presented on a dry mass basis.

Precision criteria do not apply to the GEA analysis of the UDS.

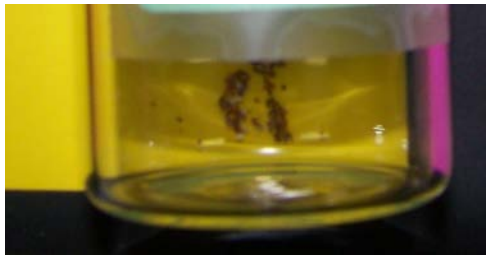
The analyte concentration 1-σ uncertainties varied from 3 to 7%; see Appendix C for the full analytical report.

The BTR directed PNNL to perform additional analysis of the UDS, including fusion dissolution followed by ICP-OES and AEA screen analysis. To prepare for this analysis, the UDS were subsampled using the Y-splitter in a cascade to create small subsamples appropriate for fusion.

One of the splits from each subsample was fractionated by panning off the rolling spherical resin beads into a vial separate from the non-rolling/non-resin material. The goal of the fractionation process was to provide a sample for XRD analysis that contained minimal resin beads and thus minimize the amorphous component background in the powder diffraction pattern. Masses from each fraction were collected. The fractionation was not perfect; some cross-contamination between resin and non-resin components was observed (see Figure 5.17 for examples). However general trends in the approximate mass distribution were recorded. The approximate OIER mass fractions in the UDS samples are provided in Table 5.25. The calculated OIER mass fraction in the sample (before acid digestion) was calculated according to Equation 4.2 and is provided in Table 5.25. This calculation approach assumes no resin mass was lost during the acid digestion processing.

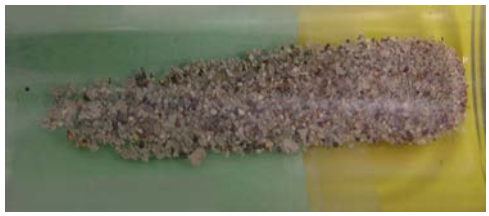


TI040-792S-2r



TI040-793S-2r

TI040-792S-2x
−2,000-µm /+500-µm sieve Fraction
Parent sample TI008-MA



TI040-793S-2x
−500-µm sieve Fraction
Parent sample TI008-SA

Figure 5.17. KW210-CC-N UDS Phase Split Fractions (top OIER; bottom non-OIER fraction)

Table 5.25. Dry Mass Fractionation of OIER in the KW210-CC-N Sieve Fractions

Sieve Fraction	Parent Sample ID	OIER in UDS, wt%	Calculated OIER in Dry Parent Sample, wt%
−500 µm	TI008-SA	2.3	1.1
	TI008-SB	3.7	1.7
		48% RPD	48% RPD
−2,000 µm/+500 µm	TI008-MA	51	44
	TI008-MB	49	42
		3.4% RPD	4.1% RPD

One subsample from each non-resin material sieve fraction was analyzed by XRD. The XRD patterns are compiled in Figure 5.18. Each XRD sample consisted primarily of silica quartz (SiO_2) and anorthite, $Ca(Al_2Si_2O_8)$. Table 5.26 lists the crystalline phases identified in the UDS, along with the sample progenitor IDs, ICDD card number, and relative intensities. All reflections were identified.

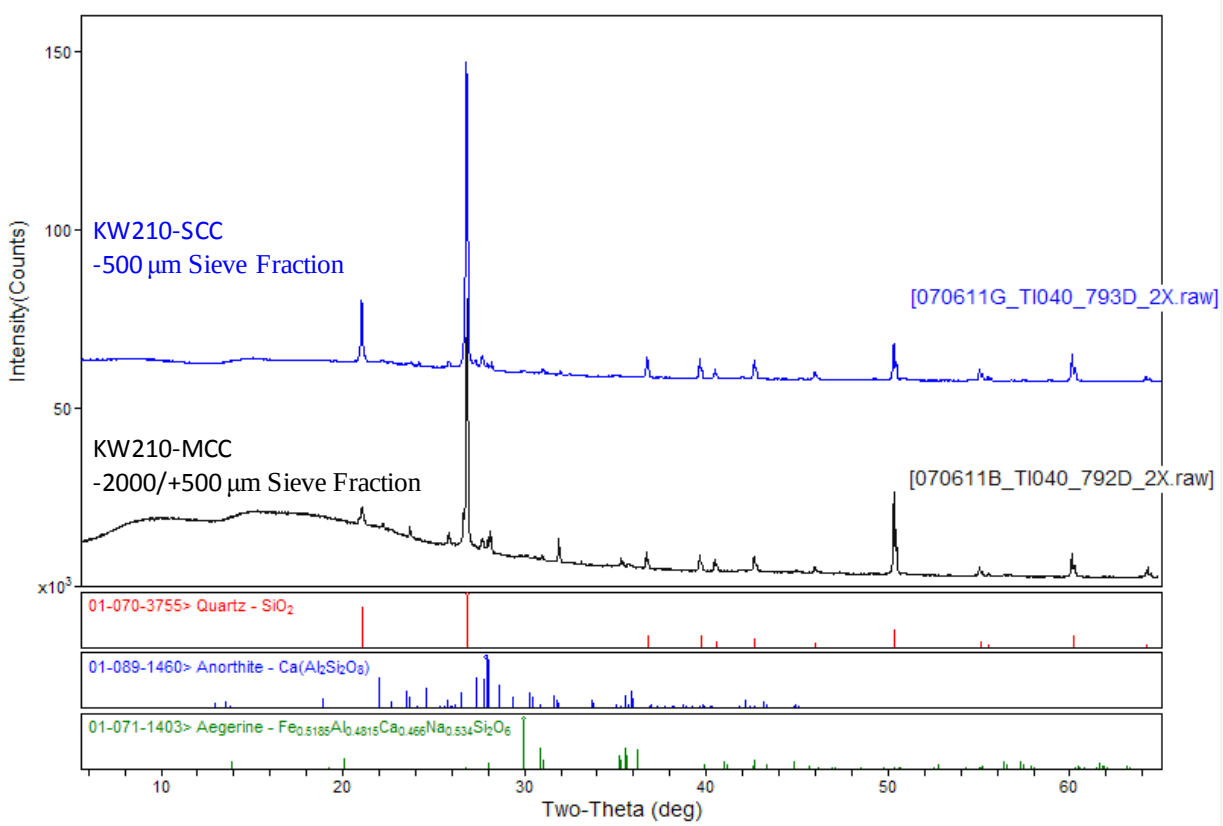


Figure 5.18. XRD Patterns of KW210-CC-N Container Composite Sieve Fraction Sample UDS

Table 5.26. Summary of Crystalline Phases Found via X-Ray Powder Diffraction on UDS Following Acid Digestion and Bulk OIER Removal from KW210-CC-N Sieve Fraction Samples

XRD Sample ID	(RPL/ASO Parent ID) Sieve Fraction	ICDD Card Number	Relative Intensity[(a)]	Formula	Common Phase Name
TI040-792D-2x	(11-0792-129-D) −2,000 µm/ +500 µm	01-070-3755	Major	SiO_2	Quartz
		01-089-1460	Major	$Ca(Al_2Si_2O_8)$	Anorthite
		01-071-1403	Minor	$Ca_{0.466}Fe_{0.5185}Al_{0.815}Na_{0.534}O_6Si_2$	Aegerine
TI040-793D-2x	(11-0793-129-D) −500 µm	01-070-3755	Major	SiO_2	Quartz
		01-089-1460	Major	$Ca(Al_2Si_2O_8)$	Anorthite
		01-071-1403	Minor	$Ca_{0.466}Fe_{0.5185}Al_{0.815}Na_{0.534}O_6Si_2$	Aegerine

(a) Crystalline components above 10 wt% are classified as major phases, between 1-10 wt% as minor phases, and under 1 wt% as possible phases. The XRD report is provided in Appendix F.

The UDS subsample splits (containing OIER) from the container composite MCC and SCC sieve fraction samples were further split into small representative aliquots applicable for KOH/KNO_3 fusion (0.1 to 0.2 g). After fusion the digestates were split. One split was analyzed by ICP-OES for AOIs Al, Ca, Fe, S, Si, U, and Zr; the other split was analyzed for gross alpha and AEA screen for a $^{239+240}Pu$ measurement. The fusion and analyses were conducted according to ASR 8878 and Special Instructions (Appendix D).[(a)] This analysis was supplemental scope beyond that defined in the QAPjP/SAP. The QC sample results were briefly discussed in Section 4.8; more detail can be found in Appendix D.

Table 5.27 provides the AOI results and their relative mass and activity fractionations to the UDS (results of ICP-OES opportunistic analytes and total alpha can be found in Appendix D). Analyte fractionation to the UDS was calculated according to Equation 4.1 where C_{UDS} was determined from the analysis of the fused sample. The pre-digest dry mass (g_S) and UDS mass (g_{UDS}) values are reported in Table 5.19 and the concentrations of analytes in the acid digestate (C_S) are reported in Table 5.20. The companion figure, Figure 5.19, presents the Al, Fe, S, U and Pu retention in the UDS as a function of the OIER content in the dry parent sample (before subjection to PNL-ALO-129 acid digestion, see Table 5.25). In Figure 5.19, the MCC and SCC sample results are overlaid on the core sample results (Figure 4.17). All of the relationships regarding analyte retention in UDS described in Section 4.8 were confirmed by the additional container composite sample data, with the exception that the MCC fraction appeared to result in an enhanced U retention (data points well above the trend line).

The direct relationship between the OIER content in the parent sample material to Fe, U, and Pu retention in the UDS suggests that a fraction of the analyte anionic or cationic species exchanged onto the OIER during the PNL-ALO-129 acid digestion and solids rinse steps. Similarly, the exchange of analytes onto OIER was reported previously with increases in Fe and $^{239+240}Pu$ concentrations by factors of 17 and 290, respectively, when a K Basin OIER (NRW-37) was exposed to a 6 M HNO_3 dissolver solution at 95°C for 7 hr (Schmidt et al. 1999). In this same 1999 study, however, the U concentration increased by only a factor of 1.07. In contrast, the present work (8 M HNO_3 combined with equal volume of 8 M HCl dissolver solution) resulted in a U retention in UDS that generally tracked well with the $^{239+240}Pu$ retention – with the exception of the OIER-rich MCC sample where U retention exceeded Pu retention by a factor of ~2.6 (see Figure 5.19). Also Fe retention was greater than Pu retention. Acid type and strength are known to affect the affinity of specific cationic/anionic analyte exchange onto OIER (Coleman 1965). Therefore, the fraction of Fe, U, and $^{239+240}Pu$ presented in Table 5.27 likely does not represent the analyte fractions in the sludge OIER currently contained in SCS-CON-210 because these analytes were not dissolved *in-situ* and the contacting medium is essentially pH 7 water (i.e., not acidic). Additional analyses of undigested OIER would be required to confirm this assessment.

(a) The UDS associated with the core samples were also processed as part of ASR 8878. Core sample 11-0420-129-D and container composite sample 11-0793-129-D were split and run as batch duplicates. All process QC samples were shared between the core and container composite samples.

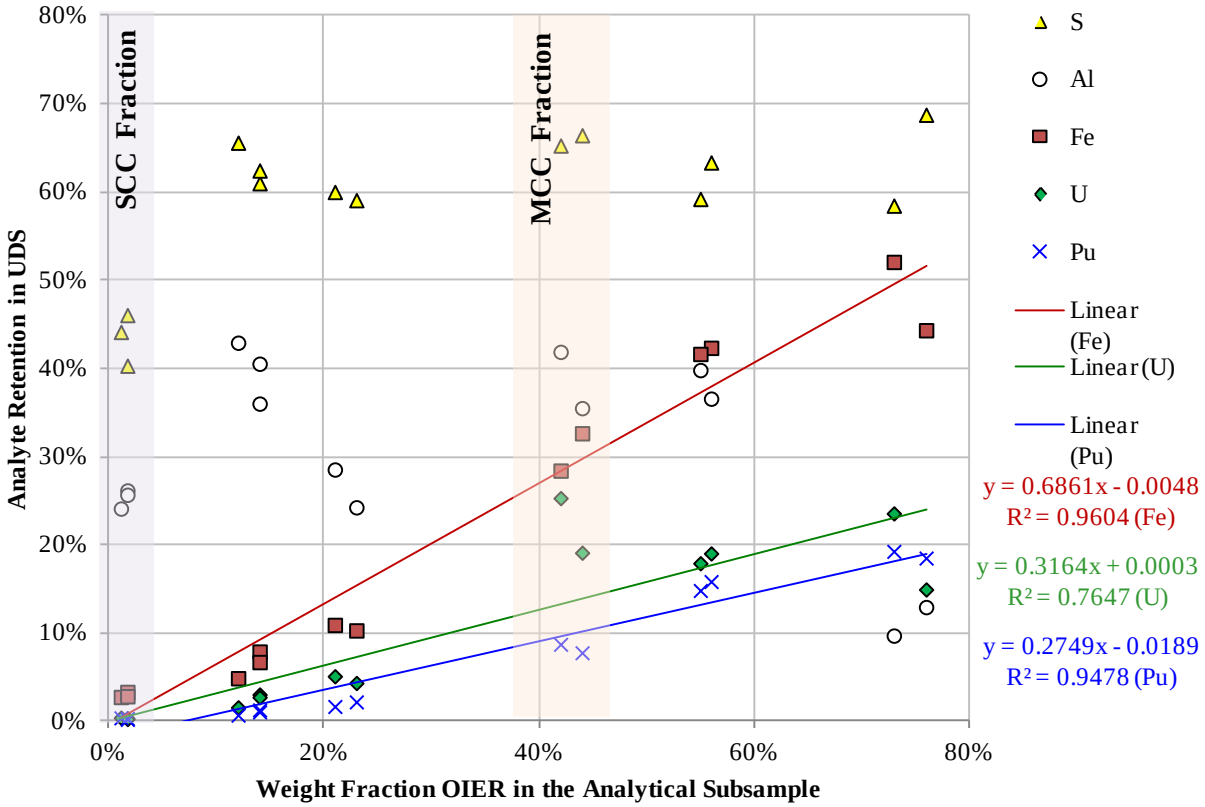
Table 5.27. Analytes in UDS Fusion, Dry UDS Mass Basis (ASR 8878)

		KW210-MCC		KW210-SCC		
Sieve Fraction:		−2,000 µm / +500 µm		−500 µm		
Parent Sample ID:		TI008-MA	TI008-MB	TI008-SA	TI008-SB	TI008-SB (Dup.)
Parent RPL/ASO ID:		11-0792-129-S	11-0792-129-D	11-0793-129-S	11-0793-129-D	11-0793-129-D
Daughter Sample ID:		TI040-0792S-1c	TI040-0792D-1c	TI040-0793S-1a	TI040-0793D-1b	TI040-0793D-2b
Daughter RPL/ASO ID:	BLK-1279	11-1289-115-S	11-1290-115-S	11-1287-115-S	11-1288-115-S	11-1288-115-D
Analyte		(µg/g) or (µCi/g)				
Al	<1,400	[11,000]	12,900	25,000	26,200	26,900
Ca	<1,500	<1,400	[2,200]	[3,000]	[3,100]	[3,200]
Fe	<570	20,900	23,000	8,360	9,780	8,750
S	<480	21,000	20,300	3,880	[3,600]	[3,000]
Si	<1,600	189,000	189,000	309,000	375,000	405,000
U	<31	8,320	9,910	1,610	1,270	1,040
Zr	<38	[37]	<27	[190]	[240]	[190]
$^{239+240}$Pu	6.83E-5	2.13E-1	1.82E-1	4.10E-1	3.45E-1	2.39E-1
Relative Amount of Analyte in the UDS						
Al	--	[36%]	42%	24%	26%	26%
Ca	--	NA	[16%]	[40%]	[45%]	[44%]
Fe	--	33%	28%	2.8%	3.3%	2.9%
S	--	66%	65%	44%	[46%]	[40%]
Si	--	NA	NA	NA	NA	NA
U	--	19%	25%	0.43%	0.36%	0.28%
Zr	--	[5.3%]	NA	[17%]	[23%]	[19%]
$^{239+240}$Pu	--	7.8%	8.7%	0.41%	0.34%	0.23%

Analyte results shown in brackets indicate that the analyte concentrations were greater than the MDL but less than the EQL. Less-than values represent the instrument detection limit multiplied by the process factors. See Appendix D for measurement uncertainties.

Percent values in brackets indicate one of the input parameters was <EQL but >MDL.

NA: Not Applicable; an input factor was <MDL or, in the case of Si, the analyte was not expected to dissolve in the PNL-ALO-129 acid digestion.



- *Pu, U, and Fe increased in the UDS as OIER content increased in the parent sample.*
- *Al and S in the UDS showed no relationship to the OIER content.*
- *Linear trend lines are provided for illustrative purposes and do not attempt to represent a model fit.*
- *The MCC and SCC sample results are overlaid on the core sample results.*

Figure 5.19. Relationship of Al, Fe, S, U, and Pu to OIER Content in the Parent Analytical Sample

The total relative activity and mass distributions of Pu, U, and Fe in the MCC and SCC sieve fraction UDS versus the acid digestates were compared. This comparison required the analyte concentration be multiplied by the mass of the component (i.e., the mass and activity balances were performed relative to the calculated sum of the SCC and MCC components). The reporting mass basis of the analyte concentration and the total component mass are summarized in Table 5.28.

Table 5.28. Sieve Fraction Samples Mass Basis used to Create Figure 5.20

	SCC		MCC		LCC
	Whole Sample	UDS	Whole Sample	UDS	Whole Sample
Dry Mass of Sieve Fraction	21.5 g	9.88 g	55.2 g	47.4 g	0.73 g
Sample Fraction Analyzed	Acid Digestate	UDS	Acid Digestate	UDS	Not Analyzed
Reporting Mass Basis of Analytes	Per Gram Dry Initial Sample	Per Gram Dry UDS	Per Gram Dry Initial Sample	Per Gram Dry UDS	Not Analyzed

Figure 5.20 compares the $^{239+240}$Pu, U, Fe and ^{137}Cs total analyte content in each sieve fraction (combined red and blue bars) relative to the analyte concentration that dissolved in the PNL-ALO-129 acid digestion (blue bars) and the normalized analyte concentration (see Equation 4.4) associated with the UDS (red bars). (Figure 5.20 is complimentary to Figure 5.16 and Table 5.22, with the enhancement that the UDS components and U and Fe distributions are incorporated.) The total Pu content was primarily in the SCC fraction with a very small total fraction associated with the UDS. Significant U and Fe content are associated with the MCC fraction. As previously noted, the ^{137}Cs is largely associated with the MCC fraction.

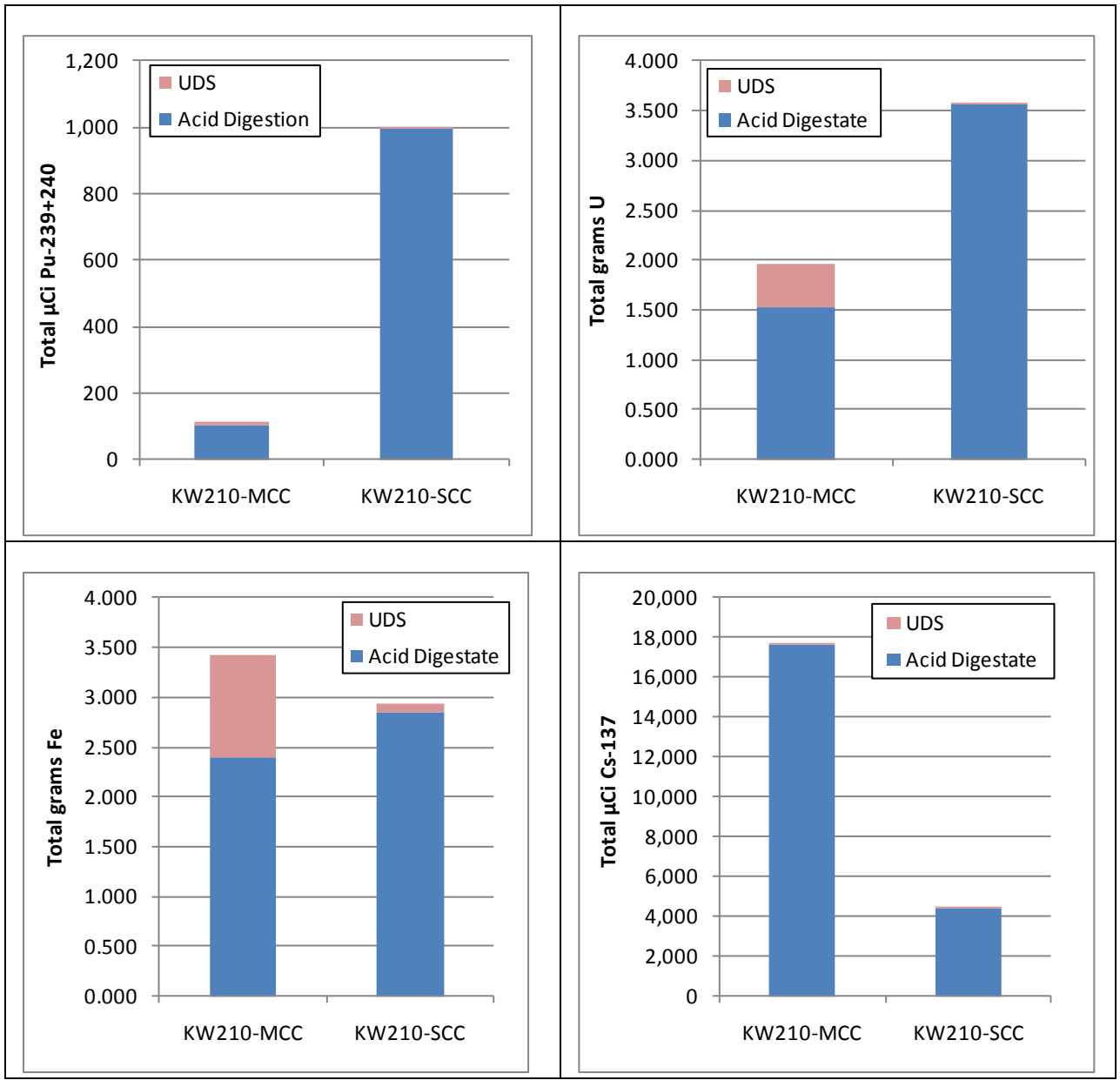


Figure 5.20. Total $^{239+240}$Pu, U, Fe and ^{137}Cs Content and Partitions Between Acid Digestate and UDS Phases for the MCC and SCC Sieve Fractions

An additional GEA analysis of the MCC sieve fraction samples was directed by the BTR to provide data on gamma activity in the resin bead fraction before acid dissolution. (This analysis was supplemental scope beyond that defined in the QAPjP/SAP.) Samples TI008-MK and TI008-ML (air dried, but not subjected to acid dissolution) were combined and the spherical particles (resin bead fraction) were separated from the non-spherical particles (i.e., irregular/granular-shaped particulate fraction). Particle separation was performed by placing the sample in the middle of a folded piece of paper, the paper was elevated, and spherical particles preferentially separated from less spherical particles via effects of gravity and friction. The samples were processed in this fashion over several iterations before effective separation was achieved. Some of the resin beads remained in the non-resin fraction as shown in Figure 5.21.

KW210-MK&ML Resin Beads KW210 MK & ML Residual Solids

Figure 5.21. Undigested KW210-MCC Sample Split for GEA

Table 5.29 provides the GEA analysis results on the resin bead and irregular/granular (residual) solids fractions under ASR 8919. See Appendix D for the ASR, analysis details, and measurement uncertainties.

The distribution of the total ^{137}Cs content between the resin and the non-resin components in the MCC fraction was assessed. The total resin component in the MCC fraction was derived from the mass fraction of the resin in the MCC sample (43% average, see Table 5.25) and the total measured MCC mass (55.2 g see Table 5.7). The non-resin mass component was calculated by difference. Each mass fraction was multiplied by the ^{137}Cs concentration assocatied with the fraction. The input data and ^{137}Cs distribtuion is summarized in Table 5.30. Only 10% of the ^{137}Cs is associated with the OIER. A component other than OIER apparently is associated with the bulk of the ^{137}Cs activity in the MCC fraction. Mordenite is an obvious consideration; however, crystalline phases of mordenite were not observed by XRD in the MCC sample (see Tables 5.10 and 5.26). However, anorthite is prominent in the XRD pattern of the digestion heel and is known to have a high affinity for ^{137}Cs with distribution coefficients, K_ds, of about 440 mL/g for sorption from a 7.9 pH, 0.03 N NaCl solution (Mucciardi et al. 1979). The sorption is slightly reversible with K_d of the desorption reaction about 44 mL/g using 5.13 N NaCl. More generally, studies of the sorption of ^{137}Cs onto Hanford soils have been summarized and shown to be high with K_ds, ranging around 1,000 mL/g, observed in tests with dilute solutions (Table 5 of Cantrell et al. 2003). However, the attribution of the high ^{137}Cs retention in the solid phase to anorthite is speculative and would require further experimentation to confirm.

Table 5.29. Radionuclide Results in KW210-MCC Sieve Fraction Separated Solids (Resin Beads and Irregular/Granular Particulate), Dry Mass Basis (ASR 8919)

	KW210-MCC	
Sieve Fraction:	−2,000 μm / +500 μm	
Sample ID:	TI008-MK and TI008-ML Combined	
RPL/ASO ID:	11-1555	11-1556
Fraction ID:	KW210 MK & ML Resin Beads	KW210 MK & ML Residual Solids[(b)]
Fraction mass, g[(a)]:	0.3049	0.3591
Analyte	(μCi/g)	(μCi/g)
GEA, μCi/g, Reference Date January 11, 2011		
^{60}Co	2.22E-2	7.60E-2
^{134}Cs	<4.E-3	<3.E-2
^{137}Cs	6.97E+1	4.56E+2
^{152}Eu	<7.E-3	<4.E-2
^{154}Eu	9.37E-2	<6.E-2
^{155}Eu	<8.E-2	<7.E-1
^{241}Am	7.76E-1	<2.E+0

(a) The combined mass of TI008-MK and TI008-ML equals 1.0305 g. Total mass recovery equals 64%. More details of the separation work can be found in LRB BNW-60605 pages 85-92 and are provided in DVD-52578-PNNL-20650 Rev. 2, Section III, September 2013.

(b) The residual solids are composed of irregular/granular particles of non-resin material.

All analyte concentrations are presented on a dry mass basis.

Precision criteria do not apply to the GEA analysis of the fraction-separated MCC sample.

The analyte concentration 1-σ uncertainties varied from 3 to 13%; see Appendix D for the full analytical report.

Table 5.30. ^{137}Cs Distribution in Components of the MCC Fraction

	55.2 g MCC Fraction	
Mass/Activity Balance Basis:	Resin component	Non-Resin component
^{137}Cs concentration	69.7 μCi/g	456 μCi/g
Mass of component	23.7 g	31.5 g
Total ^{137}Cs activity	1,650 μCi	14,300 μCi
^{137}Cs distribution in MCC	10%	90%

6.0 Records

Multiple record materials have been collected in the execution of the K Basin sludge processing and analysis. Records that are not ASO-specific are included in this report data package (in the appendices or DVD-52578-PNNL-20650 Rev. 2, Sections I-IV, September 2013). The contents of the DVD are stored in the PNNL records electronic database Total Records Information Management (TRIM) records system. The long-term storage of ASO-specific records are maintained in the National Archives and Records Administration (NARA) Federal Records Center (Seattle, Washington) (hardcopy), which in turn is based on DOE and other Federal records management requirements. This section describes where records are located and how they can be retrieved if not specifically included in this data package.

6.1 ASO Records

Sample results and supporting data are maintained in the ASO records. The records are inventoried by ASR number, PNNL project number, and RPL sample ID. The ASR and sample ID information, including the cross reference to client ID are maintained on the ASO sample tracking database (Microsoft Access database). In accordance with PNNL records management requirements, ASO records are prepared for transmittal to long-term records storage on a periodic basis, and upon project close-out. Records are boxed and the transmittal paperwork includes an inventory of the items in each box. This inventory includes a description of the record type, associated ASR number and the data type included (i.e., ICP-OES results for ASR XXXX). The ASO database is updated to reference the transmittal box ID to the associated ASR and data type sent in that transmittal. (Note: All records submitted for storage created by PNNL under the Battelle/DOE Operating Contract [DE-ACO5-76RL01830] are transferred to the NARA Federal Records Center in Seattle, Washington.) The ASO records include (but are not limited to) the following:

- bench sheets (digestion, dilutions, separation, run log records)
- analysis records (computer-generated printouts for counting instruments, ICP-OES)
- calibration records (certificates, initial and continuing calibrations, control charts)
- calculation of data to produce final summary data reports
- QE surveillance reports.

Table 6.1 summarizes the relevant record identifiers that will aid in record retrieval and points of contact/position titles that can facilitate records recovery.

6.2 Isotopic Distribution Mass Spectrometry Records

Radiochemical purification of the U and Pu fractions were conducted by the ASO; records specific to this process are handled as discussed in Section 6.1. All other records supporting the U and Pu TIMS analyses are provided in the data package, Appendix E. These include final analysis reports, sample run logs, standard certificates, computer-generated result printouts, and data calculations.

Table 6.1. Records Summary and Contacts for Retrieval

Laboratory Name	PNNL Project Number	ASR Number	RPL Numbers
Pacific Northwest National Laboratory	52578	8798	11-0419 – 11-0422
		8824	11-0791 – 11-0967
		8878	11-1279 – 11-1290
		8919	11-1555 – 11-1556
Contacts for Records Retrieval			
Position		Name	Contact Information
Project Manager for Project 52578		Clark Carlson	509-375-5334 clark.carlson@pnnl.gov
ASO Lead		Karl Pool	509-375-5246 karl.pool@pnnl.gov
ASO Administrator		Truc Trang-Le	509-375-5457 truc.trangle@pnnl.gov
PNNL Records Management; Records Storage and Retrieval		Terri Mooney	509-375-3798 terri.mooney@pnnl.gov

6.3 XRD Records

Most XRD records are associated with the powder diffraction computer files. These are processed on the computer running Jade software; final output data are images of raw and processed spectra. Other records include the analysis request form, the preparation record and the scan record. Original record printouts are maintained in the project file. Reproductions of these records are provided in Appendix F as part of the data package.

6.4 Particle Density Records

All records supporting the particle density measurements are provide in the data package, Appendix G. These include the bench sheets (calibration verification, sample mass and density calculations) and standard calibration certificates.

6.5 Rheology Records

All records supporting rheology measurements are provide in the data package, Appendix H. These include the bench sheets for performance verifications and standard calibration certificates.

6.6 Particle Size Distribution Records

All records supporting particle size distribution measurements are provided in the data package, Appendix I. These include the bench sheets for performance verifications and standard calibration certificates.

7.0 References

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Appendix A

Completed Chain of Custody Forms

Appendix A

Completed Chain of Custody Forms

This appendix contains images of the completed chain-of-custody (COC) forms submitted with each polybottle pair containing K Basin sludge. Table A.1 provides a cross-reference of the sample identification (ID) to the COC number, the sample date, and the receipt date at the Radiochemical Processing Laboratory in the order of sample date.

All eleven of the COC documents received with KW210 core sample bottles incorrectly listed Quality Assurance Project Plan/Sampling and Analysis Plan (QAPjP/SAP) KBC-40467. The correct QAPjP/SAP for sludge in the Engineered Container SCS-CON-210 is KBC-33786 Rev. 2 (Baker et al. 2009).

Table A.1. Listing of Individual COC Forms in Appendix A

Client Sample ID	COC No.	Sample Date	Receipt Date
KW210-A1-A	6695	12/29/10	1/4/11
KW210-A1-B	6695	12/29/10	1/4/11
KW210-A1-C	6697	12/30/10	1/4/11
KW210-A1-D	6697	12/30/10	1/4/11
KW210-A4-A	6662	11/17/10	11/18/10
KW210-A4-B	6662	11/17/10	11/18/10
KW210-A4-C	6664	11/18/10	12/2/10
KW210-A4-D	6664	11/18/10	12/2/10
KW210-A4-E	6668	11/19/10	12/6/10
KW210-A4-F	6668	11/19/10	12/6/10
KW210-B1-A	6674	12/1/10	12/8/10
KW210-B1-B	6674	12/1/10	12/8/10
KW210-B1-C	6678	12/7/10	12/14/10
KW210-B1-D	6678	12/7/10	12/14/10
KW210-B1-F[(a)]	6682	12/9/10	12/16/10
KW210-B1-G	6682	12/9/10	12/16/10
KW210-B3-A	6686	12/15/10	12/27/10
KW210-B3-B	6686	12/15/10	12/27/10
KW210-B3-C	6689	12/17/10	12/27/10
KW210-B3-D	6689	12/17/10	12/27/10
KW210-B3-E	6692	12/27/10	12/29/10
KW210-B3-F	6692	12/27/10	12/29/10

(a) Polybottle ID KW210-B1-E was omitted in the sequence; no such sample was collected or provided.

KBC PROJECT CHAIN OF CUSTODY

COC No. 6695 Date 12/29/10 Field Logbook No. n/a

Point of Contact _____ Phone No. _____ MSIN X4-01

Delivered to: ☐ OS Engineer ☐ 1706 KE Counting Facility ☐ 222-S Lab ☐ WSCF ☒ Other 325 LAB

Sampled By R R Ramirez (Print) _____ (Sign)

See Sample Analysis Request for individual containers and analysis.

Sample Number	Location/Description	Sample Date	Sample Time	Matrix*	Comments
KW210-A1-A	SCS-CON-210-SL	12/29/10	1021	SL	
KW210-A1-B	SCS-CON-210-SL	12/29/10	1021	SL	

*Matrix

S	Soil	DS	Drum Solids
SE	Sediment	DL	Drum Liquids
SO	Solid	T	Tissue
SL	Sludge/Slurry	WI	Wipe
W	Water	L	Liquid
O	Oil	V	Vegetation
A	Air	X	Other
AF	Air Filter		

Special Instructions: PRESERVATIVE USED – NONE
ANALYSIS REQUIRED - REF KBC 40467
DISPOSAL PER KBC 40467

Samples Transferred to new COC:
☒ No ☐ Yes, new COC No. _____

CHAIN OF POSSESSION

Relinquished By	Date	Time	Received By	Date	Time
R R Ramirez	12-29-10	1037	Sample Cart	12-29-10	1037
Sample Cart	12-30-10	1007	R R Ramirez	12-30-10	1007
R R Ramirez	12-30-10	1022	SWB	12-30-10	1022
SWB	1-4-11	1740	Howard Sommerville	1-4-11	1740
Howard Sommerville	1-4-11	19:30	Bea Palma	1/4/11	19:30

FINAL SAMPLE DISPOSITION

Disposal Method _____

Disposed By: _____ (Print) _____ (Sign) _____ (Date) _____ (Time)

NOTE: This form is to accompany the sample(s) at all times.

BC-6000-560 (02/05)

KBC PROJECT CHAIN OF CUSTODY

COC No. 6697 Date 12/30/10 Field Logbook No. N/A

Point of Contact LANCE ROSS Phone No. 373-4494 MSIN X4-01

Delivered to: ☐ OS Engineer ☐ 1706 KE Counting Facility ☐ 222-S Lab ☐ WSCF ☒ Other 325 LAB

Sampled By R R RAMIREZ (Print) (Sign)

See Sample Analysis Request for individual containers and analysis.

Sample Number	Location/Description	Sample Date	Sample Time	Matrix*	Comments
KW210-A1-C	SCS-CON210/SL	12/30/10	1032	SL	
KW210-A1-D	SCS-CON210/SL	12/30/10	1032	SL	

*Matrix

S	Soil	DS	Drum Solids
SE	Sediment	DL	Drum Liquids
SO	Solid	T	Tissue
SL	Sludge/Slurry	WI	Wipe
W	Water	L	Liquid
O	Oil	V	Vegetation
A	Air	X	Other
AF	Air Filter		

Special Instructions: PRESERVATIVE USED - NONE
ANALYSIS REQ - RDF KBC 40467
DISPOSAL per KBC 40467

Samples Transferred to new COC:
☒ No ☐ Yes, new COC No. _____

CHAIN OF POSSESSION

Relinquished By	Date	Time	Received By	Date	Time
R R RAMIREZ	~~12-30-10~~ 12-30-10	1047	SAMPLE CART	12-30-10	1047
SAMPLE CART	1-4-11	1415	DE FUNK	1-4-11	1415
~~DE FUNK~~ SAMPLE CART 1-4-11					
DE FUNK	1-4-11	1435	SWB	1-4-11	1435
SWB	1-4-11	1740	Howard Summerville	1-4-11	1740
Howard Summerville	1-4-11	19:30	Ben Palma	1/4/11	19:30

FINAL SAMPLE DISPOSITION

Disposal Method _____

Disposed By: Print / Sign / Date / Time

NOTE: This form is to accompany the sample(s) at all times.

BC-6000-560 (02/05)

CHPRC 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 105KW, 100K Area
City, State, Zip Hanford Site, 99354
Contact Jim Mathews
Phone 509-373-4598

2. SHIP TO ☐ U.S. DEPT. OF ENERGY C/0
Company PNNL
Address 325 Building, 300 Area
City, State, Zip Hanford Site, Wa 99354
Attention Fran Steen/Tony DeGuia
Phone 376-3171 (High Level Rad Facility)

6. SHIPMENT AUTHORIZATION NUMBER LJ019

7. EMERGENCY RESPONSE
Telephone 509-373-0911
Emergency Response Guide(s) 163

HM 8. Proper Shipping Name: RQ Radioactive Material, transported under special arrangement, fissile excepted — PRI HAZ 7 | SUB HAZ NA | UN ID UN2919

9. No. Pkg.	Model Package	COC/Spec	Serial No.	Seal No.	Isotopes	C.S.I.	T.I.	Bq/Package	Gr. Wt. Kg.
1	SWB	Type A	*	*	Am-241, Pu-239, Pu-240, *	NA	<0.5	.1054 TBq	1213Kg

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

11.
☐ Highway Route Controlled Quantity
☐ Exclusive Use Shipment with instructions
☒ Placards Applied Radioactive
☒ Fissile Excepted, Grams 1.293
☒ UN ID Marking UN 2919

12. LABELS APPLIED
Radioactive Yellow - II NA

13. ADDITIONAL LABELS / MARKINGS
*Pu-241, Pu-238 source term based on Radcalc

HM 8. Proper Shipping Name: RQ Radioactive Material, transported under special arrangement, fissile excepted — PRI HAZ 7 | SUB HAZ NA | UN ID UN2919

9. No. Pkg.	Model Package	COC/Spec	Serial No.	Seal No.	Isotopes	C.S.I.	T.I.	Bq/Package	Gr. Wt. Kg.
1	SWB	Type A	*	*	Am-241, Pu-239, Pu-240, *	NA	<0.5	.1054 TBq	1213Kg

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

11.
☐ Highway Route Controlled Quantity
☐ Exclusive Use Shipment with instructions
☒ Placards Applied Radioactive
☒ Fissile Excepted, Grams 1.293
☒ UN ID Marking UN 2919

12. LABELS APPLIED
Radioactive Yellow - II NA

13. ADDITIONAL LABELS / MARKINGS
*Pu-241, Pu-238 source term based on Radcalc

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

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 LABELS / MARKINGS

14. Shipment DE-Ci: 0.132 (ICRP-72)

Shipment Totals	C.S.I.	T.I.	Bq/Package	Gr. Wt. Kg.
	NA	<0.5	.2108 TBq	2426Kg

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

Dose Rate @ 1 Meter from Surface of Package
☒ <0.005 or <0.5 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
☒ <Tbl. 2-2 HNF-5173 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. L100009 | Date 1-4-11

16. TRANSPORTER
Vehicle Number G8200631
DRIVER SIGNATURE / PRINT NAME Jason Summerell

17. RECEIVER
RECEIVER SIGNATURE / PRINT NAME Benjamin Palma | Date 1/4/11

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 James Lindholm | Print Name James Lindholm | On behalf of DOE-RL | Date 1/4/11 | Organization CSG

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

*Seal Nº - 060113 TID# - 23484 / 23485 *Seal Nº 060115 TID# 23486 / 23487

A-6004-858 (REV 0)

KBC PROJECT CHAIN OF CUSTODY

COC No. 6662 Date 11/17/10 Field Logbook No. HNF-V692

Point of Contact L. Ross Phone No. 373-4424 MSIN X4-01

Delivered to: ☐ OS Engineer ☐ 1706 KE Counting Facility ☐ 222-S Lab ☐ WSCF ☒ Other 325 LAB

Sampled By R R Ramirez (Print) (Sign)

See Sample Analysis Request for individual containers and analysis.

Sample Number	Location/Description	Sample Date	Sample Time	Matrix*	Comments
KW210-A4-A	SCS ~~RM 210~~ COV-210-SL	11/17/10	1348	SL	
KW210-A4-B	SCS COV-210 FSL	11/17/10	1348	SL	

*Matrix

S	Soil	DS	Drum Solids
SE	Sediment	DL	Drum Liquids
SO	Solid	T	Tissue
SL	Sludge/Slurry	WI	Wipe
W	Water	L	Liquid
O	Oil	V	Vegetation
A	Air	X	Other
AF	Air Filter		

Special Instructions: PRESERVATIVE USED - NONE
ANALYSIS REQUIRED - REF KBC-404-67
DISPOSAL PER KBC-40467

Samples Transferred to new COC:
☒ No ☐ Yes, new COC No. _____

CHAIN OF POSSESSION

Relinquished By (Print / Sign)	Date / Time	Received By (Print / Sign)	Date / Time
R R Ramirez	11/17/10 1407	SAMPLE CART	11/17/10 1407
SAMPLE CART	11-18-10 0950	DE FUNK	11-18-10 0950
DE Funk	11-18-10 1013	PIG PEN	11-18-10 1013
Pig Pen	11/18/10 2 PM	Doylec Gunter	11/18/10 2 15PM
Doylec Gunter	11/18/10 5 30	Mike Hughes	11/18/10 5:35 PM
Mike Hughes	11-18-10 7:25 PM	Dustin Blundon	725 PM ~~Date~~ Time 11-18-10 ~~Time~~ Date

FINAL SAMPLE DISPOSITION

Disposal Method _____

Disposed By: _____ (Print) _____ (Sign) _____ (Date) _____ (Time)

NOTE: This form is to accompany the sample(s) at all times.

BC-6000-560 (02/05)

CHPRC RADIOACTIVE SHIPMENT RECORD | Page 1 of 1 | 4. Ship Prepaid | 5. Via Motor

1. SHIP FROM U.S. DEPT. OF ENERGY C/O
Company CHPRC
Address 105KW, 100K Area
City, State, Zip Hanford Site, 99354
Contact Jim Mathews
Phone 509-373-4598

2. SHIP TO ☐ U.S. DEPT. OF ENERGY C/0
Company PNNL
Address 325 Building, 300 Area
City, State, Zip Hanford Site, Wa 99354
Attention Fran Steen/Tony DeGuia
Phone 376-3171 (High Level Rad Facility)

6. SHIPMENT AUTHORIZATION NUMBER LJ010

7. EMERGENCY RESPONSE
Telephone 509-373-0911
Emergency Response Guide(s) 163

HM 8. Proper Shipping Name: RQ Radioactive Material, transported under special arrangement, fissile excepted | PRI HAZ 7 | SUB HAZ NA | UN ID UN2919

9. No. Pkg.	Model Package	COC/Spec	Serial No.	Seal No.	Isotopes	C.S.I.	T.I.	Bq/Package	Gr. Wt. Kg.
1	SWB	Type A	060113	*	Am-241, Pu-239, Pu-240, *	NA	.5	.1054 TBq	1213Kg

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

11.
☐ Highway Route Controlled Quantity
☐ Exclusive Use Shipment with instructions
☒ Placards Applied Radioactive
☒ Fissile Excepted, Grams 1.293
☒ UN ID Marking UN 2919

12. LABELS APPLIED
Radioactive Yellow - II

13. ADDITIONAL LABELS / MARKINGS
*Pu-241, Pu-238 source term based on Radcalc

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

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 LABELS / MARKINGS

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

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 LABELS / MARKINGS

14. Shipment DE-Ci: 0.066 (ICRP-72)

Shipment Totals	C.S.I.	T.I.	Bq/Package	Gr. Wt. Kg.
	NA	.5	.1054 TBq	1213Kg

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
☒ <Tbl. 2-2 HNF-5173 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. 105 KW | Survey No. L1000136 | Date 11-18-10

16. TRANSPORTER
Vehicle Number G8200631
DRIVER SIGNATURE Michael Hughes | PRINT NAME Michael Hughes

17. RECEIVER
RECEIVER SIGNATURE Mike Hughes Dustin Rhinehart | PRINT NAME Michael Hughes Dustin Rhinehart | Date 11/18/10

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
James Lindholm	James Lindholm		11-18-10	CSG

19. AUTHORIZATION FOR SHIPMENT

AIR TRANSPORT CERTIFICATION	CARGO AIRCRAFT	PASSENGER AIRCRAFT	Pkg. Dimensions (cm)
☐ N/A	☐ Cargo Aircraft Only Labels Applied	☐ Ltd Qty ☐ Research/Medical Diagnosis ☐ <3 T.I. ☐ Human Medical Research	

20. OFFSITE AUTHORIZATION

Survey No.	Date Shipped	Routing	ETA
Approved for Shipment Offsite			Date

23309 / 23310

A-6004-858 (REV 0)

KBC PROJECT CHAIN OF CUSTODY

COC No. 6664 Date 11/18/10 Field Logbook No. HNF-V-692

Point of Contact L. ROSS Phone No. 373-4424 MSIN X4-01

Delivered to: ☐ OS Engineer ☐ 1706 KE Counting Facility ☐ 222-S Lab ☐ WSCF ☒ Other 325 LAB

Sampled By R R RAMIREZ (Print) (Sign)

See Sample Analysis Request for individual containers and analysis.

Sample Number	Location/Description	Sample Date	Sample Time	Matrix*	Comments
KW210-A4-C	SCS CON-210/SL	11/18/10	1359	SL	
KW210-A4-D	SCS CON-210/SL	11/18/10	1359	SL	

*Matrix

S	Soil	DS	Drum Solids
SE	Sediment	DL	Drum Liquids
SO	Solid	T	Tissue
SL	Sludge/Slurry	WI	Wipe
W	Water	L	Liquid
O	Oil	V	Vegetation
A	Air	X	Other
AF	Air Filter		

Special Instructions: PRESERVITIVE USED – NONE
ANALYSIS REQUIRED – REF KBC – 40467
DISPOSAL – PER KBC – 40467

Samples Transferred to new COC:
☒ No ☐ Yes, new COC No. _____

CHAIN OF POSSESSION

Relinquished By	Date	Time	Received By	Date	Time
RR RAMIREZ	11/18/10	1417	SAMPLE CART	11/18/10	1417
Sample cart	11-19-2010	10:55	Gcompean	11-19-2010	10:55
Gcompean	11-19-2010	11:00	Pig Pen	11-19-2010	11:00
Pig Pen	11/29/10	150 PM	SWB	11/29/10	152 PM
SWB	12/2/10	535	FRANK BURKSFIELD	12/2/10	537
FRANK BURKSFIELD	12/2/10	731 PM	Nicole Green	12-2-10	1930

FINAL SAMPLE DISPOSITION

Disposal Method _____

Disposed By: _____ (Print) _____ (Sign) _____ (Date) _____ (Time)

NOTE: This form is to accompany the sample(s) at all times.

BC-6000-560 (02/05)

CHPRC 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 105KW,100K Area
City, State, Zip Hanford Site, 99354
Contact Jim Mathews
Phone 509-373-4598

2. SHIP TO ☐ U.S. DEPT. OF ENERGY C/0
Company PNNL
Address 325 Building, 300 Area
City, State, Zip Hanford Site, Wa 99354
Attention Fran Steen/Tony DeGuia
Phone 376-3171 (High Level Rad Facility)

6. SHIPMENT AUTHORIZATION NUMBER: LJ011

7. EMERGENCY RESPONSE
Telephone 509-373-0911
Emergency Response Guide(s): 163

HM 8. Proper Shipping Name: RQ Radioactive Material, transported under special arrangement, fissile excepted — PRI HAZ 7 — SUB HAZ NA — UN ID UN2919

9. No. Pkg.	Model Package	COC/Spec	Serial No.	Seal No.	Isotopes	C.S.I.	T.I.	Bq/Package	Gr. Wt. Kg.
1	SWB	Type A	060115	*	Am-241, Pu-239, Pu-240, *	NA	0.5	.1054 TBq	1213Kg

10. Identify for Normal Form Only
Physical Form: Liquid
Chemical Form: Elemental

11.
☐ Highway Route Controlled Quantity
☐ Exclusive Use Shipment with instructions
☒ Placards Applied: Radioactive
☒ Fissile Excepted, Grams: 1.293
☒ UN ID Marking: UN 2919

12. LABELS APPLIED
Radioactive Yellow - II

13. ADDITIONAL LABELS / MARKINGS
*Pu-241, Pu-238 source term based on Radcalc

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

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 LABELS / MARKINGS

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

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 LABELS / MARKINGS

14. Shipment DE-Ci: 0.066 (ICRP-72)

Shipment Totals	C.S.I.	T.I.	Bq/Package	Gr. Wt. Kg.
	NA	0.5	.1054 TBq	1213Kg

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
☒ <Tbl. 2-2 HNF-5173 Limits

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

Bldg. 105kw | Survey No. L1000183 | Date 12-2-10

16. TRANSPORTER

Vehicle Number	DRIVER SIGNATURE	PRINT NAME
G8200631	[signature]	FRANK BURKSFIELD

17. RECEIVER

RECEIVER SIGNATURE	PRINT NAME	Date
Nicole Green/NicoleGreen		12-2-10

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
[signature]	James Lindholm		12-2-10	CSG

19. AUTHORIZATION FOR SHIPMENT

AIR TRANSPORT CERTIFICATION	CARGO AIRCRAFT	PASSENGER AIRCRAFT	Pkg. Dimensions (cm)
☐ N/A	☐ Cargo Aircraft Only Labels Applied	☐ Ltd Qty ☐ Research/Medical Diagnosis ☐ <3 T.I. ☐ Human Medical Research	

20. OFFSITE AUTHORIZATION

Survey No.	Date Shipped	Routing	ETA
Approved for Shipment Offsite			Date

A-6004-858 (REV 0)

*TID- 23313 | 23314

KBC PROJECT CHAIN OF CUSTODY

COC No. 6668 Date 11/19/10 Field Logbook No. HNF-V-692

Point of Contact L. ROSS Phone No. 373-4424 MSIN X4-01

Delivered to: ☐ OS Engineer ☐ 1706 KE Counting Facility ☐ 222-S Lab ☐ WSCF ☒ Other 325 LAB

Sampled By RR Ramirez (Print) (Sign)

See Sample Analysis Request for individual containers and analysis.

Sample Number	Location/Description	Sample Date	Sample Time	Matrix*	Comments
KW210-A4-E	SCS-CON 210/SL	11/19/10	1320	SL	STRM 11/19/10
KW210-A4-F	SCS-CON-210/SL	11/19/10	1320	SL	STRM 11/19/10

*Matrix

S	Soil	DS	Drum Solids
SE	Sediment	DL	Drum Liquids
SO	Solid	T	Tissue
SL	Sludge/Slurry	WI	Wipe
W	Water	L	Liquid
O	Oil	V	Vegetation
A	Air	X	Other
AF	Air Filter		

Special Instructions: PRESERVATIVE USED – NONE
ANALYSIS REQUIRED – REF KBC – 40467
DISPOSAL PER KBC – 40467
Samples Transferred to new COC:
☒ No ☐ Yes, new COC No. _____

CHAIN OF POSSESSION

Relinquished By		Received By	
Print: RR RAMIREZ Sign: RRR	Date: 11/19/10 Time: 1335	Print: SAMPLE CART Sign: Sample Cart	Date: 11/19/10 Time: 1335
Print: Sample Cart Sign: Sample Cart	Date: 11/29/10 Time: 1010	Print: DE Frank Sign: DE Frank	Date: 11/29/10 Time: 1010
Print: DE Frank Sign: DE Frank	Date: 11/29/10 Time: 1033	Print: PIG PEN Sign: PIG PEN	Date: 11-29-10 Time: 1033
Print: Pig Pen Sign: Pig Pen	Date: 12/6/10 Time: 945	Print: SWB Sign: SWB	Date: 12/6/10 Time: 950
Print: SWB Sign: SWB	Date: 12-6-10 Time: 5:30 PM	Print: FRANK BURKSFIELD Sign: FRANK BURKSFIELD	Date: 12-6-10 Time: 5:30PM
Print: FRANK BURKSFIELD Sign: FRANK BURKSFIELD	Date: 12-6-10 Time: 7:25 PM	Print: T. Smith Sign: T. Smith	Date: 12-6-10 Time: 7:25 pm

FINAL SAMPLE DISPOSITION

Disposal Method _____

Disposed By:

Print	Sign	Date	Time

NOTE: This form is to accompany the sample(s) at all times.

BC-6000-560 (02/05)

CHPRC 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 105KW,100K Area
City, State, Zip Hanford Site, 99354
Contact Jim Mathews
Phone 509-373-4598

2. SHIP TO ☐ U.S. DEPT. OF ENERGY C/O
Company PNNL
Address 325 Building, 300 Area
City, State, Zip Hanford Site, Wa 99354
Attention Fran Steen/Tony DeGuia
Phone 376-3171 (High Level Rad Facility)

6. SHIPMENT AUTHORIZATION NUMBER LJ012

7. EMERGENCY RESPONSE
Telephone 509-373-0911
Emergency Response Guide(s) 163

HM 8. Proper Shipping Name: RQ Radioactive Material, transported under special arrangement,fissile excepted — PRI HAZ 7 — SUB HAZ NA — UN ID UN2919

9. No. Pkg.	Model Package	COC/Spec	Serial No.	Seal No.	Isotopes	C.S.I.	T.I.	Bq/Package	Gr. Wt. Kg.
1	SWB	Type A	060113	*	Am-241,Pu-239,Pu-240,*	NA	0.5	.1054 TBq	1213Kg

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

11.
☐ Highway Route Controlled Quantity
☐ Exclusive Use Shipment with instructions
☒ Placards Applied Radioactive
☒ Fissile Excepted, Grams 1.293
☒ UN ID Marking UN 2919

12. LABELS APPLIED
Radioactive Yellow - II

13. ADDITIONAL LABELS / MARKINGS
*Pu-241,Pu-238 source term based on Radcalc

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

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 LABELS / MARKINGS

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

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 LABELS / MARKINGS

14. Shipment DE-Ci: 0.066 (ICRP-72)

Shipment Totals	C.S.I.	T.I.	Bq/Package	Gr. Wt. Kg.
	NA	0.5	.1054 TBq	1213Kg

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

Dose Rate @ 1 Meter from Surface of Package
☐ <0.005 or _____ mSv/hr
<0.5 or <0.5 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
☒ <Tbl. 2-2 HNF-5173 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-1000200 | Date 12-6-10

16. TRANSPORTER
Vehicle Number G8200631
DRIVER SIGNATURE [signature] PRINT NAME FRANK BURKSFIELD

17. RECEIVER
RECEIVER SIGNATURE [signature] PRINT NAME T. Smith
[signature] KDW504 Date 12-6-10

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
[signature]	James Lindholm		12-6-10	CSG

19. AUTHORIZATION FOR SHIPMENT

AIR TRANSPORT CERTIFICATION	CARGO AIRCRAFT	PASSENGER AIRCRAFT	Pkg. Dimensions (cm)
☐ N/A	☐ Cargo Aircraft Only Labels Applied	☐ Ltd Qty ☐ Research/Medical Diagnosis ☐ <3 T.I. ☐ Human Medical Research	

20. OFFSITE AUTHORIZATION

Survey No.	Date Shipped	Routing	ETA
Approved for Shipment Offsite			Date

A-6004-858 (REV 0)

*TID-23317/23318

KBC PROJECT CHAIN OF CUSTODY

COC No. 6674 Date 12/1/10 Field Logbook No. N/A

Point of Contact Lance Ross Phone No. 373-4494 MSIN X4-01

Delivered to: ☐ OS Engineer ☐ 1706 KE Counting Facility ☐ 222-S Lab ☐ WSCF ☒ Other 325 Lab

Sampled By Print DE Funk Sign DE Funk

See Sample Analysis Request for individual containers and analysis.

Sample Number	Location/Description	Sample Date	Sample Time	Matrix*	Comments
KW210-B1-A	SCS-Lon210/SL	12/1/10	1014	SL	
KW210-B1-B	SCS-Lon210/SL	12/1/10	1014	SL	

*Matrix

S	Soil	DS	Drum Solids
SE	Sediment	DL	Drum Liquids
SO	Solid	T	Tissue
SL	Sludge/Slurry	WI	Wipe
W	Water	L	Liquid
O	Oil	V	Vegetation
A	Air	X	Other
AF	Air Filter		

Special Instructions: Preservative Used - None
Analysis Req. Ref KBC 33786 or 40467
Disposal Per KBC-33786 or 40467
Samples Transferred to new COC:
☒ No ☐ Yes, new COC No. _____

CHAIN OF POSSESSION

Relinquished By		Received By	
Print DE Funk	Date 12/1/10	Print Sample Cart	Date 12/1/10
Sign DE Funk	Time 1036	Sign Sample Cart	Time 1036
Print SAMPLE CART	Date 12/7/10	Print DE Funk	Date 12/7/10
Sign SAMPLE CART	Time 0750	Sign DE Funk	Time 0950
Print DE Funk	Date 12/7/10	Print Pig Pen	Date 12/7/10
Sign DE Funk	Time 1008	Sign Pig Pen	Time 1008
Print Pig Pen	Date 12/8/10	Print SWB	Date 12/8/10
Sign Pig Pen	Time 922	Sign Swb	Time 930 AM
Print SWB	Date 12/8/10	Print FRANK BURKSFIELD	Date 12/8/10
Sign Swb	Time 515	Sign FBurksfield	Time 5:15
Print FRANK BURKSFIELD	Date 12/8/10	Print T. Smith	Date 12/8/2010
Sign FBurksfield	Time 1800	Sign	Time 1900

FINAL SAMPLE DISPOSITION

Disposal Method _____

Disposed By:

Print _____ Sign _____ Date _____ Time _____

NOTE: This form is to accompany the sample(s) at all times.

BC-6000-560 (02/05)

CHPRC 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 105KW, 100K Area
City, State, Zip Hanford Site, 99354
Contact Jim Mathews
Phone 509-373-4598

2. SHIP TO ☐ U.S. DEPT. OF ENERGY C/O
Company PNNL
Address 325 Building, 300 Area
City, State, Zip Hanford Site, Wa 99354
Attention Fran Steen/Tony DeGuia
Phone 376-3171 (High Level Rad Facility)

6. SHIPMENT AUTHORIZATION NUMBER LJ013

7. EMERGENCY RESPONSE
Telephone 509-373-0911
Emergency Response Guide(s) 163

HM 8. Proper Shipping Name: RQ Radioactive Material, transported under special arrangement, fissile excepted

PRI HAZ	SUB HAZ	UN ID
7	NA	UN2919

9. No. Pkg.	Model Package	COC/Spec	Serial No.	Seal No.	Isotopes	C.S.I.	T.I.	Bq/Package	Gr. Wt. Kg.
1	SWB	Type A	060115	*	Am-241, Pu-239, Pu-240, *	NA	0.5	.1054 TBq	1213Kg

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

11.
☐ Highway Route Controlled Quantity
☐ Exclusive Use Shipment with instructions
☒ Placards Applied Radioactive
☒ Fissile Excepted, Grams 1.293
☒ UN ID Marking UN 2919

12. LABELS APPLIED
Radioactive Yellow - II

13. ADDITIONAL LABELS / MARKINGS
*Pu-241, Pu-238 source term based on Radcalc

HM 8. Proper Shipping Name:

PRI HAZ	SUB HAZ	UN ID

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 LABELS / MARKINGS

HM 8. Proper Shipping Name:

PRI HAZ	SUB HAZ	UN ID

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 LABELS / MARKINGS

14. Shipment DE-Ci: 0.066 (ICRP-72)

Shipment Totals	C.S.I.	T.I.	Bq/Package	Gr. Wt. Kg.
	NA	<0.5	.1054 TBq	1213Kg

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
☒ <Tbl. 2-2 HNF-5173 Limits

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

Bldg. 105KW | Survey No. L-100215 | Date 12/8/10

16. TRANSPORTER

Vehicle Number	DRIVER SIGNATURE	PRINT NAME
G8200631	[signature]	FRANK BURKSFIELD

17. RECEIVER

RECEIVER SIGNATURE	PRINT NAME	Date
[signature]	T. Smith	12/8/2010

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
[signature]	James Lindholm		12-8-10	CSG

19. AUTHORIZATION FOR SHIPMENT

AIR TRANSPORT CERTIFICATION	CARGO AIRCRAFT	PASSENGER AIRCRAFT	Pkg. Dimensions (cm)
☐ N/A	☐ Cargo Aircraft Only Labels Applied	☐ Ltd Qty ☐ Research/Medical Diagnosis ☐ <3 T.I. ☐ Human Medical Research	

20. OFFSITE AUTHORIZATION

Survey No.	Date Shipped	Routing	ETA
Approved for Shipment Offsite			Date

*TID-23463/23319

A-6004-858 (REV 0)

KBC PROJECT CHAIN OF CUSTODY

COC No. 6678 Date 12/7/10 Field Logbook No. N/A

Point of Contact Lance Ross Phone No. 373-4494 MSIN X4-01

Delivered to: ☐ OS Engineer ☐ 1706 KE Counting Facility ☐ 222-S Lab ☐ WSCF ☒ Other 325 LAB

Sampled By DE Funk (Print) (Sign)

See Sample Analysis Request for individual containers and analysis.

Sample Number	Location/Description	Sample Date	Sample Time	Matrix*	Comments
KW210-B1-C	SCS-Con210 /SL	12-7-10	1356	SL	
KW210-B1-D	SCS-Con210/SL	12-7-10	1356	SL	
~~KW 210-B1-E~~ DE 12/8/10	~~SCS Con210 /SL~~	~~12-7-10~~	~~1356~~	~~SL~~	SAMPLE Discarded per NCR

*Matrix

S	Soil	DS	Drum Solids
SE	Sediment	DL	Drum Liquids
SO	Solid	T	Tissue
SL	Sludge/Slurry	WI	Wipe
W	Water	L	Liquid
O	Oil	V	Vegetation
A	Air	X	Other
AF	Air Filter		

Special Instructions: Preservative Used – None
Analysis Req. Ref. KBC-40467
Disposal Per. KBC 40467
Samples Transferred to new COC:
☒ No ☐ Yes, new COC No. _____

CHAIN OF POSSESSION

Relinquished By	Date	Time	Received By	Date	Time
DE Funk / DE Funk	12/7/10	1425	SAMPLE CART / SAMPLE CART	12/7/10	1425
SAMPLE CART / SAMPLE CART	12/9/10	0950	DE Funk / DE Funk	12/9/10	0950
DE Funk / DE Funk	12/9/10	1024	Pig Pen / Pig Pen	12/9/10	1024
Pig Pen / Pig Pen	12/14/10	955	SWB / SWB	12/14/10	1000
SWB / SWB	12/14/10	5-35	Howard Sydnerville / Howard Sydnerville	12/14/10	5-35
Howard Sydnerville / Howard Sydnerville	12-14-10	7:15 AM	Tim Smith / Tim Smith	12/14/10	1915 2015

FINAL SAMPLE DISPOSITION

Disposal Method _____

Disposed By:

Print _____ Sign _____ Date _____ Time _____

NOTE: This form is to accompany the sample(s) at all times.

BC-6000-560 (02/05)

CHPRC 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 105KW, 100K Area
City, State, Zip Hanford Site, 99354
Contact Jim Mathews
Phone 509-373-4598

2. SHIP TO ☐ U.S. DEPT. OF ENERGY C/0
Company PNNL
Address 325 Building, 300 Area
City, State, Zip Hanford Site, Wa 99354
Attention Fran Steen/Tony DeGuia
Phone 376-3171 (High Level Rad Facility)

6. SHIPMENT AUTHORIZATION NUMBER: LJ015

7. EMERGENCY RESPONSE
Telephone 509-373-0911
Emergency Response Guide(s): 163

HM 8. Proper Shipping Name: RQ Radioactive Material, transported under special arrangement, fissile excepted — PRI HAZ 7 | SUB HAZ NA | UN ID UN2919

9. No. Pkg.	Model Package	COC/Spec	Serial No.	Seal No.	Isotopes	C.S.I.	T.I.	Bq/Package	Gr. Wt. Kg.
1	SWB	Type A	06013	*	Am-241, Pu-239, Pu-240, *	NA	<0.5	.1054 TBq	1213Kg

10. Identify for Normal Form Only
Physical Form: Liquid
Chemical Form: Elemental

11.
☐ Highway Route Controlled Quantity
☐ Exclusive Use Shipment with instructions
☒ Placards Applied: Radioactive
☒ Fissile Excepted, Grams: 1.293
☒ UN ID Marking: UN 2919

12. LABELS APPLIED
Radioactive Yellow - II

13. ADDITIONAL LABELS / MARKINGS
*Pu-241, Pu-238 source term based on Radcalc

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

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 LABELS / MARKINGS

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

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 LABELS / MARKINGS

14. Shipment DE-Ci: 0.066 (ICRP-72)

Shipment Totals	C.S.I.	T.I.	Bq/Package	Gr. Wt. Kg.
	NA	<0.5	.1054 TBq	1213Kg

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

Dose Rate @ 1 Meter from Surface of Package
☐ <0.005 or N/A mSv/hr
<0.5 or <0.5 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
☐ <Tbl. 2-2 HNF-5173 Limits

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

Bldg. 105 KW | Survey No. L-1000240 | Date 12-14-10

16. TRANSPORTER

Vehicle Number	DRIVER SIGNATURE	PRINT NAME
G8200631	Howard Summerville	Howard Summerville

17. RECEIVER

RECEIVER SIGNATURE	PRINT NAME	Date
	T. Smith	12-14-10

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
James Lindholm	James Lindholm		12/14/10	CSG

19. AUTHORIZATION FOR SHIPMENT

AIR TRANSPORT CERTIFICATION	CARGO AIRCRAFT	PASSENGER AIRCRAFT	Pkg. Dimensions (cm)
☐ N/A	☐ Cargo Aircraft Only Labels Applied	☐ Ltd Qty ☐ Research/Medical Diagnosis ☐ <3 T.I. ☐ Human Medical Research	

20. OFFSITE AUTHORIZATION

Survey No.	Date Shipped	Routing	ETA

Approved for Shipment Offsite — Date

A-6004-858 (REV 0)

*TID - 23464 / 23465

KBC PROJECT CHAIN OF CUSTODY

COC No. 6682 Date 12/9/10 Field Logbook No. N/A

Point of Contact Lance Ross Phone No. 373-4494 MSIN X4-01

Delivered to: ☐ OS Engineer ☐ 1706 KE Counting Facility ☐ 222-S Lab ☐ WSCF ☒ Other 325 LAB

Sampled By D.E. Funk (Print) (Sign)

See Sample Analysis Request for individual containers and analysis.

Sample Number	Location/Description	Sample Date	Sample Time	Matrix*	Comments
KW210-B1-F	SCS-CON210/SL	12/9/10	1404	SL	
KW210-B1-G	SCS-CON210/SL	12/9/10	1404	SL	

*Matrix

S	Soil	DS	Drum Solids
SE	Sediment	DL	Drum Liquids
SO	Solid	T	Tissue
SL	Sludge/Slurry	WI	Wipe
W	Water	L	Liquid
O	Oil	V	Vegetation
A	Air	X	Other
AF	Air Filter		

Special Instructions: Preservative Used - None
Analysis Req. Ref. KBC-40467
Disposal Per. Ref. KBC-40467
Samples Transferred to new COC:
☒ No ☐ Yes, new COC No. _____

CHAIN OF POSSESSION

Relinquished By (Print / Sign)	Date / Time	Received By (Print / Sign)	Date / Time
DE Funk / DE Funk	12/9/10 / 1427	SAMPLE CART / SAMPLE CART	12/9/10 / 1427
SAMPLE CART / SAMPLE CART	12/14/10 / 1340	DE Funk / DE Funk	12/14/10 / 1340
DE Funk / DE Funk	12/14/10 / 1400	Pig Pen / Pig Pen	12/14/10 / 1400
Pig Pen / Pig Pen	12/16/10 / 935	SWB / SWB	12/16/10 / 940
SWB / SWB	12/16/10 / 545	Michael Hughes / Michael Hughes	12-16-10 / 546
Mike Hughes / Mike Hughes	12-16-10 / 1912	T. Smith / T. Smith	12-16-10 / 1913

FINAL SAMPLE DISPOSITION

Disposal Method _____

Disposed By: (Print / Sign / Date / Time)

NOTE: This form is to accompany the sample(s) at all times. BC-6000-560 (02/05)

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 105KW,100K Area
City, State, Zip Hanford Site, 99354
Contact Jim Mathews
Phone 509-373-4598

2. SHIP TO ☐ U.S. DEPT. OF ENERGY C/0
Company PNNL
Address 325 Building, 300 Area
City, State, Zip Hanford Site, Wa 99354
Attention Fran Steen/Tony Deguia
Phone 376-3171 (High Level Rad Facility)

6. SHIPMENT AUTHORIZATION NUMBER LJ016

7. EMERGENCY CONTACT INFO
Telephone 509-373-0911
ERG #s 163
Charge Code NA

8. HM UN ID Proper Shipping Name: PRI HAZ SUB HAZ
RQ UN2919 Radioactive Material, transported under special arrangement, fissile excepted 7 NA

9. No. Pkg.	Model Package	COC/Spec	Serial No.	Seal No.	Isotopes	C.S.I.	T.I.	Bq/Package	Gr. Wt. Kg.
1	SWB	Type A	060115	*	Am-241, Pu-239, Pu-240, *	NA	<0.5	.1054 TBq	1213Kg

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

11.
☐ Highway Route Controlled Quantity
☐ Exclusive Use Shipment with instructions
☒ Placards Applied Radioactive
☒ Fissile Excepted, Grams 1.293
☒ UN ID Marking UN 2919

12. LABELS APPLIED
Radioactive Yellow- II

13. ADDITIONAL INFO / LABELS / MARKINGS
*Pu-241, Pu-238 source term based on Radcalc

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:
0.066 (ICRP-72)

Shipment Totals	C.S.I.	T.I.	Bq/Package	Gr. Wt. Kg.
	NA	<0.5	.1054 TBq	1213Kg

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

Dose Rate @ 1 Meter from Surface of Package
☐ <0.005 or N/A mSv/hr
<0.5 or <0.5 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
☐ <Tbl. 2-2 HNF-5173 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. 105 KW | Survey No. L-1000250 | Date 12-16-10

16. TRANSPORTER
Vehicle Number G8200631
DRIVER SIGNATURE Michael Hughes
PRINT NAME Michael Hughes

17. RECEIVER
RECEIVER SIGNATURE
PRINT NAME T. Smith
Date 12-16-10

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 James Lindholm | Print Name James Lindholm | On behalf of DOE-RL | Date 12/16/10 | Organization C5G

19. AUTHORIZATION FOR SHIPMENT

AIR TRANSPORT CERTIFICATION	CARGO AIRCRAFT	PASSENGER AIRCRAFT	Pkg. Dimensions (cm)
☐ N/A	☐ Cargo Aircraft Only Labels Applied	☐ Ltd Qty ☐ Research/Medical Diagnosis ☐ <3 T.I. ☐ Human Medical Research	

20. OFFSITE AUTHORIZATION

Survey No.	Date Shipped	Routing	ETA

Approved for Shipment Offsite | Date

RTID- 23467 / 23466

A-6003-214.1 (REV 4)

KBC PROJECT CHAIN OF CUSTODY

COC No. 6686 Date 12/15/2010 Field Logbook No. N/A

Point of Contact Lance Ross Phone No. 373-4494 MSIN X4-01

Delivered to: ☐ OS Engineer ☐ 1706 KE Counting Facility ☐ 222-S Lab ☐ WSCF ☒ Other 325 Lab

Sampled By Adam Compean (Print) Adam Compean (Sign)

See Sample Analysis Request for individual containers and analysis.

Sample Number	Location/Description	Sample Date	Sample Time	Matrix*	Comments
KW210-B3-A	scs-con 210/SL	12-15-10	10:02	SL	
KW210-B3-B	scs-con210/SL	12-15-10	10:02	SL	

*Matrix

S	Soil	DS	Drum Solids
SE	Sediment	DL	Drum Liquids
SO	Solid	T	Tissue
SL	Sludge/Slurry	WI	Wipe
W	Water	L	Liquid
O	Oil	V	Vegetation
A	Air	X	Other
AF	Air Filter		

Special Instructions: Preservative used - None
Analysis Req' Ref KBC 40467
Disposal Per KBC 40467

Samples Transferred to new COC:
☒ No ☐ Yes, new COC No. ____

CHAIN OF POSSESSION

Relinquished By	Date	Time	Received By	Date	Time
Adam Compean	12-15-2010	10:17	Sample cart	12-15-2010	10:17
Sample cart	12-16-10	14:11	Adam Compean	12-16-10	14:11
Adam Compean	12-16-10	14:28	Pig Pen	12/16/10	14:28
Pig Pen	12/17/10	9:55	SWB	12/17/10	10:05
SWB	12/27/10	5:35	FRANK BARKSFIELD	12/27/10	5:40
FRANK BARKSFIELD	12/27/10	7:15	Dustin Blandon	12-27-10	19:17

FINAL SAMPLE DISPOSITION

Disposal Method ____

Disposed By:

Print ____ Sign ____ Date ____ Time ____

NOTE: This form is to accompany the sample(s) at all times.

BC-6000-560 (02/05)

KBC PROJECT CHAIN OF CUSTODY

COC No. 6689 Date 12/17/2010 Field Logbook No. N/A

Point of Contact Lance Ross Phone No. 373-4494 MSIN X4-01

Delivered to: ☐ OS Engineer ☐ 1706 KE Counting Facility ☐ 222-S Lab ☐ WSCF ☒ Other 325 Lab

Sampled By Adam Compean (Print) Adam Compean (Sign)

See Sample Analysis Request for individual containers and analysis.

Sample Number	Location/Description	Sample Date	Sample Time	Matrix*	Comments
KW210-B3-C	SCS-con210/SL	12-17-2010	9:50	SL	
KW210-B3-D	SCS-con210/SL	12-17-2010	9:50	SL	

*Matrix

S	Soil	DS	Drum Solids
SE	Sediment	DL	Drum Liquids
SO	Solid	T	Tissue
SL	Sludge/Slurry	WI	Wipe
W	Water	L	Liquid
O	Oil	V	Vegetation
A	Air	X	Other
AF	Air Filter		

Special Instructions: Preservative Used - None
Analysis Req. Ref 40467
Disposal Req KPC 40467

Samples Transferred to new COC:
☐ No ☐ Yes, new COC No. _____

CHAIN OF POSSESSION

Relinquished By (Print / Sign)	Date / Time	Received By (Print / Sign)	Date / Time
Adam Compean / Adam Compean	12/17/2010 / 9:58	Sample cart / Sample Cart	12/17/2010 / 9:58
Sample cart / Sample Cart	12-27-2010 / 10:12	Adam Compean / Adam Compean	12-27-2010 / 10:12
Adam Compean / Adam Compean	12-27-2010 / 10:24	Pig Pen / Pig Pen	12-27-2010 / 10:24
Pig Pen / Pig Pen	12/27/10 / 205	SWB / SWB	12/27/10 / 210
SWB / SWB	12/27/10 / 535	FRANK BURKSFIELD / [signature]	12/27/10 / 540
FRANK BURKSFIELD / [signature]	12/27/10 / 715	Dustin Blundun / [signature]	12-27-10 / 1918

FINAL SAMPLE DISPOSITION

Disposal Method _____

Disposed By:

Print _____ Sign _____ Date _____ Time _____

NOTE: This form is to accompany the sample(s) at all times.

BC-6000-560 (02/05)

CHPRC 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 105KW, 100K Area
City, State, Zip Hanford Site, 99354
Contact Jim Mathews
Phone 509-373-4598

2. SHIP TO ☐ U.S. DEPT. OF ENERGY C/0
Company PNNL
Address 325 Building, 300 Area
City, State, Zip Hanford Site, Wa 99354
Attention Fran Steen/Tony DeGuia
Phone 376-3171 (High Level Rad Facility)

6. SHIPMENT AUTHORIZATION NUMBER LJ017

7. EMERGENCY RESPONSE
Telephone 509-373-0911
Emergency Response Guide(s) 163

HM 8. Proper Shipping Name: RQ — Radioactive Material, transported under special arrangement, fissile excepted — PRI HAZ 7 — SUB HAZ NA — UN ID UN2919

9. No. Pkg.	Model Package	COC/Spec	Serial No.	Seal No.	Isotopes	C.S.I.	T.I.	Bq/Package	Gr. Wt. Kg.
1	SWB	Type A	*	*	Am-241, Pu-239, Pu-240, *	NA	<0.5	.1054 TBq	1213Kg

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

11.
☐ Highway Route Controlled Quantity
☐ Exclusive Use Shipment with instructions
☒ Placards Applied Radioactive
☒ Fissile Excepted, Grams 1.293
☒ UN ID Marking UN 2919

12. LABELS APPLIED
Radioactive Yellow - II NA

13. ADDITIONAL LABELS / MARKINGS
*Pu-241, Pu-238 source term based on Radcalc

HM 8. Proper Shipping Name: RQ — Radioactive Material, transported under special arrangement, fissile excepted — PRI HAZ 7 — SUB HAZ NA — UN ID UN2919

9. No. Pkg.	Model Package	COC/Spec	Serial No.	Seal No.	Isotopes	C.S.I.	T.I.	Bq/Package	Gr. Wt. Kg.
1	SWB	Type A	*	*	Am-241, Pu-239, Pu-240, *	NA	<0.5	.1054 TBq	1213Kg

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

11.
☐ Highway Route Controlled Quantity
☐ Exclusive Use Shipment with instructions
☒ Placards Applied Radioactive
☒ Fissile Excepted, Grams 1.293
☒ UN ID Marking UN 2919

12. LABELS APPLIED
Radioactive Yellow - II NA

13. ADDITIONAL LABELS / MARKINGS
*Pu-241, Pu-238 source term based on Radcalc

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

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 LABELS / MARKINGS

14. Shipment DE-Ci: 0.132 (ICRP-72)

Shipment Totals	C.S.I.	T.I.	Bq/Package	Gr. Wt. Kg.
	NA	<0.5	.2108 TBq	2426Kg

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
☑ <Tbl. 2-2 HNF-5173 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-1000278 | Date 12-27-10

16. TRANSPORTER
Vehicle Number G8200631
DRIVER SIGNATURE F. BARKSFIELD
PRINT NAME

17. RECEIVER
RECEIVER SIGNATURE Destra Blanda
PRINT NAME
Date 12/27/10

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 James Lindholm | Print Name James Lindholm | On behalf of DOE-RL | Date 12-27-10 | Organization CSG

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

* Seal No# 040448 TID# 23471/23470 * Seal No.# -060113 TID# 23469/23468

A-6004-858 (REV 0)

KBC PROJECT CHAIN OF CUSTODY

COC No. 6692 Date 12-27-2010 Field Logbook No. N/A

Point of Contact Lance Ross Phone No. 373-4494 MSIN V4-01

Delivered to: ☐ OS Engineer ☐ 1706 KE Counting Facility ☐ 222-S Lab ☐ WSCF ☒ Other 325 Lab

Sampled By Adam Compean (Print) Adam Compean (Sign)

See Sample Analysis Request for individual containers and analysis.

Sample Number	Location/Description	Sample Date	Sample Time	Matrix*	Comments
KW210-B3-E	SCS-con 210/SL	12-27-10	14:06	SL	
KW210-B3-F	SCS-con 210/SL	12-27-10	14:06	SL	

*Matrix

S	Soil	DS	Drum Solids
SE	Sediment	DL	Drum Liquids
SO	Solid	T	Tissue
SL	Sludge/Slurry	WI	Wipe
W	Water	L	Liquid
O	Oil	V	Vegetation
A	Air	X	Other
AF	Air Filter		

Special Instructions: Preservative used – none
Analysis Req. Ref KBC 40467
Disposal Per KBC 40467

Samples Transferred to new COC:
☒ No ☐ Yes, new COC No. _____

CHAIN OF POSSESSION

Relinquished By	Date	Time	Received By	Date	Time
Adam Compean	12-27-2010	14:28	Sample cart	12-27-2010	14:28
Sample cart	12-28-2010	14:03	Adam Compean	12-28-2010	14:03
Adam Compean	12-28-2010	14:11	Pig Pen	12-28-2010	14:11
Pig Pen	12/29/10	922	SWB	12-29-10	925
SWB	12/29/10	540	Mike J Hallenbeck	12/29/10	5:40
Mike J Hallenbeck	12/29/10	7:25	Ben Talma	12/29/10	7:25am

FINAL SAMPLE DISPOSITION

Disposal Method _____

Disposed By: _____ (Print) _____ (Sign) _____ (Date) _____ (Time)

NOTE: This form is to accompany the sample(s) at all times.

BC-6000-560 (02/05)

~~CHPRC~~ 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 105KW,100K Area
City, State, Zip Hanford Site, 99354
Contact Jim Mathews
Phone 509-373-4598

2. SHIP TO ☐ U.S. DEPT. OF ENERGY C/0
Company PNNL
Address 325 Building, 300 Area
City, State, Zip Hanford Site, Wa 99354
Attention Fran Steen/Tony DeGuia
Phone 376-3171 (High Level Rad Facility)

6. SHIPMENT AUTHORIZATION NUMBER LJ018

7. EMERGENCY RESPONSE
Telephone 509-373-0911
Emergency Response Guide(s) 163

HM 8. Proper Shipping Name: | PRI HAZ | SUB HAZ | UN ID
RQ Radioactive Material, transported under special arrangement,fissile excepted | 7 | NA | UN2919

9. No. Pkg.	Model Package	COC/Spec	Serial No.	Seal No.	Isotopes	C.S.I.	T.I.	Bq/Package	Gr. Wt. Kg.
1	SWB	Type A	060115	*	Am-241,Pu-239,Pu-240,*	NA	20.5	.1054 TBq	1213Kg

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

11.
☐ Highway Route Controlled Quantity
☐ Exclusive Use Shipment with instructions
☒ Placards Applied Radioactive
☒ Fissile Excepted, Grams 1.293
☒ UN ID Marking UN 2919

12. LABELS APPLIED
Radioactive Yellow - II

13. ADDITIONAL LABELS / MARKINGS
*Pu-241,Pu-238 source term based on Radcalc

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

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 LABELS / MARKINGS

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

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 LABELS / MARKINGS

14. Shipment DE-Ci: 0.066 (ICRP-72)

Shipment Totals	C.S.I.	T.I.	Bq/Package	Gr. Wt. Kg.
	NA	20.5	.1054 TBq	1213Kg

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
☑ <Tbl. 2-2 HNF-5173 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-1000297 | Date 12-29-10

16. TRANSPORTER

Vehicle Number	DRIVER SIGNATURE	PRINT NAME
G8200631	Mike Hallenbeck	Mike J. Hallenbeck

17. RECEIVER

RECEIVER SIGNATURE	PRINT NAME	Date
Ron Palma	Ron Palma	12-29-10

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
James Lindholm	James Lindholm		12-29-10	CSG

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-6004-858 (REV 0)

#TID-23476 / 23477

Appendix B

ASR 8798 and Results

Appendix B

ASR 8798 and Results

Appendix B contains the signed Analytical Service Request (ASR) 8798 and special instructions generated for Analytical Support Operations (ASO) laboratory analysis of the KW210 core composite samples. The special instructions delineated sample-specific information, required detection limits, quality control (QC) sample requirements, and reporting requirements. All analytical reports generated by ASO in response to the ASR are provided in Appendix B. Analytical reports provide sample results; associated uncertainties; and a discussion of sample processing, QC results, compliance with QC sample requirements, and data limitations. Occurrence Reports are also provided. These were prepared when issues were identified, and resolution was required while work was in process.

Two correspondence letters to the Buyer's Technical Representative (BTR, WW Rutherford) from the PNNL K Basin Sludge Project Manager (CD Carlson) are also provided. Letter 52578-2011-L06 details the ASO evaluation of the generally low matrix spike sample recoveries for Se, Sb, and Tl in the K Basin sludge matrix. Letter 59205-2011-L04 discusses the fact that not all of the QC sample requirements delineated by the Quality Assurance Project Plan/Sampling and Analysis Plan (QAPjP/SAP) were successfully met (some analyte spike recoveries exceeded acceptance criteria and in some cases the Pb detection limit was not met). A crosswalk of the QC failures with the Waste Isolation Pilot Plant Hazardous Waste Permit (Table C3-9) is included. Although this letter specifically discusses the Settler Sludge samples collected from Engineered Container SCS-CON-230, the BTR provided direction that the discussion and type of action to be taken in the event of QC sample failure was generally applicable to the sludge from SCS-CON-210 (as well as other engineered containers).

The following table of contents shows that pagination specific to Appendix B was applied to the ASR, the analytical reports and attachment, Occurrence Reports, and reference correspondence letters. This pagination is sequential and consistent (e.g., page B.#).

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Analytical Service Request (ASR)

ASR-FY2006-ASO Rev. 0.doc

(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 K FISKUM
Phone: 376-7015 MSIN: P7-25

Project Number: 52578
Work Package: see Special Instructions

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)
  see attached special instructions.
  ☐ 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: 8/16/11
- ♦ **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 Special Instructions
- ♦ **Requested Analytical Work Completion Date:** April 27, 2011
  (Note: Priority rate charge for < 10 business day turn-around time)
- ♦ **Negotiated Commitment Date:** 5/27/11
  (*To be completed by ASO Lead*) KNP

Waste Designation Information

- ♦ **Sample Information Check List Attached?** ☒ No ☐ Yes
  If no, Reference Doc Attached: _____
  or, Previous ASR Number: 8420
  or, Previous RPL Number: _____

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

Send Report To: SK Fiskum MSIN P7-25 & Matt Fountain
R. Shimskey MSIN P7-22

Additional or Special Instructions See Attached.

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

Date Delivered: 2/24/11
Delivered By (optional): SFO techs
Time Delivered (optional): _____
Group ID (optional): _____
CMC Waste Sample? ☒ No ☐ Yes

Received By: _____

ASR Number: 8798 Rev.: 00
RPL Numbers: 11-0419 - 11-0422
(first and last)

ASO Work Accepted By: KN Pool **Signature/Date:** 3/4/11

Analytical Services Request (ASR)

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

Provide Analytes of Interest and Required Detection limits - ☐ Below ☐ Attached

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
11-0419	See Attached Special Instructions for client specific ID's for the individual analyses fractions (Table 2)	KW-Basin Sludge Core Samples	1) Digest 107 (SAL) a) Uranium Oxidation State b) ICP/OES for Uranium (7th rinse and U-metal fractions) 2) Direct Analyses a) TIC/TOC 3) Digest - 129 (SAL) a) U-KPA b) U-TIMS (Prep) c) Pu- TIMS (Prep) d) Pu-AEA e) Am-AEA f) Np-AEA g) GEA h) Sr-90 i) IPC/OES 4) Undissolved Solids Analyses a) GEA (from 129 digest performed in SAL)	Digest-107 UOS ICP	
11-0420				TIC/TOC Digest-129 U-KPA U-TIMS PU-TIMS	
11-0421				PUAEA AmAEA NpAEA GEA SR-90	
11-0422				ICP/OES GEA	

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.

Special Instructions for K-Basin Container SCS-CON-210
Core Sample Analysis:
KW210-A1, -A4, -B1, and –B3

Cognizant Scientist: SK Fiskum Date: 2/24/11
SK Fiskum

Project Manager: CD Carlson Date: 2/24/2011
CD Carlson

Project Quality Engineer: DS Coffey Date: 2-24-11
DS Coffey

ASO Lead: KN Pool Date: 2/24/11
KN Pool

STP Concurrence: WW Rutherford Date: 2/24/11
WW Rutherford

Special Instructions for K-Basin Container SCS-CON-210 Core Sample Analysis: KW210-A1, -A4, -B12, and –B3

Cognizant Scientists:
SK Fiskum, 376-7015
RW Shimskey, 376-3183
CD Carlson, 376-2912

Four core composite materials containing liquid and sludge from K West (KW) Basin Engineered Container SCS-CON-210 are presented for analysis. The sample processing and analysis schematic for a single core composite is shown in Figure 1. The processing, prior to the Analytical Support Organization (ASO)-required analyses (non-highlighted blocks), is covered in separate project-specific test instructions, 52578-TI001 and -TI002. The ASO-specific processing is shown in the highlighted blocks. The solid slurry samples have already been aliquoted.

Although most work is being performed under the ASO QA Plan, some activities are outside of the scope of the plan such as sample preparation in the High- Level Radiochemistry Facility (HLRF), X-ray diffraction (XRD), and thermal ionization mass spectrometry (TIMS).[(a)]

The material has been received in the Laboratory and logged into the Radioactive Material Tracking (RMT) system as RMT #37737; the Sample Information Checklist (SICL) is tied to this RMT number. The sample labels are color coded in association with the specific container and core composite and Radiochemical Processing Laboratory (RPL) ID numbers (where possible; the Oak Ridge centrifuge tubes are not color-coded). The sample-specific IDs, the RPL assigned sample IDs, and the sampling date (at the K Basin) are cross-referenced in Table 1. **Note that a 6-month hold time applies to ICP-OES metals analysis.**

Table 1. Core Sample ID, RPL ID, Sampling Date, and Holding Time Cross-Reference

Core Sample ID	RPL ID	Sample Date (T_0)	End of 6-Month Hold Time
KW210-A1	11-0419	12/29/10	6/29/11
KW210-A4	11-0420	11/17/10	5/17/11
KW210-B1	11-0421	12/01/10	6/01/11
KW210- B3	11-0422	12/15/10	6/15/11

(a) Inductively coupled plasma mass spectrometry (ICP-MS) may be used as contingency in the event TIMS is not available.

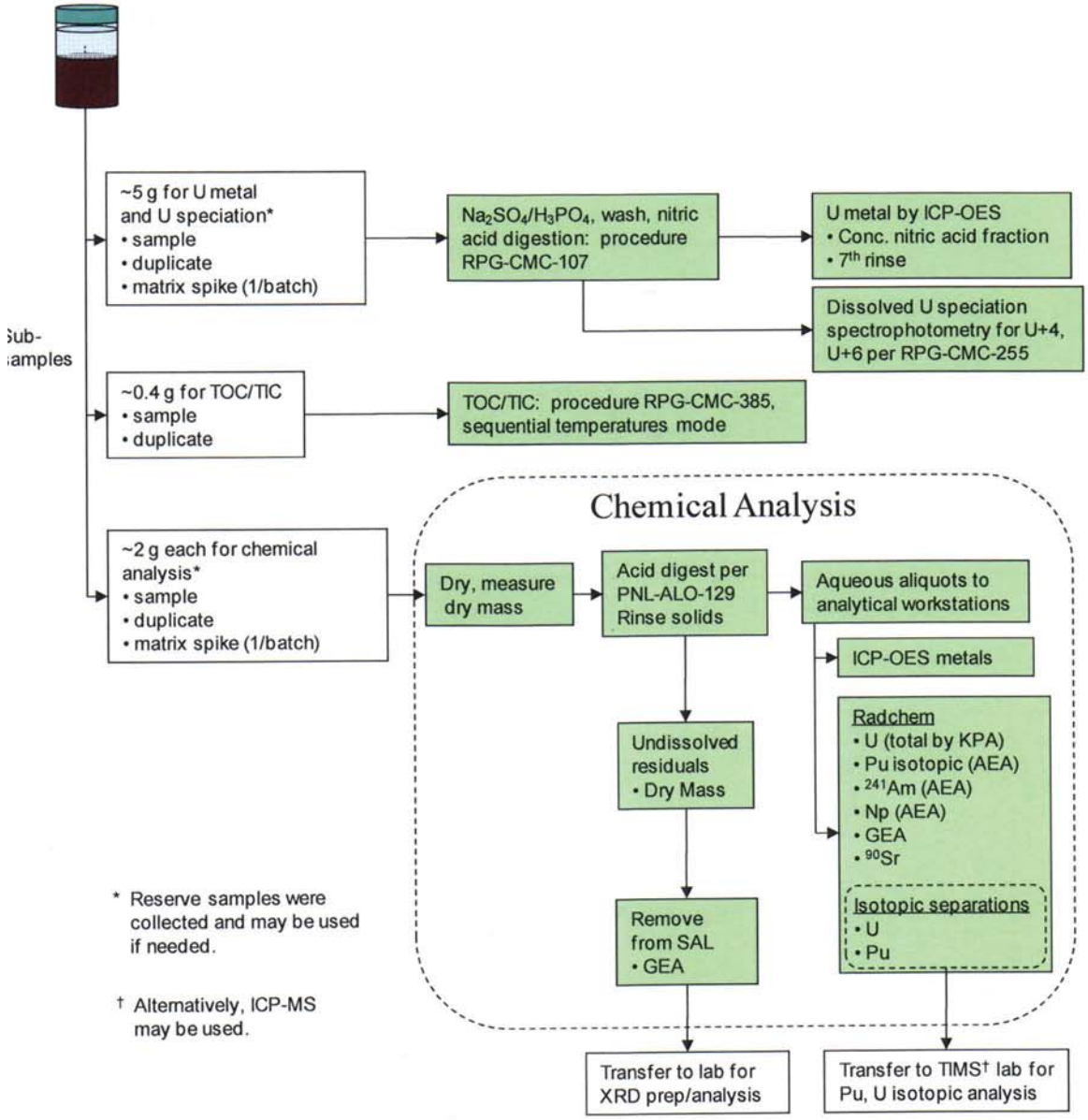

Figure 1. Analysis Schematic for Core Composites

The Chemical Measurement Center needs to charge analyses to the following work packages:

H41220	RPG-CMC-107 Rev. 1 Acid Digest	H41216	GEA
H41220	U Metal Analysis ICP-OES		Pu-AEA, Am-AEA
H41221	U Oxide speciation		Sr-90
H41215	TOC/TIC		U-KPA
H41215	PNL-ALO-129 Rev. 0 Acid Digest		Preparations for TIMS (U and Pu)
H41215	ICP-OES		

All dewatered sample solids masses have been calculated based on measures recorded from HLRF processing activities as recorded in Test Instructions 52578-TI001 and -TI002; these solids masses are reproduced in Table 2. The shaded reserve samples were collected as backups and are not expected to be analyzed.

Table 2. Cross-Reference of Sample ID, RPL ID, Dewatered Mass, and Analysis

Sample ID	RPL ID	Container Tare, g	Dewatered Solids Mass, $g_{dewatered}$	Analysis
KW-210-A1	11-0419			
TI001-A1-B	11- 0419-129-S	34.0686	2.05	Chemical analysis
TI001-A1-C	11-0419 -129-D	34.3212	2.28	Chemical analysis duplicate
TI001-A1-D				Chemical analysis reserve
TI001-A1-E				Chemical analysis reserve
TI001-A1-F				Chemical analysis reserve
TI001-A1-G				Chemical analysis reserve
TI001-A1-H	11- 0419-C-S	24.5816	1.3503	TOC/TIC
TI001-A1-J	11-0419-C-D	24.5729	1.0210	TOC/TIC duplicate
TI001-A1-P	11-0419-107-S	37.8452*	3.89	U metal
TI001-A1-Q	11-0419 -107-D	37.5330*	4.55	U metal duplicate
TI001-A1-R				U metal reserve
TI001-A1-S				U metal reserve
TI001-A1-T				U metal reserve
TI001-A1-U				U metal reserve
KW-210-A4	11-0420			
TI002-A4-B	11-0420-129-S	34.5564	2.30	Chemical analysis
TI002-A4-C	11-0420-129-D	35.0352	2.61	Chemical analysis duplicate
TI002-A4-D				Chemical analysis reserve
TI002-A4-E				Chemical analysis reserve
TI002-A4-F				Chemical analysis reserve
TI002-A4-G				Chemical analysis reserve
TI002-A4-H	11-0420-C-S	24.6518	1.4289	TOC/TIC
TI002-A4-J	11-0420-C-D	24.5934	2.0583	TOC/TIC duplicate
TI002-A4-P	11-0420-107-S	38.2432*	4.80	U metal
TI002-A4-Q	11-0420-107-D	37.6535*	3.99	U metal
TI002-A4-R				U metal reserve
TI002-A4-S				U metal reserve
TI002-A4-T				U metal reserve
TI002-A4-U				U metal reserve
KW-210-B1	11-0421			
TI002-B1-B	11-0421-129-S	33.1842	1.84	Chemical analysis
TI002-B1-C	11-0421-129-D	34.1788	1.98	Chemical analysis duplicate
TI002-B1-D	11-0421-129-MS	33.4949	2.15	Chemical analysis MS
TI002-B1-E				Chemical analysis reserve
TI002-B1-F				Chemical analysis reserve
TI002-B1-G				Chemical analysis reserve
TI002-B1-H	11-0421-C-S	24.5452	1.7868	TOC/TIC

Sample ID	RPL ID	Container Tare, g	Dewatered Solids Mass, $g_{dewatered}$	Analysis
TI002-B1-J	11-0421-C-D	24.5051	2.0942	TOC/TIC duplicate
TI002-B1-P	11-0421-107-S	37.9626*	4.18	U metal
TI002-B1-Q	11-0421-107-D	38.1402*	4.62	U metal
TI002-B1-R	11-0421-107-MS	38.7078*	3.97	U metal MS
TI002-B1-S				U metal reserve
TI002-B1-T				U metal reserve
TI002-B1-U				U metal reserve
KW-210-B3	11-0422			
TI001-B3-B	11-0422-129-S	33.9427	2.26	Chemical analysis
TI001-B3-C	11-0422-129-D	33.6711	2.24	Chemical analysis duplicate
TI001-B3-D				Chemical analysis reserve
TI001-B3-E				Chemical analysis reserve
TI001-B3-F				Chemical analysis reserve
TI001-B3-G				Chemical analysis reserve
TI001-B3-H	11-0422-C-S	24.6681	1.8483	TOC/TIC
TI001-B3-J	11-0422-C-D	24.7512	1.5905	TOC/TIC duplicate
TI001-B3-P	11-0422-107-S	37.4950*	4.33	U metal
TI001-B3-Q	11-0422-107-D	38.1267*	3.93	U metal duplicate
TI001-B3-R				U metal reserve
TI001-B3-S				U metal reserve
TI001-B3-T				U metal reserve
TI001-A1-U				U metal reserve
NA				
Purolite® + Grafoil®	11-0423-C-S	--	--	TOC/TIC
Purolite® + Grafoil®	11-0423-C-D	--	--	TOC/TIC duplicate
* Oak Ridge tube tares include a magnetic stir bar. Container tares were collected from balance N04143 on February 16, 17, 21, and 22, 2011.				

Analytes of interest and required detection limits are provided in Table 4; quality control (QC) sample acceptance criteria are provided in Table 5. One sample per analytical batch needs to be used to prepare a matrix spike (MS) sample. Subsampling of the stock material is a laborious process; a series of reserve samples from each core composite have been collected during the initial aliquoting process. The reserve samples should be used in case of catastrophic failure (i.e., spilling) of the primary or duplicate sample. The reserve samples were also collected in case the duplicate result did not agree with the primary result and a re-analysis might be required. The reserve samples may also be forwarded for analysis in place of the primary or duplicate samples based on the prerogative of the Cognizant Scientist (pending observations of actual mass collected, or other issues).

The total organic carbon (TOC) and total inorganic carbon (TIC) samples have been delivered directly to the TOC/TIC workstation following clean removal from the HLRF.

U-Metal Analysis and Uranium Oxide Speciation

U Speciation Sample Preparation

The samples (located in the Shielded Analytical Laboratory [SAL]) are provided to ASO in tared Oak Ridge tubes (suitable for sample digestion). The ASO is responsible for preparing the preparation blank (PB), the laboratory control sample (LCS), and the MS samples.

- The PB will consist of Hanford sand or equivalent and follow all processes that the samples are subjected to.
- The LCS will consist of Hanford sand, U_3O_8, (a mixture of U[IV] and U[VI]), and U metal beads.
- The MS will consist of a sample with U metal bead(s) added to it by ASO.

The samples will be processed per ASO Procedure, RPG-CMC-107, Rev. 1, *Sample Preparation for Determination of Uranium Metal Concentrations in Sludge*. At least three contacts with Na_2SO_4/H_3PO_4 shall be conducted to dissolve non-metal U species and at least 7 rinses conducted on the residual solids.

The dissolved (metal) U and seventh rinse solution U concentrations shall be determined by ICP-OES, (RPG-CMC-211, Rev. 3, *Determination of Elemental Composition by Inductively Coupled Argon Plasma Optical Emission Spectrometry [ICP-OES]*). The seventh rinse solution U concentrations should be <100 µg U/mL; the ICP-OES MDL for these samples shall be ≤1 µg U/mL. The ASO staff is directed to promply notify SK Fiskum /CD Carlson/DS Coffey if the 7th rinse is >100 µg U/mL, thus enabling PNNL to fulfill its commitment to the client for prompt notication of a potential issue with the analysis. **The duplicate precision criterion (Table 5) only applies to the dissolved U metal sample portion; it does not apply to the 7th rinse solution.**

Submit the Na_2SO_4/H_3PO_4 sample solutions containing the U(IV) and U(VI) for analysis by spectrophotometry (per RPG-CMC-255, Rev. 0, *Sample Preparation and Analysis for Determining Uranium Oxide Oxidation States in K Basin Sludges*).

U Speciation Reporting Units

Report the U metal results as **µg U per g of dewatered sludge,** where the mass of dewatered sludge in the analysis aliquot is taken from Table 2. **Report the seventh rinse sample U concentration as µg U per mL of solution. Report the U(IV) and U(VI) oxidation states as percentages.**

TOC/TIC Analysis Samples

TOC/TIC Preparation

The TOC/TIC slurry samples need to be analyzed at the work station (fume hood). These samples pose a contact dose hazard because containment is not in the hot cell. Furthermore samples will be dried as part of the analysis processing, and will also pose a contamination hazard after processing.

Damp or wet samples will need to be dried prior to subsampling and aliquot weighing. Drying can be conducted by simple air drying or heating to 105°C. In either case, constant dry mass will need to be ascertained prior to subsampling. The sample will need to be subsampled at the

workstation to provide a sample, duplicate and triplicate (for each sample) for analysis. One of the samples per analytical batch will need to be subsampled again to support an analytical spike (AS). Subsampling at the workstation will need to incorporate care to provide representative subsamples to optimize chances to meet precision measurement requirements; however, it is understood that subsampling will be conducted on a best-effort basis.

Analyze the samples per RPG-CMC-385, Rev. 2, *Carbon Measured in Solids, Sludge, and Liquid Matrices* furnace oxidation method with sequential temperature analysis mode. The TOC will be determined from C combustion at 600ºC; the same sample material will then be subjected to 1000ºC to measure all other (inorganic) C. The inorganic C measurement is expected to include Grafoil® and carbonates.

The ASO is responsible for preparing each sample in duplicate, as well as the LCS and blank spike (BS) and AS QC samples. The AS and LCS/BS samples are to include the carbon standards glucose and $CaCO_3$.

A sample of dry Purolite® NRW37 resin beads combined with a sample of Grafoil® also needs to be prepared by ASO to be run with the sample batch in duplicate, first analysis at the beginning of the analytical batch and the duplicate analysis at the end of the analytical batch. This material will be processed as a sample (11-0423) to evaluate method performance on the more-difficult-to-oxidize resin beads and Grafoil®. Report TOC and TIC recoveries of these two constituents. If the recovery of either of these constituents is >125% or <75%, contact SK Fiskum/R Shimskey (Cognizant Scientists).

TOC/TIC Reporting Units

Report the TOC and TIC concentrations as $\mu g\ C/g_{dry}$, where g_{dry} is determined at the workstation. Report the dry sample mass subjected to analysis.

Chemical Analysis Samples

Acid Digestion

Preparation

Note: Balances used to support the nuclear materials (Pu, Am, Np, and U) analysis require stringent daily balance checks on each day the balance is used . Review section on "Quality Control" before measuring and recording sample mass data. To this end, all masses collected in SAL must have the linearity check conducted and recorded.

The analytical samples are provided to ASO in tared Oak Ridge tubes (suitable for sample digestion). Analytical samples need to be dried to constant mass at 105°C. Determine **and report** the net dry sample mass.

Acid digest the dried solids according to PNL-ALO-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. Provide a copy of the ASO preparation bench sheet to SK Fiskum and RW Shimskey.

Residual solids remaining after acid digestions are expected and of great 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 solution with the acid digestion solution.

Videotape the inside Oak Ridge tubes to show characteristics of the wet solids/residues; provide video to SK Fiskum or RW Shimskey.

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/RW Shimskey. Additional processing of residual solids will depend on the mass relative to the starting (wet/dewatered) material. Additional processing may include:

- Removal from the SAL and gamma count
- Then transfer solids outside of ASO to Doinita Neiner for XRD preparation and analysis.

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 $g_{residual}$.

ICP-OES

ICP-OES Analysis

Acid digestate aliquots shall be analyzed at the ICP-OES workstation according to RPG-CMC-211, Rev. 3, *Determination of Elemental Composition by Inductively Coupled Argon Plasma Optical Emission Spectrometry (ICP-OES)*.

ICP-OES Reporting Units

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

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 3. 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 3. Authorized Radiochemistry Procedures for K Basin Sludge Analysis

Analyte	Separation	Mounting	Counting/Analysis
GEA	NA	NA	RPG-CMC-450, Rev. 1 *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. 0, *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. 0, *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. 0, *Coprecipitation Mounting of Actinides for Alpha Spectroscopy*	RPG-CMC-422, Rev. 2 *Solutions Analysis: Alpha Spectrometry*

Analyte	Separation	Mounting	Counting/Analysis
^{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 preparation	RPG-CMC-455, Rev. 0, *Separation of Uranium and Plutonium for Isotopic Analysis by Mass Spectroscopy*	NA	NA/Deliver to Pu Isotopic Analysis Workstation
U isotopic preparation	RPG-CMC-4017, Rev. 0, *Analysis of Environmental Water Samples for Actinides and Strontium-90*	NA	NA/Deliver to U Isotopic Analysis Workstation

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

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.

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 5 do not apply to the undissolved solids.

U KPA

Preparation and Analysis

Acid digestate aliquots shall be diluted and analyzed according to RPG-CMC-4014, Rev. 1, *Uranium by Kinetic Phosphorescence Analysis.* **The U KPA analysis requires stringent QC sample processing (refer to Table 5) to demonstrate accuracy:**

- One sample per analytical batch is to be spiked with U. This will be a sample digestate spiked with U at the workstation, i.e., a post-digestion MS. The calculated U recovery is required to be within 75% to 125%.
- A minimum of two different calibration verification checks need to be run with each analytical batch and an assessment of their accuracies reported (±7.5% of true value).
- An independent U BS sample needs to be run with each analytical batch. This will be an independent U BS sample processed through the acid digestion (PNL-ALO-129 acid digestion) (see Table 5). The BS recovery is required to be 80% to 120%.

U KPA Reporting Units

Report solids sample analyte concentrations as $\mu g/g_{dry}$ (U KPA) where g_{dry} is the initial dry sample mass determined prior to the start of the 129 acid digestion.

Archive of SAL-Prepared Chemical Analysis Samples

The acid digest preparations will result in a ~50- to 100-mL volume. This solution will be apportioned to the laboratory as needed to conduct work-station-specific analyses. At the time the acid digestates are apportioned to the analysis work locations, ~15-mL aliquot from each preparation should be collected and removed from SAL and stored as an archive sample. The

vials need to be labeled with the following information: date, RPL-ID, sample ID, matrix, and hazard. The remaining portion of the acid digestion preparations may be disposed of.

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. 7) 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.

NOTE: Balances used to support the nuclear materials (Pu, Am, Np, and U) analysis require more stringent daily balance checks on each day the balance is used. In this case, the ASO SAL-related operations must use at least 3 masses (e.g., A, B, and A+B) to assess balance accuracy and linearity. The balance control checks must be traceable in the records supporting these analyses. It is suggested that the daily control check log sheets be provided as part of the analytical report; however the ASO may choose alternate means to establish traceability. Tolerances for control checks for the balance in SAL cell 2 are provided in Appendix A. **If masses were inadvertently collected on a balance without the daily check, then the ASO Lead must immediately upon discovery inform the QE, Cognizant Scientist and the Project Manager.**

The balance checks at the fumehood workstations may be checked per the normal process where one calibration verification check mass is measured daily when then balance is used (PNL-ASO-052, Rev. 1). This is allowed because the fumehood operations use volume measures or do not directly support nuclear materials accountability (e.g., TOC/TIC).

Solids matrix preparative analysis QC is to include a preparation blank (PB) sample, sample duplicate (provided), LCS (or BS) and a sample MS (a separate sample is provided for spiking by the ASO) as indicated in Table 5. The BS for radioisotopes will be generated after acid digestion on a sample split, i.e., post-digestion spikes. The MS for radioisotopes and U-KPA will be generated after acid digestion on a sample split, i.e., post-digestion spikes.

The ASO is responsible for preparing the PB, LCS/BS, and spiking MS QC samples. The preparative MS and LCS/BS are to include all the analytes of interest: Ag, Al, As, Ba, Be, Ca, Cd, Cr, Fe, Na, Ni, Mg, Pb, Sb, Se, Tl, U, V, Zn, and Zr. A second preparative BS is to include U only.

The duplicate, LCS/BS, and MS sample QC acceptance criteria are provided in Table 5. The following section discusses actions to take in the event of QC failures.

Note: The ASO staff must inform/report QC failures promptly to the ASO Lead who will in turn inform the Cognizant Scientist with copy to CD Carlson and the K Basin QE (Deborah Coffey), thus enabling PNNL to fulfill its commitment to the client for prompt notification of such failures. The impact of any QC 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 affected samples in the processing batch must be re-prepared and re-analyzed for the failed analytes, availability of samples permitting.
3. In the case of multi-elemental methods (ICP-OES), isolated QC failure(s) may be communicated to SK Fiskum, RW Shimskey, Clark Carlson and the K Basin QE by the ASO Lead for an assessment of the impact on data interpretation. If the data are acceptable as-is, the project staff will communicate in writing (e.g., e-mail message) that the data may be reported; the e-mail acceptance and resulting limitations on the data from the QC sample failure(s) shall be included in the final analytical report.
4. 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. 0.2, *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. 0.2.

Data Report **Cover Page** Contents:

- Header identifying name/address of laboratory
- Client Name:
- Project #/WP #(s):
- ASR #(s):
- 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 and title (sample processing and analysis) and revision number
- M&TE (serial number or property number):
- Reference Date (when applicable):
- Client ID and 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
- Analysis dates
- Deviations from the written procedure, if any
- Method specific Sample Preparation, Separation, Mounting and Counting Methods as applicable
- Reporting Basis/Units
- QC Criteria/Results
- Limitations of the Data Interferences/Resolution Uncertainty
- Comments

Data Summary Contents:

- Client ID
- RPL ID with full ASO-related suffixes (e.g., 129-S)
- Results and result uncertainties

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 lifetime 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.

The isotopic preparations will require limited reporting since these procedures do not provide analytical results. Needed components (cover page and narrative) are described as follows:

Data Report **Cover Page** Contents:

- Header identifying name/address of laboratory
- Client Name:
- Project #/WP #(s):
- ASR #(s):

- Total # Samples:
- Report Date:
- Report Revision Date: when applicable
- Filename(s): where data files can be accessed
- Dates of Sample Processing:
- Procedure(s): Number and title (sample processing and analysis) and revision number
- M&TE (serial number or property number):
- Reference (i.e., Separation) Date (when applicable):
- Client ID and 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 process and limitations on the analysis results
Narrative Contents:

- Sample Description and Analyses
- Sample dissolution/preparation dates
- Sample Preparation
- Method specific Sample Preparation, Separation
- Deviations from the written procedure, if any
- Comments

Table 4. 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
	$\mu g/g_{(dry)}$	
Ag	2.0E+01	ICP-OES
Al	1.0E+02	
As	2.0E+01	
Ba	4.0E+02	
Be	2.0E+01	
Ca	2.0E+02	
Cd	4.0E+00	
Cr	2.0E+01	
Fe	1.0E+02	
Mg	2.0E+02	
Na	2.0E+02	
Ni	2.0E+01	
Pb	2.0E+01	
Sb	2.0E+01	
Se	2.0E+01	
Tl	2.0E+01	
V	2.0E+01	
Zn	2.0E+01	
Zr	2.0E+01	
U	1.0E+03	
U	1.0E+01	Kinetic phosphorescence analysis
Total organic carbon	2.0E+02 (as C)	Furnace oxidation, sequential temperatures mode
Total inorganic carbon	2.0E+02 (as C)	
	wt% of settled sludge	
U metal	0.01	Selective dissolution and U analysis by KPA or ICP-OES
U oxide speciation	N/A	Selective dissolution and spectrophotometry
Note: The detection limits (DL) shown in this table are based on dry mass, except for the U metal analysis, whereas the DL in the QAPjP/SAP are based on settled solids (wet) mass basis. These DL values are therefore, tighter than those in the QAPjP/SAP.		

Table 5. Analytical Quality Control Acceptance Criteria

Analyte	Analytical Technique	LCS or BS % Recovery[a]	MS or AS % Recovery[b]	Duplicate RPD[c]
		Pre-digestion spike		
As identified in Table 4	ICP-OES	80 - 120%	80 - 120%	≤20%
TOC and TIC	Furnace oxidation, sequential temperatures	75 - 125%	75 - 125%	≤20%[f]
U total	KPA	80 - 120%	N/A	≤20%
U metal	Selective dissolution and ICP-OES or KPA	75 - 125%	N/A	≤20%
U oxide speciation	Selective dissolution and spectrophotometry	[e]	N/A	N/A
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%
U total	KPA	(See pre-digestion BS) Two Calibration Checks: 92.5 – 107.5%	75 - 125%	≤20% (as indicated above)
As identified in Table 4	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 1, a counter control source is checked daily and must be within ±3 sigma or ±3%, whichever is greater, of the control value.

(e) The LCS U_3O_8 accepted ratios are 33.3% U(IV) and 66.6% U(VI). The acceptable U(VI) range is 62 to 72% or ±5% window. Thus the acceptable U(IV) range is 28 to 38% or ±5% window.

(f) Calculate the RSD for the triplicate TIC/TOC measurements.

References

52578-TI001, Rev. 0, K West Basin Sample Receipt and Preparation of Consolidated Core Composites: SCS-CON-210-A1 and SCS-CON-210-B3

52578-TI002, Rev. 0, K West Basin Sample Receipt and Preparation of Consolidated Core Composites: SCS-CON-210-A4 and SCS-CON-210-B1

ASO-QAP-001, Rev. 7, *Analytical Support Operations Quality Assurance Plan*

PNL-ASO-052, Rev. 1, *Balance Performance Checks*

PNL-ASO-058, Rev. 0.2, *ASO Data Reporting*

RPG-CMC-107, Rev. 1, *Sample Preparation for Determination of Uranium Metal Concentrations in Sludge*

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

RPG-CMC-211, Rev. 3, *Determination of Elemental Composition by Inductively Coupled Argon Plasma Optical Emission Spectrometry (ICP-OES)*

RPG-CMC-255, Rev. 0, *Sample Preparation and Analysis for Determining Uranium Oxide Oxidation States in K Basin Sludges*

RPG-CMC-385, Rev. 2, *Carbon Measured in Solids, Sludge, and Liquid Matrices*

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

RPG-CMC-450, Rev. 1, *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. 0, *Coprecipitation Mounting of Actinides for Alpha Spectroscopy*

RPG-CMC-4014, Rev. 1 *Uranium by Kinetic Phosphorescence Analysis*

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

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

Appendix A

SAL Balance Control Checks and Tolerance Limits

The balance control checks will document accuracy and linearity by measure of three masses for the analytical balance Sartorius MSG234 (M&TE 40023610) and (if needed) Mettler PR5003DR balance (SN: 1120100979).

Tolerances for the three different mass measures are provided. These tolerances have been reviewed and approved by Safeguards (client Terri Welsh, Mission Suppprt Alliance).

Radiochemical Processing Laboratory
Battelle, Pacific Northwest National Laboratory

K-Basin Project **Type of Balance:** ***Mettler PR5003DR***

Balance Information
Identificaiton (e.g. WD34567)
1120100979
Location (e.g. Building/Room)
RPL, SAL Cell 2
Calibration Due Date
Aug-2011

Check Weight (CW) Information	#1	CW Units	#2	CW Units	#3	CW Units
CW Identification	Megatron					
CW Assigned Mass:	50.003	g	999.996	g	2999.97	g
Range Low Mass (+Units) &:	49.984	g	999.720	g	2999.10	g
Range High Mass (+Units):	50.021	g	1000.272	g	3000.84	g

(updated on 9/30/10. skf)

Radiochemical Processing Laboratory
Battelle, Pacific Northwest National Laboratory

K-Basin Project **Type of Balance:** ***Sartorius MSG234***

Balance Information
Identificaiton (e.g. WD34567)
40023610/WD68444
Location (e.g. Building/Room)
RPL/201 Cell 2
Calibration Due Date
Feb-2011

Check Weight (CW) Information	#1	CW Units	#2	CW Units	#3	CW Units
CW Identification	"K-10"		"Alien"		"Megatron" + "Alien"	
CW Assigned Mass:	10.0000	g	100.0002	g	150.0004	g
Range Low Mass (+Units) &:	10.0000	g	99.9998	g	149.9995	g
Range High Mass (+Units):	10.0010	g	100.0015	g	3000.8390	g

(updated on 9/30/10. skf)

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

Mass Measurements

Project / WP#:	52578/H41215
ASR#:	8798
Client:	S. Fiskum
Total # of Samples:	9

Engineered Container & Core ID	Sample IDs	RPL Number
KW-210-A1	TI001-A1-B, -A1-C	11-0419-129-S, -129-D
KW-210-A4	TI002-A4-B, -A4-C	11-0420-129-S, -129-D
KW-210-B1	TI002-B1-B, -B1-C, -B1-D	11-0421-129-S, -129-D, -129-MS
KW-210-B3	TI001-B3-B, -B3-C	11-0422-129-S, -129-D

Revision 1

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, 3-1-11 (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:	RPL-OP-001, Rev. 9, *Routine Research Operations* PNL-ASO-052, Rev. 1, *Balance Performance Checks*
Analysis Date or Date Range:	3/1/11 thru 3/18/11
Technician/Analyst:	J. Chenault
Analysis Data (File):	ASR 8798 dried solids memo.doc
CMC Project 98620 File:	File Plan 5871: T 59205 8798: Sample preparation and analysis records;
M&TE Number(s):	40023610 – SAL balance Cell 2

Preparer / 7/28/11 Date

Reviewer / 7-28-11 Date

MEMORANDUM

Pacific Northwest NATIONAL LABORATORY | Battelle The Business of Innovation

Date:	**July 27, 2011**	Project No.:	**52578**
To:	**Sandy Fiskum**		
From:	**Karl Pool**		
Subject:	**Initial dried solids and residual dried solids masses for ASR 8798**	Internal Distribution:	**File/LB**

Revision 1 – Report revised to correct the volumes of reagent acids used in the acid digestion of dried sludge.

Prior to ASO sample digestion, all slurry samples for ASR 8798 were dried to constant weight at 105°C in pre-tared Teflon Oak Ridge digestion tubes in the Shielded Analytical Laboratory (SAL) 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 acid volume used was proportioned to the mass of sample. Samples that had mass of 1 g or less used 8 mL of each acid. Samples that had mass of 1 to 1.5 g used 12 mL of each acid. At the completion of the acid digestion, the samples were cooled and the residual (un-dissolved solids) were allowed to settle. The digestate with settled residual was then centrifuged for approximately 10 minutes to consolidate the solids. The supernatant solution was carefully removed using transfer pipettes. The residual solids were each rinsed 3 consecutive times using ~ 12 to 16 mL of the same reagent acids (1:1 combination of 8M HNO_3 + 8M HCl) used in the initial acid digestion. The centrifuged solids were dislodged from the bottom of the centrifuge tube using a vortex mixer after each addition of the rinse acids. The acid rinse/solids were centrifuged for approximately 10 minutes after each acid rinse addition. The remaining rinsed solids were returned to the drying oven for drying. The residual solids were dried in the drying oven for several days at 105°C. The residual solids in the Oak Ridge tubes were dried to constant weight.

The initial acid digestate and acid rinse solutions were collected and brought to a final volume of 100 mL in plastic volumetric flasks. The solutions in the volumetric flask were thoroughly mixed prior to sub-sampling for chemical and radiochemical analyses.

MEMORANDUM

Pacific Northwest NATIONAL LABORATORY | Battelle The Business of Innovation

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)
11-0419-129-S	**TI001-A1-B**	**1.1233**	**0.7262**
11-0419-129-D	**TI001-A1-C**	**1.2773**	**0.8140**
11-0420-129-S	**TI002-A4-B**	**1.4258**	**1.0004**
11-0420-129-D	**TI002-A4-C**	**1.5994**	**1.1212**
11-0421-129-S	**TI002-B1-B**	**0.7459**	**0.6192**
11-0421-129-D	**TI002-B1-C**	**0.8192**	**0.7206**
11-0422-129-S	**TI001-B3-B**	**1.0626**	**0.9163**
11-0422-129-D	**TI001-B3-C**	**1.0515**	**0.9157**
11-0421-129-MS	**TI002-B1-D**	**0.8818**	**0.7621**

E54-1900-001 (8/98)

Battelle PNNL/RPL/Inorganic Analysis ... ICP-OES Analysis Report
PO Box 999, Richland, Washington 99352

Project / WP#: 52578 / H41215
ASR#: 8798
Client: S. Fiskum
Total Samples: 8 (solids)

	First	Last
RPL#:	11-0419	11-0422
Client ID:	TI001-A1-B	TI001-B3-C

Sample Preparation: PNL-ALO-129, "HNO_3-HCl Extraction of Solids for Metals Analysis Using a Dry-Block Heater", 03/11/11 (SAL/ng).

Procedure: RPG-CMC-211, "Determination of Elemental Composition by Inductively Coupled Argon Plasma Optical Emission Spectrometry (ICP-OES)", Rev. 3.

Analyst: J. Carter

Analysis Date (File): 03-15-2011 (C0353)
03-16-2011 (C0354)

See Chemical Measurement Center 98620 file: ICP-325-405-3
(Calibration and Maintenance Records)

M&TE Number: N827583 (PerkinElmer 5300DV ICP-OES, S/N 077N5122002)
WD66158 (Sartorius ME414S Balance, S/N 224066373)

5/31/11
Preparer

6/3/11
Review and Concur

Battelle PNNL/RPL/Inorganic Analysis ... ICP-OES Analysis Report

Four samples in duplicate were provided for ICP-OES analysis under Analytical Service Request (ASR) 8798. The samples and duplicates were obtained from sub-samples prepared in the High Level Radiochemistry Facility (HLRF). Each of the samples was processed in the Shielded Analytical Laboratory (SAL) following RPL procedure PNL-ALO-129 using ~1 to 2 grams of sample with dilution to a final volume of 100 mL.

Analytes of interest (AOIs) were specified in the ASR, and are listed in the upper section of the attached ICP-OES Data Report. The quality control (QC) results for these AOIs have been evaluated and are presented below. Analytes other than AOIs are reported in the bottom section of the report, but have not been fully evaluated for QC performance. The results are reported on a mass basis ($\mu g/g_{dry}$) for each detected analyte based on the original sample mass. All data have been adjusted for laboratory processing factors and instrument dilutions. There were several QC issues associated with the present analyses. These issues are discussed below and also in Occurrence Report 52578-04-20-11 and PNNL Letter 52578-2011-L06.

Except for As, Sb, Se, and Tl, the results are reported for 10x diluted samples. As, Sb, Se, and Tl are reported for undiluted samples (1x) in order to obtain the necessary sensitivity. Analyzing the samples at 1x, however, does introduce potential matrix issues as both the sample density and the acid content at 1x are considerably higher than the normal matrix seen by the instrument (~0.5 M nitric acid).

Minimum Method Detection Limit (MDL) values were specified in the ASR. Except for Pb and Se in Samples 11-0421-129-S and 11-0421-129-D, MDL levels were met for all AOIs. For the Pb, however, the observed levels exceeded the MDL requirements thereby mitigating this issue. Selenium, however, was <IDL for the two samples and, therefore, the MDL was not met.

Calibration of the ICP-OES was done following the manufacturer's recommended calibration procedure and using multi-analyte custom standard solutions traceable to the National Institute of Standards and Technology (NIST). Midrange calibration verification standards (MCVA and MCVB) were used to verify acceptance of the two-point calibration curves obtained for each analyte, and also used for continuing calibration verification.

The controlling documents were ASO-QAP-001 and ASR-8798 Special Instructions. Instrument calibrations, QC checks and blanks (e.g., ICV/ICB, CCV/CCB, LLS, ICS), blank spike, matrix spike, post spike, duplicate, and serial dilution were conducted during the analysis run. The blank spike and matrix spike were prepared using a custom solution consisting of aliquots of BPNL-QC-1A and -2B, INT-QC-TCLP-A, and CG-SB1-1.

Preparation Blank (PB):

A preparation blank (reagents only) was prepared for the extraction process. The concentrations of all AOIs were within the acceptance criteria of ≤EQL (estimated quantitation level) or less than ≤5% of the concentration in the sample.

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

A blank spike was prepared for the extraction process. Recovery values are listed for all analytes included in the spike that were measured at or above the EQL. The recovery values were within the acceptance criterion of 80% to 120% for all AOIs meeting the above requirement.

Matrix-Spiked Sample:

A matrix spike was prepared for the extraction process using Sample 11-0421. Recovery values are listed for all analytes included in the spike that were measured at or above the EQL. For AOIs meeting the above requirement, the recovery values for Sb (45%), Tl (30%), Zn (74%), and Zr (244%) were outside the acceptance criterion of 80% to 120%. These recovery results are likely from a combination of factors, including 1) sample heterogeneity in the sludge, 2) matrix issues associated with the very high Al, Fe, and U in the samples, and 3) spike levels that are relatively close to the instrument EQL values. Note that all the analytes did pass the post-spike analysis acceptance range of 80 to 120% (see below).

Duplicate Relative Percent Difference (RPD):

Duplicates were prepared for each of the four samples. RPDs are listed for all analytes that were measured at or above the EQL. RPDs for Sample 11-0422 were within the acceptance criterion of ≤20% for AOIs meeting the above requirement. For Samples 11-0419 (Be-37.7%, Cr-24.5%, Ni-36.5%), 11-0420 (Be-58.5%, Cr-51.0%, Mg-25.4%, Ni-47.3%, Zn-27.3%, 86.4%), and 11-0421 (Be-144.1%, Fe-24.4%, Ni-22.0%, Zn-47.3%, Zr-135.0%), however, the RPDs were outside the acceptance criterion. The high RPDs are likely from sample heterogeneity in the samples and/or from the sub-sampling in the HLRF.

Post-Spike/Analytical Spike Sample (A Component):

A post spike (A Component) was conducted on Sample 11-0421. Recovery values are listed for all analytes in the spike that were measured at or above the EQL, and that had a spike concentration ≥25% of that in the sample. The recovery values were within the acceptance criterion of 80% to 120% for all AOIs meeting the above requirements, including those analytes that failed the matrix-spike analysis discussed above.

Post Spike/Analytical Spike Sample (B Component):

A post spike (B Component) was conducted on Sample 11-0421. Recovery values are listed for all analytes in the spike that were measured at or above the EQL, and that had a spike concentration ≥25% of that in the sample. The recovery value for uranium (the only AOI in the B Component) was within the acceptance criterion of 80% to 120%.

Serial dilution:

Five-fold serial dilution was conducted on Sample 11-0421. Percent differences (%Ds) are listed for all analytes that had a concentration at or above the EQL in the diluted sample. The %Ds were within the acceptance criterion of ≤10% for all AOIs meeting the above requirement.

Other QC:

Barium was slightly above the EQL (~0.002 µg/mL) for the first of three ICSs for the 10x dilution run. This is believed to be from carry-over of Ba from the proceeding CCV. This small carry-over, however, had no statistical effect on the reported data as the level of Ba in the ICSs was negligible compared to the levels of Ba in the samples themselves. All other instrument-related QC tests for the AOIs passed within the appropriate acceptance criteria.

Comments:

1) The "Final Results" have been corrected for all laboratory dilutions performed on the samples during processing and analysis, unless specifically noted.
2) Instrument detection limits (IDL) and estimated quantitation limits (EQL) shown are for acidified water and/or fusion flux matrices as applicable. Detection limits for other matrices may be determined if requested. Method detection limits (MDL) can be estimated by multiplying the IDL by the "Multiplier". The estimated quantitation limit (EQL) for each concentration value can be obtained by multiplying the EQL by the "Multiplier".
3) Routine precision and bias is typically ±15% or better for samples in dilute, acidified water (e.g. 2% v/v HNO_3 or less) at analyte concentrations > EQL up to the upper calibration level. This also presumes that the total dissolved solids concentration in the sample is less than 5000 µg/mL (0.5 per cent by weight). Note that bracketed values listed in the data report are within the MDL and the EQL, and have potential uncertainties greater than 15%. Concentration values < MDL are listed as "- -". Note, that calibration and QC standards are validated to a precision of ±10%.
4) Absolute precision, bias and detection limits may be determined on each sample if required by the client. The maximum number of significant figures for all ICP measurements is two.
5) Analytes included in the spike A component (for the AS/PS) are; Ag, Al, As, B, Ba, Be, Bi, Ca, Cd, Co, Cr, Cu, Fe, K, Li, Mg, Mn, Mo, Na, Ni, P, Pb, Sb, Se, Si, Sn, Sr, Ta, Ti, Tl, V, W, Y, Zn, and Zr. Analytes included in the spike B component are; Ce, Dy, Eu, La, Nd, Pd, Rh, Ru, S, Te, Th, and U.

Battelle PNNL/RPG/Inorganic Analysis ... ICPOES Data Report

	Run Date >	3/15/2011	3/16/2011	3/15/2011	3/16/2011	3/15/2011	3/16/2011	3/15/2011	3/16/2011	3/15/2011	3/16/2011
	Multiplier >	878.6	87.9	890.2	89.0	782.9	78.3	701.4	70.1	625.2	62.5
Zr-	RPL/LAB >	11-0419-129-PB @ 10x	11-0419-129-PB	11-0419-129-S @ 10x	11-0419-129-S	11-0419-129-D @ 10x	11-0419-129-D	11-0420-129-S @ 10x	11-0420-129-S	11-0420-129-D @ 10x	11-0420-129-D
Instr. Det. Limit (IDL)	Est. Quant. Limit (EQL)	Client ID >	PB		TI001-A1-B		TI001-A1-C		TI002-A4-B		TI002-A4-C	
(µg/mL)	(µg/mL)	(Analyte)	(µg/g)	(µg/g)	(µg/g)	(µg/g)	(µg/g)	(µg/g)	(µg/g)	(µg/g)	(µg/g)	(µg/g)
0.0019	0.019	Ag	--		[5.5]		[3.8]		[2.8]		[2.8]	
0.0046	0.046	Al	--		32,500		32,600		20,700		22,400	
0.0960	0.960	As		--		89.2		98.4		72.3		72.7
0.0001	0.002	Ba	[0.24]		192		199		135		143	
0.0001	0.001	Be	--		6.99		10.2		12.2		6.70	
0.0022	0.044	Ca	--		7,640		6,750		6,690		7,740	
0.0015	0.015	Cd	--		35.9		33.5		29.8		34.6	
0.0020	0.020	Cr	[3.4]		354		277		569		337	
0.0045	0.045	Fe	--		86,800		88,600		96,400		93,700	
0.0006	0.012	Mg	--		1,140		1,010		730		943	
0.0036	0.072	Na	[14]		708		655		1,420		1,650	
0.0026	0.026	Ni	--		185		128		334		206	
0.0210	0.210	Pb	--		345		345		238		264	
0.0220	0.220	Sb		--		[8.6]		[8.0]		[9.4]		[8.5]
0.1700	1.700	Se		--		--		--		--		--
0.0860	0.860	Tl		--		[9.2]		[8.7]		[6.6]		[6.8]
0.0120	0.120	U	--		93,800		103,000		82,800		73,600	
0.0011	0.022	V	--		[15]		[15]		[13]		14.2	
0.0056	0.056	Zn	--		469		398		291		383	
0.0008	0.008	Zr	--		334		369		373		148	
Other Analytes												
0.0057	0.057	B	[39]		52.9		[38]		[26]		[25]	
0.0160	0.160	Bi	[59]		[120]		[110]		[100]		[86]	
0.0087	0.087	Ce	--		79.2		74.9		63.1		55.9	
0.0025	0.025	Co	--		[4.6]		[3.3]		[6.3]		[2.4]	
0.0016	0.016	Cu	--		1,680		1,070		510		487	
0.0012	0.012	Dy	--		--		--		--		--	
0.0003	0.003	Eu	--		[2.3]		2.34		2.00		2.02	
0.0220	0.220	K	619		1,240		1,230		967		912	
0.0012	0.012	La	--		24.5		24.7		20.1		20.9	
0.0005	0.005	Li	--		[3.2]		3.67		[2.1]		[2.4]	
0.0002	0.002	Mn	--		343		291		313		297	
0.0070	0.070	Mo	--		[43]		[44]		[42]		[35]	
0.0027	0.027	Nd	--		86.9		92.6		54.0		50.6	
0.0510	0.510	P	[47]		[430]		[370]		[210]		[240]	
0.0048	0.048	Pd	--		--		--		--		--	
0.0088	0.088	Rh	--		--		--		--		--	
0.0044	0.044	Ru	--		[19]		[21]		[13]		[18]	
0.1600	1.600	S	--		7,060		6,130		6,140		7,120	
0.0130	0.130	Si	--		749		678		592		610	
0.0140	0.140	Sn	[35]		[88]		[91]		[67]		[56]	
0.0000	0.000	Sr	[0.041]		63.8		57.8		43.2		49.8	
0.0190	0.190	Ta	--		--		--		--		--	
0.0160	0.160	Te	--		--		[16]		--		--	
0.0050	0.050	Th	--		--		--		--		--	
0.0005	0.005	Ti	--		464		358		382		374	
0.0150	0.150	W	--		--		--		--		--	
0.0003	0.005	Y	--		7.32		6.86		5.43		5.66	

1) "--" indicates the value is < MDL. The method detection limit (MDL) = IDL times the "multiplier" near the top of each column. The estimated sample quantitation limit = EQL (in Column 2) times the "multiplier". Overall error for values ≥ EQL is estimated to be within ±15%.

2) Values in brackets [ ] are ≥ MDL but < EQL, with errors likely to exceed 15%.

ASR 8798 Final from C0354-Rep S. Fiskum (ASR-8798 129's 1x).XLS

Battelle PNNL/RPG/Inorganic Analysis ... ICPOES Data Report

Run Date >	3/15/2011	3/16/2011	3/15/2011	3/16/2011	3/15/2011	3/16/2011	3/15/2011	3/16/2011
Multiplier >	1340.7	134.1	1220.7	122.1	941.1	94.1	951.0	95.1
RPL/LAB >	11-0421-129-S @ 10x	11-0421-129-S	11-0421-129-D @ 10x	11-0421-129-D	11-0422-129-S @ 10x	11-0422-129-S	11-0422-129-D @ 10x	11-0422-129-D
Client ID >	TI002-B1-B		TI002-B1-C		TI001-B3-B		TI001-B3-C	
(Analyte)	(µg/g)	(µg/g)	(µg/g)	(µg/g)	(µg/g)	(µg/g)	(µg/g)	(µg/g)
Ag	[4.2]		[3.5]		[2.0]		[3.9]	
Al	15,600		15,500		12,400		12,100	
As		[46]		[41]		[54]		[50]
Ba	75.4		78.0		66.8		62.4	
Be	3.03		18.6		2.76		2.51	
Ca	7,410		6,590		8,350		7,510	
Cd	65.5		57.4		48.8		48.8	
Cr	287		274		225		218	
Fe	35,100		27,500		31,800		27,800	
Mg	582		648		588		582	
Na	1,710		1,610		1,460		1,340	
Ni	223		178		157		156	
Pb	319		322		313		287	
Sb		--		[4.6]		[5.1]		[3.9]
Se		--		--		--		--
Tl		--		--		--		--
U	51,900		45,600		55,700		51,000	
V	[9.8]		[11]		[9.8]		[8.4]	
Zn	394		243		211		200	
Zr	77.6		400		91.8		97.2	

B	[52]		[47]		[31]		[26]	
Bi	[140]		[150]		[120]		[100]	
Ce	[32]		[45]		[48]		[53]	
Co	[5.8]		[6.0]		[4.7]		[2.9]	
Cu	1,190		1,170		822		807	
Dy	--		--		[1.7]		--	
Eu	[1.3]		[1.7]		[0.86]		[1.1]	
K	1,340		1,120		657		696	
La	19.8		18.9		21.9		17.9	
Li	--		--		[0.85]		[0.70]	
Mn	222		227		214		193	
Mo	[27]		[30]		[27]		[27]	
Nd	[35]		41.6		57.6		45.7	
P	[450]		[470]		[470]		[480]	
Pd	--		--		--		--	
Rh	--		--		--		--	
Ru	[13]		[12]		[18]		[14]	
S	9,720		8,930		9,470		8,870	
Si	697		656		492		482	
Sn	286		[160]		[71]		[58]	
Sr	27.3		26.2		23.7		21.6	
Ta	--		--		--		--	
Te	--		--		--		--	
Th	--		--		--		--	
Ti	197		205		138		127	
W	--		--		--		--	
Y	[4.4]		[4.7]		5.08		[4.5]	

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QC Performance 3/16/2011

Criteria >	≤ 20%	≤ 20%	≤ 20%	≤ 20%	≤ 20%	≤ 20%	≤ 20%	≤ 20%	80%-120%	80%-120%
QC ID >	11-0419 Dup		11-0420 Dup		11-0421 Dup		11-0422 Dup		LCS/BS	
Analytes	RPD (%)	RPD (%)	RPD (%)	RPD (%)	RPD (%)	RPD (%)	RPD (%)	RPD (%)	%Rec	%Rec
Ag									96	
Al	0.4		7.6		0.8		2.1		101	
As		9.9		0.6						85
Ba	3.8		5.5		3.3		6.9		97	
Be	37.7		58.5		144.1		9.3		98	
Ca	12.4		14.6		11.6		10.6		99	
Cd	7.0		15.0		13.2		0.1		100	
Cr	24.5		51.0		4.6		3.0		95	
Fe	2.1		2.9		24.4		13.7		105	
Mg	12.1		25.4		10.8		1.1		102	
Na	7.8		14.9		6.4		8.5		103	
Ni	36.5		47.3		22.0		0.8		101	
Pb	0.0		10.2		0.8		8.7		100	
Sb										89
Se										89
Tl										84
U	9.6		11.7		13.0		8.9		100	
V									94	
Zn	16.3		27.3		47.3		5.3		93	
Zr	9.8		86.4		135.0		5.7		104	
Other Analytes										
B									96	
Bi										
Ce	5.7		12.1						97	
Co										
Cu	44.5		4.7		1.3		1.8		105	
Dy										
Eu			1.0							
K	0.7		5.8		18.0		5.6		101	
La	0.5		3.6		4.6		20.0		103	
Li									100	
Mn	16.3		5.4		2.4		10.6		104	
Mo									101	
Nd	6.3		6.5				23.1		99	
P									97	
Pd										
Rh										
Ru										
S	14.1		14.8		8.5		6.5			
Si	9.9		2.9		6.0		2.0		103	
Sn										
Sr	9.8		14.1		4.2		9.1		101	
Ta										
Te										
Th									101	
Ti	25.6		2.2		3.7		8.8		104	
W										
Y	6.6		4.3							

Shaded results are outside the acceptance criteria.

nr = spike concentration less than 25% of sample concentration. Matrix effects can be assessed from the serial dilution.

Battelle PNNL/RPG/Inorganic Analysis ... ICPOES Data Report

Criteria >	80%-120%	80%-120%	80%-120%	80%-120%	80%-120%	80%-120%	≤ 10%	≤ 10%
QC ID >	11-0421 MS		11-0421 + AS-A		11-0421 + AS-B		11-0421 5-fold Serial Dil	
Analytes	%Rec	%Rec	%Rec	%Rec	%Rec	%Rec	%Diff	%Diff
Ag	100		99					
Al	nr		102				1.6	
As		90		97				
Ba	104		101				3.1	
Be	105		103					
Ca	nr		101				1.8	
Cd	90		104					
Cr	102		101				0.2	
Fe	nr		99				2.1	
Mg	118		101				8.3	
Na	119		100				0.3	
Ni	105		105				2.0	
Pb	105		100					
Sb		45		95				
Se		96		99				
Tl		30		92				
U	nr				98		1.7	
V	99		99					
Zn	74		99				6.8	
Zr	244		104				3.5	
Other Analytes								
B	95		104					
Bi			95					
Ce	99				102			
Co			100					
Cu	nr		104				0.5	
Dy					102			
Eu					99			
K	101		103				16.5	
La	103				103			
Li	109		102					
Mn	107		103				1.1	
Mo	88		100					
Nd	105				103			
P	116		103					
Pd					99			
Rh					104			
Ru					102			
S	nr				102			
Si	31		115				42.6	
Sn			103					
Sr	114		104				2.5	
Ta			103					
Te					101			
Th	99				102			
Ti	92		105				13.7	
W			109					
Y			100					

Shaded results are outside the acceptance criteria.

nr = spike concentration less than 25% of sample concentration. Matrix effects can be assessed from the serial dilution.

ASR 8798 Final from C0354-Rep S. Fiskum (ASR-8798 129's 1x).XLS

TIC/TOC Report – Furnace Oxidation Method

Project Number:	52578
Charge Code:	H41215
ASR Number:	8798
Client:	SK Fiskum
Total Samples:	5

RPL Sample ID	Client sample ID
11-0419 -C-S,-D	KW-210-A1 / TI001-A1-H,J
11-0420 -C-S,-D	KW-210-A4 / TI002-A4-H,J
11-0421 -C-S,-D	KW-210-B1 / TI002-B1-H,J
11-0422 -C-S,-D	KW-210-B3 / TI001-B3-H,J
11-0423 -C-S,-D	Purolite® +Grafoil®

Analysis Procedure	RPG-CMC-385, Rev. 2, *Carbon Measured in Solids, Sludges, and Liquid Matrices*
Prep Procedure	None
Analyst	D Kallsen and C Rutherford
Analysis Date	6/3/2011 to 6/28/2011
Cal/Verify Standards	TOC: Sigma 99.5% Glucose CMS#239432 TIC: Mallinckrodt CMS#239430(ICV/CCV)
LCS Standards	TOC: Sigma 99.5% Glucose CMS#332698 (LCS) TIC: JT Baker CMS#239496 (LCS)
Excel Data Files	TC-11-3 Fiskum/Shimsky 8798 directory (11 files)
M&TE Numbers	Carbon System (WD13071-701)
	Balance (360-06-01-023/37050057)
All Analysis Records	System File TC-11-3

Karl N. Kool For D. Baldwin 7/20/11
Prepared By **Date**

M W Thiss 7/20/11
Reviewed By **Date**

TIC/TOC Report – Furnace Oxidation Method

Introduction

The total organic carbon/total inorganic carbon (TOC/TIC) analyses of the samples submitted under Analytical Service Request (ASR) 8798 were performed by the furnace oxidation method using the "Sequential Temperatures" option described in ASO procedure, RPG-CMC-385, Rev.2, *Carbon Measured in Solids, Sludges, and Liquid Matrices.* Controlling documents for this work were ASO-QAP-001 and the ASR 8798 Special Instructions.

The purpose of the sequential temperatures mode is to minimize impact of possible non-homogeneity in sample material. Therefore, instead of TOC and TIC/total carbon (TC) being run on separate samples, they are run on a single sample at sequential temperatures. The furnace is first run at the initial temperature of 600°C and all TOC measured, then the temperature is ramped to the final temperature of 1000°C and TIC is measured. TC is determined by the summation of TIC and TOC concentration values.

Samples

The core sample ID, the sample ID, RPL ID, and analysis date are shown in Table 1. The two Purolite® +Grafoil® mixed samples were analyzed at the beginning and end of the batches. The Special Instructions request all core samples be analyzed in at least duplicate. Because of the potential nonhomogeneous nature of these samples, all core samples were prepared and run in triplicate.

Table 1. Listing of Core Sample ID, Sample ID, RPL ID, and Analysis Date

Sample ID	RPL ID	Analysis date
KW-210-A1		
TI001-A1-H	11- 0419-C-S	6/7/2011
TI001-A1-J	11-0419-C-D	6/8/2011
KW-210-A4		
TI002-A4-H	11-0420-C-S	6/9/2011
TI002-A4-J	11-0420-C-D	6/14/2011
KW-210-B1		
TI002-B1-H	11-0421-C-S	6/15/2011
TI002-B1-J	11-0421-C-D	6/16/2011
KW-210-B3		
TI001-B3-H	11-0422-C-S	6/21/2011
TI001-B3-J	11-0422-C-D	6/23/2011
Purolite® + Grafoil®	11-0423-C-S	6/3/2011
Purolite® + Grafoil®	11-0423-C-D	6/28/2011

TIC/TOC Report – Furnace Oxidation Method

TOC/TIC Preparation

The samples were dried prior to sub-sampling and weighing. Drying was conducted by heating to 105°C for several hours or overnight, until constant dry mass was obtained and documented. The samples were sub-sampled at the workstation to provide (for each sample) a sample and duplicate, then each sample or duplicate was measured in triplicate. Two of the samples per analytical batch were selected for sub-sampling again to support two analytical spikes (AS). These were 11-0419-C-S (TI001-A1-H) and 11-0422-C-D (TI001-B3-J). Sub-sampling at the workstation required care to provide representative subsamples to optimize chances to meet precision measurement requirements; however, it is understood that sub-sampling was conducted on a best-effort basis, as per the ASR special instructions.

The samples were analyzed per RPG-CMC-385, Rev. 2, *Carbon Measured in Solids, Sludge, and Liquid Matrices*, sequential temperatures mode. The TOC was determined from combustion at 600°C; the same sample material was then subjected to 1000°C to measure all other (inorganic) C.

The ASO analyst prepared each sample in triplicate, as well as preparing the laboratory control sample (LCS) and blank spike (BS) and analytical spike (AS) QC samples. The AS and LCS/BS samples included the carbon standards: glucose and $CaCO_3$.

A sample of dry Purolite® NRW37 resin beads combined with Grafoil® also was prepared by the ASO analyst to be run with the sample batch in duplicate at the beginning of the batches and the end of the batches—this mixed material was processed as sample 11-0423.

The TOC and TIC concentration is reported here as $\mu g\ C/g_{dry}$, where g_{dry} was determined as measured at the workstation after the above-described drying process. The solids drying information is reported in Table 13.

Discussion of Results

Table 2 provides a summary of all TOC/TIC results, with a summary of attained MDL, precision, and QC sample results reported. All sample results are corrected for average percent recovery of system calibration verification check standards and are corrected for contribution from the blank, as per procedure.

The following approach was used in deriving the average result and precision for results presented in Table 2.

> U flagged (<MDL) data is not to be included in calculations of averaged data.
> J flagged (between MDL and EQL; with EQL= 5*MDL) data will be used to calculate averaged sample results (unless there is a reason not to have confidence in the J-flagged value).
> RPDs can be calculated and reported when there are two J flagged values or one J flagged value and one non-flagged value.

TIC/TOC Report – Furnace Oxidation Method

Tables 3-12 provide the complete TOC/TIC individual results and average results, with sample weights, MDL, precision, QC sample results, and QC data flags reported.

Three method QC sample failures were observed in this work. 1) One procedural requirement is that the 1st and 2nd ICBs agree within 5ug. This QC failure was seen on one analysis (batch 6) shown on Table 8. 2) A procedural requirement is that the 1st and 2nd ICVs agree within 5%. This QC failure was seen on 5 of the 10 batches, occurring for both TOC and TIC runs. The likely cause of this QC failure was early in the day operation. This QC failure caused no negative effect on results since all performance check standard recoveries met acceptance criteria. 3) On Table 4 (batch 2), the AS for TOC failed at 132% recovery. The DQO requirement is for 75%-125% recovery. The ICB/CCBs and ICV/CCVs for this batch all met requirements. The later AS for TOC (Table 11, batch 9) came in also slightly high at 115% recovery, but met the DQO requirement. There is currently no obvious reason for this AS failure. These method QC sample failures are documented in Occurrence Report 52578-07-19-11.

Several instances of failure to meet MDL requirements were seen. The ASR Special Instructions requirement for MDL was 200 ugC/g. The MDL value for each sample is directly correlated to the sample mass used for the measurement and the instrument detection limit (IDL) for the batch. The IDL for the batch is determined using the blank data for each batch. The variation in IDL from batch to batch and the ALARA-based need to keep the sample sizes small resulted in some of the MDLs being far greater than the 200 µgC/g level. The MDL limit was exceeded on 34 sample analyses spread over all 10 batches. In most of these, the sample result was well above MDL, and those samples in which the result was near the MDL all occurred on TIC analyses. There is likely little impact on data quality.

Recoveries for the special materials analyzed, Purolite® and Grafoil®, ranged from 91 to 108% recovery for carbon, for both TOC and TIC, using the sequential temperatures mode of operation. The percent recovery of carbon is based on theoretical percent carbon for the pure materials. The special materials were analyzed in duplicate at both the beginning of this work, in batch 1 and at the end of this work, in batch 10. The precision of measuring these materials was estimated for Purolite® with RPDs of 4-6% for TOC and for Grafoil® with RPDs of 1-3% for TIC.

Table 13 shows the sample drying weight data and percent water weight loss for each later step in drying. Each analyzed TOC/TIC sample was then sub-sampled respectively from each of these dried lots. All samples were dried to constant mass at ~105°C to assure that the sample mass did not contain water; extra water content would contribute to low bias for TOC/TIC results.

TIC/TOC Report – Furnace Oxidation Method

Table 2. Summary of Sample Results with MDL, Precision, and QC

Sample ID	RPL ID	Sample Weight (g)	TOC MDL (µgC/g)	Average TOC (n=3) (µgC/g)	RSD/ RPD (%)	TIC MDL (ugC/g)	Average TIC (n=3) (µgC/g)	RSD/ RPD (%)	QC BS/LCS TOC/TIC (% Rec)	QC AS TOC/TIC (% Rec)
KW-210-A1										
TI001-A1-H	11- 0419-C-S	0.0326 0.0165 0.0058	40 80 **230**	137,000	27	59 120 **340**	4,110	83	98/95	132/97
TI001-A1-J	11-0419-C-D	0.0184 0.0241 0.0097	62 47 120	140,000	35	110 84 **210**	1,080	81	101/92	--
KW-210-A4										
TI002-A4-H	11-0420-C-S	0.0189 0.0340 0.0577	84 47 27	69,300	39	**200** 110 64	2,020	85	103/92	--
TI002-A4-J	11-0420-C-D	0.0200 0.0648 0.0362	150 47 84	87,700	38	**250** 77 140	596	18	93/92	--
KW-210-B1										
TI002-B1-H	11-0421-C-S	0.0132 0.0035 0.0188	**1500** **5600** **1100**	514,000	1	**660** **2500** **460**	2,790	n/a	115/90	--
TI002-B1-J	11-0421-C-D	0.0046 0.0101 0.0148	**2700** **1200** **830**	481,000	8	**3600** **1600** **1100**	1,800	n/a	106/80	--
KW-210-B3										
TI001-B3-H	11-0422-C-S	0.0096 0.0261 0.0053	**540** **200** **980**	395,000	15	**470** 170 **840**	833	8	99/97	--
TI001-B3-J	11-0422-C-D	0.0106 0.0219 0.0141	**350** 170 **260**	367,000	3	**420** **200** **310**	3,650	38	110/88	115/87
Client Control Samples			**TOC MDL (µgC/g)**	**Average TOC (n=2) (µgC/g)**	**RPD (%)**	**TIC MDL (ugC/g)**	**Average TIC (n=2) (µgC/g)**	**RPD (%)**		
Purolite® + Grafoil®	11-0423-C-S	(b)	**590** **320**	624,000 (%Rec = 91.0 and 94.7)	4	**610** **650**	1,060,000 (%Rec = 106.9 and 105.6) (d)	1	--	--
Purolite® + Grafoil®	11-0423-C-D	(c)	**640** **610**	707,000 (%Rec = 108.0 and 102.3)	6	180 **330**	967,000 (%Rec = 95.7 and 98.6) (d)	3	--	--

(a) n/a = one or more results were less than MDL and if only one result was affected, then an RPD and not an RSD was calculated. If two results were affected, then the RPD cannot be calculated and n/a was reported.
(b) Purolite® sample weight = 0.0029g and 0.0053g; Grafoil® sample weights = 0.0046g and 0.0044g.
(c) Purolite® sample weight = 0.0044g and 0.0047g; Grafoil® sample weights = 0.0080g and 0.0044g.
(d) %Rec based on 67.2% carbon in Purolite® and 99.5% carbon in Grafoil®.
MDLs shown **BOLD** are above the requested MDL level in the Special Instructions.

Quality Control Discussion

The performance verification standards for TOC analysis are solid pure chemicals from Sigma and VWR (α-D-glucose for TOC) and from Mallinckrodt and JT Baker (calcium carbonate for TIC). The identification of the standards and their Chemical Management System (CMS) numbers are included on the raw data benchsheets for traceability.

The QC samples for the method included calibration blanks, calibration verification check samples, sample duplicates (laboratory) and/or triplicates, laboratory control sample/blank spike (LCS/BS), and analytical spike (AS). The ASR indicates that the analyses are to be performed with QC per ASO-QAP-001 and the ASR 8798 Special Instructions.

Analysis QC

Calibration Check Standards: The calibration of the coulometer analysis system was checked by analysis of calibration verification check standards analyzed at the beginning and end of the analysis run. The average recovery from the calibration check standards is applied as a correction factor to obtain the final result for the samples. The average recovery for all batches for TOC ranged from 99.9% to 107.2% recovery, and for TIC the average recovery ranged from 94.8% to 100.7%; within the method DQO of 90 to110% for the initial calibration verification (ICV) sample and 85 to 115% for the continuing calibration verification (CCV) samples, meeting the DQO criteria.

Laboratory Control Sample/Blank Spike: An LCS/BS was analyzed in each of the eight core sample daily batches, not including the two special materials batches, from 6/7/2011 to 6/23/2011, for a total of 8 LCS/BS samples. The % recovery for TOC ranged from 93.1% to 115.4% recovery, and for TIC ranged from 80.2% to 96.7% recovery. The LCS/BS recoveries are awithin acceptance criterion of 75% to 125%.

Duplicates/Triplicates: Precision of the carbon measurements is demonstrated by calculating the relative percent difference (RPD) between sample and duplicate, or relative standard deviation (RSD) between sample, duplicate, and triplicate. No duplicates were provided by the client, so laboratory-prepared duplicates and triplicates were prepared and analyzed. Each core sample was analyzed in triplicate, due to potential homogeneity concerns, while the special materials of Purolite® and Grafoil® were analyzed in duplicate. Precision in each case was determined based on results for all three replicates for the core samples or duplicates for the special materials.

For the special materials Purolite® and Grafoil®, the RPD met the DQO of < 20% RPD for both TOC and TIC, for all samples. The TOC RPD values ranged from 4% to 6% and the TIC RPD ranged from 1% to 3%.

The RSD values for TOC ranged from 1% to 39%. The RPD/RSD for TIC were n/a for two batches, and ranged from 8-85% RPD/RSD in the remaining batches. The RPD/RSD exceeding acceptance limits are documented in Occurrence Report 52578-07-19-11.

Analytical Spike: The accuracy of the carbon measurements can be estimated by the recovery from the AS. An analytical spike was prepared from solid TIC standard and solid TOC standard, and was used as analytical spikes on two samples: batch 2: 11-0419-C-S (TI001-A1-H) and batch 9: 11-0422 (TI001-B3-J). The AS recovery was 131.8% and 115.4% recovery for TOC and was 97.1% and 87.5% recovery for TIC. Of the four recovery values, three met the acceptance criteria of 75% to 125%, while one value for TOC, for TI001-A1-H, failed the acceptance criteria.

Deviation from Procedure:

TIC/TOC Report – Furnace Oxidation Method

None.

General Comments

1) Routine precision and accuracy are typically ±15% or better for non-complex samples that are free of interferences.
2) The estimated quantitation limit (EQL) is defined as 5 times the MDL. Results <5xMDL have higher uncertainties, and RPDs (or RSDs, if applicable) are not calculated if the results are <5xMDL.
3) For TOC and TIC, the analysis IDL, in µgC, is based on the standard deviation calculated from the number (n) of system blanks analyzed with the batch of samples, with the standard deviation multiplied by the Student's t values for n-1 degrees of freedom to establish the daily analysis. The MDL, in µgC/g, is sample-size dependent and based on the IDL and sample weight.

Spreadsheet Attachments:

1) 8798 Table 3
2) 8798 Table 4
3) 8798 Table 5
4) 8798 Table 6
5) 8798 Table 7
6) 8798 Table 8
7) 8798 Table 9
8) 8798 Table 10
9) 8798 Table 11
10) 8798 Table 12
11) 8798 Solids Drying Table 13

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Final Reported Carbon Results for ASR 8798

		Carbon @600C (TOC)							Carbon @1000C (TIC)						
RPL Number	Sample ID	Smpl Wt g	MDL ugC/g	Results ugC/g	Ref %Rec	QC Flag	Average ugC/g	*%RSD or* RPD(%)	Smpl Wt g	MDL ugC/g	Results ugC/g	Ref %Rec	QC Flag	Average ugC/g	*%RSD or* RPD(%)
11-0423-C-S	Purolite Beads/Grafoil	0.0029	590	612,000	91.0				0.0046	610	1,060,000	106.9			
11-0423-C-D	Purolite Beads/Grafoil	0.0053	320	636,000	94.7		624,000	4	0.0044	650	1,050,000	105.6		1,060,000	1

Ref %Rec = Measured recovery based on Special Material "Reference" carbon value.
TIC = Total Inorganic Carbon TOC = Total Organic Carbon RPD = Relative Percent Difference %RSD = %Relative Standard Deviation *(italics)* MDL = Method Detection Limit n/a = not applicable

QC Data Flags:
U = No carbon measured (detected) above the MDL
J = Carbon measured above MDL but less than the estimated quantitation limit (<5*MDL); results are qualitative

Quality Control Evaluations

Instrument QC	QC Acceptance Criteria	Comments
ICB (Proc)	1st/2nd ICB to within 5 ug	Acceptance Criteria Met
CCB (Proc)	Within 15% or 5 ug of 1st ICB	Acceptance Criteria Met for All Analyses
ICV - A (QAP)	1st/2nd Recovery within 90-110%	Acceptance Criteria Met
ICV - B (QAP)	1st/2nd ICV RPD ≤5%	Acceptance Criteria Met
CCV (QAP)	Recovery within 85-115%	Acceptance Criteria Met for All Analyses

Analysis QC	QC Acceptance Criteria		Comments
Duplicate (QAP/Client)	RPD less than or equal to (%)	20	Acceptance Criteria Met for All Analyses
Multiple Reps (Client)	%RSD less than or equal to (%)	20	Acceptance Criteria Met for All Analyses

Final Reported Carbon Results for ASR 8798

RPL Number	Sample ID	Carbon @600C (TOC) Smpl Wt g	MDL ugC/g	Results ugC/g	QC Flag	Average ugC/g	%RSD or RPD(%)	Carbon @1000C (TIC) Smpl Wt g	MDL ugC/g	Results ugC/g	QC Flag	Average ugC/g	%RSD or RPD(%)	Estimated TC (Nominal)(a) Results ugC/g	Average ugC/g	%RSD or RPD(%)
11-0419-C-S	TI001-A1-H	0.0326	40	131,000				0.0326	59	7,880				139,000		
11-0419-C-Dup	TI001-A1-H	0.0165	80	178,000				0.0165	120	3,190				181,000		
11-0419-C-Trip	TI001-A1-H	0.0058	230	104,000		137,000	*27*	0.0058	340	1,240	J	4,110	*83*	105,000	141,000	*27*

QC Results		Carbon @600C (TOC) % Recovery	Carbon @1000C (TIC) % Recovery	Estimated TC (Nominal)(a) % Recovery
Blank Spike/LCS	LCS/BS	98	95	n/a
11-0419-C-S	Analytical Spike	132	97	112
0	Analytical Spike	n/a	n/a	n/a
		n/a	n/a	n/a

(a) Estimated TC (Nominal) is sum of TIC and TOC and represents an estimate of the total carbon concentration based on the sum of "measured" and non-detected (U-flagged) results.
TIC = Total Inorganic Carbon TOC = Total Organic Carbon RPD = Relative Percent Difference %RSD = %Relative Standard Deviation *(italics)* MDL = Method Detection Limit n/a = not applicable

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ICB (Proc)	1st/2nd ICB to within 5 ug	Acceptance Criteria Met
CCB (Proc)	Within 15% or 5 ug of 1st ICB	Acceptance Criteria Met for All Analyses
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ICV - B (QAP)	1st/2nd ICV RPD ≤5%	Acceptance Criteria Met
CCV (QAP)	Recovery within 85-115%	Acceptance Criteria Met for All Analyses

Analysis QC	QC Acceptance Criteria		Comments
LCS/BS (QAP/Client)	Percent Recovery within (%)	75 to 125	Acceptance Criteria Met
Duplicate (QAP/Client)	RPD less than or equal to (%)	20	Acceptance Criteria Met for All Analyses
AS (QAP/Client)	Pecent Recovery within (%)	75 to 125	Acceptance Criteria Not Met. See Narrative for Discussion of Failure(s)
Multiple Reps (Client)	%RSD less than or equal to (%)	20	Acceptance Criteria Not Met (Failure(s) Flagged with "*" in Table); See Narrative for Discussion of Failure(s)

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Final Reported Carbon Results for ASR 8798

		Carbon @600C (TOC)						Carbon @1000C (TIC)						Estimated TC (Nominal)(a)		
RPL Number	Sample ID	Smpl Wt g	MDL ugC/g	Results ugC/g	QC Flag	Average ugC/g	*%RSD or* RPD(%)	Smpl Wt g	MDL ugC/g	Results ugC/g	QC Flag	Average ugC/g	*%RSD or* RPD(%)	Results ugC/g	Average ugC/g	*%RSD or* RPD(%)
11-0419-C-D	TI001-A1-J	0.0184	62	130,000				0.0184	110	644				131,000		
11-0419-C-D-Dup	TI001-A1-J	0.0241	47	193,000				0.0241	84	2,090				195,000		
11-0419-C-D-Trip	TI001-A1-J	0.0097	120	97,400		140,000	*35*	0.0097	210	507	J	1,080	*81*	97,900	141,000	*35*

QC Results		Carbon @600C (TOC) % Recovery	Carbon @1000C (TIC) % Recovery	Estimated TC (Nominal)(a) % Recovery
Blank Spike/LCS	LCS/BS	101	92	n/a
		n/a	n/a	
0	Analytical Spike	n/a	n/a	n/a
		n/a	n/a	n/a

(a) Estimated TC (Nominal) is sum of TIC and TOC and represents an estimate of the total carbon concentration based on the sum of "measured" and non-detected (U-flagged) results.
TIC = Total Inorganic Carbon TOC = Total Organic Carbon RPD = Relative Percent Difference %RSD = %Relative Standard Deviation *(italics)* MDL = Method Detection Limit n/a = not applicable

QC Data Flags:
U = No carbon measured (detected) above the MDL
J = Carbon measured above MDL but less than the estimated quantitation limit (<5*MDL); results are qualitative

Quality Control Evaluations

Instrument QC	QC Acceptance Criteria	Comments
ICB (Proc)	1st/2nd ICB to within 5 ug	Acceptance Criteria Met
CCB (Proc)	Within 15% or 5 ug of 1st ICB	Acceptance Criteria Met for All Analyses
ICV - A (QAP)	1st/2nd Recovery within 90-110%	Acceptance Criteria Met
ICV - B (QAP)	1st/2nd ICV RPD ≤5%	Acceptance Criteria Not Met; See Narrative for Discussion of Failure(s)
CCV (QAP)	Recovery within 85-115%	Acceptance Criteria Met for All Analyses

Analysis QC	QC Acceptance Criteria		Comments
LCS/BS (QAP/Client)	Percent Recovery within (%)	75 to 125	Acceptance Criteria Met
Duplicate (QAP/Client)	RPD less than or equal to (%)	20	Acceptance Criteria Met for All Analyses
AS (QAP/Client)	Pecent Recovery within (%)	75 to 125	Acceptance Criteria Met
Multiple Reps (Client)	%RSD less than or equal to (%)	20	Acceptance Criteria Not Met (Failure(s) Flagged with "*" in Table); See Narrative for Discussion of Failure(s)

Final Reported Carbon Results for ASR 8798

RPL Number	Sample ID	Carbon @600C (TOC) Smpl Wt g	MDL ugC/g	Results ugC/g	QC Flag	Average ugC/g	%RSD or RPD(%)	Carbon @1000C (TIC) Smpl Wt g	MDL ugC/g	Results ugC/g	QC Flag	Average ugC/g	%RSD or RPD(%)	Estimated TC (Nominal)(a) Results ugC/g	Average ugC/g	%RSD or RPD(%)
11-0420-C-S	TI002-A4-H	0.0189	84	40,900				0.0189	200	2,360				43,300		
11-0420-C-S-Dup	TI002-A4-H	0.0340	47	72,300				0.0340	110	3,550				75,800		
11-0420-C-S-Trip	TI002-A4-H	0.0577	27	94,700		69,300	*39*	0.0577	64	163	J	2,020	*85*	94,900	71,300	*37*

QC Results		Carbon @600C (TOC) % Recovery	Carbon @1000C (TIC) % Recovery	Estimated TC (Nominal)(a) % Recovery
Blank Spike/LCS	LCS/BS	103	92	n/a
		n/a	n/a	n/a
0	Analytical Spike	n/a	n/a	n/a
		n/a	n/a	n/a

(a) Estimated TC (Nominal) is sum of TIC and TOC and represents an estimate of the total carbon concentration based on the sum of "measured" and non-detected (U-flagged) results.
TIC = Total Inorganic Carbon TOC = Total Organic Carbon RPD = Relative Percent Difference %RSD = %Relative Standard Deviation *(italics)* MDL = Method Detection Limit n/a = not applicable

QC Data Flags:
U = No carbon measured (detected) above the MDL
J = Carbon measured above MDL but less than the estimated quantitation limit (<5*MDL); results are qualitative

Quality Control Evaluations

Instrument QC	QC Acceptance Criteria	Comments
ICB (Proc)	1st/2nd ICB to within 5 ug	Acceptance Criteria Met
CCB (Proc)	Within 15% or 5 ug of 1st ICB	Acceptance Criteria Met for All Analyses
ICV - A (QAP)	1st/2nd Recovery within 90-110%	Acceptance Criteria Met
ICV - B (QAP)	1st/2nd ICV RPD ≤5%	Acceptance Criteria Not Met; See Narrative for Discussion of Failure(s)
CCV (QAP)	Recovery within 85-115%	Acceptance Criteria Met for All Analyses

Analysis QC	QC Acceptance Criteria		Comments
LCS/BS (QAP/Client)	Percent Recovery within (%)	75 to 125	Acceptance Criteria Met
Duplicate (QAP/Client)	RPD less than or equal to (%)	20	Acceptance Criteria Met for All Analyses
AS (QAP/Client)	Pecent Recovery within (%)	75 to 125	n/a
Multiple Reps (Client)	%RSD less than or equal to (%)	20	Acceptance Criteria Not Met (Failure(s) Flagged with "*" in Table); See Narrative for Discussion of Failure(s)

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Final Reported Carbon Results for ASR 8798

		Carbon @600C (TOC)						Carbon @1000C (TIC)						Estimated TC (Nominal)(a)		
RPL Number	Sample ID	Smpl Wt g	MDL ugC/g	Results ugC/g	QC Flag	Average ugC/g	*%RSD or* RPD(%)	Smpl Wt g	MDL ugC/g	Results ugC/g	QC Flag	Average ugC/g	*%RSD or* RPD(%)	Results ugC/g	Average ugC/g	*%RSD or* RPD(%)
11-0420-C-D	TI002-A4-J	0.0200	150	51,500				0.0200	250	590	J			52,100		
11-0420-C-D-Dup	TI002-A4-J	0.0648	47	93,900				0.0648	77	491				94,400		
11-0420-C-D-Trip	TI002-A4-J	0.0362	84	118,000		87,700	*38*	0.0362	140	707		596	*18*	118,000	88,300	*38*

QC Results		Carbon @600C (TOC) % Recovery	Carbon @1000C (TIC) % Recovery	Estimated TC (Nominal)(a) % Recovery
Blank Spike/LCS	LCS/BS	93	92	n/a
		n/a	n/a	n/a
0	Analytical Spike	n/a	n/a	n/a
		n/a	n/a	n/a

(a) Estimated TC (Nominal) is sum of TIC and TOC and represents an estimate of the total carbon concentration based on the sum of "measured" and non-detected (U-flagged) results.
TIC = Total Inorganic Carbon TOC = Total Organic Carbon RPD = Relative Percent Difference %RSD = %Relative Standard Deviation *(italics)* MDL = Method Detection Limit n/a = not applicable

QC Data Flags:
U = No carbon measured (detected) above the MDL
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Quality Control Evaluations

Instrument QC	QC Acceptance Criteria	Comments
ICB (Proc)	1st/2nd ICB to within 5 ug	Acceptance Criteria Met
CCB (Proc)	Within 15% or 5 ug of 1st ICB	Acceptance Criteria Met for All Analyses
ICV - A (QAP)	1st/2nd Recovery within 90-110%	Acceptance Criteria Met
ICV - B (QAP)	1st/2nd ICV RPD ≤5%	Acceptance Criteria Not Met; See Narrative for Discussion of Failure(s)
CCV (QAP)	Recovery within 85-115%	Acceptance Criteria Met for All Analyses

Analysis QC	QC Acceptance Criteria		Comments
LCS/BS (QAP/Client)	Percent Recovery within (%)	75 to 125	Acceptance Criteria Met
Duplicate (QAP/Client)	RPD less than or equal to (%)	20	Acceptance Criteria Met for All Analyses
AS (QAP/Client)	Pecent Recovery within (%)	75 to 125	n/a
Multiple Reps (Client)	%RSD less than or equal to (%)	20	Acceptance Criteria Not Met (Failure(s) Flagged with "*" in Table); See Narrative for Discussion of Failure(s)

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Final Reported Carbon Results for ASR 8798

		Carbon @600C (TOC)						Carbon @1000C (TIC)						Estimated TC (Nominal)(a)		
RPL Number	Sample ID	Smpl Wt g	MDL ugC/g	Results ugC/g	QC Flag	Average ugC/g	*%RSD or* RPD(%)	Smpl Wt g	MDL ugC/g	Results ugC/g	QC Flag	Average ugC/g	*%RSD or* RPD(%)	Results ugC/g	Average ugC/g	*%RSD or* RPD(%)
11-0421-C-S	TI002-B1-H	0.0132	1,500	518,000				0.0132	660	< 660	U			518,000		
11-0421-C-S-Dup	TI002-B1-H	0.0035	5,600	516,000				0.0035	2,500	2,790	J	2,790	n/a	518,000		
11-0421-C-S-Trip	TI002-B1-H	0.0188	1,100	510,000		514,000	*1*	0.0188	460	< 460	U			510,000	516,000	*1*

QC Results		Carbon @600C (TOC) % Recovery	Carbon @1000C (TIC) % Recovery	Estimated TC (Nominal)(a) % Recovery
Blank Spike/LCS	LCS/BS	115	90	n/a
		n/a	n/a	
0	Analytical Spike	n/a	n/a	
		n/a	n/a	n/a

(a) Estimated TC (Nominal) is sum of TIC and TOC and represents an estimate of the total carbon concentration based on the sum of "measured" and non-detected (U-flagged) results.
TIC = Total Inorganic Carbon TOC = Total Organic Carbon RPD = Relative Percent Difference %RSD = %Relative Standard Deviation *(italics)* MDL = Method Detection Limit n/a = not applicable

QC Data Flags:
U = No carbon measured (detected) above the MDL
J = Carbon measured above MDL but less than the estimated quantitation limit (<5*MDL); results are qualitative

Quality Control Evaluations

Instrument QC	QC Acceptance Criteria	Comments
ICB (Proc)	1st/2nd ICB to within 5 ug	Acceptance Criteria Not Met; See Narrative for Discussion of Failure(s)
CCB (Proc)	Within 15% or 5 ug of 1st ICB	Acceptance Criteria Met for All Analyses
ICV - A (QAP)	1st/2nd Recovery within 90-110%	Acceptance Criteria Met
ICV - B (QAP)	1st/2nd ICV RPD ≤5%	Acceptance Criteria Not Met; See Narrative for Discussion of Failure(s)
CCV (QAP)	Recovery within 85-115%	Acceptance Criteria Met for All Analyses

Analysis QC	QC Acceptance Criteria		Comments
LCS/BS (QAP/Client)	Percent Recovery within (%)	75 to 125	Acceptance Criteria Met
Duplicate (QAP/Client)	RPD less than or equal to (%)	20	Acceptance Criteria Met for All Analyses
AS (QAP/Client)	Pecent Recovery within (%)	75 to 125	Acceptance Criteria Met
Multiple Reps (Client)	%RSD less than or equal to (%)	20	Acceptance Criteria Met for All Analyses

Final Reported Carbon Results for ASR 8798

RPL Number	Sample ID	Carbon @600C (TOC) Smpl Wt g	MDL ugC/g	Results ugC/g	QC Flag	Average ugC/g	*%RSD or* RPD(%)	Carbon @1000C (TIC) Smpl Wt g	MDL ugC/g	Results ugC/g	QC Flag	Average ugC/g	*%RSD or* RPD(%)	Estimated TC (Nominal)(a) Results ugC/g	Average ugC/g	*%RSD or* RPD(%)
11-0421-C-D	TI002-B1-J	0.0046	2,700	439,000				0.0046	3,600	< 3600	U			443,000		
11-0421-C-D-Dup	TI002-B1-J	0.0101	1,200	505,000				0.0101	1,600	1,800	J	1,800	n/a	507,000		
11-0421-C-D-Trip	TI002-B1-J	0.0148	830	499,000		481,000	*8*	0.0148	1,100	< 1100	U			500,000	483,000	*8*

QC Results		Carbon @600C (TOC) % Recovery	Carbon @1000C (TIC) % Recovery	Estimated TC (Nominal)(a) % Recovery
Blank Spike/LCS	LCS/BS	106	80	n/a
		n/a	n/a	
0	Analytical Spike	n/a	n/a	
		n/a	n/a	n/a

(a) Estimated TC (Nominal) is sum of TIC and TOC and represents an estimate of the total carbon concentration based on the sum of "measured" and non-detected (U-flagged) results.
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Final Reported Carbon Results for ASR 8798

		Carbon @600C (TOC)						Carbon @1000C (TIC)						Estimated TC (Nominal)(a)		
RPL Number	Sample ID	Smpl Wt g	MDL ugC/g	Results ugC/g	QC Flag	Average ugC/g	*%RSD or* RPD(%)	Smpl Wt g	MDL ugC/g	Results ugC/g	QC Flag	Average ugC/g	*%RSD or* RPD(%)	Results ugC/g	Average ugC/g	*%RSD or* RPD(%)
11-0422-C-S	TI001-B3-H	0.0096	540	356,000				0.0096	470	800	J			356,000		
11-0422-C-S-Dup	TI001-B3-H	0.0261	200	366,000		361,000	0	0.0261	170	865		833	8	367,000	362,000	0
11-0422-C-S-Trip	TI001-B3-H	0.0053	980	464,000		395,000	*15*	0.0053	840	< 840	U			465,000	396,000	*15*

QC Results		Carbon @600C (TOC) % Recovery	Carbon @1000C (TIC) % Recovery	Estimated TC (Nominal)(a) % Recovery
Blank Spike/LCS	LCS/BS	99	97	n/a
		n/a	n/a	
0	Analytical Spike	n/a	n/a	
		n/a	n/a	n/a

(a) Estimated TC (Nominal) is sum of TIC and TOC and represents an estimate of the total carbon concentration based on the sum of "measured" and non-detected (U-flagged) results.
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Quality Control Evaluations

Instrument QC	QC Acceptance Criteria	Comments
ICB (Proc)	1st/2nd ICB to within 5 ug	Acceptance Criteria Met
CCB (Proc)	Within 15% or 5 ug of 1st ICB	Acceptance Criteria Met for All Analyses
ICV - A (QAP)	1st/2nd Recovery within 90-110%	Acceptance Criteria Met
ICV - B (QAP)	1st/2nd ICV RPD ≤5%	Acceptance Criteria Met
CCV (QAP)	Recovery within 85-115%	Acceptance Criteria Met for All Analyses

Analysis QC	QC Acceptance Criteria		Comments
LCS/BS (QAP/Client)	Percent Recovery within (%)	75 to 125	Acceptance Criteria Met
Duplicate (QAP/Client)	RPD less than or equal to (%)	20	Acceptance Criteria Met for All Analyses
AS (QAP/Client)	Pecent Recovery within (%)	75 to 125	Acceptance Criteria Met
Multiple Reps (Client)	%RSD less than or equal to (%)	20	Acceptance Criteria Met for All Analyses

PNNL Radiochemical Sciences and Engineering Group
TOC and TIC Data Analysis and Report

Sequential Furnace Oxidation Worksheet
ASR: 8798

Final Reported Carbon Results for ASR 8798

		Carbon @600C (TOC)						Carbon @1000C (TIC)						Estimated TC (Nominal)(a)		
RPL Number	Sample ID	Smpl Wt g	MDL ugC/g	Results ugC/g	QC Flag	Average ugC/g	*%RSD or* RPD(%)	Smpl Wt g	MDL ugC/g	Results ugC/g	QC Flag	Average ugC/g	*%RSD or* RPD(%)	Results ugC/g	Average ugC/g	*%RSD or* RPD(%)
11-0422-C-D	TI001-B3-J	0.0106	350	353,000				0.0106	420	5,180				359,000		
11-0422-C-D-Dup	TI001-B3-J	0.0219	170	378,000				0.0219	200	3,300				381,000		
11-0422-C-D-Trip	TI001-B3-J	0.0141	260	371,000		367,000	*3*	0.0141	310	2,470		3,650	*38*	373,000	371,000	*3*

QC Results		Carbon @600C (TOC) % Recovery	Carbon @1000C (TIC) % Recovery	Estimated TC (Nominal)(a) % Recovery
Blank Spike/LCS	LCS/BS	110	88	n/a
11-0422-C-D	Analytical Spike	115	87	100
0	Analytical Spike	n/a	n/a	
		n/a	n/a	n/a

(a) Estimated TC (Nominal) is sum of TIC and TOC and represents an estimate of the total carbon concentration based on the sum of "measured" and non-detected (U-flagged) results.
TIC = Total Inorganic Carbon TOC = Total Organic Carbon RPD = Relative Percent Difference %RSD = %Relative Standard Deviation *(italics)* MDL = Method Detection Limit n/a = not applicable

QC Data Flags:
U = No carbon measured (detected) above the MDL
J = Carbon measured above MDL but less than the estimated quantitation limit (<5*MDL); results are qualitative

Quality Control Evaluations

Instrument QC	QC Acceptance Criteria	Comments
ICB (Proc)	1st/2nd ICB to within 5 ug	Acceptance Criteria Met
CCB (Proc)	Within 15% or 5 ug of 1st ICB	Acceptance Criteria Met for All Analyses
ICV - A (QAP)	1st/2nd Recovery within 90-110%	Acceptance Criteria Met
ICV - B (QAP)	1st/2nd ICV RPD ≤5%	Acceptance Criteria Met
CCV (QAP)	Recovery within 85-115%	Acceptance Criteria Met for All Analyses

Analysis QC	QC Acceptance Criteria		Comments
LCS/BS (QAP/Client)	Percent Recovery within (%)	75 to 125	Acceptance Criteria Met
Duplicate (QAP/Client)	RPD less than or equal to (%)	20	Acceptance Criteria Met for All Analyses
AS (QAP/Client)	Pecent Recovery within (%)	75 to 125	Acceptance Criteria Met
Multiple Reps (Client)	%RSD less than or equal to (%)	20	Acceptance Criteria Not Met (Failure(s) Flagged with "*" in Table); See Narrative for Discussion of Failure(s)

PNNL Radiochemical Sciences and Engineering Group
TOC and TIC Data Analysis and Report

Sequential Furnace Oxidation Worksheet
ASR: 8798

Final Reported Carbon Results for ASR 8798

		Carbon @600C (TOC)							Carbon @1000C (TIC)						
RPL Number	Sample ID	Smpl Wt g	MDL ugC/g	Results ugC/g	Ref %Rec	QC Flag	Average ugC/g	RPD(%)	Smpl Wt g	MDL ugC/g	Results ugC/g	Ref %Rec	QC Flag	Average ugC/g	RPD(%)
11-0423-C-S	Purolite Beads/Grafoil	0.0044	640	726,000	108.0				0.0080	180	953,000	95.7			
11-0423-C-D	Purolite Beads/Grafoil	0.0047	610	687,000	102.3		707,000	6	0.0044	330	981,000	98.6		967,000	3

Ref %Rec = Measured recovery based on Special Material "Reference" carbon value.
TIC = Total Inorganic Carbon TOC = Total Organic Carbon RPD = Relative Percent Difference %RSD = %Relative Standard Deviation *(italics)* MDL = Method Detection Limit n/a = not applicable

QC Data Flags:
U = No carbon measured (detected) above the MDL
J = Carbon measured above MDL but less than the estimated quantitation limit (<5*MDL); results are qualitative

Quality Control Evaluations

Instrument QC	QC Acceptance Criteria	Comments
ICB (Proc)	1st/2nd ICB to within 5 ug	Acceptance Criteria Met
CCB (Proc)	Within 15% or 5 ug of 1st ICB	Acceptance Criteria Met for All Analyses
ICV - A (QAP)	1st/2nd Recovery within 90-110%	Acceptance Criteria Met
ICV - B (QAP)	1st/2nd ICV RPD ≤5%	Acceptance Criteria Met
CCV (QAP)	Recovery within 85-115%	Acceptance Criteria Met for All Analyses

Analysis QC	QC Acceptance Criteria		Comments
Duplicate (QAP/Client)	RPD less than or equal to (%)	20	Acceptance Criteria Met for All Analyses
Multiple Reps (Client)	%RSD less than or equal to (%)	20	Acceptance Criteria Met for All Analyses

File: 8798 Solids Drying Table 13

	RPL Sample ID	Client sample ID	TARE WEIGHT (g)	INITIAL SAMPLE WEIGHT PLUS TARE (g)	1st Drying Weight (g)	2nd Drying Weight (g)	Difference from weight 1 and 2	3rd Drying Weight (g)	Difference from weight 2 and 3
				Date: 6-6-11 Time: 0800	Date: 6-6-11 Time: 1530	Date: 6-7-11 Time: 0900		Date: 6-8-11 Time: 0800	
1	11-0419-C-S	TI001-A1-H	24.5816	25.7897	25.4580	25.3978	7.38%	25.3974	0.05%
2	11-0419-C-D	TI001-A1-J	24.5729	25.2505	25.2214	25.2085	2.03%	25.2084	0.02%
				Date: 6-8-11 Time: 1530	Date: 6-9-11 Time: 0800	Date: 6-9-11 Time: 1200		Date: 6-9-11 Time: 1500	
3	11-0420-C-S	TI002-A4-H	24.6518	26.0905	25.5789	25.5757	0.35%	25.5755	0.02%
4	11-0420-C-D	TI002-A4-J	24.5934	26.6675	25.9122	25.9055	0.51%	25.9055	0.00%
				Date: 6-14-11 Time: 0800	Date: 6-14-11 Time: 1600	Date: 6-15-11 Time: 0830		Date: 6-15-11 Time: 1500	
5	11-0421-C-S	TI002-B1-H	24.5452	26.3562	25.3492	25.3427	0.82%	25.3421	0.08%
6	11-0421-C-D	TI002-B1-J	24.5051	26.6011	25.4525	25.4445	0.85%	25.4441	0.04%
				Date: 6-20-11 Time: 1430	Date: 6-21-11 Time: 0830	Date: 6-21-11 Time: 1200		Date: 6-21-11 Time: 1500	
7	11-0422-C-S	TI001-B3-H	24.6681	26.4794	25.6035	25.6030	0.05%	25.6028	0.02%
8	11-0422-C-D	TI002-B3-J	24.7512	26.2731	25.5979	25.5968	0.13%	25.5967	0.01%

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

Gamma Energy Analysis (GEA)

Project / WP#: 52578/ H41216
ASR#: 8798.00
Client: SK Fiskum
Total Samples: 9

RPL ID	Client Sample ID
11-0419-129-S, -D	TI001-A1-B, -C
11-0420-129-S, -D	TI002-A4-B, -C
11-0421-129-S, -D, -MS	TI002-B1-B, -C, -D
11-0422-129-S, -D	TI001-B3-B, -C

Analysis Type:	GEA- for all positively measured or non-detected isotopes of interest.
Sample Processing Prior to Radiochemical Processing/Analysis	☐ None ☒ Digested as per PNL-ALO-129, Rev.0 *HNO_3-HCl Acid Extraction of Solids Using a Dry Block Heater*, Analyst: N. Green (03/11/2011) ☐ Fusion as per PNL-ALO-115, Rev. 1, *Solubilization of Metals from Solids Using a KOH-KNO_3 Fusion* ☐ Other: Preparation may also involve attaining a GEA geometry that is compatible with the calibration geometry.
Analysis Procedure:	RPG-CMC-450, Rev. 1, *Gamma Energy Analysis (GEA) and Low-Energy Photon Spectrometry (LEPS)*
Reference Date:	*January 11, 2011 @ 15:14:41 p.m.
Analysis Date or Date Range:	March 14-22, 2011
Technician/Analyst:	T Trang-Le
Rad Chem Electronic Data File:	*11-0419_Fiskum.xls*
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,D,E,G,H,K,L,N,T (M & TE unique identifiers in attached supporting documents)*

T Trang-Le / 6/7/11
Prepare **Date**

Reviewer / 6/7/11
Date

*Reference date used is the same date and time used for samples submitted under ASR 8725 as directed by S. Fiskum.

SAMPLE RESULTS

Activities for all gamma emitters detected in these samples are presented in an attached Excel spreadsheet for 8798.00. 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 uncertainty 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 residual solids that did not dissolve during acid digestion.

ASO Project File, ASR-8798 has been created for this report including all appropriate supporting records which may include the Pipette Performance Verification form, Standard Certificates, Laboratory Bench Record, Shielded Analytical Laboratory Bench Sheet, 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 dried solids samples were prepared in the shielded analytical lab (SAL) hot cells by the Project and provided to the Analytical Support Operations (ASO) under Analytical Service Request (ASR) 8798 for chemical and radio-analytical analyses.

ASO received each sample in a Teflon Oak Ridge centrifuge tube, dried the contents of each centrifuge tube to a constant weight @ 105°C, and then leached the dried solids with hot dilute nitric and hydrochloric acids per procedure PNL-ALO-129. The leachate solution was used for radioanalytical analyses and ICP metals analysis; only GEA data are included in this report. At the completion of the acid digestion, residual unsolubilized material remained. The residual solids were dried @ 105° C to constant weight and the dried residuals were transferred to clean 22 mL glass scintillation vials for load out and GEA analyses. The residual solids do have measurable activity and those results are included in this report.

Batch quality control (QC) samples were prepared with the digestion of the initial dried solids and includes duplicates of each sample and an acid digestion 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. 7); 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):

An acid digestion blank was prepared with the preparation of the sludge samples in the SAL, but not for the preparation of the residual solids in the SAL.

ASO-QAP-001 requires that 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 l less than sample minimum detectable activity (MDA) – (See comments section). Cs-137 was

detected in the blank at a level less than 500,000x the level present in the samples. For isotopes of interest that were not detected in the PB and samples, the PB MDC was less than the MDC measured in the samples. The isotopes of interest that were above the MDC in the samples are all much 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 sample. 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. (The sample identified as "MS" was prepared for ICP/OES analyses only. This MS sample is also being reported for GEA as additional information for the project.)

Duplicate Relative Percent Difference (RPD):

Duplicate sample results are required to agree within <20% relative percent difference, taking into consideration the statistical uncertainties in the data. Duplicate sample results for sludge samples ranged from 0.4 to 48% RPD. The duplicate RPD Data Quality Objective of ± 20% does not apply to the residual solids results. The two sludge samples results where the ± 20% RPD was exceeded are detailed below and are documented in Occurrence Report 52578-04-18-11.

For Sludge samples:

RPL Number	Client Sample ID	Isotopes	RPD Differences
11-0419-129-S, -D	TI001-A1-B, -C	Co-60	48%
11-0421-129-S, -D	TI002-B1-B, -C	Eu-155	32%

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. 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 PNL-ALO-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 Issues

None.

Uncertainty

For gamma counting, the uncertainty in the counting data, calibration, photon abundance and the nuclear half-life are included in the calculation of the total uncertainty along with a systematic uncertainty for sample prep. The Canberra Genie2000 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.

Solids 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 ~10 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 mostly silica (however, 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 have been corrected 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. Occurrence Report 52578-04-18-11 documents DQO related issue(s) with this report: RPD's outside the <20% requirement.

For Sludge Samples:

RPL Number	Client Sample ID	Isotopes	RPD Differences
11-0419-129-S, -D	TI001-A1-B, -C	Co-60	48%
11-0421-129-S, -D	TI002-B1-B, -C	Eu-155	32%

Attachment: Data Report Sample Results for ASR 8798.

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

filename 11-0419 Fiskum
3/28/2011

Client: Fiskum
ASR 8798

Project: 52578

Prepared by: C. Soderquist 3-28-2011

Concur: T Trang-Le 3-28-2011

Procedure: PNL-ALO-129, Rev. 0 HNO_3-HCl Acid Extraction of Solids Using a Dry-Block Heater
RPG-CMC-450, Rev. 1 Gamma Energy Analysis (GEA) and Low-Energy Photon Spectrometry (LEPS)

Reference date: January 11, 2011 @ 15:14:41

M & TE: Gamma Detectors C,D,E,G,K,T

Gamma Emitters in Dry K Basin Sludge
$\mu Ci/g_{dry}$ with 1-sigma Uncertainty

Sample ID	RPL ID		Co-60	Cs-134	Cs-137	Eu-152	Eu-154	Eu-155	Am-241
TI001-A1-B	11-0419-129-S		**2.30E-1 ± 3%**	<3.E-3	**3.68E+2 ± 3%**	<5.E-3	**7.30E-1 ± 2%**	**1.18E-1 ± 7%**	**1.96E+1 ± 4%**
TI001-A1-C	11-0419-129-D		**1.41E-1 ± 3%**	<6.E-3	**3.93E+2 ± 3%**	<8.E-3	**7.59E-1 ± 2%**	**1.26E-1 ± 11%**	**2.13E+1 ± 4%**
		RPD	48%	-	7%	-	4%	7%	8%
TI002-A4-B	11-0420-129-S		**1.34E-1 ± 3%**	<3.E-3	**3.38E+2 ± 4%**	<5.E-3	**5.84E-1 ± 2%**	<6.E-2	**1.64E+1 ± 4%**
TI002-A4-C	11-0420-129-D		**1.21E-1 ± 3%**	<6.E-3	**3.81E+2 ± 4%**	<9.E-3	**5.41E-1 ± 2%**	<1.E-1	**1.39E+1 ± 4%**
		RPD	10%	-	12%	-	8%	-	17%
TI002-B1-B	11-0421-129-S		**7.34E-2 ± 3%**	<4.E-3	**1.43E+2 ± 3%**	<4.E-3	**4.71E-1 ± 2%**	**8.71E-2 ± 8%**	**1.41E+1 ± 4%**
TI002-B1-C	11-0421-129-D		**7.31E-2 ± 3%**	<3.E-3	**1.68E+2 ± 5%**	<7.E-3	**5.16E-1 ± 2%**	**1.20E-1 ± 17%**	**1.68E+1 ± 20%**
		RPD	0.4%	-	16%	-	9%	32%	17%
TI002-B1-D	11-0421-129-MS		**6.73E-2 ± 4%**	<5.E-3	**1.50E+2 ± 5%**	<1.E-2	**4.76E-1 ± 2%**	<7.E-2	**1.38E+1 ± 4%**
TI001-B3-B	11-0422-129-S		**6.86E-2 ± 3%**	<5.E-3	**1.13E+2 ± 3%**	<1.E-2	**5.28E-1 ± 2%**	**8.04E-2 ± 18%**	**1.48E+1 ± 4%**
TI001-B3-C	11-0422-129-D		**6.39E-2 ± 3%**	<5.E-3	**1.02E+2 ± 3%**	<1.E-2	**5.14E-1 ± 2%**	**7.35E-2 ± 19%**	**1.39E+1 ± 4%**
		RPD	7%	-	10%	-	3%	9%	6%
Acid digestion blank	11-0419-129-PB		<4.E-5	<4.E-5	**2.19E-4 ± 11%**	<2.E-4	<1.E-4	<2.E-4	<3.E-4
	Requested Detection Limits		1.0E-2	2.0E-1	1.5E-2	1.5E+0	1.5E+0	2.0E+0	1.5E+1

The matrix spike sample TI002-B1-D was spiked with non-radioactive metals only, and is essentially a triplicate analysis for radiochemistry.

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

filename 11-0419 Fiskum
3/30/2011

Client: Fiskum Project: 59205
ASR 8798

Prepared by: C. Soderquist 3-30-11

Concur: T Trang-Le 3/30/11

Procedure: PNL-ALO-129, Rev. 0 HNO_3-HCL Acid Extraction of Solids Using a Dry-Block Heater
RPG-CMC-450, Rev. 1 Gamma Energy Analysis (GEA) and Low-Energy Photon Spectrometry (LEPS)
Reference date: January 11, 2011 @ 15:14:41
M & TE: Gamma Detectors C,D,E,G,H,L K & N

Gamma Emitters in Acid-Insoluble Residues
Measured Activities, $\mu Ci/g_{residual}$ with 1-sigma uncertainty

Sample ID	Lab ID	Co-60	Sb-125	Sn-126	Cs-134	Cs-137	Eu-152	Eu-154	Eu-155	Am-241
TI001-A1-B	11-0419-129-S	**4.51E-4 ± 2%**	**2.52E-3 ± 7%**	**8.14E-5 ± 29%**	**1.23E-4 ± 23%**	**2.05E+0 ± 2%**	< 1E-4	**2.43E-3 ± 2%**	< 1E-3	**6.62E-2 ± 4%**
TI001-A1-C	11-0419-129-D	**5.29E-4 ± 4%**	**1.94E-3 ± 16%**	< 3E-4	< 1E-4	**2.18E+0 ± 3%**	< 1E-4	**1.75E-3 ± 5%**	< 9E-4	**4.80E-2 ± 4%**
	RPD	16%	26%	--	--	6%	--	33%	--	32%
TI002-A4-B	11-0420-129-S	**1.93E-4 ± 4%**	<8.E-4	<2.E-4	<2.E-5	**1.59E+0 ± 4%**	<5.E-5	**2.22E-3 ± 2%**	<5.E-4	**6.98E-2 ± 4%**
TI002-A4-C	11-0420-129-D	**3.76E-4 ± 4%**	**1.53E-3 ± 37%**	**3.25E-5 ± 25%**	<5.E-5	**2.77E+0 ± 2%**	<9.E-5	**2.07E-3 ± 3%**	**7.79E-4 ± 18%**	**5.37E-2 ± 4%**
	RPD	64%	--	--	--	54%	--	7%	--	26%
TI002-B1-B	11-0421-129-S	**4.51E-4 ± 3%**	**4.04E-3 ± 4%**	**4.96E-4 ± 2%**	<5.E-5	**8.67E-1 ± 2%**	<2.E-4	**2.30E-3 ± 2%**	<5.E-4	**8.72E-2 ± 3%**
TI002-B1-C	11-0421-129-D	**7.18E-4 ± 3%**	**4.69E-3 ± 5%**	**6.00E-4 ± 4%**	<3.E-5	**8.03E-1 ± 6%**	<7.E-5	**3.24E-3 ± 2%**	**3.21E-4 ± 29%**	**1.31E-1 ± 4%**
	RPD	46%	15%	19%	--	8%	--	34%	--	40%
TI002-B1-D	11-0421-129-MS	**1.04E-3 ± 5%**	**3.01E-3 ± 24%**	<2.E-4	<9.E-5	**2.23E+0 ± 5%**	<3.E-4	**3.65E-3 ± 3%**	<3.E-3	**1.48E-1 ± 20%**
TI001-B3-B	11-0422-129-S	**4.09E-4 ± 3%**	**3.41E-3 ± 5%**	**3.73E-4 ± 4%**	<2.E-5	**6.91E-1 ± 3%**	<6.E-5	**3.70E-3 ± 2%**	**5.87E-4 ± 17%**	**1.22E-1 ± 4%**
TI001-B3-C	11-0422-129-D	**4.42E-4 ± 2%**	**3.22E-3 ± 2%**	**3.98E-4 ± 2%**	<2.E-5	**5.73E-1 ± 2%**	**7.97E-5 ± 9%**	**3.39E-3 ± 2%**	**6.80E-4 ± 3%**	**1.14E-1 ± 2%**
	RPD	8%	6%	6%	--	19%	--	9%	--	7%
Requested Detection Limits		1.0E-2	--	--	2.0E-1	1.5E-2	1.5E+0	1.5E+0	2.0E+0	1.5E+1

The matrix spike sample TI002-B1-D was spiked with non-radioactive metals only, and is essentially a triplicate analysis for radiochemistry.

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	WD25687	EG&G Ortec	GEM15180P	35TP11079A	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

Project / WP#:	52578/H41216
ASR#:	8798
Client:	S. Fiskum
Total # of Samples:	4

Engineered Container & Core ID	Sample IDs	RPL Number
KW-210-A1	TI001-A1-B, -A1-C	11-0419-129-S, -129-D
KW-210-A4	TI002-A4-B, -A4-C	11-0420-129-S, -129-D
KW-210-B1	TI002-B1-B, -B1-C	11-0421-129-S, -129-D
KW-210-B3	TI001-B3-B, -B3-C	11-0422-129-S, -129-D

Analysis Type:	AEA – Pu-238, Pu-239+240
Sample Processing Prior to Radiochemical Processing/Analysis	☐ None ☒ Digested as per PNL-ALO-129, Rev. 0, *HNO_3-HCl Acid Extraction of Solids Using a Dry Block Heater, 3-7-11 (N. Green)* ☐ 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*
Total Alpha and Beta Preparation Procedure:	RPG-CMC-4001, Rev. 1, *Source Preparation For Gross Alpha and Gross Beta analyses.*
Technician/Analyst:	K. Carson, 3-28-11
Spike Standard ID's	R-569-a-3 (Sr-90), R-485-b-5 (Pu-239)
Analysis Procedure	RPG-CMC-408, Rev. 2, *Total Alpha and Beta Analysis*
Technician/Analyst:	T. Trang-Le, 3-29-11
Plutonium Separation Procedure:	RPG-CMC-4017, Rev. 0, *Analysis of Environmental Water Samples for Actinides and Strontium- 90*
Technician/Analyst:	K. Carson, 4-4-11
Spike and Tracer Standard ID's:	R-485-b-5 (Pu-239), R-533-a-7 (Pu-242 tracer)
Co-Precipitation Procedure:	RPG-CMC-496, Rev. 0, *Co-precipitation Mounting of Actinides for Alpha Spectroscopy*
Technician/Analyst:	K. Carson, 4-8-11
Analysis Procedure:	RPG-CMC-422, Rev. 2, *Solutions Analysis: Alpha Spectrometry*
Reference Date:	Same as counting dates
Analysis Date or Date Range:	4/8/11 and 4/12/11
Technician/Analyst:	T. Trang-Le
Analysis Data (File):	11-0419 Fiskum.xls, Alpha Beta
CMC Project 98620 File:	File Plan 5871: T 52578 8798: 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: 6/7/11

T Trang-Le / Reviewer / Date: 6/7/11

Sample Results

See attached data report, Pu-238 and Pu-239+240 Sample Results for ASR 8798. All data are reported in units of uCi/g-$_{dry}$ K-Basin sludge with a 1-σ uncertainty (see comments).

Sample Preparation, Separation, Mounting and Counting Methods

Four K-Basin sludge samples were prepared in the shielded analytical laboratory (SAL) hot cells by the Project and provided to the Analytical Support Operations (ASO) under Analytical Service Request (ASR) 8798 for chemical and radio chemical analyses. ASO received each sample in a Teflon Oak Ridge centrifuge tube, dried the contents of each centrifuge tube to a constant weight, and then leached the dried solids with hot dilute nitric and hydrochloric acids per procedure PNL-ALO-129. The leachate solution was used for radiochemical analyses and ICP metals analysis; only Pu-AEA is reported in this report. Visible residual solids remained following the PNL-ALO-129 digestion.

Following the digestion process, the Pu was separated from the sludge 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 April 8th and 12th, 2011; 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 Pu 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. These procedures are not required to be pre-approved by K-Basin project staff as they are used to generate opportunistic data only. The sum of the major alpha emitters, in this case Pu-238, Pu-239+240 and Am-241, agrees very well with the gross alpha results.

QUALITY CONTROL RESULTS

Quality control (QC) samples prepared in the SAL hot cells include a hot cell process blank (PB), sample duplicates for each sample, and a matrix spike (MS – for ICP/OES only). Additional QC samples were prepared prior to separations; these include a laboratory separation blank, a reagent blank spike (BS, spiked with Pu-239), and a matrix spike (MS) made by adding Pu-239 standard to a diluted sample.

Performance acceptance criteria for these samples are established by both the K Basin data quality objectives (as provided as special instruction in the ASR) and the ASO Quality Assurance (QA) Plan (ASO-QAP-001, Rev. 7). Except where noted, the Pu-AEA K-Basin DQO and ASO QAP criteria are the same. In cases where the QC criteria are different, K-Basin DQO's will take precedence. 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 Pu-242 tracer corrects for radiochemical yield and mathematically removes the detector counting efficiency from the results calculations. Tracer recover 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 94% to 108%.

Hot Cell Process Blank (PB) and Laboratory separations blank (LB):

The Pu-238 and Pu-239+240 activities 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 Pu-238 and Pu-239+240 activities are significantly less than 5% of any sample activity. There are no acceptance criteria for LB. The detection limits achieved for the Pu-238 and Pu-239+240 were below the required detection limit of 1.0E-03 $\mu Ci/g\text{-}_{dry}$

Blank Spike (BS) – reagent spike:

The BS (Pu-239) recovery of 100% meets the acceptance criteria of 80% to 120% recovery.

Matrix Spike (MS):

The MS recovery of 101% meets the acceptance criterion of 75% to 125% recovery. Note: the MS sample was prepared after digestion, by adding a known Pu-239 standard quantity to an aliquot of the leachate. Sample number 11-0422-129-S (TI001-B3-B) was selected as the matrix spike sample.

Duplicate -- Relative Percent Difference (RPD):

Duplicate results are required to agree within <20% RPD. Duplicate results range from 2% to 13% RPD; thus meeting the <20% requirement. See Assumptions and Limitations of the Data.

Instrument Quality Control

Counter control source counting is not required when samples are spiked with tracers. When tracers are used, the absolute counting efficiencies are not used.

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.

Battelle PNNL/RPL/ASO Radiochemistry Analysis Report

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 and aliquot dilution (to avoid excessive consumption of spike and avoid creating unnecessary waste). The MS prepared with this sample batch is considered a PS, since the Pu-239 spike was not added prior to the digestion process.

3. Each alpha spectrum was examined for potential interferences from other alpha emitting isotopes. Pu-242 and Am-243 tracers are added at the start of the chemical separation process. The presence of Am-243 in the Pu-AEA would indicate interference from Am-241 on the Pu-238 peak. The alpha spectra were evaluated to verify the removal of Am from the Pu. There was no evidence of interfering isotopes, including Am.

4. The 1-sigma uncertainty represents the total propagated error associated with processing and counting operations and includes weighing errors, volume uncertainties, and counting error.

5. 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.

6. The Laboratory Blank (LB) is prepared using laboratory reagents and provides data only on the cleanliness of the radiochemistry preparation process. LB results are not normalized to processing or dilution factors associated with the samples.

Attachment: Data Report -- Sample Results for ASR 8798

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

Americium 241 Analysis

Project / WP#:	52578/H41216
ASR#:	8798
Client:	S. Fiskum
Total # of Samples:	4

Engineered Container & Core ID	Sample IDs	RPL Number
KW-210-A1	TI001-A1-B, -A1-C	11-0419-129-S, -129-D
KW-210-A4	TI002-A4-B, -A4-C	11-0420-129-S, -129-D
KW-210-B1	TI002-B1-B, -B1-C	11-0421-129-S, -129-D
KW-210-B3	TI001-B3-B, -B3-C	11-0422-129-S, -129-D

Analysis Type:	AEA – Am-241
Sample Processing Prior to Radiochemical Processing/Analysis	☐ None ☒ Digested as per PNL-ALO-129, Rev. 0, *HNO_3-HCl Acid Extraction of Solids Using a Dry Block Heater, 3-7-11 (N. Green)* ☐ 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*
Total Alpha and Beta Preparation Procedure:	RPG-CMC-4001, Rev. 1, *Source Preparation For Gross Alpha and Gross Beta analyses.*
Technician/Analyst:	K. Carson, 3-28-11
Spike Standard ID's	R-569-a-3 (Sr-90), R-485-b-5 (Pu-239)
Analysis Procedure	RPG-CMC-408, Rev. 2, *Total Alpha and Beta Analysis*
Technician/Analyst:	T. Trang-Le, 3-29-11
Americium Separation Procedure:	RPG-CMC-4017, Rev. 0, *Analysis of Environmental Water Samples for Actinides and Strontium- 90*
Technician/Analyst:	K. Carson, 4-4-11
Spike and Tracer Standard ID's:	R-542-a-3 (Am-241), R-571-a-1 (Am-243 tracer)
Co-Precipitation Procedure:	RPG-CMC-496, Rev. 0, *Co-precipitation Mounting of Actinides for Alpha Spectroscopy*
Technician/Analyst:	K. Carson, 4-8-11
Analysis Procedure:	RPG-CMC-422, Rev. 2, *Solutions Analysis: Alpha Spectrometry*
Reference Date:	Same as counting dates
Analysis Date or Date Range:	4/11/11 and 4/12/11
Technician/Analyst:	T. Trang-Le
Analysis Data (File):	11-0419 Fiskum.xls, Alpha Beta
CMC Project 98620 File:	File Plan 5871: T 52578 8798: 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: 6/7/11

Reviewer: T Trang-Le / Date: 6/7/11

Sample Results

See attached data report, Am-241 Sample Results for ASR 8798. All data are reported in units of uCi/g-$_{dry}$ K-Basin sludge with a 1-σ uncertainty (see comments).

Sample Preparation, Separation, Mounting and Counting Methods

Four K-Basin sludge samples were prepared in the shielded analytical laboratory (SAL) hot cells by the Project and provided to the Analytical Support Operations (ASO) under Analytical Service Request (ASR) 8798 for chemical and radiochemical analyses. ASO received each sample to a Teflon Oak Ridge centrifuge tube, dried the contents of each centrifuge tube to a constant weight, and then leached the dried solids with hot dilute nitric and hydrochloric acids per procedure PNL-ALO-129. The leachate solution was used for radiochemical analyses and ICP metals analysis; only Am isotopic results are reported in this report. Visible residual solids remained following the PNL-ALO-129 digestion.

Following the digestion process, the Pu and U were first removed from the leachate by anion exchange chromatography using procedure RPG-CMC-4017. The Am was separated from the load and wash solution using Eichrom TRU resin per procedure RPG-CMC-4017. The separated Am fraction was then mounted for alpha spectrometry by co-precipitation using procedure RPG-CMC-496, then counted by alpha spectroscopy using procedure RPC-CMC-422. The samples were counted on April 11th and 12th, 2011; 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. These procedures are not required to be pre-approved by K-Basin project staff as they are used to generate opportunistic data only. The sum of the major alpha emitters, in this case Pu-238, Pu-239+240 and Am-241, agrees very well with the gross alpha results.

QUALITY CONTROL RESULTS

Quality control (QC) samples prepared in the SAL hot cells include a hot cell process blank (PB), sample duplicates for each sample, and a matrix spike (MS – for ICP/OES only). 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 Am-241 standard to a diluted sample.

Performance acceptance criteria for these samples are established by both the K Basin data quality objectives (as provided as special instruction in the ASR) and the ASO Quality Assurance (QA) Plan (ASO-QAP-001, Rev. 7). Except where noted, the Am-AEA K-Basin DQO and ASO QAP criteria are the same. In cases where the QC criteria are different, K-Basin DQO's take precedence. The QC sample results for Am-AEA have been evaluated and are discussed below. A summary of the Am-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 recover 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 92% to 114%.

Hot Cell Process Blank (PB) and Laboratory separations blank (LB):

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 (see comment section below). The detection limit achieved for the Am-241 is below the required detection limit of 1.0E-03 $\mu Ci/g\text{-}_{dry}$

Blank Spike (BS) – reagent spike:

The BS recovery of 102% meets the acceptance criteria of 80% to 120% recovery.

Matrix Spike (MS):

The MS recovery of 97% meets the acceptance criterion of 75% to 125% recovery. Note: the MS sample was prepared after digestion, by adding a known Am-241 standard quantity to an aliquot of the leachate. Sample number 11-0422-129-S (TI001-B3-B) was selected as the matrix spike sample.

Duplicate -- Relative Percent Difference (RPD):

Duplicate results are required to agree within <20% RPD. Duplicate results range from 2% to 18% RPD; thus meeting the <20% requirement. See Assumptions and Limitations of the Data.

Instrument Quality Control

Counter control source counting is not required when samples are spiked with tracers. When tracers are used, the absolute counting efficiencies are not used.

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 and dilution (to avoid excessive consumption of spike and avoid creating unnecessary waste). The MS prepared with this batch of samples is considered a PS, since the Am-241 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.

Attachment: Data Report -- Sample Results for ASR 8798

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

Neptunium 237 Analyses

Project / WP#:	52578/H41216
ASR#:	8798
Client:	S. Fiskum
Total # of Samples:	4

Engineered Container & Core ID	Sample IDs	RPL Number
KW-210-A1	TI001-A1-B, -A1-C	11-0419-129-S, -129-D
KW-210-A4	TI002-A4-B, -A4-C	11-0420-129-S, -129-D
KW-210-B1	TI002-B1-B, -B1-C	11-0421-129-S, -129-D
KW-210-B3	TI001-B3-B, -B3-C	11-0422-129-S, -129-D

Analysis Type:	AEA – Np-237
Sample Processing Prior to Radiochemical Processing/Analysis	☐ None ☒ Digested as per PNL-ALO-129,Rev. 0, *HNO_3-HCl Acid Extraction of Solids Using a Dry Block Heater, 3-7-11 (N. Green)* ☐ 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*
Total Alpha and Beta Preparation Procedure:	RPG-CMC-4001, Rev. 1, *Source Preparation For Gross Alpha and Gross Beta analyses.*
Technician/Analyst:	K, Carson, 3-28-11
Spike Standard ID's	R-569-a-3 (Sr-90), R-485-b-5 (Pu-239)
Analysis Procedure	RPG-CMC-408, Rev. 2, *Total Alpha and Beta Analysis*
Technician/Analyst:	T. Trang-Le, 3-29-11
Neptunium Separation Procedure:	RPG-CMC-4017, Rev. 0, *Analysis of Environmental Water Samples for Actinides and Strontium- 90*
Technician/Analyst:	K. Carson, 4-8-11
Spike Standard ID's:	R-439-c-13 (Np-237)
Co-Precipitation Procedure:	RPG-CMC-496, Rev. 0, *Co-precipitation Mounting of Actinides for Alpha Spectroscopy*
Technician/Analyst:	K. Carson, 4-14-11
Analysis Procedure:	RPG-CMC-422, Rev. 2, *Solutions Analysis: Alpha Spectrometry*
Reference Date:	Same as counting dates
Analysis Date or Date Range:	4/14/11 to 4/19/11
Technician/Analyst:	T. Trang-Le
Analysis Data (File):	11-0419 Fiskum.xls, Alpha Beta
CMC Project 98620 File:	File Plan 5871: T 52578 8798: 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: 6/7/11

Reviewer: T Trang-Le / Date: 6/7/11

Sample Results

See attached data report, Sample Results for ASR 8798. All data are reported in units of uCi/g-$_{dry}$ K-Basin sludge with a 1-σ uncertainty unless noted otherwise (see comments).

Sample Preparation, Separation, Mounting and Counting Methods

Four K-Basin sludge samples were prepared in the shielded analytical laboratory (SAL) hot cells by the Project and provided to the Analytical Support Operations (ASO) under Analytical Service Request (ASR) 8798 for chemical and radio chemical analyses. ASO received each sample in a Teflon Oak Ridge centrifuge tube, dried the contents of each centrifuge tube to a constant weight, and then leached the dried solids with hot dilute nitric and hydrochloric acids per procedure PNL-ALO-129. The leachate solution was used for radio chemical analyses and ICP metals analysis; only Np-AEA is reported in this report. Visible residual solids remained following the PNL-ALO-129.

Following the digestion process, the Np was separated from the sludge leachate using anion exchange chromatography using procedure RPG-CMC-4017. The ion exchange separation process was repeated to in order to eliminate the interference from U-234. 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. U-238 was observed at very low levels in all of the samples alpha spectra. The highest level of U-238 observed in the alpha spectra corresponds to ~9% of the Np-237 activity for the following three samples: TI001-A1-C (11-0419-129-D), TI002-B1-B (11-0421-129-S) and TI001-B3-B (11-0422-129-S).

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. These procedures are not required to be pre-approved by K-Basin project staff as they are used to generate informational data only. The sum of the major alpha emitters, in this case Pu-238, Pu-239+240 and Am-241, agree very well with the gross alpha results.

QUALITY CONTROL RESULTS

Quality control (QC) samples prepared in the SAL hot cells include a hot cell process blank (PB), sample duplicates for each sample, and a matrix spike (MS – ICP/OES only). 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.

Performance acceptance criteria for these samples are established by both the K Basin data quality objectives (as provided as special instruction in the ASR) and the ASO Quality Assurance (QA) Plan (ASO-QAP-001, Rev. 7). Except where noted, the Np-AEA K-Basin DQO and ASO QAP criteria are the same. In cases where the QC criteria are different, K-Basin DQO's will take precedence. The QC sample results for Np-AEA have been evaluated and are discussed below. A summary of the Np-AEA analysis results, including QC sample performance, is given in the attached data report.

Tracer:

Tracer is not used for analyses of Np.

Hot Cell Process 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 5% of the sample isotope concentration or less than sample minimum detectable activity (MDA) – (see comments) The PB Np-237 activity is significantly less than 5% of any sample activity. There are no acceptance criteria for LB. The detection limit achieved for the Np-237 is below the required detection limit of 1.0E-03 $\mu Ci/g_{dry}$

Blank Spike (BS) – reagent spike:

The BS recovery of 102% meets the acceptance criteria of 80% to 120% recovery.

Matrix Spike (MS):

The MS recovery of 103% 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 11-0422-129-S (TI001-B3-B) was selected as the matrix spike sample.

Duplicate -- Relative Percent Difference (RPD):

Duplicate results are required to agree within <20% RPD. Duplicate results range from 2% to 82% RPD. The RPD for sample pair TI001-A1-B (11-0419-129-S) and TI001-A1-C (11-0419-129-D) is 22% and for sample pair TI002-B1-B (11-0421-129-S) and TI002-B1-C (11-0421-129-D) is 82%. 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. The duplicate RPD's exceeding the 20% acceptance limit are documented in occurrence report 52578-05-27-11.

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.

Battelle PNNL/RPL/ASO Radiochemistry Analysis Report

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 8798.

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

Sr-90 by Liquid Scintillation Spectrometry

Project / WP#:	52578/H41216
ASR#:	8798
Client:	S. Fiskum
Total # of Samples:	4

Engineered Container & Core ID	Sample IDs	RPL Number
KW-210-A1	TI001-A1-B, -A1-C	11-0419-129-S, -129-D
KW-210-A4	TI002-A4-B, -A4-C	11-0420-129-S, -129-D
KW-210-B1	TI002-B1-B, -B1-C	11-0421-129-S, -129-D
KW-210-B3	TI001-B3-B, -B3-C	11-0422-129-S, -129-D

Analysis Type:	Sr-90
Sample Processing Prior to Radiochemical Processing/Analysis	☐ None ☒ Digested as per PNL-ALO-129, Rev. 0, *HNO_3-HCl Acid Extraction of Solids Using a Dry Block Heater, 3-7-11 (N. Green)* ☐ 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:	K. Carson, 3-28-11
Spike Standard ID's	R-569-a-3 (Sr-90), R-485-b-5 (Pu-239)
Analysis Procedure	RPG-CMC-408, Rev. 2, *Total Alpha and Beta Analysis*
Technician/Analyst:	T. Trang-Le, 3-29-11
Separation Procedure:	RPG-CMC-476, Rev. 0, *Strontium Separation Using Eichrom Strontium Resin*
Spike Standard ID:	R-569-a-3 (Sr-90)
Separation Date:	4/1/11 10:45 a.m.
Technician/Analyst:	K. Carson
Analysis Procedure:	RPG-CMC-474, Rev. 1, *Measurement of Alpha and Beta Activity by Liquid Scintillation Spectrometry*
Reference Date:	N/A
Analysis Date or Date Range:	4/7/11 (first count), 4/13/11 & 4/14/11 (second count)
Technician/Analyst:	T. Trang-Le
Rad Chem Electronic Data File:	11-0419 Fiskum.xls; Worksheet Alpha Beta
ASO Project 98620 File:	File Plan 5871: T 52578 8798: 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: 6/16/11

Reviewer / Date: T Trang-Le / 6/16/11

Battelle PNNL/RPL/ASO Radiochemistry Analysis Report

SAMPLE RESULTS

See attached data report, Sample Results for ASR 8798. All sample data are reported in $\mu Ci/g_{dry}$ K-Basin sludge with a 1-σ uncertainty (see Comments).

Sample preparation, separation, mounting, and counting

Four K-Basin sludge samples were prepared in the shielded analytical laboratory (SAL) hot cells by the Project and provided to the Analytical Support Operations (ASO) under Analytical Service Request (ASR) 8798 for chemical and radioanalytical analyses. ASO received each sample in a Teflon Oak Ridge centrifuge tube, dried the contents of each centrifuge tube to a constant weight, and then leached the dried solids with hot dilute nitric and hydrochloric acids per procedure PNL-ALO-129. This digestion process was conducted in the SAL hot cells. The leachate solution was used for radio-analytical analyses and ICP metals analysis; only Sr-90 is discussed in this report. Visible residual solids remained following the PNL-ALO-129 digestion.

Following the digestion process, the Sr-90 was separated from the leachate using Eichrom strontium resin per procedure RPG-CMC-476, then counted by liquid scintillation counting (LSC) per procedure RPG-CMC-474.

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. These procedures are not required to be pre-approved by K-Basin project staff as they are used to generate informational data only. The sum of the major beta emitters, in this case Sr/Y-90 and Cs-137, agree very well with the gross beta results.

QUALITY CONTROL RESULTS

Radioanalytical quality control (QC) samples prepared in the SAL hot cells include a hot cell process blank (PB), and sample duplicates for each sample; 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).

Performance acceptance criteria for these samples are established by both the K Basin data quality objectives (as provided as special instruction in the ASR) and the ASO Quality Assurance (QA) Plan (ASO-QAP-001, Rev. 7). Except where noted, the Sr-90 K-Basin DQO and ASO QAP criteria are the same. The quality control (QC) sample results for Sr-90 have been evaluated and are discussed below. A summary of the Sr-90 analysis results, including QC sample performance, is given in the attached data report. The calculated Sr-90 MDC (minimum detectable concentration) for all samples was less than the K-Basin method detection limit requirement of 1.0E00 $\mu Ci/g_{dry}$.

Instrument Calibration Control

Hot Cell Process Blank (PB) 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 <5% of all sample results; thus meeting the acceptance criteria. There are no acceptance criteria for LB (see Comments).

Blank Spike (BS) – reagent spike:

The BS recovery of 99% 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 TI001-B3-B (11-0422-129-S).

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 results range from 0.5% to 11% 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 (PM) and repairs are performed by the vendor under our PM 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.

Battelle PNNL/RPL/ASO Radiochemistry Analysis Report

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 "11-0419 Fiskum" has been created for this report. Supporting records such as Pipette Performance Verification forms, Laboratory Bench Record, Shielded Analytical Laboratory (SAL) 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 8798

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

filename 11-0419 Fiskum
4/21/2011

Client: S. Fiskum Project 52578 Prepared by: C Soderquist 4-21-2011
ASR 8798

Concur: T Trang-Le 4/21/2011

Procedures: PNL-ALO-129, Rev. 0 HNO_3-HCl Acid Extraction of Solids Using a Dry-Block Heater
RPG-CMC-4001, Rev 1, Source Preparation for Gross Alpha and Gross Beta Analysis
RPG-CMC-408, Rev 2, Total Alpha and Total Beta Analysis
RPG-CMC-476, Rev 0, Strontium Separation Using Eichrom Strontium Resin
RPG-CMC-474, Rev 1, Measurement of Alpha and Beta Activity by Liquid Scintillation Spectrometry
RPG-CMC-4017, Rev 0, Analysis of Environmental Water Samples for Actinides and Strontium-90
RPG-CMC-422, Rev 2, Solutions Analysis: Alpha Spectrometry
RPG-CMC-496, Rev 0, Coprecipitation Mounting of Actinides for Alpha Spectrometry

		Measured Activity, **µCi per gram of dry solids** ± 1s uncertainty						
Sample ID	Lab ID	**Gross Alpha**	**Gross Beta**	**Sr-90**	**Np-237**	**Pu-238**	**Pu-239+240**	**Am-241**
TI001-A1-B	11-0419-129-S	**4.02E+1 ± 2%**	**1.09E+3 ± 4%**	**3.44E+2 ± 2%**	**1.74E-3 ± 11%**	**2.27E+0 ± 3%**	**1.78E+1 ± 2%**	**1.82E+1 ± 2%**
TI001-A1-C	11-0419-129-D	**4.47E+1 ± 2%**	**1.05E+3 ± 4%**	**3.37E+2 ± 2%**	**2.16E-3 ± 10%**	**2.56E+0 ± 3%**	**1.92E+1 ± 2%**	**1.97E+1 ± 2%**
	RPD	11%	3%	2%	22% (1.5 s.d.)	12%	7%	8%
TI002-A4-B	11-0420-129-S	**3.72E+1 ± 2%**	**8.76E+2 ± 3%**	**2.71E+2 ± 2%**	**1.30E-3 ± 11%**	**2.30E+0 ± 3%**	**1.66E+1 ± 2%**	**1.71E+1 ± 2%**
TI002-A4-C	11-0420-129-D	**3.21E+1 ± 2%**	**9.53E+2 ± 4%**	**2.72E+2 ± 2%**	**1.19E-3 ± 11%**	**2.01E+0 ± 4%**	**1.47E+1 ± 2%**	**1.43E+1 ± 2%**
	RPD	15%	8%	0.5%	9%	13%	12%	18%
TI002-B1-B	11-0421-129-S	**3.04E+1 ± 2%**	**4.26E+2 ± 4%**	**1.39E+2 ± 2%**	**1.78E-3 ± 18%**	**1.59E+0 ± 6%**	**1.23E+1 ± 3%**	**1.51E+1 ± 2%**
TI002-B1-C	11-0421-129-D	**3.14E+1 ± 2%**	**4.61E+2 ± 4%**	**1.55E+2 ± 2%**	**7.49E-4 ± 29%**	**1.65E+0 ± 4%**	**1.26E+1 ± 2%**	**1.48E+1 ± 2%**
	RPD	3%	8%	11%	82% (2.6 s.d.)	3%	2%	2%

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

filename 11-0419 Fiskum
4/21/2011

		Measured Activity, **µCi per gram of dry solids** ± 1s uncertainty						
Sample ID	Lab ID	**Gross Alpha**	**Gross Beta**	**Sr-90**	**Np-237**	**Pu-238**	**Pu-239+240**	**Am-241**
TI001-B3-B	11-0422-129-S	**3.23E+1 ± 2%**	**3.83E+2 ± 4%**	**1.44E+2 ± 2%**	**1.14E-3 ± 19%**	**1.85E+0 ± 4%**	**1.36E+1 ± 2%**	**1.54E+1 ± 2%**
TI001-B3-C	11-0422-129-D	**3.11E+1 ± 2%**	**3.62E+2 ± 4%**	**1.34E+2 ± 2%**	**1.12E-3 ± 14%**	**1.77E+0 ± 5%**	**1.26E+1 ± 2%**	**1.47E+1 ± 2%**
	RPD	4%	6%	8%	2%	5%	7%	5%
	Hot cell acid digestion blank	< 1.E-4	< 4.E-4	< 1.E-4	< 2.E-6	< 3.E-6	< 3.E-6	5.67E-6 ± 27%
	Lab blank	< 2.E-6	< 4.E-6	< 2.E-5	< 5.E-8	< 4.E-8	< 4.E-8	3.35E-8 ± 31%
	Reagent spike	95%	100%	99%	102%	--	100%	102%
	Matrix spike	*	107%	98%	103%	--	101%	97%
	Matrix spike sample	TI001-B3-B	TI001-B3-B	TI001-B3-B	TI001-B3-B	--	TI001-B3-B	TI001-B3-B
	Requested Detection Limits	--	--	1.E+0	1.E-3	1.E-3	1.E-3	1.E-3

* spike was too small

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	WD25687	EG&G Ortec	GEM15180P	35TP11079A	Ge	15	Y

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

Uranium by Kinetic Phosphorescence Analysis (KPA)

Project / WP#:	52578/H41216
ASR#:	8798
Client:	S. Fiskum
Total Samples:	4

Engineered Container & Core ID	Sample IDs	RPL Number
KW-210-A1	TI001-A1-B, -A1-C	11-0419-129-S, -129-D
KW-210-A4	TI002-A4-B, -A4-C	11-0420-129-S, -129-D
KW-210-B1	TI002-B1-B, -B1-C	11-0421-129-S, -129-D
KW-210-B3	TI001-B3-B, -B3-C	11-0422-129-S, -129-D

Analysis Type:	U/KPA
Sample Processing Prior to Radiochemical Processing/Analysis	☐ None ☒ Digested as per PNL-ALO-129, Rev. 0, *HNO_3-HCl Acid Extraction of Solids Using a Dry Block Heater, 3-7-11 (N. Green)* ☐ Other:
Matrix Spike:	R608c – Matrix Spike, Expiration 1/1/12 R608e – Lab Reagent Spike, Expiration – 1/1/12 RS-301 – Blank Spike, Expiration 1/3/12
Analysis Procedure:	RPG-CMC-4014, Rev. 1, *Uranium by Kinetic Phosphorescence Analysis*
Analysis Date:	5/26/11
Technician/Analyst:	K. Carson
Rad Chem Electronic Data File:	11-0419 Fiskum.xls; UKPA
ASO Project 98620 File:	File Plan 5871: T 52578 8798: Sample preparation and analysis records; T3 standard certificates and preparation. Also, balance calibration and performance check records.
M&TE Number(s):	Chemcheck Instruments, Inc., Model: KPA 11, WC47898, RPL 525; KPAWIN Software, Version 1.2.8 Release 19 July 2000.

Preparer / Date: 6/17/11

Reviewer / Date: 6/17/2011

Sample Results

See attached data report, Sample Results for ASR 8798. All data are reported in units of μg U/g_{dry} K-Basin sludge with uncertainty reported as ± 1-sigma error. Calculated uncertainty includes measurement error of the instrument and dilution error.

Radiochemistry Systems file, 11-0419 Fiskum.xls, has been created for this report. Supporting records include Pipette Performance Verification forms, standard certificates, Laboratory Bench Records, and Shielded Analytical Laboratory Bench Sheet. Balance calibration records can be found in the ASO Records.

Sample Preparation and Analysis

Four K-Basin sludge samples were prepared in the shielded analytical lab (SAL) hot cells by the Project and provided to the Analytical Support Operations (ASO) under Analytical Service Request (ASR) 8798 for chemical and radio-analytical analyses. ASO received each sample in a Teflon Oak Ridge centrifuge tube, dried the contents of each centrifuge tube to a constant weight, and then leached the dried solids with hot dilute nitric and hydrochloric acids per procedure PNL-ALO-129. The leachate solution was used for radio-analytical analyses and ICP metals analysis; only U is reported in this report. Visible residual solids remained following the PNL-ALO-129 digestion.

Total uranium was measured directly in the acid digestions by kinetic phosphorescence analysis, without chemical separation. Sample digestates required an additional dilution of 10,000x to decrease sample concentrations to be within the measurable calibration range.

At the project's request, the results of all of the KPA uranium standards run concurrent with this ASR have been tabulated and attached to this report.

Quality control (QC) samples prepared include a hot cell blank, a laboratory blank, a hot cell reagent spike (BS, prepared with the samples in the hot cell), reagent (blank) spike (BS, at the instrument), a post digestion matrix spike (MS), and sample duplicates of all four samples analyzed. Minimum QC sample frequency was 2/batch.

The quality control (QC) sample results for the Uranium/KPA analysis have been evaluated and are discussed below.

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. 7); 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

Instrument Calibration Control

The U/KPA analysis has a DQO for the two calibration verification check standards analyzed at the beginning of the batch analysis. The DQO is 92.5% to 107.5% recovery of the expected value. The run was bracketed by analysis of 2 sets of the 2 calibration verification check standards. The % recovery results from the high and low pairs respectively were as follows: 103% and 102%, and 102% and 102%.

Tracer:

There is no tracer used in ASO U KPA analyses.

Hot Cell and Reagent Blank (PB):

For UKPA, the PB is required to be < EQL (estimated quantitation limit, here 0.10 µg/mL); sample results associated with the analytical run can generally be reported when the sample concentration exceeds 20 times the PB concentration. The hot cell blank was 1.99E-2 ± 7% (µg/g dry sludge) and the lab blank was <2E-5 (µg/mL). Samples were reported at a range from 4.88E+4 to 1.08E+5µg/g dry sludge exceeding the blank concentrations more than 20 times, thereby meeting the MDL requirements.

Blank Spike (BS):

The hot cell reagent spike recovery was 103% and the reagent spike prepared at the instrument was 107%, within the acceptance criteria of 80% to 120%.

Matrix Spike (MS):

The matrix spike (TI001-B3-C) recovery was 103%, within the acceptance criteria of 75-125%. Note: The MS sample was prepared "after" digestion, by adding a known U standard quantity to an aliquot of the leachate.

Duplicate Relative Percent Difference (RPD):

Duplicate results are required to agree within <20% relative percent difference. Duplicate results ranged from 6% to 13% RPD and were within the acceptance limits.

Assumption and Limitations of the Data

None

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.

Attachments : 1) Data Report Sample Results for ASR 8798

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

filename 11-0419 Fiskum
6/1/2011

Prepared by: C. Soderquist 6-8-2011

Concur: T Trang-Le 6/8/2011

Client: S. Fiskum
ASR 8798

Project 52578

Procedures: PNL-ALO-129 Rev. 0, HNO_3-HCl Acid Extraction of Solids Using a Dry-Block Heater
RPG-CMC-4014, Rev 1, Uranium by Kinetic Phosphorescence Analysis

Sample ID	Lab ID		Uranium by KPA, µg U/g $_{dry}$	± 1s
TI001-A1-B	11-0419-129-S		**9.80E+4**	**± 4%**
TI001-A1-C	11-0419-129-D		**1.08E+5**	**± 4%**
		RPD	9%	
TI002-A4-B	11-0420-129-S		**8.79E+4**	**± 4%**
TI002-A4-C	11-0420-129-D		**7.71E+4**	**± 4%**
		RPD	13%	
TI002-B1-B	11-0421-129-S		**5.55E+4**	**± 4%**
TI002-B1-C	11-0421-129-D		**4.88E+4**	**± 4%**
		RPD	13%	
TI001-B3-B	11-0422-129-S		**5.62E+4**	**± 4%**
TI001-B3-C	11-0422-129-D		**5.31E+4**	**± 4%**
		RPD	6%	
Acid digestion blank	11-0419-129-PB		1.99E-2	± 7%
		Hot cell reagent spike	103%	
		Lab reagent spike	107%	
		Lab matrix spike	103%	
		Requested Detection Limit	10 µg/g	

May 26 2011 KPA

Chemchek KPA 11-R

Sample ID	Sample Description	Sample Analysis Date	Analysis Range	Final Result	Final Result Uncert	Sample Intercept	Sample Lifetime	Sample R^2	Reference Ratio
Background	Instrument Background	5/26/2011 8:12	Low			773	382	0.945	1.000
Background	Instrument Background	5/26/2011 8:12	High			6	327	0.564	1.000
R609b #1	R609b 0.991 µg/mL #1	5/26/2011 8:14	High			77899	364	1.000	0.994
R609b #2	R609b 0.991 µg/mL #2	5/26/2011 8:17	High			78419	368	1.000	0.993
R609b #3	R609b 0.991 µg/mL #3	5/26/2011 8:21	High			78847	368	1.000	0.989
R609f #1	R609f 9.93E-3 µg/mL #1	5/26/2011 8:24	Low			81212	371	1.000	0.982
R609f #2	R609f 9.93E-3 µg/mL #2	5/26/2011 8:27	Low			79972	371	1.000	0.989
R609f #3	R609f 9.93E-3 µg/mL #3	5/26/2011 8:30	Low			80311	371	1.000	0.985
R608b #1	R608b 1.01 µg/mL cal ck #1 103%	5/26/2011 8:32	High	1.04E+00	3.14E-02	82084	365	1.000	0.992
R608f #1	R608f 1.01E-2 µg/mL cal ck #1 102%	5/26/2011 8:35	Low	1.03E-02	1.10E-04	83469	364	1.000	0.985
Blank #1	Instrument Blank	5/26/2011 8:38	Low	0.00E+00	0.00E+00	0	0	0.000	0.986
11-0419-S	11-0419-S	5/26/2011 8:40	High	1.08E-01	3.28E-03	8532	353	1.000	1.001
11-0419-D	11-0419-D	5/26/2011 8:44	High	1.35E-01	4.11E-03	10640	356	0.999	1.000
11-0420-S	11-0420-S	5/26/2011 8:47	High	1.23E-01	3.74E-03	9757	354	1.000	1.003
11-0420-D	11-0420-D	5/26/2011 8:50	High	1.21E-01	3.68E-03	9553	352	1.000	1.007
11-0421-S	11-0421-S	5/26/2011 8:53	High	4.06E-02	1.23E-03	3209	352	1.000	1.006
11-0421-D	11-0421-D	5/26/2011 8:56	High	3.92E-02	1.20E-03	3097	354	0.999	0.998
11-0422-S	11-0422-S	5/26/2011 8:59	High	5.86E-02	1.79E-03	4639	356	0.999	1.003
11-0422-D	11-0422-D	5/26/2011 9:02	High	5.48E-02	1.70E-03	4334	356	0.998	1.003
11-0419-BS	11-0419-129-BS-U-KPA	5/26/2011 9:05	High	5.22E-01	1.58E-02	41281	339	1.000	0.996
11-0419-PB	11-0419-PB	5/26/2011 9:08	Low	2.26E-04	3.21E-06	1833	329	0.997	0.988
R608b #2	R608b 1.01 µg/mL cal ck #2 102%	5/26/2011 9:11	High	1.03E+00	3.13E-02	81627	368	1.000	0.992
R608f #2	R608f 1.01E-2 µg/mL cal ck #2 102%	5/26/2011 9:13	Low	1.03E-02	1.10E-04	83525	373	1.000	0.980
Blank #2	Instrument Blank	5/26/2011 9:16	Low	0.00E+00	0.00E+00	0	0	0.000	0.985

May 26 2011 KPA

LD-0422-D	Lab Dup 11-0422-D	5/26/2011 9:19	High	5.58E-02	1.69E-03	4412	354	1.000	0.995
MS-0422-D	1 mL 11-0422-D + 1 ml R608c	5/26/2011 9:21	High	2.85E-01	8.64E-03	22527	357	1.000	0.996
RS-0419	Reagent spike 1 mL R608e direct	5/26/2011 9:26	High	5.33E-02	1.62E-03	4218	354	1.000	0.993
BLK-0419	Reagent blank 1 mL 0.5 M HNO3	5/26/2011 9:29	Low	0.00E+00	0.00E+00	0	0	0.000	0.985
R608b #3	R608b 1.01 µg/mL cal ck #3 **1o2%**	5/26/2011 9:32	High	1.03E+00	3.12E-02	81617	367	1.000	0.982
R608f #3	R608f 1.01E-2 µg/mL cal ck #3 **1o2%**	5/26/2011 9:34	Low	1.03E-02	1.10E-04	83180	373	1.000	0.977
Blank #3	Instrument Blank	5/26/2011 9:37	Low	0.00E+00	0.00E+00	0	0	0.000	0.970

5/26/11

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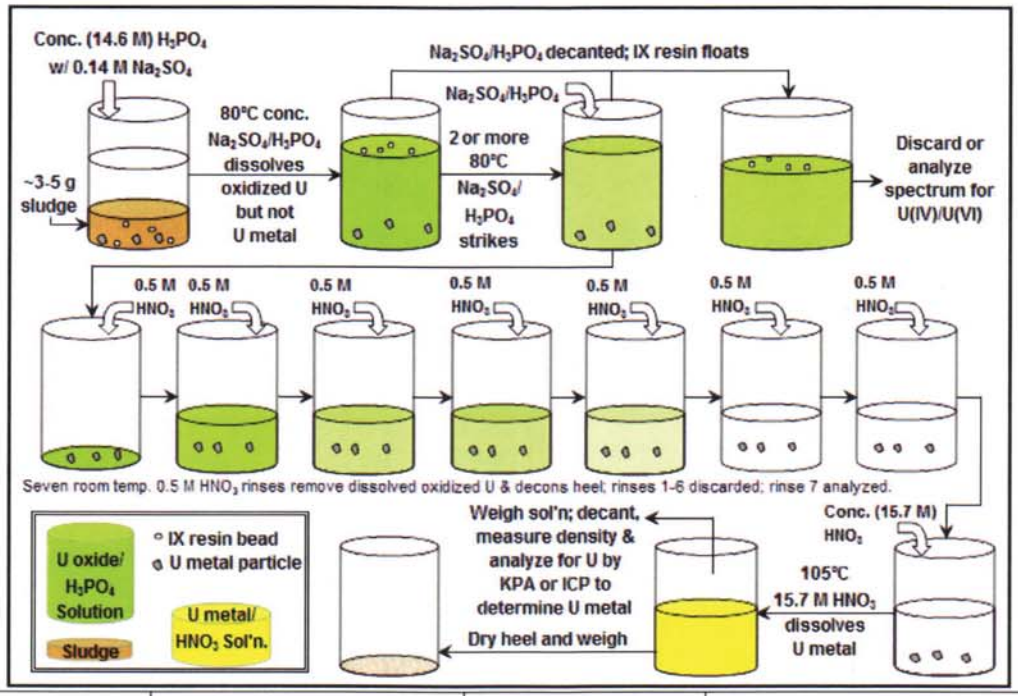

Client:	Sandy Fiskum	***ASR #:***	8798
Project #:	52578	***# Samples:***	9+1 standard+1 process blank
Charge Code:	H41220		
RPL Numbers:	11-0419-107-LCS, -B2, -S, -D; 11-0420-107-S, -D; 11-0421-107-S, -D, -MS; 11-0422-107-S, -D		

Procedure, Analysis, System, and Records Information

Analysis Procedure:	RPG-CMC-211, Rev. 3. *Determination of Elemental Composition by Inductively Coupled Argon Plasma Optical Emission Spectrometry (ICP-OES)*
Sample Prep:	RPG-CMC-107, Rev. 1, *Sample Preparation for Determination of Uranium Metal Concentrations in Sludge* PNL-ALO-501, Rev. 0, *Laboratory Procedure for Measurement of Physical and Rheological Properties of Solutions, Slurries, and Sludges*
Analyst:	Cal Delegard/Nicole Green (digestion) and Jenn Carter (ICP-OES)
Analysis Dates:	Digestion – 2 March 2011; ICP – 4 March 2011
Calibration Date/ Filename:	ICP-325-405-3 in Chemical Measurement Center 98620 file; 4 March 2011
Cal/Ver Stds Prep Date:	LCS: 25 February 2011
Sample Analysis Excel Data Files:	ICP: • C0350, 4 March 2011
M &TE Numbers	Balances 1114123894, Lab 516 and 25608154, Lab 312 (U metal beads; LCS and MS) Balance N04143 (Container Tare) Balance 40023610, SAL (solution weights); Balance 360-06-01-026 (solution density) Balance Mettler AT400 M19445 (ICP-OES) ICP-OES instrument N827583 (Lab 405)
All Analysis Records	Chemical Measurement Center 98620: RIDS U-Metal System File

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Prepared By — Date: 21 June 2011

Reviewed By — Date: 06/21/2011

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Revision 1

The report was revised to provide amended values for [U_{metal}] in Table A.1 for the Process Blank 2 sample.

Sample Results

See Attachment 1: Uranium Metal Sample Results ASR 8798.

Sample Analysis/Results Discussion

The results of uranium metal concentration analysis of nine samples of K-Basin settler tube sludge submitted under Analytical Service Request (ASR) 8798 are reported. Nine samples were delivered for analysis as wet slurries with an overburden of supernatant water. The samples were analyzed in a single batch.

The samples for uranium metal analysis were digested according to the PNNL Technical Procedure, RPG-CMC-107, Rev. 1, *Sample Preparation for Determination of Uranium Metal Concentrations in Sludge*. The contacts with 0.14 M Na_2SO_4 in concentrated H_3PO_4 are designed to dissolve and remove the accompanying uranium oxides. ASR 8798 Special Instructions specified a minimum of three contacts with 0.14 M Na_2SO_4 in concentrated H_3PO_4. The second and third contact solutions were colorless to the eye, indicating good dissolution of the contained uranium oxides.

The residues from the Na_2SO_4/H_3PO_4 contacts were rinsed sequentially seven times in 0.5 M HNO_3 and the rinsed heels, containing the undissolved uranium metal, were digested in concentrated HNO_3 to dissolve the uranium metal, all according to the digestion procedure, RPG-CMC-107, Rev. 1. For each concentrated HNO_3 digestate sample, mass and density were measured and uranium concentration in the digestate was measured by inductively coupled plasma optical emission spectrometry (ICP-OES) according to RPG-CMC-211, Rev. 3, *Determination of Elemental Composition by Inductively Coupled Argon Plasma Optical Emission Spectrometry (ICP-OES)*. The sample digestion procedure followed under RPG-CMC-107 is outlined in the Figure at the top of page 1.

Data Limitations

None.

Quality Control Discussion

The method performance is evaluated against the acceptance criteria provided in ASR 8798. Values at the Process Quantitation Limit (0.03 wt% U in sludge) or greater are considered to be quantified with an uncertainty of ±25% (at 2σ, relative) or better.

Blanks: Reagent blanks are analyzed at least once in every batch of 20 samples. Blanks of the 0.5 M HNO_3 rinse solution and the concentrated HNO_3 metal digestion solution were prepared with the sample batch; this result is in the supporting ICP-OES report. A blank containing only Hanford sand was included in the batch and was taken through all of the procedure steps. The blanks were near or below the

analytical detection limits established for the ICP-OES (0.012 µg/mL). The blank level for the 0.5 M HNO_3 reagent solution (Table A.1) was generally only a small contributor to the observed 7th rinse solution concentrations and, even when near the observed rinse solution concentrations, did not interfere with the key observations of rinse solution concentration which become important at values greater than about 100 µg/mL. Similarly, the concentrated HNO_3 blank solution concentration contributed negligibly to the 17.8 to 5,920 µg/mL digestate concentrations. Because the objective of the 7th rinse is to achieve low uranium concentrations (indicating low carryover from dissolved uranium oxides), the requirement that the prep blank concentrations be <5% of the measured concentration values in the samples does not apply to the 7th rinse concentrations.

Seventh Rinses: The uranium concentrations in the 7th 0.5 M HNO_3 rinses indicate the thoroughness of oxidized (i.e., non-metallic) uranium removal from the sludge. In developing this analytical technique (Delegard et al. 2008), uranium concentrations in the 7th rinse less than ~100 µg U/mL gave good indication that the uranium oxides were dissolved and their solutions removed from the heel.

As shown in Table A.1, all of the 7th rinse solution concentrations were less than 100 µg/mL in uranium. The rinse concentrations ranged from 0.08 to 0.81 µg U/mL and indicate complete removal of uranium oxide from the sample. The uranium concentrations in the 7th rinse were examined along with other batch quality control indicators, including duplicates, LCS results, and process blanks, in making data quality judgments.

Laboratory Control Samples (LCS): A single LCS was analyzed with the K Basin sludge samples. The LCS contained ~0.5 g of U_3O_8 (weighed to the nearest 0.001 g), ~0.6 g of Hanford sand (from Arid Lands Ecology, ALE, Reserve; weighed to the nearest 0.001 g), and ~0.011 g of uranium metal (weighed to the nearest 0.00001 g) present as a single near-spherical bead.

The LCS uranium metal recovery was 102% and was within the acceptance limits of 75-125% specified in the procedure RPG-CMC-107 and in the ASR 8798 Special Instructions.

Duplicate (Precision): A duplicate sample is required to be analyzed in every batch of 20 samples or fewer. This requirement was met.

Prior to this Project work, the MDL, 0.011 wt% U_{metal} on a settled sludge basis, was determined by evaluating uranium metal analysis recovery data (defined as PDL in Attachment J of Fiskum et al. 2009). The ASR specifies an MDL of 0.01 wt%, settled sludge basis. With the exception of one sample, all wt% results were above the ASR-specified MDL. Refer to footnote (a) of Table A.1.

The QAPjP/SAP (KBC-33786, Rev. 2) specifies that a ±20% precision criterion for duplicates is to be applied when $[U_{metal}]$ >0.1 wt%. The $[U_{metal}]$ >0.1 wt% threshold was exceeded for one KW-210 duplicate sample set. The RPD for duplicate KW-210-A4 samples was 54%. The Occurrence Report 52578-05-26-11 was generated to document the duplicate RPD values that did not meet the acceptance limits.

Matrix Spike: Based on the QAPjP/SAP (KBC-33786, Rev. 2), uranium metal measurement accuracy is to be assessed by evaluation of the LCS recoveries. However, a matrix spike (MS) sample was included to provide additional indicators of method performance. To prepare the MS sample, a uranium metal bead spike was added to one of three sludge subsamples taken from the same (B1) core. The amount of U metal added as MS was about 20-times greater than the average amount of uranium metal found present in the other two B1 core subsamples. The MS recovery, adjusted for the uranium metal quantity expected based on the average concentration found in the companion subsamples, was 99%.

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Deviations from Procedure

There were no deviations from the procedure.

General Comments

In June 2009, based on discussions between PNNL and CHPRC, it was determined that matrix spikes were to be included within each analytical batch to be processed for uranium metal and uranium oxide oxidation state distribution. While the matrix spike samples were intended to provide an additional assessment of the procedure performance, performance or failure criteria for matrix spike recoveries were not established.

References

ASO-QAP-001, Rev. 7. 2009. *Analytical Support Operations (ASO) Quality Assurance Plan*, Pacific Northwest National Laboratory, Richland, WA.

Delegard CH, SI Sinkov, AJ Schmidt, and JW Chenault. 2008. *Uranium Metal Analysis via Selective Dissolution*. PNNL-17800, Pacific Northwest National Laboratory, Richland, WA.

Fiskum, SK, JV Crum, D Neiner, CJ Thompson, JM Billing, RC Daniel, BM Oliver, T Trang-Le, SJ Bos, CH Delegard, KN Pool, MW Urie, CA Burns, MK Edwards, AJ Schmidt, CD Carlson, LR Greenwood, RW Shimskey, DS Coffey, SA Jones, and CZ Soderquist, 2009, *Characterization of K-Basin Containerized Sludge Samples Collected from Engineered Containers SCS-CON-240, 250, 260, and 220*, PNNL-19035, Pacific Northwest National Laboratory, Richland, WA.

KBC-33786, Rev. 2, *Quality Assurance Project Plan / Sampling and Analysis Plan for Sludge in the KW Engineered Containers*, CH2M Hill Plateau Remediation Company, Richland, WA. December 2009.

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Attachment 1 Uranium Metal Concentration Sample Results for ASR 8798

The analytical results for uranium metal concentration in the sludge samples are presented in Table A.1 in the dewatered sludge. Quality assurance data (i.e., blank concentrations, LCS yield, MS yield) also are presented.

Table A.1. Sample and Quality Assurance Analytical Results for Uranium Metal Concentration

ASO ID	RPL Sample ID	Sample Description / Client Core ID	Sample Wt., g	[U], µg/mL		U_{metal} added, g	U_{metal} found, g	[U_{metal}]		U_{metal} Rec., %	RPD or RSD, %
				Digest	7th rinse			µg/g [a]	wt%[a]		
11-0419-107-B3,B4	PB3, PB4	HNO_3 reagents		<0.300	<0.012						
11-0419-107-B2	Process Blank 2	Hanford sand	0.604	<0.300	0.023		<3.53E-6	*<6*	*<0.0006*		
11-0419-107-LCS	LCS	U_{metal}/U_3O_8	1.1169	941	0.405	0.01087	0.01114	9970	0.997	102	
11-0419-107-S	TI001-A1-P	KW-210-A1	3.89	278	0.362		0.00267	685	0.0685		(b)
11-0419-107-D	TI001-A1-Q	KW-210-A1	4.55	508	0.546		0.00530	1160	0.116		
11-0420-107-S	TI002-A4-P	KW-210-A4	4.80	5920	0.814		0.05730	11900	1.19		**54** RPD [c]
11-0420-107-D	TI002-A4-Q	KW-210-A4	3.99	2530	0.583		0.02730	6850	0.685		
11-0421-107-S	TI002-B1-P	KW-210-B1	4.18	17.8	0.080		0.00017	*<110*	*<0.011*		(b)
11-0421-107-D	TI002-B1-Q	KW-210-B1	4.62	79.3	0.186		0.00088	191	0.0191		
11-0422-107-S	TI001-B3-P	KW-210-B3	4.33	68.2	0.519		0.00076	176	0.0176		(b)
11-0422-107-D	TI001-B3-Q	KW-210-B3	3.93	45.0	0.335		0.00051	130	0.0130		
11-0421-107-MS	TI002-B1-R	KW-210-B1	3.97	1270	0.092	0.01053	0.01093	*102*[d]	*0.0102*[d]	99	

(a) Values in *italics* represent [U_{metal}] results that are below the process detection limit (PDL) of 0.011 wt% (110 µg/g). The process quantitation limit (PQL) is 0.03 wt% (300 µg/g). Values at the PQL or greater are considered to be quantified with an uncertainty of ±25% (at 2σ, relative) or better. See Attachment J of Fiskum et al. (2009).

(b) RPD not calculated because some or all values are below 0.1 wt% U_{metal} (1000 µg/g).

(c) RPD exceeds ±20% criterion established by the QAPjP/SAP (KBC-33786, Rev. 2).

(d) The amount of added U metal spike was deducted from the total uranium to determine wt% U_{metal} for this matrix-spiked sample. The [U_{metal}] result, even though it is below the process detection limit, is reported because it is derived by taking the difference between total uranium and the spike amount.

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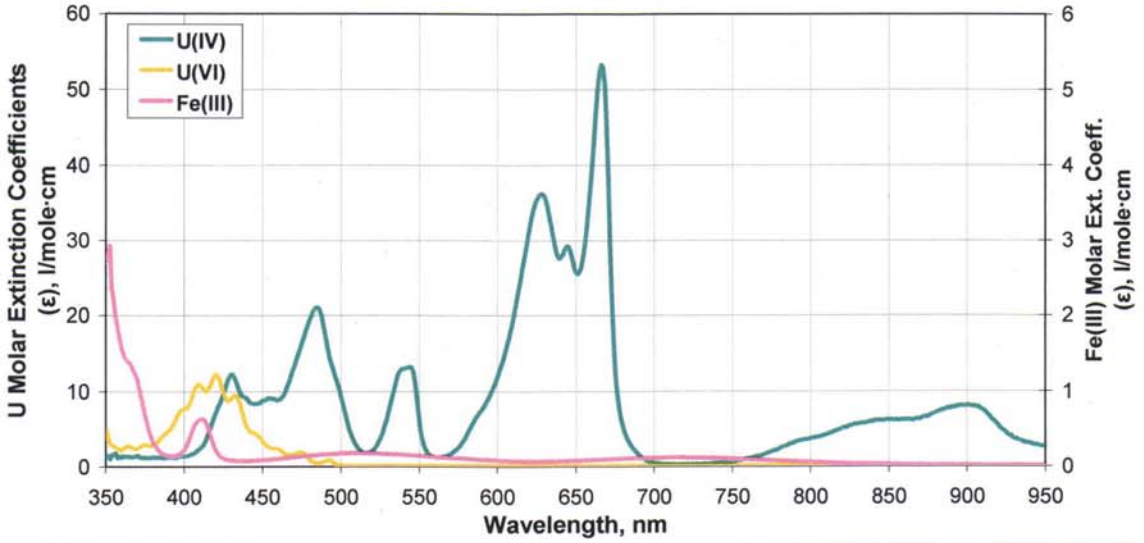

Client:	Sandy Fiskum	***ASR #:***	8798
Project #:	52578	***# Samples:***	9 samples, 1 standard, 1 blank
Charge Code:	H41221		
RPL Numbers:	11-0419-107-LCS, -B2, -S, -D; 11-0420-107-S, -D; 11-0421-107-S, -D, -MS; 11-0422-107-S, -D		

Procedure, Analysis, System, and Records Information

Analysis Procedure:	RPG-CMC-255, Rev. 0, *Sample Preparation and Analysis for Determining Uranium Oxide Oxidation States in K Basin Sludges*
Sample Prep:	RPG-CMC-107, Rev. 1, *Sample Preparation for Determination of Uranium Metal Concentrations in Sludge*
Analyst:	Sergey Sinkov
Analysis Dates:	8 March 2011
Calibration Date/ Filename:	12/2008 RPG-CMC-255 – U Oxidation State Calculation – Final.xls
Cal/Ver Stds Prep Date:	25 February 2011
Sample Analysis Excel Data Files:	030811 Spectra of H3PO4 digestates from hot cell - ASR 8798.xls RPG-CMC-255 – U Oxidation State Calculation – ASR8798.xls
M &TE Numbers	Spectral Instruments SI 400 (property number WD13494) in Lab 516
All Analysis Records	Chemical Measurement Center 98620: Fileplan U Oxide System File

Prepared By — Date: 28 APRIL 2011

Reviewed By — Date: 04/28/2011

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Sample Results

See Attachment: Uranium Oxide Oxidation States Sample Results for ASRs 8798.

Sample Analysis/Results Discussion

The results for the uranium oxidation state distribution of the uranium oxides in nine samples of K-Basin settler tube sludge submitted under Analytical Service Request (ASR) 8798 are reported. All samples were from container KW210.

The samples were prepared according to the PNNL Technical Procedure, RPG-CMC-107, Rev. 1, *Sample Preparation for Determination of Uranium Metal Concentrations in Sludge*, to dissolve and remove the accompanying uranium oxides. The digestates from the uranium oxide dissolutions were analyzed by spectrophotometry to determine the oxidation state distributions [to the U(IV) and U(VI) oxidation states] of the constituent uranium oxides according to the PNNL Technical Procedure, RPG-CMC-255, Rev. 0, *Sample Preparation and Analysis for Determining Uranium Oxide Oxidation States in K Basin Sludges*.

Data Limitations

The U(IV) and U(VI) oxidation state ratios should be used with caution. Ferrous iron, Fe(II), which is present in magnetite that has been observed in K Basin sludge, has been shown to reduce U(VI) to U(IV) in Na_2SO_4/H_3PO_4 solutions; the amount of Fe(II) is not known in these samples. The U(VI) fraction can be considered a lower bounding case and the U(IV) fraction can be considered upper bounding case.

Quality Control Discussion

The uranium oxidation state method performance is evaluated against the acceptance criteria established within procedure RPG-CMC-255, Rev. 0, *Sample Preparation and Analysis for Determining Uranium Oxide Oxidation States in K Basin Sludge*, any client specified special instructions provided in the ASR.

Blanks: The uranium oxide and many of the accompanying sludge phases were dissolved at ~80°C by three sequential contacts with 0.14 M Na_2SO_4 in concentrated H_3PO_4. In accordance with the procedure, RPG-CMC-107, Rev. 1, *Sample Preparation for Determination of Uranium Metal Concentrations in Sludge*, a process blank of 0.14 M Na_2SO_4 in concentrated H_3PO_4 was prepared per the requirement that one process blank be prepared and analyzed at least once in every batch of 20 samples. The process blank contained ~0.6 g of Hanford sand and showed <0.001 M each of U(IV) and U(VI), below the minimum concentrations detectible by the uranium oxidation state method, thus indicating the desired lack of interference by U(IV) and U(VI) contamination.

Laboratory Control Samples (LCS): A single Laboratory Control Sample (LCS) must be analyzed at least once in every batch of 20 samples. One LCS QC sample was analyzed in parallel with the K Basin sludge samples within the batch of 9 samples. The LCS QC sample contained ~0.5 g of U_3O_8 and ~0.6 g of Hanford sand (each weighed to the nearest 0.001 g) and ~0.01 g of uranium metal (weighed to the nearest 0.00001 g) present as a single near-spherical bead. The U(IV) and U(VI) uranium oxidation state distributions in the dissolved U_3O_8 were measured to determine analytical performances of the uranium oxide oxidation state distribution analyses. Because U_3O_8 is effectively one part U(IV) and two parts

U(VI) on a mole basis, the U_3O_8 dissolved in the Na_2SO_4/H_3PO_4 solution ideally yields 33.3% U(IV) and 66.7% U(VI). Per the procedure, the relative amount of U(IV) in the U_3O_8 LCS should vary by no more than 5%, absolute, from the ideal 33.3% (i.e., from 28.3 to 38.3%). The QAPjP/SAP specifies that the LCS values differ by no more than 25%, relative, from the true values and thus affords greater tolerance margins (KBC-33786, Rev. 2). The LCS for this batch contained 33.7% U(IV) and 66.3% U(VI) and thus met the target performances for both the procedure and the less stringent QAPjP/SAP.

Duplicate (Precision): A duplicate sample is required to be analyzed in every batch (of 20 samples or fewer) to allow determination of method precision. This requirement was met. Each of the four samples was measured at least in duplicate. One sample pair yielded results that could be used to estimate sample duplicate precision for U(IV), 1.7 RPD, and U(VI), 14 RPD. There was no precision Data Quality Objective specified by the Project.

Per the ASO QA Plan (Pool and Coffey 2009), the relative percent difference (RPD) is calculated in cases where both U(IV) and U(VI) concentrations are measured at or above the 0.001 M minimum detectible concentration for two samples. The ≥0.001 M U(IV) and U(VI) concentration criteria were met for one of the four samples (from core KW210-A1). The other three samples had <0.001 M U(VI). The reported RPD for the U(IV) mole%, shown in Table A.1, was 1.7% and the RPD for U(VI) was 14%. No requirement on uranium oxidation state RPD was provided in the ASR special instructions.

The QAPjP/SAP states that results should agree ±5% (absolute) if concentrations greater than 0.001 M in each oxidation state are present (KBC-33786, Rev. 2). The individual KW210-A1 uranium oxidation state values are within ±5%, absolute, of their average or 84.2 to 94.2% for U(IV) and 5.8 to 15.8% for U(VI). Therefore, this objective was met.

A single matrix spike (MS) sample was run for KW210-B1. This MS contained an added bead of uranium metal. The uranium metal spike should not alter the uranium oxide oxidation state distribution. Like the associated sample and duplicate uranium oxidation state analyses for KW210-B1, the U(VI) concentration for the MS was <0.001 M U(VI). No Data Quality Objectives are associated with the oxidation state result for the MS sample.

Deviations from Procedure

There were no deviations from the procedure RPG-CMC-255, Rev. 0, *Sample Preparation and Analysis for Determining Uranium Oxide Oxidation States in K Basin Sludges*.

References

KBC-33786, Rev. 2, *Quality Assurance Project Plan / Sampling and Analysis Plan for Sludge in the KW Engineered Containers*, CH2M Hill Plateau Remediation Company, Richland, WA. December 2009.

Pool, KN, and DS Coffey. 2009. *Analytical Support Operations (ASO) Quality Assurance Plan*, ASO-QAP-001, Rev. 7, Pacific Northwest National Laboratory, Richland, WA.

Attachment

URANIUM OXIDE OXIDATION STATES SAMPLE RESULTS FOR ASR 8798

The analytical results for uranium oxide oxidation state in the digested sludge samples are presented in Table A.1. Values of the average oxidation state also are presented with the RPD in Table A.1 for samples where the U(IV) and U(VI) concentrations were greater than the detection limit. The U(VI) concentrations for samples from KW210-A4, -B1, and -B3 were below the 0.001 M detection limit, so the mole% of oxidation species were calculated as the limiting concentration, i.e., the U(IV) percentage is greater than the value listed in Table A.1.

Table A.1. Uranium Oxide Oxidation States Sample Results ASR 8798

ASO ID	RPL Sample ID	Sample Description / Client Core ID	Concentration, M		Ox'n. State, % [a]		RPD, % [b]		Quality	
			U(IV)	U(VI)	U(IV)	U(VI)	U(IV)	U(VI)	U(IV)	U(VI)
11-0419-107-B1	Process Blank 1	Na_2SO_4/H_3PO_4	<0.001	<0.001	NA	NA	NA	NA	NA	NA
11-0419-107-B2	Process Blank 2	Hanford sand	<0.001	<0.001	NA	NA	NA	NA	NA	NA
11-0419-107-LCS	(U_{metal}/U_3O_8) - LCS	LCS	0.0169	0.0332	33.7	66.3	NA	NA	Pass [c]	Pass [c]
11-0419-107-S	TI001-A1-P	KW210-A1	0.0129	0.0017	88.4	11.6	1.70	14.0	Pass [d]	Pass [d]
11-0419-107-D	TI001-A1-Q	KW210-A1	0.0160	0.0018	90.0	10.0				
11-0420-107-S	TI002-A4-P	KW210-A4	0.0160	<0.001	>94.1	<5.9	NA	NA	NA	NA
11-0420-107-D	TI002-A4-Q	KW210-A4	0.0104	<0.001	>91.2	<8.8			NA	NA
11-0421-107-S	TI002-B1-P	KW210-B1	0.0069	<0.001	>87.4	<12.6	NA	NA	NA	NA
11-0421-107-D	TI002-B1-Q	KW210-B1	0.0076	<0.001	>88.4	<11.6			NA	NA
11-0422-107-S	TI001-B3-P	KW210-B3	0.0085	<0.001	>89.5	<10.5	NA	NA	NA	NA
11-0422-107-D	TI001-B3-Q	KW210-B3	0.0096	<0.001	>90.5	<9.5			NA	NA
11-0421-107-MS	TI002-B1-R	KW210-B1	0.0061	<0.001	>85.9	<14.1	NA	NA	NA	NA

(a) The U(IV) and U(VI) oxidation state ratios for the sludge samples should be used with caution. Ferrous iron, Fe(II), which is present in magnetite that has been observed in K Basin sludge, has been shown to reduce U(VI) to U(IV) in Na_2SO_4/H_3PO_4 solutions; the amount of Fe(II) is not known in these samples. The U(VI) fraction can be considered a lower bounding case and the U(IV) fraction can be considered upper bounding case.
(b) RPD for U(IV)% and U(VI)% in samples 11-0419-107-S and -D [100×(difference/mean)].
(c) The LCS requirement per the procedure for U(IV) is from 28.3 to 38.3%, ±5% (absolute), of the theoretical 33.3% U(IV) in U_3O_8, and 61.7 to 71.7%, ±5% (absolute), of the theoretical 66.7% U(VI) in U_3O_8. The QAPjP/SAP requirement is ±25% (relative; KBC-33786, Rev. 2). Both criteria were met.
(d) The QAPjP/SAP states that the results should agree ±5% (absolute; KBC-33786, Rev. 2) if concentrations greater than 0.001 M in each oxidation state are present. The values for KW210-A1 are within ±5%, absolute, of average and thus lie within the ranges 84.2 to 94.2% for U(IV) and 5.8 to 15.8% for U(VI). This criterion was met.

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February 15, 2011

52578-2011-L06

WW Rutherford
K Basin Sludge Treatment Project, Manager Sampling and Characterization
CH2M Hill Plateau Remediation Company
P.O. Box 1600
Richland, WA 99352

Dear Mr. Rutherford:

EVALUATION OF LOW Sb, Se, and Tl RECOVERIES IN K-BASIN MATRIX QC SAMPLES

During the inductively coupled plasma optical emission spectrometry (ICP-OES) analyses of K-Basin samples, the recovery quality control samples of antimony, selenium and thallium have not met the requirements of the QjPAP/SAP. The Analytical Services Organization (ASO) staff evaluated the issues involved and have prepared the attached document provide a summary of the issues leading to this failure per your request.

This letter will be referenced in future ASO ICP-OES reports when similar low matrix spiked analyte recoveries are found as the means to help explain the underpinning issues.

Should you have any questions please feel free to call me (376-2912) or Sandy Fiskum (376-7015).

Sincerely,

Clark Carlson
PNNL Project Manager

Enclosures

cc: WW Rutherford, MS A3-06
RB Baker, MS A3-06
STP Project File, (N. Fouad, H6-08)

bcc: CD Carlson, P7-25
SK Fiskum, P7-25
RM Shimskey, P7-27
AJ Schmidt, P8-60
KN Pool, P7-25
BM Oliver, P7-22
52578 Project File (PDF)

Evaluation of Low Sb, Se, and Tl Recoveries in K-Basin Matrix QC Samples

Inductively coupled argon plasma optical emission spectroscopy (ICP/OES) analyses of K-basin samples have demonstrated less than ideal performance in the quantification of Se, Sb and Tl at the detection limits requested by the K-basin project. Specifically, the matrix spike (MS) (and Se post spike [PS]) quality control samples run with the sample batches showed recoveries ranging from 95% to as low as 48% (summarized in Table 1). The acceptance criteria for the MS and PS are 80 to 120% recoveries. The batch laboratory control sample (LCS) recoveries (matrix-free condition) met acceptance criteria of 80 to 120%. Sample heterogeneity was considered to have factored into poor MS recoveries; however these elements in the MS samples recovered consistently <100% indicating a consistent negative bias as opposed to a random variation where some recoveries >100% would be expected. The generally consistent low MS and PS recoveries for these elements prompted a more detailed look at the method capability, instrument performance, and spectral interferences for these analytes in the K Basin sludge matrix.

Table 1. QC Summary of Selected ASRs (% Recovery)

	MS			LCS			PS		
	Sb	Se	Tl	Sb	Se	Tl	Sb	Se	Tl
ASR 8420	--	--	--	--	--	--	86	77	83
ASR 8429	79	Not reported	76	104	108	95	88	Not reported	80
ASR 8453/8487	83	81	70	86	84	80	94	95	85
ASR 8608	59	48	61	99	107	98	91	79	87
Average	74	65	69	96	100	91	90	84	84

Method Capability – The ICP/OES technique is limited in detection capability for Se, Sb and Tl due to their poor emission intensities. The current instrument detection limits (IDL) for these elements by the Analytical Support Operations (ASO) ICP/OES are 0.17ppm, 0.022ppm and 0.086ppm respectively. The IDL values are determined annually using solutions that are matrix matched to the matrices most commonly analyzed by the ASO. The method detection limit for these elements is directly affected by the processing factor associated with the sample preparation. To determine the method detection limit (MDL) for each sample, the IDL is multiplied by the processing factor associated with the sample preparation. To obtain the best possible detection capability for the K-basin samples, the digest solutions coming from the hotcell were run without additional dilution (run direct as-received) on the ICP/OES.

A more detailed evaluation was conducted on ASR 8608 samples, which demonstrated the highest variance from the QAPjP/SAP acceptance criteria. Table 2 summarizes the actual detection limits achieved for each ASR 8608 sample compared to the required detection limit requested by the project.[(a)] The sample specific MDL's were~1.1x to 10x lower than the client-requested detection limits.

(a) Larger sample size and smaller digestate volumes might be realized; however the concentration of matrix analytes (e.g., U and Fe) can cause additional interferences.

Selenium is the most challenging of the elements with MDL levels ~1.1x (TI21-MC) to 1.7x (TI021-A) of the client-required MDL, leaving little room for additional dilution. With K Basin samples containing upwards of 34 wt% Fe and U, application of a nominal process factor of 100 results in a ICP/OES analysis solution containing 3,300ppm Fe and 3,500ppm U.

Table 2. Achieved Detection Limits for Each K-Basin Sample (ASR 8608)

	Requested Detection limit	ASO Instrument Detection limit	TI021-A 10-0509-129-S (MDL) PF = 65.9	TI021-B 10-0509-129-D (MDL) PF = 80.1	TI021-MC 10-0513-129-S (MDL) PF = 109.9	TI021-MD 10-0513-129-D (MDL) PF = 104.7	TI21-SC 10-0514-129-S (MDL) PF = 107.5	TI21-SD 10-0514-129-D (MDL) PF = 94.5
	PPM	PPM	PPM	PPM	PPM	PPM	PPM	PPM
Se	20	0.17	11.2	13.6	18.7	17.8	18.3	16.1
Sb	20	0.022	1.4	1.8	2.4	2.3	2.4	2.1
Tl	20	0.086	5.7	6.9	9.5	9.0	9.2	8.1

Instrument Performance – During the analyses of the K-basin samples, numerous instrument quality control samples were run to verify the instrument performed within specification during the entire analyses process. Instrument QC samples include initial and continuing calibration verification standards, initial and continuing calibration blanks, low level standard and interference check standard (ICS). All instrument QC passed the method acceptance criteria for Se, Sb, and Tl for all the ASR's listed in Table 1. Ongoing instrument performance has also been confirmed through performance evaluation (PE) sample programs. The ASO has passed the most current PE sample for Se, Sb and Tl and historically has passed on these 4 elements.

Spectral Interferences – The ICP/OES system software captures the spectral data collected for each sample during the analyses run. The spectral data can be retrieved and evaluated by the ICP/OES scientist. The K-basin samples, where the undiluted digestates were analyzed for Se, Sb and Tl, were processed knowing the sample solution matrix did not match the matrix used for calibration and calibration verification standards. In this case, the K-basin samples contained a mix of HNO_3 and HCl diluted to a nominal concentration of 1M to 2M. The ICP/OES calibration and calibration verification solutions use 0.8M HNO_3 sample matrix. Additionally the high concentrations of Fe and U in the undiluted digestate samples typically caused these analytes to saturate the detector resulting in the instrument not using the inter-element correction (IEC) factors for Fe and U[(b)]. To mitigate the effect of not applying IECs, the ICP/OES scientist carefully evaluated the spectral lines to be used for Se, Sb and Tl in the presence of high concentrations of Fe and U. The spectral lines selected for Se, Sb and Tl provided the highest sensitivity with the lowest spectral interference potential.

(b) The instrument software does not perform the inter-element correction because the concentration of the saturating element(s) is unknown, thus the appropriate factor to apply to the other elements present in the spectra cannot be determined. The instrument flags the element as "Saturated" so the user is aware the IEC are not being applied for that element.

Critical to the proper quantification of all elements by the ICP/OES technique is determination of the back ground subtraction points used for each spectral peak. Background subtraction points are carefully selected by the ICP/OES scientist to optimize the analysis method. These points are selected using the solution matrix that is most commonly run on the instrument, 0.8M HNO_3. As noted earlier, the sample matrix analyzed for the K-basin samples was a mix of HNO_3 and HCl. The spectra showed a substantial increase in background levels in the spectral regions of the 4 target analytes. The background levels not only increased but the shape of the background baseline also changed, showing more sloping and irregular contour. The Se was especially impacted due to a large peak shoulder coming from the Fe emission peak pulling the background levels up. This change in background baseline impacted how the background subtraction corrects the spectral peaks for the region of interest used for sample quantification.

As an example of these matrix effects, the Se spectra for samples 10-0509-129-S (blue) and matrix spike sample 10-0509-129-MS (white) are shown in Fig. 1. The highlighted green data points represent the detector array pixels sampled for Se for 10-0509-129-S. The green “cross” indicates the location of the single spectral point in the method used for background subtraction. The large peak at the right side is a secondary peak from the Fe component in the sample. As is evident, the signal intensities for the five highlighted points are all lower than the background subtraction line represented by the horizontal white dashed line, which results in a negative concentration value for Se of -1.8ppm after background subtraction. No other points near the Se peak emission signal are less than the observed Se peak area intensity or less than the selected background point. Note that defining the background within the integration peak area (which would result in a calculated 0 ppm Se as opposed to a negative Se concentration) is not a defensible practice. The corresponding five pixels for 10-0509-129-MS (not highlighted) result in a background-corrected concentration of 1.9ppm. For this particular sample matrix, there is no background location within the integration window which would provide for a positive corrected concentration value for the sample.

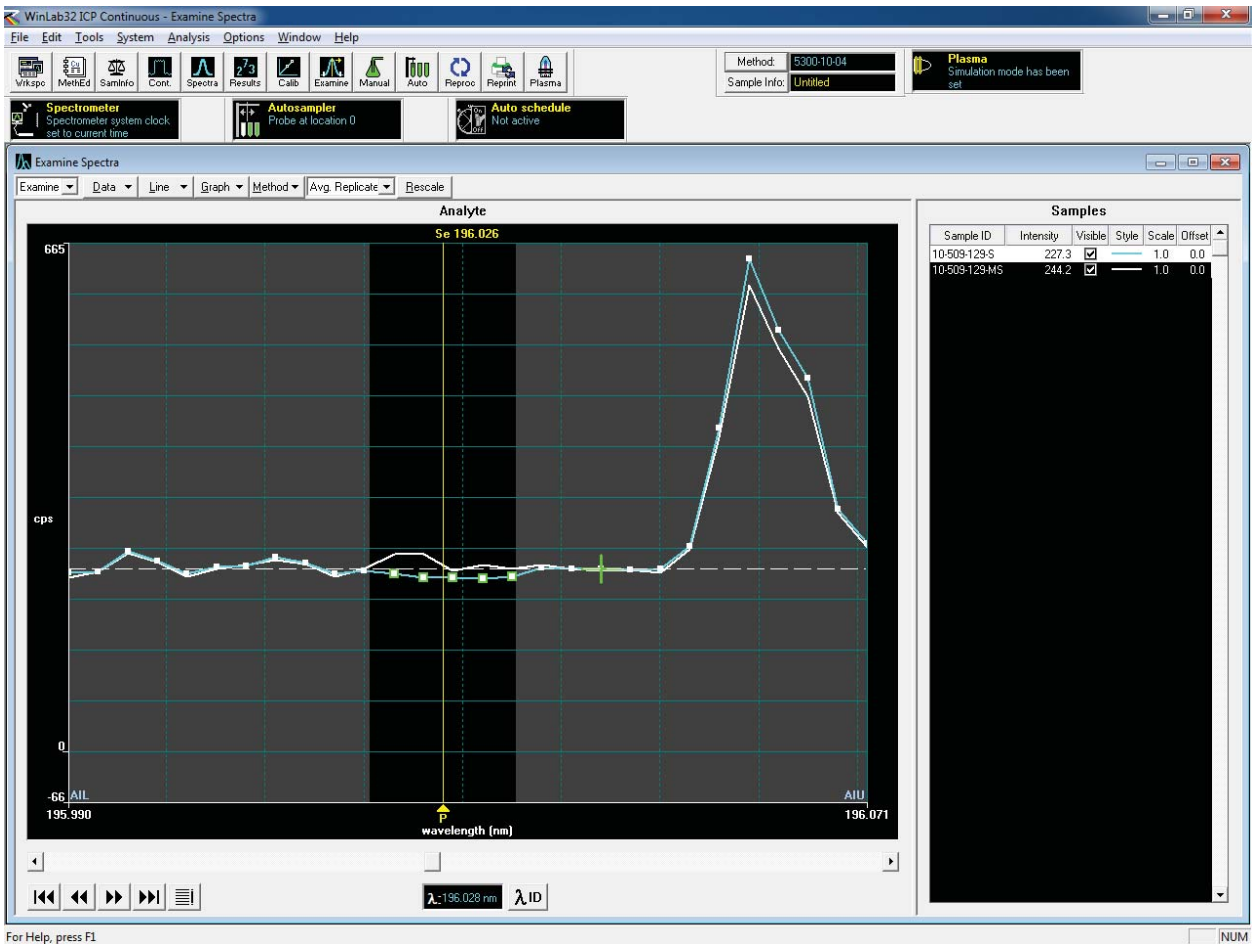

Figure 1. ICP-OES Spectra for Sample 10-0509-129-S (Blue and Green Highlight) and Matrix Spike Sample 10-0509-129-MS (White)

The matrix-specific QC samples were equally affected by the background baseline shape and the resulting over-correction from the background subtraction. The ASO does not use negative concentration results when determining sample and matrix QC concentrations. Results that are below the optimized background subtraction points indicate the concentration present is so close to the background that slight fluctuations in the integrations of the signal will cause some results to be slightly negative while others may be slightly positive. All negative concentration results are treated as 0.0ppm concentration for the specific element. Samples from ASR 8608 were investigated more thoroughly. The Se concentrations detected at the instrument (not dilution-corrected) are summarized in Table 3.

Table 3. Se Results for ASR 8608, Sample 10-0509

	Measured ppm	Reported Values and Recoveries	Alternative Recovery Calculation
10-0509-129-S	-1.8	<MDL	<MDL
10-0509-129-D	-1.1	<MDL	<MDL
10-0509-129-MS	1.9	+(1.9 – 0)/4.0 = 48%	(1.9 – -1.8)/4.0 = 93%
PS[(a)]	3.97	(3.97 – 0)/ 5.0 = 79%	(3.97 – -0.9)/5.0 = 97%
(a) The action of post-spiking resulted in 2x sample dilution.			

The negative Se concentration (measured at the instrument, not dilution corrected) calculated for the sample (10-0509-129-S) was -1.8ppm and for the duplicate (10-0509-129-D) was -1.1ppm. The actual amount of Se spike present in the matrix spike sample was 4ppm. The measured concentration of Se in the MS sample (at the instrument and not dilution corrected) was 1.9ppm. The actual sample result of -1.8ppm gets zeroed out instead of added back into the MS result. If the sample result of -1.8ppm is not zeroed out and added back to the MS results, the MS Se concentration would be 3.7ppm (93% recovery). The situation is similar for the PS sample. In this case, sample 10-0509-129-D was diluted 2× with the PS solution. The Se concentration in the PS solution was 10ppm; after 2× dilution it was 5ppm at the instrument. The measured Se concentration of the PS was 3.97ppm. The measured sample Se concentration is -1.8ppm, thus -0.90ppm in the PS solution due to the 2× dilution. Since the sample result is zeroed out, the PS result is calculated using the 3.96ppm result and does not add back in the -0.90ppm from the sample. Had the -0.90ppm been added back to the PS sample, the result would have been 4.87ppm or 97% recovery.

Similar spectral plots of Sb and Tl for sample 10-0509-129-S (blue trace with green highlight) and 10-0509-129-MS (white trace) are shown in Figs. 2 and 3, respectively. In Fig. 2, the Sb spectra are positive for both the sample and MS, and thus the location of the background point did not contribute to the MS under-recovery. In Fig. 3, the Tl value is slightly negative for the sample, but this was not a significant contributor to the MS under-recovery. Thus, relocation of the background point to provide a positive Tl signal for the sample would not have increased the MS Tl recovery to any significant extent. Thus there appear to be additional effects associated with the sample matrix that are influencing the calculated under-recovery of the Sb and Tl matrix spikes that are not well understood.

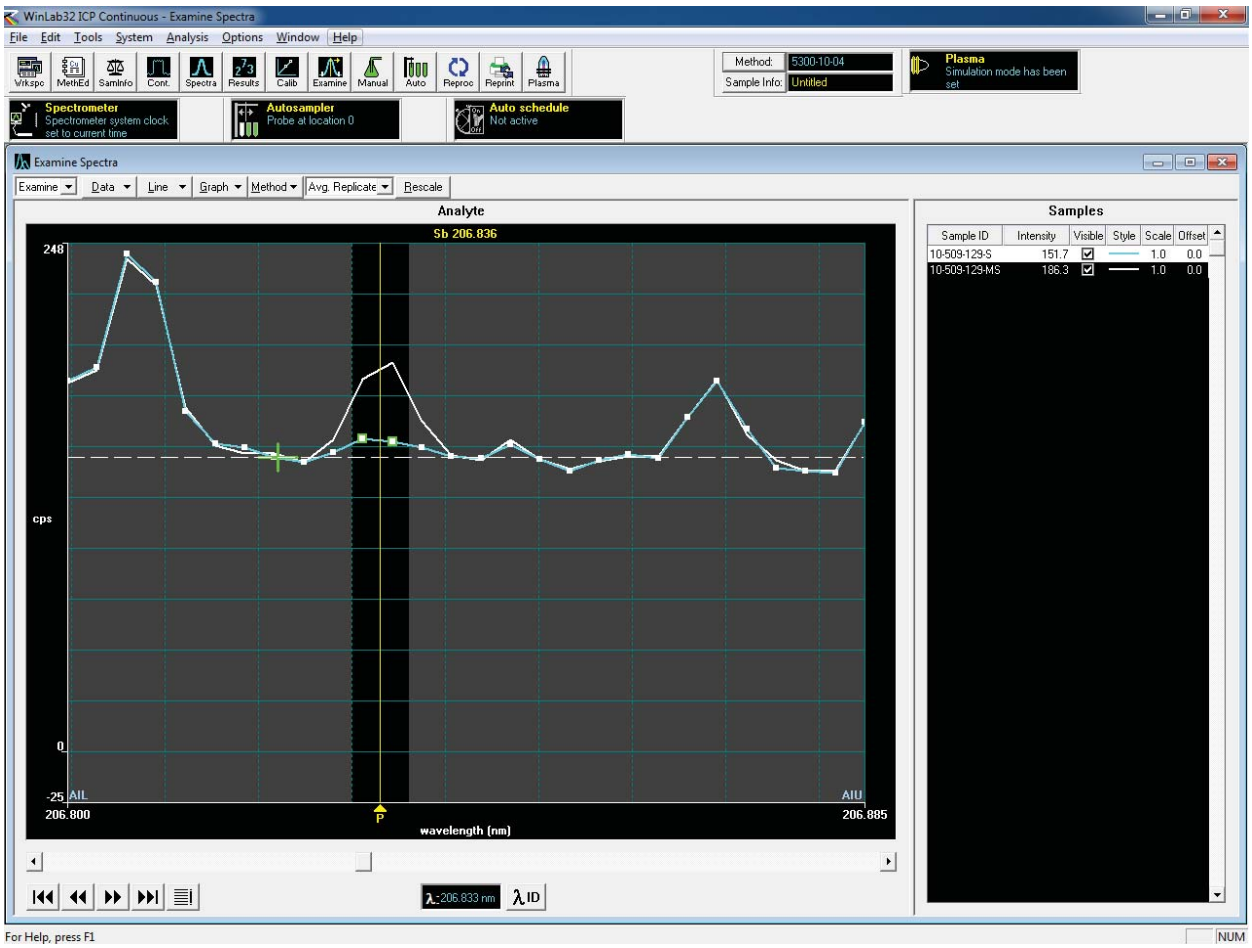

Figure 2. Sb Spectra for 10-0509-129-S (Blue and Green Highlight) and Matrix Spike Sample 10-0509-129-MS (White)

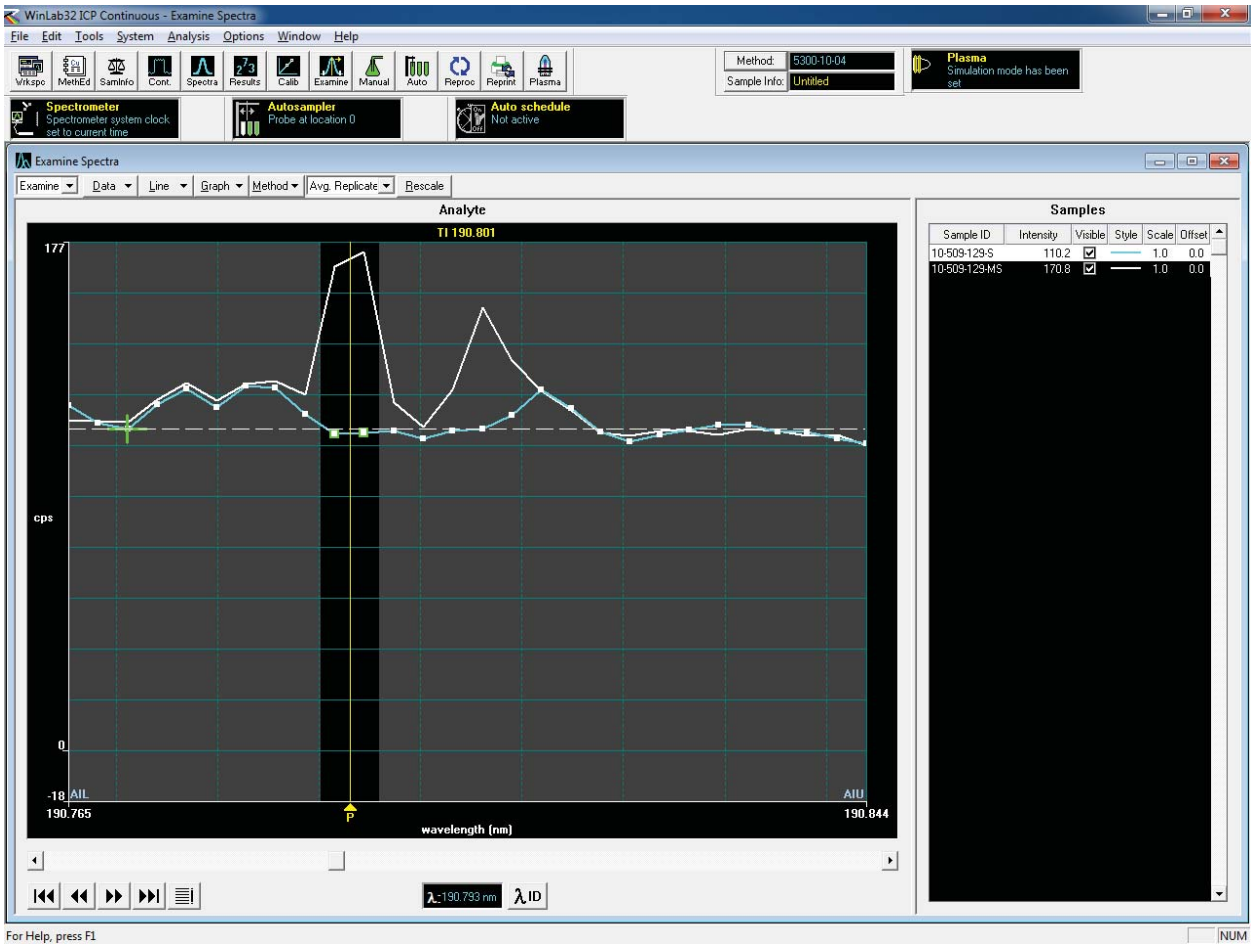

Figure 3. Tl Spectra for 10-0509-129-S (Blue and Green Highlight) and Matrix Spike Sample 10-0509-129-MS (White)

The same affects in spectral background were observed in previously analyzed K-basin samples from earlier core sample analyses. The background corrections applied to the Se, Sb and Tl for the current K-basin work are consistent with back ground corrections applied to the earlier K-basin work.

Spiking Levels – The matrix related issues are confounded by the spiking levels (i.e., low concentrations) that are optimally designed for Toxic Characteristic Leaching Procedure (TCLP) related work. Some laboratories overcome shifting background issues by using matrix QC standards at higher concentrations, thus the analyte emission signal is substantially above background and shifting background changes have minor impacts on matrix QC sample recoveries. The ASO has not taken this approach, but instead has built our matrix QC standards to be near the regulatory threshold levels for the TCLP samples.[(c)]

(c) Spiking at levels near the regulatory threshold provides more accurate representation on how the analytes of interest are behaving in the sample matrix near the regulatory limit. Over-spiking (spikes for TCLP analytes significantly above the regulatory threshold) can provide a false representation of quantification of the target analyte near the regulatory threshold.

Alternate analytical techniques that provide lower detection capability for Se, Sb and Tl (graphite furnace atomic absorption) are commonly used in the regulatory community to quantify these elements. This capability is not currently available through the PNNL ASO.

Conclusions – The limitations of method capability for these elements and the low levels present in the matrix QC standards are expected to produce similar low recoveries for the MS and PS samples associated with the K Basin samples high in Fe and U concentrations. The low Se recovery in the MS samples would suggest a persistent low bias in the measured sample concentration at or near the detection limit. Future analyses of K-Basin samples for Se, Sb and Tl will likely result in matrix QC sample spike recoveries falling near or below the 80% acceptance threshold. Future reports for these analytes may include a statement such as "Se, Sb, Tl analyte concentrations at or near the MDL are likely biased low" depending on the batch-specific MS and PS sample recovery evaluations. Reference to this letter transmittal will be included in the future reports as means for explaining low bias issues.

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March 14, 2011

59205-2011-L04

WW Rutherford
K Basin Sludge Treatment Project, Manager Sampling and Characterization
CH2M Hill Plateau Remediation Company
P.O. Box 1600
Richland, WA 99352

Dear Mr. Rutherford:

ISSUES WITH MEETING LEAD METHOD DETECTION LIMIT AND QUALITY CONTROL SAMPLE ACCEPTANCE CRITERIA

During the analytical campaigns PNNL has been performing for the Sludge Treatment Project of CHPRC, there have been several cases where we have not met various Quality Control (QC) requirements, including detection limits and spike recoveries. The QC samples which have not been missed are those related to the functioning of the instruments and the appropriateness of the methods used (i.e. preparation blanks, laboratory control samples, initial and continuing verification samples). The samples that have been missed have generally been the result of sample inhomogeneity (whether associated with sub-sampling in the hot cells or at the basins) and/or complications from the sample matrix. A point that needs to be made at this juncture is that just because a QC sample result did not meet its acceptance criteria, it does not mean there was a failure of the analysis. Two main reasons that we have observed failures in the QC sample results are associated with the 1) sample matrix complexity and the potential for inhomogeneity when the sample is subdivided into small subsamples–samples sizes are limited to reduce dose consequences to analysts and to accommodate the digestion or processing procedure sample size limitations; and 2) sample matrix inter-element effects. This letter will concentrate on the failures of meeting the required inductively coupled plasma-optical emission spectroscopy (ICP-OES) QC acceptance limits for the samples analyzed under ASR 8725, but also apply to past QC issues and may be indicated in future results. The table at the end of this letter shows the crosswalk between the results and responses by PNNL and the WIPP Table C3-9. As a part of the corrective actions for not meeting the QC criteria, this letter will be included in the final data packages for both Containers SCS-CON 230 and SCS-CON 210.

An issue that needs to be addressed when discussing QC sample analysis failure is the intended application of the digestion and analysis procedures. SW-846 and other USEPA procedures are generally intended for common matrices, such as soils and surface and ground waters. The samples from the K-

Basins are more complex containing target analytes in a U-rich environment ;[1] these matrices are not considered when the USEPA developed method QC sample acceptance criteria.

During the ICP-OES analysis performed on ASR 8725 samples, there were four kinds of QC failures observed:

1) the lead (Pb) method detection limit (MDL) was higher than the QAPjP/SAP-required MDL (20 µg/g);
2) the precision of ±20% relative percent difference (RPD, based on duplicate sample analysis) was not met for beryllium (Be) in core A3 and for calcium (Ca), chromium (Cr), nickel (Ni), and zirconium (Zr) in core B4; and
3) the accuracy (80 to 120%, matrix spike sample analyte recovery on core sample A3) was not met for arsenic (As), selenium (Se), chromium (Cr) and thallium (Tl); and
4) the accuracy (<10%, serial dilution on core sample A2) was not met for arsenic (As).

The failure to meet the MDL for lead (Pb) is directly related to the process factor (overall dilution the sample is subjected before measuring ICP-OES analytes). The dilutions required to bring the samples to the bench for handling (addressing ALARA concerns) in addition to the dilutions required to reduce the effect of interferents (uranium in this case) make the effective sample-specific MDL slightly higher than the limit required by the QAPjP/SAP of 20 µg/g, but still below the value of 100 µg/g required by WIPP in Table B3-8.[2] Efficacy of measuring the Pb concentration on less-diluted samples was evaluated. The U interference on the Pb emission line was significant and required an inter-element correction; however the U concentration in the less-diluted sample exceeded the linear range on the ICP-OES, precluding the correct application of an inter-element correction. In order to rectify this issue, two possible corrections could be made. The first would be to perform a different analysis technique, such as Atomic Absorption Spectroscopy, which we are not set up to perform and would require sending the samples off for analysis at another laboratory. The second potential correction would be to separate out the interferents, which would require significant expense and lead time for method qualification.

The precision failures of three analytes (Cr, Ni, and Zr) in the B4 core samples are indicative of the variable presence of stainless steel and/or zircaloy cladding. A slight inhomogeneity issue with stainless steel and/or zircaloy particles and their partition to the analytical subsamples would explain the differences between duplicates. The failure of the precision for Ca cannot be simply explained. Reanalysis could be performed, but the likelihood of inhomogeneity in any additional analyses would be present and may affect other analytes as well.

The accuracy failures for selenium and thallium in the MS sample are associated with the spectral background issues discussed in the memo sent to you earlier.[3] The matrix spike and serial dilution for arsenic did not meet the required limits, but both of these together indicate a matrix interference. This could be adjusted by using a higher dilution, but this will again cause a missing of the required detection

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([1]) The U emission spectrum is replete with lines; high-U concentration solutions require extensive dilution such that inter-element corrections can be applied.
([2]) WIPP document for 100 ug/g Pb dl.
([3]) 52578-2011-L06, to WW Rutherford from CD Carlson, 2/15/11.

limit. The blank spike sample analyte recoveries met all acceptance criteria, further indicating the MS sample analyte recovery failures were confounded by the sample matrix.

The crosswalk of the QC failures with the WIPP Hazardous Waste Permit Table C3-9 is as follows:

QC Sample	Results obtained by PNNL on WIPP Metals under ASR-8725	Recommend Action based on WIPP Criteria (Table 3C-9) **if criteria failed**	Actual Action taken by PNNL **(if a failure occurred)**
Method Performance Samples	Semi-Annual Performance samples meet criteria	NA	None
Laboratory blanks	Ca and U greater than detection limit	If analyte values are ≤10x blank value and ≥5X PRQL- samples must be re-digested and reanalyzed	All sample analyses for Ca and U are ≥10x blank value and ≥5X PRQL – no action needed
Matrix spikes	As, Se and Tl all have low recovery. Cr has high recovery	No action required for matrix related exceedances – only listed as a non-conformance	Se and Tl issue evaluated in PNNL letter 52578-2011-L06. As low, so result is reported as suspect. Cr high, so value is upper bounding case – no action taken but result indicated in the final verified data package
Matrix Spike duplicate	NA	No action required for matrix related exceedances – only listed as a non-conformance	NA
Initial Calibration	All calibration completed	Correct issue and repeat	NA
Continuing Calibration	CCV[1] complete and within specifications	Correct issue, recalibrate and repeat	NA
Serial Dilution	Dilution failure for As	Flag data if result is >50x IDL	Some of the As sample results are >50x IDL – failure will be indicated in the final verified data package.
Interference Correction verification	ICV[2] complete and within specification	Recalibrate and repeat analysis – if this does not correct, identify as a nonconformance	NA

Laboratory Control Samples	LCS all within specifications	Re-digest and reanalyze all samples	NA
Blind Audit Samples	NA	NA	NA

[1] Initial Calibration Verification sample
[2] Continuing Calibration Verification sample

Sincerely,

Clark Carlson
PNNL Project Manager

Enclosures

cc: WW Rutherford, MS A3-06
RB Baker, MS A3-06
JA Pottmeyer, MS A3-06
STP Project File, (FE Wickstrand, R1-29)

bcc: CD Carlson, P7-25
SK Fiskum, P7-25
RM Shimskey, P7-27
AJ Schmidt, P8-60
KN Pool, P7-25
DS Coffey, P7-25
52578 Project File (PDF)

52578-04-18-11

K-Basin Occurrence Report

Title: ASR 8798 GEA, Duplicate results exceed 20% requirement		Date: 4-18-11
Unique Identifier: 52578-04-18-11		
Primary Person Identifying Issue: Truc Trang-Le		
Distribution: Email and posting on K-Basin Share-Point	Clark Carlson	
	Deborah Coffey	Andy Schmidt
	Sandy Fiskum	Rick Shimskey
	Karl Pool	Truc Trang Le, Project file for ASR 8798
Date Submitted to K-Basin Records:	6-13-2011	

Occurrence Description: ASR#: 8798

The duplicate relative percent difference (RPD) for Co-60 on samples TI-001-A1-B and TI001-A1-C was 48%. The duplicate RPD for Eu-155 on samples TI-002-B1-B and TI002-B1-C was 32%. Both of these RPDs exceed the acceptance limit of ± 20%.

Impact of Occurrence:

There is no impact due to the RPDs exceeding the acceptance limits for these two isotopes.

The GEA results for the other 4 measured isotopes for the duplicate pair of TI-001-A1-A and TI001-A1-B ranged from 4 to 7%. This is a good indication that the majority of the target isotopes are agreeing well between sample and duplicate.

The GEA results for the other 4 measured isotopes for the duplicate pair of TI-001-B1-B and TI001-B1-C ranged from 0.4 to 16%. This is a good indication that the majority of the target isotopes are agreeing well between sample and duplicate.

The overall activity ($\mu Ci/g_{dry}$) present in each sample is dominated by Cs-137 and Am-241 (over 100x more activity than present from the Co-60 or Eu-155).

Significant Condition Adverse to Quality? Yes _____ No Y

Evaluated by: [signature] Date: 6/13/2011

Further Disposition: ☒ No ☐ Yes If yes, ☐ Nonconformance? ☐ Deficiency?

Date Occurrence Closed: 6-13-2011

Signature/Date of Responsible QE: DS Coffey 6/13/11

Signature/Date of Responsible K-Basin Manager: [signature] 6/13/2011

Comments:

52578-04-20-11

K-Basin Occurrence Report

Title: ASR 8798 – Duplicate and Matrix Spike Results Exceed Acceptance Limits for ICP/OES Analyses		Date: 4-20-11
Unique Identifier: 52578-04-20-11		
Primary Person Identifying Issue: Brian Oliver		
Distribution: Email and posting on K-Basin Share-Point	Clark Carlson	
	Deborah Coffey	Andy Schmidt
	Sandy Fiskum	Rick Shimskey
	Karl Pool	Truc Trang Le, Project file for ASR 8798
Date Submitted to K-Basin Records:	8 June 2011	

Occurrence Description: ASR#: 8798

1) Duplicate ICP/OES RPD exceed ± 20%
   a) For duplicate sample pair TI001-A1-B and TI001-A1-C, the following are a list of elements where the RPD's exceeded ± 20%: Be (37.7%), Cr (24.5%) and Ni (36.5%)
   b) For duplicate sample pair TI002-A4-B and TI002-A4-C, the following are a list of elements where the RPD's exceeded ± 20%: Be (58.5%), Cr (51.0%), Mg (25.4%), Ni (47.3%), Zn (27.3%) and (Zr 86.4%).
   c) For duplicate sample pair TI002-B1-B and TI002-B1-C, the following are a list of elements where the RPD's exceeded ± 20%: Be (144.1%), Fe (24.4%), Ni (22.0%), Zn (47.3%) and Zr (135.0%)
2) Matrix Spike recoveries exceeded the acceptance range of 80 to 120% recovery
   a) The following are the list of elements that exceeded the 80 to 120% recovery acceptance range are: (Sample TI002-B1-D was used as the matrix spike sample).
      i) Sb – 45% recovery
      ii) Tl – 30 % recovery
      iii) Zn – 74 % recovery
      iv) Zr – 244 % recovery

   NOTE: All the analytes that did not pass the matrix spike acceptance criteria did pass the post spike acceptance range of 80 to 120% recovery.

Impact of Occurrence:
There is no impact resulting from this occurrence. The variability in recovery of elements from the sample and duplicate is indicative of heterogeneity of the acid soluable component of the composited sludge. The post spike recovery indicates that dilution of the "matrix" improves the recovery of the elements.

Significant Condition Adverse to Quality? Yes _____ No X

Evaluated by: [signature] Date: 6/8/2011

Further Disposition: ☒ No ☐ Yes If yes, ☐ Nonconformance? ☐ Deficiency?

Date Occurrence Closed: 6-8-2011

Signature/Date of Responsible QE: Deborah Coffey 6-8-2011

Signature/Date of Responsible K-Basin Manager: [signature] 6/8/2011

Comments:

K-Basin Occurrence Report

Title: ASR 8798 Duplicate U-Metal Result Exceeds 20%		Date: 5-26-11
Unique Identifier: 52578-05-26-11		
Primary Person Identifying Issue: C. Delegard		
Distribution: Email and posting on K-Basin Share-Point	Clark Carlson	
	Deborah Coffey	Andy Schmidt
	Sandy Fiskum	Rick Shimskey
	Karl Pool	Truc Trang Le, Project file for ASR 8798
Date Submitted to K-Basin Records:	8 June 2011	

Occurrence Description: ASR 8798:

The duplicate RPD for sample pair TI002-A4-P (11-0420-107-S) and TI002-A4-Q (11-0420-407-D) is 54%, exceeding the acceptance criterion of ± 20%. The U-metal wt% result for sample TI002-A4-P is 1.19 wt% and for sample TI002-A4-Q is 0.685 wt%.

Impact of Occurrence:

There is no impact due to this occurrence. Variation in the U-metal content of K-Basin sludge samples is not unexpected.

Significant Condition Adverse to Quality? Yes _____ No Y

Evaluated by: [signature] Date: 6/8/2011

Further Disposition: ☒ No ☐ Yes If yes, ☐ Nonconformance? ☐ Deficiency?

Date Occurrence Closed: 8 June 2011

Signature/Date of Responsible QE: Deborah Coffey 6/8/2011

Signature/Date of Responsible K-Basin Manager: [signature] 6/8/2011

Comments:

K-Basin Occurrence Report

Title: ASR 8798 - Duplicate RPDs for Np-237 Exceed 20% Acceptance Limit.		Date: 5-27-11
Unique Identifier: 52578-05-27-11		
Primary Person Identifying Issue: K. Pool		
Distribution: Email and posting on K-Basin Share-Point	Clark Carlson	
	Deborah Coffey	Andy Schmidt
	Sandy Fiskum	Rick Shimskey
	Karl Pool	Truc Trang Le, Project file for ASR 8798
Date Submitted to K-Basin Records:	6-15-2011	

Occurrence Description: ASR 8798

1) The duplicate results for duplicate pair samples TI001-A1-B (11-0419-129-S) and TI001-A1-C (11-0419-129-D) and duplicate pair samples TI002-B1-B (11-0421-129-S) and TI002-B1-C (11-0421-129-D) exceeded the acceptance limit of 20% RPD for Np-237 (22% RPD) and (82% RPD) respectively.

Mean difference (MD) may be used to compare two radiochemical determinations that are reported with a total propagated uncertainty. The MD determines if the results are statistically different at the 95% confidence level. The MD is calculated using:

$$MD = \frac{|R_1 - R_2|}{\sqrt{a_1^2 + a_2^2}}$$

where:

- R_1 = first sample result
- R_2 = second sample result
- a_1 = two sigma total propagated uncertainty of first result
- a_2 = two sigma total propagated uncertainty of second result.

When the MD result is greater than or equal to 1.96, there is 95% confidence that the two results are not equal.

The mean difference for the duplicate pairs that did not meet the acceptance criteria are as follows:

Np-237 (TI001-A1 pair) $MD = \frac{|(1.74E-3)-(2.16E-3)|}{\sqrt{(3.83E-4)^2+(4.32E-4)^2}} = 7.27\text{E-}01$

Np-237 (TI002-B1 pair) $MD = \frac{|(1.78E-3)-(7.49E-4)|}{\sqrt{(6.23E-4)^2+(4.33E-4)^2}} = 1.36\text{E+}00$

In both cases, the MD calculation shows the results are less than 1.96, indicating the sample and duplicate results are statistically equal.

Impact of Occurrence:

There is no impact from this occurrence.

The sample and duplicate results have 1-σ counting uncertainties that range from 10 to 29%. This indicates the sample and duplicate results are close to the detection limit. The mean difference calculation shows the sample and duplicate results are statistically equal.

Significant Condition Adverse to Quality? Yes _____ No V

Evaluated by: [signature] Date: 6/15/2011

Further Disposition: ☑ No ☐ Yes If yes, ☐ Nonconformance? ☐ Deficiency?

Date Occurrence Closed: 6-15-2011

Signature/Date of Responsible QE: [signature] 6-15-2011

Signature/Date of Responsible K-Basin Manager: [signature] 6/15/2011

Comments:

K-Basin Occurrence Report

Title: Drying of Samples for TOC/TIC Analyses, ASR 8798	Date: 6-30-11
Unique Identifier: 52578-06-30-11	
Primary Person Identifying Issue: Clark Carlson	

Distribution: Email and posting on K-Basin Share-Point	Clark Carlson	Karl Pool
	Deborah Coffey	Andy Schmidt
	Sandy Fiskum	Rick Shimskey
	Matt Fountain	Truc Trang Le, Project file for ASR 8798
Date Submitted to K-Basin Records:	20 July 2011	

Occurrence Description:
QAPjP/SAP KBC-33786, Rev. 2 specified sample analysis for total organic carbon (TOC) only (refer to Table 3-4a and Figure 3-4) and indicated that the samples were to be analyzed on a wet (settled basis). However, in accordance with the ASO ASR 8798 Special Instructions reviewed and approved by the Buyer's Technical Representative (BTR), the samples were dried at 105°C before implementing ASO procedure, RPG-CMC-385, *Carbon Measured in Solids, Sludge, and Liquid Matrices* for TOC and TIC analyses.

Impact of Occurrence:
Drying of the samples to constant weight would remove any organic species (e.g. organic solvents) that have an appreciable vapor pressure at or near 105°C. However, due to the small sample size and the environment in which the samples were weighed, the loss of water during weighing operation would be a potential issue, leading to large uncertainty on the mass basis of the analysis results. Please see the attached discussion for a complete analysis of this issue.

Significant Condition Adverse to Quality? Yes _____ No X

Evaluated by: [signature] Date: 7/20/2011

Further Disposition: X No ☐ Yes If yes, ☐ Nonconformance? ☐ Deficiency?

Date Occurrence Closed: 7-20-11

Signature/Date of Responsible QE: DS Coffey 7-20-11

Signature/Date of Responsible K-Basin Manager: [signature]

Comments:

Total Carbon/Total Organic Carbon Analysis

In the QAPjP/SAP, KBC-33786, Revision 2 for Container KW-210, Table 3-4a on page 3.21, the sample preparation step for determining total organic carbon (TOC) is to collect settled sludge sub-samples for testing. Samples were actually collected from what is termed an "as-sampled" basis, where water has been removed from the sludge such that the sludge has enough strength to suspend dense particles such as U metal during the course of the subsampling activity. The as-settled sludge density is smaller than the as-sampled sludge density. A small subsample of strengthened sludge is transferred to a vial for clean transfer from the hot cell; the air headspace is substantial. During analysis, the TOC samples are dried prior to placing them in the furnace to perform the total combustion of the samples. It is assumed that the reason for requesting the analysis on the wet sample mass is to minimize the loss of the more volatile organic species and to assess the carbon concentration on an as-settled sludge mass basis. The reason the laboratory dries the samples prior to analysis will be discussed in the following paragraph.

During the initial analysis of the containerized sludges from Containers SCS-CON-220, 240, 250 and 260, the TOC was performed using the hot persulfate method, which is a wet chemical method and lends itself well to analyzing wet samples, although the samples were dried to minimize the issue of water evaporation during weighing and confounding the reporting mass basis. Because of the presence of chemical oxidation resistant OIER beads, the hot persulfate method was shown to be inadequate to get a true reading of organic carbon content and the high temperature furnace method was used on subsequent samples. The high temperature method is generally used to determine total carbon and total organic carbon. This procedure was later modified to include a two-step heating regimen to differentiate the contribution of Grafoil® from the organic carbon in the individual samples. The high temperature carbon combustion apparatus is in an open hood so there are potential issues with contamination and dose to staff. Generally, samples are heat-dried (105°C) to constant weight prior to analysis since wet samples in the high temperature furnace may spatter during initial heating, which could send some of the material out of the combustion area of the furnace, resulting in incomplete combustion of the sample and underestimation of carbon in the sample.

Many of the samples analyzed for this project have high carbon content due to the presence of resin beads and Grafoil®, so small sample sizes are required to prevent a swamping of the detector. Small samples sizes are also desirable from an ALARA perspective because the samples are relatively high dose. When small sample sizes are used, the analysis of wet samples is problematic. During the transfer of a wet sample with a relatively high surface area to mass ratio from the sample vial to the analysis boat followed by weighing, a measureable amount of water is lost to evaporation. This water loss will affect the true sample mass and any correlation to the as-settled solid mass will not be possible, giving results that cannot be tied back to a known water content.

One final consideration is that any of the more volatile organic species will have losses due to the handling of the samples during all steps from collection in the basin to processing in the HLRF. Sample temperatures were not controlled, head spaces during processing were not minimized, and holding times were not applied, therefore an accurate determination of more volatile species would not be possible for these samples.

For the reasons discussed above, all of the samples for carbon analysis were dried prior to sample analysis.

K-Basin Occurrence Report

Title: TOC/TIC method QC, Duplicate/Replicate and Analytical Spike not meeting method and project specific acceptance criteria.		**Date: 7-19-11**
Unique Identifier: 52578-07-19-11		
Primary Person Identifying Issue: D. Baldwin		
Distribution: Email and posting on K-Basin Share-Point	Clark Carlson	
	Deborah Coffey	Andy Schmidt
	Sandy Fiskum	Rick Shimskey
	Karl Pool	Truc Trang Le, Project file for **ASR 8798**
Date Submitted to K-Basin Records:	7-21-11	

Occurrence Description: ASR 8798:

The following list of items detail the issues identified with the TIC/TOC analyses:

1) Method QC related issues:

   a) 1st and 2nd Initial Calibration Blanks (ICB) are to agree within 5 µg. The first and second ICB's for run #6 (TI002-B1-H) differed by 5.6 µg.

   b) 1st and 2nd Initial Calibration Verification (ICV) standards are to agree within 5%. In 4 of the 10 TOC runs, the 1st and 2nd ICV's exceeded the 5% agreement criteria.
      i) Run #2 (TI001-A1-H) – The ICV's are 104% and 110%.
      ii) Run #4 (TI002-A4-H) – The ICV's are 100% and 107%.
      iii) Run #5 (TI002-A4-J) – The ICV's are 100% and 108%.
      iv) Run #6 (TI002-B1-H) – The ICV's are 91% and 105%.

      In 4 of the 10 TIC runs, the 1st and 2nd ICV's exceeded the 5% agreement criteria.
      i) Run #3 (TI001-A1-J) - The ICV's are 95% and 103%.
      ii) Run #5 (TI002-A4-J) – The ICV's are 92% and 100%.
      iii) Run #6 (TI002-B1-H) – The ICV's are 95% and 110%.
      iv) Run #7 (TI002-B1-J) – The ICV's are 90% and 97%.

NOTE: All ICV results are within the method acceptance limits of 90% to 110%.

2) Project specific acceptance criteria issues:

   a) The method detection limit (MDL) is required to be 200µgC/g.
   A total of 56 sample runs (TOC and TIC combined) were performed for the samples in the ASR. The 200µg/g MDL requirement was exceeded in 34 of those runs. The MDL for each sample run is mathematically derived from the sample size used and the instrument detection limit for the batch (run#). Sample sizes were intentionally kept small to limit technician exposure to radiation (ALARA). The small sample sizes along with the variation in the IDL from batch to batch caused the MDL values to exceed the 200µg/g. The method is capable of running larger sample sizes and thus able to achieve MDL values much lower than 200µg/g. In this case, due to the intentional use of small sample sizes, the MDL of 200µg/g could not be

met for all sample runs.

3) Analyses QC issues:

   a) The Analytical Spike (AS) recovery is required to be within 75% to 125%. The AS recovered at 132% (TOC) for the AS in run #2. The other AS run with this batch (run #9) recovered at 115% (TOC). The TIC recoveries are 97% for run #2 and 88% for run #9. There is currently no obvious reason for the high AS recovery for TOC in run #2.

4) Duplicate and Triplicate precision is required to be $\leq 20\%$.

   Each core sample was analyzed in triplicate, due to potential homogeneity concerns, while the special materials of Purolite® and Grafoil® were analyzed in duplicate. Precision in each case was determined based on results for all three replicates for the core samples or duplicates for the special materials.

   The following approach was used in deriving the precision for results.

   Relative Percent Differences (RPDs) can be calculated and reported when there are two J flagged values or one J flagged value and one non-flagged value.

   NOTE: The customary reporting basis for RPD's uses only results that are greater than the Estimated Quantitation Limit (EQL) (not J-flagged). For this report, it was agreed to use results that were greater than the instrument detection limit and less than the EQL in the RPD/Relative Standard Deviation (RSD) calculations.

   The following summarized the RPD/RSD issues:
   a) Run #2 (TI001-A1-H) – TOC (27%), TIC (83%)
   b) Run #3 (TI001-A1-J) – TOC (35%), TIC (81%)
   c) Run #4 (TI002-A4-H) – TOC (39%), TIC (85%)
   d) Run #5 (TI002-A4-J) – TOC (38%)
   e) Run #9 (TI001-B3-J) – TIC (38%)

Impact of Occurrence:

1) Method QC related issues:
   a) For run #6, the two initial TOC ICB results differed by 5.6 µgC and are required to be no greater than 5 µgC. The run IDL for TOC is determined by the average of the blanks run for the day. The end of day blank was between the two initial blanks. The average blank is subtracted from the sample result to correct for the background. In this case the run #6 average blank for the day is 10 µgC. The TOC triplicate sample results ranged from 1850 µgC to 9690 µgC. The small change that resulted in the two ICB differing by 5.6 µgC has negligible impact on the final TOC results.
   b) The 1st and 2nd ICV's are to agree within 5% - The cases where the initial two ICV's did not achieve agreement within 5% (TOC and/or TIC); the ICV results were all within the method acceptance range of 90% to 110%. No impact to reported results.

2) Project specific acceptance criteria :
   a) MDL is required to be 200 µgC/g of lower - The IDL for the batch is determined using the

blank data for each batch. The variation in IDL from batch to batch and the ALARA-based need to keep the sample sizes small resulted in some of the MDLs being far greater than the 200 µgC/g level. Most of the sample result was well above MDL, and those samples in which the result was near the MDL all occurred on TIC analyses. There is likely little impact.

3) Analysis QC Issues:
   a) The Analytical Spike (AS) recovery is required to be within 75% to 125%. The AS recovered at 132% (TOC) for the AS in run #2. The other AS run with this batch (run #9) recovered at 115% (TOC). The TIC recoveries are 97% for run #2 and 88% for run #9. There is currently no obvious reason for the high AS recovery for TOC in run #2. The ICV's and LCS/BS all recovered within the acceptance limits for this run.

4) Duplicate and Triplicate precision is required to be ≤ 20%.
   a) The variability observed with the duplicate and triplicate data is indicative of heterogeneity of the parent sample coupled with the small sample sizes used for the analyses. The data for the triplicate measurements provides some indication of the range of C from each subsample of the parent.

Significant Condition Adverse to Quality? Yes _____ No X

This determination will be made by Clark Carlson. This section may not apply to all systems evaluated. When used, document who made the determination of significant condition.

Review other ORs to evaluate if this is a recurring problem. Provide a short summary of what was done to determine the impact of the occurrence and the extent of the problem.

Evaluated by: [signature] **Date:** 7/20/2011

Further Disposition: ☒ **No** ☐ **Yes** **If yes,** ☐ **Nonconformance?** ☐ **Deficiency?**

This will be partially based on project managers' discussions with the client.

Date Occurrence Closed: 7-20-11

Signature/Date of Responsible QE: [signature] 7-20-11

Signature/Date of Responsible K-Basin Manager: [signature]

Comments:

Attachments may be added as needed. Identification of specific affected samples is useful.

Appendix C

ASR 8824 and Results

Appendix C

ASR 8824 and Results

Appendix C contains the signed Analytical Service Request (ASR) 8824 and special instructions generated for Analytical Support Operations (ASO) laboratory analysis of the KW210 container composite samples. The special instructions delineated sample-specific information, required detection limits, quality control (QC) sample requirements, and reporting requirements. All analytical reports generated by the ASO in response to the ASR are provided in Appendix C. Analytical reports provide sample results; associated uncertainties; and a discussion of sample processing, QC results, compliance with QC sample requirements, and data limitations. Occurrence Reports are also provided. These were prepared when issues were identified, and resolution was required while work was in process.

The following table of contents shows that pagination specific to Appendix C was applied to the ASR, the analytical reports, and Occurrence Reports. This pagination is sequential and consistent (e.g., page C.#).

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Analytical Service Request (ASR)

ASR-FY2006-ASO Rev. 0.doc

(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: S. K. Fiskum
Print Name: S K Fiskum
Phone: 375-5677 MSIN: P7-25

Project Number: 52578
Work Package: various

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: July 1, 2011
- ♦ **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: Special Instructions
- ♦ **Requested Analytical Work Completion Date:** May 20
  (Note: Priority rate charge for < 10 business day turn-around time)
- ♦ **Negotiated Commitment Date:** 5/20/11
  (To be completed by ASO Lead) KNP

Waste Designation Information

- ♦ **Sample Information Check List Attached?** ☒ No ☐ Yes
  If no, Reference Doc Attached: _____
  or, Previous ASR Number: 8798
  or, Previous RPL Number: _____

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

Send Report To: SK Fiskum MSIN P7-25
LA Snow MSIN P7-25

Additional or Special Instructions See attached

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

Date Delivered: 4/6/11
Delivered By (optional): Cen Palma / SK Fiskum
Time Delivered (optional): _____
Group ID (optional): _____
CMC Waste Sample? ☒ No ☐ Yes

Received By: _____
ASR Number: 8824 Rev.: 00
RPL Numbers: 11-0791, -0792, 0793, 0967
(first and last)

ASO Work Accepted By: KN Pool **Signature/Date:** Kenneth N. Pool 4/15/11

Analytical Services Request (ASR)

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

Provide Analytes of Interest and Required Detection limits - ☐ **Below** ☐ **Attached**

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**
11-0791	**See Attached Special Instructions for client specific ID's for the individual analyses fractions (Table 2)**	**KW-Basin Sieved Fraction Sample**	1) Direct - GEA (only)	GEA	Radchem
11-0792		**KW-Basin Sieved Fraction Samples**	1) Digest 107 (SAL) a) Uranium Oxidation State b) ICP/OES for Uranium (7th rinse and U-metal fractions) 2) Digest - 129 (SAL) a) Alpha-AEA b) GEA c) IPC/OES 3) Undissolved Solids Analyses a) GEA (from 129 digest performed in SAL)	Digest-107 UOS ICP/OES Digest-129 Alpha AEA ICP/OES	Lab Analyses ↓ Radchem Lab Analyses
11-0793				GEA	Radchem
11-0967	**See Attached Special Instructions for client specific ID's for the individual analyses fractions (Table 2)**	**KW-Basin Sludge Core Samples**	1) Digest 107 (SAL) a) Uranium Oxidtation State b) ICP/OES for Uranium (7th rinse and U-metal fractions)	Digest 107 UOS ICP	Lab Analyses Lab Analyses
	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.				

Special Instructions for K-Basin Container SCS-CON-210
Container Composite Sample Analysis (Project 52578)

Cognizant Scientist: SK Fiskum Date: 4/5/11
SK Fiskum

Project Manager: Date: 4/11/11
CD Carlson

Project Quality Engineer: DS Coffey Date: 4-5-2011
DS Coffey

ASO Lead: Date: 4/5/11
KN Pool

STP Concurrence: Date: 4/5/11
WW Rutherford

Special Instructions for K-Basin Container SCS-CON-210 Container Composite Sample Analysis

Cognizant Scientists:
SK Fiskum, 376-7015
RW Shimskey, 376-3183
LA Snow, 376-4628
CD Carlson, 376-2912

Material from the K West (KW) Basin Engineered Container SCS-CON-210 was combined into a container composite and then sieve-fractionated. Materials from +2000 µm, between 500 and 2000 µm, and -500 µm are presented for analysis. One of the core samples from this container, KW210-A4, will also be analyzed in duplicate. (This sample was analyzed per ASR 8798 and preliminary results indicated high RPDs were obtained; the re-analysis will better clarify the U metal content.)

The material has been received in the Radiochemistry Processing Laboratory (RPL) and logged into the Radioactive Material Tracking (RMT) system as RMT #37737; the Sample Information Checklist (SICL) is tied to this RMT number. The sample-specific IDs, the RPL/Analytical Support Organization (ASO) assigned sample IDs, and the first core sampling date (at the K Basin) are cross-referenced in Table 1. **Note that the 6-month hold time noted below applies to ICP-OES metals analysis on the PNL-ALO-129, Rev. 0 acid digest.**

Table 1. Core Sample ID, RPL ID, Sampling Date, and Holding Time Cross-Reference

Core Sample ID	RPL/ASO ID	Sample Date (T_0)	End of 6-Month Hold Time
KW210-LCC	11-0791	11/17/10	5/17/11
KW210-MCC	11-0792		
KW210-SCC	11-0793		
KW210-A4	11-0967	NA	NA

The sample processing and analysis schematic is shown in Figure 1. The processing, prior to the ASO-required analyses (non-highlighted blocks), is covered in a separate project-specific test instruction, 52578-TI008. The ASO-specific processing is shown in the highlighted blocks. The samples have already been aliquoted, so no further subdivision by ASO is required.

Although most work is being performed under the ASO Quality Assurance (QA) Plan, some activities shown in Figure 1are outside of the scope of the plan such as X-ray diffraction (XRD) particle density, and particle size distribution (PSD).

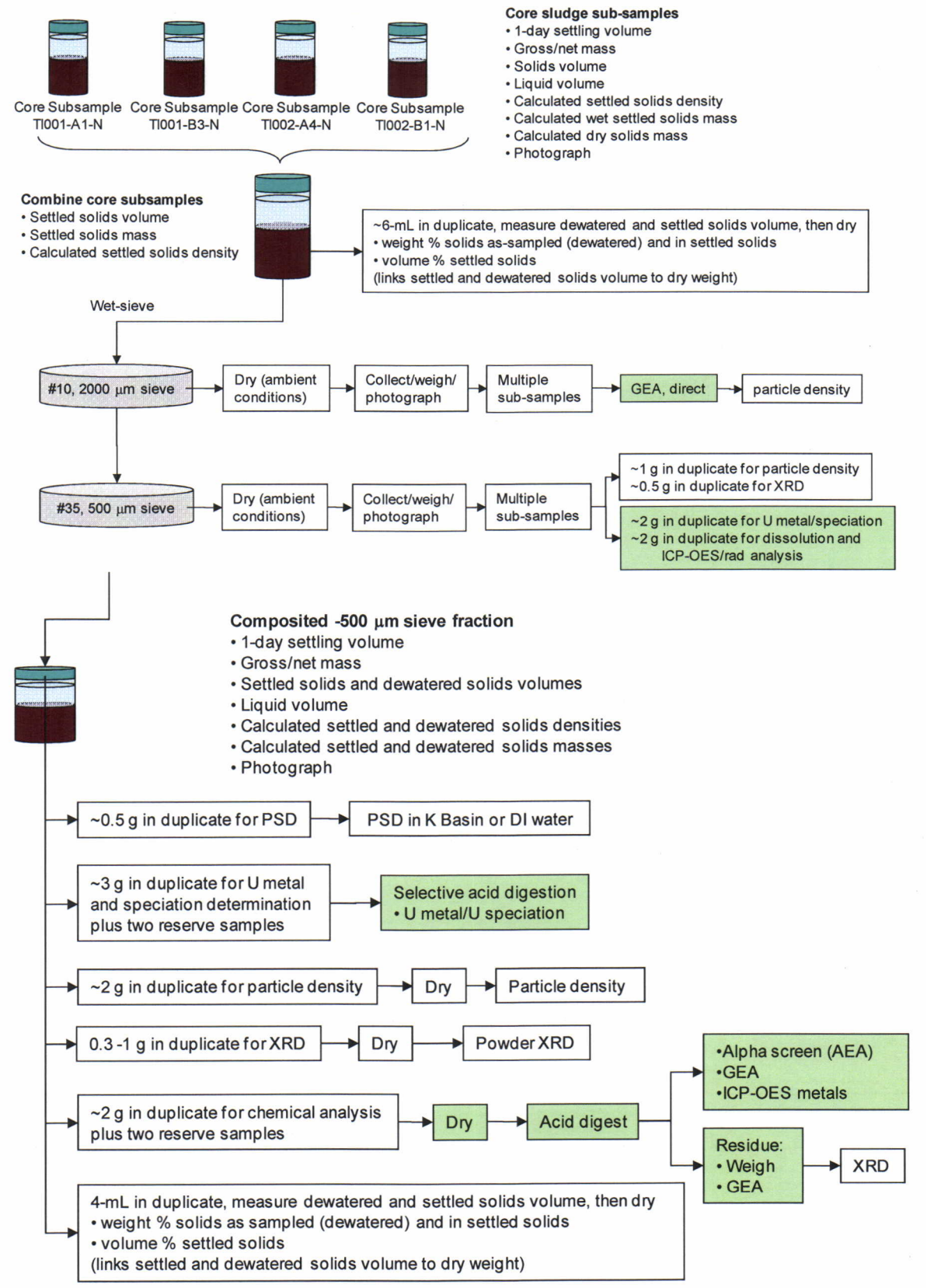


Figure 1. Analysis Schematic for KW210 Container Composite

The Chemical Measurement Center needs to charge analyses to the following work packages:

H41220	RPG-CMC-107, Rev. 1, Acid Digest
H41220	U Metal Analysis ICP-OES
H41221	U Oxide speciation
H41216	GEA
	Alpha-AEA
H41215	PNL-ALO-129, Rev. 0, Acid Digest
	ICP-OES

All sample solids masses have been calculated based on measures recorded from the Shielded Analytical Laboratory (SAL) processing activities as recorded in Test Instruction, 52578-TI008; these solids masses are reproduced in Table 2. The shaded (reserve) samples were collected as back-up samples and are not expected to be analyzed.

Table 2. Cross-Reference of Sample ID, RPL ID, Dewatered Mass, and Analysis

Sample ID	RPL ID	Oak Ridge Tube Tare, g	Dry or Dewatered Solids Mass, $g_{dewatered}$	Analysis
KW210-LCC	11-0791		**dry**	
TI008-LA	11-0791-S	NA	0.7263	Analytical (direct GEA only)
KW210-MCC	11-0792		**dry**	
TI008-MA	11-0792-129-S	NA	1.5122	Analytical
TI008-MB	11-0792-129-D	NA	1.9603	Analytical duplicate
TI008-MC	11-0792-129-MS	NA	1.3960	Analytical matrix spike/reserve
TI008-MD	11-0792-Archive	NA	1.1887	Analytical reserve
TI008-ME	11-0792-107-S	NA	2.5202	U metal/ speciation
TI008-MF	11-0792-107-D	NA	2.6176	U metal/ speciation dup
TI008-MG	11-0792-107-MS	NA	2.4524	U metal matrix spike/reserve
KW210-SCC	11-0793		**dewatered**	
TI008-SA	11-0793-129-S	32.4119	1.9371	Analytical
TI008-SB	11-0793-129-D	32.1468	2.2705	Analytical duplicate
TI008-SC	11-0793-129-MS	33.0204	1.9678	Analytical matrix spike
TI008-SD	11-0793-Archive	33.4496	2.8118	Analytical reserve
TI008-SE	11-0793-107-S	38.9594	2.1878	U metal/ speciation
TI008-SF	11-0793-107-D	38.8666	3.5278	U metal/speciation dup.
TI008-SG	11-0793-107-MS	38.8473	4.1801	U metal matrix spike
KW210-A4				
TI002-A4-R	11-0967-107-S	34.3532	4.37	U metal only
TI002-A4-S	11-0967-107-D	33.1604	4.36	U metal only, duplicate

Analytes of interest and required detection limits are provided in Table 4; quality control (QC) sample acceptance criteria are provided in Table 5. One sample per analytical batch needs to be used to prepare a matrix spike (MS) sample. Subsampling of the stock material is a laborious

process; a series of reserve samples from each core composite have been collected during the initial aliquoting process. The reserve samples should be used in case of catastrophic failure (i.e., spilling) of the primary or duplicate sample. The reserve samples were also collected in case the duplicate result did not agree with the primary result and a re-analysis might be required. The reserve samples may also be forwarded for analysis in place of the primary or duplicate samples based on the prerogative of the Cognizant Scientist (pending observations of actual mass collected, or other issues).

Uranium Metal Analysis and Uranium Oxide Speciation

U Metal/U Speciation Sample Preparation

The wet sample slurries (located in the SAL) are provided to ASO in tared Oak Ridge tubes (suitable for sample digestion). The dry samples are provided in 5-mL plastic vials (pour solids into Oak Ridge centrifuge tubes). The ASO is responsible for preparing the preparation blank (PB), the laboratory control sample (LCS), and the MS samples.

- The PB will consist of Hanford sand or equivalent and follow all processes that the samples are subjected to.
- The LCS will consist of Hanford sand, U_3O_8, (a mixture of U[IV] and U[VI]), and U metal beads.
- The MS will consist of a sample with U metal bead(s) added to it by ASO.

The samples will be processed per ASO Procedure, RPG-CMC-107, Rev. 1, *Sample Preparation for Determination of Uranium Metal Concentrations in Sludge*. At least three contacts with Na_2SO_4/H_3PO_4 shall be conducted to dissolve non-metal U species and at least 7 rinses conducted on the residual solids.

The dissolved (metal) U and seventh rinse solution U concentrations shall be determined by ICP-OES, (RPG-CMC-211, Rev. 3, *Determination of Elemental Composition by Inductively Coupled Argon Plasma Optical Emission Spectrometry [ICP-OES]*). The seventh rinse solution U concentrations should be <100 µg U/mL; the ICP-OES MDL for these samples shall be ≤1 µg U/mL. The ASO staff is directed to promply notify SK Fiskum /CD Carlson/DS Coffey if the 7th rinse is >100 µg U/mL, thus enabling PNNL to fulfill its commitment to the client for prompt notication of a potential issue with the analysis. **The duplicate precision criterion (Table 5) only applies to the dissolved U metal sample portion; it does not apply to the 7th rinse solution.**

Submit the Na_2SO_4/H_3PO_4 sample solutions containing the U(IV) and U(VI) for analysis by spectrophotometry (per RPG-CMC-255, Rev. 0, *Sample Preparation and Analysis for Determining Uranium Oxide Oxidation States in K Basin Sludges*). Note that the re-analysis of KW210-A4 samples do not require the U(IV)/U(VI) ratio analysis.

U Metal and Speciation Reporting Units

Report the U metal results as **µg U per g of dewatered sludge,** where the mass of dewatered sludge in the analysis aliquot is taken from Table 2. **Report the seventh rinse sample U concentration as µg U per mL of solution. Report the U(IV) and U(VI) oxidation states as percentages.**

Chemical Analysis Samples

Acid Digestion

Preparation

Note: Balances used to support the nuclear materials (Pu, Am, Np, and U) analysis require stringent daily balance checks on each day the balance is used . Review section on "Quality Control" before measuring and recording sample mass data. To this end, all masses collected in SAL must have the linearity check conducted and recorded.

The analytical slurry samples are provided to ASO in tared Oak Ridge tubes (suitable for sample digestion); the analytical dry solids samples are provided in 5-mL plastic vials. The wet slurry analytical samples need to be dried to constant mass at 105°C. **Determine and report the net dry sample mass.**

Acid digest the dried solids according to PNL-ALO-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. Provide a copy of the ASO preparation bench sheet to SK Fiskum and LA Snow.

Residual solids remaining after acid digestions are expected and of great 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 solution 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/LA Snow.** Additional processing of residual solids will depend on the mass relative to the starting (wet/dewatered) material. Additional processing may include:

- Remove from the SAL for pictures
- Direct gamma count
- Transfer solids outside of ASO to Doinita Neiner for XRD preparation and analysis.

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 $g_{residual}$.

ICP-OES

ICP-OES Analysis

Acid digestate aliquots (PNL-ALO-129) shall be analyzed for the Table 4 analytes of interest (Ag, Al, As, Ba, Be, Ca, Cd, Cr, Fe, Na, Ni, Mg, Pb, Sb, Se, Tl, U, V, Zn, and Zr) at the ICP-OES workstation according to RPG-CMC-211, Rev. 3, *Determination of Elemental Composition by Inductively Coupled Argon Plasma Optical Emission Spectrometry (ICP-OES)*.

ICP-OES Reporting Units

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

Radiochemistry

Preparations and Analysis

Acid digestate aliquots (PNL-ALO-129) 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 3. No procedure or procedure revision substitutions are to be made without prior written authorization; requests shall be made by the ASO Lead and submitted to SK Fiskum or CD Carlson to forward to the client for approval.

Report analytes defined in Table 4 plus any other observed analytes.

Table 3. Authorized Radiochemistry Procedures for K Basin Sludge Analysis

Analyte	Separation	Mounting	Counting/Analysis
GEA	NA	NA	RPG-CMC-450, Rev. 1 *Gamma Energy Analysis (GEA) and Low-Energy Photon Spectrometry (LEPS)*
Alpha screen /AEA	NA	RPG-CMC-496, Rev. 0, *Coprecipitation Mounting of Actinides for Alpha Spectroscopy*	RPG-CMC-422, Rev. 2 *Solutions Analysis: Alpha Spectrometry*

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.

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 5 do not apply to the undissolved solids.**

Archive of SAL-Prepared Chemical Analysis Samples

The PNL-ALO-129 acid digest preparations will result in a ~50- to 100-mL volume. This solution will be apportioned to the laboratory as needed to conduct work-station-specific analyses. At the time the acid digestates are apportioned to the analysis work locations, ~15-mL aliquot from each preparation should be collected and removed from SAL and stored as an archive sample. The vials need to be labeled with the following information: date, RPL-ID, sample ID, matrix, and hazard. The remaining portion of the acid digestion preparations may be disposed of.

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. 7) 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. Where there is a discrepancy between the data quality objectives (DQOs) in the Special Instructions and the Quality Assurance Project Plan/Sampling and Analysis Plan (QAPjP/ SAP)for Sludge in the KW Engineered Containers; KBC-33786, Rev. 2, December 2009, the QAPjP/SAP takes precedence.

NOTE: Balances used to support the nuclear materials (Pu, Am, Np, and U) analysis require more stringent daily balance checks on each day the balance is used. To maintain consistency with project-established hot cell practices, the ASO SAL-related operations must use at least 3 masses (e.g., A, B, and A+B) to provide an indication of balance accuracy and linearity. The balance control checks must be traceable in the records supporting these analyses. It is suggested that the daily control check log sheets be provided as part of the analytical report; however the ASO may choose alternate means to establish traceability. Acceptance limits for control checks for the balance in SAL cell 2 are provided in Appendix A. **If masses are inadvertently collected on a balance without the daily calibration verification check, then the ASO Lead must immediately upon discovery inform the QE, Cognizant Scientist and the Project Manager.**

The balance **calibration verification** checks at the fumehood workstations may be made per the normal process where one calibration verification check weight (mass) is measured daily when then balance is used (as per the ASO procedure, PNL-ASO-052, Rev. 1, *Balance Performance Checks*). This is allowed because the fumehood operations use volume measures or do not directly support nuclear materials accountability.

Solids matrix preparative analysis QC is to include a PB sample, sample duplicate (provided), LCS (or BS) and a sample MS (a separate sample is provided for spiking by the ASO) as indicated in Table 2 and Table 5. The BS for the AEA screen will be generated after acid digestion on a sample split, i.e., post-digestion spike (PS). The MS for alpha AEA screen will be generated after acid digestion on a sample split, i.e., PS.

The ASO is responsible for preparing the PB, LCS/BS, and spiking MS QC samples. The preparative MS and LCS/BS are to include all the analytes of interest: Ag, Al, As, Ba, Be, Ca, Cd, Cr, Fe, Na, Ni, Mg, Pb, Sb, Se, Tl, U, V, Zn, and Zr.

The duplicate, LCS/BS, and MS sample QC acceptance criteria are provided in Table 5. The following section discusses actions to take in the event of QC failures.

Note: The ASO staff must inform/report QC failures promptly to the ASO Lead who will in turn inform the Cognizant Scientist with copy to CD Carlson and the K Basin QE (Deborah Coffey), thus enabling PNNL to fulfill its commitment to the client for prompt notification of such failures. The impact of any QC 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 affected samples in the processing batch must be re-prepared and re-analyzed for the failed analytes, availability of samples permitting.

3. In the case of multi-elemental methods (ICP-OES), isolated QC failure(s) may be communicated to SK Fiskum, RW Shimskey, Clark Carlson and the K Basin QE, DS Coffey, by the ASO Lead for an assessment of the impact on data interpretation. If the data are acceptable as-is, the project staff will communicate in writing (e.g., e-mail message) that the

data may be reported; the e-mail acceptance and resulting limitations on the data from the QC sample failure(s) shall be included in the final analytical report.

4. 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. 0.2, *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. 0.2.

Data Report **Cover Page** Contents:

- Header identifying name/address of laboratory
- Client Name:
- Project #/WP #(s):
- ASR #(s):
- 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 and title (sample processing and analysis) and revision number
- M&TE (serial number or property number):
- Reference Date (when applicable):
- Client ID and 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
- Analysis dates
- Deviations from the written procedure, if any
- Method specific Sample Preparation, Separation, Mounting and Counting Methods as applicable
- Reporting Basis/Units
- QC Criteria/Results

- Limitations of the Data Interferences/Resolution Uncertainty
- Comments

Data Summary Contents:
- Client ID
- RPL ID with full ASO-related suffixes (e.g., 129-S)
- Results and result uncertainties

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 lifetime 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 4. 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 KW210 core sample analysis measured by GEA for ASR 8798.)
^{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	
$^{241}Am/^{238}Pu$	1.0E-03	Alpha screen/ AEA
$^{239+240}Pu$	1.0E-03	
	$\mu g/g_{(dry)}$	
Ag	2.0E+01	ICP-OES
Al	1.0E+02	
As	2.0E+01	
Ba	4.0E+02	
Be	2.0E+01	
Ca	2.0E+02	
Cd	4.0E+00	
Cr	2.0E+01	
Fe	1.0E+02	
Mg	2.0E+02	
Na	2.0E+02	
Ni	2.0E+01	
Pb	2.0E+02	
Sb	2.0E+01	
Se	2.0E+02	
Tl	2.0E+01	
V	2.0E+01	
Zn	2.0E+01	
Zr	2.0E+01	
U	1.0E+03	
	wt% of as-sampled sludge	
U metal	0.01	Selective dissolution and U analysis by KPA or ICP-OES
U oxide speciation	N/A	Selective dissolution and spectrophotometry
Note: The detection limits (DL) shown in this table are based on dry mass, except for the U metal analysis, whereas the DL in the QAPjP/SAP are based on settled solids (wet) mass basis. These DL values are therefore, more restrictive than those in the QAPjP/SAP.		

Table 5. Analytical Quality Control Acceptance Criteria

Analyte	Analytical Technique	LCS or BS % Recovery[a]	MS or AS % Recovery[b]	Duplicate RPD[c]
		Pre-digestion spike		
As identified in Table 4	ICP-OES	80 - 120%	80 - 120%	≤20%
U metal	Selective dissolution and ICP-OES	75 - 125%	N/A	≤20% where [U] >0.1 wt%
U oxide speciation	Selective dissolution and spectrophotometry	[e]	N/A	N/A
Analyte	**Analytical Technique**	**LCS or BS % Recovery[a]**	**Matrix Spike % Recovery[b]**	**Duplicate RPD[c]**
		Post-digestion spike		
Alpha screen (spike material is ^{239}Pu)	AEA	80 - 120%	75 - 125%	≤25%
As identified in Table 4	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. The ASO calculates RPD from the absolute value of the difference between a sample and duplicate.

(d) Per RPG-CMC-450, Rev 1, a counter control source is checked daily and must be within ±3 sigma or ±3%, whichever is greater, of the control value.

(e) The LCS U_3O_8 accepted ratios are 33.3% U(IV) and 66.6% U(VI). The acceptable U(VI) range is 62 to 72% or ±5% window. Thus the acceptable U(IV) range is 28 to 38% or ±5% window.

References

52578-TI008, Rev. 0, Preparation of K West Basin Consolidated Container Composite for SCS-CON-210

ASO-QAP-001, Rev. 7, *Analytical Support Operations Quality Assurance Plan*

PNL-ASO-052, Rev. 1, *Balance Performance Checks*

PNL-ASO-058, Rev. 0.2, *ASO Data Reporting*

Hanford Analyical Services Quality Assurance Requirments Document (HASQARD), 2007, DOE/RL-96-68, Revsion 3, Department of Energy Richland Office, Richland Washington

RPG-CMC-107, Rev. 1, *Sample Preparation for Determination of Uranium Metal Concentrations in Sludge*

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

RPG-CMC-211, Rev. 3, *Determination of Elemental Composition by Inductively Coupled Argon Plasma Optical Emission Spectrometry (ICP-OES)*

RPG-CMC-255, Rev. 0, *Sample Preparation and Analysis for Determining Uranium Oxide Oxidation States in K Basin Sludges*

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

RPG-CMC-450, Rev. 1, *Gamma Energy Analysis (GEA) and Low-Energy Photon Spectrometry (LEPS)*

RPG-CMC-496, Rev. 0, *Coprecipitation Mounting of Actinides for Alpha Spectroscopy*

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

Appendix A

SAL Balance Control Checks and Acceptance Limits

The balance control checks will document accuracy and linearity by measure of three masses for the analytical balance Sartorius MSG234 (M&TE 40023610).

Acceptance limits for the three different mass measures are provided. These acceptance limits have been reviewed and approved by Safeguards (client Terri Welsh, Mission Suppprt Alliance) in 2010. Re-evaluation of the check weights following the 2/2011 calibration on the MSG234 were conducted. The previously approved acceptance limit were slightly more restrictive than the recalculated acceptance limits from 10 measures of each weight following the February 2011 re-calibration. The more restrictive acceptance limit will be applied for conservativism.

Radiochemical Processing Laboratory
Battelle, Pacific Northwest National Laboratory

K-Basin Project **Type of Balance:** ***Sartorius MSG234***

Balance Information
Identificaiton (e.g. WD34567)
40023610/WD68444
Location (e.g. Building/Room)
RPL/201 Cell 2
Calibration Due Date
Feb-2012

Check Weight (CW) Information	#1	CW Units	#2	CW Units	#3	CW Units
CW Identification	"K-10"		"Alien"		"Megatron" + "Alien"	
CW Assigned Mass:	10.0000	g	100.0002	g	150.0004	g
Range Low Mass (+Units) &:	10.0000	g	99.9998	g	149.9995	g
Range High Mass (+Units):	10.0010	g	100.0015	g	3000.8390	g

(updated on 3/23/11. skf)

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

Mass Measurements

Project / WP#:	52578/H41215
ASR#:	8824
Client:	S. Fiskum
Total # of Samples:	5

Engineered Container & Core ID	Sample IDs	RPL Number
KW-210-MCC	TI008-MA, -MB	11-0792-129-S, -129-D
KW-210-SCC	TI008-SA, -SB, -SC	11-0793-129-S, -129-D, -129-MS

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:	RPL-OP-001, Rev. 9, *Routine Research Operations* PNL-ASO-052, Rev. 1, *Balance Performance Checks*
Analysis Date or Date Range:	4/15/11 thru 4/28/11
Technician/Analyst:	J. Chenault
Analysis Data (File):	ASR 8824 dried solids memo.doc
CMC Project 98620 File:	File Plan 5871: T 52578 8824: Sample preparation and analysis records;
M&TE Number(s):	40023610 – SAL balance Cell 2

Preparer / 5/19/11 Date

Reviewer / 20 May 2011 Date

MEMORANDUM

Pacific Northwest NATIONAL LABORATORY | Battelle The Business of Innovation

Date:	**May 18, 2011**	Project No.:	**52578**
To:	**Sandy Fiskum**		
From:	**Karl Pool**	Internal Distribution:	**File/LB**
Subject:	**Initial dried solids and residual dried solids masses for ASR 8824**		

Prior to ASO sample digestion, the previously air dried and slurry samples for ASR 8824 were dried to constant weight at 105°C in pre-tared Teflon Oak Ridge digestion tubes in the Shielded Analytical Laboratory (SAL) 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 acid volume used was proportioned to the mass of sample. Samples that had mass of 1 g or less used 8 mL of each acid, samples that had mass of 1 to 1.5 g used 12 mL of each acid and samples which had mass of 1.5 to 2.0 g used 16 mL of each acid. At the completion of the acid digestion, the samples were cooled and the residual (un-dissolved solids) were allowed to settle. The digestate with settled residual was then centrifuged for approximately 10 minutes to consolidate the solids. The supernatant solution was carefully removed using transfer pipettes. The residual solids were each rinsed 3 consecutive times using ~ 12 to 16 mL of the same reagent acids (1:1 combination of 8M HNO_3 + 8M HCl) used in the initial acid digestion. The centrifuged solids were dislodged from the bottom of the centrifuge tube using a vortex mixer after each addition of the rinse acids. The acid rinse/solids were centrifuged for approximately 10 minutes after each acid rinse addition. The remaining rinsed solids were returned to the drying oven for drying. The residual solids in the Oak Ridge tubes were dried in the drying oven for several days at 105°C to constant weight. The dry masses of the solids after digestion are presented in the Table 1.

The initial acid digestate and acid rinse solutions were collected and brought to a final volume of 100 mL in plastic volumetric flasks. The solutions in the volumetric flask were thoroughly mixed prior to sub-sampling for chemical and radiochemical analyses.

MEMORANDUM

Pacific Northwest NATIONAL LABORATORY | Battelle The Business of Innovation

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)
11-0792-129-S	**TI008-MA**	**1.3641**	**1.1750**
11-0792-129-D	**TI008-MB**	**1.7754**	**1.5186**
11-0793-129-S	**TI008-SA**	**0.8873**	**0.4087**
11-0793-129-D	**TI008-SB**	**1.0879**	**0.5009**
11-0793-129-MS	**TI008-SC**	**0.8999**	**0.3934**

E54-1900-001 (8/98)

Battelle PNNL/RPL/Inorganic Analysis ... ICP-OES Analysis Report
PO Box 999, Richland, Washington 99352

Project / WP#: 52578 / H41220
ASR#: 8824
Client: S. Fiskum
Total Samples: 4 (solids)

	First	Last
RPL#:	11-0792-S	11-0793-D
Client ID:	TI008-MA	TI008-SB
Sample Preparation: PNL-ALO-129, Rev. 0, "HNO_3-HCl Extraction of Solids for Metals Analysis Using a Dry-Block Heater", 4/20/11 (SAL/ng).		

Procedure: RPG-CMC-211, "Determination of Elemental Composition by Inductively Coupled Argon Plasma Optical Emission Spectrometry (ICP-OES)", Rev. 3.

Analyst: J. Carter

Analysis Date (File): 04-26-2011 (C0378)

See Chemical Measurement Center 98620 file: ICP-325-405-3
(Calibration and Maintenance Records)

M&TE Number: N827583 (PerkinElmer 5300DV ICP-OES, S/N 077N5122002)
WD66158 (Sartorius ME414S Balance, S/N 224066373)

5/17/11
Preparer

5/19/11
Review and Concur

Battelle PNNL/RPL/Inorganic Analysis ... ICP-OES Analysis Report

Two samples in duplicate were provided for ICP-OES analysis under Analytical Service Request (ASR) 8824. The samples and duplicates were obtained from sub-samples prepared in the High Level Radiochemistry Facility (HLRF). Each of the samples was processed in the Shielded Analytical Laboratory (SAL) following RPL procedure PNL-ALO-129 using ~1 to 2 grams of sample with dilution to a final volume of 100 mL.

Analytes of interest (AOIs) were specified in the ASR, and are listed in the upper section of the attached ICP-OES Data Report. The quality control (QC) results for these AOIs have been evaluated and are presented below. Analytes other than AOIs are reported in the bottom section of the report, but have not been fully evaluated for QC performance. The results are reported on a mass basis ($\mu g/g_{dry}$) for each detected analyte based on the original sample mass. All data have been adjusted for laboratory processing factors and instrument dilutions. There were several QC issues associated with the present analyses. These issues are discussed below and also in Occurrence Report (OR) 52578-04-29-11.

Except for As, Sb, Se, and Tl, the results are reported for 10x diluted samples. As, Sb, Se, and Tl are reported for undiluted samples (1x) in order to obtain the necessary sensitivity. Analyzing the samples at 1x, however, does introduce potential matrix issues as both the sample density and the acid content at 1x are considerably higher than the normal matrix seen by the instrument (~0.5 M nitric acid).

Minimum Method Detection Limit (MDL) values were specified in the ASR. Except for Pb in Sample 11-0793-S, MDL levels were met for all AOIs. However, the observed level of Pb exceeded the MDL requirements thereby making this issue moot.

Calibration of the ICP-OES was done following the manufacturer's recommended calibration procedure and using multi-analyte custom standard solutions traceable to the National Institute of Standards and Technology (NIST). Midrange calibration verification standards (MCVA and MCVB) were used to verify acceptance of the two-point calibration curves obtained for each analyte, and also used for continuing calibration verification.

The controlling documents were ASO-QAP-001 and ASR-8824 Special Instructions. Instrument calibrations, QC checks and blanks (e.g., ICV/ICB, CCV/CCB, LLS, ICS), blank spike, matrix spike, post spike, duplicates, and serial dilution were conducted during the analysis run. The blank spike and matrix spike were prepared using a custom solution consisting of aliquots of BPNL-QC-1A and -2B, INT-QC-TCLP-A, and CG-SB1-1.

Preparation Blank (PB):

A preparation blank (reagents only) was prepared for the extraction process. Except for Na (71.5 µg/g), the concentrations of all AOIs were within the acceptance criteria of ≤EQL (estimated quantitation level) or less than ≤5% of the concentration in the sample. Sodium exceeded the 5% requirement for Samples 11-0793-S and 11-0793-D, and is addressed in OR 52578-04-29-11.

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

A blank spike was prepared for the extraction process. Recovery values are listed for all analytes included in the spike that were measured at or above the EQL. Recovery values for the AOIs meeting this requirement ranged from 86 to 108%, and were within the acceptance criterion of 80% to 120%.

Matrix-Spiked Sample:

A matrix spike was prepared for the extraction process using Sample 11-0793. Recovery values are listed for all analytes included in the spike that were measured at or above the EQL. The recovery values for the AOIs meeting these requirements ranged from 78 to 162%, and except for Ca, Na, and Tl, were within the acceptance criterion of 80% to 120%. However, Ca, Na, and Tl were also analyzed in the post-spike (discussed below) and were all recovered within the acceptance criterion.

Duplicate Relative Percent Difference (RPD):

Duplicates were prepared for each of the two samples. RPDs are listed for all analytes that were measured at or above the EQL. RPDs for the AOIs meeting this requirement ranged from 0.7 to 143.2%, and except for Ba, Cr, Fe, Ni, Pb, and Zr for Sample 11-0792, were within the acceptance criterion of ≤20%. The high RPDs for 11-0792 are likely from sample heterogeneity and/or from the sub-sampling in the HLRF, and are addressed in OR 52578-04-29-11.

Post-Spike/Analytical Spike Sample (A Component):

A post spike (A Component) was conducted on Sample 11-0793-S. Recovery values are listed for all analytes in the spike that were measured at or above the EQL, and that had a spike concentration ≥25% of that in the sample. Recovery values for the AOIs meeting these requirements ranged from 92 to 114%, and were within the acceptance criterion of 80% to 120%.

Post Spike/Analytical Spike Sample (B Component):

A post spike (B Component) was conducted on Sample 11-0793-S. Recovery values are listed for all analytes in the spike that were measured at or above the EQL, and that had a spike concentration ≥25% of that in the sample. The recovery value for U was 102%, and was within the acceptance criterion of 80% to 120.

Serial dilution:

Five-fold serial dilution was conducted on Sample 11-0792-S. Percent differences (%Ds) are listed for all analytes that had a concentration at or above the EQL in the diluted sample. The %Ds for the AOIs meeting this requirement ranged from 0.2 to 4.7%, and were within the acceptance criterion of ≤10%.

Other QC:

Ba, Be, and Zn were slightly above the EQL for some of the Interference Check Standards (ICSs) analyzed during the run. In the case of Ba and Zn, the issue is believed to be from small impurities in the ICS solution. In the case of Be, the issue is from a small

background shift in the instrument for the ICS runs. In all three cases, these results had no statistical effect on the reported data. All other instrument-related QC tests for the AOIs passed within the appropriate acceptance criteria.

Comments:

1) The "Final Results" have been corrected for all laboratory dilutions performed on the samples during processing and analysis, unless specifically noted.
2) Instrument detection limits (IDL) and estimated quantitation limits (EQL) shown are for acidified water and/or fusion flux matrices as applicable. Detection limits for other matrices may be determined if requested. Method detection limits (MDL) can be estimated by multiplying the IDL by the "Multiplier". The estimated quantitation limit (EQL) for each concentration value can be obtained by multiplying the EQL by the "Multiplier".
3) Routine precision and bias is typically ±15% or better for samples in dilute, acidified water (e.g. 2% v/v HNO_3 or less) at analyte concentrations > EQL up to the upper calibration level. This also presumes that the total dissolved solids concentration in the sample is less than 5000 µg/mL (0.5 per cent by weight). Note that bracketed values listed in the data report are within the MDL and the EQL, and have potential uncertainties greater than 15%. Concentration values < MDL are listed as "- -". Note, that calibration and QC standards are validated to a precision of ±10%.
4) Absolute precision, bias and detection limits may be determined on each sample if required by the client. The maximum number of significant figures for all ICP measurements is two.
5) Analytes included in the spike A component (for the AS/PS) are; Ag, Al, As, B, Ba, Be, Bi, Ca, Cd, Co, Cr, Cu, Fe, K, Li, Mg, Mn, Mo, Na, Ni, P, Pb, Sb, Se, Si, Sn, Sr, Ta, Ti, Tl, V, W, Y, Zn, and Zr. Analytes included in the spike B component are; Ce, Dy, Eu, La, Nd, Pd, Rh, Ru, S, Te, Th, and U.

Battelle PNNL/RPL/Inorganic Analysis ... ICPOES Data Report

		Run Date >	4/26/2011	4/26/2011	4/26/2011	4/26/2011	4/26/2011	4/26/2011	4/26/2011
		Multiplier >	1.0	78.2	782.1	73.3	733.1	56.3	563.3
		RPL/LAB >	405 diluent	11-0792-129-PB	11-0792-129-PB @ 10x	11-0792-129-S	11-0792-129-S @ 10x	11-0792-129-D	11-0792-129-D @ 10x
Instr. Det. Limit (IDL)	Est. Quant. Limit (EQL)	Client ID >	Lab diluent	PB		TI008-MA		TI008-MB	
(µg/mL)	(µg/mL)	Analytes	(µg/mL)	(µg/g)	(µg/g)	(µg/g)	(µg/g)	(µg/g)	(µg/g)
0.0019	0.019	Ag	--		--		[7.8]		--
0.0046	0.046	Al	--		--		17,200		15,300
0.0960	0.960	As	--	--		[35]		[31]	
0.0001	0.002	Ba	[0.0001]		[0.097]		141		316
0.0001	0.001	Be	--		--		6.44		6.62
0.0022	0.044	Ca	--		--		9,220		9,800
0.0015	0.015	Cd	--		--		36.6		38.1
0.0020	0.020	Cr	--		--		137		209
0.0045	0.045	Fe	--		[3.8]		37,100		49,500
0.0006	0.012	Mg	[0.0008]		--		675		808
0.0036	0.072	Na	[0.015]		71.5		1,450		1,570
0.0026	0.026	Ni	--		--		99.2		149
0.0210	0.210	Pb	--		--		175		260
0.0220	0.220	Sb	--	--		[5.8]		[4.1]	
0.1700	1.700	Se	--	--		--		--	
0.0860	0.860	Tl	--	--		--		--	
0.0120	0.120	U	--		--		30,300		25,000
0.0011	0.022	V	--		--		[14]		16.6
0.0056	0.056	Zn	--		[6.7]		250		240
0.0008	0.008	Zr	--		--		573		94.9
Other Analytes		Other Analytes							
0.0057	0.057	B	[0.027]		[22]		[24]		[17]
0.0160	0.160	Bi	--		[59]		[63]		[43]
0.0087	0.087	Ce	--		--		[25]		[17]
0.0025	0.025	Co	--		--		[4.2]		[3.3]
0.0016	0.016	Cu	--		--		423		403
0.0012	0.012	Dy	--		--		--		--
0.0003	0.003	Eu	--		--		[0.25]		[0.58]
0.0220	0.220	K	--		[120]		461		479
0.0012	0.012	La	--		--		13.0		13.5
0.0005	0.005	Li	--		5.39		5.83		5.25
0.0002	0.002	Mn	--		--		250		305
0.0070	0.070	Mo	--		--		[20]		[17]
0.0027	0.027	Nd	[0.0028]		--		24.6		[13]
0.0510	0.510	P	--		--		[120]		[150]
0.0048	0.048	Pd	--		--		[4.6]		[3.1]
0.0088	0.088	Rh	--		[7.8]		--		--
0.0044	0.044	Ru	--		--		--		--
0.1600	1.600	S	--		--		9,130		9,230
0.0130	0.130	Si	--		[13]		583		506
0.0140	0.140	Sn	--		--		[22]		[29]
0.0000	0.000	Sr	[0.0000]		[0.025]		51.3		54.9
0.0190	0.190	Ta	--		--		--		--
0.0160	0.160	Te	--		--		--		[13]
0.0050	0.050	Th	--		--		[34]		[26]
0.0005	0.005	Ti	--		--		221		394
0.0150	0.150	W	--		--		--		--
0.0003	0.005	Y	--		--		[2.4]		2.97

1) "--" indicates the value is Shaded results are outside the acceptance criteria.
near the top of each column. The estimated sample quantitation limit = EQL (in Column 2)
times the "multiplier". Overall error for values ≥ EQL is estimated to be within ±15%.
2) Values in brackets [ ] are ≥ MDL but < EQL, with errors likely to exceed 15%.

Battelle PNNL/RPL/Inorganic Analysis ... ICPOES Data Report

Run Date >	4/26/2011	4/26/2011	4/26/2011	4/26/2011
Multiplier >	112.7	1127.0	91.9	919.2
RPL/LAB >	11-0793-129-S	11-0793-129-S @ 10x	11-0793-129-D	11-0793-129-D @ 10x
Client ID >	TI008-SA		TI008-SB	
(Analyte)	(µg/g)	(µg/g)	(µg/g)	(µg/g)
Ag		46.7		45.2
Al		36,200		34,000
As	182		167	
Ba		81.4		82.0
Be		15.9		15.2
Ca		2,080		1,770
Cd		51.5		45.5
Cr		714		671
Fe		135,000		130,000
Mg		1,090		981
Na		449		384
Ni		214		194
Pb		925		936
Sb	[15]		[17]	
Se	--		--	
Tl	[15]		[12]	
U		172,000		160,000
V		58.2		56.0
Zn		505		474
Zr		432		363
B		[36]		[36]
Bi		[120]		[98]
Ce		155		136
Co		[5.4]		[3.9]
Cu		1,920		1,820
Dy		--		--
Eu		4.09		4.41
K		541		470
La		43.1		40.0
Li		14.1		11.8
Mn		279		253
Mo		[68]		[64]
Nd		146		135
P		819		734
Pd		[6.9]		[6.8]
Rh		--		--
Ru		[37]		[31]
S		2,260		1,940
Si		1,220		1,280
Sn		[61]		[68]
Sr		15.9		14.1
Ta		--		--
Te		[52]		[26]
Th		205		180
Ti		453		400
W		--		[26]
Y		11.3		10.7

Battelle PNNL/RPL/Inorganic Analysis ... ICPOES Data Report

QC Performance 4/26/2011

Criteria >	≤ 20%	≤ 20%	≤ 20%	≤ 20%	80%-120%	80%-120%	80%-120%	80%-120%
QC ID >	11-0792 Dup		11-0793 Dup		LCS/BS		11-0793 MS	
Analytes	RPD (%)	RPD (%)	RPD (%)	RPD (%)	%Rec	%Rec	%Rec	%Rec
Ag				3.3		101		102
Al		11.4		6.4		103		nr
As			8.1		89		90	
Ba		76.7		0.7		102		101
Be		2.8		4.7		103		102
Ca		6.0		16.3		102		162
Cd		3.9		12.4		106		107
Cr		41.7		6.3		98		104
Fe		28.7		3.7		104		nr
Mg		17.9		10.5		103		119
Na		8.0		15.6		103		123
Ni		40.2		9.8		106		107
Pb		39.1		1.2		108		107
Sb					89		87	
Se					90		91	
Tl					86		78	
U		18.9		7.3		102		nr
V				3.9		103		103
Zn		4.1		6.4		100		103
Zr		143.2		17.3		107		nr
Other Analytes								C
B						104		105
Bi								
Ce				12.8		101		98
Co								
Cu		4.7		5.5		110		nr
Dy								
Eu				7.6				
K		3.7		14.0		105		109
La		4.1		7.6		99		101
Li		10.5		17.6		113		108
Mn		19.8		10.0		107		113
Mo						105		109
Nd				7.3		97		101
P				10.9				118
Pd								
Rh								
Ru								
S		1.1		15.5				99
Si		14.1		4.6		108		0
Sn								
Sr		6.7		12.1		103		128
Ta								
Te								
Th				13.0		100		101
Ti		56.1		12.5		106		107
W								
Y				5.7				

Shaded results are outside the acceptance criteria.

Battelle PNNL/RPL/Inorganic Analysis ... ICPOES Data Report

QC Performance 4/26/2011

Criteria >	80%-120%	80%-120%	80%-120%	≤ 10%	≤ 10%
QC ID >	11-0793 + AS-A		11-0793 + AS-B	11-0792 5-Fold Serial Dil.	
Analytes	%Rec	%Rec	%Rec	%Diff	%Diff
Ag		103			
Al		nr			0.5
As	100				
Ba		101			1.0
Be		99			4.7
Ca		101			0.2
Cd		92			
Cr		104			2.7
Fe		nr			1.2
Mg		98			2.0
Na		98			4.6
Ni		106			2.6
Pb		114			
Sb	96				
Se	98				
Tl	89				
U			102		0.7
V		97			
Zn		103			4.5
Zr		102			1.0
Other Analytes					
B		101			
Bi		92			
Ce			105		
Co		96			
Cu		nr			0.5
Dy			101		
Eu			102		
K		99			
La			101		
Li		105			
Mn		108			1.8
Mo		105			
Nd			103		
P		96			
Pd			99		
Rh			98		
Ru			101		
S			102		0.6
Si		98			3.4
Sn		85			
Sr		102			0.3
Ta		98			
Te			101		
Th			102		
Ti		112			2.1
W		98			
Y		98			

Shaded results are outside the acceptance criteria.

nr = spike concentration less than 25% of sample concentration. Matrix effects can be assessed from the serial dilu

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

Gamma Energy Analysis (GEA)

Project / WP#: 52578/ H41216
ASR#: 8824.00
Client: SK Fiskum
Total Samples: 3 Samples

Engineered Container and Core ID	RPL ID	Client Sample ID
KW210-LCC	11-0791-129-S	TI008-LA
KW210-MCC	11-0792-129-S, -D	TI008-MA, -MB
KW210-SCC	11-0793-129-S, -D, -MS	TI008-SA, -SB, -SC

Analysis Type:	GEA- for all positively measured or non-detected isotopes
Sample Processing Prior to Radiochemical Processing/Analysis	☒ None (TI008-LA) Only ☐ Digested as per RPG-CMC-128, Rev. 0 *HNO_3-HCl Acid Extraction of Liquids for Metals Analysis Using a Dry Block Heater* ☒ Digested as per PNL-ALO-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: Preparation may also involve attaining a GEA geometry that is compatible with the calibration geometry.
Analysis Procedure:	RPG-CMC-450, Rev. 1 *Gamma Energy Analysis (GEA) and Low-Energy Photon Spectrometry (LEPS)*
Reference Date:	* January 11, 2011 @ 15:14:41
Analysis Date or Date Range:	May 3-24, 2011
Technician/Analyst:	T Trang-Le
Rad Chem Electronic Data File:	*11-0792 Fiskum.xls*
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):	*Detector C,D,E,G,K,T (M & TE unique identifiers in attached supporting documents)*

*Reference date used is the same date and time used for samples submitted under ASR 8725 as directed by S. Fiskum.

T Trang-Le / 7/15/11
Prepare **Date**

Reviewer / 7/15/11
Date

SAMPLE RESULTS

Activities for all gamma emitters detected in these samples are presented in an attached Excel spreadsheets for ASR 8824.00. All sludge data are reported in units of $\mu Ci/g_{dry}$ and all residual solids are reported as $\mu Ci/g_{residual}$, both 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 residual solids that did not dissolve during acid digestion.

ASO Project File, ASR 8824 has been created for this report including all appropriate supporting records which may include the Pipette Performance Verification form, Standard Certificates, Laboratory Bench Record, Shielded Analytical Laboratory Bench Sheet, 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 sludge samples were prepared in the shielded analytical lab (SAL) hot cells by the Project and provided to the Analytical Support Operations (ASO) under Analytical Service Request (ASR) 8824 for chemical and radioanalytical analyses.

Sample TI008-LA was received from client pre-dried and analyzed by GEA "as received."

ASO received each sample requiring acid digestion in Teflon Oak Ridge centrifuge tubes, dried the contents of each centrifuge tube to a constant weight, and then leached the dried solids with hot dilute nitric and hydrochloric acids per procedure PNL-ALO-129. The leachate solution was used for radioanalytical analyses and ICP metals analysis; only GEA data are included in this report. At the completion of the acid digestion, residual unsolubilized material remained. The residual solids were dried to constant weight and the dried residuals were transferred to clean 22 mL glass scintillation vials for load out and GEA analyses. The residual solids do have measurable activity and those results are included in this report.

Quality control (QC) samples prepared include: 1) the duplicate pairs; TI008-MA, -MB and TI008-SA, -SB, 2) the 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. 7); 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):

A reagent blank was prepared with the preparation of the sludge samples in the SAL, but not for the preparation of the residual solids in the SAL.

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 sample. 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 or LCS samples analyzed for ASO GEA analyses. Instrument performance is assessed by the analyses of daily control counts and weekly background counts, as discussed below. (The sample identified as "MS" was prepared for ICP/OES analyses only. This MS sample is also being reported for GEA as additional information for the project.)

Duplicate Relative Percent Difference (RPD):

Duplicate results for the sludge are required to agree within ≤20% relative percent difference, taking into consideration the statistical uncertainties in the data. The residual solids results are not subject to the duplicate acceptance criteria. Duplicate results for the sludge samples ranged from 4% to 31%

For Sludge samples:

RPL Number	Client Sample ID	Isotopes	RPD Differences
11-0792-129-S, -D	TI008-MA, -MB	Co-60 Eu-154	31% 26%

Occurrence report (52578-06-27-11) documents DQO related issue(s) with this report: RPD's outside the ≤20% requirement.

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 used for the 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 an 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 PNL-ALO-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 efficiency, 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. A conservative estimate is that 2% is the lowest uncertainty possible for our GEA measurements taking into account systematic uncertainties in gamma calibration standards.

The samples were counted at about 10 cm from the detector to minimize dead time due to the high sample activities. Counting at such long distances also minimizes the differences between the sample geometry and the calibration geometry. Calibrations are performed using water samples whereas the samples consisted of mostly silica (however, the exact sample mineralogical composition and associated density were not known). The small sample size (<1g) minimized the gamma absorption effects; therefore, density corrections were not applied.

Comments

1. The results have been corrected 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. Occurrence report (52578-06-27-11) documents DQO related issue(s) with this report: RPD's outside the ≤20% requirement.

For Sludge samples:

RPL Number	Client Sample ID	Isotopes	RPD Differences
11-0792-129-S, -D	TI008-MA, -MB	Co-60 Eu-154	31% 26%

Attachment: Data Report Sample Results for ASR 8824.

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

filename 11-0792 Fiskum
5/19/2011

Client: Fiskum
ASR 8824

Project: 52578

Prepared by: T Trang-Le 5/19/11

Concur: C Soderquist 5-19-2011

Procedure: RPG-CMC-450, Rev. 1 Gamma Energy Analysis (GEA) and Low-Energy Photon Spectrometry (LEPS)

Reference date: January 11, 2011 @ 15:14:41
M & TE: Gamma Detector T

		Measured Activities, $\mu Ci/_{g\ dry}$ with 1-sigma uncertainty						
Sample ID	**RPL ID**	**Co-60**	**Cs-134**	**Cs-137**	**Eu-152**	**Eu-154**	**Eu-155**	**Am-241**
TI008-LA	11-0791-S	**1.48E-1 ± 2%**	<6.E-4	**6.25E+1 ± 4%**	<6.E-4	**5.64E-2 ± 2%**	<9.E-3	**1.04E+0 ± 10%**
	Requested Detection Limits	1.0E-2	2.0E-1	1.5E-2	1.5E+0	1.5E+0	2.0E+0	1.5E+1

This sample was counted for gamma emitters as received, in the same vials received from the project. No blank accompanied these samples.

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

filename 11-0792 Fiskum
5/9/2011

Client: Fiskum
ASR 8824

Project: 52578

Prepared by: T Trang-Le 5/9/11

Concur: C Soderquist 5-16-11

Procedure: PNL-ALO-129, Rev. 0, HNO3-HCl Acid Extraction of Solids Using a Dry-Block Heater
RPG-CMC-450, Rev. 1 Gamma Energy Analysis (GEA) and Low-Energy Photon Spectrometry (LEPS)

Reference date: January 11, 2011 @ 15:14:41

M & TE: Gamma Detector C,D,E,G,K

			Measured Activities, µCi per gram of acid-insoluble residue ± 1s uncertainty							
Sample ID	**RPL ID**		**Co-60**	**Cs-134**	**Sb-125**	**Cs-137**	**Eu-152**	**Eu-154**	**Eu-155**	**Am-241**
TI008-MA	11-0792-129-S		**4.08E-4 ± 5%**	<9.E-5	<1.E-3	**1.19E+0 ± 6%**	<1.E-4	**9.22E-4 ± 7%**	<7.E-4	**1.68E-2 ± 4%**
TI008-MB	11-0792-129-D		**2.89E-4 ± 7%**	<1.E-4	<3.E-3	**2.89E+0 ± 3%**	<1.E-4	**1.17E-3 ± 6%**	<2.E-3	**1.46E-2 ± 6%**
		RPD	34%	--	--	83%	--	24%	--	14%
TI008-SA	11-0793-129-S		**1.12E-3 ± 4%**	<7.E-5	**6.38E-3 ± 7%**	**5.83E-1 ± 4%**	<2.E-4	**1.30E-3 ± 7%**	<1.E-3	**3.65E-2 ± 7%**
TI008-SB	11-0793-129-D		**7.98E-4 ± 4%**	<6.E-5	**3.86E-3 ± 11%**	**6.13E-1 ± 4%**	<2.E-4	**1.22E-3 ± 5%**	<1.E-3	**3.30E-2 ± 5%**
		RPD	34%	--	49%	5%	--	6%	--	10%
TI008-SC	11-0793-129-MS		**2.24E-3 ± 3%**	<1.E-4	**5.13E-3 ± 12%**	**1.15E+0 ± 3%**	<2.E-4	**2.68E-3 ± 4%**	<1.E-3	**4.35E-2 ± 5%**
	Requested Detection Limits		1.0E-2	2.0E-1	-	1.5E-2	1.5E+0	1.5E+0	2.0E+0	1.5E+1

These five samples were counted for gamma emitters as received, in the same vials received from the project. No blank accompanied these samples.

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

filename 11-0792 Fiskum
6/10/2011

Client: Fiskum
ASR 8824

Project: 52578

Prepared by: T Trang-Le 6-13-2011

Concur: C. Soderquist 6-13-2011

Procedures: PNL-ALO-129, Rev. 0, HNO_3-HCl Acid Extraction of Solids Using a Dry-Block Heater
RPG-CMC-450, Rev. 1 Gamma Energy Analysis (GEA) and Low-Energy Photon Spectrometry (LEPS)
Reference date: January 11, 2011 @ 15:14:41
M & TE: Gamma Detector E, T

		Measured Activities, µCi per gram of dry K Basin sludge ± 1s uncertainty						
Sample ID	**RPL ID**	**Co-60**	**Cs-134**	**Cs-137**	**Eu-152**	**Eu-154**	**Eu-155**	**Am-241**
TI008-MA	11-0792-129-S	**9.10E-2 ± 3%**	<9.E-3	**2.96E+2 ± 4%**	<7.E-3	**1.52E-1 ± 3%**	<1.E-1	**2.45E+0 ± 10%**
TI008-MB	11-0792-129-D	**6.69E-2 ± 5%**	<1.E-2	**3.44E+2 ± 3%**	<7.E-3	**1.17E-1 ± 5%**	<1.E-1	**2.26E+0 ± 11%**
	RPD	31%	--	15%	--	26%	--	8%
TI008-SA	11-0793-129-S	**2.07E-1 ± 3%**	<8.E-3	**2.08E+2 ± 4%**	<1.E-2	**1.52E+0 ± 2%**	**2.55E-1 ± 10%**	**3.94E+1 ± 10%**
TI008-SB	11-0793-129-D	**1.94E-1 ± 3%**	<8.E-3	**2.03E+2 ± 4%**	<1.E-2	**1.46E+0 ± 2%**	**2.36E-1 ± 9%**	**3.65E+1 ± 10%**
	RPD	6%	--	2%	--	4%	8%	8%
TI008-SC	11-0793-129-MS	**2.21E-1 ± 3%**	<8.E-3	**2.21E+2 ± 4%**	<1.E-2	**1.58E+0 ± 2%**	**2.43E-1 ± 10%**	**4.09E+1 ± 10%**
Hot cell acid digestion blank		<1.E-5	<1.E-5	**3.98E-4 ± 3%**	<4.E-5	<3.E-5	<5.E-5	<2.E-4
Requested Detection Limits		1.0E-2	2.0E-1	1.5E-2	1.5E+0	1.5E+0	2.0E+0	1.5E+1

These samples were dried to constant weight, then digested in an HNO_3-HCl mixture. The matrix spike sample listed (TI008-SC) was spiked with stable metals, not radionuclides, and is essentially a triplicate analysis for radiochemistry.

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	WD25687	EG&G Ortec	GEM15180P	35TP11079A	Ge	15	Y

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

Alpha Spectrometry - Total Alpha

Project / WP#:	52578/H41216
ASR#:	8824
Client:	S. Fiskum
Total # of Samples:	4

Engineered Container & Core ID	Sample IDs	RPL Number
KW-210-MCC	TI008-MA	11-0792-129-S
	TI008-MB	11-0792-129-D
KW-210-SCC	TI008-SA	11-0793-129-S
	TI008-SB	11-0793-129-D

Analysis Type:	AEA – Total Alpha
Sample Processing Prior to Radiochemical Processing/Analysis	☐ None ☒ Digested as per PNL-ALO-129, Rev. 0, *HNO_3-HCl Acid Extraction of Solids Using a Dry Block Heater, 4-20-11 (N. Green)* ☐ Fusion as per PNL-ALO-115 Rev. 1.1, *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*
Total Alpha Preparation Procedure:	RPG-CMC-4001, Rev. 1, *Source Preparation For Gross Alpha and Gross Beta analyses.*
Technician/Analyst:	L. Darnell, 5-25-11
Spike Standard ID's	R-485-a-10 (Pu-239)
Analysis Procedure	RPG-CMC-408, Rev. 2, *Total Alpha and Beta Analysis*
Technician/Analyst:	L. Darnell, 5-25-11
Co-Precipitation Procedure:	RPG-CMC-496, Rev. 0, *Co-precipitation Mounting of Actinides for Alpha Spectroscopy*
Technician/Analyst:	L. Darnell, 5-31-11
Spike and Tracer Standard ID's:	R-485-a-10 (Pu-239)
Analysis Procedure:	RPG-CMC-422, Rev. 2, *Solutions Analysis: Alpha Spectrometry*
Reference Date:	Same as analyses dates
Analysis Date or Date Range:	6/1/11
Technician/Analyst:	T. Trang-Le
Analysis Data (File):	11-0792 Fiskum.xls, Alpha Beta
CMC Project 98620 File:	File Plan 5871: T 52578 8824: 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: 6/29/11

T Trang-Le
Reviewer / Date: 6/29/11

Sample Results

See attached data report, Sample Results for ASR 8824. All data are reported in units of μCi/g K-Basin dry sludge with a 1-σ uncertainty (see comments).

Sample Preparation, Separation, Mounting and Counting Methods

Four K-Basin sludge samples were prepared in the shielded analytical laboratory (SAL) hot cells by the Project and provided to the Analytical Support Operations (ASO) under Analytical Service Request (ASR) 8824 for chemical and radiochemical analyses. ASO received each sample in a Teflon Oak Ridge centrifuge tube, dried the contents of each centrifuge tube to a constant weight, and then leached the dried solids with hot dilute nitric and hydrochloric acids per procedure PNL-ALO-129. The leachate solution was used for radiochemical analyses and ICP metals analysis; only Alpha-AEA is reported in this report. Visible residual solids remained following the PNL-ALO-129 digestion.

Following the digestion process, an aliquot of the digestate was mounted for alpha spectrometry by co-precipitation using procedure RPG-CMC-496, then counted by alpha spectroscopy using procedure RPC-CMC-422. The samples were counted on June 1st, 2011; no decay corrections were made.

Although not formally requested by the ASR, gross alpha analyses was performed on each sample to obtain information to estimate aliquot size for the alpha-AEA and for checking the internal consistency of the alpha isotopic data. The gross alpha results are included in the data tables as supplemental information only. Gross alpha activity was measured by evaporating small aliquots of leachate onto counting planchets per procedure RPG-CMC-4001 and counting per procedure RPG-CMC-408. These procedures are not required to be pre-approved by K-Basin project staff as they are used to generate opportunistic data only. The sum of the major alpha emitters, in this case Pu-239+240 and Pu-238 + Am-241, agrees very well with the gross alpha results.

QUALITY CONTROL RESULTS

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

Performance acceptance criteria for these samples are established by both the K Basin data quality objectives (as provided as special instruction in the ASR) and the ASO Quality Assurance (QA) Plan (ASO-QAP-001, Rev. 7). Except where noted, the Alpha-AEA K-Basin DQO and ASO QAP criteria are the same. In cases where the QC criteria are different, K-Basin DQO's take precedence. 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:

Tracer is not used for this analysis.

Hot Cell Process Blank (PB) and Laboratory separations blank (LB):

The alpha isotopic (Pu-239+240, Pu-238+Am-241 and Cm-243+244) activities 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 alpha isotopic activities are significantly less than 5% of any sample activity. There are no acceptance criteria for LB. The detection limit achieved in the PNL-ALO-129 acid digestion blank for Pu-239+240 is 4.43 E-5 $\mu Ci/g_{dry\ sludge}$, for Am-241+Pu-238 is 6.47 E-5 $\mu Ci/g_{dry\ sludge}$ and for Cm-243+244 is 1.36 E-5 $\mu Ci/g_{dry\ sludge}$. The achieved detection limits are all below the requested detection limit of 1.0E-3 $\mu Ci/g_{dry\ sludge}$.

Blank Spike (BS) – reagent spike:

The BS recovery of 100% (Pu-239) meets the acceptance criteria of 80% to 120% recovery.

Matrix Spike (MS):

The MS recovery of 96% (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 11-0792-129-D (TI008-MB) was selected as the matrix spike sample.

Duplicate - Relative Percent Difference (RPD):

Duplicate results are required to agree within <20% RPD. Duplicate results (samples TI008-MA and TI008-MB duplicate) for Pu-238+Am-241 is 34%, and for Pu-239+240 is 29% and for Cm-243+244 is 52% thus exceeding the acceptance criteria for duplicate precision. Occurrence Report 52578-06-27-11 has been generated to document the duplicate results exceeding the acceptance limits.

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

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 and dilution (to avoid excessive consumption of spike and avoid creating unnecessary waste). The MS prepared with this batch of samples is considered a PS, since the Pu-239 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.

Attachment: Data Report -- Sample Results for ASR 8824

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

filename 11-0792 Fiskum
6/15/2011

Client: Fiskum
ASR 8824

Project: 52578

Prepared by: C S. deRyuiter 6-15-2011

Concur: T Trang-Le 6/15/2011

Procedures: PNL-ALO-129, Rev. 0, HNO_3-HCl Acid Extraction of Solids Using a Dry-Block Heater
RPG-CMC-4001, Rev 1, Source Preparation for Gross Alpha and Gross Beta Counting
RPG-CMC-408, Rev 2, Total Alpha and Beta Analysis
RPG-CMC-496, Rev 0, Coprecipitation Mounting of Actinides for Alpha Spectrometry
RPG-CMC-422, Rev 2,Solutions Analysis: Alpha Spectrometry

Count date: 5/25/11 and 6/1/11

M & TE: Ludlum alpha counters, Alpha spectrometry counting system

Measured Activities, µCi per gram of dry K Basin sludge ± 1s uncertainty

Sample ID	RPL ID	Gross Alpha	Pu-239+240	Pu-238 + Am-241	Cm-243+244
TI008-MA	11-0792-129-S	**5.78E+0 ± 2%**	**2.17E+0 ± 2%**	**3.61E+0 ± 2%**	**5.03E-3 ± 16%**
TI008-MB	11-0792-129-D	**4.15E+0 ± 2%**	**1.62E+0 ± 2%**	**2.56E+0 ± 2%**	**2.96E-3 ± 20%**
	RPD	33%	29%	34%	52%
TI008-SA	11-0793-129-S	**1.02E+2 ± 1%**	**4.56E+1 ± 2%**	**5.03E+1 ± 2%**	<4.E-2
TI008-SB	11-0793-129-D	**9.74E+1 ± 1%**	**4.72E+1 ± 2%**	**5.05E+1 ± 2%**	**5.98E-2 ± 32%**
	RPD	5%	3%	1%	--
Hot cell acid digestion blank		<2.E-4	4.43E-5 ± 8%	6.47E-5 ± 7%	1.36E-5 ± 15%
Lab blank		<3.E-6	<5.E-8	<6.E-8	<4.E-8
Reagent spike		90%	100%	--	--
Matrix spike (TI008-MB)		75%	96%	--	--
Requested Detection Limits		--	1.0E-3	1.0E-3	--

These samples were dried to constant weight, then digested in an HNO_3-HCl mixture in a hot cell. The acid digestion solution was used to measure the alpha emitters listed on this page.

Radiochemistry M&TE List

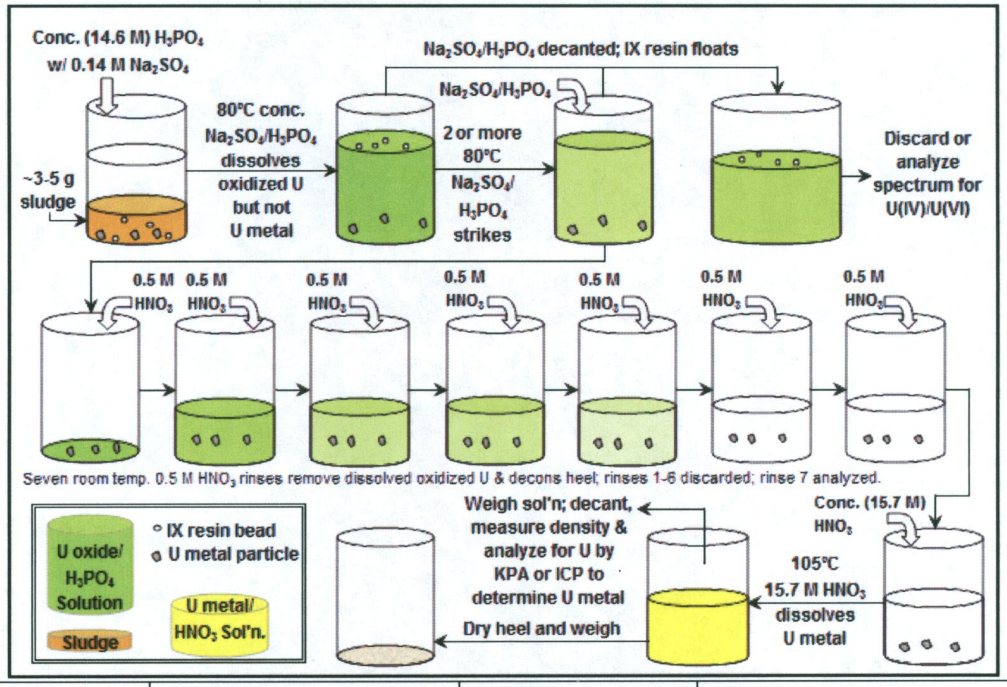
ID	Number of Detectors	Property #	Model or Serial #	Location, in Bldg. 325	Procedures using the M&TE	PM Contract?
Dual Alpha Beta Gas Proportional Counters						
Oxford LB4100	16	WD13066	L8285-O	425	Total alpha, total beta, Sr/Y90, Tc99	Y
Alpha Counters						
Ludlum	10	multiple	multiple	425	Total Alpha	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	WD25687	EG&G Ortec	GEM15180P	35TP11079A	Ge	15	Y

Battelle - Pacific Northwest National Laboratory
Analytical Support Operations –
Uranium Metal Concentration in K Basin Sludge Report – Revision 2

PO Box 999, Richland, Washington 99352



Client:	Sandy Fiskum	***ASR #:***	8824
Project #:	52578	***# Samples:***	7+1 standard+1 process blank
Charge Code:	H41220		
RPL Numbers:	11-0792-107-LCS, -B2, -S, -D; 11-0793-107-S, -D, -MS; 11-0967-107-S, -D		

Procedure, Analysis, System, and Records Information

Sample Prep:	RPG-CMC-107, Rev. 1, *Sample Preparation for Determination of Uranium Metal Concentrations in Sludge* PNL-ALO-501, Rev. 0, *Laboratory Procedure for Measurement of Physical and Rheological Properties of Solutions, Slurries, and Sludges*
Analysis Procedure:	RPG-CMC-211, Rev. 3. *Determination of Elemental Composition by Inductively Coupled Argon Plasma Optical Emission Spectrometry (ICP-OES)*
Analyst:	Cal Delegard/Nicole Green (digestion) and Jenn Carter (ICP-OES)
Analysis Dates:	Digestion – 13 April 2011; ICP – 19 April 2011
Calibration Date/ Filename:	ICP-325-405-3 in Chemical Measurement Center 98620 file; 4 March 2011
Cal/Ver Stds Prep Date:	LCS: 8 April 2011
Sample Analysis Excel Data Files:	ICP: C0374, 19 April 2011
M &TE Numbers	Balances 1114123894, Lab 516 and 25608154, Lab 312 (U metal beads--LCS & MS) Balance N04143 (Container Tare) Balance 40023610, SAL (solution weights); Balance 360-06-01-037 (sol'n. density) Balance Mettler AT400 M19445 (ICP-OES) ICP-OES instrument N827583 (Lab 405)
All Analysis Records	Chemical Measurement Center 98620: RIDS U-Metal System File

Battelle - Pacific Northwest National Laboratory
Analytical Support Operations –
Uranium Metal Concentration in K Basin Sludge Report – Revision 2
PO Box 999, Richland, Washington 99352

16 Aug 2013
Prepared By Date

08/19/2013
Reviewed By Date

Battelle - Pacific Northwest National Laboratory Analytical Support Operations – Uranium Metal Concentration in K Basin Sludge Report – Revision 2

PO Box 999, Richland, Washington 99352

Revisions 1 and 2

Revision 1 to the report was made to remove "less than" symbols (<) from the values for U_{metal} found, g, and $[U_{metal}]$, expressed in µg/g and wt%, in Table A.1 for the Process Blank 2 sample.

Revision 2 to the report was made to correct the number of samples delivered as wet slurries from two to five and the number delivered as dried solids from five to two in the first paragraph of the section "Sample Analysis/Results Discussion". The drying method also was further clarified to be "air-dried". A new second paragraph in the "Sample Analysis/Results Discussion" section was added to explain the mass differences for the KW-210-MCC samples between those reported in ASR 8824 and those presented in Table A.1 of Attachment 1. Finally, the explanatory paragraph in Attachment 1 was corrected to describe the KW-210-SCC samples as being dewatered (not dried) and the drying method further clarified to be "air-dried".

Sample Results

See Attachment 1: Uranium Metal Sample Results ASR 8824.

Sample Analysis/Results Discussion

The results of uranium metal concentration analysis of seven samples of K-Basin sludge submitted under Analytical Service Request (ASR) 8824 are reported. Five samples were delivered for analysis as wet slurries with an overburden of supernatant water and two as air-dried solids. The samples were analyzed in a single batch.

The bulk KW-210-MCC material was air-dried in the hot cell before mixing, aliquoting, and weighing into 5-mL plastic vials. The MCC splits used for the uranium metal digestions then were transferred to Oak Ridge centrifuge tubes, as required under RPG-CMC-107, Rev. 1, and the amounts transferred determined by weighing. The transferred amounts were nearly quantitative (~99 wt%) with the materials not transferred likely retained within the plastic 5-mL vials by electrostatics. Because of this retention, the aliquot masses in ASR 8824 are slightly higher than those given in Table A.1 of Attachment 1.

The samples for uranium metal analysis were digested according to the PNNL Technical Procedure, RPG-CMC-107, Rev. 1, *Sample Preparation for Determination of Uranium Metal Concentrations in Sludge*. The contacts with 0.14 M Na_2SO_4 in concentrated H_3PO_4 are designed to dissolve and remove the accompanying uranium oxides. ASR 8824 Special Instructions specified a minimum of three contacts with 0.14 M Na_2SO_4 in concentrated H_3PO_4. The second and third contact solutions were colorless to the eye, indicating good dissolution of the contained uranium oxides.

The residues from the Na_2SO_4/H_3PO_4 contacts were rinsed sequentially seven times in 0.5 M HNO_3 and the rinsed heels, containing the undissolved uranium metal, were digested in concentrated HNO_3 to dissolve the uranium metal, all according to the digestion procedure, RPG-CMC-107. For each concentrated HNO_3 digestate sample, mass and density were measured and uranium concentration in the digestate was measured by inductively coupled plasma optical emission spectrometry (ICP-OES) according to RPG-CMC-211, Rev. 3, *Determination of Elemental Composition by Inductively Coupled*

Argon Plasma Optical Emission Spectrometry (ICP-OES). The sample digestion procedure followed under RPG-CMC-107 is outlined in the Figure at the top of page 1.

Data Limitations

None.

Quality Control Discussion

The method performance is evaluated against the acceptance criteria provided in ASR 8824. Values at the Process Quantitation Limit (0.03 wt% U in sludge) or greater are considered to be quantified with an uncertainty of ±25% (at 2σ, relative) or better.

Blanks: Reagent blanks are analyzed at least once in every batch of 20 samples. Blanks of the 0.5 M HNO_3 rinse solution and the concentrated HNO_3 metal digestion solution were prepared with the sample batch; this result is in the supporting ICP-OES report. A blank containing only Hanford sand was included in the batch and was taken through all of the procedure steps. The blanks were near or below the analytical detection limits established for the ICP-OES (0.012 µg/mL). The blank level for the 0.5 M HNO_3 reagent solution (Table A.1) was generally only a small contributor to the observed 7th rinse solution concentrations and, even when near the observed rinse solution concentrations, did not interfere with the key observations of rinse solution concentration which become important at values greater than about 100 µg/mL. Similarly, the concentrated HNO_3 blank solution concentration contributed negligibly to the 177 to 5,340 µg/mL digestate concentrations.

Seventh Rinses: The uranium concentrations in the 7th 0.5 M HNO_3 rinses indicate the thoroughness of oxidized (i.e., non-metallic) uranium removal from the sludge. In developing this analytical technique (Delegard et al. 2008), uranium concentrations in the 7th rinse less than ~100 µg U/mL gave good indication that the uranium oxides were dissolved and their solutions removed from the heel.

As shown in Table A.1, all of the 7th rinse solution concentrations were less than 100 µg/mL in uranium. The rinse concentrations ranged from 0.04 to 1.5 µg U/mL and indicate complete removal of uranium oxide from the sample. The uranium concentrations in the 7th rinse were examined along with other batch quality control indicators, including duplicates, LCS results, and process blanks, in making data quality judgments.

Laboratory Control Samples (LCS): A single LCS was analyzed with the K Basin sludge samples. The LCS contained ~0.5 g of U_3O_8 (weighed to the nearest 0.001 g), ~0.6 g of Hanford sand (from Arid Lands Ecology, ALE, Reserve; weighed to the nearest 0.001 g), and ~0.012 g of uranium metal (weighed to the nearest 0.00001 g) present as a single near-spherical bead.

The LCS uranium metal recovery was 103% and was within the acceptance limits of 75-125% specified in the procedure RPG-CMC-107 and in the ASR 8824 Special Instructions.

Duplicate (Precision): A duplicate sample is required to be analyzed in every batch of 20 samples or fewer. This requirement was met.

Prior to this Project work, the MDL, 0.011 wt% U_{metal} on a settled sludge basis, was determined by evaluating uranium metal analysis recovery data (defined as PDL in Attachment J of Fiskum et al. 2011). The ASR specifies an MDL of 0.01 wt%, settled sludge basis. All sample wt% results were above the ASR-specified MDL. Refer to footnote (a) of Table A.1.

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The QAPjP/SAP (KBC-33786, Rev. 2) specifies that a ±20% precision criterion for duplicates is to be applied when [U_{metal}] >0.1 wt%. The [U_{metal}] >0.1 wt% threshold was exceeded for the KW-210-MCC and KW-210-A4 duplicate sample sets. The RPD for duplicate KW-210-MCC samples was 86% and the RPD for KW-210-A4 was 4%. Occurrence Report 52578-05-19-2011 was generated to document that the duplicate RPD values for KW-210-MCC did not meet the acceptance limits.

Matrix Spike: Based on the QAPjP/SAP (KBC-33786, Rev. 2), uranium metal measurement accuracy is to be assessed by evaluation of the LCS recoveries. However, a matrix spike (MS) sample was included to provide additional indicators of method performance. To prepare the MS sample, a uranium metal bead spike was added to one of three sludge subsamples taken from the same set of KW-210-SCC samples. The amount of U metal added as MS was about 7-times greater than the average amount of uranium metal found present in the other two -SCC subsamples. The MS recovery, adjusted for the uranium metal quantity expected based on the average concentration found in the companion subsamples, was 123%.

Deviations from Procedure

There were no deviations from the procedure.

General Comments

In June 2009, based on discussions between PNNL and CHPRC, it was determined that matrix spikes were to be included within each analytical batch to be processed for uranium metal and uranium oxide oxidation state distribution. While the matrix spike samples were intended to provide an additional assessment of the procedure performance, performance or failure criteria for matrix spike recoveries were not established.

References

ASO-QAP-001, Rev. 7. 2009. *Analytical Support Operations (ASO) Quality Assurance Plan*, Pacific Northwest National Laboratory, Richland, WA.

Delegard CH, SI Sinkov, AJ Schmidt, and JW Chenault. 2008. *Uranium Metal Analysis via Selective Dissolution*. PNNL-17800, Pacific Northwest National Laboratory, Richland, WA.

Fiskum SK, JM Billing, SJ Bos, CA Burns, CD Carlson, DS Coffey, JV Crum, RC Daniel, CH Delegard, MK Edwards, OT Farmer, LR Greenwood, SA Jones, D Neiner, BM Oliver, KN Pool, AJ Schmidt, RW Shimskey, SI Sinkov, SZ Soderquist, CJ Thompson, ML Thomas, T Trang-Le, and MW Urie. 2011. *Characterization Data Package for Containerized Sludge Samples Collected from Engineered Containers SCS-CON-240, 250, 260, and 220*. PNNL-19035, Rev. 1, Pacific Northwest National Laboratory, Richland, WA.

KBC-33786, Rev. 2, *Quality Assurance Project Plan / Sampling and Analysis Plan for Sludge in the KW Engineered Containers*, CH2M Hill Plateau Remediation Company, Richland, WA. December 2009.

Attachment 1 Uranium Metal Concentration Sample Results for ASR 8824

The analytical results for uranium metal concentration in the air-dried (KW-210-MCC) and dewatered (KW-210-A4 and KW-210-SCC) sludge samples are presented in Table A.1. Quality assurance data (i.e., blank concentrations, LCS yield, MS yield) also are presented.

Table A.1. Sample and Quality Assurance Analytical Results for Uranium Metal Concentration

ASO ID	RPL Sample ID	Sample Description / Client Core ID	Sample Wt., g	[U], µg/mL		U_{metal} added, g	U_{metal} found, g	$[U_{metal}]$		U_{metal} Rec., %	RPD or RSD, %
				Digest	7th rinse			µg/g [a]	wt% [a]		
11-0792-107-B3,B4	PB3, PB4	HNO_3 reagents		<0.120	<0.012						
11-0792-107-B2	Process Blank 2	Hanford sand	0.602	0.64	<0.012		3.88E-6	*6.5*	*0.00065*		
11-0792-107-LCS	LCS	U_{metal}/U_3O_8	1.1159	1360	0.146	0.01191	0.01223	11000	1.10	103	
11-0792-107-S	TI008-ME	KW-210-MCC	2.49	1190	1.50		0.01064	4260	0.426		**86** RPD [b]
11-0792-107-D	TI008-MF	KW-210-MCC	2.60	591	0.359		0.00443	1700	0.170		
11-0793-107-S	TI008-SE	KW-210-SCC	2.19	168	0.042		0.00147	674	0.0674		[c]
11-0793-107-D	TI008-SF	KW-210-SCC	3.53	177	0.082		0.00150	426	0.0426		
11-0967-107-S	TI002-A4-R	KW-210-A4	4.37	5340	0.475		0.03607	8250	0.825		4 RPD
11-0967-107-D	TI002-A4-S	KW-210-A4	4.36	4150	0.937		0.03467	7950	0.795		
11-0793-107-MS	TI008-SG	KW-210-SCC	4.18	1870	0.170	0.01081	0.01559	1140 [d]	0.114 [d]	123	

[a] Values in *italics* represent $[U_{metal}]$ results that are below the process detection limit (PDL) of 0.011 wt% (110 µg/g). The process quantitation limit (PQL) is 0.03 wt% (300 µg/g). Values at the PQL or greater are considered to be quantified with an uncertainty of ±25% (at 2σ, relative) or better. See Attachment J of Fiskum et al. (2011).

[b] RPD exceeds ±20% criterion established by the QAPjP/SAP (KBC-33786, Rev. 2).

[c] RPD not calculated because some or all values are below 0.1 wt% U_{metal}.

[d] The amount of added U metal spike was deducted from the total uranium to determine wt% U_{metal} for this matrix-spiked sample.

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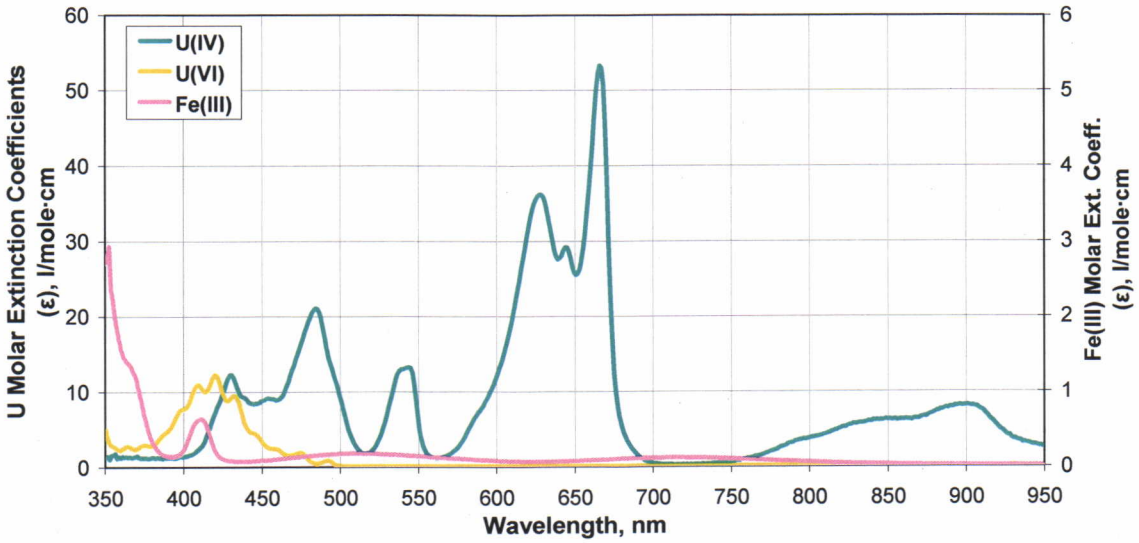

Client:	Sandy Fiskum	***ASR #:***	8824
Project #:	52578	***# Samples:***	7 samples, 1 standard, 1 blank
Charge Code:	H41221		
RPL Numbers:	11-0792-107-LCS, -B2, -S, -D; 11-0793-107-S, -D, -MS; 11-0967-107-S, -D		

Procedure, Analysis, System, and Records Information

Analysis Procedure:	RPG-CMC-255, Rev. 0, *Sample Preparation and Analysis for Determining Uranium Oxide Oxidation States in K Basin Sludges*
Sample Prep:	RPG-CMC-107, Rev. 1, *Sample Preparation for Determination of Uranium Metal Concentrations in Sludge*
Analyst:	Sergey Sinkov
Analysis Dates:	15-19 April 2011
Calibration Date/ Filename:	12/2008 RPG-CMC-255 – U Oxidation State Calculation – Final.xls
Cal/Ver Stds Prep Date:	8 April 2011
Sample Analysis Excel Data Files:	RPG-CMC-255 – U Oxidation State Calculation – ASR 8824.xls 041811 Spectra of H3PO4 digestates from hot cell – ASR 8824.xls
M &TE Numbers	Spectral Instruments SI 400 (property number WD13494) in Lab 516
All Analysis Records	Chemical Measurement Center 98620: Fileplan U Oxide System File

Prepared By — Date: 19 MAY 2011

Reviewed By — Date: 05/19/2011

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Sample Results

See Attachment: Uranium Oxide Oxidation States Sample Results for ASR 8824.

Sample Analysis/Results Discussion

The results for the uranium oxidation state distribution of the uranium oxides in seven samples of K-Basin settler tube sludge submitted under Analytical Service Request (ASR) 8824 are reported. All samples were from container KW210.

The samples were prepared according to the PNNL Technical Procedure, RPG-CMC-107, Rev. 1, *Sample Preparation for Determination of Uranium Metal Concentrations in Sludge*, to dissolve and remove the accompanying uranium oxides. There were no problems with the digestions. The digestates from the uranium oxide dissolutions were analyzed by spectrophotometry to determine the oxidation state distributions [to the U(IV) and U(VI) oxidation states] of the constituent uranium oxides according to the ASO procedure RPG-CMC-255, Rev. 0, *Sample Preparation and Analysis for Determining Uranium Oxide Oxidation States in K Basin Sludges*.

Two additional samples from KW210-A4 also were run with the present batch to provide supplemental information to results obtained under ASR 8798 for KW210-A4.

Data Limitations

The U(IV) and U(VI) oxidation state ratios should be used with caution. Ferrous iron, Fe(II), which is present in magnetite that has been observed in K Basin sludge, has been shown to reduce U(VI) to U(IV) in Na_2SO_4/H_3PO_4 solutions; the amount of Fe(II) is not known in these samples. The U(VI) fraction can be considered a lower bounding case and the U(IV) fraction can be considered upper bounding case.

Quality Control Discussion

The uranium oxidation state method performance is evaluated against the acceptance criteria established within procedure RPG-CMC-255, Rev. 0, *Sample Preparation and Analysis for Determining Uranium Oxide Oxidation States in K Basin Sludge*, and any client specified special instructions provided in the ASR.

Blanks: The uranium oxide and many of the accompanying sludge phases were dissolved at ~80°C by three sequential contacts with 0.14 M Na_2SO_4 in concentrated H_3PO_4. In accordance with the procedure, RPG-CMC-107, Rev. 1, *Sample Preparation for Determination of Uranium Metal Concentrations in Sludge*, a process blank of 0.14 M Na_2SO_4 in concentrated H_3PO_4 was prepared per the requirement that one process blank be prepared and analyzed at least once in every batch of 20 samples. The process blank contained ~0.6 g of Hanford sand and showed <0.001 M each of U(IV) and U(VI), below the minimum concentrations detectible by the uranium oxidation state method. The process blank showed negligible uranium concentration indicating negligible contamination.

Laboratory Control Samples (LCS): A single Laboratory Control Sample (LCS) was analyzed, meeting the requirement that at least one LCS to be analyzed in parallel with every batch of 20 samples. The

present batch had 9 K Basin sludge samples. The LCS sample contained ~0.5 g of U_3O_8 and ~0.6 g of Hanford sand (each weighed to the nearest 0.001 g) and ~0.01 g of uranium metal (weighed to the nearest 0.00001 g) present as a single near-spherical bead. The U(IV) and U(VI) uranium oxidation state distributions in the dissolved U_3O_8 were measured to determine analytical performances of the uranium oxide oxidation state distribution analyses. Because U_3O_8 is effectively one part U(IV) and two parts U(VI) on a mole basis, the U_3O_8 dissolved in the Na_2SO_4/H_3PO_4 solution ideally yields 33.3% U(IV) and 66.7% U(VI). Per the procedure, the relative amounts of U(IV) and U(VI) in the U_3O_8 LCS should vary by no more than 5%, absolute, from the ideal 33.3% (i.e., from 28 to 38%) and 66.7% (i.e., from 62 to 72%), respectively. The QAPjP/SAP (KBC-33786, Rev. 2) specifies that the LCS values differ by no more than 25%, relative, from the true values and thus affords greater tolerance margins. The LCS sample for this batch contained 34.2% U(IV) and 65.8% U(VI) and thus met the target performances for both the procedure and the less stringent QAPjP/SAP.

Duplicate (Precision): A duplicate sample is required to be analyzed in every batch (of 20 samples or fewer) to allow determination of method precision. This requirement was met. Each of the three samples was measured at least in duplicate.

Per the ASO QA Plan (ASO-QAP-001, Rev. 7), the relative percent difference (RPD) is calculated in cases where both U(IV) and U(VI) concentrations are measured at or above the 0.001 M minimum detectible concentration for two samples. The ≥0.001 M U(IV) and U(VI) concentration criteria were met for one of the three samples (from the small size fraction of the KW210 core composite, KW210-SCC). The other two samples had <0.001 M U(VI). The reported RPD for KW210-SCC for the U(IV) mole%, shown in Table A.1, was 7.0% and the RPD for U(VI) was 35%. No RPD requirement for uranium oxidation state is provided in the ASR special instructions.

The QAPjP/SAP states that the results should agree ±5% (absolute) if concentrations greater than 0.001 M in each oxidation state are present (KBC-33786, Rev. 2). The individual KW210-SCC uranium oxidation state values are within ±5%, absolute, of their average or 78.2 to 88.2% for U(IV) and 11.8 to 21.8% for U(VI). Therefore, this objective was met.

A single matrix spike (MS) sample was run for KW210-SCC. This MS contained an added bead of uranium metal. The uranium metal spike should not alter the uranium oxide oxidation state distribution. The U(IV) and U(VI) percentage values for the MS were between, and thus were consistent with, the values found for the associated sample and duplicate uranium oxidation state analyses for KW210-SCC. No Data Quality Objectives are associated with the oxidation state result for the MS sample.

Deviations from Procedure

There were no deviations from the procedure RPG-CMC-255, Rev. 0, *Sample Preparation and Analysis for Determining Uranium Oxide Oxidation States in K Basin Sludges*.

References

ASO-QAP-001, Rev. 7. 2009. *Analytical Support Operations (ASO) Quality Assurance Plan*, Pacific Northwest National Laboratory, Richland, WA.

KBC-33786, Rev. 2, *Quality Assurance Project Plan / Sampling and Analysis Plan for Sludge in the KW Engineered Containers*, CH2M Hill Plateau Remediation Company, Richland, WA. December 2009.

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Attachment

URANIUM OXIDE OXIDATION STATES SAMPLE RESULTS FOR ASR 8824

The analytical results for uranium oxide oxidation state in the digested sludge samples are presented in Table A.1. Values of the average oxidation state also are presented with the RPD in Table A.1 for samples where the U(IV) and U(VI) concentrations were greater than the detection limit. The U(VI) concentrations for samples from KW210-MCC and -A4 were below the 0.001 M detection limit, so the mole% of oxidation species were calculated as the limiting concentration, i.e., the U(IV) percentage is greater than the value listed in Table A.1.

Table A.1. Uranium Oxide Oxidation States Sample Results ASR 8824

ASO ID	RPL Sample ID	Sample Description / Client Core ID	Concentration, M		Ox'n. State, % [a]		RPD, % [b]		Quality	
			U(IV)	U(VI)	U(IV)	U(VI)	U(IV)	U(VI)	U(IV)	U(VI)
11-0792-107-B1	Process Blank 1	Na_2SO_4/H_3PO_4	<0.001	<0.001	NA	NA	NA	NA	NA	NA
11-0792-107-B2	Process Blank 2	Hanford sand	<0.001	<0.001	NA	NA	NA	NA	NA	NA
11-0792-107-LCS	(U_{metal}/U_3O_8) - LCS	LCS	0.0105	0.0202	34.2	65.8	NA	NA	Pass [c]	Pass [c]
11-0792-107-S	TI008-ME	KW-210-MCC	0.0040	<0.001	>80.0	<20.0	NA	NA	NA	NA
11-0792-107-D	TI008-MF	KW-210-MCC	0.0040	<0.001	>80.1	<19.9			NA	NA
11-0793-107-S	TI008-SE	KW-210-SCC	0.0098	0.0024	80.3	19.7	7.0	35	Pass [d]	Pass [d]
11-0793-107-D	TI008-SF	KW-210-SCC	0.0153	0.0025	86.1	13.9				
11-0967-107-S	TI002-A4-R	KW-210-A4	0.0111	<0.001	>91.8	<8.2	NA	NA	NA	NA
11-0967-107-D	TI002-A4-S	KW-210-A4	0.0117	<0.001	>92.1	<7.9			NA	NA
11-0793-107-MS	TI008-SG	KW-210-SCC	0.0213	0.0035	85.8	14.2	NA	NA	NA	NA

(a) The U(IV) and U(VI) oxidation state ratios for the sludge samples should be used with caution. Ferrous iron, Fe(II), which is present in magnetite that has been observed in K Basin sludge, has been shown to reduce U(VI) to U(IV) in Na_2SO_4/H_3PO_4 solutions; the amount of Fe(II) is not known in these samples. The U(VI) fraction can be considered a lower bounding case and the U(IV) fraction can be considered upper bounding case.

(b) RPD for U(IV)% and U(VI)% in samples 11-0419-107-S and -D [100×(difference/mean)].

(c) The LCS requirement per the procedure for U(IV) is from 28.3 to 38.3%, ±5% (absolute), of the theoretical 33.3% U(IV) in U_3O_8, and 61.7 to 71.7%, ±5% (absolute), of the theoretical 66.7% U(VI) in U_3O_8. The QAPjP/SAP requirement is ±25% (relative; KBC-33786, Rev. 2). Both criteria were met.

(d) The QAPjP/SAP states that the results should agree ±5% (absolute; KBC-33786, Rev. 2) if concentrations greater than 0.001 M in each oxidation state are present. The values for KW210-SCC are within ±5%, absolute, of average and thus lie within the ranges 78.2 to 88.2% for U(IV) and 11.8 to 21.8% for U(VI). This criterion was met.

52578-04-29-11

K-Basin Occurrence Report

Title: ASR 8824 Duplicates Exceeded 20% Requirement for ICP/OES from -129 Acid Digestion and [Na] in PB Greater Than 5% Present in the Samples		Date: 4-29-11
Unique Identifier: 52578-04-29-11		
Primary Person Identifying Issue: Brian Oliver		
Distribution: Email and posting on K-Basin Share-Point	Clark Carlson	
	Deborah Coffey	Andy Schmidt
	Sandy Fiskum	Rick Shimskey
	Karl Pool	Truc Trang Le, Project file for ASR 8824
Date Submitted to K-Basin Records:	8 June 2011	

Occurrence Description: ASR#: 8824

1) ICP/OES duplicate RPD's for sample and duplicate (TI008-MA and TI008-MB) exceed the 20% RPD requirement for the following target analytes:
   a) Ba – 76.7%
   b) Cr – 41.7%
   c) Fe – 28.7%
   d) Ni – 40.2%
   e) Pb – 39.1%
   f) Zr - 143.2%

2) The Na concentration in the process blank (71.5ppm) exceeds 5% of the Na level present in samples TI008-SA (Na blank is 15.9% of the sample result) and TI008-SB (Na blank is 18.6% of the sample result).

Impact of Occurrence:

The variability between sample and duplicate is an indication of primary sample heterogeneity.

The elevated Na in the process blank could results in a positive bias for the Na results for this batch run.

Significant Condition Adverse to Quality? Yes _____ No X

Evaluated by: [signature] Date: 6/8/2011

Further Disposition: ☒ No ☐ Yes If yes, ☐ Nonconformance? ☐ Deficiency?

Date Occurrence Closed: 6/8/2011

Signature/Date of Responsible QE: Deborah Coffey 6/8/2011

Signature/Date of Responsible K-Basin Manager: [signature] 6/8/2011

Comments:

8doc 6-30-11
52578-06-27-11

K-Basin Occurrence Report

Title: ASR 8824 GEA and Alpha-AEA duplicate results exeed ± 20% requirement.		Date: 06-27-11
Unique Identifier: 52578-06-27-11		
Primary Person Identifying Issue: Truc Trang-Le		
Distribution: Email and posting on K-Basin Share-Point	Clark Carlson	
	Deborah Coffey	Andy Schmidt
	Sandy Fiskum	Rick Shimskey
	Karl Pool	Truc Trang Le, Project file for ASR 8824
Date Submitted to K-Basin Records:	6-30-2011	

Occurrence Description: ASR: 8824

GEA Analyses:
The duplicate relative percent difference (RPD) for Co-60 and Eu-154 on samples TI008-MA and TI008-MB is 31% and 26% respectively. Both of these RPDs exceed the acceptance limit of ± 20%.

Alpha AEA Analyses:
The duplicate relative percent difference (RPD) for Pu-238+Am-241 is 34%, for Pu-239+240 is 29% and for Cm-243+244 is 52% for samples TI008-MA and TI008-MB. These RPDs exceed the accetance limit of ± 20%.

Impact of Occurrence:

There is no impact due to the RPDs exceeding the acceptance limit for these two isotopes.

The variation observed is reflective of the heterogeneous nature of the mid fraction samples.

Significant Condition Adverse to Quality? Yes _____ No X

Evaluated by: [signature] Date: 6/30/2011

Further Disposition: ☒ No ☐ Yes If yes, ☐ Nonconformance? ☐ Deficiency?

Date Occurrence Closed: 6-30-2011

Signature/Date of Responsible QE: Deborah Coffey 6-30-2011

Signature/Date of Responsible K-Basin Manager: [signature] 6/30/2011

Comments:

K-Basin Occurrence Report

Title: U-metal RPD exceeds 20%		**Date: 5-19-11**
Unique Identifier: 52578-05-19-11		
Primary Person Identifying Issue: C. Delegard		
Distribution: Email and posting on K-Basin Share-Point	Clark Carlson	
	Deborah Coffey	Andy Schmidt
	Sandy Fiskum	Rick Shimskey
	Karl Pool	Truc Trang Le, Project file for **ASR 8824**
Date Submitted to K-Basin Records:	7-28-2011	

Occurrence Description: ASR 8824:

The duplicate RPD for results for samples TI008-ME (11-0792-107-S) and TI008-MF (11-0792-107-D) was 86%, exceeding the acceptance criteria of 20%. Both the sample and duplicate sample have U-metal concentrations greater than 0.1 weight %, TI008-ME (0.426 wt. %) and TI008-MF (0.170 wt. %).

Impact of Occurrence:

The sample masses used for the U-metal analyses were 2.49g for the sample and 2.60g for the duplicate. Both the sample and duplicate sample have U-metal concentrations greater than 0.1 weight %, TI008-ME (0.426 wt. %) and TI008-MF (0.170 wt. %). Both results are above the process quantitation limit of 0.03 weight% U. The variability in the results is likely due to sample inhomogeniety.

Significant Condition Adverse to Quality? Yes _____ No X

Evaluated by: _____ **Date:** 7/28/11

Further Disposition: ☒ **No** ☐ **Yes** **If yes,** ☐ **Nonconformance?** ☐ **Deficiency?**

Date Occurrence Closed: 7-28-2011

Signature/Date of Responsible QE: Deborah Coffey 7/28/2011

Signature/Date of Responsible K-Basin Manager: _____ 7/28/2011

Comments:

Appendix D

ASR 8878 and ASR 8919 Results

Appendix D

ASR 8878 and ASR 8919 Results

Appendix D contains the signed Analytical Services Request (ASR) 8878 and Special Instructions for the undissolved solids (UDS) of selected subsamples from the SCS-CON-210The UDS were obtained following the acid digestion conducted according to PNL-ALO-129, Rev. 0, *HNO_3-HCl Acid Extraction of Solids Using a Dry-Block Heater*. Weighed UDS samples were fused and the fusion flux dissolved according to procedure PNL-ALO-115, Rev. 1, *Solubilization of Metals from Solids Using a KOH-KNO_3 Fusion*. The resulting solutions were analyzed for gross alpha, alpha isotopes by alpha energy analysis (AEA screen), and inductively coupled plasma-optical emission spectrometry (ICP-OES).

Appendix D also includes the signed ASR 8919 Request for the GEA analysis of the spherical (resin bead fraction) and non-spherical (residual solids fraction) particle fractions separated from the merge of −2,000-µm /+500-µm (MCC) sieve fraction samples (TI008-MK and TI008-ML).

All analytical reports generated by the Analytical Support Operations (ASO) laboratory in response to these ASRs are provided in Appendix D. Analytical reports provide sample results, associated uncertainties, and a discussion of sample processing, quality control results, compliance with QC sample requirements, and data limitations. An Occurrence Report was also provided. These was prepared when issues were identified, and resolution was required while work was in process.

The following table of contents shows that pagination specific to Appendix D was applied to the ASRs, the analytical reports, and Occurrence Report. This pagination is sequential and consistent (e.g., page D.#).

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ASR 8878 Alpha Spectrometry—Total Alpha Report Analysis Report..... D.26
Radiochemistry Data Report—Sample Results for ASR 8878..... D.30
Occurrence Report 52578-08-31-11 ..... D.33
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ASR 8919 Gamma Energy Analysis Narrative Report..... D.36

Analytical Service Request (ASR)

ASR-FY2006-ASO Rev. 0.doc

(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: SK. Fiskum
Print Name: SK Fiskum
Phone: 375-5677 MSIN: P7-25

Project Number: 52578
Work Package: #41215 / #41216

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: August 16, 2011
- ♦ **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 Special Instructions

- ♦ **Requested Analytical Work Completion Date:** July 13, 2011
  (Note: Priority rate charge for < 10 business day turn-around time)
- ♦ **Negotiated Commitment Date:** 7/29/11
  (*To be completed by ASO Lead*) KNP

Waste Designation Information

- ♦ **Sample Information Check List Attached?** ☒ No ☐ Yes
  If no, Reference Doc Attached: _____
  or, Previous ASR Number: 8798
  or, Previous RPL Number: _____

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

Send Report To: SK Fiskum MSIN P7-25 LA Snow P7-25
MS Fountain MSIN P7-25

Additional or Special Instructions See attached

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

Date Delivered: 6/16/11
Delivered By (optional): SK Fiskum
Time Delivered (optional): _____
Group ID (optional): _____
CMC Waste Sample? ☒ No ☐ Yes

Received By: T Trang-Le

ASR Number: 8878 Rev.: 00
RPL Numbers: (11-1279) → (11-1290)
(first and last)

ASO Work Accepted By: KN Pool **Signature/Date:** Kurt N. Pool 6/22/11

Analytical Services Request (ASR)

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

Provide Analytes of Interest and Required Detection limits - ☐ **Below** ☐ **Attached**

ASO Staff Use Only				ASO Staff Use Only	
RPL Number	Client Sample ID	Sample Description (& Matrix if varies)	Analyses Requested	**Test**	**Library**
11-1279	TI040-0419S-1b			Fusion-115	
11-1280	TI040-0419D-2b			ICP	lab Analysis
11-1281	TI040-0420S-2c			Alpha	Radchem
11-1282	TI040-0420D-1c			Alpha-AEA	↓
	TI040-0420D-2c This is a batch duplicate				
11-1283	TI040-0421S-2b				
11-1284	TI040-0421D-2b	**KW-Basin Core 210 residual dried solids from 129 digest**	1) Fusion - 115 (Lab 420) a) ICP/OES (Al, Ca, Fe, Si, S, Zr, U) b) Gross Alpha c) Total Alpha - AEA		
11-1285	TI040-0422S-1c				
11-1286	TI040-0422D-1c				
11-1287	TI040-0793S-1a				
11-1288	TI040-0793D-1b				
	TI040-0793D-2b This is a batch duplicate				
11-1289	TI040-0792S-1c				
11-1290	TI040-0792D-1c				

Note: Samples are being provided as pre-aliquoted by the client. The masses of pre-aliquoted sample are included in the ASR Special Instructions. Capture weights of sample transferred to the crucible as the weight used for the fusion.

Special Instructions for K-Basin SCS-CON-210 Core Sample
Undissolved Sludge:
Fusion Preparation and Analysis
Project 52578

Cognizant Scientist: Sandra R. Fiskum Date: 6/16/11
SK Fiskum

Project Manager: Date: 6/16/2011
CD Carlson

Project Quality Engineer: D. S. Coffey Date: 6-16-2011
DS Coffey

ASO Lead: Date: 6/16/11
KN Pool

STP Concurrence: Date: 6/16/11
WW Rutherford

Special Instructions for K-Basin SCS-CON-210 Core Sample Undissolved Sludge: Fusion Preparation and Analysis Project 52578

Cognizant Scientists:
SK Fiskum, 375-5677
MS Fountain, 375-5311
CD Carlson, 375-5334

Core and composite materials containing liquid and sludge from K West (KW) Basin Engineered Container 210 (i.e., SCS-CON-210) were processed for analysis using the ASO procedure PNL-ALO-129 acid digestion according to ASR 8798 (core samples) and ASR 8824 (container composite samples). Significant dry mass fraction of material did not dissolve. The composition of these undissolved solids (UDS) is of interest. The UDS are presented for fusion-aided dissolution followed by ICP-OES analysis, gross alpha, and alpha energy analysis (AEA) screen. The samples are provided in masses suitable for direct fusion processing (splitting conducted per Test Instruction 52578-TI040); no additional subsampling should be attempted due to the heterogenous nature of the material. The sample batch duplicates (2) have already been aliquoted. All samples probably contain significant sand and organic ion exchange resin beads.

A cross-reference of sample IDs and sample mass is provided in Table 1.

Table 1. Associated Core Sample ID, Sample ID, ASO/RPL ID, and Sample Mass Cross-Reference

Associated Core Sample ID	Sample ID	ASO/RPL ID		Sample Mass, g
KW210-A1	TI040-0419S-1b	11-1279		0.1589
	TI040-0419D-2b	11-1280		0.1559
KW210-A4	TI040-0420S-2c	11-1281		0.1331
	TI040-0420D-1c	11-1282		0.1116
	TI040-0420D-2c	11-1282D	Batch duplicate	0.1140
KW210-B1	TI040-0421S-2b	11-1283		0.1554
	TI040-0421D-1b	11-1284		0.1698
KW210-B3	TI040-0422S-1c	11-1285		0.1238
	TI040-0422D-1c	11-1286		0.1360
KW210-CC -500 µm	TI040-0793S-1a	11-1287		0.1795
	TI040-0793D-1b	11-1288		0.1202
	TI040-0793D-2b	11-1288D	Batch duplicate	0.1124
KW210-CC +500/-2000 µm	TI040-0792S-1c	11-1289		0.1485
	TI040-0792D-1c	11-1290		0.1967

The following work packages apply

- Sample processing by PNL-ALO-115 fusion and ICP-OES analysis: H41215
- Radiochemistry analysis: H41216

Analytes of interest and targeted detection limits are provided in Table 3; the QAPjP/SAP KBC-33786, Rev. 2 did not delineate these follow-on analyses for UDS and as such, MDLs were not specified. The quality control (QC) sample acceptance criteria are provided in Table 4; these criteria are consistent with Hanford Analytical Services Quality Assurance Requirements (HASQARD) requirements. Although a 6-month holding time was implemented for the original sample ICP-OES analysis, a holding time does not apply to these undissolved solids.

Chemical Analysis Samples

Fusion

Preparation
Fusion of the solids is to be conducted according to the ASO procedure, PNL-ALO-115, Rev. 1, *Solubilization of Metals from Solids Using a KOH-KNO_3 Fusion*. Report observations associated with the dissolution processing.

ICP-OES

ICP-OES Analysis
Fusion-prepared aliquots shall be analyzed at the ICP-OES workstation according to RPG-CMC-211, Rev. 3, *Determination of Elemental Composition by Inductively Coupled Argon Plasma Optical Emission Spectrometry (ICP-OES)*.

ICP-OES Reporting Units
Report solids sample analyte concentrations as µg/g.

Radiochemistry

Preparations and Analysis
Fusion-prepared aliquots shall be prepared according to procedures identified 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	Mounting	Counting/Analysis
Gross Alpha	RPG-CMC-4001, Rev. 1, *Source Preparation for Gross Alpha and Gross Beta Analysis*	RPG-CMC-408, Rev. 2, *Total Alpha and Beta Analysis*
AEA Screen	RPG-CMC-496, Rev. 0, *Coprecipitation Mounting of Actinides for Alpha Spectroscopy*	RPG-CMC-422, Rev. 2 *Solutions Analysis: Alpha Spectrometry*

Radiochemistry Reporting Units

Report solids sample analyte concentrations as µCi/g.

Archive of Prepared Chemical Analysis Samples

The fusion preparations will result in a ~50- to 100-mL volume. This solution will be apportioned to the laboratory as needed to conduct work-station-specific analyses. At the time the fusion preparations are apportioned to the analysis work locations, ~15-mL aliquot from each preparation should be collected and and reserved as an archive sample. The vials need to be labeled with the following information: date, RPL-ID, sample ID, matrix, and hazard. The remaining portion of the fusion preparations may be disposed of.

Quality Control

All work is required to be conducted to the requirements of the HASQARD, Rev. 3 document. Performing work to the requirements of the ASO QA Plan (ASO-QAP-001, Rev. 7) 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.

NOTE: The balance checks at the fumehood workstations shall be checked per the normal process where one calibration verification check mass is measured daily when then balance is used (PNL-ASO-052, Rev. 1).

Matrix preparative analysis QC is to include a preparation blank (PB), sample/sample duplicate (2 sample duplicates provided), and an LCS (Montana Soil, SRM 2710) as indicated in Table 4. The BS and MS for the AEA will be generated after fusion on a sample split, i.e., post-fusion spikes.

The ASO is responsible for preparing the PB and LCS. The LCS, and post-fusion BS/MS sample QC acceptance criteria are provided in Table 4.

Note: The ASO staff must inform/report QC failures promptly to the Cognizant Scientists with copy to CD Carlson and the K Basin QE (Deborah Coffey), thus enabling PNNL to fulfill its commitment to the client for prompt notification of such failures. The impact of any QC 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 affected samples in the processing batch must be re-prepared and re-analyzed for the failed analytes, availability of samples permitting.
3. In the case of multi-elemental methods (ICP-OES), isolated QC failure(s) may be communicated to SK Fiskum, MS Fountain, CD Carlson and the K Basin QE by the ASO Lead for an assessment of the impact on data interpretation.

QAPjP/SAP KBC-33786, Rev. 2 does not apply precision criteria to analysis of the UDS, therefore the QAPjP/SAP DQOs for sample duplicate pairs do not apply. Precision will be calculated in terms of relative percent difference (RPD) for two sample split duplicate pairs (11-1282/11-1282D and 11-1288/11-1288D).

Reporting

The analytical data report shall be prepared in accordance with procedure PNL-ASO-058, Rev. 0.2, *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. 0.2.

Data Report **Cover Page** Contents:

- Header identifying name/address of laboratory
- Client Name:
- Project #/WP #(s):
- ASR #(s):
- 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 and title (sample processing and analysis) and revision number
- M&TE (serial number or property number):
- Reference Date (when applicable):
- Client ID and 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
- Analysis dates
- Deviations from the written procedure, if any
- Method specific Sample Preparation, Separation, Mounting and Counting Methods as applicable
- Reporting Basis/Units
- QC Criteria/Results
- Limitations of the Data Interferences/Resolution Uncertainty
- Comments

Data Summary Contents:

- Client ID
- RPL ID with full ASO-related suffixes (e.g., 115-S)
- Results and result uncertainties

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 lifetime 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. Target Method Detection Limits for Solids

Analyte	Solids	Analysis Method
	µCi/g	
Total Alpha	1.0E-03	Gross Alpha
^{241}Am / ^{238}Pu	1.0E-03	AEA Screen
$^{239+240}Pu$	1.0E-03	
Identify and quantify any other spectral peak		
	µg/g	
Al	3.0E+03	ICP-OES[(a)]
Ca	3.0E+03	
Fe	1.0E+03	
S	1.0E+03	
Si	3.0E+03	
Zr	2.0E+02	
U	1.0E+03	

(a) Selected analytes were measured in the parent sample at >5,000 µg/g (dry mass basis).
(b) The targeted MDL is based on the fusion flux matrix adjusted instrument detection limit (IDL) multiplied by a nominal process factor of 5,000. The MDL is intended to be a guide to the analytical staff when selecting aliquot sizes and conducting dilutions. It is understood that matrix effects may confound the achieved MDL.

Table 4. Analytical Quality Control Sample Acceptance Criteria

Analyte	Analytical Technique	LCS % Recovery[a]	
		Pre-digestion spike	
Al, Fe, Si, Ca in Montana I Soil SRM 2710	ICP-OES	80 - 120%	
Analyte	**Analytical Technique**	**BS % Recovery[a]**	**MS % Recovery[b]**
		Post-digestion spike	
Gross Alpha (spike material is ^{239}Pu)	Alpha scintillation	80 - 120%	75 - 125%
AEA Screen (spike material is ^{239}Pu)	AEA	80 - 120%	75 - 125%

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. 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.

References

52578-TI040, Rev. 0, Processing of the Undissolved Solids Samples from the K West Basin Core Samples Collected from SCS-CON-210 for Additional Analysis

ASO-QAP-001, Rev. 7, *Analytical Support Operations Quality Assurance Plan*

PNL-ASO-052, Rev. 1, *Balance Performance Checks*

PNL-ASO-058, Rev. 0.2, *ASO Data Reporting*

PNL-ALO-115, Rev. 1, *Solubilization of Metals from Solids Using a KOH-KNO_3 Fusion*

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

RPG-CMC-211, Rev. 3, *Determination of Elemental Composition by Inductively Coupled Argon Plasma Optical Emission Spectrometry (ICP-OES)*

RPG-CMC-408, Rev. 2, *Total Alpha and Beta Analysis*

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

RPG-CMC-496, Rev. 0, *Coprecipitation Mounting of Actinides for Alpha Spectroscopy*

RPG-CMC-4001, Rev. 1, *Source Preparation for Gross Alpha and Gross Beta Analysis*

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

KBC-33786, Rev. 2, Baker RB, JL Westcott, TL Welsh, JA Pottmeter, and AJ Schmidt. 2009*Quality Assurance Project Plan/Sampling and Analysis Plan for Sludge in the KW Engineered Containers*. CH2M Hill Plateau Remediation, Company, Richland, Washington.

Battelle PNNL/RPL/Inorganic Analysis ... ICP-OES Analysis Report
PO Box 999, Richland, Washington 99352

Project / WP#: 52578 / H41215
ASR#: 8878
Client: S. Fiskum
Total Samples: 14 (solids)

	First	Last
RPL#:	11-1279	11-1290
Client ID:	TI040-0419S-1b	TI040-0792D-1c
Sample Preparation: PNL-ALO-115, Rev. 1.1, "Solubilization of Metals from Solids using a $KOH-KNO_3$ Fusion", 7/18/11 (SRPL/ld).		

Procedure: RPG-CMC-211, Rev. 3, "Determination of Elemental Composition by Inductively Coupled Argon Plasma Optical Emission Spectrometry (ICP-OES)",.

Analyst: J. Carter

Analysis Date (File): 07-29-2011 (C0399)
08-01-2011 (C0400)

See Chemical Measurement Center 98620 file: ICP-325-405-3
(Calibration and Maintenance Records)

M&TE Number: N827583 (PerkinElmer 5300DV ICP-OES, S/N 077N5122002)
WD66158 (Sartorius ME414S Balance, S/N 224066373)

BM Ellen 8/18/11
Preparer

C Carlson 8/26/11
Review and Concur

Battelle PNNL/RPL/Inorganic Analysis ... ICP-OES Analysis Report

Fourteen samples were provided for ICP-OES analysis under Analytical Service Request (ASR) 8878. The samples were residual dried solids from an earlier digestion preparation. Each of the samples was processed in the Sample Receiving and Preparation Laboratory (SRPL) following RPL procedure PNL-ALO-115 using ~0.1 gram of sample with dilution to a final volume of 100 mL.

Analytes of interest (AOIs) were specified in the ASR, and are listed in the upper section of the attached ICP-OES Data Report. The quality control (QC) results for these AOIs have been evaluated and are presented below. Analytes other than AOIs are reported in the bottom section of the report, but have not been fully evaluated for QC performance. The results for Sr are not reported because of issues with the instrument calibration for that analyte.

The results are reported on a mass basis (µg/g) and have been adjusted for laboratory processing factors and instrument dilutions. Minimum Method Detection Limit (MDL) values were specified in the ASR. MDL levels were met for all AOIs.

Calibration of the ICP-OES was done following the manufacturer's recommended calibration procedure and using multi-analyte custom standard solutions traceable to the National Institute of Standards and Technology (NIST). Midrange calibration verification standards (MCVA and MCVB) were used to verify acceptance of the two-point calibration curves obtained for each analyte, and also used for continuing calibration verification.

The controlling documents were ASO-QAP-001 and ASR-8878 Special Instructions. Instrument calibrations, QC checks and blanks (e.g., ICV/ICB, CCV/CCB, LLS, ICS), blank spike, post spike, duplicates, and serial dilution were conducted during the analysis run. The blank spike was prepared using NIST SRM-2710 (Montana Soil).

Preparation Blank (PB):

A preparation blank (reagents only) was prepared for the extraction process. The concentrations of all AOIs were within the acceptance criteria of ≤EQL (estimated quantitation level) or less than ≤5% of the concentration in the sample.

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

An LCS was prepared for the extraction process. Recovery values are listed for all analytes included in the spike that were measured at or above the EQL. Recovery values for AOIs meeting this requirement ranged from 95 to 107%, and were within the acceptance criterion of 80% to 120%.

Matrix-Spiked Sample:

No matrix spike was prepared for the extraction process.

Duplicate Relative Percent Difference (RPD):

Duplicates were prepared for two of the samples (11-1282 and 11-1288). RPDs are listed for all analytes that were measured at or above the EQL. RPDs for AOIs meeting this requirement ranged from 2.6 to 20.6%. Per the ASR, no acceptance criterion are specified for the duplicate RPDs.

Post-Spike/Analytical Spike Sample (A Component):

Post-spike (A Component) analyses were performed on Samples 11-1279 and 11-1285. Recovery values are listed for all analytes in the spike that were measured at or above the EQL, and that had a spike concentration ≥25% of that in the sample. Recovery values for AOIs meeting these requirements ranged from 99 to 119%, and were within the acceptance criterion of 80% to 120%.

Post Spike/Analytical Spike Sample (B Component):

Post-spike (B Component) analyses were performed on Samples 11-1279 and 11-1285. Recovery values are listed for all analytes in the spike that were measured at or above the EQL, and that had a spike concentration ≥25% of that in the sample. Recovery values for AOIs meeting these requirements ranged from 96 to 107%, and were within the acceptance criterion of 80% to 120%.

Serial dilution:

Five-fold serial dilution was performed on Samples 11-1279 and 11-1285. Percent differences (%Ds) are listed for all analytes that had a concentration at or above the EQL in the diluted sample. The %Ds for AOIs meeting this requirement ranged from 0.5 to 6.1%, and were within the acceptance criterion of ≤10%.

Other QC:

Silicon was outside the acceptance range for two of the CCV's, and was the result of carry-over from a previous HF rinse applied to the system nebulizer and quartz spray chamber as part of in-process preventative maintenance. After a suitable rinse-out period, the Si fell within the acceptance criterion. All other instrument-related QC tests for the AOIs passed within the appropriate acceptance criteria.

Comments:

1) The "Final Results" have been corrected for all laboratory dilutions performed on the samples during processing and analysis, unless specifically noted.
2) Instrument detection limits (IDL) and estimated quantitation limits (EQL) shown are for acidified water and/or fusion flux matrices as applicable. Detection limits for other matrices may be determined if requested. Method detection limits (MDL) can be estimated by multiplying the IDL by the "Multiplier". The estimated quantitation limit (EQL) for each concentration value can be obtained by multiplying the EQL by the "Multiplier".
3) Routine precision and bias is typically ±15% or better for samples in dilute, acidified water (e.g. 2% v/v HNO_3 or less) at analyte concentrations > EQL up to the upper calibration level. This also presumes that the total dissolved solids concentration in the sample is less than 5000 µg/mL (0.5 per cent by weight). Note that bracketed values listed in the data report are within the MDL and the EQL, and have potential uncertainties greater than 15%. Concentration values < MDL are listed as "- -". Note, that calibration and QC standards are validated to a precision of ±10%.

4) Absolute precision, bias and detection limits may be determined on each sample if required by the client. The maximum number of significant figures for all ICP measurements is two.
5) Analytes included in the spike A component (for the AS/PS) are; Ag, Al, As, B, Ba, Be, Bi, Ca, Cd, Co, Cr, Cu, Fe, K, Li, Mg, Mn, Mo, Na, Ni, P, Pb, Sb, Se, Si, Sn, Sr, Ta, Ti, Tl, V, W, Y, Zn, and Zr. Analytes included in the spike B component are; Ce, Dy, Eu, La, Nd, Pd, Rh, Ru, S, Te, Th, and U.

Battelle PNNL/RPG/Inorganic Analysis ... ICPOES Data Report

		Run Date >	7/29/2011	7/29/2011	7/29/2011	7/29/2011
		Multiplier >	3518.6	3148.6	3205.1	3782.1
		RPL/LAB >	BLK-1279 @ 5x	11-1279 @ 5x	11-1280 @ 5x	11-1281 @ 5x
Instr. Det. Limit (IDL)	Est. Quant. Limit (EQL)	Client ID >	Prep Blank	TI040-0419S 1b	TI040-0419D 2b	TI040-0420S 2c
(µg/mL)	(µg/mL)	(Analyte)	(µg/g)	(µg/g)	(µg/g)	(µg/g)
0.4064	4.064	Al	--	20,100	16,400	22,200
0.4345	4.345	Ca	--	[3,400]	[2,100]	[2,700]
0.1614	1.614	Fe	--	16,500	16,000	7,080
0.1356	1.356	S	--	16,400	13,900	16,700
0.4424	4.424	Si	--	276,000	268,000	350,000
0.0088	0.176	U	--	7,840	7,370	1,920
0.0109	0.109	Zr	--	694	579	623
Other Analytes						
0.0014	0.014	Ag	[10.0]	--	--	--
0.0730	0.730	As	--	--	--	--
0.0046	0.046	B	[27]	[52]	[110]	[42]
0.0078	0.078	Ba	--	656	511	840
0.0001	0.001	Be	--	[0.32]	[0.22]	--
0.0120	0.120	Bi	--	--	--	--
0.0022	0.022	Cd	--	--	--	[63]
0.0066	0.066	Ce	--	--	--	--
0.0027	0.027	Co	[32]	[14]	--	--
0.0016	0.016	Cr	[6.0]	[40]	[35]	[19]
0.0543	0.543	Cu	--	--	--	--
0.0014	0.014	Dy	--	--	--	--
0.0004	0.004	Eu	--	[2.1]	[2.1]	17.9
0.0000	0.000	K	na	na	na	na
0.0017	0.017	La	--	174	232	369
0.0003	0.003	Li	--	9.45	--	--
0.0150	0.150	Mg	--	1,540	1,100	1,090
0.0166	0.166	Mn	--	[92]	[62]	--
0.0063	0.063	Mo	--	--	--	--
2.0840	20.840	Na	--	--	--	--
0.0041	0.041	Nd	--	--	--	--
0.0000	0.000	Ni	na	na	na	na
0.0690	0.690	P	--	--	--	--
0.0603	0.603	Pb	--	--	--	--
0.0052	0.052	Pd	--	--	--	--
0.0098	0.098	Rh	--	--	--	--
0.0066	0.066	Ru	--	--	--	--
0.0340	0.340	Sb	--	--	--	--
0.2400	2.400	Se	--	--	--	--
0.0240	0.240	Sn	--	--	--	--
0.0019	0.019	Sr	--	89.8	78.5	111
0.0200	0.200	Ta	--	--	--	--
0.0160	0.160	Te	--	--	[60]	[65]
0.0028	0.028	Th	--	--	--	--
0.0035	0.035	Ti	--	2,370	1,940	716
0.0460	0.460	Tl	--	--	--	--
0.0007	0.021	V	[3.5]	[8.6]	[6.7]	[15]
0.0190	0.190	W	--	--	--	--
0.0002	0.002	Y	--	[4.5]	[2.2]	[2.7]
0.1315	1.315	Zn	--	--	--	9,160

1) "--" indicates the value is < MDL. The method detection limit (MDL) = IDL times the "multiplier" near the top of each column. The estimated sample quantitation limit = EQL (in Column 2) times the "multiplier". Overall error for values ≥ EQL is estimated to be within ±15%.

2) Values in brackets [ ] are ≥ MDL but < EQL, with errors likely to exceed 15%.

na = not applicable; KOH flux and Ni crucible or Na_2O_2 flux and Zr crucible for fusion preparations, or Si for HF assisted digests.

ASR 8878 Part 1 Final from C0399 S. Fiskum (ASR-8878).XLS

Battelle PNNL/RPG/Inorganic Analysis ... ICPOES Data Report

QC Performance 7/29/2011

Criteria >	≤ 20%	80%-120%	80%-120%	80%-120%	80%-120%	≤ 10%
QC ID >	Dup (none)	LCS/BS	MS (none)	11-1279 + AS-A	11-1279 + AS-B	11-1279 5-fold Serial Dil
Analytes	RPD (%)	%Rec	%Rec	%Rec	%Rec	%Diff
Al		95		104		
Ca		[99]		100		
Fe		97		99		
S					98	
Si		107		101		0.5
U					96	6.1
Zr				102		
Other Analytes						
Ag				91		
As				100		
B				99		
Ba		93		92		
Be				101		
Bi				92		
Cd				106		
Ce					96	
Co				97		
Cr				102		
Cu		99		106		
Dy					100	
Eu					97	
K		na	na	na	na	na
La					100	
Li				102		
Mg		96		101		
Mn		100		100		
Mo				97		
Na				97		
Nd					94	
Ni		na	na	na	na	na
P				100		
Pb		97		100		
Pd					89	
Rh					99	
Ru					93	
Sb				96		
Se				101		
Sn				96		
Sr		97		103		
Ta				99		
Te					98	
Th					95	
Ti		93		100		2.5
Tl				91		
V				97		
W				101		
Y		84		99		
Zn		100		100		

Shaded results are outside the acceptance criteria.

nr = spike concentration less than 25% of sample concentration. Matrix effects can be assessed from the serial dilution.

na = not applicable; KOH flux and Ni crucible or Na_2O_2 flux and Zr crucible for fusion preparations, or Si for HF assisted digests.

Battelle PNNL/RPG/Inorganic Analysis ... ICPOES Data Report

		Run Date >	8/1/2011	8/1/2011	8/1/2011	8/1/2011	8/1/2011	8/1/2011	8/1/2011
		Multiplier >	4533.1	4456.3	3217.5	2944.6	4061.7	3684.6	2796.4
		RPL/LAB >	11-1282 @ 5x	DUP-1282 @ 5x	11-1283 @ 5x	11-1284 @ 5x	11-1285 @ 5x	11-1286 @ 5x	11-1287 @ 5x
Instr. Det. Limit (IDL)	Est. Quant. Limit (EQL)	Client ID >	TI040-0420D 1C	TI040-0420D 2c	TI040-0421S 2b	TI040-0421D 1b	TI040-0422S 1c	TI040-0422D 1c	TI040-0793S 1a
(µg/mL)	(µg/mL)	(Analyte)	(µg/g)	(µg/g)	(µg/g)	(µg/g)	(µg/g)	(µg/g)	(µg/g)
0.4064	4.064	Al	[18,000]	21,800	[2,800]	[1,900]	[8,300]	[9,200]	25,000
0.4345	4.345	Ca	[3,300]	[2,900]	--	--	--	--	[3,000]
0.1614	1.614	Fe	11,500	9,650	33,700	34,000	27,100	22,800	8,360
0.1356	1.356	S	15,900	16,900	25,800	14,300	19,000	14,800	3,880
0.4424	4.424	Si	316,000	355,000	38,200	22,100	127,000	161,000	309,000
0.0088	0.176	U	3,310	2,970	11,000	16,000	15,200	12,800	1,610
0.0109	0.109	Zr	847	--	--	--	--	--	[190]
Other Analytes									
0.0014	0.014	Ag	--	--	--	--	[6.2]	--	--
0.0730	0.730	As	--	--	--	--	--	--	--
0.0046	0.046	B	[25]	--	[32]	[19]	[51]	--	[44]
0.0078	0.078	Ba	737	785	[68]	[43]	363	467	947
0.0001	0.001	Be	--	[0.50]	--	[0.19]	--	--	[0.39]
0.0120	0.120	Bi	--	--	--	--	--	--	--
0.0022	0.022	Cd	--	--	--	--	--	--	--
0.0066	0.066	Ce	--	--	--	--	--	--	--
0.0027	0.027	Co	--	[15]	[37]	[43]	[34]	[21]	[24]
0.0016	0.016	Cr	72.9	[31]	[38]	47.5	[39]	[41]	139
0.0543	0.543	Cu	--	--	--	--	--	--	--
0.0014	0.014	Dy	[9.6]	--	--	--	--	--	[4.9]
0.0004	0.004	Eu	[3.2]	[1.8]	[1.8]	[1.7]	--	[1.6]	--
0.0000	0.000	K	na	na	na	na	na	na	na
0.0017	0.017	La	670	--	--	--	--	--	--
0.0003	0.003	Li	[3.5]	[1.2]	--	--	--	--	[1.4]
0.0150	0.150	Mg	1,410	947	[350]	[140]	[120]	[140]	2,210
0.0166	0.166	Mn	[110]	[95]	--	[110]	--	--	[130]
0.0063	0.063	Mo	--	--	--	--	--	--	--
2.0840	20.840	Na	--	--	--	--	--	--	--
0.0041	0.041	Nd	--	--	--	--	--	--	--
0.0000	0.000	Ni	na	na	na	na	na	na	na
0.0690	0.690	P	--	--	--	--	--	--	--
0.0603	0.603	Pb	--	--	--	--	--	--	--
0.0052	0.052	Pd	--	--	--	--	--	--	--
0.0098	0.098	Rh	--	[48]	--	--	--	--	--
0.0066	0.066	Ru	[35]	--	--	--	--	--	--
0.0340	0.340	Sb	--	--	--	--	--	--	--
0.2400	2.400	Se	--	--	--	--	--	--	--
0.0240	0.240	Sn	--	--	--	--	--	--	--
0.0019	0.019	Sr	(a)	(a)	(a)	(a)	(a)	(a)	(a)
0.0200	0.200	Ta	--	--	--	--	--	--	--
0.0160	0.160	Te	--	--	--	--	--	--	--
0.0028	0.028	Th	--	--	--	--	--	--	--
0.0035	0.035	Ti	1,470	1,010	345	385	351	455	3,540
0.0460	0.460	Tl	--	--	--	--	--	--	--
0.0007	0.021	V	[21]	[18]	[11]	[2.1]	[7.0]	--	[11]
0.0190	0.190	W	--	--	--	--	--	--	--
0.0002	0.002	Y	[6.6]	[4.4]	[1.3]	--	[0.81]	--	13.0
0.1315	1.315	Zn	--	--	--	--	--	--	--

1) "--" indicates the value is < MDL. The method detection limit (MDL) = IDL times the "multiplier" near the top of each column. The estimated sample quantitation limit = EQL (in Column 2) times the "multiplier". Overall error for values ≥ EQL is estimated to be within ±15%.

2) Values in brackets [ ] are ≥ MDL but < EQL, with errors likely to exceed 15%.

na = not applicable; KOH flux and Ni crucible or Na_2O_2 flux and Zr crucible for fusion preparations, or Si for HF assisted digests.

(a) = Sr results are not reported (calibration issues).

Battelle PNNL/RPG/Inorganic Analysis ... ICPOES Data Report

Run Date >	8/1/2011	8/1/2011	8/1/2011	8/1/2011
Multiplier >	4223.0	4537.2	3328.9	2510.0
RPL/LAB >	11-1288 @ 5x	DUP-1288 @ 5x	11-1289 @ 5x	11-1290 @ 5x
Client ID >	TI040-0793D 1b	TI040-0793D 2b	TI040-0792S 1c	TI040-0792D 1c
(Analyte)	(µg/g)	(µg/g)	(µg/g)	(µg/g)
Al	26,200	26,900	[11,000]	12,900
Ca	[3,100]	[3,200]	--	[2,200]
Fe	9,780	8,750	20,900	23,000
S	[3,600]	[3,000]	21,000	20,300
Si	375,000	405,000	189,000	189,000
U	1,270	1,040	8,320	9,910
Zr	[240]	[190]	[37]	--
Ag	--	--	[4.8]	[4.8]
As	--	--	--	--
B	[31]	--	--	--
Ba	966	1,050	423	499
Be	[0.57]	[0.36]	[0.23]	[0.30]
Bi	--	--	--	--
Cd	--	--	--	--
Ce	--	--	--	--
Co	[26]	[36]	[15]	[16]
Cr	[67]	84.0	1,290	[34]
Cu	--	--	--	--
Dy	--	--	--	[4.9]
Eu	--	--	--	[1.2]
K	na	na	na	na
La	--	--	--	--
Li	[1.2]	--	[2.1]	--
Mg	2,470	2,110	551	728
Mn	[140]	[140]	727	[52]
Mo	--	--	--	--
Na	--	--	--	--
Nd	--	--	--	--
Ni	na	na	na	na
P	--	--	--	--
Pb	--	--	--	--
Pd	--	--	--	--
Rh	--	--	--	--
Ru	--	--	--	--
Sb	--	--	--	--
Se	--	--	--	--
Sn	--	--	--	--
Sr	(a)	(a)	(a)	(a)
Ta	--	--	--	--
Te	--	--	--	--
Th	--	--	--	--
Ti	3,390	3,060	1,190	1,190
Tl	--	--	--	--
V	[15]	[16]	[19]	[14]
W	--	--	--	--
Y	[5.3]	[6.4]	[3.5]	[1.4]
Zn	--	--	--	--

Battelle PNNL/RPG/Inorganic Analysis ... ICPOES Data Report

QC Performance 8/1/2011

Criteria >	≤ 20%	≤ 20%	80%-120%	80%-120%	80%-120%	80%-120%	≤ 10%
QC ID >	11-1282 Dup	11-1288 Dup	LCS/BS (none)	MS (none)	11-1285 + AS-A	11-1285 + AS-B	11-1285 5-fold Serial Dil
Analytes	RPD (%)	RPD (%)	%Rec	%Rec	%Rec	%Rec	%Diff
Al		2.6			106		
Ca					107		
Fe	17.8	11.1			113		
S	6.0					107	
Si	11.8	7.7			119		1.7
U	10.9	20.6				99	1.2
Zr					111		
Other Analytes							
Ag					103		
As					115		
B					107		
Ba	6.4	8.1			109		
Be					108		
Bi					104		
Cd					113		
Ce						100	
Co					111		
Cr					110		
Cu					116		
Dy						103	
Eu						103	
K	na	na	na	na	na	na	na
La						103	
Li					104		
Mg	39.4	15.5			108		
Mn					109		
Mo					109		
Na					105		
Nd						99	
Ni	na	na	na	na	na	na	na
P					111		
Pb					113		
Pd						96	
Rh						97	
Ru						102	
Sb					110		
Se					113		
Sn					106		
Sr	(a)	(a)	(a)	(a)	(a)	(a)	(a)
Ta					113		
Te						105	
Th						105	
Ti	37.8	10.3			111		
Tl					102		
V					104		
W					111		
Y					107		
Zn					116		

Shaded results are outside the acceptance criteria.

nr = spike concentration less than 25% of sample concentration. Matrix effects can be assessed from the serial dilution.

na = not applicable; KOH flux and Ni crucible or Na_2O_2 flux and Zr crucible for fusion preparations, or Si for HF assisted digests.

(a) = Sr results are not reported (calibration issues).

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

Gross Alpha

Project / WP#:	52578/H41216
ASR#:	8878
Client:	S. Fiskum
Total # of Samples:	14

Sample IDs	RPL Number		Sample IDs	RPL Number
TI040-0419S-1b	11-1279-115-S		TI040-0422S-1c	11-1285-115-S
TI040-0419D-2b	11-1280-115-S		TI040-0422D-1c	11-1286-115-S
TI040-0420S-2c	11-1281-115-S		TI040-0793S-1a	11-1287-115-S
TI040-0420D-1c	11-1282-115-S		TI040-0793D-1b	11-1288-115-S
TI040-0420D-2c	11-1282-115-D		TI040-0793D-2b	11-1288-115-D
TI040-0421S-2b	11-1283-115-S		TI040-0792S-1c	11-1289-115-S
TI040-0421D-1b	11-1284-115-S		TI040-0792D-1c	11-1290-115-S

Analysis Type:	Gross Alpha
Sample Processing Prior to Radiochemical Processing/Analysis	☐ None ☐ Digested as per PNL-ALO-129, Rev. 0, *HNO_3-HCl Acid Extraction of Solids Using a Dry Block Heater* ☒ Fusion as per PNL-ALO-115, Rev. 1.1, *Solubilization of Metals from Solids Using a KOH-KNO_3 Fusion, 7-18-11 (L. Darnell)* ☐ 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:	L. Darnell, 7-19-11
Spike Standard ID's:	R-485-a-10 (Pu-239)
Analysis Procedure:	RPG-CMC-408, Rev. 2, *Total Alpha and Beta Analysis*
Reference Date:	Not applicable
Analysis Date(s) or Date Range:	7-20-11 and 7-21-11
Technician/Analyst:	L. Darnell
Analysis Data (File):	11-1279 Fiskum.xls
CMC Project 98620 File:	File Plan 5871: T 52578 8878: 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):	Ludlum alpha scintillation detectors – See attached M&TE list

Preparer / 8/31/11 Date

T Trang-Le / 8/31/11
Reviewer Date

Sample Results

See attached data report, Sample Results for ASR 8878. All data are reported in units of µCi/g dry sample with a 1-σ uncertainty (see comments).

Sample Preparation, Separation, Mounting and Counting Methods

Fourteen samples submitted under Analytical Service Request (ASR) 8878 were analyzed for Gross Alpha. All the samples were prepared in laboratory 420 following RPL procedure PNL-ALO-115. Each sample was provided to the laboratory such that the entire mass of the sample could be used for the fusion dissolution. The entire contents of each vial was emptied into tared Ni crubles and then weighed. The weight obtained by the laboratory is the weight used for all dilution calculations. The resulting solution was used for radiochemical analyses; only Gross Alpha is reported under this report.

Following the fusion dissolution process, the digestates were prepared for counting without chemical separation. The digestates were diluted to bring the alpha activity down to levels needed for alpha counting and reduce the solids loading that occurs due to the fusion flux matrix. The diluted digestate was mounted for gross alpha counting using procedure RPG-CMC-4001, then counted using alpha counters per procedure RPG-CMC-408.

QUALITY CONTROL RESULTS

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

Performance acceptance criteria for these samples are established by both the K Basin data quality objectives (as provided as special instruction in the ASR) and the ASO Quality Assurance (QA) Plan (ASO-QAP-001, Rev. 7). Except where noted, the Gross Alpha K-Basin DQO and ASO QAP criteria are the same. In cases where the QC criteria are different, K-Basin DQO's will take precedence. The QC sample results for Gross Alpha have been evaluated and are discussed below. A summary of the Gross Alpha 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) – see comments. The PB gross alpha activity is less than the MDA . There are no acceptance criteria for LB. The requested detection limit of 1.0 E-03 µCi/g is lower than what can be achieved for this sample set. The sample matrix of dissolved fusion flux results in solid formation on the counting disc upon evaporation that causes absorption of alpha. Each sample counting disc was counted for 120 minutes. The achievable detection limit for the samples was 2.0 E-02 µCi/g. Occurrence report 52578-08-31-11 has been generated to document the achieved MDL not meeting the requested MDL.

Blank Spike (BS) – reagent spike:

The BS (Pu-239) recovery of 96% meets the acceptance criteria of 80% to 120% recovery.

Matrix Spike (MS):

The MS (Pu-239) recovery of 90% meets the acceptance criterion of 75% to 125% recovery. Note: the MS sample was prepared after digestion, by adding a known Pu-239 standard quantity to an aliquot of the leachate. Sample number 11-1290 (TI040-0792D-1c) was selected as the matrix spike sample.

Duplicate -- Relative Percent Difference (RPD):

Duplicate precision was not a required DQO for this samples set.

Instrument Quality Control

Alpha Ludlum detectors 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 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

None

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 Pu-239 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 includes weighing errors, volume uncertainties, and counting error.
4. The Laboratory Blank (LB) is prepared using laboratory reagents and provides data only on the cleanliness of the radiochemistry preparation process. LB results are not normalized to processing or dilution factors associated with the samples.
5. 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 8878

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

Alpha Spectrometry - Total Alpha

Project / WP#:	52578/H41216
ASR#:	8878
Client:	S. Fiskum
Total # of Samples:	14

Sample IDs	RPL Number		Sample IDs	RPL Number
TI040-0419S-1b	11-1279-115-S		TI040-0422S-1c	11-1285-115-S
TI040-0419D-2b	11-1280-115-S		TI040-0422D-1c	11-1286-115-S
TI040-0420S-2c	11-1281-115-S		TI040-0793S-1a	11-1287-115-S
TI040-0420D-1c	11-1282-115-S		TI040-0793D-1b	11-1288-115-S
TI040-0420D-2c	11-1282-115-D		TI040-0793D-2b	11-1288-115-D
TI040-0421S-2b	11-1283-115-S		TI040-0792S-1c	11-1289-115-S
TI040-0421D-1b	11-1284-115-S		TI040-0792D-1c	11-1290-115-S

Analysis Type:	AEA – Total Alpha
Sample Processing Prior to Radiochemical Processing/Analysis	☐ None ☐ Digested as per PNL-ALO-129, Rev. 0, *HNO_3-HCl Acid Extraction of Solids Using a Dry Block Heater* ☒ Fusion as per PNL-ALO-115 Rev. 1.1, *Solubilization of Metals from Solids Using a KOH-KNO_3 Fusion, (L. Darnell, 7-18-11)* ☐ Other:
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:	L. Darnell, 7-19-11
Spike Standard ID's	R-485-a-10 (Pu-239)
Analysis Procedure	RPG-CMC-408, Rev. 2, *Total Alpha and Beta Analysis*
Technician/Analyst:	L. Darnell, 7-20-11 and 7-21-11
Co-Precipitation Procedure:	RPG-CMC-496, Rev. 0, *Co-precipitation Mounting of Actinides for Alpha Spectroscopy*
Technician/Analyst:	L. Darnell, 7-26-11
Spike and Tracer Standard ID's:	R-485-a-10 (Pu-239)
Analysis Procedure:	RPG-CMC-422, Rev. 2, *Solutions Analysis: Alpha Spectrometry*
Reference Date:	Same as analyses dates
Analysis Date or Date Range:	7-27-11 and 7-28-11
Technician/Analyst:	T. Trang-Le
Analysis Data (File):	11-1279 Fiskum.xls
CMC Project 98620 File:	File Plan 5871: T 52578 8878: 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

K. L. Darnell / 8/31/11
Preparer **Date**

T Trang-Le / 8/31/11
Reviewer **Date**

Sample Results

See attached data report, Sample Results for ASR 8878. All data are reported in units of μCi/g dry sample with a 1-σ uncertainty (see comments).

Sample Preparation, Separation, Mounting and Counting Methods

Fourteen samples submitted under Analytical Service Request (ASR) 8878 were analyzed for Alpha-AEA. All the samples were prepared in laboratory 420 following RPL procedure PNL-ALO-115. Each sample was provided to the laboratory such that the entire mass of the sample could be used for the fusion dissolution. The entire contents of each vial was emptied into tared Ni crubles and then weighed. The weight obtained by the laboratory is the weight used for all dilution calculations.

Following the fusion digestion process, an aliquot of the digestate was mounted for alpha spectrometry by co-precipitation using procedure RPG-CMC-496, then counted by alpha spectroscopy using procedure RPC-CMC-422. The samples were counted on from July 27th to July 28th, 2011; no decay corrections were made.

QUALITY CONTROL RESULTS

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

Performance acceptance criteria for these samples are established by both the K Basin data quality objectives (as provided as special instruction in the ASR) and the ASO Quality Assurance (QA) Plan (ASO-QAP-001, Rev. 7). Except where noted, the Alpha-AEA K-Basin DQO and ASO QAP criteria are the same. In cases where the QC criteria are different, K-Basin DQO's take precedence. 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:

Tracer is not used for this analysis.

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

The alpha isotopic (Pu-239+240, Pu-238+Am-241 and Cm-243+244) activities 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 alpha isotopic activities are significantly less than 5% of any sample activity. There are no acceptance criteria for LB. The detection limit achieved in the fusion preparation blank for Pu-239+240 is 6.83 E-5 µCi/g, for Pu-238+Am-241 is < 5.0 E-5 µCi/g and for Cm-243+244 is <1.0 E-5 µCi/g. The achieved detection limits are all below the requested detection limit of 1.0E-3 $\mu Ci/g_{residual}$.

Blank Spike (BS) – reagent spike:

The BS recovery of 92% (Pu-239) meets the acceptance criteria of 80% to 120% recovery.

Matrix Spike (MS):

The MS recovery of 100% (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 11-1290-115-S (TI040-0792D-1c) was selected as the matrix spike sample.

Duplicate - Relative Percent Difference (RPD):

Duplicate precision was not a required DQO for this samples set.

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

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 and dilution (to avoid excessive consumption of spike and avoid creating unnecessary waste). The MS prepared with this batch of samples is considered a PS, since the Pu-239 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.

Attachment: Data Report -- Sample Results for ASR 8878

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

filename 11-1279 Fiskum
8/15/2011

Client: S. Fiskum
ASR 8878

Prepared by: C. Soderquist 8-15-11

Concur: T Trang-Le 8/15/11

Procedures: PNL-ALO-115, Rev 1.1, Solubilization of Metals from Solids Using a KOH-KNO3 Fusion
RPG-CMC-4001, Rev 1, Source Preparation for Gross Alpha and Gross Beta Analysis
RPG-CMC-408, Rev 2, Total Alpha and Total Beta Analysis
RPG-CMC-496, Rev 0, Coprecipitation Mounting of Actinides for Alpha Spectroscopy
RPG-CMC-422, Rev 2, Solutions Analysis: Alpha Spectrometry

M&TE: Ludlum alpha counters, Alpha spectrometry counting system

Reference date July 28, 2011

Sample	Lab ID	Measured Activity, **µCi per gram** of dry sample ± 1s						
		Gross Alpha		**Pu-239+240**		**Pu-238+Am-241**		**Cm-243+244**
TI040-0419S-1b	11-1279-115 S	**5.56E-1**	**± 3%**	**4.77E-1**	**± 2%**	**1.08E-1**	**± 3%**	< 1.E-3
TI040-0419D-2b	11-1280-115 S	**7.32E-1**	**± 3%**	**6.82E-1**	**± 2%**	**1.31E-1**	**± 3%**	< 8.E-4
TI040-0420S-2c	11-1281-115 S	**2.38E-1**	**± 5%**	**1.70E-1**	**± 2%**	**6.99E-2**	**± 3%**	< 8.E-5
TI040-0420D-1c	11-1282-115 S	**3.41E-1**	**± 5%**	**2.79E-1**	**± 2%**	**8.37E-2**	**± 2%**	< 2.E-4
TI040-0420D-2c	11-1282-115 D	**3.10E-1**	**± 5%**	**2.30E-1**	**± 2%**	**7.23E-2**	**± 3%**	< 2.E-4
	RPD	9%		19%		15%		--
TI040-0421S-2b	11-1283-115 S	**3.49E+0**	**± 2%**	**3.36E+0**	**± 2%**	**5.10E-1**	**± 3%**	< 1.E-3
TI040-0421D-1b	11-1284-115 S	**3.62E+0**	**± 2%**	**3.41E+0**	**± 2%**	**5.33E-1**	**± 3%**	< 2.E-3
TI040-0422S-1c	11-1285-115 S	**3.05E+0**	**± 2%**	**2.97E+0**	**± 2%**	**4.77E-1**	**± 3%**	< 2.E-3
TI040-0422D-1c	11-1286-115 S	**2.73E+0**	**± 2%**	**2.53E+0**	**± 2%**	**4.16E-1**	**± 3%**	< 2.E-3
TI040-0793S-1a	11-1287-115 S	**5.20E-1**	**± 3%**	**4.10E-1**	**± 2%**	**7.72E-2**	**± 2%**	< 3.E-4

Sample	Lab ID	Measured Activity, **µCi per gram** of dry sample ± 1s						
		Gross Alpha		**Pu-239+240**		**Pu-238+Am-241**		**Cm-243+244**
TI040-0793D-1b	11-1288-115 S	**4.06E-1**	**± 4%**	**3.45E-1**	**± 2%**	**6.46E-2**	**± 3%**	< 2.E-4
TI040-0793D-2b	11-1288-115 D	**3.51E-1**	**± 5%**	**2.39E-1**	**± 2%**	**4.58E-2**	**± 3%**	< 2.E-4
	RPD	15%		36%		34%		--
TI040-0792S-1c	11-1289-115 S	**2.99E-1**	**± 5%**	**2.13E-1**	**± 2%**	**4.66E-2**	**± 3%**	< 1.E-4
TI040-0792D-1c	11-1290-115 S	**2.19E-1**	**± 5%**	**1.82E-1**	**± 2%**	**3.55E-2**	**± 3%**	< 1.E-4
	Fusion blank	< 1.E-2		6.83E-5	± 23%	< 5.E-5		< 1.E-5
	Lab blank	< 2.E-6		< 4.E-8		< 3.E-8		< 2.E-8
	Reagent spike	96%		92%		--		--
	Matrix spike (TI040-0792D-1c)	90%		100%		--		--
	Requested detection limit	1.E-3		1.E-3		1.E-3		--

These samples are acid-insoluble residues from a hot cell acid digestion of K Basin sludge samples. These samples were delivered to the analytical lab and dissolved for analysis by fusion with KOH-KNO_3. Alpha emitters were measured in the fusion solution.

Results are given per gram of dry insoluble residue. The sample masses were provided by the Project. The duplicate samples were provided by the Project; the lab did not split or sub-sample any samples.

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	WD25687	EG&G Ortec	GEM15180P	35TP11079A	Ge	15	Y

K-Basin Occurrence Report

Title: ASR 8878 – Gross Alpha – Achieved detection limit for samples was greater than the requested detection limit of 1.0E-03µCi/g.		**Date: 08-31-11**
Unique Identifier: 52578-08-31-11		
Primary Person Identifying Issue: Karl Pool		
Distribution: Email and posting on K-Basin Share-Point	Clark Carlson	Karl Pool
	Deborah Coffey	Andy Schmidt
	Sandy Fiskum	Rick Shimskey
	Matt Fountain	Truc Trang Le, Project file for **ASR 8878**
Date Submitted to K-Basin Records:	9-1-2011	

Occurrence Description: ASR 8878:

1) Requested detection limit of 1.0E-03 µCi/g was not achieved on the gross alpha samples –

   The ASR requests a method detection limit of 1.0E-03 µCi/g for gross alpha that is not achievable in the sample matrix. The solids samples are dissolved using KOH/KNO_3 fusion. The resulting solution is high in dissolved solids, primarily from the fusion flux used. When gross alpha samples are prepared for analyses, the sample size used takes into account the solids loading that will occur when the sample dries on the counting disk. The solids loading significantly attenuates the alpha emitters on the counting disk. A small sample size is thus needed to minimize solids loading. Each sample was counted for 120 minutes with a resulting MDL as high as 2.0 E-02 µCi/g.

Impact of Occurrence:

There is no impact associated with this occurrence. Each sample in the set had measureable alpha activity. The lowest alpha activity measured in the sample set was 2.19E-01 µCi/g.

Significant Condition Adverse to Quality? Yes _____ No ✓

Evaluated by: _____ **Date:** 9/1/2011

Further Disposition: ☒ **No** ☐ **Yes** **If yes,** ☐ **Nonconformance?** ☐ **Deficiency?**

Date Occurrence Closed: 9-1-2011

Signature/Date of Responsible QE: _____ 9-1-2011

Signature/Date of Responsible K-Basin Manager: _____ 9/1/2011

Comments:

Analytical Service Request (ASR)

ASR-FY2006-ASO Rev. 0.doc

(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: MKEdwards
Print Name: MK Edwards
Phone: 375-5666 MSIN: P7-25

Project Number: 52578
Work Package: H41216

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: _____

- ♦ **Requested Analytical Work Completion Date:** 8/22/2011
  (Note: Priority rate charge for < 10 business day turn-around time)
- ♦ **Negotiated Commitment Date:** 8/22/11
  (*To be completed by ASO Lead*) KNP

Waste Designation Information

- ♦ **Sample Information Check List Attached?** ☒ No ☐ Yes
  If no, Reference Doc Attached: _____
  or, Previous ASR Number: 8824
  or, Previous RPL Number: _____

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

Send Report To: MK Edwards MSIN 8 P7-25
SK Fiskum MSIN P7-25

Additional or Special Instructions _____

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

Date Delivered: 8/10/11
Delivered By (optional) _____
Time Delivered (optional) _____
Group ID (optional) _____
CMC Waste Sample? ☒ No ☐ Yes

Received By: I Rang-te
ASR Number: 8919 Rev.: 00
RPL Numbers: (11-1555) - (11-1556)
(first and last)

ASO Work Accepted By: KN Pool **Signature/Date:** KN Pool 8/11/11

Analytical Service Request (ASR)

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

Provide Analytes of Interest and required Detection limits - ☐ Below ☐ Attached

ASO Staff Use Only RPL Number	Client Sample ID	Sample Description (& Matrix, If Varies)	Analysis Requested	ASO Staff Use Only Test	ASO Staff Use Only Library
11-1555	KW 210 mk2ML Resin beads	Resin beads	GEA	GEA	Radchem
11-1556	KW 210 mk2ML Residual Solids	Silica	GEA	↓	↓
		Reference date:	Jan 11, 2011 C 15:14:41 PM		

ASR # 8919 Rev.: 00 Page 1 of 1

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

Gamma Energy Analysis (GEA)

Project / WP#: 52578/ H41216
ASR#: 8919.00
Client: MK Edwards
Total Samples: 2 Samples

RPL ID	Client Sample ID
11-1555	KW210 MK & ML Resin Beads
11-1556	KW210 MK & ML Residual Solids

Analysis Type:	GEA- for all positively measured or non-detected isotopes
Sample Processing Prior to Radiochemical Processing/Analysis	☒ None ☐ Digested as per RPG-CMC-128, Rev. 0 *HNO_3-HCl Acid Extraction of Liquids for Metals Analysis Using a Dry Block Heater* ☐ Digested as per PNL-ALO-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: Preparation may also involve attaining a GEA geometry that is compatible with the calibration geometry.
Analysis Procedure:	RPG-CMC-450, Rev. 1 *Gamma Energy Analysis (GEA) and Low-Energy Photon Spectrometry (LEPS)*
Reference Date:	* January 11, 2011 @ 15:14:41
Analysis Date or Date Range:	August 10, 2011
Technician/Analyst:	MG Cantaloub
Rad Chem Electronic Data File:	*11-1555 Edwards.xls*
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):	*Detector T (M & TE unique identifiers in attached supporting documents)*

*Reference date used is the same date and time used for samples submitted under ASR 8725 as directed by S. Fiskum.

T Trang-Le / 8/31/11
Prepare **Date**

Reviewer / 8/31/11
Date

SAMPLE RESULTS

Activities for all gamma emitters detected in these samples are presented in an attached Excel spreadsheets for ASR 8919.00. All residual solids are reported as µCi/g , both 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 µCi/g where g residual is the mass of residual solids that did not dissolve during acid digestion.

ASO Project File, ASR 8919 has been created for this report including all appropriate supporting records which may include the Pipette Performance Verification form, Standard Certificates, Laboratory Bench Record, Shielded Analytical Laboratory Bench Sheet, 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

Solid samples were received from client pre-dried and analyzed by GEA "as received."

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. 7); 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):

There is no process blank in this batch. No sample preparation was performed, the samples were analyzed "as received".

Required Detection Limits

The ASR doesn't have detection limit requirements for residual solid or resin samples.

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

There were no BS, MS or LCS 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):

There is no duplicate sample pairs for this ASR.

Replicate Relative Standard Deviation (RSD):

There were no replicate samples for this ASR.

Instrument Calibration and Quality Control

Gamma detectors are calibrated using multi-isotope standards that are NIST-traceable and prepared in the identical counting geometry used for the 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 an 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

None.

Interferences/Resolution

None.

Uncertainty

For gamma counting, the uncertainty in the efficiency, 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. A conservative estimate is that 2% is the lowest uncertainty possible for our GEA measurements taking into account systematic uncertainties in gamma calibration standards.

The samples were counted at about 20.5 inches from the detector to minimize dead time due to the high sample activities. Counting at such long distances also minimizes the differences between the sample geometry and the calibration geometry. Calibrations are performed using water samples whereas the samples consisted of mostly silica (however, the exact sample mineralogical composition and associated density were not known). The small sample size (<1g) minimized the gamma absorption effects; therefore, density corrections were not applied.

Comments

1. The results have been corrected 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.

Attachment: Data Report Sample Results for ASR 8919.

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

filename 11-1555 Edwards
8/22/2011

Client: M. Edwards
ASR: 8919

Project: 52578
WP: H41216

Prepare by: T Trang-le 8-22-11

Concur: C Soderquist 8-22-11

Procedures: RPG-CMC-450, Rev. 1 Gamma Energy Analysis (GEA) and Low-Energy Photon Spectrometry (LEPS)
Count date: 10-Aug-11
Reference date: 1/11/2011 15:14
M&TE: Detector T

Sample	Lab ID	Measured Activity, **µCi per gram** ± 1s						
		Co-60	**Cs-134**	**Cs-137**	**Eu-152**	**Eu-154**	**Eu-155**	**Am-241**
KW210 MK & ML Resin Beads	11-1555	**2.22E-2 ± 5%**	< 4.E-3	**6.97E+1 ± 3%**	< 7.E-3	**9.37E-2 ± 4%**	< 8.E-2	**7.76E-1 ± 13%**
KW210 MK & ML Residual Solids	11-1556	**7.60E-2 ± 10%**	< 3.E-2	**4.56E+2 ± 3%**	< 4.E-2	< 6.E-2	< 7.E-1	< 2.E+0

These samples were counted as received from the project, using the sample masses provided by the project. All detected gamma emitters are reported.

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	WD25687	EG&G Ortec	GEM15180P	35TP11079A	Ge	15	Y

Appendix E

Isotopic Analysis Request and Results

Appendix E

Isotopic Analysis Request and Results

Appendix E contains the signed analysis request for the Pu and U isotopic analyses of Engineered Container SCS-CON-210 core samples. The analysis request delineates sample-specific information, required precision, quality control (QC) sample requirements, and reporting requirements. The core samples were initially processed by the Analytical Services Operations (ASO) laboratory according to Analytical Services Request (ASR) 8798 (see Appendix B). This included acid digestion according to PNL-ALO-129, Rev. 0, *HNO_3-HCl Acid Extraction of Solids Using a Dry-Block Heater*. The resulting solutions were processed by the ASO to isolate the U component and the Pu component as two separate fractions per sample. The ASO staff then provided the purified fractions to the thermal ionization mass spectrometry (TIMS) workstation for analysis. The ASO documented the TIMS preparative work in the "Thermal Ionization Mass Spectrometry Sample Preparation" summary report (provided in Appendix E).

The TIMS analytical report with the sample isotopic distributions, associated uncertainties, and a discussion of sample processing, QC results, data limitations, and Excel data reduction spreadsheet (KW210 TIMS data June 23, 2011.xls) are provided in Appendix E. Occurrence Reports are prepared when issues are identified, and resolution is required while work is in process; one Occurrence Report was issued and is also provided. A portion of the data reduction spreadsheet, ASR 8798 data reduction Rev. 1.xlsx, which is relevant to the isotopic calculations, is also provided. The isotopic calculations data reduction spreadsheet renormalized the TIMS-reported isotopic distribution with ^{238}Pu concentration from Pu alpha energy analysis, converted U and Pu isotopic results from atom percent to mass percent, and then converted isotope fraction to activity and mass concentrations (dry and wet settled sludge mass basis). The full calculation spreadsheet is provided with the supplemental information as part of the complete data package.

The following table of contents shows that pagination specific to Appendix E was applied to the TIMS analysis request, the ASO preparation and TIMS analysis reports, the supporting TIMS data, Occurrence Report 52578-05-09-11, and the surveillance report (generated by the Pacific Northwest National Laboratory Quality Engineer). This pagination is sequential and consistent (e.g., page E.#).

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Thermal Ionization Mass Spectrometry

Analysis Request for Plutonium and Uranium Isotopes in K Basin KW210 Core Samples

Cognizant Scientist: SK. Fiskum Date: 4/13/11
SK Fiskum

Analyst: SJ Bos Date: 4/14/11
SJ Bos

Project Quality Engineer: DS Coffey Date: 4-14-11
DS Coffey

Project Manager: CD Carlson Date: 4/14/2011
CD Carlson

STP Concurrence: WW Rutherford Date: 4/14/11
WW Rutherford

Thermal Ionization Mass Spectrometry

Analysis Request for Plutonium and Uranium Isotopes in

K Basin KW210 Core Samples

Requester	SK Fiskum	**Work Package**	H41217
Signature		**Project Number**	52578
		Subtask	TIMS
Date Submitted:		**Date Required:**	4/11/11
Sample Description:	Four core samples were collected from the Large Engineered Container, SCS-CON-210, containing sludge located at the K West Basin. Subsamples have been collected from each of the core samples and dissolved according to PNL-ALO-129, Rev. 0, *HNO_3-HCl Acid Extraction of Solids Using a Dry-Block Heater*. Aliquots of the sample digestates were prepared for Pu TIMS analysis by the ASO using the separation procedure RPG-CMC-455, Rev. 0, *Separation of Uranium and Plutonium for Isotopic Analysis by Mass Spectroscopy*. Aliquots of the sample digestates were prepared for U TIMS analysis by the ASO using the separation procedure RPG-CMC-4017, Rev. 0, *Analysis of Environmental Water Samples for Actinides and Strontium-90.* The sample IDs and corresponding core sample IDs are provided.		

Project Sample ID	**RPL/ASO Sample ID—Uranium Isotopic**	**RPL/ASO Sample ID—Plutonium Isotopic**	**K Basin Core Sample ID**
TI001-A1-B	11- 0419-129-S-U	11- 0419-129-S-Pu	KW210-A1
TI001-A1-C	11-0419 -129-D-U	11-0419 -129-D-Pu	KW210-A1 duplicate
TI002-A4-B	11-0420-129-S-U	11-0420-129-S-Pu	KW210-A4
TI002-A4-C	11-0420-129-D-U	11-0420-129-D-Pu	KW210-A4 duplicate
TI002-B1-B	11-0421-129-S-U	11-0421-129-S-Pu	KW210-B1
TI002-B1-C	11-0421-129-D-U	11-0421-129-D-Pu	KW210-B1 duplicate
TI001-B3-B	11-0422-129-S-U	11-0422-129-S-Pu	KW210-B3
TI001-B3-C	11-0422-129-D-U	11-0422-129-D-Pu	KW210-B3 duplicate
NA	11-0419-129-PB-U	11-0419-129-PB-Pu	ASO preparation blank

Preparations and Analysis

The Analytical Support Operations (ASO) was directed to provide purified sample fractions with sufficient analyte concentrations to create a measureable signal by Thermal Ionization Mass Spectrometry

(TIMS). Therefore, the ASO preparation shall not be considered complete until satisfactory isotopic results are obtained. It is understood that the preparation blank (PB) may not have sufficient analyte concentration to meet this objective. The ASO does not provide a laboratory control sample (LCS) or a matrix spike (MS) sample.

TIMS-specific sample preparation, processing, and analyses shall be conducted according to PNNL-98523-264, *Mass Spectrometer Isotopic Analysis*, Rev. 2. Procedure substitutions or procedure revision substitutions are ***not*** to be made without prior written authorization from SK Fiskum or CD Carlson and may result in the need to issue a K Basin Occurrence Report. Should insufficient analyte concentration be found to conduct an analysis, contact SK Fiskum and KA Pool, who will set up an additional sample purification process to be performed by the ASO.

Quality Control

The TIMS analysis processing will need to adhere to the QC defined in the technical procedure PNNL-98523-264, *Mass Spectrometer Isotopic Analysis*, Rev. 2 and project-specific quality control as follows:

Duplicates	The ^{239}Pu, ^{240}Pu, ^{241}Pu, and ^{242}Pu atom percents for a given core sample and duplicate shall agree within ±20% where the isotopic abundance is greater than 1%.
	The ^{233}U, ^{234}U, ^{235}U, ^{236}U, and ^{238}U atom percents shall agree within ±20% for a given core and duplicate sample where the isotopic abundance is greater than 1%.
Reference Standards	At least one traceable reference standard must be analyzed at the beginning of the analytical batch. The results for ^{238}Pu, ^{239}Pu, ^{240}Pu, ^{241}Pu, ^{242}Pu atom percents shall be within 6% of the certified value where the isotope atom percent of the certified value is >0.1.
	Traceable reference standards must be analyzed at the beginning of the analytical batch. The results for ^{234}U, ^{235}U, ^{236}U, and ^{238}U atom percents shall be within 2% of the certified value where the isotope atom percent of the certified value is >0.1.

Note that the Reference Standard acceptance criteria identified in the QAPjP/SAP KBC33786, Rev. 2 were developed specifically for ^{239}Pu and ^{240}Pu (±6%) and for ^{235}U and ^{238}U (±2%). These criteria are conservatively applied to other (minor) isotopes in the Standard Reference Material (SRM) is where the minor isotope concentration exceeds 0.1 atom%.

Note: The TIMS analysis staff must inform/report QC failures promptly to the Cognizant Scientists with copy to CD Carlson and the K Basin QE (Deborah Coffey), thus enabling PNNL to fulfill its commitment to the client for prompt notification of such failures. The impact of any QC failure on data quality or project schedule (e.g., re-runs) will be assessed in conjunction with the client.

TIMS Reporting

Report ^{238}Pu, ^{239}Pu, ^{240}Pu, ^{241}Pu, and ^{242}Pu atom percents for each sample in a tabular format.

Report ^{233}U, ^{234}U, ^{235}U, ^{236}U, and ^{238}U atom percents for each sample in a tabular format.

Provide a report with the following elements: a data report **Cover Page**, a **Narrative**, the **Data Summary**, the signed **Analysis Request Form** and the **Supporting Data**.

Data Report **Cover Page** Contents (see 52578-ADM-001 *K Basin Requests for Analysis and Guidelines for Data Reporting and Technical, QE, and Spreadsheet Reviews* and Attachment 1 for an example):

- Header identifying name/address of laboratory
- Client Name:
- Project #/WP #(s):
- Total # Samples:
- Report Date:
- Report Revision Date: when applicable
- Filename(s): where data files can be accessed
- Dates of Sample Analysis:
- Procedure(s): Number and title (sample processing and analysis) and revision number
- M&TE (serial number or property number):
- Reference Date (e.g., sample run date):
- Client ID and RPL/ASO ID Sample Numbers:
- Report Review and Approval:

  Preparer _____Date_____
  (Printed name and signature of responsible individual)

  Technical Review _____Date _____
  (Printed name and signature of technical reviewer)

  Quality Engineer Review_____Date _____
  (Printed name and signature of quality engineering reviewer)

  Project Manager Review_____Date _____
  (Printed name and signature of Project Management reviewer)

Narrative Contents:

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

Data Summary Contents: (This is a tabular summary with the following components)

- Client ID
- RPL ID with full ASO-related suffixes (e.g., 129-S)
- TIMS ID if used
- Sample results and result uncertainties
- Standard results and result uncertainties

Supporting Data

- Standards certificates
- Signature and date of person who performed the test and recorded the data
- Analytical bench sheets or copies of LRB pages (including evidence of technical review)
- Hand/spreadsheet calculation review documentation
- Computer printouts of the raw data

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 as soon as practical after completion of analysis.

Attachment 1. Example Data Reporting Cover Sheet

Plutonium and Uranium Isotopic Analysis for KW210 Core Samples

Battelle—Pacific Northwest National Laboratory
Radiochemical Sciences and Engineering Group
PO Box 999, Richland, Washington 99352
Project: #52578

Client: SK Fiskum

Sample ID's:

Project Sample ID	RPL/ASO Sample ID—Uranium Isotopic	RPL/ASO Sample ID—Plutonium Isotopic	K Basin Core Sample ID
TI001-A1-B	11- 0419-129-S-U	11- 0419-129-S-Pu	KW210-A1
TI001-A1-C	11-0419 -129-D-U	11-0419 -129-D-Pu	KW210-A1 duplicate
TI002-A4-B	11-0420-129-S-U	11-0420-129-S-Pu	KW210-A4
TI002-A4-C	11-0420-129-D-U	11-0420-129-D-Pu	KW210-A4 duplicate
TI002-B1-B	11-0421-129-S-U	11-0421-129-S-Pu	KW210-B1
TI002-B1-C	11-0421-129-D-U	11-0421-129-D-Pu	KW210-B1 duplicate
TI001-B3-B	11-0422-129-S-U	11-0422-129-S-Pu	KW210-B3
TI001-B3-C	11-0422-129-D-U	11-0422-129-D-Pu	KW210-B3 duplicate
NA	11-0419-129-PB-U	11-0419-129-PB-Pu	ASO preparation blank

Total Samples: 18

Procedure: PNNL-98523-264, *Mass Spectrometer Isotopic Analysis*, Rev. 2

Analyst:

Analysis (Reference) Dates:

Instrument: Manufacturer, Model, Unique Identifier (property or serial number), Instrument Software (name, version, date)

Electronic Report File name: File name and file location

_____
Prepared By: {type name here} / Date

_____
Technical Review By: {type name here} / Date

_____
QE Review By: {type name here} / Date

_____
Project Management Release / Date

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

Thermal Ionization Mass Spectrometry Sample Preparation

Project / WP#:	52578/H41216
ASR#:	8798
Client:	S. Fiskum
Total # of Samples:	4

Revision 1

Engineered Container & Core ID	Sample IDs	RPL Number
KW-210-A1	TI001-A1-B, -A1-C	11-0419-129-S, -129-D
KW-210-A4	TI002-A4-B, -A4-C	11-0420-129-S, -129-D
KW-210-B1	TI002-B1-B, -B1-C	11-0421-129-S, -129-D
KW-210-B3	TI001-B3-B, -B3-C	11-0422-129-S, -129-D

Analysis Type:	TIMS Sample Preparation
Sample Processing Prior to Radiochemical Processing/Analysis	☐ None ☒ Digested as per PNL-ALO-129, Rev. 0, *HNO_3-HCl Acid Extraction of Solids Using a Dry Block Heater, 3-7-11 (N. Green)* ☐ 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
Separation Procedures:	RPG-CMC-4017, Rev. 0, *Analysis of Environmental Water Samples for Actinides and Strontium- 90.* RPG-CMC-455, Rev.0, *Separation of Uranium and Plutonium for Isotopic Analysis by Mass Spectroscopy*
Spike Standard ID:	N/A
Separation Dates:	4/22/11 – 4/27/11
Technician/Analyst:	K. Carson
Rad Chem Electronic Data File:	N/A
ASO Project 98620 File:	File Plan 5871: T 52578 8798: 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):	N/A

Preparer / Date: 6/30/11

T Trang-Le / 6/30/11
Reviewer / Date

Revision 1

This revision is being issued to correct the procedure references for the Pu and U Thermal Ionization Mass Spectrometry (TIMS) preparations.

Sample preparation and separation

Four K-Basin sludge samples were prepared in the Shielded Analytical Laboratory (SAL) hot cells by the Project and provided to the Analytical Support Operations (ASO) under Analytical Service Request (ASR) 8798 for chemical and radioanalytical analyses. ASO received each sample in a Teflon Oak Ridge centrifuge tube, dried the contents of each centrifuge tube to a constant weight, and then leached the dried solids with hot dilute nitric and hydrochloric acids per procedure PNL-ALO-129, Rev. 0, *HNO_3-HCl Acid Extraction of Solids Using a Dry Block Heater*. This digestion process was conducted in the SAL hot cells. The leachate solution was used for radioanalytical analysis, ICP metals analysis and isotopic distribution of Pu and U by (TIMS); only TIMS sample preparation is discussed in this report.

Analytical Support Operations (ASO) performed the initial chemical separation for sample aliquouts that are transferred to the TIMS for isotopic ratio determinations from the PNL-ALO-129 digests. The text below provides a description of the chemical separation performed on samples for TIMS analyses.

The ASO utilizes procedure RPG-CMC-4017, Rev. 0, *Analysis of Environmental Water Samples for Actinides and Sr-90* to obtain the uranium chemical fraction used for TIMS analyses. The uranium is chemically separated using ion exchange chromatography; the uranium chemical fraction is collected and then concentrated in Teflon conical vials. The uranium fraction samples are then sent to the TIMS laboratory for analysis.

The ASO utilizes procedure RPG-CMC-455, Rev. 0, *Separation of Uranium and Plutonium for Isotopic Analysis by Mass Spectrometry* to obtain the plutonium chemical fraction used for TIMS analysis. The plutonium is chemically separated using ion exchange chromatography; the plutonium chemical fraction is collected and then concentrated in Teflon conical vials. The plutonium fraction samples are then sent to the TIMS laboratory for analysis.

Preparation Blank

A preparation blank (PB) was included in the SAL digestion of the K-Basin sludge samples and the PB sample was also processed for Pu and U TIMS. The aliquot size used for the preparation blank was 1.0 mL of undiluted digestate while the aliquot sized used for the samples was 0.1 mL of the undiluted digestate.

Plutonium and Uranium Isotopic Analysis for KW210 Core Samples

Battelle—Pacific Northwest National Laboratory
Radiochemical Sciences and Engineering Group
PO Box 999, Richland, Washington 99352
Project: #52578

July 15, 2011

Sample ID's:

Project Sample ID	ASO Sample ID	ASO Sample ID	Sample Description
TI001-A1-B	11-0419-129-S-U	11-0419-129-S-Pu	KW210-A1
TI001-A1-C	11-0419-129-D-U	11-0419-129-D-Pu	KW210-A1 duplicate
TI002-A4-B	11-0420-129-S-U	11-0420-129-S-Pu	KW210-A4
TI002-A4-C	11-0420-129-D-U	11-0420-129-D-Pu	KW210-A4 duplicate
TI002-B1-B	11-0421-129-S-U	11-0421-129-S-Pu	KW210-B1
TI002-B1-C	11-0421-129-D-U	11-0421-129-D-Pu	KW210-B1 duplicate
TI001-B3-B	11-0422-129-S-U	11-0422-129-S-Pu	KW210-B3
TI001-B3-C	11-0422-129-D-U	11-0422-129-D-Pu	KW210-B3 duplicate
NA	11-0419-129-PB-U	11-0419-129-PB-Pu	ASO preparation blank

Procedure: PNNL-98523-264 Rev. 2, *Mass Spectrometer Isotopic Analysis*

Analyst: Stan Bos

Analysis Dates: U-TIMS 5/6/11 and 5/7/2011, Pu-TIMS 5/18 and 5/25/2011

Instrument: CEC, model 21-703 (M&TE# 467806) Property # WB76849, Instrument Software Isoto_26.prg Rev. A, (3/2000)

Electronic Report File name: https://pnlweb.pnl.gov/projects/KBasin_2\Plutonium and Uranium Isotopic Analysis for KW210 Core Samples

S J Bos 7/15/11
Prepared By: Stan Bos / Date

Jennifer C Braley 7/15/2011
Technical Review By Jen Braley / Date

Deborah Coffey 20 July 2011
QE Review By: Deborah Coffey / Date

7/21/2011
Project Management Release / Date

MEMORANDUM

Pacific Northwest
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Date: **July 15, 2011**
To: **Sandy Fiskum**
From: **Stan Bos**
Subject: **Pu and U Isotopic analysis**

Project No.: **52578**
Internal Distribution: **File/LB**

Samples of K-Basin sludge material were analyzed for uranium and plutonium isotopic abundance by thermal ionization mass spectrometry (TIMS). Sample sets consisted of four samples each prepared in duplicate along with one preparation blank. Samples were delivered on May 3, 2011 in two sets; one containing the uranium fraction and the second containing the plutonium fraction.

The uranium preparations arrived at the mass spectrometer lab in 20cc glass vials. One milliliter of nitric acid was added to each vial and 5µL of sample was pipetted and dried onto rhenium filaments for analysis. Uranium isotopic analysis began on May 3, 2011 running NIST uranium isotopic standard U-030 and continued on May 6 and 9, 2010. Seven samples were analyzed on May 6, 2011, but due to a temperature swing in the laboratory analysis had to be stopped where the mass spectrometer went out of calibration. Analyses were restarted on May 9, 2011 rerunning samples 11-0422-129-SU, 11-420-129-D-U and running 11-0422-129-D-U. The uranium isotopic abundance results and are reported in Table 1.

Plutonium preparations were received either dry or with up to 30µL of solution in conical shaped Teflon vials. For the samples that were dry or nearly dry, 25µL of 4M nitric acid was added to the vial and 10µL of solution were pipetted and dried on a rhenium filament for analysis. Plutonium isotopic analysis was performed on May 17 and 18, 2011 running NIST isotopic standard Pu-946 before and after sample analysis. Three samples 11-0420-129-S-P, 11-0420-129-D-P, and 11-0421-129-D-P, had inconsistent results and were reanalyzed on May 25, 2011. Results of the analyses are reported in Table 3. Sample signal was adequate for the analyses but was not comparable to the signal strength of the standards. While the instrument was producing ~10 volt signals on Pu-946 standards, the samples were providing ~1 volt signals. It was noted that there was a buildup of residue on the filament which might have caused the signal to be depressed.

A preparation blank was included with each set of samples. In both sample sets the preparation blank arrived dry and were diluted the same as the associated samples. Five microliters of the preparation blank for uranium and 10µL for plutonium was loaded onto the rhenium filament and installed in the mass spectrometer. For both the U and Pu preparation blanks, there was not adequate signal at the major analyte mass to

warrant an attempt at isotopic analysis. Preparation blanks are reported as not detected (ND) for all isotopes.

Separations:

In preparation for TIMS analyses, the plutonium and uranium were chemically separated by Analytical Support Operations (ASO) staff using ion exchange procedures RPG-CMC-4017 (Rev 0) for uranium and RPG-CMC-455 (Rev 0) for the plutonium. Separations procedures were run concurrently on April 22, 2011. These procedures segregate the uranium and plutonium into separate and purified fractions. This process is necessary to eliminate mass interferences common to both elements (e.g., mass 238 as Pu and U) as well as to reduce other materials that otherwise would create mass loading on the filament. The PNL-ALO-129 acid digestion preparation blank (generated during processing in the hot cells) was also prepared for TIMS in parallel with the samples

Analysis:

The mass spectrometer is designed to analyze one sample at a time. Samples arrive at the mass spectrometry laboratory in either 20cc glass vials or 5cc conical Teflon vials. Depending on the condition of the sample they may be diluted in nitric acid or may be run without dilution. A portion of the sample solution is pipetted onto a rhenium filament then taken to dryness by electrically heating the filament. Samples are then loaded into the mass spectrometer for analysis.

TIMS measurements are made with the instrument cycling through each isotope analyte, taking measurements at each stop and averaging the results at a given mass. This routine is repeated 9 times calculating the isotope ratios after each cycle. An average isotope ratio is computed at the end of the analytical routine. From the isotopic ratios, atom percent and mass percent are calculated. Estimates of uncertainty are calculated from the standard deviation of the isotopic ratios for the minor peaks. For the major peak the uncertainty is calculated from the sum of the uncertainties in minor peaks.

Isotopic abundance analysis was performed using the CEC 21-703 thermal ionization mass spectrometer (M&TE 467806) following procedure PNNL-98523-264 (Rev 2). A record of the analyses was recorded in laboratory record book 56999 pages 77 and 78. Samples were assigned RPL numbers 11-0419 through 11-0422. Analytical requests forms were prepared for the U and Pu TIMS analyses and are attached to this report. Work package H41217 has been charged for this analysis.

Calibration:

Instrument performance checks for uranium isotopic analysis are made using an isotopic reference standard supplied by NIST. Uranium isotopic standard U-030 was analyzed on May 3, 2011 to verify the instrument output and testing the mass discrimination correction. No adjustment was made to the mass discrimination correction. Standards were analyzed on May 6, and 9, 2011 with a standard being run before the samples on May 6 and before and after samples on May 9, 2011. Results of 5 runs of U-030 are reported and averaged in the uranium standards Table 2. The performance criterion for the TIMS instrument is agreement of ±2% for the major (>0.1 atom percent) isotopes to the certified standard. Pooled averages for all U-030 standard runs met the ±2% criteria for the major isotopes, U^{235} and U^{238}, but did not meet the criteria for minor isotope U^{236} where the percent difference was -2.66%. Occurrence Report 52578-05-09-11 has been issued to address the impact of reference standard failure.

Instrument performance checks for plutonium isotopic analysis are made using NIST Pu-946 isotopic reference standard. Prior to starting sample analysis on May 17, 2011 three runs of Pu-946 were made to verify the instrument output and the mass discrimination setting. No adjustment was made to the mass discrimination correction. Standards were also analyzed before and after samples on May 18 and 25, 2011. Results of nine Pu-946 analyses are reported in the Pu isotopic standards Table 4. The performance criterion for the TIMS analysis is agreement of ±6% for the major (>0.1 atom percent) isotopes. This criterion was met for all isotopes.

Relative percent difference of duplicate samples:

Duplicate preparations were analyzed and relative percent differences of the results were calculated. A performance agreement of ±20 % was requested. This criterion was met for the major isotopes of uranium but was not attained for isotope U-234 on samples 11-0422-129-S-U and -D-U. The ±20 RPD was not attained on the U-233 isotope likely due to the low abundance of the isotope in the sample. Performance agreement for Pu-241 was not met on one set of duplicate pairs (TI001-A1-B and TI001-A1-C at 27% RPD). Per the analysis request, duplicate precision criteria did not apply to Pu^{238} because of the recognition that the U^{238} isobar may interfere (despite efforts to create a clean Pu-U separation by the ASO. The QAPjP/SAP KBC-33786, Rev. 2 Tables 3-7 indicates that the duplicate precision only applies to major isotopes where a "major isotope" was defined as isotope abundance greater than 1% of the total sample. The U^{233}, U^{234}, Pu^{238}, Pu^{241} and Pu^{242} were each present at <0.7 atom%, therefore the precision criterion does not apply to these isotopes. Occurrence Report 52578-05-09-11 has been issued to address the impact of duplicate sample RPD failures.

Sample Identification:

Project Sample ID	ASO Sample ID	ASO Sample ID	Sample Description
TI001-A1-B	11-0419-129-S-U	11-0419-129-S-Pu	KW210-A1
TI001-A1-C	11-0419-129-D-U	11-0419-129-D-Pu	KW210-A1 duplicate
TI002-A4-B	11-0420-129-S-U	11-0420-129-S-Pu	KW210-A4
TI002-A4-C	11-0420-129-D-U	11-0420-129-D-Pu	KW210-A4 duplicate
TI002-B1-B	11-0421-129-S-U	11-0421-129-S-Pu	KW210-B1
TI002-B1-C	11-0421-129-D-U	11-0421-129-D-Pu	KW210-B1 duplicate
TI001-B3-B	11-0422-129-S-U	11-0422-129-S-Pu	KW210-B3
TI001-B3-C	11-0422-129-D-U	11-0422-129-D-Pu	KW210-B3 duplicate
NA	11-0419-129-PB-U	11-0419-129-PB-Pu	ASO preparation

MEMORANDUM

Pacific Northwest
NATIONAL LABORATORY
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Table 1. Uranium Isotopic Analysis in KW210 Core Samples
By Thermal Ionization Mass Spectrometry

Final Reported Results

TIMS CEC21-703 (M&TE 4678061)

Sample #	Sample ID	Filament	Run Date	U-233	U-234			U-235			U-236			U-238		
				At%	At%	Std Dev	%RSD	At%	Std Dev	%RSD	At%	Std Dev	%RSD	At%	Std Dev	%RSD
TI001-A1-B	11-0419-129-S-U	1111010	05/06/11	<0.003	0.008	± 0.001	15.84	0.711	± 0.012	1.64	0.073	± 0.005	6.83	99.208	± 0.018	0.02
TI001-A1-C	11-0419-129-D-U	1111011	05/06/11	<0.003	0.007	± 0.001	16.44	0.709	± 0.008	1.11	0.076	± 0.002	3.17	99.208	± 0.011	0.01
			Average =		0.008			0.710			0.075			99.208		
			RPD (%)		14			0.4			4			0		
	11-0419-129-PB-U	1111012	05/06/11	(ND)	(ND)			(ND)			(ND)			(ND)		
TI002-A4-B	11-0420-129-S-U	11111013	05/06/11	<0.003	0.007	± 0.003	41.18	0.702	± 0.017	2.39	0.072	0.004	6.06	99.220	± 0.024	0.02
TI002-A4-C	11-0420-129-D-U	1111014	05/09/11	<0.003	0.007	± 0.002	27.18	0.705	± 0.027	3.77	0.074	0.007	9.66	99.214	± 0.036	0.04
			Average =		0.007			0.704			0.073			99.217		
			RPD (%)		0			0.4			1			0.006		
TI002-B1-B	11-0421-129-S-U	1111015	05/06/11	<0.003	0.007	± 0.002	36.69	0.706	± 0.041	5.87	0.071	0.010	13.34	99.216	± 0.053	0.05
TI002-B1-C	11-0421-129-D-U	1111016	05/06/11	<0.003	0.007	± 0.002	29.43	0.704	± 0.011	1.56	0.072	0.006	7.92	99.217	± 0.019	0.02
			Average =		0.007			0.705			0.072			99.217		
			RPD (%)		0			0.3			1			0.000		
TI001-B3-B	11-0422-129-S-U	1111017	05/09/11	<0.003	0.008	± 0.003	37.16	0.710	± 0.012	1.69	0.071	0.006	8.20	99.211	± 0.021	0.02
TI001-B3-C	11-0422-129-D-U	1111018	05/09/11	<0.003	0.011	± 0.005	41.52	0.714	± 0.019	2.69	0.077	0.006	7.76	99.198	± 0.030	0.03
			Average =		0.010			0.712			0.074			99.205		
			RPD (%)		30			0.6			8.0			0.000		

Results have been renormalized to remove the U^{233} values found on the run sheets.
The Process Blank had inadequate signal at the major analyte mass attempt perform isotopic analysis reported as ND not detected.

Table 2. Uranium Isotopic Analysis in KW210 Core Samples
By Thermal Ionization Mass Spectrometry

Summary of Standard Results - Standard: U-030														
			U-234			U-235			U-236			U-238		
	Run Date	Filament	Atom%	1−σ	RSD(%)	Atom%	1−σ	RSD(%)	Atom%	1−σ	RSD(%)	Atom%	1−σ	RSD(%)
Cert Value	n/a	n/a	0.0190	± 0.0001	n/a	3.046	± 0.0030	n/a	0.0204	± 0.0010	n/a	96.915	± 0.003	n/a
Pooled	n/a	n/a	0.0191	± 0.0007	3.42	3.019	± 0.011	0.38	0.0199	± 0.0015	7.40	96.94	± 0.011	0.01
		%Difference =	0.61			-0.88			-2.66			0.03		
		Acceptable range	0.0186 to 0.0194			2.99 to 3.11			0.0200 to 0.0208			94.98 to 0.98.85		
1	05/03/11	0301114.1	0.0187	± 0.0023	12.5	3.001	± 0.026	0.86	0.0216	± 0.0031	14.3	96.96	± 0.03	0.03
2	05/06/11	0301116.1	0.0195	± 0.0025	12.8	3.017	± 0.031	1.0	0.0193	± 0.0019	9.8	96.94	± 0.03	0.04
3	05/06/11	0301117.1	0.0201	± 0.0021	10.6	3.030	± 0.023	0.75	0.0178	± 0.0024	13.5	96.93	± 0.03	0.03
4	05/09/11	0301118.1	0.0188	± 0.0032	16.9	3.020	± 0.042	1.4	0.0197	± 0.0015	7.6	96.94	± 0.05	0.05
5	05/09/11	0301119.1	0.0185	± 0.0021	11.1	3.027	± 0.023	0.76	0.0209	± 0.0017	8.1	96.93	± 0.03	0.03

The pooled results for uranium isotopes U^{234}, U^{235} and U^{238} meet the acceptance criteria of ±2% of the certified value, isotope with abundances <0.1 atom percent did not meet the ± 2% criteria. Each analysis run of U^{235} and U^{238} met the acceptance criteria of ±2%. The shaded results indicate a response exceeding the ±2% criterion; see the Occurrence Report 52578-05-09-11-18-11.

Note: Decay correction is not required for uranium isotopic standards.

Table 3. Plutonium Isotopic Analysis in KW210 Core Samples
By Thermal Ionization Mass Spectrometry

Final Reported Results

TIMS CEC21-703 (M&TE 4678061)

Sample #	Sample	Filament	Run Date	Pu-238			Pu-239			Pu-240			Pu-241			Pu-242		
				At%	Std Dev	%RSD	At%	Std Dev	%RSD	At%	Std Dev	%RSD	At%	Std Dev	%RSD	At%	Std Dev	%RSD
TI001-A1-B	11-0419-129-S-Pu	1211010	05/18/11	0.095	± 0.052	54.9	86.1	± 0.7	0.8	12.9	± 0.5	3.91	0.674	± 0.104	15.5	0.231	± 0.067	28.9
TI001-A1-C	11-0419-129-D-Pu	1211011	05/18/11	0.155	± 0.037	24.1	86.2	± 0.5	0.5	12.9	± 0.4	2.73	0.515	± 0.047	9.1	0.250	± 0.032	12.9
			Average =	0.125			86.1	± 0.1		12.9			0.595			0.240		
			RPD	48			0.1			0.0			27			8		
NA	11-0419-129-PB-Pu	1211012	05/18/11	(ND)			(ND)			(ND)			(ND)			(ND)		
TI002-A4-B	11-0420-129-S-Pu	1211013	05/25/11	0.330	± 0.154	46.5	85.7	± 0.4	0.5	13.2	± 0.2	1.18	0.531	± 0.042	8.0	0.253	± 0.036	14.0
TI002-A4-C	11-0420-129-D-Pu	1211014	05/25/11	0.232	± 0.094	40.4	86.1	± 0.4	0.4	13.0	± 0.2	1.65	0.507	± 0.032	6.3	0.228	± 0.025	10.9
			Average =	0.281			85.9			13.1			0.519			0.240		
			RPD	35			0.4			2			5			11		
TI002-B1-B	11-0421-129-S-Pu	1211015	05/18/11	0.079	± 0.026	33.6	86.4	± 0.5	0.5	12.8	± 0.3	2.50	0.486	± 0.075	15.4	0.227	± 0.034	14.9
TI002-B1-C	11-0421-129-D-Pu	1211016	05/25/11	0.142	± 0.034	24.0	86.2	± 0.4	0.5	13.0	± 0.3	2.31	0.484	± 0.044	9.0	0.234	± 0.030	13.0
			Average =	0.110			86.3			12.9			0.485			0.230		
			RPD	57			0.3			1			0.3			3		
TI001-B3-B	11-0422-129-S-Pu	1211017	05/18/11	0.100	± 0.028	27.6	86.2	± 0.5	0.6	12.9	± 0.3	2.53	0.535	± 0.102	19.0	0.229	± 0.037	16.3
TI001-B3-C	11-0422-129-D-Pu	1211018	05/18/11	0.235	± 0.137	58.1	85.9	± 1.1	1.3	13.2	± 0.7	5.31	0.521	± 0.201	38.6	0.209	± 0.081	38.7
			Average =	0.167			86.0			13.0			0.528			0.219		
			RPD	81			0.4			2			3			9		

Shaded RPD's indicate the values >20%; in each case, one or more of the duplicate values were less than the 1 atom percent threshold where the 20% acceptance criterion applies (QAPjP/SAP KBC-33786, Rev. 2).

The Process Blank had inadequate signal at the major analyte mass attempt perform isotopic analysis reported as ND not detected.

Table 4. Plutonium Isotopic Analysis in KW210 Core Samples
By Thermal Ionization Mass Spectrometry

Summary of Standard Results - Standard: Pu-946																	
			Pu-238			Pu-239			Pu-240			Pu-241			Pu-242		
	Run Date	Filament	Atom%	1−σ	RSD(%)	Atom%	1−σ	RSD(%)	Atom%	1−σ	RSD(%)	Atom%	1−σ	RSD(%)	Atom%	1−σ	RSD(%)
Cert Value	n/a	n/a	0.188	± 0.007	n/a	86.134	± 0.015	n/a	12.480	± 0.015	n/a	0.613	± 0.005	n/a	0.586	± 0.003	n/a
Pooled	n/a	n/a	0.188	± 0.005	2.79	86.18	± 0.052	0.06	12.44	± 0.052	0.42	0.617	± 0.0079	1.27	0.583	± 0.008	1.45
		%Difference =	0.3			0.05			-0.354			0.7			-0.4		
		Acceptable range	0.176 to 0.199			80.97 to 91.30			11.73 to 13.23			0.576 to 0.650			0.550 to 0.621		
1	05/17/11	9461106.1	0.189	± 0.007	3.59	86.23	0.12	0.13	12.39	0.07	0.60	0.620	± 0.020	3.27	0.575	0.014	2.42
2	05/17/11	9461106.2	0.184	± 0.006	3.12	86.21	0.08	0.10	12.41	0.05	0.42	0.612	± 0.012	1.96	0.583	0.014	2.37
3	05/17/11	9461106.3	0.181	± 0.009	4.89	86.14	0.14	0.16	12.47	0.09	0.75	0.631	± 0.023	3.57	0.584	0.015	2.61
4	05/18/11	9461107.1	0.188	± 0.012	6.19	86.19	0.23	0.26	12.45	0.18	1.46	0.609	± 0.017	2.83	0.571	0.017	2.91
5	05/18/11	9461107.2	0.196	± 0.012	5.89	86.21	0.15	0.17	12.40	0.09	0.74	0.609	± 0.021	3.48	0.584	0.023	3.87
6	05/18/11	9461108.1	0.196	± 0.039	20.00	86.24	0.44	0.50	12.35	0.28	2.29	0.618	± 0.085	13.7	0.588	0.028	4.83
7	05/25/11	9461109.1	0.187	± 0.007	4.00	86.10	0.20	0.23	12.53	0.15	1.23	0.609	± 0.021	3.52	0.579	0.017	2.97
8	05/25/11	9461109.2	0.189	± 0.022	11.50	86.11	0.24	0.28	12.48	0.15	1.20	0.622	± 0.034	5.46	0.600	0.035	5.91
9	05/25/11	94611010.1	0.184	± 0.011	5.90	86.16	0.17	0.19	12.44	0.12	0.95	0.625	± 0.022	3.55	0.588	0.014	2.34

The individual and pooled results for all plutonium isotopes meet the acceptance criteria of ±6% of the certified value.
Pu-946 isotope values are decay corrected to 5-17-2011.

RPL K Basin Project 52578	Document Review Record

Title:	Plutonium and Uranium Isotopic Analysis for KW210 Core Samples

Please return the completed form to:		Comments Due:
Organization/Department:	**Designated Reviewer:**	**Reviewer Signature/Date:** (Upon completion of review)
D9H63	JC Braley	Jenifer C Braley

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	1/1	E	A 'was' was present when it should be a 'were'	corrected S J Bos
2	1/1	E	table 3 should be Table 3	corrected S J Bos
3	1/2	E	Period after one Rev and not the other, should be stricken for consistency	Corrected S J Bos
4	1/3	E	Table 3-7 should be Tables 3-7	Corrected S J Bos
5				
6				
7				
8				
END				

Concur with comment resolution, review complete.	Comments Resolved By:
Jenifer C Braley 7/13/11	S J Bos 7/14/11
Reviewer Signature Date	Author Signature Date

I reviewed the underpinning spreadsheet (data entries and calculations for file KW210 TIMS data June 2011.xls) and data incorporation to the report. Discussions in the report are accurate and the conclusions are sound. I also acknowledge that while Sandy Fiskum suggested the verbiage for this disclosure, I, Jenifer C. Braley, concur with this statement.

Title:	**TIMS Isotopic Data for KW Basin Core KW230 (?)**
File Name	
Revision Number:	1
Date Prepared:	6/15/2011
Prepared by:	Stan Bos
General Statement of Purpose:	Spreadsheet calculates renormalizes data when run cycles or isotopes must be removed for the data reduction, uncertainties and presents the data in a format that can be pasted into reports .
General Description of Approach:	
Urainium std	Uranium isotopic standards
U TIMS	Isotope ratio analysis of the urainium fractions
Final Results 1	Report table for plutioium fractions

Plutonium std	Plutonium 946 isotope ratio standards
Pu TIMS	Isotope ratio analysis of the plutonium fractions
Final Results 2	Report table for plutioium fractions

TIMS Isotopic Standard Worksheet

Select Isotopes to Report	U-234	U-235	U-236	U-238

	At% U-234	At% U-235	At% U-236	At% U-238
Pooled Average	0.019107	3.019149	0.019868	96.941876
Pooled STD	0.002427	0.029286	0.002484	0.034197
RSD (%)	12.70	0.97	12.50	0.04

1

Output Results -- TIMS CEC21-703 (M&TE 4678061)					
No.	Std ID/Filament ID/Date	Use?	U-234 Ratio	U-235 Ratio	U-236 Ratio	U-238 Ratio
1	U-030	Yes	0.0001724	0.0308798	0.0001998	1.0000000
	0401114.1	Yes	0.0001539	0.0306582	0.0002461	1.0000000
	05/03/11	Yes	0.0001774	0.0312367	0.0002200	1.0000000
		Yes	0.0002066	0.0313486	0.0002818	1.0000000
		Yes	0.0002068	0.0312343	0.0001997	1.0000000
		Yes	0.0001960	0.0305761	0.0002075	1.0000000
		Yes	0.0002131	0.0309255	0.0001794	1.0000000
		Yes	0.0002312	0.0308760	0.0002154	1.0000000
		Yes	0.0001791	0.0308527	0.0002517	1.0000000
	TIMS Ratio Average		**0.0001929**	**0.0309542**	**0.0002224**	**1.0000000**
	TIMS Ratio Std Dev		**0.0000241**	**0.0002663**	**0.0000318**	**0.0003222**
	TIMS Avg At%		**0.0187076**	**3.0012726**	**0.0215614**	**96.9584584**
	Avg At% Std Dev		**0.0023344**	**0.0258164**	**0.0030853**	**0.0312361**
	%RSD		**12.48**	**0.86**	**14.31**	**0.03**
	Comments:					

Reported Results -- TIMS CEC21-703 (M&TE 4678061)				
U-234 Ratio	U-235 Ratio	U-236 Ratio	U-238 Ratio
0.0001724	0.0308798	0.0001998	1.0000000
0.0001539	0.0306582	0.0002461	1.0000000
0.0001774	0.0312367	0.0002200	1.0000000
0.0002066	0.0313486	0.0002818	1.0000000
0.0002068	0.0312343	0.0001997	1.0000000
0.0001960	0.0305761	0.0002075	1.0000000
0.0002131	0.0309255	0.0001794	1.0000000
0.0002312	0.0308760	0.0002154	1.0000000
0.0001791	0.0308527	0.0002517	1.0000000
0.0001929	**0.0309542**	**0.0002224**	**1.0000000**
0.0000241	**0.0002663**	**0.0000318**	**0.0003222**
0.0187076	**3.0012726**	**0.0215614**	**96.9584584**
0.0023344	**0.0258164**	**0.0030853**	**0.0312361**
12.48	**0.86**	**14.31**	**0.03**

Ratio Average
Ratio Std Dev
Avg At%
Avg At% SD
%RSD

Calculated At% for Each Individual Scan (For Reference Only)				
Indiv. Ratio Sum	At% U-234	At% U-235	At% U-236	At% U-238
1.031252	0.0167	2.9944	0.0194	96.9695
1.031058	0.0149	2.9755	0.0239	96.9877
1.031644	0.0172	3.0279	0.0213	96.9336
1.031837	0.0200	3.0381	0.0273	96.9145
1.031641	0.0200	3.0276	0.0194	96.9330
1.030980	0.0190	2.9657	0.0201	96.9951
1.031318	0.0207	2.9986	0.0174	96.9633
1.031323	0.0224	2.9938	0.0209	96.9629
1.031264	0.0174	2.9917	0.0244	96.9665

Results based on At% from individual Scans (For Reference Use Only)				
At% Average	0.0187	3.0013	0.0216	96.9585
At% Std Dev	0.0023	0.0250	0.0031	0.0304
%RSD	12.47	0.83	14.30	0.03

2

Output Results -- TIMS CEC21-703 (M&TE 4678061)					
No.	Std ID/Filament ID/Date	Use?	U-234 Ratio	U-235 Ratio	U-236 Ratio	U-238 Ratio
2	U-030	No	0.0001608	0.0285448	0.0023970	1.0000000
	0401116.1	Yes	0.0001702	0.0308260	0.0001655	1.0000000
	05/06/11	Yes	0.0002109	0.0310466	0.0001864	1.0000000
		Yes	0.0002290	0.0307557	0.0002278	1.0000000
		Yes	0.0002337	0.0314748	0.0002030	1.0000000
		Yes	0.0002009	0.0313715	0.0001987	1.0000000
		Yes	0.0001689	0.0307662	0.0002154	1.0000000
		Yes	0.0001782	0.0315104	0.0001879	1.0000000
		Yes	0.0002153	0.0311957	0.0002103	1.0000000
	TIMS Ratio Average		**0.0001964**	**0.0308324**	**0.0004436**	**1.0000000**
	TIMS Ratio Std Dev		**0.0000276**	**0.0009872**	**0.0007328**	**0.0016676**
	TIMS Avg At%		**0.0190440**	**2.9891649**	**0.0430022**	**96.9487889**
	Avg At% Std Dev		**0.0026734**	**0.0879532**	**0.0710489**	**0.1616674**
	%RSD		**14.04**	**2.94**	**165.20**	**0.17**
	Comments:					

Reported Results -- TIMS CEC21-703 (M&TE 4678061)				
U-234 Ratio	U-235 Ratio	U-236 Ratio	U-238 Ratio
0.0001702	0.0308260	0.0001655	1.0000000
0.0002109	0.0310466	0.0001864	1.0000000
0.0002290	0.0307557	0.0002278	1.0000000
0.0002337	0.0314748	0.0002030	1.0000000
0.0002009	0.0313715	0.0001987	1.0000000
0.0001689	0.0307662	0.0002154	1.0000000
0.0001782	0.0315104	0.0001879	1.0000000
0.0002153	0.0311957	0.0002103	1.0000000
0.0002069	**0.0311184**	**0.0001994**	**1.0000000**
0.0000258	**0.0003155**	**0.0000194**	**0.0003508**
0.0194749	**3.0167524**	**0.0193283**	**96.9444444**
0.0024999	**0.0305901**	**0.0018846**	**0.0349745**
12.84	**1.01**	**9.75**	**0.04**

Ratio Average
Ratio Std Dev
Avg At%
Avg At% SD
%RSD

Calculated At% for Each Individual Scan (For Reference Only)				
Indiv. Ratio Sum	At% U-234	At% U-235	At% U-236	At% U-238
1.031162	0.0165	2.9894	0.0160	96.9780
1.031444	0.0204	3.0100	0.0181	96.9515
1.031213	0.0222	2.9825	0.0221	96.9732
1.031912	0.0226	3.0501	0.0197	96.9075
1.031771	0.0195	3.0405	0.0193	96.9207
1.031151	0.0164	2.9837	0.0209	96.9791
1.031877	0.0173	3.0537	0.0182	96.9108
1.031621	0.0209	3.0239	0.0204	96.9348

Results based on At% from individual Scans (For Reference Use Only)				
At% Average	0.0195	3.0167	0.0193	96.9445
At% Std Dev	0.0025	0.0297	0.0019	0.0340
%RSD	12.83	0.98	9.76	0.04

3

Output Results -- TIMS CEC21-703 (M&TE 4678061)					
No.	Std ID/Filament ID/Date	Use?	U-234 Ratio	U-235 Ratio	U-236 Ratio	U-238 Ratio
3	U-030	Yes	0.0001801	0.0315838	0.0001869	1.0000000
	0401117.1	Yes	0.0002123	0.0309041	0.0002033	1.0000000
	05/06/11	Yes	0.0002069	0.0309864	0.0001665	1.0000000
		Yes	0.0001839	0.0313066	0.0002222	1.0000000
		Yes	0.0001904	0.0316178	0.0002065	1.0000000
		Yes	0.0001924	0.0311908	0.0001908	1.0000000
		Yes	0.0002359	0.0312499	0.0001478	1.0000000
		Yes	0.0002347	0.0312294	0.0001703	1.0000000
		Yes	0.0002292	0.0312850	0.0001573	1.0000000
	TIMS Ratio Average		**0.0002073**	**0.0312615**	**0.0001835**	**1.0000000**
	TIMS Ratio Std Dev		**0.0000220**	**0.0002353**	**0.0000248**	**0.0002821**
	TIMS Avg At%		**0.0200951**	**3.0302391**	**0.0177881**	**96.9318777**
	Avg At% Std Dev		**0.0021315**	**0.0228097**	**0.0024008**	**0.0273421**
	%RSD		**10.61**	**0.75**	**13.50**	**0.03**
	Comments:					

Reported Results -- TIMS CEC21-703 (M&TE 4678061)				
U-234 Ratio	U-235 Ratio	U-236 Ratio	U-238 Ratio
0.0001801	0.0315838	0.0001869	1.0000000
0.0002123	0.0309041	0.0002033	1.0000000
0.0002069	0.0309864	0.0001665	1.0000000
0.0001839	0.0313066	0.0002222	1.0000000
0.0001904	0.0316178	0.0002065	1.0000000
0.0001924	0.0311908	0.0001908	1.0000000
0.0002359	0.0312499	0.0001478	1.0000000
0.0002347	0.0312294	0.0001703	1.0000000
0.0002292	0.0312850	0.0001573	1.0000000
0.0002073	**0.0312615**	**0.0001835**	**1.0000000**
0.0000220	**0.0002353**	**0.0000248**	**0.0002821**
0.0200951	**3.0302391**	**0.0177881**	**96.9318777**
0.0021315	**0.0228097**	**0.0024008**	**0.0273421**
10.61	**0.75**	**13.50**	**0.03**

Ratio Average
Ratio Std Dev
Avg At%
Avg At% SD
%RSD

Calculated At% for Each Individual Scan (For Reference Only)				
Indiv. Ratio Sum	At% U-234	At% U-235	At% U-236	At% U-238
1.031951	0.0175	3.0606	0.0181	96.9038
1.031320	0.0205	2.9966	0.0197	96.9631
1.031360	0.0201	3.0044	0.0161	96.9594
1.031733	0.0178	3.0344	0.0215	96.9262
1.032015	0.0184	3.0637	0.0200	96.8978
1.031574	0.0187	3.0236	0.0185	96.9392
1.031644	0.0229	3.0292	0.0143	96.9336
1.031634	0.0228	3.0272	0.0165	96.9336
1.031672	0.0222	3.0325	0.0152	96.9301

Results based on At% from individual Scans (For Reference Use Only)				
At% Average	0.0201	3.0302	0.0178	96.9319
At% Std Dev	0.0021	0.0221	0.0024	0.0267
%RSD	10.62	0.73	13.49	0.03

4

Output Results -- TIMS CEC21-703 (M&TE 4678061)					
No.	Std ID/Filament ID/Date	Use?	U-234 Ratio	U-235 Ratio	U-236 Ratio	U-238 Ratio
4	U-030	Yes	0.0001714	0.0304685	0.0001792	1.0000000
	0401118.1	Yes	0.0002171	0.0311156	0.0001936	1.0000000
	05/09/11	Yes	0.0001252	0.0312051	0.0001929	1.0000000
		Yes	0.0002064	0.0314997	0.0002045	1.0000000
		Yes	0.0002314	0.0315185	0.0001904	1.0000000
		Yes	0.0002047	0.0313511	0.0002276	1.0000000
		Yes	0.0001689	0.0315855	0.0002101	1.0000000
		Yes	0.0002152	0.0304205	0.0002072	1.0000000
		Yes	0.0002010	0.0311937	0.0002213	1.0000000
	TIMS Ratio Average		**0.0001935**	**0.0311509**	**0.0002030**	**1.0000000**
	TIMS Ratio Std Dev		**0.0000327**	**0.0004317**	**0.0000155**	**0.0004799**
	TIMS Avg At%		**0.0187561**	**3.0198236**	**0.0196770**	**96.9417433**
	Avg At% Std Dev		**0.0031730**	**0.0418497**	**0.0015041**	**0.0465269**
	%RSD		**16.92**	**1.39**	**7.64**	**0.05**
	Comments:					

Reported Results -- TIMS CEC21-703 (M&TE 4678061)				
U-234 Ratio	U-235 Ratio	U-236 Ratio	U-238 Ratio
0.0001714	0.0304685	0.0001792	1.0000000
0.0002171	0.0311156	0.0001936	1.0000000
0.0001252	0.0312051	0.0001929	1.0000000
0.0002064	0.0314997	0.0002045	1.0000000
0.0002314	0.0315185	0.0001904	1.0000000
0.0002047	0.0313511	0.0002276	1.0000000
0.0001689	0.0315855	0.0002101	1.0000000
0.0002152	0.0304205	0.0002072	1.0000000
0.0002010	0.0311937	0.0002213	1.0000000
0.0001935	**0.0311509**	**0.0002030**	**1.0000000**
0.0000327	**0.0004317**	**0.0000155**	**0.0004799**
0.0187561	**3.0198236**	**0.0196770**	**96.9417433**
0.0031730	**0.0418497**	**0.0015041**	**0.0465269**
16.92	**1.39**	**7.64**	**0.05**

Ratio Average
Ratio Std Dev
Avg At%
Avg At% SD
%RSD

Calculated At% for Each Individual Scan (For Reference Only)				
Indiv. Ratio Sum	At% U-234	At% U-235	At% U-236	At% U-238
1.030819	0.0166	2.9558	0.0174	97.0102
1.031526	0.0210	3.0165	0.0188	96.9437
1.031523	0.0121	3.0251	0.0187	96.9440
1.031911	0.0200	3.0526	0.0198	96.9076
1.031940	0.0224	3.0543	0.0185	96.9048
1.031783	0.0198	3.0385	0.0221	96.9196
1.031965	0.0164	3.0607	0.0204	96.9026
1.030843	0.0209	2.9510	0.0201	97.0080
1.031616	0.0195	3.0238	0.0215	96.9353

Results based on At% from individual Scans (For Reference Use Only)				
At% Average	0.0188	3.0198	0.0197	96.9418
At% Std Dev	0.0032	0.0406	0.0015	0.0453
%RSD	16.91	1.34	7.63	0.05

5

Output Results -- TIMS CEC21-703 (M&TE 4678061)					
No.	Std ID/Filament ID/Date	Use?	U-234 Ratio	U-235 Ratio	U-236 Ratio	U-238 Ratio
5	U-030	Yes	0.0002232	0.0313623	0.0002166	1.0000000
	0401119.1	Yes	0.0002029	0.0312000	0.0002143	1.0000000
	05/09/11	Yes	0.0001916	0.0316136	0.0002108	1.0000000
		Yes	0.0002075	0.0309867	0.0002099	1.0000000
		Yes	0.0001829	0.0309076	0.0002145	1.0000000
		Yes	0.0002027	0.0313242	0.0002487	1.0000000
		Yes	0.0001810	0.0312149	0.0002455	1.0000000
		Yes	0.0001806	0.0314781	0.0002047	1.0000000
		Yes	0.0001491	0.0310020	0.0001881	1.0000000
	TIMS Ratio Average		**0.0001913**	**0.0312322**	**0.0002159**	**1.0000000**
	TIMS Ratio Std Dev		**0.0000213**	**0.0002375**	**0.0000174**	**0.0002762**
	TIMS Avg At%		**0.0185411**	**3.0274297**	**0.0209279**	**96.9331013**
	Avg At% Std Dev		**0.0020610**	**0.0230225**	**0.0016878**	**0.0267713**
	%RSD		**11.12**	**0.76**	**8.06**	**0.03**
	Comments:					

Reported Results -- TIMS CEC21-703 (M&TE 4678061)				
U-234 Ratio	U-235 Ratio	U-236 Ratio	U-238 Ratio
0.0002232	0.0313623	0.0002166	1.0000000
0.0002029	0.0312000	0.0002143	1.0000000
0.0001916	0.0316136	0.0002108	1.0000000
0.0002075	0.0309867	0.0002099	1.0000000
0.0001829	0.0309076	0.0002145	1.0000000
0.0002027	0.0313242	0.0002487	1.0000000
0.0001810	0.0312149	0.0002455	1.0000000
0.0001806	0.0314781	0.0002047	1.0000000
0.0001491	0.0310020	0.0001881	1.0000000
0.0001913	**0.0312322**	**0.0002159**	**1.0000000**
0.0000213	**0.0002375**	**0.0000174**	**0.0002762**
0.0185411	**3.0274297**	**0.0209279**	**96.9331013**
0.0020610	**0.0230225**	**0.0016878**	**0.0267713**
11.12	**0.76**	**8.06**	**0.03**

Ratio Average
Ratio Std Dev
Avg At%
Avg At% SD
%RSD

Calculated At% for Each Individual Scan (For Reference Only)				
Indiv. Ratio Sum	At% U-234	At% U-235	At% U-236	At% U-238
1.031802	0.0216	3.0396	0.0210	96.9178
1.031617	0.0197	3.0244	0.0208	96.9352
1.032016	0.0186	3.0633	0.0204	96.8977
1.031404	0.0201	3.0043	0.0204	96.9552
1.031305	0.0177	2.9969	0.0208	96.9645
1.031776	0.0196	3.0360	0.0241	96.9203
1.031631	0.0175	3.0258	0.0228	96.9338
1.031863	0.0175	3.0506	0.0198	96.9121
1.031339	0.0145	3.0060	0.0182	96.9613

Results based on At% from individual Scans (For Reference Use Only)				
At% Average	0.0185	3.0274	0.0209	96.9331
At% Std Dev	0.0021	0.0223	0.0017	0.0260
%RSD	11.11	0.74	8.06	0.03

PNNL - Radiochemical Processing and Engineering Group

Procedure: PNNL-98323-264, Rev 2

Summary of Standard Results - Standard: U-030

	Run Date	Filament	U-234			U-235			U-236			U-238					
			Atom%	1-σ	RSD(%)	Atom%	1-σ	RSD(%)	Atom%	1-σ	RSD(%)	Atom%	1-σ	RSD(%)	Atom%	1-σ	RSD(%)
Cert Value	n/a	n/a	0.0190	± 0.0001	n/a	3.046	± 0.0030	n/a	0.0204	± 0.0010	n/a	96.915	± 0.003	n/a			n/a
Pooled	n/a	n/a	0.0191	± 0.0007	3.42	3.019	± 0.0114	0.38	0.0199	± 0.0015	7.40	96.942	± 0.011	0.01			
	%Difference =		0.61			-0.883			-2.66			0.028					
1	05/03/11	0301114.1	0.0187	± 0.0023	12.48	3.001	± 0.0258	0.86	0.0216	± 0.0031	14.31	96.958	± 0.031	0.03			
2	05/06/11	0301116.1	0.0195	± 0.0025	12.84	3.017	± 0.0306	1.01	0.0193	± 0.0019	9.75	96.944	± 0.035	0.04			
3	05/06/11	0301117.1	0.0201	± 0.0021	10.61	3.030	± 0.0228	0.75	0.0178	± 0.0024	13.50	96.932	± 0.027	0.03			
4	05/09/11	0301118.1	0.0188	± 0.0032	16.92	3.020	± 0.0418	1.39	0.0197	± 0.0015	7.64	96.942	± 0.047	0.05			
5	05/09/11	0301119.1	0.0185	± 0.0021	11.12	3.027	± 0.0230	0.76	0.0209	± 0.0017	8.06	96.933	± 0.027	0.03			

Page 1 of 1

TIMS Isotopic Sample Worksheet

Select Isotopes to Report	U-233	U-234	U-235	U-236	U-238

Output Results – TIMS CEC21-703 (M&TE 4678061)

No.	Smpl ID/Filament ID/Date	Use?	U-233 Ratio	U-234 Ratio	U-235 Ratio	U-236 Ratio	U-238 Ratio
1	11-0419-129-S-U	Yes	0.0000000	0.0000745	0.0073871	0.0008011	1.0000000
	1111010	Yes	0.0000000	0.0000758	0.0071360	0.0006794	1.0000000
	05/06/11	Yes	0.0000000	0.0000748	0.0071318	0.0006903	1.0000000
		Yes	0.0000000	0.0000786	0.0071274	0.0007567	1.0000000
		Yes	0.0000000	0.0000998	0.0070314	0.0007477	1.0000000
		Yes	0.0000000	0.0000670	0.0072920	0.0008204	1.0000000
		Yes	0.0000000	0.0000909	0.0070186	0.0006840	1.0000000
		Yes	0.0000000	0.0000625	0.0071631	0.0007198	1.0000000
		Yes	0.0000000	0.0000947	0.0072125	0.0007537	1.0000000
	TIMS Ratio Average		**0.0000000**	**0.0000798**	**0.0071667**	**0.0007392**	**1.0000000**
	TIMS Ratio Std Dev		**0.0000000**	**0.0000126**	**0.0001174**	**0.0000505**	**0.0001806**
	TIMS Avg At%		**0.0000000**	**0.0079212**	**0.7109878**	**0.0733377**	**99.2077533**
	Avg At% Std Dev		**#DIV/0!**	**0.0012543**	**0.0116507**	**0.0050103**	**0.0179153**
	%RSD		**#DIV/0!**	**15.84**	**1.64**	**6.83**	**0.02**
	Comments:						

Reported Results – TIMS CEC21-703 (M&TE 4678061)

U-233 Ratio	U-234 Ratio	U-235 Ratio	U-236 Ratio	U-238 Ratio
0.0000000	0.0000745	0.0073871	0.0008011	1.0000000
0.0000000	0.0000758	0.0071360	0.0006794	1.0000000
0.0000000	0.0000748	0.0071318	0.0006903	1.0000000
0.0000000	0.0000786	0.0071274	0.0007567	1.0000000
0.0000000	0.0000998	0.0070314	0.0007477	1.0000000
0.0000000	0.0000670	0.0072920	0.0008204	1.0000000
0.0000000	0.0000909	0.0070186	0.0006840	1.0000000
0.0000000	0.0000625	0.0071631	0.0007198	1.0000000
0.0000000	0.0000947	0.0072125	0.0007537	1.0000000
Ratio Average	**0.0000000**	**0.0000798**	**0.0071667**	**0.0007392**	**1.0000000**
Ratio Std Dev	**0.0000000**	**0.0000126**	**0.0001174**	**0.0000505**	**0.0001806**
Avg At%	**0.0000000**	**0.0079212**	**0.7109878**	**0.0733377**	**99.2077533**
Avg At% SD	**#DIV/0!**	**0.0012543**	**0.0116507**	**0.0050103**	**0.0179153**
%RSD	**#DIV/0!**	**15.84**	**1.64**	**6.83**	**0.02**

Calculated At% for Each Individual Scan (For Reference Only)

Indiv. Ratio Sum	At% U-233	At% U-234	At% U-235	At% U-236	At% U-238
1.008263	0.0000	0.0074	0.7327	0.0795	99.1805
1.007891	0.0000	0.0075	0.7080	0.0674	99.2171
1.007897	0.0000	0.0074	0.7076	0.0685	99.2165
1.007963	0.0000	0.0078	0.7071	0.0751	99.2100
1.007879	0.0000	0.0099	0.6976	0.0742	99.2183
1.008179	0.0000	0.0066	0.7233	0.0814	99.1887
1.007794	0.0000	0.0090	0.6964	0.0679	99.2267
1.007945	0.0000	0.0062	0.7107	0.0714	99.2117
1.008061	0.0000	0.0094	0.7155	0.0748	99.2004

Results based on At% from individual Scans (For Reference Use Only)

At% Average	0.0000	0.0079	0.7110	0.0733	99.2078
At% Std Dev	0.0000	0.0013	0.0115	0.0050	0.0178
%RSD	#DIV/0!	15.84	1.62	6.82	0.02

Output Results – TIMS CEC21-703 (M&TE 4678061)

No.	Smpl ID/Filament ID/Date	Use?	U-233 Ratio	U-234 Ratio	U-235 Ratio	U-236 Ratio	U-238 Ratio
2	11-0419-129-D-U	Yes	0.0000000	0.0000507	0.0072942	0.0007406	1.0000000
	1111011	Yes	0.0000000	0.0000764	0.0071751	0.0007371	1.0000000
	05/06/11	Yes	0.0000000	0.0000657	0.0071938	0.0007816	1.0000000
		Yes	0.0000000	0.0000531	0.0070837	0.0008105	1.0000000
		Yes	0.0000000	0.0000588	0.0070954	0.0007680	1.0000000
		Yes	0.0000000	0.0000683	0.0070771	0.0007373	1.0000000
		Yes	0.0000000	0.0000796	0.0071418	0.0007686	1.0000000
		Yes	0.0000000	0.0000760	0.0072212	0.0007510	1.0000000
		Yes	0.0000000	0.0000783	0.0070528	0.0007698	1.0000000
	TIMS Ratio Average		**0.0000000**	**0.0000674**	**0.0071483**	**0.0007627**	**1.0000000**
	TIMS Ratio Std Dev		**0.0000000**	**0.0000111**	**0.0000795**	**0.0000242**	**0.0001148**
	TIMS Avg At%		**0.0000000**	**0.0066900**	**0.7091763**	**0.0756685**	**99.2084653**
	Avg At% Std Dev		**#DIV/0!**	**0.0011001**	**0.0078895**	**0.0023998**	**0.0113894**
	%RSD		**#DIV/0!**	**16.44**	**1.11**	**3.17**	**0.01**
	Comments:						

Reported Results – TIMS CEC21-703 (M&TE 4678061)

U-233 Ratio	U-234 Ratio	U-235 Ratio	U-236 Ratio	U-238 Ratio
0.0000000	0.0000507	0.0072942	0.0007406	1.0000000
0.0000000	0.0000764	0.0071751	0.0007371	1.0000000
0.0000000	0.0000657	0.0071938	0.0007816	1.0000000
0.0000000	0.0000531	0.0070837	0.0008105	1.0000000
0.0000000	0.0000588	0.0070954	0.0007680	1.0000000
0.0000000	0.0000683	0.0070771	0.0007373	1.0000000
0.0000000	0.0000796	0.0071418	0.0007686	1.0000000
0.0000000	0.0000760	0.0072212	0.0007510	1.0000000
0.0000000	0.0000783	0.0070528	0.0007698	1.0000000
Ratio Average	**0.0000000**	**0.0000674**	**0.0071483**	**0.0007627**	**1.0000000**
Ratio Std Dev	**0.0000000**	**0.0000111**	**0.0000795**	**0.0000242**	**0.0001148**
Avg At%	**0.0000000**	**0.0066900**	**0.7091763**	**0.0756685**	**99.2084653**
Avg At% SD	**#DIV/0!**	**0.0011001**	**0.0078895**	**0.0023998**	**0.0113894**
%RSD	**#DIV/0!**	**16.44**	**1.11**	**3.17**	**0.01**

Indiv. Ratio Sum	At% U-233	At% U-234	At% U-235	At% U-236	At% U-238
1.008086	0.0000	0.0050	0.7236	0.0735	99.1979
1.007989	0.0000	0.0076	0.7118	0.0731	99.2075
1.008041	0.0000	0.0065	0.7136	0.0775	99.2023
1.007947	0.0000	0.0053	0.7028	0.0804	99.2115
1.007922	0.0000	0.0058	0.7040	0.0762	99.2140
1.007883	0.0000	0.0068	0.7022	0.0732	99.2179
1.007990	0.0000	0.0079	0.7085	0.0763	99.2073
1.008048	0.0000	0.0075	0.7164	0.0745	99.2016
1.007901	0.0000	0.0078	0.6998	0.0764	99.2161

Results based on At% from individual Scans (For Reference Use Only)

At% Average	0.0000	0.0067	0.7092	0.0757	99.2085
At% Std Dev	0.0000	0.0011	0.0078	0.0024	0.0113
%RSD	#DIV/0!	16.44	1.11	3.17	0.01

Output Results – TIMS CEC21-703 (M&TE 4678061)

No.	Smpl ID/Filament ID/Date	Use?	U-233 Ratio	U-234 Ratio	U-235 Ratio	U-236 Ratio	U-238 Ratio
3	11-0419-129-PB-U	No					
	1111012	No					
	05/06/11	No					
		No					
		No					
		No					
		No					
		No					
	TIMS Ratio Average						
	TIMS Ratio Std Dev						
	TIMS Avg At%						
	Avg At% Std Dev						
	%RSD						
	Insufficient analyte to run isotopic analysis.						

Reported Results – TIMS CEC21-703 (M&TE 4678061)

U-233 Ratio	U-234 Ratio	U-235 Ratio	U-236 Ratio	U-238 Ratio
Ratio Average				
Ratio Std Dev				
Avg At%				
Avg At% SD				
%RSD				

Indiv. Ratio Sum	At% U-233	At% U-234	At% U-235	At% U-236	At% U-238

Results based on At% from individual Scans (For Reference Use Only)

At% Average					
At% Std Dev					
%RSD					

Output Results – TIMS CEC21-703 (M&TE 4678061)

No.	Smpl ID/Filament ID/Date	Use?	U-233 Ratio	U-234 Ratio	U-235 Ratio	U-236 Ratio	U-238 Ratio
4	11-0420-129-S-U	Yes	0.0000000	0.0000460	0.0071569	0.0006965	1.0000000
	11111013	Yes	0.0000000	0.0000370	0.0072004	0.0006575	1.0000000
	05/06/11	Yes	0.0000000	0.0000893	0.0070636	0.0006696	1.0000000
		Yes	0.0000000	0.0000409	0.0069314	0.0007204	1.0000000
		Yes	0.0000000	0.0001148	0.0072038	0.0007654	1.0000000
		Yes	0.0000000	0.0000588	0.0070515	0.0007196	1.0000000
		Yes	0.0000000	0.0000775	0.0071077	0.0007614	1.0000000
		Yes	0.0000000	0.0001026	0.0072254	0.0007657	1.0000000
		Yes	0.0000000	0.0000526	0.0066953	0.0007737	1.0000000
	TIMS Ratio Average		**0.0000000**	**0.0000688**	**0.0070707**	**0.0007255**	**1.0000000**
	TIMS Ratio Std Dev		**0.0000000**	**0.0000283**	**0.0001688**	**0.0000440**	**0.0002411**
	TIMS Avg At%		**0.0000000**	**0.0068296**	**0.7015490**	**0.0719872**	**99.2196343**
	Avg At% Std Dev		**#DIV/0!**	**0.0028121**	**0.0167509**	**0.0043632**	**0.0239262**
	%RSD		**#DIV/0!**	**41.18**	**2.39**	**6.06**	**0.02**
	Comments:						

Reported Results – TIMS CEC21-703 (M&TE 4678061)

U-233 Ratio	U-234 Ratio	U-235 Ratio	U-236 Ratio	U-238 Ratio
0.0000000	0.0000460	0.0071569	0.0006965	1.0000000
0.0000000	0.0000370	0.0072004	0.0006575	1.0000000
0.0000000	0.0000893	0.0070636	0.0006696	1.0000000
0.0000000	0.0000409	0.0069314	0.0007204	1.0000000
0.0000000	0.0001148	0.0072038	0.0007654	1.0000000
0.0000000	0.0000588	0.0070515	0.0007196	1.0000000
0.0000000	0.0000775	0.0071077	0.0007614	1.0000000
0.0000000	0.0001026	0.0072254	0.0007657	1.0000000
0.0000000	0.0000526	0.0066953	0.0007737	1.0000000
Ratio Average	**0.0000000**	**0.0000688**	**0.0070707**	**0.0007255**	**1.0000000**
Ratio Std Dev	**0.0000000**	**0.0000283**	**0.0001688**	**0.0000440**	**0.0002411**
Avg At%	**0.0000000**	**0.0068296**	**0.7015490**	**0.0719872**	**99.2196343**
Avg At% SD	**#DIV/0!**	**0.0028121**	**0.0167509**	**0.0043632**	**0.0239262**
%RSD	**#DIV/0!**	**41.18**	**2.39**	**6.06**	**0.02**

Indiv. Ratio Sum	At% U-233	At% U-234	At% U-235	At% U-236	At% U-238
1.007899	0.0000	0.0046	0.7101	0.0691	99.2163
1.007895	0.0000	0.0037	0.7144	0.0652	99.2167
1.007823	0.0000	0.0089	0.7009	0.0664	99.2238
1.007693	0.0000	0.0041	0.6878	0.0715	99.2366
1.008084	0.0000	0.0114	0.7146	0.0759	99.1981
1.007830	0.0000	0.0058	0.6997	0.0714	99.2231
1.007947	0.0000	0.0077	0.7052	0.0755	99.2116
1.008094	0.0000	0.0102	0.7167	0.0760	99.1971
1.007522	0.0000	0.0052	0.6645	0.0768	99.2535

Results based on At% from individual Scans (For Reference Use Only)

At% Average	0.0000	0.0068	0.7015	0.0720	99.2196
At% Std Dev	0.0000	0.0028	0.0166	0.0044	0.0238
%RSD	#DIV/0!	41.16	2.37	6.06	0.02

Output Results – TIMS CEC21-703 (M&TE 4678061)

No.	Smpl ID/Filament ID/Date	Use?	U-233 Ratio	U-234 Ratio	U-235 Ratio	U-236 Ratio	U-238 Ratio
5	11-0420-129-D-U	Yes	0.0000000	0.0000655	0.0074149	0.0008708	1.0000000
	11111014	Yes	0.0000000	0.0002472	0.0076350	0.0009256	1.0000000
	05/06/11	Yes	0.0000000	0.0000283	0.0068409	0.0005721	1.0000000
		Yes	0.0000000	0.0002796	0.0072713	0.0004664	1.0000000
		Yes	0.0000000	0.0000256	0.0069032	0.0009798	1.0000000
		Yes	0.0000000	0.0001744	0.0076256	0.0006624	1.0000000
		Yes	0.0000000	0.0000528	0.0068810	0.0007216	1.0000000
		Yes	0.0000000	0.0000646	0.0070489	0.0007624	1.0000000

Reported Results – TIMS CEC21-703 (M&TE 4678061)

U-233 Ratio	U-234 Ratio	U-235 Ratio	U-236 Ratio	U-238 Ratio
0.0000000	0.0000655	0.0074149	0.0008708	1.0000000
0.0000000	0.0002472	0.0076350	0.0009256	1.0000000
0.0000000	0.0000283	0.0068409	0.0005721	1.0000000
0.0000000	0.0002796	0.0072713	0.0004664	1.0000000
0.0000000	0.0000256	0.0069032	0.0009798	1.0000000
0.0000000	0.0001744	0.0076256	0.0006624	1.0000000
0.0000000	0.0000528	0.0068810	0.0007216	1.0000000
0.0000000	0.0000646	0.0070489	0.0007624	1.0000000

Indiv. Ratio Sum	At% U-233	At% U-234	At% U-235	At% U-236	At% U-238
1.008351	0.0000	0.0065	0.7353	0.0864	99.1718
1.008808	0.0000	0.0245	0.7568	0.0918	99.1269
1.007441	0.0000	0.0028	0.6790	0.0568	99.2614
1.008017	0.0000	0.0277	0.7213	0.0463	99.2046
1.007909	0.0000	0.0025	0.6849	0.0972	99.2153
1.008462	0.0000	0.0173	0.7562	0.0657	99.1609
1.007655	0.0000	0.0052	0.6829	0.0716	99.2403
1.007876	0.0000	0.0064	0.6994	0.0756	99.2186

Results based on At% from individual Scans (For Reference Use Only)

At% Average	0.0000	0.0079	0.7110	0.0733	99.2078
At% Std Dev	0.0000	0.0013	0.0115	0.0050	0.0178
%RSD	#DIV/0!	15.84	1.62	6.82	0.02

TIMS Isotopic Sample Worksheet

Select Isotopes to Report	U-233	U-234	U-235	U-236	U-238

Output Results -- TIMS CEC21-703 (M&TE 4678061)

	Use?	U-233 Ratio	U-234 Ratio	U-235 Ratio	U-236 Ratio	U-238 Ratio
	Yes	0.0000000	0.0000952	0.0067767	0.0003915	1.0000000
TIMS Ratio Average		0.0000000	0.0001148	0.0071553	0.0007058	1.0000000
TIMS Ratio Std Dev		0.0000000	0.0000954	0.0003399	0.0002031	0.0006384
TIMS Avg At%		0.0000000	0.0113892	0.7098659	0.0700259	99.2087190
Avg At% Std Dev		#DIV/0!	0.0094677	0.0337245	0.0201470	0.0633392
%RSD		#DIV/0!	83.13	4.75	28.77	0.06

Reported Results -- TIMS CEC21-703 (M&TE 4678061)

	U-233 Ratio	U-234 Ratio	U-235 Ratio	U-236 Ratio	U-238 Ratio
	0.0000000	0.0000952	0.0067767	0.0003915	1.0000000
Ratio Average	0.0000000	0.0001148	0.0071553	0.0007058	1.0000000
Ratio Std Dev	0.0000000	0.0000954	0.0003399	0.0002031	0.0006384
Avg At%	0.0000000	0.0113892	0.7098659	0.0700259	99.2087190
Avg At% SD	#DIV/0!	0.0094677	0.0337245	0.0201470	0.0633392
%RSD	#DIV/0!	83.13	4.75	28.77	0.06

Calculated At% for Each Individual Scan (For Reference Only)

1.007263	0.0000	0.0095	0.6728	0.0389	99.2789

Results based on At% from individual Scans (For Reference Use Only)

At% Average	0.0000	0.0114	0.7099	0.0700	99.2087
At% Std Dev	0.0000	0.0095	0.0334	0.0201	0.0630
%RSD	#DIV/0!	83.10	4.70	28.75	0.06

Comments:

No.	Smpl ID/Filament ID/Date	Use?	U-233 Ratio	U-234 Ratio	U-235 Ratio	U-236 Ratio	U-238 Ratio	U-233 Ratio	U-234 Ratio	U-235 Ratio	U-236 Ratio	U-238 Ratio	Indiv. Ratio Sum	At% U-233	At% U-234	At% U-235	At% U-236	At% U-238
6	11-0421-129-S-U	Yes	0.0000000	0.0000598	0.0073381	0.0007278	1.0000000	0.0000000	0.0000598	0.0073381	0.0007278	1.0000000	1.008126	0.0000	0.0059	0.7279	0.0722	99.1940
	1111015	Yes	0.0000000	0.0000902	0.0073466	0.0006970	1.0000000	0.0000000	0.0000902	0.0073466	0.0006970	1.0000000	1.008134	0.0000	0.0089	0.7287	0.0691	99.1932
	05/06/11	Yes	0.0000000	0.0000298	0.0079459	0.0005783	1.0000000	0.0000000	0.0000298	0.0079459	0.0005783	1.0000000	1.008554	0.0000	0.0030	0.7879	0.0573	99.1519
		Yes	0.0000000	0.0000611	0.0072751	0.0008526	1.0000000	0.0000000	0.0000611	0.0072751	0.0008526	1.0000000	1.008189	0.0000	0.0061	0.7216	0.0846	99.1878
		Yes	0.0000000	0.0000435	0.0070924	0.0007738	1.0000000	0.0000000	0.0000435	0.0070924	0.0007738	1.0000000	1.007910	0.0000	0.0043	0.7037	0.0768	99.2152
		Yes	0.0000000	0.0000753	0.0068096	0.0006555	1.0000000	0.0000000	0.0000753	0.0068096	0.0006555	1.0000000	1.007540	0.0000	0.0075	0.6759	0.0651	99.2516
		Yes	0.0000000	0.0001098	0.0066943	0.0006104	1.0000000	0.0000000	0.0001098	0.0066943	0.0006104	1.0000000	1.007415	0.0000	0.0109	0.6645	0.0606	99.2640
		Yes	0.0000000	0.0000811	0.0069421	0.0007295	1.0000000	0.0000000	0.0000811	0.0069421	0.0007295	1.0000000	1.007753	0.0000	0.0080	0.6889	0.0724	99.2307
		Yes	0.0000000	0.0000533	0.0065905	0.0008475	1.0000000	0.0000000	0.0000533	0.0065905	0.0008475	1.0000000	1.007491	0.0000	0.0053	0.6541	0.0841	99.2564
	TIMS Ratio Average		0.0000000	0.0000671	0.0071150	0.0007192	1.0000000	Ratio Average	0.0000000	0.0000671	0.0071150	0.0007192	1.0000000					
	TIMS Ratio Std Dev		0.0000000	0.0000246	0.0004176	0.0000960	0.0005382	Ratio Std Dev	0.0000000	0.0000246	0.0004176	0.0000960	0.0005382					
	TIMS Avg At%		0.0000000	0.0066574	0.7059179	0.0713518	99.2160729	Avg At%	0.0000000	0.0066574	0.7059179	0.0713518	99.2160729					
	Avg At% Std Dev		#DIV/0!	0.0024424	0.0414339	0.0095202	0.0533966	Avg At% SD	#DIV/0!	0.0024424	0.0414339	0.0095202	0.0533966					
	%RSD		#DIV/0!	36.69	5.87	13.34	0.05	%RSD	#DIV/0!	36.69	5.87	13.34	0.05					

Results based on At% from individual Scans (For Reference Use Only)

At% Average	0.0000	0.0067	0.7059	0.0714	99.2161
At% Std Dev	0.0000	0.0024	0.0412	0.0095	0.0531
%RSD	#DIV/0!	36.71	5.83	13.35	0.05

Comments:

No.	Smpl ID/Filament ID/Date	Use?	U-233 Ratio	U-234 Ratio	U-235 Ratio	U-236 Ratio	U-238 Ratio	U-233 Ratio	U-234 Ratio	U-235 Ratio	U-236 Ratio	U-238 Ratio	Indiv. Ratio Sum	At% U-233	At% U-234	At% U-235	At% U-236	At% U-238
7	11-0421-129-D-U	Yes	0.0000000	0.0000468	0.0071683	0.0007082	1.0000000	0.0000000	0.0000468	0.0071683	0.0007082	1.0000000	1.007923	0.0000	0.0046	0.7112	0.0703	99.2139
	1111016	Yes	0.0000000	0.0000631	0.0071429	0.0006125	1.0000000	0.0000000	0.0000631	0.0071429	0.0006125	1.0000000	1.007819	0.0000	0.0063	0.7087	0.0608	99.2242
	05/06/11	Yes	0.0000000	0.0000576	0.0070182	0.0006884	1.0000000	0.0000000	0.0000576	0.0070182	0.0006884	1.0000000	1.007764	0.0000	0.0057	0.6964	0.0683	99.2296
		Yes	0.0000000	0.0000788	0.0070003	0.0007232	1.0000000	0.0000000	0.0000788	0.0070003	0.0007232	1.0000000	1.007802	0.0000	0.0078	0.6946	0.0718	99.2258
		Yes	0.0000000	0.0000525	0.0070470	0.0008114	1.0000000	0.0000000	0.0000525	0.0070470	0.0008114	1.0000000	1.007911	0.0000	0.0052	0.6992	0.0805	99.2151
		Yes	0.0000000	0.0000909	0.0070358	0.0007708	1.0000000	0.0000000	0.0000909	0.0070358	0.0007708	1.0000000	1.007898	0.0000	0.0090	0.6981	0.0765	99.2164
		Yes	0.0000000	0.0000649	0.0070563	0.0007750	1.0000000	0.0000000	0.0000649	0.0070563	0.0007750	1.0000000	1.007896	0.0000	0.0064	0.7001	0.0769	99.2166
		Yes	0.0000000	0.0000702	0.0073415	0.0007423	1.0000000	0.0000000	0.0000702	0.0073415	0.0007423	1.0000000	1.008154	0.0000	0.0070	0.7282	0.0736	99.1912
		Yes	0.0000000	0.0001137	0.0070111	0.0007212	1.0000000	0.0000000	0.0001137	0.0070111	0.0007212	1.0000000	1.007846	0.0000	0.0113	0.6957	0.0716	99.2215
	TIMS Ratio Average		0.0000000	0.0000709	0.0070913	0.0007281	1.0000000	Ratio Average	0.0000000	0.0000709	0.0070913	0.0007281	1.0000000					
	TIMS Ratio Std Dev		0.0000000	0.0000209	0.0001104	0.0000576	0.0001889	Ratio Std Dev	0.0000000	0.0000209	0.0001104	0.0000576	0.0001889					
	TIMS Avg At%		0.0000000	0.0070389	0.7035752	0.0722411	99.2171448	Avg At%	0.0000000	0.0070389	0.7035752	0.0722411	99.2171448					
	Avg At% Std Dev		#DIV/0!	0.0020717	0.0109542	0.0057180	0.0187438	Avg At% SD	#DIV/0!	0.0020717	0.0109542	0.0057180	0.0187438					
	%RSD		#DIV/0!	29.43	1.56	7.92	0.02	%RSD	#DIV/0!	29.43	1.56	7.92	0.02					

Results based on At% from individual Scans (For Reference Use Only)

At% Average	0.0000	0.0070	0.7036	0.0722	99.2171
At% Std Dev	0.0000	0.0021	0.0109	0.0057	0.0187
%RSD	#DIV/0!	29.43	1.55	7.91	0.02

Comments:

No.	Smpl ID/Filament ID/Date	Use?	U-233 Ratio	U-234 Ratio	U-235 Ratio	U-236 Ratio	U-238 Ratio	U-233 Ratio	U-234 Ratio	U-235 Ratio	U-236 Ratio	U-238 Ratio	Indiv. Ratio Sum	At% U-233	At% U-234	At% U-235	At% U-236	At% U-238
8	11-0422-129-S-U	Yes	0.0000000	0.0000546	0.0071644	0.0007000	1.0000000	0.0000000	0.0000546	0.0071644	0.0007000	1.0000000	1.007919	0.0000	0.0054	0.7108	0.0695	99.2143
	1111017	No	0.0000000	0.0000658	0.0072367	0.0008346	1.0000000											
	05/09/11	Yes	0.0000000	0.0000413	0.0071768	0.0008351	1.0000000	0.0000000	0.0000413	0.0071768	0.0008351	1.0000000	1.008053	0.0000	0.0041	0.7119	0.0828	99.2011
		Yes	0.0000000	0.0000753	0.0069076	0.0006631	1.0000000	0.0000000	0.0000753	0.0069076	0.0006631	1.0000000	1.007646	0.0000	0.0075	0.6855	0.0658	99.2412
		Yes	0.0000000	0.0001200	0.0072180	0.0007135	1.0000000	0.0000000	0.0001200	0.0072180	0.0007135	1.0000000	1.008052	0.0000	0.0119	0.7160	0.0708	99.2013
		Yes	0.0000000	0.0001123	0.0070834	0.0007220	1.0000000	0.0000000	0.0001123	0.0070834	0.0007220	1.0000000	1.007918	0.0000	0.0111	0.7028	0.0716	99.2144
		Yes	0.0000000	0.0000804	0.0073273	0.0007594	1.0000000	0.0000000	0.0000804	0.0073273	0.0007594	1.0000000	1.008167	0.0000	0.0080	0.7268	0.0753	99.1899
		Yes	0.0000000	0.0000488	0.0071787	0.0006834	1.0000000	0.0000000	0.0000488	0.0071787	0.0006834	1.0000000	1.007911	0.0000	0.0048	0.7122	0.0678	99.2151
		Yes	0.0000000	0.0000917	0.0071811	0.0006534	1.0000000	0.0000000	0.0000917	0.0071811	0.0006534	1.0000000	1.007926	0.0000	0.0091	0.7125	0.0648	99.2136
	TIMS Ratio Average		0.0000000	0.0000767	0.0071638	0.0007294	1.0000000	Ratio Average	0.0000000	0.0000781	0.0071547	0.0007162	1.0000000					
	TIMS Ratio Std Dev		0.0000000	0.0000274	0.0001161	0.0000676	0.0002111	Ratio Std Dev	0.0000000	0.0000290	0.0001206	0.0000587	0.0002083					
	TIMS Avg At%		0.0000000	0.0076083	0.7107135	0.0723622	99.2093161	Avg At%	0.0000000	0.0077434	0.7098239	0.0710589	99.2113738					
	Avg At% Std Dev		#DIV/0!	0.0027219	0.0115152	0.0067103	0.0209474	Avg At% SD	#DIV/0!	0.0028774	0.0119640	0.0058269	0.0206683					
	%RSD		#DIV/0!	35.78	1.62	9.27	0.02	%RSD	#DIV/0!	37.16	1.69	8.20	0.02					

Results based on At% from individual Scans (For Reference Use Only)

At% Average	0.0000	0.0077	0.7098	0.0711	99.2114
At% Std Dev	0.0000	0.0029	0.0119	0.0058	0.0206
%RSD	#DIV/0!	37.16	1.67	8.19	0.02

Comments:

No.	Smpl ID/Filament ID/Date	Use?	U-233 Ratio	U-234 Ratio	U-235 Ratio	U-236 Ratio	U-238 Ratio	U-233 Ratio	U-234 Ratio	U-235 Ratio	U-236 Ratio	U-238 Ratio	Indiv. Ratio Sum	At% U-233	At% U-234	At% U-235	At% U-236	At% U-238
9	11-0422-129-D-U	Yes	0.0000000	0.0000997	0.0073447	0.0007931	1.0000000	0.0000000	0.0000997	0.0073447	0.0007931	1.0000000	1.008238	0.0000	0.0099	0.7285	0.0787	99.1830
	1111018	Yes	0.0000000	0.0000678	0.0072729	0.0007109	1.0000000	0.0000000	0.0000678	0.0072729	0.0007109	1.0000000	1.008052	0.0000	0.0067	0.7215	0.0705	99.2013
	05/09/11	Yes	0.0000000	0.0001013	0.0071895	0.0008124	1.0000000	0.0000000	0.0001013	0.0071895	0.0008124	1.0000000	1.008103	0.0000	0.0100	0.7132	0.0806	99.1962
		Yes	0.0000000	0.0001618	0.0074768	0.0008650	1.0000000	0.0000000	0.0001618	0.0074768	0.0008650	1.0000000	1.008504	0.0000	0.0160	0.7414	0.0858	99.1568
		Yes	0.0000000	0.0001960	0.0070340	0.0008425	1.0000000	0.0000000	0.0001960	0.0070340	0.0008425	1.0000000	1.008073	0.0000	0.0194	0.6978	0.0836	99.1992
		Yes	0.0000000	0.0000537	0.0069269	0.0007912	1.0000000	0.0000000	0.0000537	0.0069269	0.0007912	1.0000000	1.007772	0.0000	0.0053	0.6873	0.0785	99.2288
		Yes	0.0000000	0.0000766	0.0069584	0.0007569	1.0000000	0.0000000	0.0000766	0.0069584	0.0007569	1.0000000	1.007792	0.0000	0.0076	0.6905	0.0751	99.2268
		Yes	0.0000000	0.0001051	0.0071822	0.0007573	1.0000000	0.0000000	0.0001051	0.0071822	0.0007573	1.0000000	1.008045	0.0000	0.0104	0.7125	0.0751	99.2020
		Yes	0.0000000	0.0001228	0.0073873	0.0006755	1.0000000	0.0000000	0.0001228	0.0073873	0.0006755	1.0000000	1.008186	0.0000	0.0122	0.7327	0.0670	99.1881
	TIMS Ratio Average		0.0000000	0.0001094	0.0071970	0.0007783	1.0000000	Ratio Average	0.0000000	0.0001094	0.0071970	0.0007783	1.0000000					
	TIMS Ratio Std Dev		0.0000000	0.0000454	0.0001933	0.0000604	0.0002991	Ratio Std Dev	0.0000000	0.0000454	0.0001933	0.0000604	0.0002991					
	TIMS Avg At%		0.0000000	0.0108545	0.7139248	0.0772069	99.1980138	Avg At%	0.0000000	0.0108545	0.7139248	0.0772069	99.1980138					
	Avg At% Std Dev		#DIV/0!	0.0045067	0.0191714	0.0059927	0.0296707	Avg At% SD	#DIV/0!	0.0045067	0.0191714	0.0059927	0.0296707					
	%RSD		#DIV/0!	41.52	2.69	7.76	0.03	%RSD	#DIV/0!	41.52	2.69	7.76	0.03					

Results based on At% from individual Scans (For Reference Use Only)

At% Average	0.0000	0.0109	0.7139	0.0772	99.1980
At% Std Dev	0.0000	0.0045	0.0190	0.0060	0.0295
%RSD	#DIV/0!	41.51	2.67	7.75	0.03

Comments:

No.	Smpl ID/Filament ID/Date	Use?	U-233 Ratio	U-234 Ratio	U-235 Ratio	U-236 Ratio	U-238 Ratio	U-233 Ratio	U-234 Ratio	U-235 Ratio	U-236 Ratio	U-238 Ratio	Indiv. Ratio Sum	At% U-233	At% U-234	At% U-235	At% U-236	At% U-238
10	11-0420-129-D-U	Yes	0.0000000	0.0000694	0.0073048	0.0008158	1.0000000	0.0000000	0.0000694	0.0073048	0.0008158	1.0000000	1.008190	0.0000	0.0069	0.7245	0.0809	99.1877

TIMS Isotopic Sample Worksheet

Select Isotopes to Report	U-233	U-234	U-235	U-236	U-238

Output Results -- TIMS CEC21-703 (M&TE 4678061)						Reported Results -- TIMS CEC21-703 (M&TE 4678061)				Calculated At% for Each Individual Scan (For Reference Only)			
										1.008276	0.0000	0.0078	0.7376	0.0754	99.1792
11111014	Yes	0.0000000	0.0000787	0.0074368	0.0007600	1.0000000		0.0000000	0.0000787	0.0074368	0.0007600	1.0000000													
05/09/11	Yes	0.0000000	0.0000852	0.0071936	0.0007272	1.0000000		0.0000000	0.0000852	0.0071936	0.0007272	1.0000000	1.008986	0.0000	0.0085	0.7136	0.0721	99.2058
	Yes	0.0000000	0.0000369	0.0070726	0.0006024	1.0000000		0.0000000	0.0000369	0.0070726	0.0006024	1.0000000	1.007712	0.0000	0.0037	0.7018	0.0598	99.2347
	Yes	0.0000000	0.0000639	0.0070787	0.0007365	1.0000000		0.0000000	0.0000639	0.0070787	0.0007365	1.0000000	1.007879	0.0000	0.0063	0.7023	0.0731	99.2182
	Yes	0.0000000	0.0000659	0.0070787	0.0007250	1.0000000		0.0000000	0.0001000	0.0067040	0.0007250	1.0000000	1.007529	0.0000	0.0099	0.6654	0.0720	99.2527
	Yes	0.0000000	0.0001000	0.0067040	0.0007250	1.0000000		0.0000000	0.0000769	0.0067339	0.0008175	1.0000000	1.007628	0.0000	0.0076	0.6683	0.0811	99.2429
	Yes	0.0000000	0.0000769	0.0067339	0.0008175	1.0000000		0.0000000	0.0000769	0.0067339	0.0008175	1.0000000	1.008174	0.0000	0.0060	0.7357	0.0690	99.1893
	Yes	0.0000000	0.0000606	0.0074170	0.0006960	1.0000000		0.0000000	0.0000606	0.0074170	0.0006960	1.0000000	1.007910	0.0000	0.0102	0.6921	0.0825	99.2152
	Yes	0.0000000	0.0001025	0.0069753	0.0008318	1.0000000		0.0000000	0.0001025	0.0069753	0.0008318	1.0000000						
TIMS Ratio Average		0.0000000	0.0000749	0.0071019	0.0007458	1.0000000	Ratio Average	0.0000000	0.0000749	0.0071019	0.0007458	1.0000000	Results based on At% from individual Scans (For Reference Use Only)					
TIMS Ratio Std Dev		0.0000000	0.0000204	0.0002674	0.0003599		Ratio Std Dev	0.0000000	0.0000204	0.0002674	0.0000721	0.0003599	At% Average	0.0000	0.0074	0.7046	0.0740	99.2140
TIMS Avg At%		0.0000000	0.0074311	0.7046033	0.0739938	99.2139718	Avg At%	0.0000000	0.0074311	0.7046033	0.0739938	99.2139718	At% Std Dev	0.0000	0.0020	0.0264	0.0071	0.0155
Avg At% Std Dev		#DIV/0!	0.0020199	0.0265338	0.0071499	0.0357037	Avg At% SD	#DIV/0!	0.0020199	0.0265338	0.0071499	0.0357037	%aRSD	#DIV/0!	27.19	3.74	9.66	0.04
%RSD		#DIV/0!	27.18	3.77	9.66	0.04	%RSD	#DIV/0!	27.18	3.77	9.66	0.04						

Comments

PNNL - Radiochemical Processing and Engineering Group

Final Reported Results

TIMS CEC21-703 (M&TE 4678061)

Smpl #	Sample ID	Filament	Run Date	U-233 At%	U-233 Std Dev	U-233 %RSD	U-234 At%	U-234 Std Dev	U-234 %RSD	U-235 At%	U-235 Std Dev	U-235 %RSD	U-236 At%	U-236 Std Dev	U-236 %RSD	U-238 At%	U-238 Std Dev	U-238 %RSD
1	11-0419-129-S-U	1111010	05/06/11	<0.003			0.008	± 0.0013	15.84	0.711	± 0.0117	1.64	0.073	± 0.0050	6.83	99.208	± 0.0179	0.02
2	11-0419-129-D-U	1111011	05/06/11	<0.003			0.007	± 0.0011	16.44	0.709	± 0.0079	1.11	0.076	± 0.0024	3.17	99.208	± 0.0114	0.01
			Average =				**0.007**	**± 0.0009**	**11.92**	**0.710**	**± 0.0013**	**0.18**	**0.075**	**± 0.0016**	**2.21**	**99.208**	**± 0.0005**	**0.00**
			RPD (%)				17			0.3			3			0.001		
3	11-0419-129-PB-	1111012	05/06/11	(ND)			(ND)			(ND)			(ND)			(ND)		
4	11-0420-129-S-U	11111013	05/06/11	<0.003			0.007	± 0.0028	41.18	0.702	± 0.0168	2.39	0.072	± 0.0044	6.06	99.220	± 0.0239	0.02
10	11-0420-129-D-U	1111014	05/09/11	<0.003			0.007	± 0.0020	27.18	0.705	± 0.0265	3.77	0.074	± 0.0071	9.66	99.214	± 0.0357	0.04
			Average =				**0.007**	**± 0.0004**	**5.97**	**0.703**	**± 0.0022**	**0.31**	**0.073**	**± 0.0014**	**1.94**	**99.217**	**± 0.0040**	**0.00**
			RPD (%)				8			0.4			3			0.006		
6	11-0421-129-S-U	1111015	05/06/11	<0.003			0.007	± 0.0024	36.69	0.706	± 0.0414	5.87	0.071	± 0.0095	13.34	99.216	± 0.0534	0.05
7	11-0421-129-D-U	1111016	05/06/11	<0.003			0.007	± 0.0021	29.43	0.704	± 0.0110	1.56	0.072	± 0.0057	7.92	99.217	± 0.0187	0.02
			Average =				**0.007**	**± 0.0003**	**3.94**	**0.705**	**± 0.0017**	**0.24**	**0.072**	**± 0.0006**	**0.88**	**99.217**	**± 0.0008**	**0.00**
			RPD (%)				6			0.3			1			-0.001		
8	11-0422-129-S-U	1111017	05/09/11	<0.003			0.008	± 0.0029	37.16	0.710	± 0.0120	1.69	0.071	± 0.0058	8.20	99.211	± 0.0207	0.02
9	11-0422-129-D-U	1111018	05/09/11	<0.003			0.011	± 0.0045	41.52	0.714	± 0.0192	2.69	0.077	± 0.0060	7.76	99.198	± 0.0297	0.03
			Average =				**0.009**	**± 0.0022**	**23.66**	**0.712**	**± 0.0029**	**0.41**	**0.074**	**± 0.0043**	**5.86**	**99.205**	**± 0.0094**	**0.01**
			RPD (%)				33			0.6			8			0.013		

TIMS Isotopic Standard Worksheet

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PNNL - Radiochemical Processing and Engineering Group

Procedure: PNNL-98523-264, Rev 2

TIMS Isotopic Standard Worksheet

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PNNL - Radiochemical Processing and Engineering Group

Procedure: PNNL-98523-264, Rev 2

Summary of Standard Results - Standard: Pu-946

			Pu-238		Pu-239		Pu-240		Pu-241		Pu-242	
	Run Date	Filament	Atom%	1-σ	RSD(%)	Atom%	1-σ	RSD(%)	Atom%	1-σ	RSD(%)	Atom%	1-σ	RSD(%)
Cert Value	n/a	n/a	0.1877	± 0.0070	n/a	86.134	± 0.015	n/a	12.480	± 0.015	n/a	0.6130	± 0.0050	n/a	0.5856	± 0.003	n/a
Pooled	n/a	n/a	0.1882	± 0.005	2.79	86.175	± 0.052	0.06	12.436	± 0.052	0.42	0.6172	± 0.0079	1.27	0.5835	± 0.008	1.45
	%Difference =		0.3			0.05			-0.354			0.7			-0.4		
1	05/17/11	9461106.1	0.189	± 0.007	3.59	86.227	± 0.115	0.13	12.390	± 0.074	0.60	0.620	± 0.020	3.27	0.5749	± 0.014	2.42
2	05/17/11	9461106.2	0.184	± 0.006	3.12	86.206	± 0.083	0.10	12.415	± 0.052	0.42	0.612	± 0.012	1.96	0.5830	± 0.014	2.37
3	05/17/11	9461106.3	0.181	± 0.009	4.89	86.136	± 0.140	0.16	12.468	± 0.094	0.75	0.631	± 0.023	3.57	0.5840	± 0.015	2.61
4	05/18/11	9461107.1	0.188	± 0.012	6.19	86.185	± 0.227	0.26	12.447	± 0.181	1.46	0.609	± 0.017	2.83	0.5705	± 0.017	2.91
5	05/18/11	9461107.2	0.196	± 0.012	5.89	86.206	± 0.147	0.17	12.404	± 0.091	0.74	0.609	± 0.021	3.48	0.5844	± 0.023	3.87
6	05/18/11	9461108.1	0.196	± 0.039	20.00	86.245	± 0.435	0.50	12.354	± 0.283	2.29	0.618	± 0.085	13.69	0.5882	± 0.028	4.83
7	05/25/11	9461109.1	0.187	± 0.007	4.00	86.099	± 0.201	0.23	12.526	± 0.155	1.23	0.609	± 0.021	3.52	0.5786	± 0.017	2.97
8	05/25/11	9461109.2	0.189	± 0.022	11.50	86.110	± 0.241	0.28	12.479	± 0.150	1.20	0.622	± 0.034	5.46	0.5997	± 0.035	5.91
9	05/25/11	94611010	0.184	± 0.011	5.90	86.164	± 0.165	0.19	12.439	± 0.119	0.95	0.625	± 0.022	3.55	0.5880	± 0.014	2.34

Page 1 of 1

TIMS Isotopic Sample Worksheet

Select Isotopes to Report	Pu-238	Pu-239	Pu-240	Pu-241	Pu-242

Output Results – TIMS CEC21-703 (M&TE 4678061)

No.	Smpl ID/Filament ID/Date	Use?	Pu-238 Ratio	Pu-239 Ratio	Pu-240 Ratio	Pu-241 Ratio	Pu-242 Ratio
1	11-0419-129-S-Pu	Yes	0.0000850	1.0000000	0.1504369	0.0066372	0.0029674
	1211010	Yes	0.0016365	1.0000000	0.1603001	0.0087258	0.0014103
	05/18/11	Yes	0.0002682	1.0000000	0.1564845	0.0089409	0.0031848
		Yes	0.0012072	1.0000000	0.1422759	0.0096338	0.0036026
		Yes	0.0016136	1.0000000	0.1531110	0.0066869	0.0021807
		Yes	0.0011531	1.0000000	0.1433747	0.0090029	0.0016256
		Yes	0.0018476	1.0000000	0.1484095	0.0071204	0.0028746
		Yes	0.0008598	1.0000000	0.1470927	0.0067990	0.0028326
		Yes	0.0012315	1.0000000	0.1485962	0.0069318	0.0034269
	TIMS Ratio Average		**0.0011003**	**1.0000000**	**0.1500091**	**0.0078310**	**0.0026784**
	TIMS Ratio Std Dev		**0.0006044**	**0.0084482**	**0.0058579**	**0.0012129**	**0.0007730**
	TIMS Avg At%		**0.0947194**	**86.0867692**	**12.9137949**	**0.6741426**	**0.2305738**
	Avg At% Std Dev		**0.0520268**	**0.7272750**	**0.5042862**	**0.1044130**	**0.0665490**
	%RSD		**54.93**	**0.84**	**3.91**	**15.49**	**28.86**
	Comments:						

Reported Results – TIMS CEC21-703 (M&TE 4678061)

Pu-238 Ratio	Pu-239 Ratio	Pu-240 Ratio	Pu-241 Ratio	Pu-242 Ratio	
0.0000850	1.0000000	0.1504369	0.0066372	0.0029674	
0.0016365	1.0000000	0.1603001	0.0087258	0.0014103	
0.0002682	1.0000000	0.1564845	0.0089409	0.0031848	
0.0012072	1.0000000	0.1422759	0.0096338	0.0036026	
0.0016136	1.0000000	0.1531110	0.0066869	0.0021807	
0.0011531	1.0000000	0.1433747	0.0090029	0.0016256	
0.0018476	1.0000000	0.1484095	0.0071204	0.0028746	
0.0008598	1.0000000	0.1470927	0.0067990	0.0028326	
0.0012315	1.0000000	0.1485962	0.0069318	0.0034269	
0.0011003	**1.0000000**	**0.1500091**	**0.0078310**	**0.0026784**	**Ratio Average**
0.0006044	**0.0084482**	**0.0058579**	**0.0012129**	**0.0007730**	**Ratio Std Dev**
0.0947194	**86.0867692**	**12.9137949**	**0.6741426**	**0.2305738**	**Avg At%**
0.0520268	**0.7272750**	**0.5042862**	**0.1044130**	**0.0665490**	**Avg At% SD**
54.93	**0.84**	**3.91**	**15.49**	**28.86**	**%RSD**

Calculated At% for Each Individual Scan (For Reference Only)

Indiv. Ratio Sum	At% Pu-238	At% Pu-239	At% Pu-240	At% Pu-241	At% Pu-242
1.160127	0.0073	86.1975	12.9673	0.5721	0.2558
1.172073	0.1396	85.3189	13.6766	0.7445	0.1203
1.168878	0.0229	85.5521	13.3876	0.7649	0.2725
1.156720	0.1044	86.4514	12.2999	0.8329	0.3114
1.163592	0.1387	85.9408	13.1585	0.5747	0.1874
1.155156	0.0998	86.5684	12.4117	0.7794	0.1407
1.160252	0.1592	86.1882	12.7911	0.6137	0.2478
1.157584	0.0743	86.3868	12.7069	0.5873	0.2447
1.160186	0.1061	86.1930	12.8080	0.5975	0.2954

Results based on At% from individual Scans (For Reference Use Only)

At% Average	0.0947	86.0886	12.9120	0.6741	0.2307
At% Std Dev	0.0519	0.6655	0.4426	0.1042	0.0668
%RSD	54.82	0.77	3.43	15.45	28.97

No.	Smpl ID/Filament ID/Date	Use?	Pu-238 Ratio	Pu-239 Ratio	Pu-240 Ratio	Pu-241 Ratio	Pu-242 Ratio
2	11-0419-129-D-Pu	Yes	0.0015460	1.0000000	0.1490039	0.0052284	0.0034270
	1211011	Yes	0.0012688	1.0000000	0.1509154	0.0063275	0.0033134
	05/18/11	Yes	0.0014212	1.0000000	0.1438000	0.0056386	0.0025932
		Yes	0.0019100	1.0000000	0.1457857	0.0058049	0.0025557
		Yes	0.0019548	1.0000000	0.1553129	0.0052747	0.0026300
		Yes	0.0020885	1.0000000	0.1527571	0.0067048	0.0030795
		Yes	0.0014236	1.0000000	0.1455310	0.0061516	0.0028624
		Yes	0.0019405	1.0000000	0.1506099	0.0059771	0.0023982
		Yes	0.0026577	1.0000000	0.1544235	0.0066698	0.0032078
	TIMS Ratio Average		**0.0018012**	**1.0000000**	**0.1497933**	**0.0059775**	**0.0028964**
	TIMS Ratio Std Dev		**0.0004340**	**0.0054337**	**0.0040833**	**0.0005433**	**0.0003732**
	TIMS Avg At%		**0.1552161**	**86.1721050**	**12.9080011**	**0.5150928**	**0.2495851**
	Avg At% Std Dev		**0.0373947**	**0.4682347**	**0.3518652**	**0.0468150**	**0.0321599**
	%RSD		**24.09**	**0.54**	**2.73**	**9.09**	**12.89**
	Comments:						

Pu-238 Ratio	Pu-239 Ratio	Pu-240 Ratio	Pu-241 Ratio	Pu-242 Ratio	
0.0015460	1.0000000	0.1490039	0.0052284	0.0034270	
0.0012688	1.0000000	0.1509154	0.0063275	0.0033134	
0.0014212	1.0000000	0.1438000	0.0056386	0.0025932	
0.0019100	1.0000000	0.1457857	0.0058049	0.0025557	
0.0019548	1.0000000	0.1553129	0.0052747	0.0026300	
0.0020885	1.0000000	0.1527571	0.0067048	0.0030795	
0.0014236	1.0000000	0.1455310	0.0061516	0.0028624	
0.0019405	1.0000000	0.1506099	0.0059771	0.0023982	
0.0026577	1.0000000	0.1544235	0.0066698	0.0032078	
0.0018012	**1.0000000**	**0.1497933**	**0.0059775**	**0.0028964**	**Ratio Average**
0.0004340	**0.0054337**	**0.0040833**	**0.0005433**	**0.0003732**	**Ratio Std Dev**
0.1552161	**86.1721050**	**12.9080011**	**0.5150928**	**0.2495851**	**Avg At%**
0.0373947	**0.4682347**	**0.3518652**	**0.0468150**	**0.0321599**	**Avg At% SD**
24.09	**0.54**	**2.73**	**9.09**	**12.89**	**%RSD**

Indiv. Ratio Sum	At% Pu-238	At% Pu-239	At% Pu-240	At% Pu-241	At% Pu-242
1.159205	0.1334	86.2660	12.8540	0.4510	0.2956
1.161825	0.1092	86.0715	12.9895	0.5446	0.2852
1.153453	0.1232	86.6962	12.4669	0.4888	0.2248
1.156056	0.1652	86.5010	12.6106	0.5021	0.2211
1.165172	0.1678	85.8242	13.3296	0.4527	0.2257
1.164630	0.1793	85.8642	13.1164	0.5757	0.2644
1.155969	0.1232	86.5075	12.5895	0.5322	0.2476
1.160946	0.1671	86.1367	12.9730	0.5166	0.2066
1.166959	0.2277	85.6928	13.2330	0.5716	0.2749

Results based on At% from individual Scans (For Reference Use Only)

At% Average	0.1551	86.1733	12.9069	0.5150	0.2495
At% Std Dev	0.0369	0.4157	0.3010	0.0459	0.0318
%RSD	23.78	0.48	2.33	8.92	12.75

No.	Smpl ID/Filament ID/Date	Use?	Pu-238 Ratio	Pu-239 Ratio	Pu-240 Ratio	Pu-241 Ratio	Pu-242 Ratio
3	11-0420-129-S-Pu	Yes	0.0017050	1.0000000	0.1525954	0.0088062	0.0026110
	1211013	Yes	0.0002822	1.0000000	0.1742533	0.0135393	0.0039704
	05/18/11	Yes	0.0023773	1.0000000	0.1635464	0.0110860	0.0037637
		Yes	0.0014306	1.0000000	0.1740074	0.0089896	0.0051802
		Yes	0.0048475	1.0000000	0.1524545	0.0137738	0.0004387
		Yes	0.0016749	1.0000000	0.1711141	0.0171387	0.0031442
		Yes	0.0041744	1.0000000	0.1690548	0.0208912	0.0032413
		Yes	0.0031627	1.0000000	0.1574925	0.0156206	0.0045266
		Yes	0.0023206	1.0000000	0.1626579	0.0170369	0.0015993
	TIMS Ratio Average		**0.0024417**	**1.0000000**	**0.1641307**	**0.0140980**	**0.0031639**
	TIMS Ratio Std Dev		**0.0014206**	**0.0154941**	**0.0085805**	**0.0040302**	**0.0014628**
	TIMS Avg At%		**0.2062526**	**84.4712772**	**13.8643299**	**1.1908789**	**0.2672615**
	Avg At% Std Dev		**0.1200014**	**1.3088106**	**0.7248083**	**0.3404323**	**0.1235686**
	%RSD		**58.18**	**1.55**	**5.23**	**28.59**	**46.24**
	Comments:						

Pu-238 Ratio	Pu-239 Ratio	Pu-240 Ratio	Pu-241 Ratio	Pu-242 Ratio	
0.0017050	1.0000000	0.1525954	0.0088062	0.0026110	
0.0002822	1.0000000	0.1742533	0.0135393	0.0039704	
0.0023773	1.0000000	0.1635464	0.0110860	0.0037637	
0.0014306	1.0000000	0.1740074	0.0089896	0.0051802	
0.0048475	1.0000000	0.1524545	0.0137738	0.0004387	
0.0016749	1.0000000	0.1711141	0.0171387	0.0031442	
0.0041744	1.0000000	0.1690548	0.0208912	0.0032413	
0.0031627	1.0000000	0.1574925	0.0156206	0.0045266	
0.0023206	1.0000000	0.1626579	0.0170369	0.0015993	
0.0024417	**1.0000000**	**0.1641307**	**0.0140980**	**0.0031639**	**Ratio Average**
0.0014206	**0.0154941**	**0.0085805**	**0.0040302**	**0.0014628**	**Ratio Std Dev**
0.2062526	**84.4712772**	**13.8643299**	**1.1908789**	**0.2672615**	**Avg At%**
0.1200014	**1.3088106**	**0.7248083**	**0.3404323**	**0.1235686**	**Avg At% SD**
58.18	**1.55**	**5.23**	**28.59**	**46.24**	**%RSD**

Indiv. Ratio Sum	At% Pu-238	At% Pu-239	At% Pu-240	At% Pu-241	At% Pu-242
1.165718	0.1463	85.7841	13.0903	0.7554	0.2240
1.192045	0.0237	83.8894	14.6180	1.1358	0.3331
1.180773	0.2013	84.6903	13.8508	0.9389	0.3187
1.189608	0.1203	84.0613	14.6273	0.7557	0.4355
1.171515	0.4138	85.3596	13.0135	1.1757	0.0374
1.193072	0.1404	83.8172	14.3423	1.4365	0.2635
1.197362	0.3486	83.5170	14.1189	1.7448	0.2707
1.180802	0.2678	84.6882	13.3378	1.3229	0.3833
1.183615	0.1961	84.4870	13.7425	1.4394	0.1351

Results based on At% from individual Scans (For Reference Use Only)

At% Average	0.2065	84.4771	13.8601	1.1895	0.2668
At% Std Dev	0.1204	1.1956	0.6183	0.3339	0.1229
%RSD	58.30	1.42	4.46	28.07	46.08

No.	Smpl ID/Filament ID/Date	Use?	Pu-238 Ratio	Pu-239 Ratio	Pu-240 Ratio	Pu-241 Ratio	Pu-242 Ratio
4	11-0420-129-D-Pu	Yes	0.0017945	1.0000000	0.1457002	0.0073756	0.0037488
	1211014	Yes	0.0030190	1.0000000	0.1482431	0.0068908	0.0035860
	05/18/11	Yes	0.0018397	1.0000000	0.1478715	0.0093051	0.0023044
		Yes	0.0001918	1.0000000	0.1554446	0.0081981	0.0027384
		Yes	0.0018372	1.0000000	0.1511807	0.0092486	0.0024850
		Yes	0.0026552	1.0000000	0.1475747	0.0071436	0.0030223
		Yes	0.0017067	1.0000000	0.1559164	0.0070042	0.0029206
		Yes	0.0010349	1.0000000	0.1611785	0.0079864	0.0030880
		Yes	0.0017927	1.0000000	0.1456843	0.0077212	0.0031970
	TIMS Ratio Average		**0.0017635**	**1.0000000**	**0.1509771**	**0.0078748**	**0.0030101**
	TIMS Ratio Std Dev		**0.0008207**	**0.0075958**	**0.0053970**	**0.0009072**	**0.0004709**
	TIMS Avg At%		**0.1515541**	**85.9382999**	**12.9747162**	**0.6767507**	**0.2586791**
	Avg At% Std Dev		**0.0705322**	**0.6527711**	**0.4638086**	**0.0779609**	**0.0404693**
	%RSD		**46.54**	**0.76**	**3.57**	**11.52**	**15.64**
	Comments:						

Pu-238 Ratio	Pu-239 Ratio	Pu-240 Ratio	Pu-241 Ratio	Pu-242 Ratio	
0.0017945	1.0000000	0.1457002	0.0073756	0.0037488	
0.0030190	1.0000000	0.1482431	0.0068908	0.0035860	
0.0018397	1.0000000	0.1478715	0.0093051	0.0023044	
0.0001918	1.0000000	0.1554446	0.0081981	0.0027384	
0.0018372	1.0000000	0.1511807	0.0092486	0.0024850	
0.0026552	1.0000000	0.1475747	0.0071436	0.0030223	
0.0017067	1.0000000	0.1559164	0.0070042	0.0029206	
0.0010349	1.0000000	0.1611785	0.0079864	0.0030880	
0.0017927	1.0000000	0.1456843	0.0077212	0.0031970	
0.0017635	**1.0000000**	**0.1509771**	**0.0078748**	**0.0030101**	**Ratio Average**
0.0008207	**0.0075958**	**0.0053970**	**0.0009072**	**0.0004709**	**Ratio Std Dev**
0.1515541	**85.9382999**	**12.9747162**	**0.6767507**	**0.2586791**	**Avg At%**
0.0705322	**0.6527711**	**0.4638086**	**0.0779609**	**0.0404693**	**Avg At% SD**
46.54	**0.76**	**3.57**	**11.52**	**15.64**	**%RSD**

Indiv. Ratio Sum	At% Pu-238	At% Pu-239	At% Pu-240	At% Pu-241	At% Pu-242
1.158619	0.1549	86.3096	12.5753	0.6366	0.3236
1.161739	0.2599	86.0779	12.7604	0.5931	0.3087
1.161321	0.1584	86.1089	12.7330	0.8013	0.1984
1.166573	0.0164	85.7212	13.3249	0.7028	0.2347
1.164752	0.1577	85.8552	12.9797	0.7940	0.2134
1.160396	0.2288	86.1775	12.7176	0.6156	0.2605
1.167548	0.1462	85.6496	13.3542	0.5999	0.2501
1.173288	0.0882	85.2306	13.7373	0.6807	0.2632
1.158395	0.1548	86.3263	12.5764	0.6665	0.2760

Results based on At% from individual Scans (For Reference Use Only)

At% Average	0.1517	85.9396	12.9732	0.6767	0.2587
At% Std Dev	0.0708	0.5982	0.4088	0.0777	0.0409
%RSD	46.67	0.70	3.15	11.48	15.80

No.	Smpl ID/Filament ID/Date	Use?	Pu-238 Ratio	Pu-239 Ratio	Pu-240 Ratio	Pu-241 Ratio	Pu-242 Ratio
5	11-0421-129-S-Pu	Yes	0.0008366	1.0000000	0.1470467	0.0060678	0.0026357
	1211015	Yes	0.0005724	1.0000000	0.1426943	0.0050492	0.0029295
	05/18/11	Yes	0.0005687	1.0000000	0.1474566	0.0049801	0.0030263
		Yes	0.0008529	1.0000000	0.1523031	0.0052795	0.0027947
		Yes	0.0013468	1.0000000	0.1510944	0.0060919	0.0020114
		Yes	0.0011300	1.0000000	0.1516444	0.0064354	0.0027199
		Yes	0.0006977	1.0000000	0.1474053	0.0065609	0.0030420
		Yes	0.0013746	1.0000000	0.1503327	0.0062074	0.0020259

Pu-238 Ratio	Pu-239 Ratio	Pu-240 Ratio	Pu-241 Ratio	Pu-242 Ratio	
0.0008366	1.0000000	0.1470467	0.0060678	0.0026357	
0.0005724	1.0000000	0.1426943	0.0050492	0.0029295	
0.0005687	1.0000000	0.1474566	0.0049801	0.0030263	
0.0008529	1.0000000	0.1523031	0.0052795	0.0027947	
0.0013468	1.0000000	0.1510944	0.0060919	0.0020114	
0.0011300	1.0000000	0.1516444	0.0064354	0.0027199	
0.0006977	1.0000000	0.1474053	0.0065609	0.0030420	
0.0013746	1.0000000	0.1503327	0.0062074	0.0020259	

Indiv. Ratio Sum	At% Pu-238	At% Pu-239	At% Pu-240	At% Pu-241	At% Pu-242
1.156587	0.0723	86.4613	12.7138	0.5246	0.2279
1.151245	0.0497	86.8625	12.3948	0.4386	0.2545
1.156032	0.0492	86.5028	12.7554	0.4308	0.2618
1.161230	0.0734	86.1156	13.1157	0.4546	0.2407
1.160545	0.1160	86.1665	13.0193	0.5249	0.1733
1.161930	0.0973	86.0637	13.0511	0.5539	0.2341
1.157706	0.0603	86.3777	12.7325	0.5667	0.2628
1.159941	0.1185	86.2113	12.9604	0.5351	0.1747

TIMS Isotopic Sample Worksheet

Select Isotopes to Report	Pu-238	Pu-239	Pu-240	Pu-241	Pu-242

Output Results -- TIMS CEC21-703 (M&TE 4678061)						
	Yes	0.0008146	1.0000000	0.1422176	0.0039301	0.0024473
TIMS Ratio Average		0.0009105	1.0000000	0.1480217	0.0056225	0.0026259
TIMS Ratio Std Dev		0.0003058	0.0052736	0.0037073	0.0008679	0.0003925
TIMS Avg At%		0.0786807	86.4169427	12.7915808	0.4858773	0.2269184
Avg At% Std Dev		0.0264301	0.4557250	0.3203774	0.0749972	0.0339202
%RSD		33.59	0.53	2.50	15.44	14.95

Reported Results -- TIMS CEC21-703 (M&TE 4678061)					
	0.0008146	1.0000000	0.1422176	0.0039301	0.0024473
Ratio Average	0.0009105	1.0000000	0.1480217	0.0056225	0.0026259
Ratio Std Dev	0.0003058	0.0052736	0.0037073	0.0008679	0.0003925
Avg At%	0.0786807	86.4169427	12.7915808	0.4858773	0.2269184
Avg At% SD	0.0264301	0.4557250	0.3203774	0.0749972	0.0339202
%RSD	33.59	0.53	2.50	15.44	14.95

Calculated At% for Each Individual Scan (For Reference Only)					
1.149410	0.0709	87.0012	12.3731	0.3419	0.2129
Results based on At% from individual Scans (For Reference Use Only)					
At% Average	0.0786	86.4181	12.7907	0.4857	0.2269
At% Std Dev	0.0262	0.4071	0.2730	0.0738	0.0341
%RSD	33.36	0.47	2.13	15.20	15.02

Comments:

No.	Smpl ID/Filament ID/Date	Use?	Pu-238 Ratio	Pu-239 Ratio	Pu-240 Ratio	Pu-241 Ratio	Pu-242 Ratio
6	11-0421-129-D-Pu	Yes	0.0011152	1.0000000	0.1495502	0.0084760	0.0032501
	1211016	Yes	0.0009049	1.0000000	0.1557303	0.0074064	0.0039300
	05/18/11	Yes	0.0005398	1.0000000	0.1514265	0.0086801	0.0045646
		Yes	0.0005899	1.0000000	0.1482850	0.0045611	0.0025112
		Yes	0.0012407	1.0000000	0.1545561	0.0062610	0.0026369
		Yes	0.0011018	1.0000000	0.1559141	0.0078528	0.0035026
		Yes	0.0016259	1.0000000	0.1441689	0.0089214	0.0017276
		Yes	0.0020830	1.0000000	0.1564406	0.0074249	0.0020181
		Yes	0.0001944	1.0000000	0.1457832	0.0084650	0.0024744
	TIMS Ratio Average		0.0010440	1.0000000	0.1513172	0.0075610	0.0029573
	TIMS Ratio Std Dev		0.0005774	0.0075261	0.0046287	0.0013953	0.0009248
	TIMS Avg At%		0.0897733	85.9934393	13.0122874	0.6501935	0.2543065
	Avg At% Std Dev		0.0496498	0.6471972	0.3980373	0.1199851	0.0795251
	%RSD		55.31	0.75	3.06	18.45	31.27

Reported Results -- TIMS CEC21-703 (M&TE 4678061)					
0.0011152	1.0000000	0.1495502	0.0084760	0.0032501	
0.0009049	1.0000000	0.1557303	0.0074064	0.0039300	
0.0005398	1.0000000	0.1514265	0.0086801	0.0045646	
0.0005899	1.0000000	0.1482850	0.0045611	0.0025112	
0.0012407	1.0000000	0.1545561	0.0062610	0.0026369	
0.0011018	1.0000000	0.1559141	0.0078528	0.0035026	
0.0016259	1.0000000	0.1441689	0.0089214	0.0017276	
0.0020830	1.0000000	0.1564406	0.0074249	0.0020181	
0.0001944	1.0000000	0.1457832	0.0084650	0.0024744	
Ratio Average	0.0010440	1.0000000	0.1513172	0.0075610	0.0029573
Ratio Std Dev	0.0005774	0.0075261	0.0046287	0.0013953	0.0009248
Avg At%	0.0897733	85.9934393	13.0122874	0.6501935	0.2543065
Avg At% SD	0.0496498	0.6471972	0.3980373	0.1199851	0.0795251
%RSD	55.31	0.75	3.06	18.45	31.27

Indiv. Ratio Sum	At% Pu-238	At% Pu-239	At% Pu-240	At% Pu-241	At% Pu-242
1.162392	0.0959	86.0295	12.8657	0.7292	0.2796
1.167972	0.0775	85.6185	13.3354	0.6341	0.3365
1.165211	0.0463	85.8214	12.9956	0.7449	0.3917
1.155947	0.0510	86.5091	12.8280	0.3946	0.2172
1.164695	0.1065	85.8594	13.2701	0.5376	0.2264
1.168371	0.0943	85.5892	13.3446	0.6721	0.2998
1.156444	0.1406	86.4720	12.4666	0.7715	0.1494
1.167967	0.1783	85.6189	13.3943	0.6357	0.1728
1.156917	0.0168	86.4366	12.6010	0.7317	0.2139
Results based on At% from individual Scans (For Reference Use Only)					
At% Average	0.0897	85.9950	13.0110	0.6502	0.2541
At% Std Dev	0.0495	0.5929	0.3443	0.1202	0.0789
%RSD	55.22	0.69	2.65	18.48	31.04

No.	Smpl ID/Filament ID/Date	Use?	Pu-238 Ratio	Pu-239 Ratio	Pu-240 Ratio	Pu-241 Ratio	Pu-242 Ratio
7	11-0422-129-S-Pu	Yes	0.0007034	1.0000000	0.1489685	0.0055007	0.0032113
	1211017	Yes	0.0008044	1.0000000	0.1451277	0.0056475	0.0024258
	05/18/11	Yes	0.0011673	1.0000000	0.1535958	0.0066015	0.0027970
		Yes	0.0010867	1.0000000	0.1491745	0.0069001	0.0030832
		Yes	0.0011290	1.0000000	0.1495902	0.0058781	0.0031546
		Yes	0.0010382	1.0000000	0.1500435	0.0061142	0.0022644
		Yes	0.0012014	1.0000000	0.1489405	0.0046306	0.0021060
		Yes	0.0015597	1.0000000	0.1572736	0.0088325	0.0021779
		Yes	0.0017046	1.0000000	0.1452526	0.0057476	0.0027336
	TIMS Ratio Average		0.0011550	1.0000000	0.1497741	0.0062059	0.0026615
	TIMS Ratio Std Dev		0.0003190	0.0057195	0.0037863	0.0011808	0.0004334
	TIMS Avg At%		0.0995836	86.2220250	12.9138262	0.5350824	0.2294828
	Avg At% Std Dev		0.0275032	0.4931448	0.3264655	0.1018097	0.0373664
	%RSD		27.62	0.57	2.53	19.03	16.28

Reported Results -- TIMS CEC21-703 (M&TE 4678061)					
0.0007034	1.0000000	0.1489685	0.0055007	0.0032113	
0.0008044	1.0000000	0.1451277	0.0056475	0.0024258	
0.0011673	1.0000000	0.1535958	0.0066015	0.0027970	
0.0010867	1.0000000	0.1491745	0.0069001	0.0030832	
0.0011290	1.0000000	0.1495902	0.0058781	0.0031546	
0.0010382	1.0000000	0.1500435	0.0061142	0.0022644	
0.0012014	1.0000000	0.1489405	0.0046306	0.0021060	
0.0015597	1.0000000	0.1572736	0.0088325	0.0021779	
0.0017046	1.0000000	0.1452526	0.0057476	0.0027336	
Ratio Average	0.0011550	1.0000000	0.1497741	0.0062059	0.0026615
Ratio Std Dev	0.0003190	0.0057195	0.0037863	0.0011808	0.0004334
Avg At%	0.0995836	86.2220250	12.9138262	0.5350824	0.2294828
Avg At% SD	0.0275032	0.4931448	0.3264655	0.1018097	0.0373664
%RSD	27.62	0.57	2.53	19.03	16.28

Indiv. Ratio Sum	At% Pu-238	At% Pu-239	At% Pu-240	At% Pu-241	At% Pu-242
1.158384	0.0607	86.3272	12.8600	0.4749	0.2772
1.154005	0.0697	86.6547	12.5760	0.4894	0.2102
1.164162	0.1003	85.8987	13.1937	0.5671	0.2403
1.160245	0.0937	86.1887	12.8572	0.5947	0.2657
1.159752	0.0973	86.2253	12.8985	0.5068	0.2720
1.159460	0.0895	86.2470	12.9408	0.5273	0.1953
1.156879	0.1038	86.4395	12.8743	0.4003	0.1820
1.169844	0.1333	85.4815	13.4440	0.7550	0.1862
1.155438	0.1475	86.5472	12.5712	0.4974	0.2366
Results based on At% from individual Scans (For Reference Use Only)					
At% Average	0.0996	86.2233	12.9129	0.5348	0.2295
At% Std Dev	0.0274	0.4381	0.2758	0.0994	0.0375
%RSD	27.51	0.51	2.12	18.60	16.33

No.	Smpl ID/Filament ID/Date	Use?	Pu-238 Ratio	Pu-239 Ratio	Pu-240 Ratio	Pu-241 Ratio	Pu-242 Ratio
8	11-0422-129-D-Pu	Yes	0.0011951	1.0000000	0.1523063	0.0064310	0.0028562
	1211018	Yes	0.0008485	1.0000000	0.1525807	0.0060339	0.0021753
	05/18/11	Yes	0.0020975	1.0000000	0.1475099	0.0031842	0.0014050
		Yes	0.0024531	1.0000000	0.1618240	0.0042327	0.0026904
		Yes	0.0042151	1.0000000	0.1659229	0.0048338	0.0042287
		Yes	0.0052727	1.0000000	0.1422038	0.0102411	0.0017200
		Yes	0.0030856	1.0000000	0.1505182	0.0075363	0.0019662
		No	0.0102164	1.0000000	0.1498233	0.0054770	0.0008525
		No	0.0251029	1.0000000	0.1991176	0.0077574	-0.0109559
	TIMS Ratio Average		0.0060541	1.0000000	0.1579785	0.0061919	0.0007709
	TIMS Ratio Std Dev		0.0076837	0.0313082	0.0170009	0.0021206	0.0045030
	TIMS Avg At%		0.5170046	85.3974255	13.4909591	0.5287752	0.0658357
	Avg At% Std Dev		0.6561697	2.6736421	1.4518294	0.1810964	0.3845466
	%RSD		126.92	3.13	10.76	34.25	584.10

Reported Results -- TIMS CEC21-703 (M&TE 4678061)					
0.0011951	1.0000000	0.1523063	0.0064310	0.0028562	
0.0008485	1.0000000	0.1525807	0.0060339	0.0021753	
0.0020975	1.0000000	0.1475099	0.0031842	0.0014050	
0.0024531	1.0000000	0.1618240	0.0042327	0.0026904	
0.0042151	1.0000000	0.1659229	0.0048338	0.0042287	
0.0052727	1.0000000	0.1422038	0.0102411	0.0017200	
0.0030856	1.0000000	0.1505182	0.0075363	0.0019662	
Ratio Average	0.0027382	1.0000000	0.1532665	0.0060704	0.0024345
Ratio Std Dev	0.0015900	0.0130170	0.0081433	0.0023421	0.0009416
Avg At%	0.2351400	85.8730471	13.1614651	0.5212862	0.2090616
Avg At% SD	0.1365423	1.1178098	0.6992922	0.2011197	0.0808556
%RSD	58.07	1.30	5.31	38.58	38.68

Indiv. Ratio Sum	At% Pu-238	At% Pu-239	At% Pu-240	At% Pu-241	At% Pu-242
1.162789	0.1028	86.0002	13.0984	0.5531	0.2456
1.161638	0.0730	86.0853	13.1350	0.5194	0.1873
1.154197	0.1817	86.6404	12.7803	0.2759	0.1217
1.171200	0.2095	85.3825	13.8169	0.3614	0.2297
1.179201	0.3575	84.8032	14.0708	0.4099	0.3586
1.159438	0.4548	86.2487	12.2649	0.8833	0.1483
1.163106	0.2653	85.9767	12.9411	0.6479	0.1690
Results based on At% from individual Scans (For Reference Use Only)					
At% Average	0.2349	85.8767	13.1582	0.5216	0.2086
At% Std Dev	0.1363	1.0315	0.6136	0.2026	0.0791
%RSD	58.00	1.20	4.66	38.85	37.90

No.	Smpl ID/Filament ID/Date	Use?	Pu-238 Ratio	Pu-239 Ratio	Pu-240 Ratio	Pu-241 Ratio	Pu-242 Ratio
9	11-420-129-S-P	Yes	0.0013726	1.0000000	0.1557230	0.0063764	0.0032113
	1211013	Yes	0.0017442	1.0000000	0.1544908	0.0057209	0.0032573
	05/25/11	Yes	0.0021082	1.0000000	0.1537717	0.0063670	0.0033056
		Yes	0.0032776	1.0000000	0.1561428	0.0062485	0.0026224
		Yes	0.0047430	1.0000000	0.1502786	0.0060286	0.0021173
		Yes	0.0048676	1.0000000	0.1533688	0.0067870	0.0033412
		Yes	0.0046629	1.0000000	0.1542418	0.0060014	0.0026476
		Yes	0.0054208	1.0000000	0.1519449	0.0069198	0.0031544
		Yes	0.0064582	1.0000000	0.1546383	0.0053422	0.0029456
	TIMS Ratio Average		0.0038586	1.0000000	0.1538445	0.0061991	0.0029559
	TIMS Ratio Std Dev		0.0017923	0.0045242	0.0018225	0.0004947	0.0004147
	TIMS Avg At%		0.3299967	85.7008160	13.1846011	0.5312670	0.2533192
	Avg At% Std Dev		0.1535973	0.3877289	0.1561906	0.0424004	0.0355406
	%RSD		46.55	0.45	1.18	7.98	14.03

Reported Results -- TIMS CEC21-703 (M&TE 4678061)					
0.0013726	1.0000000	0.1557230	0.0063764	0.0032113	
0.0017442	1.0000000	0.1544908	0.0057209	0.0032573	
0.0021082	1.0000000	0.1537717	0.0063670	0.0033056	
0.0032776	1.0000000	0.1561428	0.0062485	0.0026224	
0.0047430	1.0000000	0.1502786	0.0060286	0.0021173	
0.0048676	1.0000000	0.1533688	0.0067870	0.0033412	
0.0046629	1.0000000	0.1542418	0.0060014	0.0026476	
0.0054208	1.0000000	0.1519449	0.0069198	0.0031544	
0.0064582	1.0000000	0.1546383	0.0053422	0.0029456	
Ratio Average	0.0038586	1.0000000	0.1538445	0.0061991	0.0029559
Ratio Std Dev	0.0017923	0.0045242	0.0018225	0.0004947	0.0004147
Avg At%	0.3299967	85.7008160	13.1846011	0.5312670	0.2533192
Avg At% SD	0.1535973	0.3877289	0.1561906	0.0424004	0.0355406
%RSD	46.55	0.45	1.18	7.98	14.03

Indiv. Ratio Sum	At% Pu-238	At% Pu-239	At% Pu-240	At% Pu-241	At% Pu-242
1.166683	0.1176	85.7131	13.3475	0.5465	0.2753
1.165213	0.1497	85.8212	13.2586	0.4910	0.2795
1.165553	0.1809	85.7962	13.1930	0.5463	0.2836
1.168291	0.2805	85.5951	13.3651	0.5348	0.2245
1.163168	0.4078	85.9721	12.9198	0.5183	0.1820
1.168365	0.4166	85.5897	13.1268	0.5809	0.2860
1.167554	0.3994	85.6492	13.2107	0.5140	0.2268
1.167440	0.4643	85.6575	13.0152	0.5927	0.2702
1.169384	0.5523	85.5151	13.2239	0.4568	0.2519
Results based on At% from individual Scans (For Reference Use Only)					
At% Average	0.3299	85.7010	13.1845	0.5313	0.2533
At% Std Dev	0.1533	0.3765	0.1454	0.0423	0.0355
%RSD	46.48	0.44	1.10	7.97	14.00

No.	Smpl ID/Filament ID/Date	Use?	Pu-238 Ratio	Pu-239 Ratio	Pu-240 Ratio	Pu-241 Ratio	Pu-242 Ratio
10	11-420-129-D-P	Yes	0.0012596	1.0000000	0.1480662	0.0057363	0.0027234

Reported Results -- TIMS CEC21-703 (M&TE 4678061)					
0.0012596	1.0000000	0.1480662	0.0057363	0.0027234	

Indiv. Ratio Sum	At% Pu-238	At% Pu-239	At% Pu-240	At% Pu-241	At% Pu-242
1.157786	0.1088	86.3718	12.7887	0.4955	0.2352

TIMS Isotopic Sample Worksheet

Select Isotopes to Report	Pu-238	Pu-239	Pu-240	Pu-241	Pu-242

		Output Results – TIMS CEC21-703 (M&TE 4678061)					Reported Results – TIMS CEC21-703 (M&TE 4678061)					Calculated At% for Each Individual Scan (For Reference Only)				
1211014	Yes	0.0013423	1.0000000	0.1484897	0.0063903	0.0024842	0.0013423	1.0000000	0.1484897	0.0063903	0.0024842	1.158707	0.1158	86.3031	12.8151	0.5515	0.2144
05/25/11	Yes	0.0019579	1.0000000	0.1488230	0.0064799	0.0024569	0.0019579	1.0000000	0.1488230	0.0064799	0.0024569	1.159718	0.1688	86.2279	12.8327	0.5587	0.2119
	Yes	0.0019026	1.0000000	0.1506335	0.0055799	0.0027381	0.0019026	1.0000000	0.1506335	0.0055799	0.0027381	1.160854	0.1639	86.1435	12.9761	0.4807	0.2359
	Yes	0.0030506	1.0000000	0.1530704	0.0058119	0.0027452	0.0030506	1.0000000	0.1530704	0.0058119	0.0027452	1.164678	0.2619	85.8606	13.1427	0.4990	0.2357
	Yes	0.0032202	1.0000000	0.1494253	0.0059184	0.0030105	0.0032202	1.0000000	0.1494253	0.0059184	0.0030105	1.161574	0.2772	86.0901	12.8640	0.5095	0.2592
	Yes	0.0035905	1.0000000	0.1537124	0.0060699	0.0032200	0.0035905	1.0000000	0.1537124	0.0060699	0.0032200	1.165593	0.3080	85.7933	13.1875	0.5208	0.1905
	Yes	0.0039135	1.0000000	0.1496795	0.0057392	0.0030698	0.0039135	1.0000000	0.1496795	0.0057392	0.0030698	1.162402	0.3367	86.0288	12.8767	0.4937	0.2641
	Yes	0.0040012	1.0000000	0.1547968	0.0052560	0.0023560	0.0040012	1.0000000	0.1547968	0.0052560	0.0023560	1.166482	0.3430	85.7279	13.2704	0.4568	0.2020
TIMS Ratio Average		0.0026932	1.0000000	0.1507441	0.0058949	0.0026449	Ratio Average	0.0026932	1.0000000	0.1507441	0.0058949	0.0026449	Results based on At% from individual Scans (For Reference Use Only)				
TIMS Ratio Std Dev		0.0010872	0.0000000	0.0042323	0.0002869	0.0002882	Ratio Std Dev	0.0010872	0.0000000	0.0042323	0.0002869	0.0003701	0.0002882	At% Average	0.2316	86.0608	12.9727	0.5074	0.2276
TIMS Avg At%		0.2317736	86.0602324	12.9738698	0.5073135	0.2276207	Avg At%	0.2317736	86.0602324	12.9738698	0.5073135	0.2276207	At% Std Dev	0.0931	0.3318	0.1813	0.0325	0.0249
Avg At% Std Dev		0.0935668	0.3642352	0.2140197	0.0247990	0.0247990	Avg At% SD	0.0935668	0.3642352	0.2140197	0.0318497	0.0247990	%RSD	40.19	0.39	1.40	6.40	10.96
%RSD		40.37	0.42	1.65	6.28	10.89	%RSD	40.37	0.42	1.65	6.28	10.89						
Comments:																		

No.	Simpl ID/Filament ID/Date	Use?	Pu-238 Ratio	Pu-239 Ratio	Pu-240 Ratio	Pu-241 Ratio	Pu-242 Ratio		Pu-238 Ratio	Pu-239 Ratio	Pu-240 Ratio	Pu-241 Ratio	Pu-242 Ratio	Indiv. Ratio Sum	At% Pu-238	At% Pu-239	At% Pu-240	At% Pu-241	At% Pu-242
11	11-421-129-D-P	Yes	0.0010671	1.0000000	0.1503231	0.0061799	0.0032585		0.0010671	1.0000000	0.1503231	0.0061799	0.0032585	1.160829	0.0919	86.1454	12.9496	0.5324	0.2807
	1211016	Yes	0.0014965	1.0000000	0.1547836	0.0057446	0.0028557		0.0014965	1.0000000	0.1547836	0.0057446	0.0028557	1.164880	0.1285	85.8457	13.2875	0.4911	0.2451
	05/25/11	Yes	0.0021285	1.0000000	0.1508051	0.0060632	0.0027647		0.0021285	1.0000000	0.1508051	0.0060632	0.0027647	1.161762	0.1832	86.0762	12.9807	0.5219	0.2380
		Yes	0.0011734	1.0000000	0.1463296	0.0061614	0.0024660		0.0011734	1.0000000	0.1463296	0.0061614	0.0024660	1.156130	0.1015	86.4954	12.6568	0.5329	0.2133
		Yes	0.0017046	1.0000000	0.1455919	0.0051903	0.0024916		0.0017046	1.0000000	0.1455919	0.0051903	0.0024916	1.154978	0.1476	86.5817	12.6056	0.4494	0.2157
		Yes	0.0013910	1.0000000	0.1500000	0.0047471	0.0031093		0.0013910	1.0000000	0.1500000	0.0047471	0.0031093	1.159237	0.1200	86.2656	12.9595	0.4086	0.2682
		Yes	0.0020992	1.0000000	0.1547301	0.0058725	0.0028612		0.0020992	1.0000000	0.1547301	0.0058725	0.0028612	1.165563	0.1801	85.7954	13.2751	0.5038	0.2455
		Yes	0.0017532	1.0000000	0.1481868	0.0054720	0.0021633		0.0017532	1.0000000	0.1481868	0.0054720	0.0021633	1.157575	0.1515	86.3875	12.8015	0.4727	0.1869
		Yes	0.0020429	1.0000000	0.1541788	0.0051821	0.0024220		0.0020429	1.0000000	0.1541788	0.0051821	0.0024220	1.163826	0.1755	85.9235	13.2476	0.4453	0.2081
TIMS Ratio Average			0.0016507	1.0000000	0.1505477	0.0056226	0.0027103	Ratio Average	0.0016507	1.0000000	0.1505477	0.0056226	0.0027103	Results based on At% from individual Scans (For Reference Use Only)					
TIMS Ratio Std Dev			0.0003963	0.0000000	0.0041381	0.0003529	0.0003529	Ratio Std Dev	0.0003963	0.0000000	0.0041381	0.0003529	0.0003529	At% Average	0.1422	86.1683	12.9716	0.4845	0.2335
TIMS Avg At%			0.1422375	86.1674378	12.9723067	0.4844822	0.2335358	Avg At%	0.1422375	86.1674378	12.9723067	0.4844822	0.2335358	At% Std Dev	0.0139	0.3654	0.2578	0.0435	0.0302
Avg At% Std Dev			0.0341464	0.4082690	0.2999862	0.0437259	0.0304104	Avg At% SD	0.0341464	0.4082690	0.2999862	0.0437259	0.0304104	%RSD	23.86	0.42	1.99	8.98	12.91
%RSD			24.01	0.47	2.31	9.03	13.02	%RSD	24.01	0.47	2.31	9.03	13.02						
Comments																			

Final Reported Results

TIMS CEC21-703 (M&TE 4678061)

Smpl #	Sample	Filament	Run Date	Pu-238			Pu-239			Pu-240			Pu-241			Pu-242		
				At%	Std Dev	%RSD	At%	Std Dev	%RSD	At%	Std Dev	%RSD	At%	Std Dev	%RSD	At%	Std Dev	%RSD
1	11-0419-129-S-Pu	1211010	05/18/11	0.095	± 0.052	54.93	86.09	± 0.73	0.84	12.91	± 0.50	3.91	0.674	± 0.104	15.49	0.231	± 0.067	28.86
2	11-0419-129-D-Pu	1211011	05/18/11	0.155	± 0.037	24.09	86.17	± 0.47	0.54	12.91	± 0.35	2.73	0.515	± 0.047	9.09	0.250	± 0.032	12.89
			Average =	**0.125**	**± 0.043**		**86.13**	**± 0.06**		**12.9**	**± 0.00**		**0.595**	**± 0.112**		**0.240**	**± 0.013**	
			RPD	48			0.1			0.04			27			8		
12	11-419-129-PB-P	1211012	05/18/11	(ND)			(ND)			(ND)			(ND)			(ND)		
9	11-420-129-S-P	1211013	05/25/11	0.330	± 0.154	46.55	85.70	± 0.39	0.45	13.18	± 0.16	1.18	0.531	± 0.042	7.98	0.253	± 0.036	14.03
10	11-420-129-D-P	1211014	05/25/11	0.232	± 0.094	40.37	86.06	± 0.36	0.42	12.97	± 0.21	1.65	0.507	± 0.032	6.28	0.228	± 0.025	10.89
			Average =	**0.281**	**n/a**	**n/a**	**85.88**	**n/a**	**n/a**	**13.08**	**n/a**	**n/a**	**0.519**	**n/a**	**n/a**	**0.240**	**n/a**	**n/a**
			RPD	35			0.4			1.62			5			11		
5	11-0421-129-S-Pu	1211015	05/18/11	0.079	± 0.026	33.59	86.42	± 0.46	0.53	12.79	± 0.32	2.50	0.486	± 0.075	15.44	0.227	± 0.034	14.95
11	11-421-129-D-P	1211016	05/25/11	0.142	± 0.034	24.01	86.17	± 0.41	0.47	12.97	± 0.30	2.31	0.484	± 0.044	9.03	0.234	± 0.030	13.02
			Average =	**0.110**	**± 0.045**	**40.69**	**86.29**	**± 0.18**	**0.20**	**12.9**	**± 0.13**	**0.99**	**0.485**	**± 0.001**	**0.20**	**0.230**	**± 0.005**	**2.03**
			RPD	58			0.3			1			-0.288			3		
7	11-0422-129-S-Pu	1211017	05/18/11	0.100	± 0.028	27.62	86.22	± 0.49	0.57	12.91	± 0.33	2.53	0.535	± 0.102	19.03	0.229	± 0.037	16.28
8	11-0422-129-D-Pu	1211018	05/18/11	0.235	± 0.137	58.07	85.87	± 1.12	1.30	13.16	± 0.70	5.31	0.521	± 0.201	38.58	0.209	± 0.081	38.68
			Average =	**0.167**	**± 0.096**	**57.27**	**86.05**	**± 0.25**	**0.29**	**13.0**	**± 0.18**	**1.34**	**0.528**	**± 0.010**	**1.85**	**0.219**	**± 0.014**	**6.59**
			RPD	81			0.4			2			3			9		

Spreadsheet Review Form

Spreadsheet Author Name:	SJ Bos	Review Date:	June 17, 2011
Date Prepared:	6/15/2011	Spreadsheet Subject:	Thermal ionization mass spectrometery data
Reviewer Name:	JC Braley	File Name:	KW210 TIMS data June 2011
Reviewer Title:	Scientist	Revision No.	Rev 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:
☒	100% Verification Review (Verification of all cells/calculations)		

REVIEW CHECK LIST

Spreadsheet/Calculation Identification

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

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

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

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

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

Reviewer Sign/Date: Jeff C Braley 7/15/11

76

3/1/11 K Basin "cont" SB 3-4-11

SB 030 11 12 235/238 = 3.021 / 3.046 = .992 3.05 amps

SB 11-0076 u 3/1/11 1 ul of Sample diluted in 200 ul of .5M HNO3 3.15

SB 11-0076 UD 1 ul " " 3.10

SB 11-0077u 1 ul diluted sample 3.20

SB 11 0077UD 3.15

SB 11 0078u 3.1

SB 11 0078UD 3.15

SB 11-0079u 3.2

SB 11-0079UD 3.15

SB 3/2/11

3/2/11 SB 030 11 13 5 ul 235/238 =

SB 030 11 13.1 Rerun filament 3.2 amps

3.05 amps

SB

Project No. Date of Work

Entered By SB Date 4-4-11

Disclosed To and Understood By

Signed 1. Date 4.4.11

2. Date

LRB 56999

3/2011 K-Basin 77

SB	030 11 14.1	5 ml	235/238 =	.03695/.03046 = .0004		3.25 amps
SB	030 11 15.1	5 ml	all Hi ↑	(Lab Temp To High)		3.25 amps
SB	030 11 16.1	5 ml	235/238	3.0167 / 3.046 =		3.20
SB	030 11 16.2	5 ml	Lab Temp went Hi	SB 5/6/11 SB 5/6/11 = -.010		3.2
SB	030 11 17.1	5 ml				3.2
SB	030 11 17.2	5 ml				3.2

no change to Mass Bias Factor

	Samples	dl. Metal	Lot #	HNO_3		
SB	TI 001-A1-B	11-0419-129-S-DL	11 11 010	5 ml	3.3 amps	
SB	TI 001-A1-C	11-0419-129-D-DL	11 11 011	5 ml	3.1 amps	
~~SB~~	~~TI 001~~ SB 5/6/11	11-0419-129-PB DL	11 11 012	10 ml	3.3	
SB	TI 001-A4-B	11-0420-129 S DL	11 11 013	5 ml	3.3	
SB	TI 002-A4-C	11-0420-129 D DL	11 11 014	5 ml	3.3	
SB	TI 002-~~A4-C~~ B1-B	11-~~0420~~ 0421-129 S DL SB 5/6/11	11 11 015	5 ml	3.4	
SB	TI 002-B1-C	11-0422-129 D DL	11 11 016	5 ml	3.2	
SB	TI 001-B3-B	11-0422-129 S DL	11 11 017	7 ml	3.3	
SB	TI 001-B3-C	11-0422-129 D DL SB 5/6/11	11 11 018	NOT Run		

9/11 SB TI 001-B3-B Repeat No 234 Rerun Sample

SB	030 11 18.1	5 ml	235/238	3.0198 / 3.046 =		
SB	TI-001-B3-B	5 ml	11-0422-129-S DL	11-11017		3.3 amps
SB	TI-001-B3-C		11-0422-129 D DL	11-11018		3.3
SB	TI 002-A4-C		11-0420-129-D DL	11 11014		3.3
SB	TI001-B3-B	Rerun To include	11 33	11 11017		3.3
SB	11 030	03011 19				3.3

SB	Pu 946	946 11006.1	24/239	.14369 / .14489 = .992		2.25 amps
SB	"	946 11006.2		.1437 / .1449 = .992		"
SB	Pu 946	946 11006.3				"

no change to mass bias factor SB 5/6/11

9/11 SB	Pu 946	946 11007.1 & 002 SB 5/12/11				2.25
SB	TI001-A1-B	11-0419-129-S-DL Pu	12 11 010			2.25
SB	TI 001-A1-C	11-0419-129-D-Pu	12 11 011			2.25
SB		11-0419-129-PB-Pu	12 11 012	No Signal		2.25

Project No. _____ Date of Work _____

Entered By SB Date 5/12/11

Disclosed To and Understood By

Signed 1. Jeri C Bailey Date July 13, 2011

78 K-Basin-2 cont

5/13/11	SB	TI 002-A4-B	11-0420-129-S-Pu	121013	(not used)	5-18-11 SB ~~3.5 amps~~
	SB	TI 002-A4-C	11-0420-129-D-Pu	121014	(not used)	2.3 amp
	SB	TI 002-B1-B	11-0421-129-S-Pu	121015		2.3
	SB	TI 002-B1-C	11-0421-129-D-Pu	121016	(not used)	2.35
	SB	TI 001-B3-B	SB 7/15/11 ~~11-0421~~ 0422 -129-S-Pu	121017		2.30
	SB	TI 001-B3-C	11-~~0421~~ 0422 -129-D-Pu SB 7/15/11	121018		
	SB	946 11008.1				~~2.35 amps~~
	SB 5/25/11					
5/25/11	SB	946 11009.1	~~275/2~~ 240/239 = .1455/.1449 =			2.3 amps
	SB	946 11009.2	.1449/.1449 = .000			"
	SB	TI 002-A4-B	11-0420-129-S-Pu Rerun			2.3
	SB	TI 002-A4-C	11-0420-129-~~D~~ D Pu Rerun SB 5/25/11			2.3
	SB	TI-002-B1-D	11-0421-129-S-Pu Rerun		Claw (W) signal	2.35
	SB	TI-002-B1-C	11-0421-129-D-Pu Rerun			2.30
	SB	"	"		Rerun same filament	2.3
	SB	946 11010.1				2.4

SJ Boss 7/11/2011

Project No. _____ Date of Work _____

Entered By SJ Boss Date 7/11/2011

Disclosed To and Understood By

Signed 1. Daniel C Bradley Date July 13, 2011

2. _____ Date _____

LEB 06777

MASS SPECTROMETER LABORATORIES

ANALYTICAL CHEMISTRY
325 BUILDING

CEC21-703A(M&TE 467806)
PROCEDURE NO.: PNL-MA-599, PNNL-98523-264

CUSTOMER: Sandy Fiskum RUN # 5 MASS DISC. FACTOR: .002
SAMPLE I.D.: TI001-A1-B 11-0419 -139-S-U 6 May 2011 11:45:07
LABORATORY LOG-IN NUMBER: 1111010 SB 6/13/11 ANALYST: Stan Bos
ALO LOG-IN NUMBER: 11-0419~~US~~ -S-U SB 6/13/11 LRB: BNW 56999 PAGE 76

REFERENCE MASS: 238 REVIEWED BY: PK B
DATE: 5/16/11

MEASURED MASSES:

233	234	235	236	238

RATIOS:

1.26E-5	7.45E-5	.0073871	.0008011	1
2.04E-5	7.58E-5	.007136	.0006794	1
1.06E-5	7.48E-5	.0071318	.0006903	1
3.2E-6	7.86E-5	.0071274	.0007567	1
3.3E-6	9.98E-5	.0070314	.0007477	1
1.43E-5	6.7E-5	.007292	.0008204	1
1.E-7	9.09E-5	.0070186	.000684	1
2.29E-5	6.25E-5	.0071631	.0007198	1
4.39E-5	9.47E-5	.0072125	.0007537	1

NO SCANS REJECTED AT THE 95% LEVEL

MEAN RATIO FOR EACH ISOTOPE IS:

1.46E-5	7.99E-5	.0071667	.0007392	1

STANDARD DEVIATION OF RATIO FOR EACH ISOTOPE IS:

1.35E-5	1.26E-5	.0001174	5.05E-5	0

MASS	ATOM %	MASS %	Mean Ratio	STD
233	.0014465	.0014162	.0000146	.0000135
234	.0079220	.0077897	.0000799	.0000126
235	.7109772	.7020837	.0071667	.0001174
236	.0733360	.0727268	.0007392	.0000505
238	99.2063184	99.2159837	1.0000000	0.0000000

MASS SPECTROMETER LABORATORIES

ANALYTICAL CHEMISTRY
325 BUILDING

CEC21-703A(M&TE 467806)
PROCEDURE NO.: PNL-MA-599, PNNL-98523-264

CUSTOMER: Sandy Fiskum RUN # 6 MASS DISC. FACTOR: .002
SAMPLE I.D.: TI001-A1-C 11-0419-179-D-LL 6 May 2011 12:20:54
LABORATORY LOG-IN NUMBER: 1111011 SB 6/7/11 ANALYST: Stan Bos
ALO LOG-IN NUMBER: 11-0419~~UD~~ -D-LL SB 6/13/11 LRB: BNW 56999 PAGE 76

REFERENCE MASS: 238 REVIEWED BY: PKB
DATE: 5/16/11

MEASURED MASSES:

233	234	235	236	238

RATIOS:

-3.3E-6	5.07E-5	.0072942	.0007406	1
3.5E-6	7.64E-5	.0071751	.0007371	1
9.7E-6	6.57E-5	.0071938	.0007816	1
2.7E-6	5.31E-5	.0070837	.0008105	1
1.1E-6	5.88E-5	.0070954	.000768	1
1.17E-5	6.83E-5	.0070771	.0007373	1
1.48E-5	7.96E-5	.0071418	.0007686	1
8.5E-6	7.6E-5	.0072212	.000751	1
1.26E-5	7.83E-5	.0070528	.0007698	1

NO SCANS REJECTED AT THE 95% LEVEL

MEAN RATIO FOR EACH ISOTOPE IS:

6.8E-6	6.74E-5	.0071484	.0007627	1

STANDARD DEVIATION OF RATIO FOR EACH ISOTOPE IS:

6.1E-6	1.11E-5	7.95E-5	2.42E-5	0

MASS	ATOM %	MASS %	Mean Ratio	STD
233	.0006754	.0006613	.0000068	.0000061
234	.0066902	.0065784	.0000674	.0000111
235	.7091722	.7003010	.0071484	.0000795
236	.0756665	.0750379	.0007627	.0000242
238	99.2077957	99.2174214	1.0000000	0.0000000

MASS SPECTROMETER LABORATORIES

ANALYTICAL CHEMISTRY
325 BUILDING

CEC21-703A(M&TE 467806)
PROCEDURE NO.: PNL-MA-599, PNNL-98523-264

CUSTOMER: Sandy Fiskum RUN # 7 MASS DISC. FACTOR: .002
SAMPLE I.D.: Process Blank 11-0419-129-PB-IC 6 May 2011 12:45:46
LABORATORY LOG-IN NUMBER: 1111012 5/6/11 ANALYST: Stan Bos
ALO LOG-IN NUMBER: 11-0419PB LRB: BNW 56999 PAGE 76

REFERENCE MASS: 238 REVIEWED BY: PKB
DATE: 5/16/11

MEASURED MASSES:

233	234	235	236	238

RATIOS:

2.6E-6	-.0006611	.0085665	.0002158	1
1.48E-5	1.74E-5	.0101087	.0003697	1
3.06E-5	3.01E-5	.0079469	.0003888	1

MEAN RATIO FOR EACH ISOTOPE IS:

1.6E-5	-.0002045	.008874	.0003248	1

STANDARD DEVIATION OF RATIO FOR EACH ISOTOPE IS:

1.4E-5	.0003955	.0011132	9.49E-5	0

MASS	ATOM %	MASS %	Mean Ratio	STD
233	.0015884	.0015552	.0000160	.0000140
234	-.0202700	-.0199315	-.0002045	.0003955
235	.8794804	.8684904	.0088740	.0011132
236	.0321881	.0319211	.0003248	.0000949
238	99.1070132	99.1179648	1.0000000	0.0000000

.1 Volt 238 signal
not enough sample to run isotopic analysis.
5/6/11

MASS SPECTROMETER LABORATORIES

ANALYTICAL CHEMISTRY
325 BUILDING

CEC21-703A(M&TE 467806)
PROCEDURE NO.: PNL-MA-599, PNNL-98523-264

CUSTOMER: Sandy Fiskum RUN # 8 MASS DISC. FACTOR: .002
SAMPLE I.D.: TI002-A4-B 11-0420-129-S-LL 6 May 2011 13:21:55
LABORATORY LOG-IN NUMBER: 1111013 SA 6/3/11 ANALYST: Stan Bos
ALO LOG-IN NUMBER: 11-0420SU LRB: BNW 56999 PAGE 76

REFERENCE MASS: 238 REVIEWED BY: P.K.B.
DATE: 5/16/11

MEASURED MASSES:

233	234	235	236	238
RATIOS:				
6.7E-6	4.6E-5	.0071569	.0006965	1
-1.37E-5	3.7E-5	.0072004	.0006575	1
4.1E-6	8.93E-5	.0070636	.0006696	1
-2.9E-6	4.09E-5	.0069314	.0007204	1
5.6E-6	.0001148	.0072038	.0007654	1
5.6E-6	5.88E-5	.0070515	.0007196	1
-5.1E-6	7.75E-5	.0071077	.0007614	1
1.94E-5	.0001026	.0072254	.0007657	1
5.8E-6	5.26E-5	.0066953	.0007737	1

NO SCANS REJECTED AT THE 95% LEVEL

MEAN RATIO FOR EACH ISOTOPE IS:

2.8E-6	6.88E-5	.0070707	.0007255	1

STANDARD DEVIATION OF RATIO FOR EACH ISOTOPE IS:

9.3E-6	2.83E-5	.0001688	4.4E-5	0

MASS	ATOM %	MASS %	Mean Ratio	STD
233	.0002799	.0002741	.0000028	.0000093
234	.0068289	.0067147	.0000688	.0000283
235	.7015473	.6927705	.0070707	.0001688
236	.0719858	.0713877	.0007255	.0000440
238	99.2193582	99.2288530	1.0000000	0.0000000

MASS SPECTROMETER LABORATORIES

ANALYTICAL CHEMISTRY
325 BUILDING

CEC21-703A(M&TE 467806)
PROCEDURE NO.: PNL-MA-599, PNNL-98523-264

CUSTOMER: Sandy Fiskum RUN # 5 MASS DISC. FACTOR: .002
SAMPLE I.D.: TI002-A4-C 11-0420-139-D-u 9 May 2011 15:04:19
LABORATORY LOG-IN NUMBER: 1111014 5 Bos 6/7/11 ANALYST: Stan Bos SJB 5/9/2011
ALO LOG-IN NUMBER: 11-0420DU LRB: BNW 56999 PAGE 77

REFERENCE MASS: 238 REVIEWED BY: PKB
DATE: 5/16/11

MEASURED MASSES:

233	234	235	236	238

RATIOS:

1.73E-5	6.94E-5	.0073048	.0008158	1
3.52E-5	7.87E-5	.0074368	.00076	1
1.1E-6	8.52E-5	.0071936	.0007272	1
1.76E-5	3.69E-5	.0070726	.0006024	1
-2.7E-6	6.39E-5	.0070787	.0007365	1
1.71E-5	.0001	.006704	.000725	1
1.5E-5	7.69E-5	.0067339	.0008175	1
1.06E-5	6.06E-5	.007417	.000696	1
-5.E-7	.0001025	.0069753	.0008318	1

NO SCANS REJECTED AT THE 95% LEVEL

MEAN RATIO FOR EACH ISOTOPE IS:

1.23E-5	7.49E-5	.0071019	.0007458	1

STANDARD DEVIATION OF RATIO FOR EACH ISOTOPE IS:

1.19E-5	2.04E-5	.0002675	7.21E-5	0

MASS	ATOM %	MASS %	Mean Ratio	STD
233	.0012199	.0011944	.0000123	.0000119
234	.0074303	.0073061	.0000749	.0000204
235	.7045946	.6957803	.0071019	.0002675
236	.0739927	.0733780	.0007458	.0000721
238	99.2127626	99.2223412	1.0000000	0.0000000

MASS SPECTROMETER LABORATORIES

ANALYTICAL CHEMISTRY
325 BUILDING

CEC21-703A(M&TE 467806)
PROCEDURE NO.: PNL-MA-599, PNNL-98523-264

CUSTOMER: Sandy Fiskum RUN # 10 MASS DISC. FACTOR: .002
SAMPLE I.D.: TI002-B1-B 11-0421-135-S-U 6 May 2011 14:39:12
LABORATORY LOG-IN NUMBER: 1111015 5505 6/7/11 ANALYST: Stan Bos
ALO LOG-IN NUMBER: 11-0421SU LRB: BNW 56999 PAGE 76

REFERENCE MASS: 238 REVIEWED BY:
DATE: 5/16/11

MEASURED MASSES:

233	234	235	236	238

RATIOS:

-1.53E-5	5.89E-5	.0073381	.0007278	1
-7.9E-6	9.02E-5	.0073466	.000697	1
-8.1E-6	2.98E-5	.0079459	.0005783	1
1.65E-5	6.11E-5	.0072751	.0008526	1
7.62E-5	4.35E-5	.0070924	.0007738	1
6.9E-6	7.53E-5	.0068096	.0006555	1
5.01E-5	.0001098	.0066943	.0006104	1
3.62E-5	8.11E-5	.0069421	.0007295	1
-1.1E-6	5.33E-5	.0065905	.0008475	1

NO SCANS REJECTED AT THE 95% LEVEL

MEAN RATIO FOR EACH ISOTOPE IS:

1.71E-5	6.7E-5	.007115	.0007192	1

STANDARD DEVIATION OF RATIO FOR EACH ISOTOPE IS:

3.1E-5	2.47E-5	.0004176	9.6E-5	0

MASS	ATOM %	MASS %	Mean Ratio	STD
233	.0016931	.0016577	.0000171	.0000310
234	.0066464	.0065353	.0000670	.0000247
235	.7059076	.6970769	.0071150	.0004176
236	.0713519	.0707591	.0007192	.0000960
238	99.2144010	99.2239710	1.0000000	0.0000000

MASS SPECTROMETER LABORATORIES

ANALYTICAL CHEMISTRY
325 BUILDING

CEC21-703A(M&TE 467806)
PROCEDURE NO.: PNL-MA-599, PNNL-98523-264

CUSTOMER: Sandy Fiskum RUN # 11 MASS DISC. FACTOR: .002
SAMPLE I.D.: TI002-B1-C 11-0421-139-D-u 6 May 2011 15:19:17
LABORATORY LOG-IN NUMBER: 1111016 5/6/2011 ANALYST: Stan Bos
ALO LOG-IN NUMBER: 11-0421SDU LRB: BNW 56999 PAGE 76

REFERENCE MASS: 238 REVIEWED BY:
DATE: 5/16/11

MEASURED MASSES:

233	234	235	236	238

RATIOS:

8.E-7	4.68E-5	.0071683	.0007082	1
1.55E-5	6.31E-5	.0071429	.0006125	1
1.56E-5	5.76E-5	.0070182	.0006884	1
1.54E-5	7.88E-5	.0070003	.0007232	1
3.54E-5	5.25E-5	.007047	.0008114	1
3.97E-5	9.09E-5	.0070358	.0007708	1
4.6E-5	6.49E-5	.0070563	.000775	1
4.84E-5	7.02E-5	.0073415	.0007423	1
3.81E-5	.0001137	.0070111	.0007212	1

NO SCANS REJECTED AT THE 95% LEVEL

MEAN RATIO FOR EACH ISOTOPE IS:

2.83E-5	7.09E-5	.0070913	.0007281	1

STANDARD DEVIATION OF RATIO FOR EACH ISOTOPE IS:

1.67E-5	2.09E-5	.0001104	5.76E-5	0

MASS	ATOM %	MASS %	Mean Ratio	STD
233	.0028093	.0027506	.0000283	.0000167
234	.0070374	.0069198	.0000709	.0000209
235	.7035560	.6947547	.0070913	.0001104
236	.0722399	.0716397	.0007281	.0000576
238	99.2143574	99.2239352	1.0000000	0.0000000

MASS SPECTROMETER LABORATORIES

ANALYTICAL CHEMISTRY
325 BUILDING

CEC21-703A(M&TE 467806)
PROCEDURE NO.: PNL-MA-599, PNNL-98523-264

CUSTOMER: Sandy Fiskum RUN # 6 MASS DISC. FACTOR: .002
SAMPLE I.D.: TI001-B3-B 11-0422-129-5-a 9 May 2011 15:39:26
LABORATORY LOG-IN NUMBER: 1111017 BB 5/7/11 ANALYST: Stan Bos SJB
ALO LOG-IN NUMBER: 11-0422SU LRB: BNW 56999 PAGE 77

REFERENCE MASS: 238 REVIEWED BY: PKB
DATE: 5/14/11

MEASURED MASSES:

233	234	235	236	238

RATIOS:

2.29E-5	5.46E-5	.0071644	.0007	1
8.1E-6	6.58E-5	.0072367	.0008346	1
-6.3E-6	4.13E-5	.0071768	.0008351	1
9.5E-6	7.53E-5	.0069076	.0006631	1
8.9E-6	.00012	.007218	.0007135	1
6.2E-6	.0001123	.0070834	.000722	1
3.96E-5	8.04E-5	.0073273	.0007594	1
1.67E-5	4.88E-5	.0071787	.0006834	1
1.79E-5	9.17E-5	.0071811	.0006534	1

NO SCANS REJECTED AT THE 95% LEVEL

MEAN RATIO FOR EACH ISOTOPE IS:

1.37E-5	7.67E-5	.0071638	.0007294	1

STANDARD DEVIATION OF RATIO FOR EACH ISOTOPE IS:

1.28E-5	2.74E-5	.0001161	6.76E-5	0

MASS	ATOM %	MASS %	Mean Ratio	STD
233	.0013588	.0013303	.0000137	.0000128
234	.0076087	.0074815	.0000767	.0000274
235	.7107058	.7018155	.0071638	.0001161
236	.0723609	.0717598	.0007294	.0000676
238	99.2079660	99.2176129	1.0000000	0.0000000

MASS SPECTROMETER LABORATORIES

ANALYTICAL CHEMISTRY
325 BUILDING

CEC21-703A(M&TE 467806)
PROCEDURE NO.: PNL-MA-599, PNNL-98523-264

CUSTOMER: Sandy Fiskum RUN # 4 MASS DISC. FACTOR: .002
SAMPLE I.D.: TI001-B3-C 11-422-139-1-a 9 May 2011 14:27:06
LABORATORY LOG-IN NUMBER: 1111018 SB 5/7/11 ANALYST: Stan Bos SJ Bos
ALO LOG-IN NUMBER: 11-0422DU LRB: BNW 56999 PAGE 76

REFERENCE MASS: 238 REVIEWED BY: F.K.B
DATE: 5/16/11

MEASURED MASSES:

233	234	235	236	238

RATIOS:

1.94E-5	9.97E-5	.0073447	.0007931	1
7.E-6	6.78E-5	.0072729	.0007109	1
-1.06E-5	.0001013	.0071895	.0008124	1
-4.9E-6	.0001618	.0074768	.000865	1
6.1E-6	.000196	.007034	.0008425	1
2.E-6	5.37E-5	.0069269	.0007912	1
-5.7E-6	7.66E-5	.0069584	.0007569	1
-1.28E-5	.0001051	.0071822	.0007573	1
2.96E-5	.0001228	.0073873	.0006755	1

NO SCANS REJECTED AT THE 95% LEVEL

MEAN RATIO FOR EACH ISOTOPE IS:

3.3E-6	.0001094	.007197	.0007783	1

STANDARD DEVIATION OF RATIO FOR EACH ISOTOPE IS:

1.4E-5	4.54E-5	.0001933	6.04E-5	0

MASS	ATOM %	MASS %	Mean Ratio	STD
233	.0003318	.0003248	.0000033	.0000140
234	.0108518	.0106704	.0001094	.0000454
235	.7139215	.7049918	.0071970	.0001933
236	.0772069	.0765656	.0007783	.0000604
238	99.1976881	99.2074473	1.0000000	0.0000000

U. S. Department of Commerce
Maurice H. Stans
Secretary

National Bureau of Standards
A. V. Astin, Director

Certificate

Standard Reference Material U-030
Uranium Isotopic Standard

	^{234}U	^{235}U	^{236}U	^{238}U
Atom percent	0.0190	3.046	0.0204	96.915
	±.0001	±.003	±.0001	±.003
Weight percent	0.0187	3.009	0.0202	96.953

The material consists of highly purified oxide, U_3O_8. The atomic weight of the material is calculated to be 237.958 using the nuclidic masses 234.0409; 235.0439; 236.0457 and 238.0508.

The values for ^{234}U and ^{236}U were calculated from measurements at the National Bureau of Standards. The samples were spiked with high-purity ^{233}U to approximate the ^{234}U concentration, the ratios ^{233}U to ^{234}U and ^{233}U to ^{236}U were measured on a triple-filament equipped surface ionization mass spectrometer with ion-multiplier amplifier circuits.

The values for ^{235}U and ^{238}U were calculated from measurements of the ^{235}U to ^{238}U ratio made at the National Bureau of Standards on a triple-filament, surface ionization mass spectrometer equipped with dc amplifier circuits. The observed ratios were corrected for mass discrimination effects by intercomparison with synthetic mixtures prepared at the 3 percent ^{235}U level from high-purity ^{235}U and ^{238}U.

The limits indicated for the isotopic concentrations are at least as large as the 95-percent confidence limits for a single determination, and include terms for inhomogeneities in the material as well as analytical error. The ^{235}U to ^{238}U ratio for this standard, 0.03143, is known to at least 0.1 percent.

Mass spectrometry measurements at NBS were made by E. L. Garner on solutions prepared by L. A. Machlan.

The overall direction and coordination of the technical measurements leading to certification were performed under the chairmanship of W. R. Shields.

The technical and support aspects in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by J. L. Hague.

Washington, D. C. 20234
April 21, 1969

W. Wayne Meinke, Chief
Office of Standard Reference Materials

(This certificate supersedes certificate of October 1, 1958)

MASS SPECTROMETER LABORATORIES

ANALYTICAL CHEMISTRY
325 BUILDING

CEC21-703A(M&TE 467806)
PROCEDURE NO.: PNL-MA-599, PNNL-98523-264

CUSTOMER: Sandy Fiskum RUN # 1 MASS DISC. FACTOR: .002
SAMPLE I.D.: U-030 3 May 2011 13:09:12
LABORATORY LOG-IN NUMBER: 0301114.1 ANALYST: Stan Bos
ALO LOG-IN NUMBER: LRB: BNW 56999 PAGE 76

REFERENCE MASS: 238 REVIEWED BY:
DATE: 5/16/11

MEASURED MASSES:

234	235	236	238

RATIOS:

.0001724	.0308798	.0001998	1
.0001539	.0306582	.0002461	1
.0001774	.0312367	.00022	1
.0002066	.0313486	.0002818	1
.0002068	.0312343	.0001997	1
.000196	.0305761	.0002075	1
.0002131	.0309255	.0001794	1
.0002312	.030876	.0002154	1
.0001791	.0308527	.0002517	1

NO SCANS REJECTED AT THE 95% LEVEL

MEAN RATIO FOR EACH ISOTOPE IS:

.0001929	.0309542	.0002224	1

STANDARD DEVIATION OF RATIO FOR EACH ISOTOPE IS:

2.41E-5	.0002663	3.18E-5	0

MASS	ATOM %	MASS %	Mean Ratio	STD
234	.0187075	.0184001	.0001929	.0000241
235	3.0012722	2.9645773	.0309542	.0002663
236	.0215610	.0213881	.0002224	.0000318
238	96.9584592	96.9956345	1.0000000	0.0000000

MASS SPECTROMETER LABORATORIES

ANALYTICAL CHEMISTRY
325 BUILDING

CEC21-703A(M&TE 467806)
PROCEDURE NO.: PNL-MA-599, PNNL-98523-264

CUSTOMER: Sandy Fiskum RUN # 1 MASS DISC. FACTOR: .002
SAMPLE I.D.: U-030 6 May 2011 09:53:08
LABORATORY LOG-IN NUMBER: 0301116.1 ANALYST: Stan Bos
ALO LOG-IN NUMBER: LRB: BNW 56999 PAGE 76

REFERENCE MASS: 238 REVIEWED BY:
DATE: 5/16/11

MEASURED MASSES:

234	235	236	238

RATIOS:

.0001608	.0285448	.0002397	1
.0001702	.030826	.0001655	1
.0002109	.0310466	.0001864	1
.000229	.0307557	.0002278	1
.0002337	.0314748	.000203	1
.0002009	.0313715	.0001987	1
.0001689	.0307662	.0002154	1
.0001782	.0315104	.0001879	1
.0002153	.0311957	.0002102	1

CYCLE # 1 REJECTED AT THE 95% LEVEL
DUE TO THE 235/ 238 RATIO

MEAN RATIO FOR EACH ISOTOPE IS:

.0002009	.0311184	.0001994	1

STANDARD DEVIATION OF RATIO FOR EACH ISOTOPE IS:

2.58E-5	.0003155	1.94E-5	0

MASS	ATOM %	MASS %	Mean Ratio	STD
234	.0194750	.0191551	.0002009	.0000258
235	3.0167530	2.9798744	.0311184	.0003155
236	.0193279	.0191729	.0001994	.0000194
238	96.9444441	96.9817976	1.0000000	0.0000000

MASS SPECTROMETER LABORATORIES

ANALYTICAL CHEMISTRY
325 BUILDING

CEC21-703A(M&TE 467806)
PROCEDURE NO.: PNL-MA-599, PNNL-98523-264

CUSTOMER: Sandy Fiskum RUN # 3 MASS DISC. FACTOR: .002
SAMPLE I.D.: U-030 6 May 2011 10:48:28
LABORATORY LOG-IN NUMBER: 0301117.1 ANALYST: Stan Bos
ALO LOG-IN NUMBER: LRB: BNW 56999 PAGE 76

REFERENCE MASS: 238 REVIEWED BY:
DATE: 5/16/11

MEASURED MASSES:

234	235	236	238

RATIOS:

.0001801	.0315838	.0001869	1
.0002123	.0309041	.0002033	1
.0002069	.0309864	.0001665	1
.0001839	.0313066	.0002222	1
.0001904	.0316178	.0002065	1
.0001924	.0311908	.0001908	1
.0002359	.0312499	.0001478	1
.0002347	.0312294	.0001703	1
.0002292	.031285	.0001573	1

NO SCANS REJECTED AT THE 95% LEVEL

MEAN RATIO FOR EACH ISOTOPE IS:

.0002073	.0312615	.0001835	1

STANDARD DEVIATION OF RATIO FOR EACH ISOTOPE IS:

2.2E-5	.0002353	2.48E-5	0

MASS	ATOM %	MASS %	Mean Ratio	STD
234	.0200955	.0197655	.0002073	.0000220
235	3.0302398	2.9932013	.0312615	.0002353
236	.0177867	.0176440	.0001835	.0000248
238	96.9318780	96.9693892	1.0000000	0.0000000

MASS SPECTROMETER LABORATORIES

ANALYTICAL CHEMISTRY
325 BUILDING

CEC21-703A(M&TE 467806)
PROCEDURE NO.: PNL-MA-599, PNNL-98523-264

CUSTOMER: Sandy Fiskum RUN # 2 MASS DISC. FACTOR: .002
SAMPLE I.D.: U-030 9 May 2011 12:53:55
LABORATORY LOG-IN NUMBER: 1118.1 ANALYST: Stan Bos
ALO LOG-IN NUMBER: LRB: BNW 56999 PAGE 76

REFERENCE MASS: 238 REVIEWED BY:
DATE:

MEASURED MASSES:

234	235	236	238

RATIOS:

.0001714	.0304685	.0001792	1
.0002171	.0311156	.0001936	1
.0001252	.0312051	.0001929	1
.0002064	.0314997	.0002045	1
.0002314	.0315185	.0001904	1
.0002047	.0313511	.0002276	1
.0001689	.0315855	.0002101	1
.0002152	.0304205	.0002072	1
.000201	.0311937	.0002213	1

NO SCANS REJECTED AT THE 95% LEVEL

MEAN RATIO FOR EACH ISOTOPE IS:

.0001935	.0311509	.000203	1

STANDARD DEVIATION OF RATIO FOR EACH ISOTOPE IS:

3.27E-5	.0004317	1.55E-5	0

MASS	ATOM %	MASS %	Mean Ratio	STD
234	.0187572	.0184491	.0001935	.0000327
235	3.0198226	2.9829074	.0311509	.0004317
236	.0196759	.0195180	.0002030	.0000155
238	96.9417443	96.9791255	1.0000000	0.0000000

MASS SPECTROMETER LABORATORIES

ANALYTICAL CHEMISTRY
325 BUILDING

CEC21-703A(M&TE 467806)
PROCEDURE NO.: PNL-MA-599, PNNL-98523-264

CUSTOMER: Sandy Fiskum RUN # 7 MASS DISC. FACTOR: .002
SAMPLE I.D.: U-030 9 May 2011 16:09:54
LABORATORY LOG-IN NUMBER: 0301119 ANALYST: Stan Bos
ALO LOG-IN NUMBER: LRB: BNW 56999 PAGE 77

REFERENCE MASS: 238 REVIEWED BY:
DATE: 5/16/11

MEASURED MASSES:

234	235	236	238

RATIOS:

.0002232	.0313623	.0002166	1
.0002029	.0312	.0002143	1
.0001916	.0316136	.0002108	1
.0002075	.0309867	.0002099	1
.0001829	.0309076	.0002145	1
.0002027	.0313242	.0002487	1
.000181	.0312149	.0002355	1
.0001806	.0314781	.0002047	1
.0001491	.031002	.0001881	1

NO SCANS REJECTED AT THE 95% LEVEL

MEAN RATIO FOR EACH ISOTOPE IS:

.0001913	.0312321	.0002159	1

STANDARD DEVIATION OF RATIO FOR EACH ISOTOPE IS:

2.13E-5	.0002375	1.74E-5	0

MASS	ATOM %	MASS %	Mean Ratio	STD
234	.0185408	.0182362	.0001913	.0000213
235	3.0274292	2.9904241	.0312321	.0002375
236	.0209280	.0207602	.0002159	.0000174
238	96.9331020	96.9705795	1.0000000	0.0000000

MASS SPECTROMETER LABORATORIES

ANALYTICAL CHEMISTRY
325 BUILDING

CEC21-703A(M&TE 467806)
PROCEDURE NO.: PNL-MA-599, Appl - 98523 - 264

CUSTOMER: Sandy Fiskum 53 5-18-11 RUN # 3 MASS DISC. FACTOR: 0
SAMPLE I.D.: 11-0419-129-S-U Pu 18 May 2011 10:34:02
LABORATORY LOG-IN NUMBER: 1211010 ANALYST: Stan Bos
ALO LOG-IN NUMBER: 11-0419~~DS~~ SP PKB 6/1/11 LRB: BNW 56999 PAGE 77

REFERENCE MASS: 239 REVIEWED BY: PKB
DATE: 5/18/11

MEASURED MASSES:

238	239	240	241	242
RATIOS:				
8.5E-5	1	.1504369	.0066372	.0029674
.0016365	1	.1603001	.0087258	.0014103
.0002682	1	.1564845	.0089409	.0031848
.0012072	1	.1422759	.0096338	.0036026
.0016136	1	.153111	.0066869	.0021807
.0011531	1	.1433747	.0090029	.0016256
.0018476	1	.1484095	.0071204	.0028746
.0008598	1	.1470927	.006799	.0028326
.0012315	1	.1485962	.0069318	.0034269

NO SCANS REJECTED AT THE 95% LEVEL

MEAN RATIO FOR EACH ISOTOPE IS:

.0011003	1	.1500091	.007831	.0026784

STANDARD DEVIATION OF RATIO FOR EACH ISOTOPE IS:

.0006044	0	.0058579	.0012129	.000773

MASS	ATOM %	MASS %	Mean Ratio	STD
238	.0947206	.0942657	.0011003	.0006044
239	86.0867677	86.0332792	1.0000000	0.0000000
240	12.9137967	12.9597719	.1500091	.0058579
241	.6741419	.6793609	.0078310	.0012129
242	.2305731	.2333223	.0026784	.0007730

MASS SPECTROMETER LABORATORIES

ANALYTICAL CHEMISTRY
325 BUILDING

CEC21-703A(M&TE 467806) G0536-264
PROCEDURE NO.: PNL-MA-599, PNNL-~~4836~~ Rev 7/14/11

CUSTOMER: Sandy Fiskum RUN # 5 MASS DISC. FACTOR: 0
SAMPLE I.D.: 11-0419-129-D-Pu 18 May 2011 11:17:33
LABORATORY LOG-IN NUMBER: 1211011 ANALYST: Stan Bos
ALO LOG-IN NUMBER: 11-0419~~PD~~ LRB: BNW 56999 PAGE 77

REFERENCE MASS: 239 REVIEWED BY:
DATE: 5/18/11

MEASURED MASSES:

238	239	240	241	242

RATIOS:

.001546	1	.1490039	.0052284	.003427
.0012688	1	.1509154	.0063275	.0033134
.0014212	1	.1438	.0056386	.0025932
.00191	1	.1457857	.0058049	.0025557
.0019548	1	.1553129	.0052747	.00263
.0020885	1	.1527571	.0067048	.0030795
.0014236	1	.145531	.0061516	.0028624
.0019405	1	.1506099	.0059971	.0023982
.0026577	1	.1544235	.0066698	.0032078

NO SCANS REJECTED AT THE 95% LEVEL

MEAN RATIO FOR EACH ISOTOPE IS:

.0018012	1	.1497933	.0059775	.0028964

STANDARD DEVIATION OF RATIO FOR EACH ISOTOPE IS:

.0004339	0	.0040833	.0005433	.0003732

MASS	ATOM %	MASS %	Mean Ratio	STD
238	.1552157	.1544723	.0018012	.0004339
239	86.1721060	86.1197433	1.0000000	0.0000000
240	12.9080005	12.9541324	.1497933	.0040833
241	.5150926	.5190874	.0059775	.0005433
242	.2495853	.2525646	.0028964	.0003732

MASS SPECTROMETER LABORATORIES

ANALYTICAL CHEMISTRY
325 BUILDING

CEC21-703A(M&TE 467806)
PROCEDURE NO.: PNL-MA-599, PNNL-98523-264

CUSTOMER: Sandy Fiskum RUN # 5 MASS DISC. FACTOR: 0
SAMPLE I.D.: 11-0419-129-PB-Pu 18 May 2011 11:39:26
LABORATORY LOG-IN NUMBER: 1211012 ANALYST: Stan Bos S J Bos
ALO LOG-IN NUMBER: 11-0419PB LRB: BNW 56999 PAGE 77

REFERENCE MASS: 239 REVIEWED BY: P.K.D. 6/1/11
DATE: 6/1/11

MEASURED MASSES:

238	239	240	241	242

RATIOS:

unable to run sample for Isotopic abundance

238-Pu signal too low to analyze

S J Bos 6/1/11

MASS SPECTROMETER LABORATORIES

ANALYTICAL CHEMISTRY
325 BUILDING

0420 dsc 7-1-11

CEC21-703A(M&TE 467806)
PROCEDURE NO.: PNL-MA-599, PNL-9523-264

CUSTOMER: Sandy Fiskum RUN # 3 MASS DISC. FACTOR: 0
SAMPLE I.D.: 11-~~420~~-129-S-P 25 May 2011 13:30:38
LABORATORY LOG-IN NUMBER: 1211013 ANALYST: Stan Bos
ALO LOG-IN NUMBER: 11-0420SP LRB: BNW 56999 PAGE 78

REFERENCE MASS: 239 REVIEWED BY: PKB
DATE: 6/1/11

MEASURED MASSES:

238	239	240	241	242

RATIOS:

.0013726	1	.155723	.0063764	.0032113
.0017442	1	.1544908	.0057209	.0032573
.0021082	1	.1537717	.006367	.0033056
.0032776	1	.1561428	.0062485	.0026224
.004743	1	.1502786	.0060286	.0021173
.0048676	1	.1533688	.006787	.0033412
.0046629	1	.1542418	.0060014	.0026476
.0054208	1	.1519449	.0069198	.0031544
.0064582	1	.1546383	.0053422	.0029456

NO SCANS REJECTED AT THE 95% LEVEL

MEAN RATIO FOR EACH ISOTOPE IS:

.0038506	1	.1538445	.0061991	.0029559

STANDARD DEVIATION OF RATIO FOR EACH ISOTOPE IS:

.0017923	0	.0018225	.0004948	.0004147

MASS	ATOM %	MASS %	Mean Ratio	STD
238	.3299960	.3284136	.0038506	.0017923
239	85.7008174	85.6482205	1.0000000	0.0000000
240	13.1846007	13.2316408	.1538445	.0018225
241	.5312662	.5353832	.0061991	.0004948
242	.2533196	.2563419	.0029559	.0004147

MASS SPECTROMETER LABORATORIES

ANALYTICAL CHEMISTRY
325 BUILDING

0420 DSC 7-1-11

CEC21-703A(M&TE 467806) PNNL - 95523 - 264
PROCEDURE NO.: PNL-MA-599, ~~PNNL-264~~ SB 7/14/11

CUSTOMER: Sandy Fiskum RUN # 4 MASS DISC. FACTOR: 0
SAMPLE I.D.: 11-420-129-D-Pu 25 May 2011 14:03:40
LABORATORY LOG-IN NUMBER: 1211014 ANALYST: Stan Bos
ALO LOG-IN NUMBER: 11-0420DP LRB: BNW 56999 PAGE 78

REFERENCE MASS: 239 REVIEWED BY: P.K.B.
DATE: 6/1/11

MEASURED MASSES:

238	239	240	241	242

RATIOS:

.0012596	1	.1480662	.0057363	.0027234
.0013423	1	.1484897	.0063903	.0024842
.0019579	1	.148823	.0064799	.0024569
.0019026	1	.1506335	.0055799	.0027381
.0030506	1	.1530704	.0058119	.0027452
.0032202	1	.1494253	.0059184	.0030105
.0035905	1	.1537124	.0060699	.00222
.0039135	1	.1496795	.0057392	.0030698
.0040012	1	.1547968	.005328	.002356

NO SCANS REJECTED AT THE 95% LEVEL

MEAN RATIO FOR EACH ISOTOPE IS:

.0026932	1	.1507441	.0058949	.0026449

STANDARD DEVIATION OF RATIO FOR EACH ISOTOPE IS:

.0010872	0	.0024869	.0003701	.0002882

MASS	ATOM %	MASS %	Mean Ratio	STD
238	.2317737	.2306646	.0026932	.0010872
239	86.0602213	86.0082608	1.0000000	0.0000000
240	12.9730700	13.0194852	.1507441	.0024869
241	.5073140	.5112505	.0058949	.0003701
242	.2276210	.2303390	.0026449	.0002882

MASS SPECTROMETER LABORATORIES

ANALYTICAL CHEMISTRY
325 BUILDING

CEC21-703A(M&TE 467806)
PROCEDURE NO.: PNL-MA-599, PNL-9852-264

CUSTOMER: Sandy Fiskum RUN # 9 MASS DISC. FACTOR: 0
SAMPLE I.D.: 11-0421-129-S-Pu 18 May 2011 13:43:00
LABORATORY LOG-IN NUMBER: 1211015 ANALYST: Stan Bos
ALO LOG-IN NUMBER: 11-0421SP LRB: BNW 56999 PAGE 77

REFERENCE MASS: 239 REVIEWED BY: P.K B
DATE: 5/18/11

MEASURED MASSES:

238	239	240	241	242

RATIOS:

.0008366	1	.1470467	.0060678	.0026357
.0005724	1	.1426943	.0050492	.0029295
.0005687	1	.1474566	.0049801	.0030263
.0008529	1	.1523031	.0052795	.0027947
.0013468	1	.1510944	.0060919	.0020114
.00113	1	.1516444	.0064354	.0027199
.0006977	1	.1474053	.0065609	.003042
.0013746	1	.1503327	.0062074	.0020259
.0008146	1	.1422176	.0039301	.0024473

NO SCANS REJECTED AT THE 95% LEVEL

MEAN RATIO FOR EACH ISOTOPE IS:

.0009105	1	.1480217	.0056225	.0026258

STANDARD DEVIATION OF RATIO FOR EACH ISOTOPE IS:

.0003058	0	.0037073	.0008679	.0003925

MASS	ATOM %	MASS %	Mean Ratio	STD
238	.0786802	.0783039	.0009105	.0003058
239	86.4169435	86.3650326	1.0000000	0.0000000
240	12.7915816	12.8373868	.1480217	.0037073
241	.4858774	.4896490	.0056225	.0008679
242	.2269174	.2296277	.0026258	.0003925

MASS SPECTROMETER LABORATORIES

ANALYTICAL CHEMISTRY
325 BUILDING

C421 dsc 7-1-11

CEC21-703A(M&TE 467806)
PROCEDURE NO.: PNL-MA-599, PNNL-9852-3-2011

CUSTOMER: Sandy Fiskum RUN # 7 MASS DISC. FACTOR: 0
SAMPLE I.D.: 11-421-129-D-P 25 May 2011 15:26:09
LABORATORY LOG-IN NUMBER: 1211016 ANALYST: Stan Bos
ALO LOG-IN NUMBER: 11-0421DP LRB: BNW 56999 PAGE 78

REFERENCE MASS: 239 REVIEWED BY:
DATE: 6/1/11

MEASURED MASSES:

238	239	240	241	242
RATIOS:				
.0010671	1	.1503231	.0061799	.0032585
.0014965	1	.1547836	.0057446	.0028557
.0021285	1	.1508051	.0060632	.0027647
.0011734	1	.1463296	.0061614	.002466
.0017046	1	.1455919	.0051903	.0024916
.001391	1	.15	.0047371	.0031093
.0020992	1	.1547301	.0058725	.0028612
.0017532	1	.1481868	.005472	.0021633
.0020429	1	.1541788	.0051821	.002422

NO SCANS REJECTED AT THE 95% LEVEL

MEAN RATIO FOR EACH ISOTOPE IS:

.0016507	1	.1505477	.0056225	.0027103

STANDARD DEVIATION OF RATIO FOR EACH ISOTOPE IS:

.0003963	0	.0034814	.0005074	.0003529

MASS	ATOM %	MASS %	Mean Ratio	STD
238	.1422372	.1415562	.0016507	.0003963
239	86.1674394	86.1151951	1.0000000	0.0000000
240	12.9723060	13.0186852	.1505477	.0034814
241	.4844804	.4882384	.0056225	.0005074
242	.2335370	.2363250	.0027103	.0003529

MASS SPECTROMETER LABORATORIES

ANALYTICAL CHEMISTRY
325 BUILDING

CEC21-703A(M&TE 467806)
PROCEDURE NO.: PNL-MA-599, DOE/RL-98533-264

CUSTOMER: Sandy Fiskum RUN # 11 MASS DISC. FACTOR: 0
SAMPLE I.D.: 11-0422-129-S-Pu 18 May 2011 14:53:07
LABORATORY LOG-IN NUMBER: 1211017 ANALYST: Stan Bos
ALO LOG-IN NUMBER: 11-0422SP LRB: BNW 56999 PAGE 77

REFERENCE MASS: 239 REVIEWED BY: PKB
DATE: 6/1/11

MEASURED MASSES:

238	239	240	241	242
RATIOS:				
.0007034	1	.1489685	.0055007	.0032113
.0008044	1	.1451277	.0056475	.0024258
.0011673	1	.1535958	.0066015	.002797
.0010867	1	.1491745	.0069001	.0030832
.001129	1	.1495902	.0058781	.0031546
.0010382	1	.1500435	.0061142	.0022644
.0012014	1	.1489405	.0046306	.002106
.0015597	1	.1572736	.0088325	.0021779
.0017046	1	.1452526	.0057476	.0027336

NO SCANS REJECTED AT THE 95% LEVEL

MEAN RATIO FOR EACH ISOTOPE IS:

.001155	1	.1497741	.0062058	.0026615

STANDARD DEVIATION OF RATIO FOR EACH ISOTOPE IS:

.000319	0	.0037864	.0011808	.0004334

MASS	ATOM %	MASS %	Mean Ratio	STD
238	.0995840	.0991069	.0011550	.0003190
239	86.2220265	86.1694852	1.0000000	0.0000000
240	12.9138259	12.9599564	.1497741	.0037864
241	.5350807	.5392295	.0062058	.0011808
242	.2294829	.2322219	.0026615	.0004334

SB 6/13/11

entered on wrong sheet
SB 6/13/11

MASS SPECTROMETER LABORATORIES

ANALYTICAL CHEMISTRY
325 BUILDING

CEC21-703A(M&TE 467806)
PROCEDURE NO.: PNL-MA-599, PNL-9853-264

CUSTOMER: Sandy Fiskum RUN # 12 MASS DISC. FACTOR: 0
SAMPLE I.D.: 11-0422-129-D-Pu 18 May 2011 15:20:57
LABORATORY LOG-IN NUMBER: 1211018 ANALYST: Stan Bos
ALO LOG-IN NUMBER: 11-0422DP LRB: BNW 56999 PAGE 77

REFERENCE MASS: 239 REVIEWED BY: PKB
DATE: 6/1/11

MEASURED MASSES:

238	239	240	241	242

RATIOS:

.0011951	1	.1523063	.006431	.0028562
.0008485	1	.1525807	.0060339	.0021753
.0020975	1	.1475099	.0031842	.001405
.0024531	1	.161824	.0042327	.0026904
.0042151	1	.1659229	.0048338	.0042287
.0052727	1	.1422038	.0102411	.00172
.0030856	1	.1505182	.0075363	.0019662
.0102164	1	.1498233	.005477	.0008525
.0251029	1	.1991176	.0077574	-.0109559

Sample exhausted

eliminate cycles 8+9

CYCLE # 9 REJECTED AT THE 95% LEVEL
DUE TO THE 242/ 239 RATIO

MEAN RATIO FOR EACH ISOTOPE IS:

.003673	1	.1528361	.0059963	.0022368

STANDARD DEVIATION OF RATIO FOR EACH ISOTOPE IS:

.0030261	0	.0076369	.0021784	.0010358

6/13/11

MASS	ATOM %	MASS %	Mean Ratio	STD
238	.3153493 .2351	.3138406	.0036730	.0030261
239	85.8559110 85.873	85.8041690	1.0000000	0.0000000
240	13.1218855 13.161	13.1688476	.1528361	.0076369
241	.5148135 .5213	.5188087	.0059963	.0021784
242	.1920407 .2071	.1943340	.0022368	.0010358

Re normalized To Remove cycles 8 & 9

SF 6/13/11

U. S. Department of Commerce
Malcolm Baldrige
Secretary

National Bureau of Standards
Ernest Ambler, Director

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W.Y. MATSUMOTO

National Bureau of Standards

Certificate of Analysis

Standard Reference Material 946

Plutonium Isotopic Standard

This Standard Reference Material (SRM) is certified as an isotopic standard for use in isotopic measurements of plutonium. SRM 946 consists of approximately 250 mg of plutonium in the form of plutonium sulfate tetrahydrate packaged in a glass microbottle.

	^{238}Pu	^{239}Pu	^{240}Pu	^{241}Pu	^{242}Pu
Atom Percent*	0.232 ±0.007	84.464 ±0.015	12.253 ±0.015	2.477 ±0.005	0.574 ±0.003

**As of January 1, 1982, refer to Table 1 for quarterly decay-adjusted values.*

The plutonium isotopic distribution was determined by thermal ionization mass spectrometry at the National Bureau of Standards (NBS) on samples from which the americium and uranium were removed. Because high-purity plutonium isotopes have not been used to prepare known synthetic mixtures, the accuracy is dependent on uranium and plutonium exhibiting similar behavior. The observed mass spectrometer data were corrected for mass discrimination effects using data from the analysis of uranium isotopic SRM's that had been analyzed under similar conditions.

SRM 946 contains uranium and americium isotopes, including growing-in daughters of plutonium that are isobaric with the plutonium isotopes. In addition, there may be radiation damage to the glass bottle and the teflon cap liner resulting in small glass slivers. Therefore, in its use, a chemical separation that provides a purified plutonium fraction is essential to the attainment of high accuracy.

Measurements leading to the certification of this SRM were made in the Inorganic Analytical Research Division by E.L. Garner and L.A. Machlan.

The technical and support aspects involved in the revision of this Certificate were coordinated through the Office of Standard Reference Materials by T.E. Gills.

August 19, 1982
Washington, D.C. 20234
(Revision of Certificate
dated 12-3-71)

George A. Uriano, Chief
Office of Standard Reference Materials

(over)

The decay-adjusted values for the plutonium isotopic composition, in atom percent, are tabulated below in Table 1. The half-life values, in years, used for the decay-adjustment are: ^{238}Pu, 87.74; ^{239}Pu, 24,119; ^{240}Pu, 6,560; ^{241}Pu, 14.34; and ^{242}Pu, 387,000.

Table 1
Decay-Adjusted Plutonium Isotopic Composition
Atom Percent

Date	^{238}Pu	^{239}Pu	^{240}Pu	^{241}Pu	^{242}Pu
January 1, 1982	0.232	84.464	12.253	2.477	0.574
April 1, 1982	.231	84.490	12.257	2.448	.574
July 1, 1982	.231	84.515	12.260	2.419	.575
October 1, 1982	.230	84.540	12.264	2.391	.575
January 1, 1983	.230	84.565	12.267	2.363	.575
April 1, 1983	.230	84.590	12.270	2.335	.575
July 1, 1983	.229 .225	84.614 .888	12.274 .311	2.308 .000	.575 .577
October 1, 1983	.229	84.638	12.277	2.281	.575
January 1, 1984	.229	84.662	12.280	2.253	.576
April 1, 1984	.228	84.685	12.283	2.227	.576
July 1, 1984	.228	84.709	12.286	2.201	.576
October 1, 1984	.227	84.732	12.290	2.175	.576
January 1, 1985	.227	84.755	12.293	2.149	.576
April 1, 1985	.227	84.777	12.296	2.125	.576
July 1, 1985	.226	84.799	12.299	2.100	.577
October 1, 1985	.226	84.821	12.302	2.075	.577
January 1, 1986	.225	84.843	12.305	2.050	.577
April 1, 1986	.225 .00265	84.864 .14502	12.307	2.026 .02387	.577 .00680
July 1, 1986	.225 .00265	84.885	12.310	2.003	.577
October 1, 1986	.224	84.906	12.313	1.979	.577
95% Confidence Limit:	±0.007	±0.015	±0.015	±0.005	±0.003
		85.08	12.326		

Pu SRM 946			Renorm	Decay Corrected
	10/1/1997	5/17/2011	**5/17/2011**	at%
Isotope	at%	at%	at%	**Pu Only**
238	0.2078	0.186588	**0.186625**	**0.187685**
239	85.63	85.630000	**85.647226**	**86.133715**
240	12.407	12.407000	**12.409496**	**12.479984**
241	1.1741	0.609406	**0.609529**	**0.612991**
Am-241	0	0.564694	**0.564808**	
242	0.5822	0.582200	**0.582317**	**0.585625**
	100.0011	99.979888	**100.000000**	**100.000000**
			99.435192	**Pu only**

Reviewed by: L R Greenwood 6-7-11

MASS SPECTROMETER LABORATORIES

ANALYTICAL CHEMISTRY
325 BUILDING

CEC21-703A(M&TE 467806)
PROCEDURE NO.: PNL-MA-599, PNL-9852-264

CUSTOMER: Sandy Fiskum RUN # 1 MASS DISC. FACTOR: 0
SAMPLE I.D.: Pu-946 17 May 2011 13:21:28
LABORATORY LOG-IN NUMBER: 9461106 ANALYST: Stan Bos
ALO LOG-IN NUMBER: LRB: BNW 56999 PAGE 77

REFERENCE MASS: 239 REVIEWED BY:
DATE: 5/17/11

MEASURED MASSES:

238	239	240	241	242
RATIOS:				
.0023652	1	.1440462	.0076631	.0064258
.0021445	1	.1432006	.0072838	.0067369
.0022082	1	.1419692	.0073621	.0067458
.0020985	1	.1435652	.0071028	.0067622
.0022031	1	.1439553	.0071098	.0063916
.0021513	1	.1435857	.00684	.0066657
.0021628	1	.1452246	.0071147	.006672
.0021241	1	.1435586	.0069958	.006709
.0022257	1	.1440954	.0071975	.0068974

NO SCANS REJECTED AT THE 95% LEVEL

MEAN RATIO FOR EACH ISOTOPE IS:

.002187	1	.143689	.0071855	.0066674

STANDARD DEVIATION OF RATIO FOR EACH ISOTOPE IS:

7.86E-5	0	.0008632	.0002351	.0001617

MASS	ATOM %	MASS %	Mean Ratio	STD
238	.1885823	.1876741	.0021870	.0000786
239	86.2270484	86.1723697	1.0000000	0.0000000
240	12.3898763	12.4338272	.1436890	.0008632
241	.6195844	.6243730	.0071855	.0002351
242	.5749086	.5817559	.0066674	.0001617

MASS SPECTROMETER LABORATORIES

ANALYTICAL CHEMISTRY
325 BUILDING

CEC21-703A(M&TE 467806)
PROCEDURE NO.: PNL-MA-599, Rev. 98523-264

CUSTOMER: Sandy Fiskum RUN # 2 MASS DISC. FACTOR: 0
SAMPLE I.D.: Pu-946 17 May 2011 13:37:21
LABORATORY LOG-IN NUMBER: 9461106.2 ANALYST: Stan Bos
ALO LOG-IN NUMBER: LRB: BNW 56999 PAGE 77

REFERENCE MASS: 239 REVIEWED BY: PKB
DATE: 5/17/11

MEASURED MASSES:

238	239	240	241	242
RATIOS:				
.0020323	1	.1434716	.0069617	.0070438
.002261	1	.1439947	.0070213	.0069259
.0021303	1	.1440443	.0071292	.0066145
.0021541	1	.1433893	.0070437	.0068346
.0020656	1	.1443016	.0073361	.006848
.0021787	1	.1445669	.0070319	.0066436
.0021344	1	.144987	.0070805	.0065541
.0021153	1	.1442272	.0073321	.0066799
.0020932	1	.1431098	.006992	.0067261

NO SCANS REJECTED AT THE 95% LEVEL

MEAN RATIO FOR EACH ISOTOPE IS:

.0021294	1	.1440103	.0071032	.0067634

STANDARD DEVIATION OF RATIO FOR EACH ISOTOPE IS:

6.65E-5	0	.0006004	.0001395	.0001604

MASS	ATOM %	MASS %	Mean Ratio	STD
238	.1835709	.1826866	.0021294	.0000665
239	86.2064313	86.1516228	1.0000000	0.0000000
240	12.4146099	12.4586279	.1440103	.0006004
241	.6123394	.6170710	.0071032	.0001395
242	.5830485	.5899918	.0067634	.0001604

MASS SPECTROMETER LABORATORIES

ANALYTICAL CHEMISTRY
325 BUILDING

CEC21-703A(M&TE 467806)
PROCEDURE NO.: PNL-MA-599, PNL-10533-264

CUSTOMER: Sandy Fiskum RUN # 3 MASS DISC. FACTOR: 0
SAMPLE I.D.: Pu-946 17 May 2011 13:54:47
LABORATORY LOG-IN NUMBER: 9461106.3 ANALYST: Stan Bos
ALO LOG-IN NUMBER: LRB: BNW 56999 PAGE 77

REFERENCE MASS: 239 REVIEWED BY:
DATE: 5/17/11

MEASURED MASSES:

238	239	240	241	242

RATIOS:

.0022594	1	.1447871	.0069771	.0064662
.002029	1	.1430881	.0069715	.0068219
.0021938	1	.1471769	.0074265	.0067507
.0021561	1	.1447172	.0072337	.0069118
.0021637	1	.1445398	.0078381	.0067061
.0020803	1	.1440903	.0073266	.0067266
.0020623	1	.1451425	.0074061	.0071263
.0019834	1	.1443631	.0073187	.0067984
.0019503	1	.1448737	.0074369	.006714

NO SCANS REJECTED AT THE 95% LEVEL

MEAN RATIO FOR EACH ISOTOPE IS:

.0020976	1	.1447532	.0073261	.0067802

STANDARD DEVIATION OF RATIO FOR EACH ISOTOPE IS:

.0001026	0	.001087	.0002615	.0001773

MASS	ATOM %	MASS %	Mean Ratio	STD
238	.1806782	.1798071	.0020976	.0001026
239	86.1358230	86.0807101	1.0000000	0.0000000
240	12.4684360	12.5125940	.1447532	.0010870
241	.6310425	.6359160	.0073261	.0002615
242	.5840203	.5909728	.0067802	.0001773

MASS SPECTROMETER LABORATORIES

ANALYTICAL CHEMISTRY
325 BUILDING

CEC21-703A(M&TE 467806)
PROCEDURE NO.: PNL-MA-599, DNW-98523-264

CUSTOMER: Sandy Fiskum RUN # 1 MASS DISC. FACTOR: 0
SAMPLE I.D.: Pu-946 18 May 2011 09:26:44
LABORATORY LOG-IN NUMBER: 9461107.1 ANALYST: Stan Bos
ALO LOG-IN NUMBER: LRB: BNW 56999 PAGE 77

REFERENCE MASS: 239 REVIEWED BY: PKB
DATE: 5/18/11

MEASURED MASSES:

238	239	240	241	242

RATIOS:

.0019998	1	.1477493	.0070511	.0066316
.0022519	1	.1466591	.0071096	.0065578
.002342	1	.1431908	.0072711	.0069405
.002045	1	.142464	.0070849	.0067249
.0021811	1	.1446248	.0066984	.0063056
.0023041	1	.1457228	.0072151	.0067846
.0023481	1	.1450692	.0068179	.0064383
.0020481	1	.1413035	.0070763	.0065016
.0021242	1	.1429934	.0073037	.006693

NO SCANS REJECTED AT THE 95% LEVEL

MEAN RATIO FOR EACH ISOTOPE IS:

.0021827	1	.1444196	.0070698	.0066198

STANDARD DEVIATION OF RATIO FOR EACH ISOTOPE IS:

.0001352	0	.0021019	.0002	.0001924

MASS	ATOM %	MASS %	Mean Ratio	STD
238	.1881173	.1872112	.0021827	.0001352
239	86.1852074	86.1304698	1.0000000	0.0000000
240	12.4468369	12.4909775	.1444196	.0021019
241	.6093116	.6140202	.0070698	.0002000
242	.5705268	.5773213	.0066198	.0001924

MASS SPECTROMETER LABORATORIES

ANALYTICAL CHEMISTRY
325 BUILDING

CEC21-703A(M&TE 467806)
PROCEDURE NO.: PNL-MA-599, PNL - 98533 - 264

CUSTOMER: Sandy Fiskum RUN # 2 MASS DISC. FACTOR: 0
SAMPLE I.D.: Pu-946 18 May 2011 09:43:03
LABORATORY LOG-IN NUMBER: 9461107.2 ANALYST: Stan Bos
ALO LOG-IN NUMBER: LRB: BNW 56999 PAGE 77

REFERENCE MASS: 239 REVIEWED BY:
DATE: 5/18/11

MEASURED MASSES:

238	239	240	241	242
RATIOS:				
.0024706	1	.1430085	.0073102	.0066909
.0021144	1	.1443037	.0071906	.0064261
.0023262	1	.1433183	.0070787	.006455
.0023312	1	.1432405	.0069752	.00717
.0021932	1	.1434035	.0070166	.0070888
.0021634	1	.1446059	.0066461	.0066066
.0022204	1	.1424739	.0074596	.0067674
.0021861	1	.1457711	.0070999	.0069465
.0024847	1	.1448969	.0068111	.0068629

NO SCANS REJECTED AT THE 95% LEVEL

MEAN RATIO FOR EACH ISOTOPE IS:

.0022767	1	.1438914	.0070653	.0067793

STANDARD DEVIATION OF RATIO FOR EACH ISOTOPE IS:

.0001341	0	.0010609	.0002458	.0002625

MASS	ATOM %	MASS %	Mean Ratio	STD
238	.1962644	.1953191	.0022767	.0001341
239	86.2059496	86.1512330	1.0000000	0.0000000
240	12.4042919	12.4482865	.1438914	.0010609
241	.6090745	.6137815	.0070653	.0002458
242	.5844196	.5913798	.0067793	.0002625

MASS SPECTROMETER LABORATORIES

ANALYTICAL CHEMISTRY
325 BUILDING

CEC21-703A(M&TE 467806)
PROCEDURE NO.: PNL-MA-599, PNNL-98523-264

CUSTOMER: Sandy Fiskum RUN # 13 MASS DISC. FACTOR: 0
SAMPLE I.D.: Pu-946 PIEB 5/25/11 18 May 2011 15:54:47
LABORATORY LOG-IN NUMBER: 94611008.1 ANALYST: Stan Bos
ALO LOG-IN NUMBER: LRB: BNW 56999 PAGE 78

REFERENCE MASS: 239 REVIEWED BY: P.K.B.
DATE: 5/25/11

MEASURED MASSES:

238	239	240	241	242
RATIOS:				
.0022805	1	.1470138	.0071981	.0070842
.0016822	1	.1472824	.0071703	.0068758
.0026149	1	.1391972	.0074456	.0068328
.0025598	1	.1433622	.0084116	.0063849
.0031166	1	.1418578	.0076801	.0068231
.0021294	1	.140706	.0080441	.0070711
.0017241	1	.1468405	.0050084	.0062489
.0023191	1	.1438318	.0068766	.0072836
.0020145	1	.1390538	.0066295	.0067792

NO SCANS REJECTED AT THE 95% LEVEL

MEAN RATIO FOR EACH ISOTOPE IS:

.0022712	1	.1432384	.0071627	.0068204

STANDARD DEVIATION OF RATIO FOR EACH ISOTOPE IS:

.0004542	0	.0032809	.0009806	.0003294

MASS	ATOM %	MASS %	Mean Ratio	STD
238	.1958812	.1949380	.0022712	.0004542
239	86.2446118	86.1899486	1.0000000	0.0000000
240	12.3535396	12.3973654	.1432384	.0032809
241	.6177442	.6225188	.0071627	.0009806
242	.5882232	.5952293	.0068204	.0003294

MASS SPECTROMETER LABORATORIES

ANALYTICAL CHEMISTRY
325 BUILDING

CEC21-703A(M&TE 467806)
PROCEDURE NO.: PNL-MA-599, PNNL-98573-264

CUSTOMER: Sandy Fiskum RUN # 1 MASS DISC. FACTOR: 0
SAMPLE I.D.: Pu-946 PNNL 5/25/11 25 May 2011 11:23:54
LABORATORY LOG-IN NUMBER: 94611Ø9.1 ANALYST: Stan Bos
ALO LOG-IN NUMBER: LRB: BNW 56999 PAGE 78

REFERENCE MASS: 239 REVIEWED BY: JXD
DATE: 5/25/11

MEASURED MASSES:

238	239	240	241	242
RATIOS:				
.0022267	1	.1428996	.0065841	.0066254
.002147	1	.1448841	.0070371	.0067367
.0022451	1	.147404	.0074173	.0067458
.0022628	1	.1450578	.007025	.0065672
.0019778	1	.149	.0073031	.006967
.002208	1	.1447977	.0071538	.0067846
.0021492	1	.1442185	.0071101	.0069136
.0021407	1	.1450688	.0068295	.0063065
.0022267	1	.1460089	.007197	.0068313

NO SCANS REJECTED AT THE 95% LEVEL

MEAN RATIO FOR EACH ISOTOPE IS:

.002176	1	.1454821	.007073	.0067198

STANDARD DEVIATION OF RATIO FOR EACH ISOTOPE IS:

8.7E-5	0	.0017955	.0002492	.0001998

MASS	ATOM %	MASS %	Mean Ratio	STD
238	.1873524	.1864492	.0021760	.0000870
239	86.0992015	86.0441469	1.0000000	0.0000000
240	12.5258964	12.5702630	.1454821	.0017955
241	.6089794	.6136828	.0070730	.0002492
242	.5785703	.5854581	.0067198	.0001998

MASS SPECTROMETER LABORATORIES

ANALYTICAL CHEMISTRY
325 BUILDING

CEC21-703A(M&TE 467806)
PROCEDURE NO.: PNL-MA-599, PNL-9852-264

CUSTOMER: Sandy Fiskum RUN # 2 MASS DISC. FACTOR: 0
SAMPLE I.D.: Pu-946 PMB 5/25/11 25 May 2011 11:40:26
LABORATORY LOG-IN NUMBER: 94611009.2 ANALYST: Stan Bos
ALO LOG-IN NUMBER: LRB: BNW 56999 PAGE 78

REFERENCE MASS: 239 REVIEWED BY: PKB
DATE: 5/25/11

MEASURED MASSES:

238	239	240	241	242
RATIOS:				
.0020825	1	.1470099	.0075728	.0068716
.0022889	1	.1427578	.0071397	.0066393
.0023645	1	.146598	.0068243	.007238
.0023698	1	.1446244	.0070716	.0071301
.0018694	1	.1459414	.007169	.0074977
.002626	1	.1463435	.0068182	.0071429
.0019959	1	.1419388	.0068531	.006236
.0023077	1	.1446753	.0078134	.0073431
.0018988	1	.1443857	.0077529	.0065782

NO SCANS REJECTED AT THE 95% LEVEL

MEAN RATIO FOR EACH ISOTOPE IS:

.0022004	1	.1449194	.0072239	.0069641

STANDARD DEVIATION OF RATIO FOR EACH ISOTOPE IS:

.0002531	0	.001741	.0003944	.0004117

MASS	ATOM %	MASS %	Mean Ratio	STD
238	.1894754	.1885616	.0022004	.0002531
239	86.1098155	86.0546086	1.0000000	0.0000000
240	12.4789846	12.5231639	.1449194	.0017410
241	.6220476	.6268508	.0072239	.0003944
242	.5996770	.6068150	.0069641	.0004117

MASS SPECTROMETER LABORATORIES

ANALYTICAL CHEMISTRY
325 BUILDING

CEC21-703A(M&TE 467806)
PROCEDURE NO.: PNL-MA-599, PNNL - 98523-264

CUSTOMER: Sandy Fiskum RUN # 8 MASS DISC. FACTOR: 0
SAMPLE I.D.: Pu-946 25 May 2011 15:55:53
LABORATORY LOG-IN NUMBER: 94611010.1 ANALYST: Stan Bos
ALO LOG-IN NUMBER: LRB: BNW 56999 PAGE 78

REFERENCE MASS: 239 REVIEWED BY: PKB
DATE: 5/25/11

MEASURED MASSES:

238	239	240	241	242

RATIOS:

.0022503	1	.1452927	.0078199	.0067963
.0021502	1	.1414528	.0073162	.0065748
.0019062	1	.1439105	.0069625	.0068441
.0021902	1	.1431991	.0070752	.0066905
.0020751	1	.1456688	.0073042	.0067102
.0023085	1	.1448252	.0072817	.0069726
.0022016	1	.1444237	.0072637	.0070748
.002156	1	.1446784	.006961	.0067865
.0019946	1	.1458613	.007255	.0069765

NO SCANS REJECTED AT THE 95% LEVEL

MEAN RATIO FOR EACH ISOTOPE IS:

.002137	1	.144368	.0072488	.0068251

STANDARD DEVIATION OF RATIO FOR EACH ISOTOPE IS:

.0001261	0	.0013766	.0002574	.0001596

MASS	ATOM %	MASS %	Mean Ratio	STD
238	.1841289	.1832415	.0021370	.0001261
239	86.1638925	86.1088815	1.0000000	0.0000000
240	12.4393117	12.4833840	.1443680	.0013766
241	.6245859	.6294105	.0072488	.0002574
242	.5880810	.5950826	.0068251	.0001596

K-Basin Occurrence Report

Title: TIMS RPD and SRM Issues for KW210 Core Samples	**Date:** 5/9/11
Unique Identifier: 52578-05-09-11	
Primary Person Identifying Issue: Stan Bos	
Distribution: Email and posting on K-Basin Share-Point	Clark Carlson, Matt Fountain, Deborah Coffey, Rick Shimskey, Stan Bos, Sandy Fiskum, Andy Schmidt, Jen Braley
Date Submitted to K-Basin Records:	20 July 2011

Occurrence Description:

Occurrence #1: SRM failures

The isotopic Uranium (U) Standard Reference Material (SRM) acceptance criterion of ±2% of the certified value is a requirement specified in the QAPjP/SAP, KBC-52578, Rev. 2, Table 3-7. Some of the five runs with NIST SRM U-030 exceeded the ±2% criterion for isotopes ^{234}U and ^{236}U. See Table 1, highlighted values indicate results exceeding the acceptance limits. Table 1 includes the preceding results from processing the KW230 samples for comparison (processed February/March).

Table 1. U030 SRM ^{234}U and ^{236}U Results, Five Process Runs

		U-234		U-236	
		Atom%	1-s	Atom%	1-s
	Certificate Value	0.0190	± 0.0001	0.0204	± 0.0001
	+2%	0.0194		0.0208	
	-2%	0.0186		0.0200	
Run #	Run Date	Atom%	1-s	Atom%	1-s
1	02/09/11	0.0191	± 0.003	0.0206	± 0.001
2	02/09/11	0.0187	± 0.001	0.0204	± 0.001
3	02/10/11	0.0182	± 0.001	0.0208	± 0.002
4	02/10/11	0.0200	± 0.002	0.0206	± 0.002
5	02/10/11	0.0197	± 0.003	0.0215	± 0.002
6	02/10/11	0.0208	± 0.002	0.0216	± 0.003
7	02/14/11	0.0185	± 0.002	0.0209	± 0.003
8	02/16/11	0.0203	± 0.002	0.0195	± 0.002
9	02/16/11	0.0174	± 0.001	0.0198	± 0.002
10	03/01/11	0.0190	± 0.002	0.0203	± 0.004
11	03/02/11	0.0188	± 0.002	0.0216	± 0.002
12	05/03/11	0.0187	± 0.002	0.0216	± 0.003
13	05/06/11	0.0195	± 0.002	0.0193	± 0.002
14	05/06/11	0.0201	± 0.002	0.0178	± 0.002
15	05/09/11	0.0188	± 0.003	0.0197	± 0.002
16	05/09/11	0.0185	± 0.002	0.0209	± 0.002
	May 2011 average and standard deviation	0.0191	± 0.0007	0.0199	± 0.0015

Grand average and standard deviation	0.0191	± 0.0009	0.0204	± 0.0010

Occurrence #2: RPD Failures

The ^{238}Pu isotope RPD exceeded ±20% for all duplicate sample pairs.
The ^{241}Pu RPD exceeded ±20% for KW210-A1 core sample duplicate pair (sample IDs TI001-A1-B and TI001-A1-C); 27% RPD was obtained.

Impact:
Impact of Occurrence #1:

No impact. The specified ±2% acceptance criterion was intended to apply to ^{235}U and ^{238}U (see attached correspondence). The QAPjP/SAP KBC-33786, Rev. 2, Table 3-7 did not specifiy which isotopes were were subject to this criterion. Therefore the sample analyisis request specified the ±2% criterion applied to isotopes at concentrations >0.1 atom percent. This analysis request had been reviewed by the CH2M Hill Plataeu Remeidation Co. (CHPRC) Safeguards representative and signed by the Buyer's Technical Representative (BTR); the BTR signature indicated concurrence that the DQOs were properly identified and flowed down to the analytical laboratory/staff.

The ^{234}U and ^{236}U isotopic ratios were significantly less than 0.1 atom %. The 1-σ uncertainties of the ^{234}U and ^{236}U values were large (much greater than 2%, relative) and generally bracketed the ±2% criterion range as shown in Figure 1. The averaged results for isotopes ^{234}U and ^{236}U for the 11 standard runs (processed in February/March 2011) and the 5 additional standard runs (processed in May 2011 to support KW210 samples) met the acceptance limit.

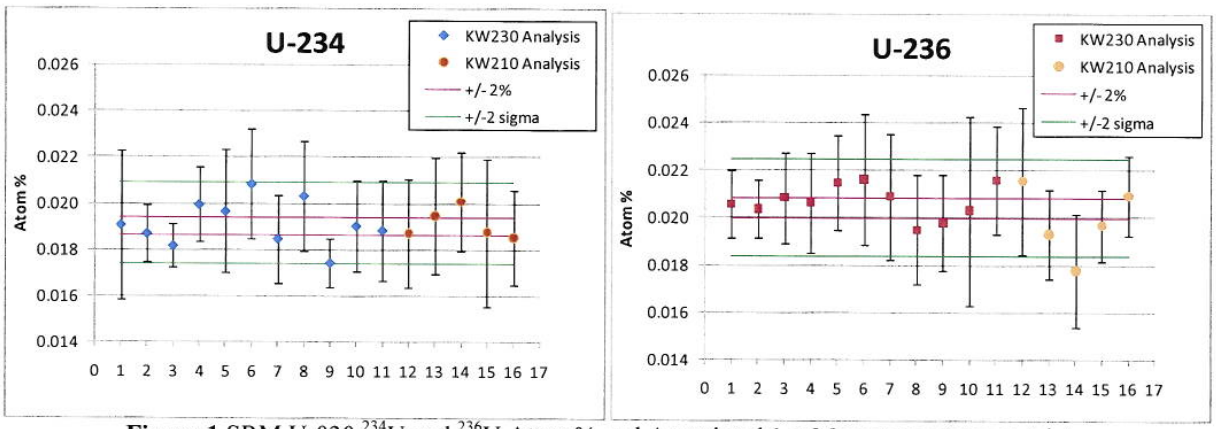


Figure 1 SRM U-030 ^{234}U and ^{236}U Atom % and Associated 1-σ Measurement Uncertainty (Also shown are the ±2% criterion and the ±2 times the standard deviation.)

Impact of Occurrence #2

Plutonium isotopic precision requirements were provided to the analyst as follows:

> The ^{239}Pu, ^{240}Pu, ^{241}Pu, and ^{242}Pu atom percents for a given core sample and duplicate shall agree within ±20% where the isotopic abundance is greater than 1%.

The analysis request specifically excluded precision criteria for the ^{238}Pu isotope because potential isobaric interference from ^{238}U was recognized as a likely possibility. The ^{238}Pu concentration will be determined strictly from the alpha energy analysis results.

There is no project impact for the ^{241}Pu high RPD result because the sample pair results were <1 atom % (specifically 0.674 and 0.515 atom %). The QAPjP/SAP specifies "For TIMS analyses, the ±20% precision applies to the isotopic abundance of major isotopes in the Main and Client Duplicate samples for each core (major isotope = isotope abundance greater than 1% of the total sample)." That said, it is noted that the value of 0.674 atom % ^{241}Pu determined from sample TI001-A1-B was greater than two standard deviations of all ^{241}Pu sample results (0.532 atom % ± 0.0605, where 0.0605 is the standard deviation determined from Excel function [STDEV]).

Significant Condition Adverse to Quality? Yes _____ No Y

Evaluated by: [signature] **Date:** 7/20/2011

Further Disposition: ☒ **No** ☐ **Yes** **If yes,** ☐ **Nonconformance?** ☐ **Deficiency?**

Date Occurrence Closed: 7-20-11

Signature/Date of Responsible QE: [signature]

Signature/Date of Responsible K-Basin Manager: [signature]

Comments:

E-mail discussion with the client describing the genesis of the standard acceptance criteria for U and Pu in the QAPjP/SAP KBC-33786 Rev. 2 is attached to this occurrence report.

Fiskum, Sandra K

From: Schmidt, Andrew J
Sent: Monday, April 11, 2011 11:59 AM
To: Welsh, Terri L; Baker, Ronald B (Ron)
Cc: Carlson, Clark D; Fiskum, Sandra K
Subject: FW: Pu and U control charts for TIMS
Attachments: TIMS Control chart Restored 1.xls; ARF-KW230_TIMS_Pu[1].pdf; ARF-KW230%20TIMS%20U[1].pdf

Terri:

Thank you! I corrected the date in the note below from 4/12/11 vs. 4/11/11).

Andy

From: Welsh, Terri L
Sent: Monday, April 11, 2011 11:48 AM
To: Schmidt, Andrew J; Baker, Ronald B (Ron)
Subject: RE: Pu and U control charts for TIMS

Andy,

The words below reflect our conversation this morning.

Terri Welsh

From: Schmidt, Andrew J [mailto:andy.schmidt@pnl.gov]
Sent: Monday, April 11, 2011 10:14 AM
To: Welsh, Terri L; Baker, Ronald B (Ron)
Subject: FW: Pu and U control charts for TIMS
Importance: High

Terri and Ron:

The purpose of this note is to confirm the technical basis for the LCS recoveries used in Pu and U TIMS, and the applicability of the QAPjP LCS accuracy (recoveries) to minor isotopes (very low abundance).

Based on our brief discussion this morning (4-11-11; AJ Schmidt and TL Welsh), the recoveries in the LCS/standard reference material for U-234 and U-236 are non-issues with respect to the QAPjP and the Safeguard's needs.

Technical basis of LCS recoveries in the QAPjP:

For U, the 2% recovery was based on the TIMS historical performance for U-238 and U-235. (Control Chart)
For Pu, the 6% recovery was based on TIMS historical performance for Pu-239 and Pu-240. (Control Chart)
Control charts we not prepared for other U and Pu isotopes.

The QAPjP is silent on the LCS/SRM recovery, in terms of applicability to low abundance isotopes. The only guidance provided is that given with respect to the 20% duplicate criterion (only applicable to isotopes greater than 1% abundance).

Consequently, a conservative and achievable approach was recommended by PNNL for LCS/SRM accuracy within the TIMS ARFs. In the TIMS ARFs, a threshold was added that the U and Pu LCS accuracies only applied to isotopes present at >0.1% abundance. The adaptation of the >0.1% threshold was previously discussed in a conversation between Fiskum and Welsh. The outcome of the discussion was reflected in the ARFs, which were approved by the BTR.

Recoveries for U-234 and U-236.

LCS recoveries for U-234 and U-236 (less than 0.1% abundance) exceeded 2%. The isotopic Uranium (U) Standard Reference Material (SRM) acceptance criterion of ±2% of the certified value is a requirement specified in the QAPjP/SAP, KBC-40467, Rev. 1, Table 3-7, but only applicable to U-235 and U-238. The ^{234}U and ^{236}U isotopic ratios were significantly less than 0.1 atom %; the sample analysis request specified that ±2% of the certified value criterion applies to values >0.1 atom %. The request had been reviewed and signed by the Buyer's Technical Representative (BTR). The 1-s uncertainties of the ^{234}U and ^{236}U values were large (much greater than 2%, relative) and generally bracketed the ±2% criterion range as shown in Figure 1.

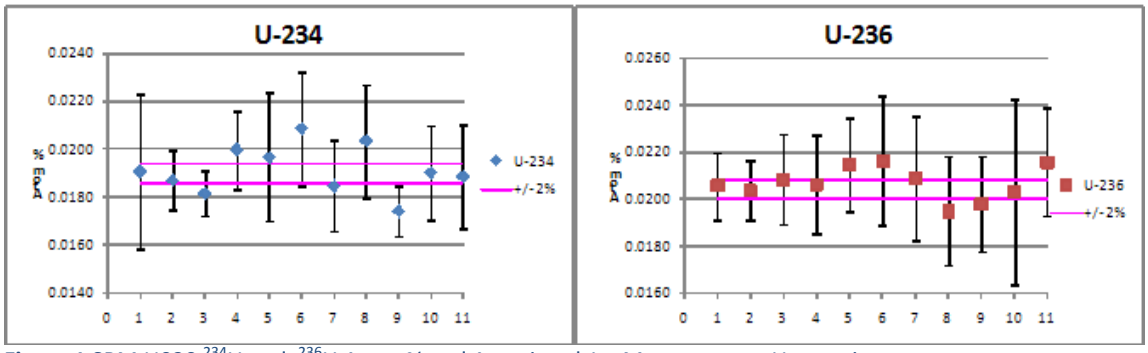


Figure 1 SRM U030 ^{234}U and ^{236}U Atom % and Associated 1-s Measurement Uncertainty

- Andy

From ARF-KW-230 TIMS Pu:

Quality Control

The TIMS analysis processing will need to adhere to the QC defined in the technical procedure PNNL-98523-264, *Mass Spectrometer Isotopic Analysis*, Rev. 2 and project-specific quality control as follows:

Duplicates	The ^{239}Pu, ^{240}Pu, ^{241}Pu, and ^{242}Pu atom percents for a given core sample and duplicate shall agree within ±20%.
Reference Standard	At least one traceable reference standard must be analyzed at the beginning of the analytical batch. The results for ^{238}Pu, ^{239}Pu, ^{240}Pu, ^{241}Pu, ^{242}Pu atom percents shall be within 6% of the certified value where the isotope atom percent of the certified value is >0.1.

From ARF-KW-230 TIMS U:

Quality Control

The TIMS analysis processing will need to adhere to the QC defined in the technical procedure PNNL-98523-264, *Mass Spectrometer Isotopic Analysis*, Rev. 2 and project-specific quality control sample requirements as follows:

Duplicates	The ^{233}U, ^{234}U, ^{235}U, ^{236}U, and ^{238}U atom percents shall agree within ±20% for a given core and duplicate sample.
Reference Standard	Traceable reference standards must be analyzed at the beginning of the analytical batch. The results for ^{234}U, ^{235}U, ^{236}U, and ^{238}U atom percents shall be within 2% of the certified value where the isotope atom percent of the certified value is >0.1.

From: Schmidt, Andrew J
Sent: Wednesday, February 02, 2011 3:14 PM
To: Welsh, Terri L
Subject: FW: Pu and U control charts for TIMS

Terri:

Stan was able to dig up the original file.

- Andy

From: Bos, Stanley J
Sent: Wednesday, February 02, 2011 2:49 PM
To: Schmidt, Andrew J
Subject: RE: Pu and U control charts for TIMS

Andy

Looks like I lucky, and found the old control chart.

Stan

From: Welsh, Terri L
Sent: Tuesday, February 01, 2011 2:58 PM
To: Schmidt, Andrew J
Subject: RE: Pu control charts

Andy,

We both should have a copy in our files. If we do other work, then the applicable SAP could reference the correct isotope for the 2% and 6% limits.

Terri

From: Schmidt, Andrew J [mailto:andy.schmidt@pnl.gov]
Sent: Monday, January 31, 2011 5:49 PM
To: Welsh, Terri L
Cc: Bos, Stanley J
Subject: FW: Pu control charts

Terri:

I have not been able to find the PDFs of Stan's Control charts in my files. Do you need these?

Stan: Do you have these available? I must have deleted the original message.

Andy

From: Welsh, Terri L
Sent: Wednesday, December 19, 2007 4:40 PM
To: Schmidt, Andrew J
Subject: RE: Pu control charts

Andy,

For the Uranium data there is definitely one data point that is a significant player in the standard deviation (also in the mean). I believe that it is an outlier and should not be included for the control limits. I think we should stay with 2% for the U TIMS. The Pu TIMS data definitely shows some scatter and we should use the 6%.

Terri

From: Schmidt, Andrew J [mailto:andy.schmidt@pnl.gov]
Sent: Wednesday, December 19, 2007 4:34 PM
To: Carlson, Clark D; Welsh, Terri L
Cc: Baker, Ronald B (Ron)
Subject: FW: Pu control charts

Clark and Terri:

Stan performed a more detailed review of the TIMS data.

Based on his recommendation, I propose we change the LSC Recovery Values in Table 3 as follows:

U TIMS, = +/- 4% (Stnd Dev. = 1.4) (note Terri: This could be dropped to 2% as we only have one outlier.... your call).

Pu TIMS, +/- 6% (Stnd. Dev = 2%)

- Andy

From: Bos, Stanley J
Sent: Wednesday, December 19, 2007 3:42 PM
To: Schmidt, Andrew J
Subject: Pu control charts

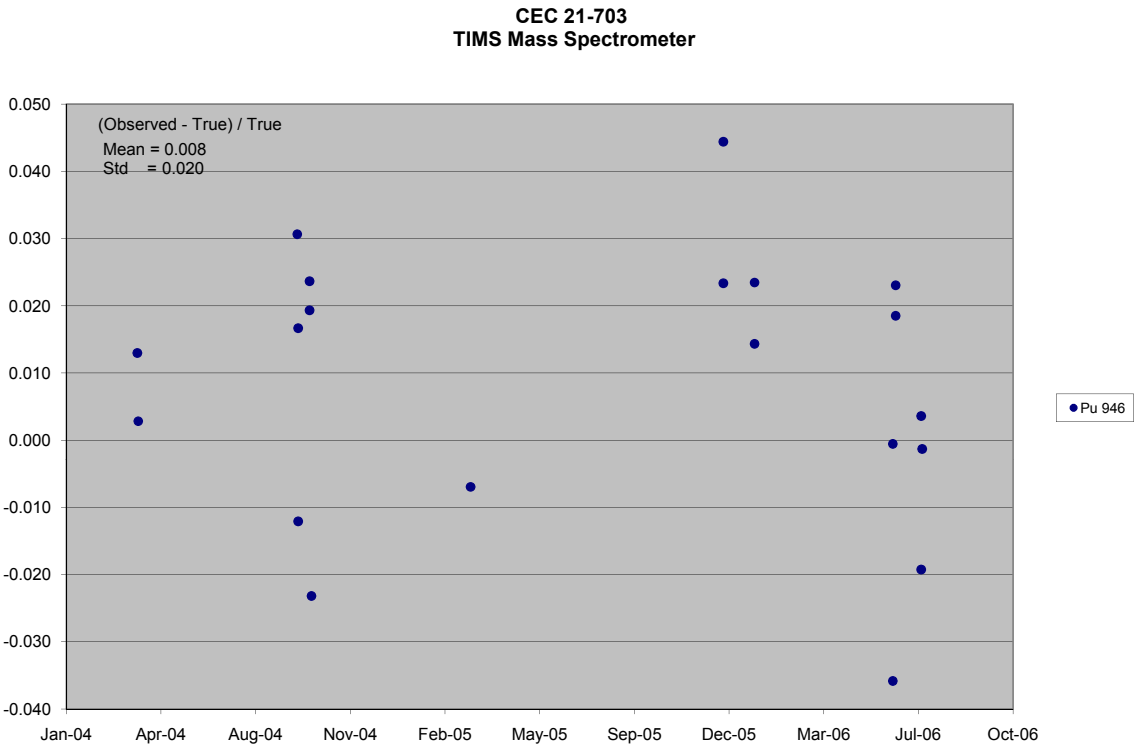
Andy

Attached is another version of the Pu control chart and the spreadsheet used to develop the chart. After our discussions concerning the variance of the Pu TIMS data, I went through the lab book and culled out a number of the values I used in the earlier control chart. The current version is data from Jan 2004 to present, I've eliminated some values where duplicate or replacement runs were made. I think this current list accurately reflects the Pu standards data that was used during analytical runs.

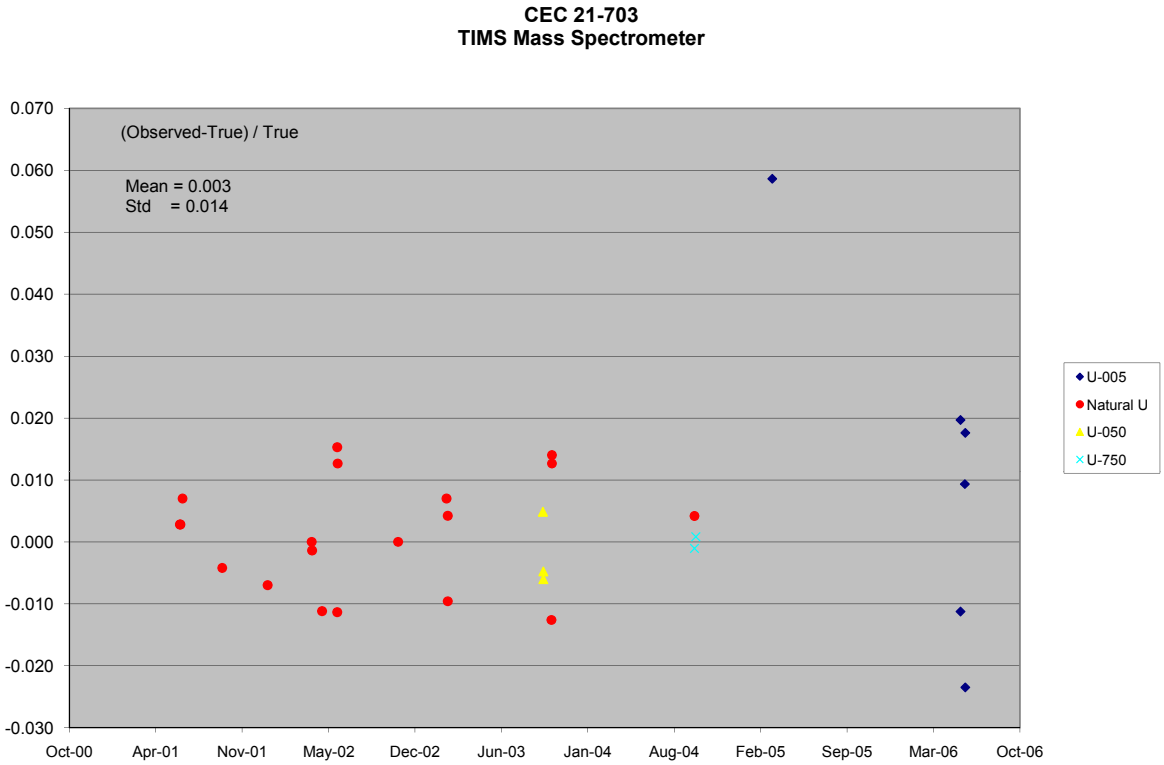
After reviewing the data, my recommendation is to use a 3 sigma uncertainty band. I believe that better reflects the scatter in the Pu standards.

Stan Bos

<<TIMS Control chart Pu .pdf>> <<TIMS Control chart.xls>>


CEC 21-703
TIMS Mass Spectrometer
(Observed - True) / True
Mean = 0.008
Std = 0.020
0.050
0.040
0.030
0.020
0.010
0.000
-0.010
-0.020
-0.030
-0.040
Jan-04
Apr-04
Aug-04
Nov-04
Feb-05
May-05
Sep-05
Dec-05
Mar-06
Jul-06
Oct-06
Pu 946

52578-05-09-11 (Attachment 1)



Pu 946

	240	239	
1/17/2000	12.47	85.674	0.1455517
1/11/2000	12.488	85.665	0.1457772
2/23/2002	12.392	85.782	0.1444592
7/27/2000	12.459	85.724	0.1453385
1/2/2001	12.457	85.727	0.1453101
7/21/2001	12.443	85.747	

			9/15/2004	946	12.461	85.894	0.145074
					RO-RT /RT		
9460401	3/29/2004	12.628	85.93	0.1470	0.013		
9460402	3/30/2004	12.51	85.989	0.1455	0.003		
9460404	9/14/2004	12.794	85.567	0.1495	0.031		
9460405	9/15/2004	12.334	86.058	0.1433	-0.012		
9460406	9/15/2004	12.643	85.72	0.1475	0.017		
9460407	9/27/2004	12.672	85.692	0.1479	0.019		
9460408	9/27/2004	12.714	85.613	0.1485	0.024		
9460409	9/29/2004	12.203	86.111	0.1417	-0.023		
9460501	3/16/2005	12.411	86.148	0.1441	-0.007		
9460601	12/8/2005	12.722	85.692	0.1485	0.023		
9460602	12/8/2005	12.966	85.575	0.1515	0.044		
9460604	1/10/2006	12.717	85.65	0.1485	0.023		
9460605	1/10/2006	12.637	85.877	0.1472	0.014		
9460606	6/5/2006	12.472	86.017	0.1450	-0.001		
9460607	6/5/2006	12.094	86.461	0.1399	-0.036		
9460608	6/8/2006	12.727	85.752	0.1484	0.023		
9460609	6/8/2006	12.691	85.889	0.1478	0.019		
9460611	7/5/2006	12.301	86.456	0.1423	-0.019		
9460612	7/5/2006	12.531	86.067	0.1456	0.004		
9460613	7/6/2006	12.460	86.000	0.1449	-0.001		

n	20
Sum	0.158
mean	0.008
std	0.020

		235	238		RO-RT/RT			
005						0.4895	99.504	0.004919
005	3/16/2005	0.518	99.464	0.005208	0.059			
	5/26/2006	0.484	99.503	0.004864	-0.011			
	5/26/2006	0.499	99.476	0.005016	0.020			
	6/5/2006	0.494	99.489	0.004965	0.009			
	6/6/2006	0.498	99.48	0.005006	0.018			
	6/6/2006	0.478	99.503	0.004804	-0.023			
nat	9/20/2001	0.717	99.278	0.007222	-0.004	0.72	99.275	0.007253
	6/14/2001	0.722	99.272	0.007273	0.003			
	6/15/2001	0.722	99.272	0.007273	0.003			
	6/20/2001	0.725	99.269	0.007303	0.007			
	1/3/2002	0.715	99.278	0.007202	-0.007			
nat	10/22/2003	0.711	99.285	0.007161	-0.013			
	10/23/2003	0.729	99.26	0.007344	0.013			
	10/23/2003	0.73	99.263	0.007354	0.014			
nat	2/21/2003	0.725	99.268	0.007303	0.007			
	11/1/2002	0.72	99.274	0.007253	0.000			
	6/13/2002	0.712	99.298	0.00717	-0.011			
	6/13/2002	0.731	99.274	0.007363	0.015			
	6/14/2002	0.729	99.26	0.007344	0.013			
	4/15/2002	0.72	99.274	0.007253	0.000			
	4/16/2002	0.719	99.274	0.007243	-0.001			
	5/9/2002	0.712	99.281	0.007172	-0.011			
	2/24/2003	0.713	99.261	0.007183	-0.010			
	2/24/2003	0.723	99.27	0.007283	0.004			
nat	9/17/2004	0.723	99.273	0.007283	0.004			
050	10/2/2003	5.033	94.89	0.05304	0.005			
050	10/3/2003	4.987	94.933	0.052532	-0.005	5.01	94.915	0.052784
050	10/3/2003	4.981	94.938	0.052466	-0.006			
750	9/17/2004	75.367	23.829	3.162827	-0.001			
750	9/20/2004	75.395	23.793	3.168789	0.001			
						75.357	23.801	3.166127
					31			
					0.090			
				mean	0.003			
				Std	0.015			

			9/15/2004	946	12.461	85.894	0.145074
					RO-RT /RT		
9460401	3/29/2004	12.628	85.93	0.146957	0.013		
9460402	3/30/2004	12.51	85.989	0.145484	0.003		
9460404	9/14/2004	12.794	85.567	0.14952	0.031		
9460405	9/15/2004	12.334	86.058	0.143322	-0.012		
9460406	9/15/2004	12.643	85.72	0.147492	0.017		
9460407	9/27/2004	12.672	85.692	0.147878	0.019		
9460408	9/27/2004	12.714	85.613	0.148505	0.024		
9460409	9/29/2004	12.203	86.111	0.141712	-0.023		
9460501	3/16/2005	12.411	86.148	0.144066	-0.007		
9460601	12/8/2005	12.722	85.692	0.148462	0.023		
9460601	12/8/2005	12.722	85.692	0.148462	0.023		
9460602	12/8/2005	12.966	85.575	0.151516	0.044		
9460604	1/10/2006	12.717	85.65	0.148476	0.023		
9460605	1/10/2006	12.637	85.877	0.147152	0.014		
9460606	6/5/2006	12.472	86.017	0.144995	-0.001		
9460607	6/5/2006	12.094	86.461	0.139878	-0.036		
9460608	6/8/2006	12.727	85.752	0.148416	0.023		
9460609	6/8/2006	12.691	85.889	0.14776	0.019		
9460611	7/5/2006	12.301	86.456	0.14228	-0.019		
9460612	7/5/2006	12.531	86.067	0.145596	0.004		
9460613	7/6/2006	12.460	86.000	0.144884	-0.001		

n	21
Sum	0.181
mean	0.009
std	0.020

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PLANNING

Topic/Issue to be evaluated: QE Review of Draft Report, Isotopic Analysis in KW230 [Sic] Core Samples (June 2011)

This report has been revised to include the addition of a data reduction spreadsheet that was included in Appendix E to the draft -210 final report and was not included in the TIMS data package submitted for QE review.

This surveillance was undertaken to evaluate the draft Report, that was wrongly titled "Isotopic Analysis in KW230 Core Samples (June 2011)" that should rightly be titled "Isotopic Analysis in KW210 Core Samples from the Settler Sludge" for the samples listed in Table 1 below.

Table 1. Sample IDs for Isotopic Analysis in KW210 Core Samples from the Settler Sludge

Project Sample ID	RPL/ASO Sample ID—Uranium	RPL/ASO Sample ID—Plutonium Isotopic	K Basin Core Sample ID
TI001-A1-B	11- 0419-129-S-U	11- 0419-129-S-Pu	KW210-A1
TI001-A1-C	11-0419 -129-D-U	11-0419 -129-D-Pu	KW210-A1 duplicate
TI002-A4-B	11-0420-129-S-U	11-0420-129-S-Pu	KW210-A4
TI002-A4-C	11-0420-129-D-U	11-0420-129-D-Pu	KW210-A4 duplicate
TI002-B1-B	11-0421-129-S-U	11-0421-129-S-Pu	KW210-B1
TI002-B1-C	11-0421-129-D-U	11-0421-129-D-Pu	KW210-B1 duplicate
TI001-B3-B	11-0422-129-S-U	11-0422-129-S-Pu	KW210-B3
TI001-B3-C	11-0422-129-D-U	11-0422-129-D-Pu	KW210-B3 duplicate
NA	11-0419-129-PB-U	11-0419-129-PB-Pu	ASO preparation blank

PERFORMANCE

Date Initiated: 28 June 2011	**Location:** RPL
Date Completed: 1 July 2011	**DRAFT Report Issued:** 1 July 2011 **FINAL Report Issued**: 21 July 2011 **Revised Report Issued:** 3 August 2011
Contacts: Stan Bos, Clark Carlson, Sandy Fiskum, and Jen Braley	**Org Code:** PE 137, D9H63
Surveillance Team: Deborah Coffey	

PRELIMINARY INFORMATION

Source of Requirement(s):

KBC-33786, Rev. 2, *Quality Assurance Project Plan/Sampling and Analysis Plan (QAPjP/SAP for Sludge in the KW Engineered Containers*, CH2M Hill Plateau Remediation Company, Richland, Washington.

HNF-36985, Rev. 3, *Data Quality Objectives for Sampling and Analysis of K Basin Sludge (DQO)*, , CH2M Hill Plateau Remediation Company, Richland, Washington.

Other Applicable Documents:

- RPL-OP-001, Rev. 9, *Routine Research Operations*
- PNNL-98523-264, Rev. 2, *Mass Spectrometer Isotopic Analysis*
- 52578-ADM-001, Rev. 0, *K Basin Requests for Analysis and Guidelines for Data Reporting and Technical, QE, and Spreadsheet Reviews.*
- Analysis Request for Plutonium and Uranium Isotopes in K Basin Core Samples from the Settler Sludge Origin, KW210 (S Fiskum to S. Bos, signed off 4/14/11)

Purpose/Scope:

The 52578 Project QE is required via the QA Program to perform a review of K Basin reports generated within the Radiochemical Processing Laboratory (RPL) to determine traceability and accuracy of data contributing to the completed report.

The draft report was reviewed to evaluate that the data are what they purport to be, that the reported results reflect what was actually done and the results meet applicable requirements. When deficiencies in the data are identified, then those deficiencies need to be documented as a K Basin Occurrence Report for the data user's review and, when applicable, resolved by corrective action.

The requirements for this work are defined in Table 3-6 and Table 3-7, KBC-33786, Rev. 2, *Quality Assurance Project Plan/Sampling and Analysis Plan (QAPjP/SAP for Sludge in the KW Engineered Containers* and in Analysis Request for Plutonium and Uranium Isotopes in K Basin Core Samples from the Settler Sludge Origin, KW210.

Requirements:

Requirements from KBC-33786, Rev. 2, the applicable QAPjP/SAP s are the following:

- Reporting requirements – atom % of U and Pu for ^{233}U, ^{234}U, ^{235}U, ^{236}U, and ^{238}U; and ^{238}Pu, ^{239}Pu, ^{240}Pu, ^{241}Pu, and ^{242}Pu.
- There is no specified reporting limit
- The DQO for preparative duplicate precision is $\pm$ 20%.
- There is no accuracy DQO for a post-prep MS

- There are DQOs for a LCS sample to be analyzed at a frequency of 1/batch (2% for U-TIMS and 6% for Pu-TIMS)

Requirements from the analysis request were the following:

Duplicates	The ^{239}Pu, ^{240}Pu, ^{241}Pu, and ^{242}Pu atom percents for a given core sample and duplicate shall agree within ±20% where the isotopic abundance is greater than 1%.
	The ^{233}U, ^{234}U, ^{235}U, ^{236}U, and ^{238}U atom percents shall agree within ±20% for a given core and duplicate sample where the isotopic abundance is greater than 1%.
Reference Standards	At least one traceable reference standard must be analyzed at the beginning of the analytical batch. The results for ^{238}Pu, ^{239}Pu, ^{240}Pu, ^{241}Pu, ^{242}Pu atom percents shall be within 6% of the certified value where the isotope atom percent of the certified value is >0.1.
	Traceable reference standards must be analyzed at the beginning of the analytical batch. The results for ^{234}U, ^{235}U, ^{236}U, and ^{238}U atom percents shall be within 2% of the certified value where the isotope atom percent of the certified value is >0.1.

Note that the Reference Standard acceptance criteria identified in the QAPjP/SAP KBC33786, Rev. 2 were developed specifically for ^{239}Pu and ^{240}Pu (±6%) and for ^{235}U and ^{238}U (±2%). These criteria are conservatively applied to other (minor) isotopes in the Standard Reference Material (SRM) is where the minor isotope concentration exceeds 0.1 atom%.

TIMS Reporting:

Report ^{238}Pu, ^{239}Pu, ^{240}Pu, ^{241}Pu, and ^{242}Pu atom percents for each sample in a tabular format.

Report ^{233}U, ^{234}U, ^{235}U, ^{236}U, and ^{238}U atom percents for each sample in a tabular format.

Provide a report with the following elements: a data report Cover Page, a Narrative, the Data Summary, the signed Analysis Request Form and the Supporting Data.

When issues were found, the cognizant staff were contacted to resolve issues prior to completion of the review.

APPROACH:

The report and supporting data package contained the following information:

1. Analysis Request for Plutonium and Uranium Isotopes in K Basin Core Samples from the Settler Sludge Origin, KW210 (S Fiskum to S. Bos, signed off 4/14/11)

2. Email submittal of draft report and Excel spreadsheets on 6/23/11

3. DRR form for Technical Reviewer, Jen Braley – This form was incomplete and lacked a title for the document reviewed and the organization of the Technical Reviewer. Although the DRR states that there were editorial comments, there was no indication what those comments were and the form did not show that the comments were resolved.

4. Cover page –Present in report attached to DRR, but missing in electronic version submitted. Cover page did not have the right title; says "Isotopic Analysis in KW230 Core Samples (June 2011)" that should rightly be titled "Isotopic Analysis in KW210 Core Samples from the Settler Sludge". Cover page did not have signatures.

5. Memo Report, Pu and U isotopic analysis (pp. 4, plus four (4) tables)

6. Mass Spec data print-outs for 8 U samples plus PB analyzed on 5/6 and 9/11; references LRB 56999 p. 76-77. Technical Review by P Berry is noted on each sample data report.

7. NBS SRM U-030 Certificate, and SRM analysis data for 5/3/11 (1); 5/6/11 (2) and 5/9/11 (2); references LRB 56999 pp. 76 & 77.
8. Mass Spec data print-outs for 8 Pu samples plus PB analyzed on 5/18 & 25/11; references LRB 56999 p. 77 & 78. Technical Review by P Berry is noted on each sample data report.

9. Summary of Standard results – for Pu 946; NBS Certificate of Analysis for SRM 946, Plutonium Isotopic Standard; and SRM analysis data for 5/17, 18 & 25/11 references LRB 56999 pp. 77 &78. See decay-corrected SRM 946 values with technical review by LR Greenwood.

10. Copies of 56999 LRB pages 77 and 78. Missing a copy of LRB page 76. Pages were not signed as to completion of a Technical Review.

11. Copy of ASR 8798 Thermal Ionization Mass Spectrometry Sample Preparation, Rev. 1 –dated 6/3011- added by QE Reviewer

12. TIMS Isotopic Data for KW Basin Cores KW230 (?), prepared 6/15/2011 by Stan Bos; Excel file KW210TIMS data June 2011.xls with worksheets:
    - Uranium Std.
    - U TIMS
    - Final Results 1
    - Plutonium Std
    - Pu TIMS
    - Final Results 2

    Included a Spreadsheet Review Form completed by J Braley that was signed, but not dated.

13. Missing a copy of draft OR 52578-05-09-11.

14. **Excel Spreadsheet ASR 8798 Data Reduction Rev. 1.xlsx, pp.4; prepared by SK Fiskum/M Fountain 7/18/2011; Technical review provided by Cal Delegard 23 July 2011; titled K Basin Radionuclide Calculations; plus a spreadsheet titled ASR 8798 Data reduction, Rev.1.xlsx TIMS Rad wet mass basis; with no indication in the data package of technical review status**
    - **Worksheet – Spreadsheet Review Form –**

- **TIMS Rad Calc**

Jen Braley was identified as the technical reviewer of the data package **with the exception of item 14 above,** and this was documented with a DRR.

The completed surveillance report will be posted to the SharePoint site and submitted as a project record. Any documentation associated with this surveillance will be available in the Lead Auditor's office, Building 325, 5-G.

RESULTS

Samples were prepared by ASO and submitted to S. Bos for TIMS analysis. ASO TIMS sample preparation activities are documented in an ASO report:

- ASR 8798 Thermal Ionization Mass Spectrometry Sample Preparation, Rev. 1 –dated 6/3011

The draft report initiated by the K Basin analysis request dated 4/14/11 was provided as the basis for the data quality review. Eight (8) samples and an ASO preparation blank (PB) were provided for analysis on 5/3/11 as noted above in Table 1.

Procedure:

- PNNL-98523-264, Rev. 2, *Mass Spectrometer Isotopic Analysis*

M&TE used:

Location	Description	Manufacturer	Model	Unique ID
RPL/320	Mass spec	CEC	21-703	467806 WB76849 SN:21793
	NBS SRM U-030	NBS		
	NBS SRM 946	NBS		

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.

Summary of Identified Concerns:

Note: Concerns identified in the review are expected to be addressed in a comment resolution meeting to be scheduled by the report author when the comments have been addressed.

A comment resolution meeting was convened on 15 July 2011; , Stan Bos and Deborah Coffey attended to disposition review comments and discuss the draft Occurrence Report, OR 52578-05-09-11. The report was re-submitted on 15 July 2011. There were no outstanding identified findings or observations.

Issues of concern:

1. Apparent data transcription errors in a few places on U results; refer to comment copy. Discussed and tracked correct data to spreadsheet/normalized data. No further action required. Closed.

2. Add footnotes to both U and Pu reporting tables to explain why PB data are ND; define ND in the table. The PB sample did not have a measureable single so that the data could not be collected. The U and Pu results are appropriately tagged with ND in the results section but ND is not defined in the table nor footnoted regarding the lack of signal. This was done; no further action required. Closed.

3. Typo in Pu-238 acceptance criteria for Pu-946 std. Says 0.176 to 00.199; should be 0.199. This was corrected no further action required. Closed.

4. DRR form for Technical Reviewer, Jen Braley – This form is incomplete and lacks a title for the document reviewed and the organization of the Technical Reviewer. Although the DRR states that there were editorial comments, there is no indication what those comments were and the form does not show that the comments were resolved. There also appeared to be information added that is not initialed and dated that was added by someone other than J. Braley. Re-done DRR provided and initialed/dated. Closed.

5. Cover page does not have the right title; says "Isotopic Analysis in KW230 Core Samples (June 2011)" that should rightly be titled "Isotopic Analysis in KW210 Core Samples from the Settler Sludge". This was corrected no further action required. Cover page does not have signatures.

6. 56999 LRB pages 77 and 78. Will need copy of LRB page 76. Pages are not signed as to completion of a Technical Review. Obtained a copy of page 76 and re-reviewed pages 77 and 78 against the data to reconcile log # issues. See issue #8. Closed.

7. Lack of Page 76 of LRB affects traceability of results for the following samples: Obtained a copy of page 76 and re-reviewed pages 77 and 78 against the data to reconcile log # issues. See issue #8. Closed.

Project Sample ID	RPL/ASO Sample ID—Uranium	RPL/ASO Sample ID—Plutonium Isotopic	K Basin Core Sample ID
TI001-A1-B	11- 0419-129-S-U	11- 0419-129-S-Pu	KW210-A1
TI001-A1-C	11-0419 -129-D-U	11-0419 -129-D-Pu	KW210-A1 duplicate
TI002-A4-B	11-0420-129-S-U	11-0420-129-S-Pu	KW210-A4
TI002-B1-C	11-0421-129-D-U	11-0421-129-D-Pu	KW210-B1 duplicate
TI001-B3-B	11-0422-129-S-U	11-0422-129-S-Pu	KW210-B3
TI001-B3-C	11-0422-129-D-U	11-0422-129-D-Pu	KW210-B3 duplicate
NA	11-0419-129-PB-U	11-0419-129-PB-Pu	ASO preparation blank

8. Two samples documented on LRB page 78 do not match the data print-out results for sample log entries 1211017 and 1211018; LRB says these were samples 11-0421-129-S-Pu and 11-0421-129D-Pu, but data print-out calls these samples 11-0422-129-S-Pu and 11-0422-129D-Pu. This discrepancy needs to be unraveled. This problem was resolved and log entries on page 78 required corrections by line-out, initial/date. Page 78 replaced in record. Closed.

9. Data print-outs for May 18th list the incorrect procedure – Should be PNNL-98523-264, Rev. 2, *Mass Spectrometer Isotopic Analysis,* but "PNL-MA-599" is listed. Correct procedure added by hand correction, initial and date. Closed.

10. Why are there no corresponding data print-outs for standards samples? Discussed. Logbook works OK. Closed.

11. Spreadsheet Review Form completed by J Braley and signed, but not dated. Corrected. Closed.

12. The practice of calculating RPD values using more significant figures than are reported leads to apparent errors in data reporting. For example, for two samples the sample and the duplicate are the same reported values which would of course be a relative percent difference of 0%, but these are reported as 8% and 6% RPD which is ridiculous. Corrected. Closed.

13. The standard analyzed for U reporting failed rather profoundly for two isotopes (^{234}U - 3 of 5 and ^{236}U 5 of 5); How does this impact the accuracy of the data collected? An Occurrence Report has been issued to address the impact of reference standard failures (OR 52578-05-09-11). Closed.

14. Although a draft K Basin Occurrence Report is mentioned in the narrative, it was not included in the data package provided for review, and due to PNNL network issues could not be reviewed as part of this surveillance. A completed and signed copy is needed to complete the surveillance. Obtained. Closed.

15. Also RPD failures needed to be added to the OR and referenced in the text. Email sent 7/18/11 on this issue. Added to OR 52578-05-09-11. Corrected. Closed.

16. **Data reduction and reporting is being performed separately from data package review, was not reviewed by the TIMS analyst, was not submitted for QE review, and the basis for data reduction, namely normalizing Pu isotopic results to the ^{238}Pu AEA results was not technically established in the TIMS review process. Data reduction and reporting also**

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involved conversion of wt% results to units of µg/g and µCi/g on a dry mass and settled mass basis. Any errors are the responsibility of the authors and technical reviewer. Basis for normalizing Pu isotopic results to the ^{238}Pu AEA was discussed with Karl Pool w.r.t. selection of $_{238}$Pu over other isotopes analyzed. No further action.

CORRECTIVE ACTION

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

Surveillance Performed By:

Deborah Coffey, Quality Engineer

Date: 3 August 2011

DISTRIBUTION:

Clark Carlson – P7-25
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Spreadsheet Review Form

Spreadsheet Author Name:	SK Fiskum/M Fountain	Review Date:	23 July 2011
Date Prepared:	7/18/2011	Spreadsheet Subject:	KW210 Core Characterization
Reviewer Name:	Cal Delegard	File Name:	ASR 8798 data reduction.xlsx
Reviewer Title:	Senior Research Scientist	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: Verification of data input to tabs "TIMS Rad Calc." and "TIMS Rad wet mass basis" and calculations for "TIMS Rad wet mass basis". Note that calc. cells locked for "TIMS Rad Calc."
☐	100% Verification Review (Verification of all cells/calculations)		

REVIEW CHECK LIST

Spreadsheet/Calculation Identification

Spreadsheet Information	Yes	No	NA
Title:			
Revision Number:			
Date Prepared:			
Prepared by:			
General Statement of Purpose:			
General Description of Approach:			
Comments: "Overview" worksheet contains all this information. ✓			

Input Values	Yes	No	NA
Are input parameters correct (verified with source?)	✓		
Are parameter units consistent?	✓		
Are input values properly referenced?	✓		
Are input uncertainties correct?			✓
Comments: Input values copied from ASO and TIMS electronic files, which are properly referenced. Cover sheet references source data for physical properties.			

Assumptions	Yes	No	NA
Are assumptions clearly stated?			
Are assumptions supported and justified?			
Are assumptions reasonable?			
Comments: "Overview" worksheet contains all this information. ✓			

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

Results/Conclusions	Yes	No	NA
Are formulas consistent in cells?	✓		
Are calculations correct?	✓		
Are conclusions consistent with results?	✓		
Are conclusions consistent with applicable limits?	✓		
Comments: See comments to "Other" in "Scope of Spreadsheet Review", above.			

Reviewer Sign/Date: 23 July 2011

Filename: ASR 8798 Data Reduction Rev1.xlsx
Tab: TIMS Rad Calc.

K Basin Radionuclide Calculations

	Container	KW210	KW210	KW210	KW210	KW210	KW210	KW210	KW210
	Core	A1	A1	A4	A4	B1	B1	B3	B3
	Sample ID	TI001-A1-B	TI001-A1-C	TI002-A4-B	TI002-A4-C	TI002-B1-B	TI002-B1-C	TI001-B3-B	TI001-B3-C
	ASO ID	11-0419-129-S	11-0419-129-D	11-0420-129-S	11-0420-129-D	11-0421-129-S	11-0421-129-D	11-0422-129-S	11-0422-129-D
		Analysis Results							
	Total U	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g
	U/KPA	98,000	108,000	87,900	77,100	55,500	48,800	56,200	53,100
	U/ICP-OES	93,800	103,000	82,800	73,600	51,900	45,600	55,700	51,000
	TIMS	Atom%	Atom%	Atom%	Atom%	Atom%	Atom%	Atom%	Atom%
	^{233}U	0	0	0	0	0	0	0	0
	^{234}U	0.008	0.007	0.007	0.007	0.007	0.007	0.008	0.011
	^{235}U	0.711	0.709	0.702	0.705	0.706	0.704	0.710	0.714
	^{236}U	0.073	0.076	0.072	0.074	0.071	0.072	0.071	0.077
	^{238}U	99.208	99.208	99.220	99.214	99.216	99.217	99.211	99.198
	^{238}Pu	0.095	0.155	0.330	0.232	0.079	0.142	0.100	0.235
	^{239}Pu	86.09	86.17	85.70	86.06	86.42	86.17	86.22	85.87
	^{240}Pu	12.91	12.91	13.18	12.97	12.79	12.97	12.91	13.16
	^{241}Pu	0.674	0.515	0.531	0.507	0.486	0.484	0.535	0.521
	^{242}Pu	0.231	0.250	0.253	0.228	0.227	0.234	0.229	0.209
	AEA	µCi/g	µCi/g	µCi/g	µCi/g	µCi/g	µCi/g	µCi/g	µCi/g
	^{238}Pu	2.27E+0	2.56E+0	2.30E+0	2.01E+0	1.59E+0	1.65E+0	1.85E+0	1.77E+0
	$^{239+240}Pu$	1.78E+1	1.92E+1	1.66E+1	1.47E+1	1.23E+1	1.26E+1	1.36E+1	1.26E+1
	^{241}Am	1.82E+1	1.97E+1	1.71E+1	1.43E+1	1.51E+1	1.48E+1	1.54E+1	1.47E+1
	^{237}Np	1.74E-3	2.16E-3	1.30E-3	1.19E-3	1.78E-3	7.49E-4	1.14E-3	1.12E-3
	GEA	µCi/g	µCi/g	µCi/g	µCi/g	µCi/g	µCi/g	µCi/g	µCi/g
	^{60}Co	2.30E-1	1.41E-1	1.34E-1	1.21E-1	7.34E-2	7.31E-2	6.86E-2	6.39E-2
	^{134}Cs	<3.E-3	<6.E-3	<3.E-3	<6.E-3	<4.E-3	<3.E-3	<5.E-3	<5.E-3
	^{137}Cs	3.68E+2	3.93E+2	3.38E+2	3.81E+2	1.43E+2	1.68E+2	1.13E+2	1.02E+2
	^{152}Eu	<5.E-3	<8.E-3	<5.E-3	<9.E-3	<4.E-3	<7.E-3	<1.E-2	<1.E-2
	^{154}Eu	7.30E-1	7.59E-1	5.84E-1	5.41E-1	4.71E-1	5.16E-1	5.28E-1	5.14E-1
	^{155}Eu	1.18E-1	1.26E-1	< 6.E-2	< 1.E-1	8.71E-2	1.20E-1	8.04E-2	7.35E-2
	^{241}Am	1.96E+1	2.13E+1	1.64E+1	1.39E+1	1.41E+1	1.68E+1	1.48E+1	1.39E+1

Entries were taken from the TIMS raw data package (U TIMS and Pu TIMS spreadsheet tabs), and the cell entries for U KPA and AEA (embedded in this spreadsheet) such that un-rounded numbers were input here. Rounding was conducted at the very end to produce results in atom%, µCi/g, and µg/g. The RPD values were based on the un-rounded or untruncated values as well.

Note that GEA (and ICP-OES U) data values were input into the spreadsheet with only 3 signficicant figures therefore, GEA and U ICP-OES data have been rounded.

The 233U values (<0.003 atom%) were set to 0 atom%.

Filename: ASR 8798 Data Reduction Rev1.xlsx
Tab: TIMS Rad Calc.

K Basin Radionuclide Calculations

	A	B	C	D	E	F	G	H	I	J	K	L
34			**Container**	**KW210**	**KW210**	**KW210**	**KW210**	**KW210**	**KW210**	**KW210**	**KW210**	
35			**Core**	**A1**	**A1**	**A4**	**A4**	**B1**	**B1**	**B3**	**B3**	
36			**Sample ID**	**TI001-A1-B**	**TI001-A1-C**	**TI002-A4-B**	**TI002-A4-C**	**TI002-B1-B**	**TI002-B1-C**	**TI001-B3-B**	**TI001-B3-C**	
37			**ASO ID**	**11-0419-129-S**	**11-0419-129-D**	**11-0420-129-S**	**11-0420-129-D**	**11-0421-129-S**	**11-0421-129-D**	**11-0422-129-S**	**11-0422-129-D**	
38			Atomic weight U	238.03	238.03	238.03	238.03	238.03	238.03	238.03	238.03	
39			^{233}U, wt% U	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	
40			^{234}U, wt% U	0.0078	0.0066	0.0067	0.0073	0.0065	0.0069	0.0076	0.0107	
41			^{235}U, wt% U	0.7021	0.7003	0.6928	0.6958	0.6971	0.6948	0.7009	0.7050	
42			^{236}U, wt% U	0.0727	0.0750	0.0714	0.0734	0.0708	0.0716	0.0705	0.0766	
43			^{238}U, wt% U	99.2174	99.2181	99.2291	99.2235	99.2256	99.2267	99.2210	99.2078	
44			Atom fraction ^{239}Pu in ^{239,240}Pu	0.86956	0.86972	0.86667	0.86900	0.87106	0.86915	0.86974	0.86710	
45			Mass fraction ^{239}Pu in ^{239,240}Pu	0.86908	0.86925	0.86618	0.86853	0.87059	0.86868	0.86926	0.86662	
46			^{239}Pu, µCi/g sludge	1.150E+01	1.235E+01	1.060E+01	9.475E+00	7.946E+00	8.085E+00	8.763E+00	8.084E+00	
47			^{240}Pu, µCi/g sludge	6.336E+00	6.797E+00	5.988E+00	5.246E+00	4.320E+00	4.471E+00	4.821E+00	4.551E+00	
48			At% $^{238}Pu_{AEA}$, w/ resp. to $^{239}Pu_{TIMS}$	0.0619	0.0651	0.0676	0.0666	0.0630	0.0638	0.0663	0.0684	
49			^{238}Pu, atom%, normalized	0.0619	0.0651	0.0678	0.0667	0.0630	0.0639	0.0663	0.0685	
50			^{239}Pu, atom%, normalized	86.1150	86.2499	85.9263	86.2026	86.4305	86.2351	86.2507	86.0165	
51			^{240}Pu, atom%, normalized	12.9180	12.9196	13.2193	12.9945	12.7936	12.9825	12.9181	13.1834	
52			^{241}Pu, atom%, normalized	0.6744	0.5156	0.5327	0.5082	0.4860	0.4849	0.5353	0.5222	
53			^{242}Pu, atom%, normalized	0.2306	0.2498	0.2540	0.2280	0.2270	0.2337	0.2296	0.2094	
54			Atomic weight Pu	239.20	239.20	239.20	239.20	239.20	239.20	239.20	239.20	
55			^{238}Pu, wt% Pu	0.0617	0.0648	0.0675	0.0664	0.0627	0.0635	0.0660	0.0682	
56			^{239}Pu, wt% Pu	86.0613	86.1970	85.8724	86.1498	86.3785	86.1824	86.1979	85.9632	
57			^{240}Pu, wt% Pu	12.9641	12.9658	13.2663	13.0410	12.8395	13.0289	12.9643	13.2305	
58			^{241}Pu, wt% Pu	0.6796	0.5196	0.5368	0.5121	0.4897	0.4886	0.5394	0.5262	
59			^{242}Pu, wt% Pu	0.2334	0.2528	0.2570	0.2307	0.2297	0.2365	0.2323	0.2119	
60												
61												
62												
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67												
68												

Filename: ASR 8798 Data Reduction Rev1.xlsx
Tab: TIMS Rad Calc.

K Basin Radionuclide Calculations

	A	B	C	D	E	F	G	H	I	J	K	L
69			**Container**	**KW210**	**KW210**	**KW210**	**KW210**	**KW210**	**KW210**	**KW210**	**KW210**	
70			**Core**	**A1**	**A1**	**A4**	**A4**	**B1**	**B1**	**B3**	**B3**	
71			**Sample ID**	**TI001-A1-B**	**TI001-A1-C**	**TI002-A4-B**	**TI002-A4-C**	**TI002-B1-B**	**TI002-B1-C**	**TI001-B3-B**	**TI001-B3-C**	
72			**ASO ID**	**11-0419-129-S**	**11-0419-129-D**	**11-0420-129-S**	**11-0420-129-D**	**11-0421-129-S**	**11-0421-129-D**	**11-0422-129-S**	**11-0422-129-D**	
73	**Isotopic Conc., Weight Basis, µg/g, as reported**		^{233}U, KPA	0.000E+0	0.000E+0	0.000E+0	0.000E+0	0.000E+0	0.000E+0	0.000E+0	0.000E+0	
74			^{234}U, KPA	7.633E+0	7.104E+0	5.903E+0	5.633E+0	3.633E+0	3.377E+0	4.279E+0	5.667E+0	
75			^{235}U, KPA	6.880E+2	7.563E+2	6.089E+2	5.364E+2	3.869E+2	3.390E+2	3.939E+2	3.743E+2	
76			^{236}U, KPA	7.127E+1	8.104E+1	6.275E+1	5.657E+1	3.927E+1	3.496E+1	3.960E+1	4.066E+1	
77			^{238}U, KPA	9.723E+4	1.072E+5	8.722E+4	7.650E+4	5.507E+4	4.842E+4	5.576E+4	5.268E+4	
78			Total Pu, ^{238}Pu basis	2.154E+2	2.311E+2	1.989E+2	1.773E+2	1.483E+2	1.512E+2	1.639E+2	1.516E+2	
79			Total Pu, ^{239,240}Pu basis	2.154E+2	2.311E+2	1.989E+2	1.773E+2	1.483E+2	1.512E+2	1.639E+2	1.516E+2	
80			^{238}Pu	1.328E-1	1.498E-1	1.342E-1	1.177E-1	9.294E-2	9.611E-2	1.082E-1	1.034E-1	
81			^{239}Pu	1.854E+2	1.992E+2	1.708E+2	1.528E+2	1.281E+2	1.303E+2	1.413E+2	1.303E+2	
82			^{240}Pu	2.793E+1	2.996E+1	2.639E+1	2.312E+1	1.904E+1	1.970E+1	2.125E+1	2.006E+1	
83			^{241}Pu	1.464E+0	1.201E+0	1.068E+0	9.080E-1	7.263E-1	7.390E-1	8.841E-1	7.978E-1	
84			^{242}Pu	5.028E-1	5.841E-1	5.113E-1	4.091E-1	3.406E-1	3.577E-1	3.808E-1	3.213E-1	
85			^{237}Np	2.472E+0	3.073E+0	1.853E+0	1.697E+0	2.536E+0	1.065E+0	1.626E+0	1.587E+0	
86			^{241}Am, AEA	5.312E+0	5.750E+0	4.976E+0	4.156E+0	4.394E+0	4.317E+0	4.491E+0	4.281E+0	
87			^{241}Am, GEA	5.712E+0	6.207E+0	4.779E+0	4.051E+0	4.109E+0	4.896E+0	4.313E+0	4.051E+0	
88												
89	**Isotopic Conc., Activity Basis, µCi/g, as reported**		^{233}U, KPA	0.000E+0	0.000E+0	0.000E+0	0.000E+0	0.000E+0	0.000E+0	0.000E+0	0.000E+0	
90			^{234}U, KPA	4.749E-2	4.420E-2	3.673E-2	3.505E-2	2.260E-2	2.101E-2	2.662E-2	3.526E-2	
91			^{235}U, KPA	1.487E-3	1.634E-3	1.316E-3	1.159E-3	8.361E-4	7.327E-4	8.513E-4	8.090E-4	
92			^{236}U, KPA	4.609E-3	5.241E-3	4.058E-3	3.659E-3	2.540E-3	2.261E-3	2.561E-3	2.629E-3	
93			^{238}U, KPA	3.268E-2	3.602E-2	2.932E-2	2.571E-2	1.851E-2	1.628E-2	1.874E-2	1.771E-2	
94			^{238}Pu	2.274E+0	2.565E+0	2.298E+0	2.015E+0	1.591E+0	1.646E+0	1.853E+0	1.771E+0	
95			^{239}Pu	1.150E+1	1.235E+1	1.060E+1	9.475E+0	7.946E+0	8.085E+0	8.763E+0	8.084E+0	
96			^{240}Pu	6.336E+0	6.797E+0	5.988E+0	5.246E+0	4.320E+0	4.471E+0	4.821E+0	4.551E+0	
97			^{241}Pu	1.519E+2	1.246E+2	1.108E+2	9.423E+1	7.538E+1	7.669E+1	9.176E+1	8.280E+1	
98			^{242}Pu	1.989E-3	2.311E-3	2.023E-3	1.619E-3	1.348E-3	1.415E-3	1.506E-3	1.271E-3	
99			^{237}Np	1.739E-3	2.162E-3	1.303E-3	1.194E-3	1.784E-3	7.490E-4	1.143E-3	1.117E-3	
100			^{241}Am, AEA	1.823E+1	1.973E+1	1.708E+1	1.426E+1	1.508E+1	1.481E+1	1.541E+1	1.469E+1	
101			^{241}Am, GEA	1.960E+1	2.130E+1	1.640E+1	1.390E+1	1.410E+1	1.680E+1	1.480E+1	1.390E+1	
102												
103												

Filename: ASR 8798 Data Reduction Rev1.xlsx
Tab: TIMS Rad Calc.

K Basin Radionuclide Calculations

	A	B	C	D	E	F	G	H	I	J	K	L
104			Container	KW210	KW210	KW210	KW210	KW210	KW210	KW210	KW210	
105			Core	A1	A1	A4	A4	B1	B1	B3	B3	
106			Sample ID	TI001-A1-B	TI001-A1-C	TI002-A4-B	TI002-A4-C	TI002-B1-B	TI002-B1-C	TI001-B3-B	TI001-B3-C	
107			ASO ID	11-0419-129-S	11-0419-129-D	11-0420-129-S	11-0420-129-D	11-0421-129-S	11-0421-129-D	11-0422-129-S	11-0422-129-D	
108	Cooling Time,	By ^{241}Am AEA		31.6	36.2	35.7	35.4	40.3	39.6	37.2	38.2	
109	y	By ^{241}Am GEA		32.8	37.5	35.1	35.0	39.1	41.9	36.5	37.2	
110												
111	Title:	K Basin Radionuclide Calculations										
112	Purpose:	Convert U, Pu, Np, and Am analytical data										
113		to specific gravimetric and activity bases.										
114												
115	Date:	18 July 2011										
116	Prep'd. by:	SK Fiskum										
117	Rev'd. by:	CH Delegard										
118												
119	Signature:											
120												
121												
122												
123												
124		Cells for data entry										
125												
126	**Comments**											
127	Entry Date: 7/18/11											
128	Filename: AS ASR 8798 Data Reduction Rev1.xlsx											
129	Reporting units are in terms of µg/g dry mass basis.											
130	Microsoft Excel 2003 SP3											
131												
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23 July 2011

Isotopic Data				
Isotope	Atomic Mass	$t_{½}$, y	Spec. Act., Ci/g	λ, y^{-1}
^{233}U	233.0396352	159200	9.636E-03	4.354E-06
^{234}U	234.0409521	245500	6.222E-03	2.823E-06
^{235}U	235.0439299	703800000	2.161E-06	9.849E-10
^{236}U	236.045568	23420000	6.467E-05	2.960E-08
^{238}U	238.0507882	4468000000	3.361E-07	1.551E-10
^{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 8798 Data Reduction Rev1.xlsx
Date: TIMS Rad wet mass basis

K Basin Radionuclide Calculations

Wet settled solids mass basis

Conversion factor, g_{dry} / $g_{as\text{-}settled}$		0.447	0.447	0.567	0.567	0.328	0.328	0.363	0.363
	Container	KW210	KW210	KW210	KW210	KW210	KW210	KW210	KW210
	Core	A1	A1	A4	A4	B1	B1	B3	B3
	Sample ID	T1001-A1-B	T1001-A1-C	T1002-A4-B	T1002-A4-C	T1002-B1-B	T1002-B1-C	T1001-B3-B	T1001-B3-C
	ASQ ID	11-0419-129-S	11-0419-129-D	11-0420-129-S	11-0420-129-D	11-0421-129-S	11-0421-129-D	11-0422-129-S	11-0422-129-D
Isotopic Conc., Weight Basis, µg/g, wet settled solids	^{233}U, KPA	0.000E+0	0.000E+0	0.000E+0	0.000E+0	0.000E+0	0.000E+0	0.000E+0	0.000E+0
	^{234}U, KPA	3.412E+0	3.176E+0	3.345E+0	3.192E+0	1.192E+0	1.108E+0	1.551E+0	2.054E+0
	^{235}U, KPA	3.075E+2	3.381E+2	3.451E+2	3.040E+2	1.269E+2	1.112E+2	1.428E+2	1.357E+2
	^{236}U, KPA	3.186E+1	3.623E+1	3.556E+1	3.206E+1	1.288E+1	1.147E+1	1.436E+1	1.474E+1
	^{238}U, KPA	4.346E+4	4.790E+4	4.943E+4	4.335E+4	1.807E+4	1.589E+4	2.021E+4	1.910E+4
	Total Pu, ^{238}Pu basis	9.629E+1	1.033E+2	1.127E+2	1.005E+2	4.866E+1	4.962E+1	5.942E+1	5.496E+1
	Total Pu, ^{239,240}Pu basis	9.629E+1	1.033E+2	1.127E+2	1.005E+2	4.866E+1	4.962E+1	5.942E+1	5.496E+1
	^{238}Pu	5.936E-2	6.694E-2	7.606E-2	6.667E-2	3.049E-2	3.153E-2	3.923E-2	3.749E-2
	^{239}Pu	8.287E+1	8.903E+1	9.681E+1	8.656E+1	4.203E+1	4.276E+1	5.122E+1	4.725E+1
	^{240}Pu	1.248E+1	1.339E+1	1.496E+1	1.310E+1	6.247E+0	6.465E+0	7.703E+0	7.272E+0
	^{241}Pu	6.544E-1	5.366E-1	6.052E-1	5.146E-1	2.383E-1	2.425E-1	3.205E-1	2.892E-1
	^{242}Pu	2.247E-1	2.611E-1	2.898E-1	2.318E-1	1.118E-1	1.174E-1	1.380E-1	1.165E-1
	^{237}Np	1.105E+0	1.374E+0	1.050E+0	9.616E-1	8.321E-1	3.494E-1	5.893E-1	5.754E-1
	^{241}Am, AEA	2.374E+0	2.570E+0	2.820E+0	2.355E+0	1.442E+0	1.416E+0	1.628E+0	1.552E+0
	^{241}Am, GEA	2.553E+0	2.775E+0	2.708E+0	2.296E+0	1.348E+0	1.606E+0	1.564E+0	1.468E+0
Isotopic Conc., Activity Basis, µCi/g, wet settled solids	^{233}U, KPA	0.000E+0	0.000E+0	0.000E+0	0.000E+0	0.000E+0	0.000E+0	0.000E+0	0.000E+0
	^{234}U, KPA	2.123E-2	1.976E-2	2.081E-2	1.986E-2	7.416E-3	6.894E-3	9.651E-3	1.278E-2
	^{235}U, KPA	6.646E-4	7.306E-4	7.457E-4	6.570E-4	2.743E-4	2.404E-4	3.086E-4	2.933E-4
	^{236}U, KPA	2.060E-3	2.343E-3	2.300E-3	2.073E-3	8.332E-4	7.417E-4	9.284E-4	9.531E-4
	^{238}U, KPA	1.461E-2	1.610E-2	1.661E-2	1.457E-2	6.073E-3	5.340E-3	6.794E-3	6.419E-3
	^{238}Pu	1.017E+0	1.146E+0	1.302E+0	1.142E+0	5.221E-1	5.400E-1	6.718E-1	6.420E-1
	^{239}Pu	5.140E+0	5.522E+0	6.005E+0	5.369E+0	2.607E+0	2.652E+0	3.177E+0	2.931E+0
	^{240}Pu	2.832E+0	3.038E+0	3.393E+0	2.973E+0	1.417E+0	1.467E+0	1.748E+0	1.650E+0
	^{241}Pu	6.791E+1	5.569E+1	6.280E+1	5.340E+1	2.473E+1	2.516E+1	3.326E+1	3.002E+1
	^{242}Pu	8.892E-4	1.033E-3	1.146E-3	9.172E-4	4.421E-4	4.643E-4	5.461E-4	4.608E-4
	^{237}Np	7.774E-4	9.663E-4	7.386E-4	6.764E-4	5.853E-4	2.457E-4	4.145E-4	4.048E-4
	^{241}Am, AEA	8.147E+0	8.819E+0	9.676E+0	8.082E+0	4.946E+0	4.860E+0	5.586E+0	5.325E+0
	^{241}Am, GEA	8.761E+0	9.521E+0	9.294E+0	7.877E+0	4.626E+0	5.512E+0	5.365E+0	5.039E+0

*Mass conversion factors were obtained from Physical Property calculation spreadsheets supporting each core processing.

	KW210	KW210	KW210	KW210	KW210	KW210	KW210	KW210
	A1	A1	A4	A4	B1	B1	B3	B3
	T1001-A1-B	T1001-A1-C	T1002-A4-B	T1002-A4-C	T1002-B1-B	T1002-B1-C	T1001-B3-B	T1001-B3-C
	11-0419-129-S	11-0419-129-D	11-0420-129-S	11-0420-129-D	11-0421-129-S	11-0421-129-D	11-0422-129-S	11-0422-129-D
ug/g	RPD	RPD	RPD	RPD	RPD	RPD	RPD	RPD
^{233}U, KPA	NA	NA	NA	NA	NA	NA	NA	NA
^{234}U, KPA	7.2%	4.7%	7.3%	**28%**
^{235}U, KPA	9.5%	13%	13%	5.1%
^{236}U, KPA	13%	10%	12%	2.6%
^{238}U, KPA	9.7%	13%	13%	5.7%
Normalized µCi/g or µg/g				
^{238}Pu	12%	13%	3.4%	4.5%
^{239}Pu	7.2%	11%	1.7%	8.1%
^{240}Pu	7.0%	13%	3.4%	5.8%
^{241}Pu	20%	16%	1.7%	10%
^{242}Pu	15%	**22%**	4.9%	17%
Atom%				
^{233}U, TIMS	NA	NA	NA	NA
^{234}U, TIMS	17%	8.4%	5.6%	**33%**
^{235}U, TIMS	0.26%	0.43%	0.33%	0.58%
^{236}U, TIMS	3.1%	2.7%	1.2%	8.3%
^{238}U, TIMS	0.0007%	0.0057%	0.0011%	0.0135%
Normalized Atom%				
^{238}Pu TIMS	5.0%	1.7%	1.4%	3.3%
^{239}Pu TIMS	0.16%	0.32%	0.23%	0.27%
^{240}Pu TIMS	0.013%	1.7%	1.5%	2.0%
^{241}Pu TIMS	**27%**	4.7%	0.2%	2.5%
^{242}Pu TIMS	8.0%	11%	2.9%	9.2%

RPDs show slight variation from the TIMS final report because these spreadsheet results were based on untruncated values (rounding at the end).

(Note that the lower the Pu isotope RPD value, the more it will be affected relative to that reported by TIMS because of the effect of ^{238}Pu renormalization and it's associated variation.)

Gray shading indicates the RPD exceeded ±20% criterion defined by the QAPjP/SAP.

Appendix F

X-Ray Diffraction (XRD) Analysis

Appendix F

X-Ray Diffraction (XRD) Analysis

Appendix F contains the X-ray diffraction (XRD) reports. The XRD analyses were conducted on the size-fractionated samples from SCS-CON-210 and the undissolved solids generated from the acid digestions resulting from the completion of Analytical Service Requests (ASR) 8798 (core samples) and 8824 (size-fractionated container composite samples). Both reports reference appendices, which are self-contained within the XRD reports.

The following table of contents shows that pagination specific to Appendix F was applied to the reports (including self-contained appendices), document review records, instrument log sheets, and surveillance reports (generated by the PNNL Quality Engineers). This pagination is sequential and consistent (e.g., page F.#).

Appendix F Table of Contents

XRD Report Supporting Analysis of KW210 Sieve Fractions F.3
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X-Ray Diffraction Analysis Report

Battelle—Pacific Northwest National Laboratory
Radiochemical Sciences and Engineering Group
PO Box 999, Richland, Washington 99352

Project: #52578
Sample ID: KW 210 Container Composites

Procedure: PNNL-RPG-268 Rev. 4, "Solids Analysis by X-Ray Diffraction"

Analyst: Doinita Neiner
Analysis Dates: 05/25/2011
Instrument: RGD # 105
Property Number: WD 59716
Serial Number: GD 3827N

DNeiner 7/20/11
Prepared By Doinita Neiner /Date

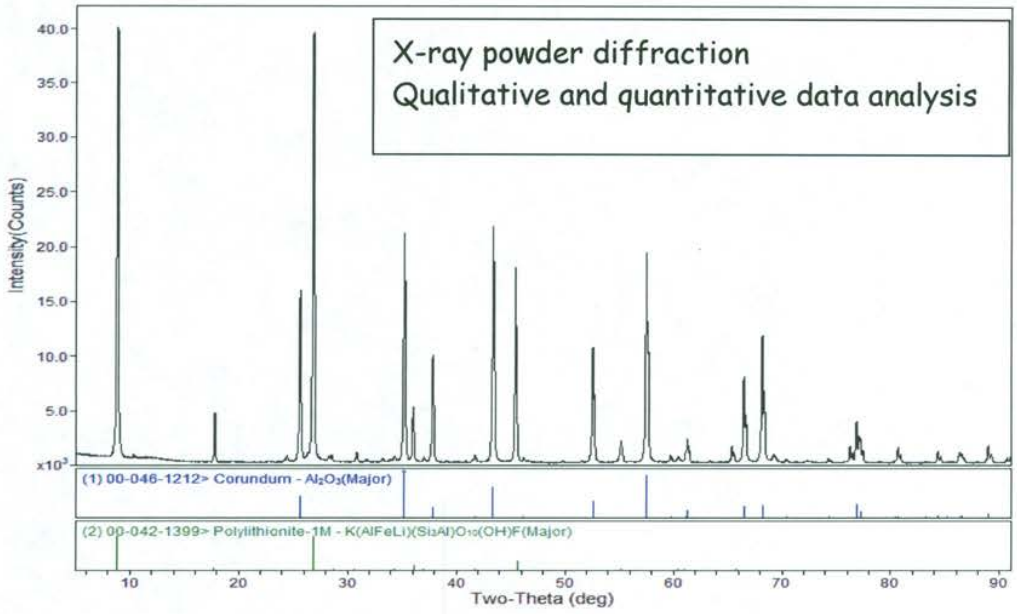
Michael W Urie 7/20/11
Reviewed By Mike Urie /Date

Deborah Middleton 7/20/11
QE Reviewed By Deborah Coffey/Date DSC 7/20/11

7/21/11
Project Management Release Clark Carlson/Date

Battelle - Pacific Northwest National Laboratory *Radiochemical Sciences and Engineering Group*

X-Ray Diffraction Data Analysis



Project Number: 52578
Charge Code: H41223
Client: S. Fiskum
Total Samples: 4 solids (See results for Client IDs)

Analysis Procedure	PNNL-RPG-268 Rev. 4, "Solids Analysis by X-Ray Diffraction"
Prep Procedure	Slurry of amyl acetate and Krylon on a glass slide covered with Kapton
Analyst	Doinita Neiner
Analysis Date	May 25, 2011
Sample Prep. Date	May 19, 2011 through May 24, 2011
Reference Standards	Rutile – SRM 674- PDF 01-077-0440
Software **Reference database**	Jade MDI 9 ICDD PDF -2 2009
M&TE Numbers	XRD System (WD 59716), RPL/317

Battelle - Pacific Northwest National Laboratory *Radiochemical Sciences and Engineering Group*

X-Ray Diffraction Data Analysis

XRD Analysis Request Form

Requester	SK Fiskum	**Work Package**	H41223
Signature	S. K. Fiskum	**Project Number**	52578
		Subtask	XRD
Date Submitted:	4/6/11	**Date Required:**	5/6/11

Treatability Study? ☑ no ☐ yes; if yes, start date:

Sample ID	Sample Description	Internal Standard Request
TI008-MH	-2000/+500 µm, air-dried solids	☐ Rutile ☐ Alumina ☑ None
TI008-MJ	-2000/+500 µm, air-dried solids duplicate	☐ Rutile ☐ Alumina ☑ None
TI008-SH	-500 µm wet slurry	☐ Rutile ☐ Alumina ☑ None
TI008-SJ	-500 µm wet slurry duplicate	☑ Rutile ☐ Alumina ☑ None

Comments: All samples are from the KW210 container composite that had been wet-sieved into three size fractions. Only samples from two fractions are provided herein. A large quantity of resin beads have been observed in the -2000/+500 µm fraction. The -500 µm sample fractions are wet slurries that will require air drying.

Identify crystalline structures in room-temperature-dried sludge. Identify hydrate compounds plus any other identifiable compounds. This includes noting the presence (or absence) of aluminum hydrates, uranium hydrides, uranium oxides as well as zirconium metal. The analytical composition (ICP-OES analytes) will be provided upon availability. Uranium is expected to be a dominant component.

All samples are to be analyzed without internal standard; one sample (selected choice above can be altered depending on quantity of sample available) are to also be run with rutile (TiO_2). Rutile X-ray diffraction angles and relative peak magnitudes will be used to help demonstrate acceptable system performance within the sample matrix and analysis process.

Please use the new Rigaku system for powder diffraction analysis if at all possible. Please use a bench sheet you develop to support sample processing and analysis and provide this original bench sheet as part of the data package.

Notes for Pattern Analyst:

XRD Mount preparation

Standard Reference Materials ID's: TiO_2: SRM 674 Al_2O_3: SRM676 Other: _____

Fixative: ☐ Collodion ☒ Krylon ☐ Other _____

Mount: ☒ Glass Slide, ☐ Kapton covered ☐ PIP Holder ☐ Other _____

Prepared by: D Meiner (Signature) 5/24/11 (Date)

XRD Operating Parameters	X-RAY 2-theta range: 5 – 65 degrees (5-65 default)
	Step size: ____ degrees (0.02 default)
	Count time: ___ second(s) (2 default) ☐ Multi-Scans

(Shaded areas to be filled out by the XRD analysis staff.)

Battelle - Pacific Northwest National Laboratory *Radiochemical Sciences and Engineering Group*

X-Ray Diffraction Data Analysis

XRD Analysis Bench Sheet

For Procedure PNNL-RPG-268, Rev. 4,

Solids Analysis by X-Ray Diffraction

Sample Preparation

Print and complete pages as needed in order to document sample preparation for all samples submitted under one Analysis Request.

Sample ID(s) T1008-MH

Sample preparation date (grinding and mounting) 5/19/11

Sample dried air-dried ☐

Internal standard: None ☒ Rutile ☐ Alumina ☐ Other ☐ describe _____

Standard ID: _____

Grinding method: Mortar & Pestle ☒ Mill ☐ Other ☐ describe _____

Fixative: ☒ Krylon ☐ Other

Mount type: Slide ☐ Other ☐ describe _____

Notes: _____

XRD Mount ID(s) T1008-MH

Sample ID(s) T1008-MY

Sample preparation date (grinding and mounting) 5/19/11

Sample dried air-dried ☐

Internal standard: None ☒ Rutile ☐ Alumina ☐ Other ☐ describe _____

Standard ID: _____

Grinding method: Mortar & Pestle ☒ Mill ☐ Other ☐ describe _____

Fixative: ☒ Krylon ☐ Other

Mount type: Slide ☒ Other ☐ describe _____

Notes: _____

XRD Mount ID(s) T1008-MY

Sample ID(s) T1008-SH

Sample preparation date (grinding and mounting) 5/24/11

Sample dried air-dried ☒

Internal standard: None ☒ Rutile ☐ Alumina ☐ Other ☐ describe _____

Standard ID: _____

Grinding method: Mortar & Pestle ☒ Mill ☐ Other ☐ describe _____

Fixative: ☒ Krylon ☐ Other

Mount type: Slide ☒ Other ☐ describe _____

Notes: _____

XRD Mount ID(s) T1008-SH

D Deiver 5/25/11
Analyst Signature/Date

Michael Lin 7/8/11
Reviewer Signature/Date

Battelle - Pacific Northwest National Laboratory *Radiochemical Sciences and Engineering Group*

X-Ray Diffraction Data Analysis

XRD Analysis Bench Sheet

For Procedure PNNL-RPG-268, Rev. 4,
Solids Analysis by X-Ray Diffraction
Sample Preparation

Print and complete pages as needed in order to document sample preparation for all samples submitted under one Analysis Request.

Sample ID(s) T1008-SJ
Sample preparation date (grinding and mounting) 5/24/11
Sample dried air-dried ☒
Internal standard: None ☒ Rutile ☐ Alumina ☐ Other ☐ describe _____
Standard ID: _____
Grinding method: Mortar & Pestle ☒ Mill ☐ Other ☐ describe _____
Fixative: ☒ Krylon ☐ Other
Mount type: Slide ☒ Other ☐ describe _____
Notes: _____
XRD Mount ID(s) T1008-SJ

Sample ID(s) T1008-SJ-
Sample preparation date (grinding and mounting) 5/27/11
Sample dried air-dried ☒
Internal standard: None ☐ Rutile ☒ Alumina ☐ Other ☐ describe _____
Standard ID: TiO_2
Grinding method: Mortar & Pestle ☒ Mill ☐ Other ☐ describe _____
Fixative: ☒ Krylon ☐ Other
Mount type: Slide ☒ Other ☐ describe _____
Notes: _____
XRD Mount ID(s) T1008-SJ-TiO_2

Sample ID(s) _____
Sample preparation date (grinding and mounting) _____
Sample dried air-dried ☐
Internal standard: None ☐ Rutile ☐ Alumina ☐ Other ☐ describe _____
Standard ID: _____
Grinding method: Mortar & Pestle ☐ Mill ☐ Other ☐ describe _____
Fixative: ☐ Krylon ☐ Other
Mount type: Slide ☐ Other ☐ describe _____
Notes: _____
XRD Mount ID(s) _____

DKeiser 5/25/11
Analyst Signature/Date

Michael Thm 7/8/11
Reviewer Signature/Date

Battelle - Pacific Northwest National Laboratory *Radiochemical Sciences and Engineering Group*

X-Ray Diffraction Data Analysis

XRD Analysis Bench Sheet

For Procedure PNNL-RPG-268, Rev. 4,
Solids Analysis by X-Ray Diffraction
Diffraction Measurements

Print and complete pages as needed in order to document sample analysis and daily calibration verification for all samples submitted under one Analysis Request.

Today's date: 05/25/11
XRD daily performance checks with NIST standard run today? ☒ NIST standard: SRM640a Si
Associated daily calibration check filename 052511a_Si
Logbook updated? ☒

XRD mount ID	XRD filename	Notes	Analyst initials
T1008-MH	T1008-MH-052511b		DN
T1008-MJ	T1008-MJ-052511C		DN
T1008-SH	T1008-SH-052511D		DN
T1008-SJ	T1008-SJ-052511E		DN
T1008-SJ TiO2	T1008-SJ-TiO2-052511F		DN

XRD Operating Parameters	
	X-RAY 2-theta range: 5 – 65 degrees (5-65 default)
	Step size: 0.02 degrees (0.02 default)
	Other XRD scan parameters: 2 deg/min, DTEX, 40 kV, 44 mA

Today's date: _____
XRD daily performance checks with NIST standard run today? ☐ NIST standard: _____
Associated daily calibration check filename _____
Logbook updated? ☐

XRD mount ID	XRD filename	Notes	Analyst initials

XRD Operating Parameters	
	X-RAY 2-theta range: ___ – ___ degrees (5-65 default)
	Step size: ____ degrees (0.02 default)
	Other XRD scan parameters:

DNeiner 5/25/11
Analyst Signature/Date

7/3/11
Reviewer Signature/Date

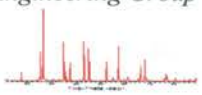


Table 1. Summary of Phases Identified in KW210 Container Composites

Sample ID RPL ID/(Client ID)	ICDD Card Number	Relative Intensity[a]	Formula	Common Phase Name
TI008-MH	01-070-3755	Major	SiO_2	Quartz
	--	Unassigned	27.3°	
TI008-MJ	01-070-3755	Major	SiO_2	Quartz
	--	Unassigned	33°, 35°-36°[b]	
TI008-SH	01-070-3755	Major	SiO_2	Quartz
	01-072-4553	Major	UO_2	Uraninite
	01-075-0944	Major	U_4O_9	Uranium oxide
	--	Unassigned	23.6°, 34.5°, 35.6°	
TI008-SJ	01-070-3755	Major	SiO_2	Quartz
	01-072-4553	Major	UO_2	Uraninite
	01-075-0944	Major	U_4O_9	Uranium oxide
	--	Unassigned	24°, 35°,35.8°, 37°, 38°, 51.5°	

(a) Crystalline components above 10 wt.% are classified as Major phases, between 1-10 wt.% as Minor phases, and under 1 wt.% as Possible phases.
(b) A broad peak was present between 35° to 36° 2-theta.

1. Sample preparation

X-ray powder diffraction (XRD) data from the samples identified in Table 2 have been examined for identification of crystalline phases present. The samples were KW210 container composite that was wet sieved into three size fractions prior to submitting for XRD analysis. Only two sieve fractions were submitted for analysis. The samples TI008-SH and TI008-SJ were air dried prior to XRD slide preparation. The samples TI008-MH and TI008-MJ were used as received.

All the samples were processed and analyzed according to procedure, Solids Analysis by X-Ray Diffraction, PNNL-RPG-268, Rev. 4. A sample was mixed with approximately 2 mL of amyl acetate in a 20-mL glass scintillation vial to produce a slurry. A small aliquot of the slurry was transferred into a mortar and ground with a pestle (all tungsten carbide components). After grinding, a small fraction of the sample was pipetted onto a Krylon-covered glass slide and allowed to dry. The dried samples were then covered with Kapton. One client sample, TI008-SJ was run in duplicate (one without internal standard and one with 3-4 mg TiO_2, rutile as internal standard). Titania (rutile structure) internal standard was used, SRM 674; the standard certificate is provided in Appendix 1.

Table 2. Sample Identification

Sample ID	Sample Description	XRD Sample ID	Internal Standard
TI008-MH	KW210 (500-2000 µm)	TI008-MH	None
TI008-MJ	KW210 (500-2000 µm)	TI008-MJ	None
TI008-SH	KW210 (< 500 µm)	TI008-SH	None
TI008-SJ	KW210 (< 500 µm)	TI008-SJ	None
		TI008-SJ-TiO_2	TiO_2

2. X-ray powder diffraction data collection

X-ray powder diffraction analyses were performed on a Rigaku Ultima IV X-ray Diffractometer (Cu Kα radiation, λ = 1.5418 Å). Data were collected in a step scan mode between 5 and 65° 2-theta with a step size of 0.02°, a speed of 2°/minute, and using a 2D detector DTEX. The X-ray tube operating conditions were 40 kV and 44 mA.

The daily performance check associated with the XRD sample files is presented in Appendix 2. This performance check meets the performance criteria (i.e., 111 Si 2-theta = 28.44°±0.06°) specified in the XRD procedure PNNL-RPG-268 Rev. 4. The NIST certificate for the standard used for the daily performance check (SRM 640a) is presented in Appendix 3.

The quality control/calibration of the XRD instrument results are provided in Appendix 4 and the standard certificate, SRM 1976 is presented in Appendix 5. The requirement for the semiannual calibration of the diffractometer (as stated in the PNNL-RPG-268 Rev. 4, is that observed d-interplanar distance for any given crystal lattice Miller indices be within ±0.02 Å (Del-d value – difference between the calculated and the observed interplanar distance) of the d-values from the ICDD (International Centre for Diffraction Data) card for alumina (corundum structure). For this quality control semiannual calibration, all Del-d values are well within the aforementioned criterion (±0.02 Å Del-d value).

3. X-ray powder diffraction data analysis

3.1. Database Search Details

Phase identification was conducted using JADE search match routines. The search was done including the following elements: C, Na, K, Mg, Ca, Al, Sr, Si, O, H, Cd, Cr, Ba, Cu, Ni, Pb, Mn, Fe, Zn, Zr, Ti and U. All diffraction patterns have been compared against three ICDD PDF-2 databases: RDB-ICSD, Inorganics, and Minerals. All of the XRD analyses included the search for uranium oxides, oxyhydroxides, hydrates and hydroxides; aluminum oxides, oxyhydroxides and hydrates; and zirconium metal and zirconia (from cladding materials). The PDF (Powder Diffraction File) cards of the crystalline phases identified in the samples analyzed in this report are given in Appendix 6.

3.2. Rietveld Refinement Details

The Rietveld method effectively separates overlapping data, thereby allowing an accurate determination of the structure/composition. Rietveld refinement on powder materials is a widely used application in determining the components of chemical mixtures. The refinement takes into account the PDF cards of the identified phases, and compares those with the calculated peaks from the observed powder pattern. The refinement generates a difference plot between an observed and a calculated powder pattern. In addition, because it calculates a powder pattern from the PDF cards of the phases taken into consideration it gives a goodness of fit for that particular phase, which is a reflection of how good the fit is between the observed and the calculated powder patterns. The refined parameters are the unit cell parameters, background, and

preferred orientation. The refinement helps decide which one of the examined phases is present in the observed powder pattern, by comparing all peak positions and intensities with calculated values (from the PDF card). The quality of the refinement can be judged based on the R factors (e.g., R_w) and the difference map (i.e., the difference between an observed and a calculated powder pattern). Typically, the better the Rietveld fit the lower the R_w factor; R_w <20% suggests a good fit, R_w <10% suggests an excellent fit. R_w values <20% have to be supported by a small difference between the calculated and observed powder patterns (i.e., minor deviations within the difference map).

4. Summary of findings

The samples represent >500/<2000 micron particle size (medium fraction) samples TI008-MH and TI008-MJ, and <500 micron particle size (small fraction) samples, TI008-SH and TI008-SJ. The medium fraction is primarily composed of quartz. The small fraction contains uraninite in addition to quartz. In addition some reflections present in the powder diffraction data were unidentified. The subsamples do not contain uranium oxyhydroxides, hydrates and hydroxides; aluminum oxides, oxyhydroxides and hydrates, or zirconium metal and zirconia (from cladding materials) as crystalline phases. A summary of phases is presented in Table 1.

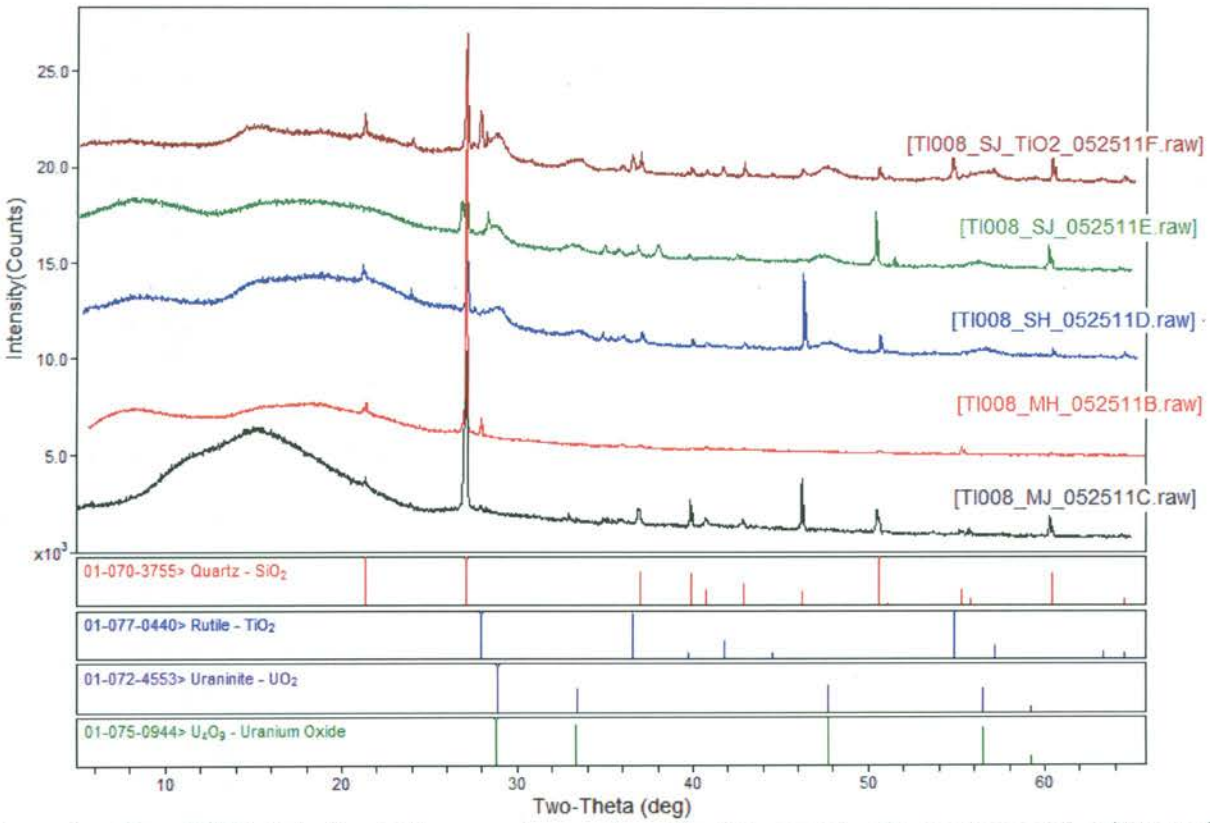


Figure 1. Raw XRD data for all the samples analyzed in this report. No background subtracted.

The following table summarizes the figures present in this report.

Table 3. Summary of Figures

Client Sample ID	Sample Description	Figures
TI008-MH	KW210 (500-2000 µm)	2
TI008-MJ	KW210 (500-2000 µm)	3
TI008-SH	KW210 (< 500 µm)	4
TI008-SJ	KW210 (< 500 µm)	5, 6

5. XRD Sample Analysis

5.1. XRD Samples TI008-MH

Figure 2 presents the raw XRD data for sample TI008-MH along with the ICDD cards of the crystalline phases identified based on the Rietveld refinement. This sample is composed of silica (quartz). There is an unidentified peak at 27.3° 2 theta and a broad peak from 5° to 12° 2-theta. The broad peak may be attributable to resin beads which could be present as an amorphous low-angle hump. The amorphous hump contributes to the high R_w = 13% seen in the Rietveld refinement presented in Figure 2.

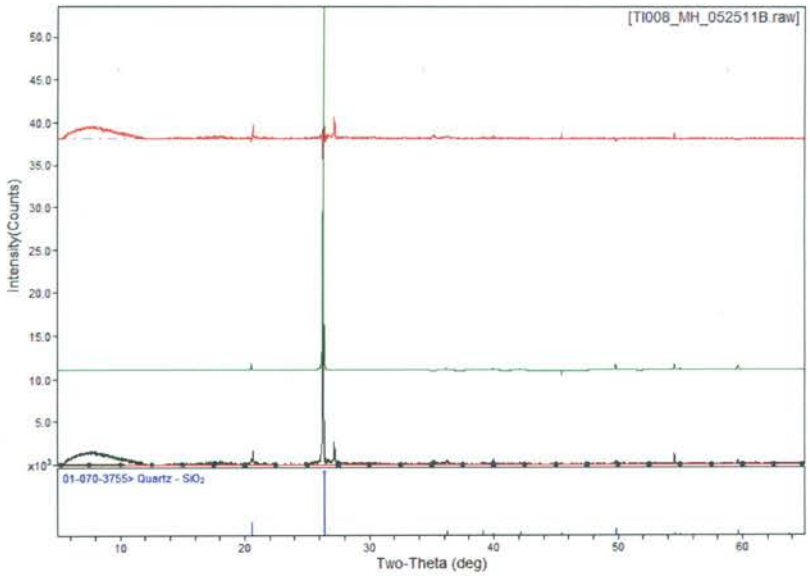


Figure 2. Rietveld refinement of the XRD data for TI008-MH. The black line is the observed powder pattern, the green line is the simulated powder pattern based on the model, and the red line is the difference between the calculated and the observed powder patterns. R_w=13% (See Section 3.2). Background subtracted spectra shown.

5.2. XRD Sample TI008-MJ

Figure 3 presents the raw XRD data for samples TI008-MJ along with the ICDD card of the crystalline phase identified based on the Rietveld refinement (R_w= 5 %), silica (quartz). In addition the peaks at 33° and a small broad peak between 35°- 36° 2-theta remain unidentified.

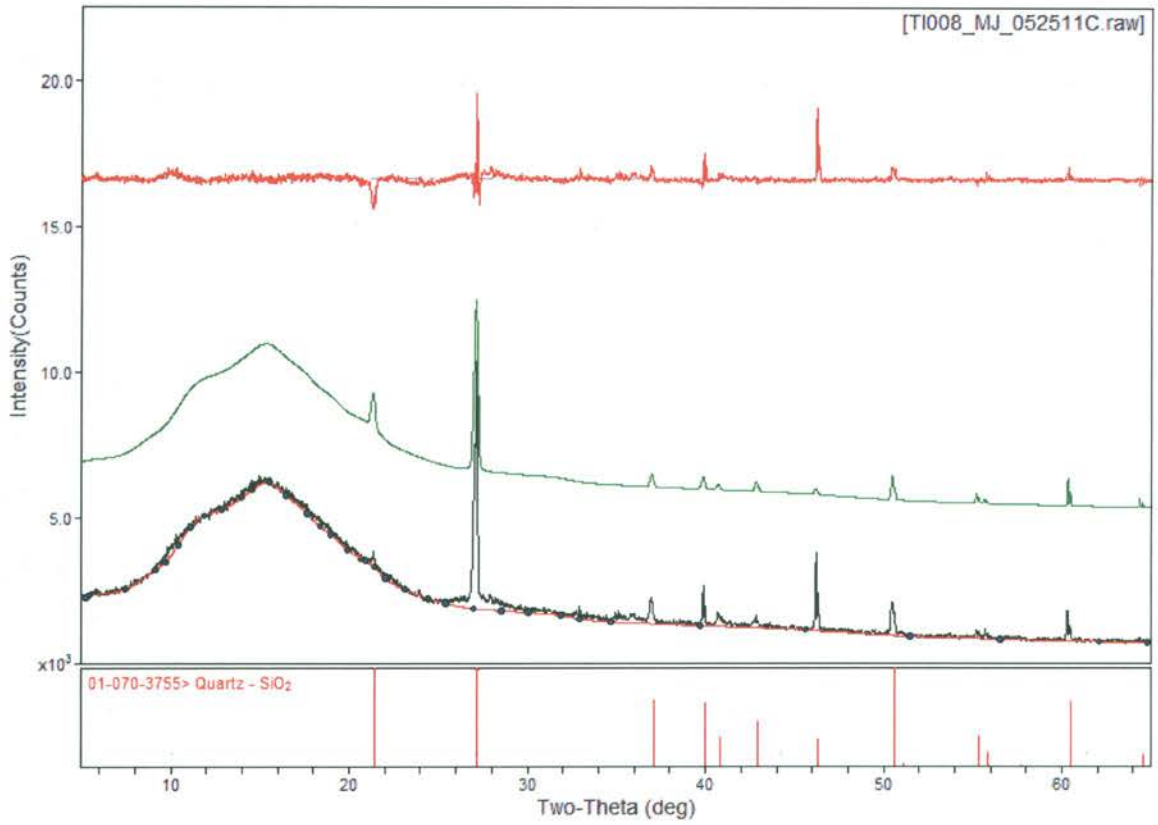


Figure 3. Rietveld refinement of the XRD data for TI008-MJ. The black line is the observed powder pattern, the green line is the simulated powder pattern based on the model, and the red line is the difference between the calculated and the observed powder patterns. R_w= 5 % (See Section 3.2). Background subtracted spectra shown.

5.3. XRD Sample TI008-SH

Figure 4 presents the Rietveld refinement (R_w= 5 %) of the raw XRD data for sample TI008-SH with the ICDD cards of the crystalline phases identified in this material. Based on this analysis the sample contains silica (quartz) and uranium oxides (UO_2 and U_4O_9) as major phases. In addition the peaks at 23.6°, 34.5° and 35.6° 2-theta, remain unidentified. The relatively big difference between the calculated and the observed diffraction patterns is due to preferred orientation of the quartz crystals.

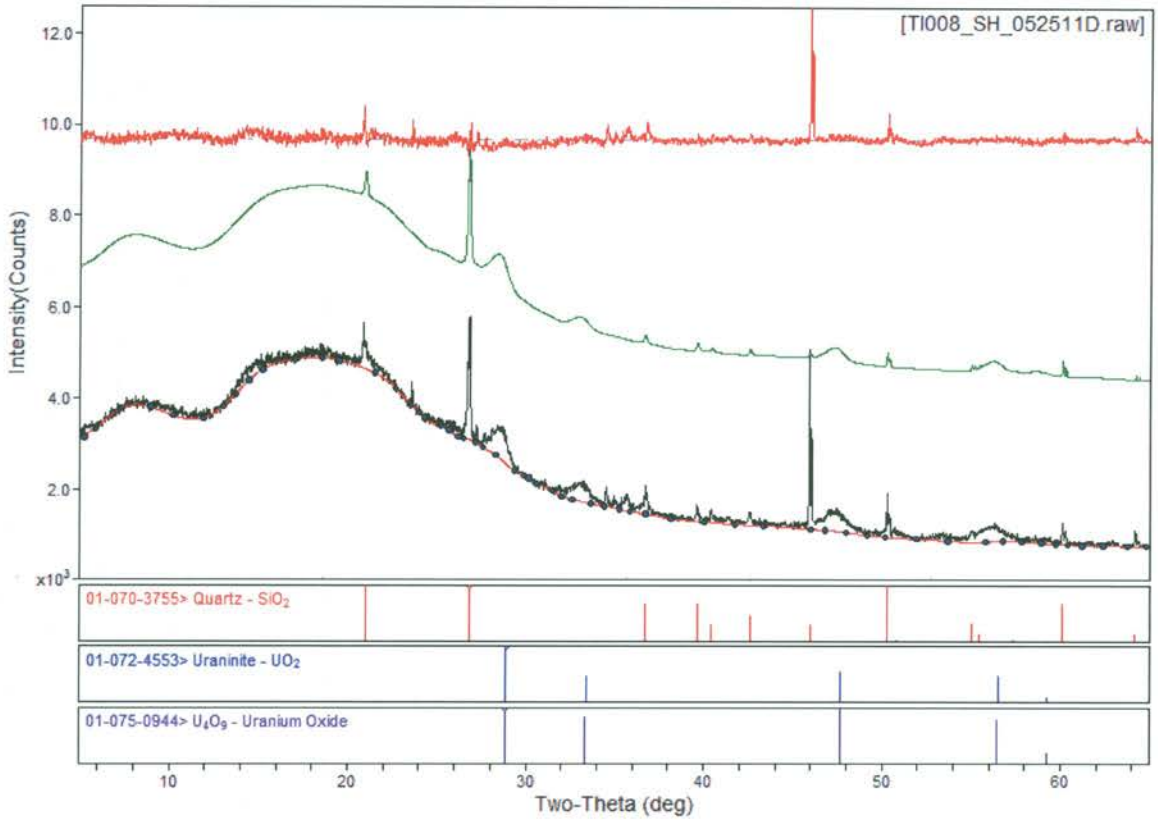


Figure 4. Rietveld refinement of the XRD data for TI008-SH. The black line is the observed powder pattern, the green line is the simulated powder pattern based on the model, and the red line is the difference between the calculated and the observed powder patterns. R_w= 5 % (See Section 3.2). No background subtracted spectra shown.

5.4. XRD Samples TI008-SJ and TI008-SJ-TiO_2

Figure 5 presents the Rietveld refinement (R_w= 8 %) of the XRD data for sample TI008-SJ with the ICDD cards of the corresponding crystalline phases. Figure 6 presents the Rietveld refinement (R_w= 5 %) data for TI008-SJ-TiO_2 with the ICDD cards of the corresponding crystalline phases. The major phases present in these samples are silica (quartz) and uranium oxides (UO_2 and U_4O_9). In addition sample TI008-SJ-TiO_2 contains TiO_2 (rutile) that was added as an internal standard. Rutile internal standard peaks, SRM 674, are consistent with the ones reported in the ICDD 01-077-0440, which means that the peak positions in the analyzed samples are reliable. In addition sample TI008-SJ has unidentified peaks at 24, 35°, 35.8°, 37°, 38° and 51.5° 2-theta. Although not as intense, these unidentified peaks are also present in TI008-SJ-TiO_2.

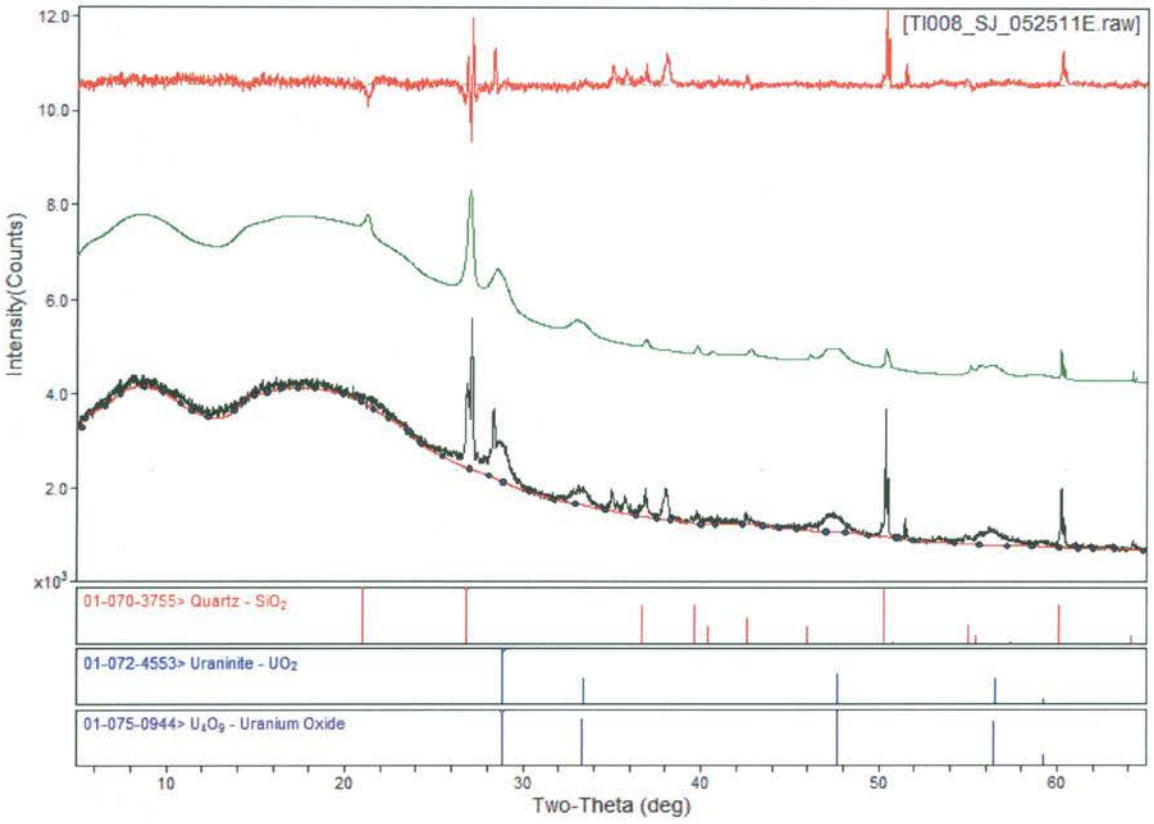


Figure 5. Rietveld refinement of the XRD data for TI008-SJ. The black line is the observed powder pattern, the green line is the simulated powder pattern based on the model, and the red line is the difference between the calculated and the observed powder patterns. R_w= 8 % (See Section 3.2). No background subtracted spectra shown.

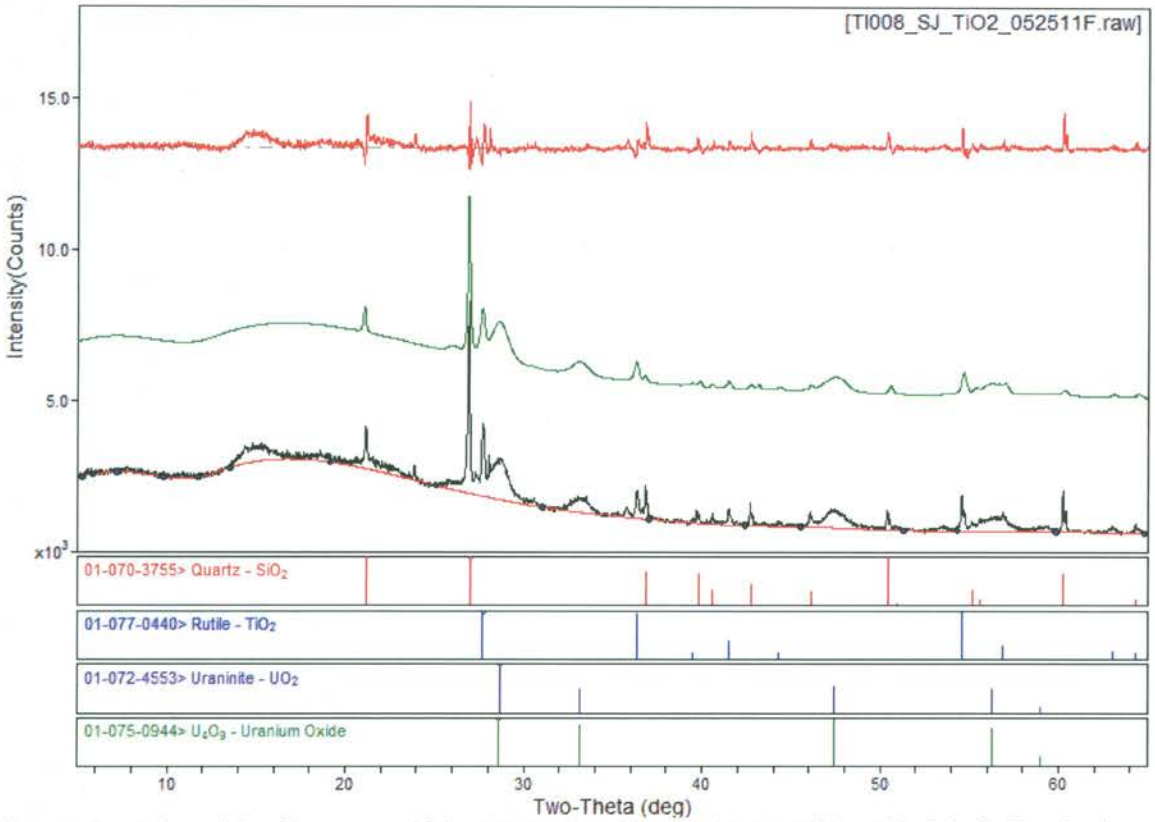
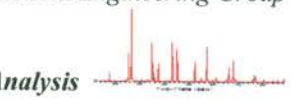


Figure 6. Rietveld refinement of the XRD data for TI008-SJ-TiO_2. The black line is the observed powder pattern, the green line is the simulated powder pattern based on the model, and the red line is the difference between the calculated and the observed powder patterns. R_w= 5 % (See Section 3.2). No background subtracted spectra shown.



Appendix 1
SRM 674 NIST Certificate

National Bureau of Standards

Certificate

Standard Reference Material 674

X-Ray Powder Diffraction Intensity Set

C. R. Hubbard

This Standard Reference Material (SRM) consists of five different phases. The phases, separately bottled, are α-Al_2O_3 (corundum structure), ZnO (wurtzite structure), TiO_2 (rutile structure), Cr_2O_3 (corundum structure), and CeO_2 (fluorite structure). These phases can be used as internal standards for quantitative analysis and as external standards for checking the intensity response of x-ray diffraction instruments. The five phases cover the range of linear absorption coefficient from 100 to 1000 cm^{-1} for CuKα radiation. The intensity values are for a constant diffracting volume (fixed divergent slit and infinitely thick mounting or equivalent).

The certified relative intensity values (CuKα radiation), for the major diffraction lines below 70° 2(θ), for each phase are given in Table 1. The certified reference intensity ratios (I/Ic) for ZnO, TiO_2, Cr_2O_3, and CeO_2, relative to α-Al_2O_3 and the lattice parameters are given in Tables 2 and 3. The uncertainties of the relative intensity and lattice parameter values are the standard deviations derived from multiple measurements. The uncertainties of the reference intensity ratio values were determined from the value of the internal inconsistency derived from all possible binary mixtures.

The average crystallite size for each of the five phases is near 2.0 μm. Crystallite size broadening of the diffraction peaks is small but present. This is particularly apparent for α-Al_2O_3, which has a nominal crystallite size less than 1.0 μm. The lattice parameters, uncorrected for refraction, were determined with a relative uncertainty of about three parts in 10^5 using an internal standard. This uncertainty in the lattice parameter must be increased by a factor of two or three if one wishes to determine absolute d-spacings as the uncertainties due to the internal standard 2θ's and to thermal expansion would need to be added.

The technical and support aspects concerning the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by R.K. Kirby.

Washington, D.C. 20234
June 1, 1983

(over)

Stanley D. Rasberry, Chief
Office of Standard Reference Materials

Table 1. Relative Intensitites of Major Lines

CuKα Radiation (μ = 124.1 cm^{-1})

hkl	2θ	I^{rel}	hkl	2θ	I^{rel}	
α-Al_2O_3[a]	(Corundum Structure)		TiO_2	(cont.)		
012	25.58°	55.4 ± 2.4	111	41.20°	21.4 ± 1.4	
104	35.13	87.4 ± 1.9	211	54.27	56.9 ± 2.8	
110	37.78	36.5 ± 1.4	220	56.51	16.9 ± 1.4	
113	43.36	100.0	002	62.76	8.2 ± 0.7	
024	52.55	45.5 ± 1.3	301	68.93	(21.1)	approximate due to
116	57.52	92.5 ± 2.6	112	69.79	(11.1)	partial overlap
214	66.54	34.7 ± 1.0				
300	68.19	55.5 ± 2.2				
ZnO	(Wurtzite Structure)		Cr_2O_3[c]	(Corundum Structure)		
100	31.75°	57.6 ± 1.1	012	24.52°	69.2 ± 1.6	
002	34.44	40.2 ± 1.4	104	33.59	100.0	
101	36.25	100.0	110	36.20	87.1 ± 2.3	
102	47.54	22.8 ± 0.5	113	41.47	32.3 ± 1.0	
110	56.55	34.4 ± 0.8	024	50.21	39.4 ± 0.8	
103	62.87	31.0 ± 0.9	116	54.81	94.5 ± 2.2	
200	66.40	4.7 ± 0.2	214	63.43	30.8 ± 1.3	
112	67.91	25.9 ± 0.6	300	65.09	40.6 ± 1.0	
TiO_2[b]	(Rutile Structure)		CeO_2[d]	(Fluorite Structure)		
110	27.42°	100.0	111	28.55°	100.0	
101	36.04	44.0 ± 1.7	200	33.08	28.0 ± 0.8	
			220	47.49	53.5 ± 2.0	
			311	57.32	43.4 ± 2.3	

[a] Two broad, very weak peaks (32.80 and 45.5° 2θ) can be detected. These lines are possibly due to γ-Al_2O_3 (PDF 29-63).

[b] Anatase was observed at ~1.0 wt. % level. Unexplained very weak peaks at 13.9 and 18.0° 2θ were also detected.

[c] Small unexplained intensity deviations from smooth background.

[d] Three unexplained, broad, very weak diffraction peaks were observed at 14.40, 16.64, and 23.75° 2θ.

Appendix 2
Daily Performance Checks

The daily performance check shows that the Rigaku Ultima IV diffractometer is within calibration per PNNL-RPG-268 Rev. 4, "Solids Analysis by X-Ray Diffraction" so no adjustments have been made. Figure 7 presents the XRD data obtained on the silicon SRM 640a standard. Table 4 contains summarized results of the daily performance check with this standard. The columns in Table 4 have the following significance:

1) 2T(r) gives the 2-theta of the reference powder pattern (ICDD card),
2) 2T(o) is the observed 2-theta,
3) Delta is the difference between the observed and the reference 2-theta,
4) d(r) is the interplanar distance d for the reference pattern (in Å),
5) d(o) is the observed d,
6) Del-d is the difference between the observed and the reference d,
7) I % is the intensity in percent (the most intense peak is always 100% and all other peaks are normalized).

Per procedure the measured 2-theta of the performance check standard (i.e., the 111 Si reference line) must be 28.44°±0.06° 2-theta; the observed 2-theta difference was 0.012 and met this requirement. Thus the performance check conducted on May 25, 2011 met this criterion.

Daily calibration report (27 to 29 2-theta) – Date 05-25-2011

Theta Calibration Report
[052511a_si.raw] si 640a - Full Chart
Reference Lines = 01-073-6978> Silicon - Si
Two-Theta Range of Fit = 27.0/29.0(deg)
Zero Offset = -0.012(deg)
ESD of Fit = 0.0°, |Delta 2-Theta| = 0.012(deg)

Table 4. Calibration Report Summary

2T(r)	2T(o)	Delta	d(r)	d(o)	Del-d	I%
28.447	28.459	0.012	3.1350	3.1337	0.0013	100.0

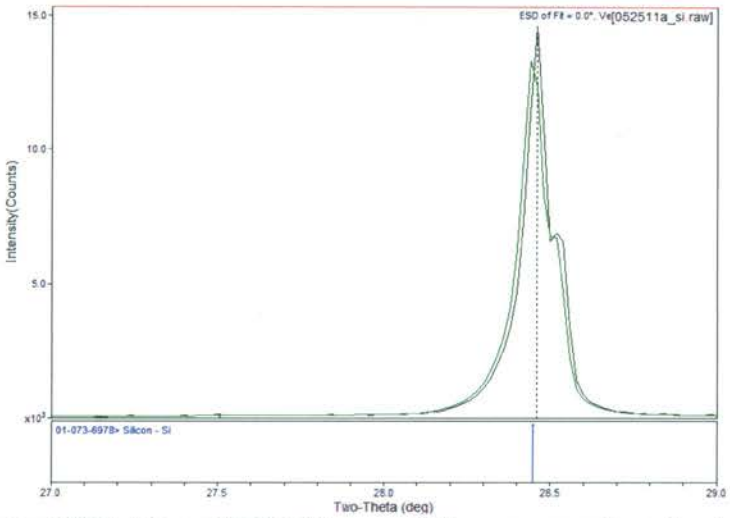


Figure 7. The raw XRD data on SRM 640a against the corresponding structure ICDD card.

Appendix 3
SRM 640a NIST Certificate

National Bureau of Standards

Certificate

Standard Reference Material 640a

Silicon Powder 2θ/d-Spacing Standard for X-Ray Diffraction

Camden R. Hubbard

This Standard Reference Material (SRM) was prepared for use as an external or internal 2θ/d-spacing calibration standard for powder diffractometry.

Electronic grade float-zone prepared silicon boules, 99.999 +% pure, were ground to pass a 75 μm (200 mesh) sieve. This powder, which was identical to SRM 640, was then jet-milled to reduce the mean particle size to about 2 μm.

A total of twelve samples, mixed with tungsten and silver internal standards [1], were measured using a high angle goniometer controlled by a minicomputer. The CuKα_1 peak position was determined by profile fitting procedures and then corrected for sample, instrumental, and physical aberrations (except refraction) through use of the internal standard lines [2]. Silicon peak positions were then corrected for effects of thermal expansion. Each corrected set was refined by a least-square routine that minimized $\Sigma(\theta_{obs}-\theta_{calc})^2$ to obtain estimates of a_i and their estimated standard errors, s_i. The weighted average of the twelve lattice parameters, uncorrected for refraction, at 25 °C is

$$\langle a \rangle = 5.430825 \pm 0.000036 \text{ Å}$$

where $\lambda(CuK\alpha_1) = 1.5405981$ Å [3]. The estimated total uncertainty given above includes contributions from three sources (listed in decreasing importance): (1) uncertainty of the lattice parameters of the tungsten and silver standards; (2) random errors of the measurements; and (3) the uncertainty in $\lambda(CuK\alpha_1)$.

The technical and support aspects concerning the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by R.K. Kirby.

Washington, D.C. 20234
February 19, 1982

George A. Uriano, Chief
Office of Standard Reference Materials

(over)

The 2θ values given in Table 1 were calculated from the certified value of the lattice parameter. The relative intensities [4], I (rel), also given in Table 1 can be used as an aid in identifying the silicon lines when SRM 640a is mixed with test materials. The uncertainty of each value of I (rel) may be as large as $\pm$ 3. Suggested methods for use of this SRM are given in references [2], [5], and [6].

Table 1

Calculated Diffraction Angles and Relative Intensities

(T = 25.0 °C)

These Values Are Not Certified

hkℓ	I (rel)	2θpeak	hkℓ	I (rel)	2θpeak
111	100	28.443°	511/333	6	94.955°
220	55	47.304	440	3	106.712
311	30	56.124	531	7	114.096
400	6	69.132	620	8	127.550
331	11	76.378	533	3	136.900
422	12	88.033	444	*	158.644

*Not measured

[1] Swanson, H.E.; McMurdie, H.F; Morris, M.C.; and Evans, E.H. (1966), Standard X-Ray Diffraction Powder Patterns, National Bureau of Standards Monograph 25, Section 4, NBS, Washington, D.C. 20234.

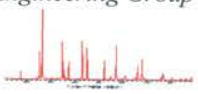
[2] Hubbard, C.R. (1982), submitted for publication in J. Appl. Cryst.

[3] Deslattes, R.D. and Henins, A. (1973), Phys. Rev. Letters, 31, 972-975.

[4] Morris, M.C.; McMurdie, H.F.; Evans, E.H.; Paretzkin, B.; deGroot, J.H.; Hubbard, C.R ; and Carmel, S.J. (1976) Standard X-Ray Diffraction Powder Patterns, National Bureau of Standards Monograph 25, Section 13, NBS, Washington, D.C. 20234.

[5] Snyder, R.L.; Hubbard, C.R.; and Panagiotopoulos, N.C., Advances in X-Ray Analysis, Vol. 25, (in press).

[6] Hubbard, C.R. (1980) Accuracy in Powder Diffraction, NBS Special Publication 567, p 489-502.



Appendix 4
Quality Control/Calibration

This control calibration measurement shows that the Rigaku Ultima IV diffractometer is within calibration per PNNL-RPG-268 Rev. 4, "Solids Analysis by X-Ray Diffraction" so no adjustments have been made. The XRD data obtained on this standard are represented in Figure 8. Table 5 contains summarized results of the quality control/calibration results obtained using NIST SRM 1976 (Corundum).

The columns in Table 5 have the following significance:

1) 2T(r) gives the 2-theta of the reference powder pattern (ICDD card),
2) 2T(o) is the observed 2-theta,
3) Delta is the difference between the observed and the reference 2-theta,
4) d(r) is the interplanar distance d for the reference pattern (in Å),
5) d(o) is the observed d,
6) Del-d is the difference between the observed and the reference d,
7) I % is the intensity in percent (the most intense peak is always 100% and all other peaks are normalized).

The requirement for this quality check (semiannual calibration verification of the diffractometer as stated in the PNNL-RPG-268 Rev. 4, is that observed d-interplanar distance for any given crystal lattice Miller indices be within ±0.02 Å of the NIST SRM 1976 ICDD card d-values. For this quality performance check, all Del-d values are well within this criterion. The ICDD card for alumina (corundum structure) is provided in Appendix 6.

Control Calibration Report (10 to 60 2 theta) – Date 01-06-2011

Theta Calibration Report
[nist_1976_dtex_010611d.raw] corundum - Full Chart
Reference Lines = 01-071-6478> Corundum - Al2O3
Two-Theta Range of Fit = 5.0/65.0(deg)
Zero Offset = 0.032(deg)

Table 5. Summary of the semiannual calibration report

2T(r)	2T(o)	Delta	d(r)	d(o)	Del-d	I%
25.573	25.541	0.032	3.4805	3.4848	0.0043	32.7
35.147	35.116	0.031	2.5512	2.5534	0.0022	100
37.77	37.739	0.03	2.3799	2.3817	0.0018	13
41.676	41.639	0.037	2.1654	2.1673	0.0019	3
43.347	43.317	0.03	2.0857	2.0871	0.0014	49.6
46.17	46.14	0.031	1.9646	1.9658	0.0012	0.5
52.544	52.515	0.029	1.7403	1.7412	0.0009	24.2
57.494	57.46	0.034	1.6016	1.6025	0.0009	80.4
59.729	59.7	0.029	1.5469	1.5476	0.0007	0.8
61.118	61.099	0.019	1.5151	1.5155	0.0004	1.4
61.299	61.261	0.039	1.511	1.5119	0.0009	14

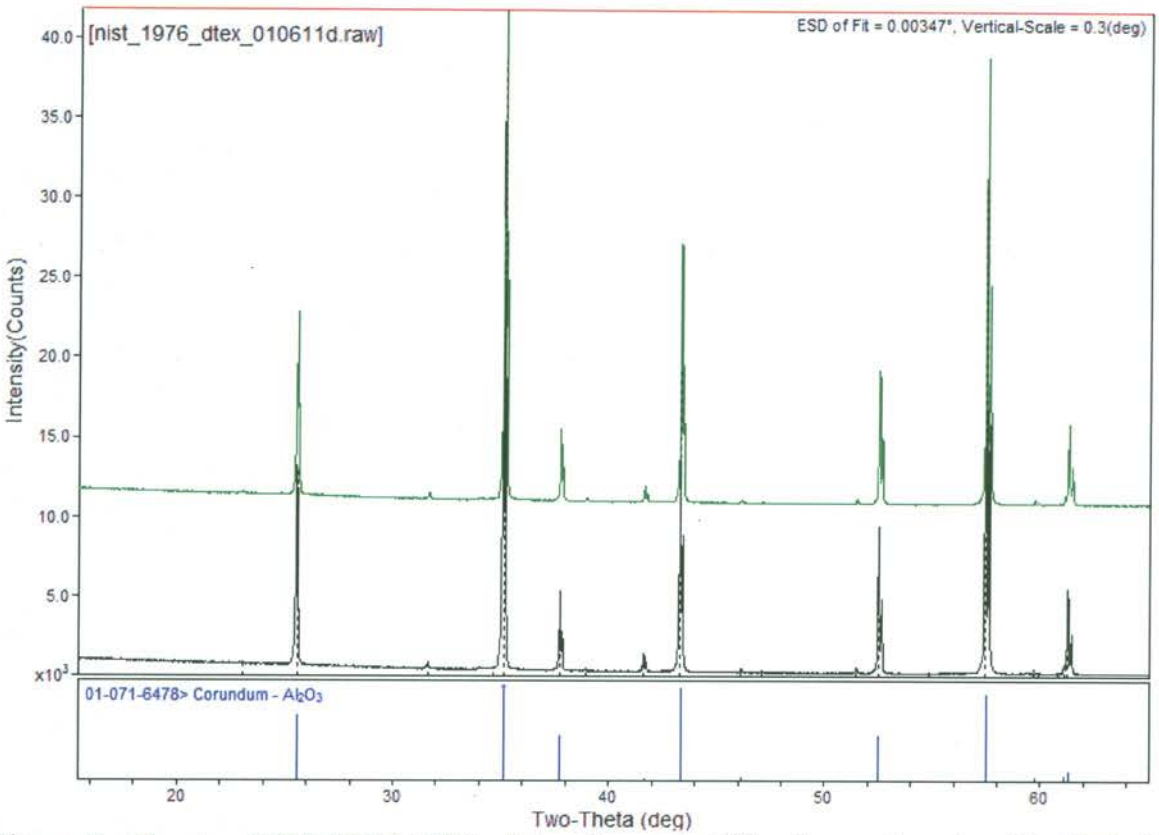
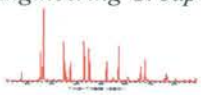


Figure 8. Alumina NIST SRM 1976 plate, observed diffraction pattern is with black (middle image) the simulated reference diffraction pattern is with green (top image) and the ICDD card is with blue (bottom image).



Appendix 5
SRM 1976 NIST Certificate

National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material 1976

Instrument Sensitivity Standard for X-ray Powder Diffraction

This Standard Reference Material (SRM) consists of a sintered alumina plate, approximately 45 mm on a side by 1.6 mm in thickness, intended for use in calibration of powder X-ray equipment for diffraction intensity as a function of 2Θ angle (instrument sensitivity). The form of the SRM serves to eliminate the variability of sample loading procedure from intensity measurements. This SRM material was chosen for consistency of microstructure with respect to grain size, shape, micro-strain and orientation. The platelets comprising this microstructure are 3 to 7 μm in diameter by 1 to 2 μm in thickness and are highly oriented with the basal plane parallel to the surface of the plate. Some amount of amorphous phase is present though no crystalline impurities could be detected.

The proper use of this SRM requires the measurement of intensity values from test equipment in a manner analogous to either of the two measurement methods used for certification. The certified parameters consist of absolute variation in intensity, 12 relative intensity values calculated from integrated intensity and peak height measurements from 25.5 to 145 degrees 2Θ, and the lattice parameters. Graphical evaluation of the ratio of data from the test equipment to the certified values will allow for an appropriate judgment as to the condition of the test equipment. This may be followed by the calculation of a correction curve, yielding standardization of instrument sensitivity and thus more accurate inter-laboratory comparisons of data involving X-ray diffraction intensity measurements.

The overall coordination of the preparation and the technical measurements leading to certification was performed by J.P. Cline of the NIST Ceramics Division. The project was the result of a round robin study carried out by R. Jenkins and W. Schreiner of the International Centre for Diffraction Data, Swarthmore, Pennsylvania.

The alumina substrates for this SRM were donated to NIST by International Business Machines Corporation, East Fishkill, New York, through the efforts of R. Anderson.

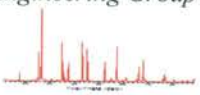
Statistical analysis was provided by S.B. Schiller and K.R. Eberhardt of the NIST Statistical Engineering Division.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Standard Reference Materials Program by N.M. Trahey.

Gaithersburg, MD 20899
November 8, 1991

William P. Reed, Chief
Standard Reference Materials Program

(over)



Certified intensity data were collected from 25 randomly selected samples on a Philips[1] diffractometer using an AEG[1] long fine-focus Cu X-ray tube operated at 1800 watts with a constant potential generator. The diffractometer was equipped with incident and diffracted beam sollar slits, graphite diffracted beam monochromator in anti-parallel geometry, and scintillation detector. The incident beam slits were of the theta compensating type, locked such that the illumination area was 12.5 mm wide at 25 degrees 2Θ. This corresponds to a slit size of 0.93 degrees; the receiving slit was 0.066 degrees. The take-off angle was 4 degrees. Samples were spun at approximately 52 rpm during data collection.

Data were collected and processed with the Siemens Diffrac500[1] version of NBS*Quant. This algorithim scans specific peaks for an integrated intensity measurement, with extended count times for each of ten points on either side of the scan range. Scan angles are indicated in Table 1; the step width was 0.005 degrees. Scan time ranged from 30 to 90 minutes per peak, for a total of 16 hours per sample. The seventh and eleventh scan ranges actually include two peaks, as noted by the two sets of miller indices listed for them in Table 1. The use of two peaks in these scan ranges ensured that intensity data were reported evenly through the desired 2Θ range.

Peak height values were manually determined, with a background subtraction, from the same scans as were the integrated intensity data. Five background data points from each end of the scan range were averaged to determine the background value. Peak height values were determined from an average of the three most intense points for data below 55 degrees 2Θ; four data points were used for the remainder of the data. Due to the proximity of the two reflections used in the seventh and eleventh relative intensity values, peak height values for these data reflect the superposition of the Cu $K\alpha_1$ and $K\alpha_2$ peaks. Thus, peak height data for these two pairs of reflections will be valid only with the use of Cu Kα radiation.

Lattice parameters were determined from the 25 samples by means of least squares refinement from 47 peak positions determined with the spline method, ranging from 22 to 152 degrees 2Θ. Peak positions were corrected for optical aberrations with the use of SRM 640b silicon powder, to generate a 2nd order polynomial external standard correction curve which was applied to peak location determinations. Measurements were made on a Siemens D500[1] diffractometer equipped with a focusing Ge incident beam monochromator, sample spinner, and a position sensitive proportional detector. Copper Kα radiation was used. Measurements were made within a period of 48 hours during which operating conditions were maintained. The machine was stabilized under such conditions for several days prior to measurements.

The intensity measurements from the SRM material are expected to show heterogeneity due to variations in preferred orientation. The heterogeneous nature of the samples requires a certification of a population of true relative intensities rather than the average relative intensity for each peak. The preferred orientation will uniformly effect the variability of all the peaks so analysis of the data assumed the intensity measurements have the same true relative variance. This allowed for pooling of the variances for both absolute and relative intensities. The exception to this assumption is the (300) peak; the crystallites in the diffracting position for this reflection must be perpendicular to the preferred orientation direction. Increased variability of intensity from this reflection is thus expected. The nominal value of the relative intensity is reported for information only; it was not used in subsequent analysis.

[1] Certain manufacturers are mentioned for informative purposes only and do not represent an endorsement by NIST.

ABSOLUTE VARIATION IN INTENSITY

Analysis of the raw integrated intensity values indicated a drift in the instrument. This drift was modeled with a quadratic equation and removed from subsequent analyses. The relative standard deviation of the raw counts, pooled from the 13 peaks is:

RSD (raw counts) = 2.21%

This value incorporates both material variability and machine imprecision. This parameter can be used to determine differences in absolute sensitivity with different SRM samples.

RELATIVE INTENSITY VARIATION

The relative intensity values displayed no drift. The pooled relative standard deviation from integrated intensity measurements of the 12 peaks is:

RSD (integrated area, relative intensity) = 2.06%

The pooled relative standard deviation of the relative intensity values determined from peak height measurements is:

RSD (peak height, relative intensity) = 2.62%

The total relative uncertainty is a multiple of the relative standard deviation. For each relative intensity, this value is:

Total Uncertainty (integrated intensity) = 6.12%
Total Uncertainty (peak height) = 7.85%

This uncertainty is the half-width of a 90% confidence, 95% coverage tolerance interval. With 90% confidence, 95% of the true relative intensities for all of the peaks of this SRM should fall within the uncertainty of the certified values.

Each SRM plate is sintered to a relatively low density allowing it to be scored and broken easily into a desired size. Care should be taken to ensure that the side opposite the label is illuminated; otherwise, results are invalid. Each plate is cylindrically symmetrical, thus orientation of the plate is not of concern. However, errors due to particle counting statistics will be reduced if a sample spinner is used. Consistency in the degree of size and micro-strain induced peak broadening of the SRM allow for the use of peak heights as a substitution for integrated intensity measurements, though precision is seen to suffer as a result. The ratio of experimental data to the certified values should be plotted as a function of 2Θ angle. If this data is patternless, and if all of the ratios fall within the total uncertainty (0.0612/0.0785) of 1, then the test equipment is in control. However, if there is a clear pattern in the data, or if the ratio for at least one peak is more than 0.0612/0.0785 from 1, then the system is out of control (see Appendix).

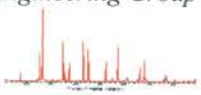


Table 1

Reflection(s) (hkl)	Scan Angles (low/high) 2Θ		Relative Intensity Integrated Area	Relative Intensity Peak Height
(012)	24.7	26.2	32.34	33.31
(104)	34.0	36.2	100.0	100.0
(113)	42.4	44.2	51.06	49.87
(024)	51.8	53.3	26.69	25.17
(116)	56.0	59.0	92.13	83.6
(300)	67.4	69.0	19.13	16.89
(1.0.10) (119)	75.7	78.2	55.57	34.61
(0.2.10)	88.1	89.7	11.76	8.99
(226)	94.3	96.0	10.14	7.25
(2.1.10)	100.1	102.0	16.13	10.94
(324) (0.1.14)	115.4	117.4	20.86	10.09
(1.3.10)	126.8	128.95	15.58	7.56
(146)	135.2	137.4	15.47	6.55
(4.0.10)	144.3	146.7	11.29	4.06

LATTICE PARAMETERS

The certified lattice parameters, from the mean of the 25 samples are:

$a = 0.4758846$ nm (4.758846 Å)

$\sigma = 0.0000109$ (0.000109)

$c = 1.299306$ nm (12.99306 Å)

$\sigma = 0.0000238$ (0.000238)

where λ ($CuK\alpha_1$) = 0.1540629 nm (1.540629Å)

Appendix: How to assess and correct instrument bias using this SRM

When looking for instrumental problems, the peaks should be considered jointly, not individually. First, plot control bands as $1 \pm U$ over the range of 2Θ, where U is the relative uncertainty (U = 0.0612 if peak areas were used, or 0.0785 if peak heights were used). Next, plot the ratio of each new measurement to the certified value on the vertical axis against the 2Θ peak location on the horizontal axis. As long as these ratios fall within the control bands and are patternless, the system cannot be labelled "out of control."

However, if there is a pattern in the ratios, then the device is introducing a systematic error, even if all of the ratios fall within the control bands. This systematic error should be corrected by modelling the ratios as a function of peak location and using the model to correct future observations on unknowns.

Let the measured relative intensities on the 12 peaks be y_i and the certified values be c_i, so the ratios to model are

$$r_i = \frac{y_i}{c_i}.$$

Fit a simple model, using ordinary least squares regression, which describes these ratios adequately as a function of peak location (x_i). The fitted model has m parameters: m=1 for a horizontal straight line, m=2 for a slanted straight line, m=3 for a quadratic, etc. The residual sum of squares from the fit, plus an allowance for the uncertainty in the certified values on the certificate, is:

$$RSS_{full} = \sum_{1}^{n} (r_i - \hat{r}_i)^2 + (n-m)\frac{(RSD)^2}{25}$$

and the residual degrees of freedom for this "full" model are n - m. The RSD = 0.0206 if peak areas were used, or 0.0262 if peak heights were used. The residual sum of squares for the horizontal line at 1 (which describes "ideal" data), plus an allowance for the uncertainty in the certified values on the certificate, is:

$$RSS_1 = \sum_{1}^{n} (r_i - 1)^2 + (n-1)\frac{(RSD)^2}{25}$$

and the residual degrees of freedom are n - 1. Then if the test statistic

$$F = \frac{\left\{RSS_1 - RSS_{full} + (m-1)\frac{(RSD)^2}{25}\right\} / (m-1)}{\left\{RSS_{full} + (n-m)\frac{(RSD)^2}{25}\right\} / (n-m)}$$

is larger than the upper 0.05 percentage point from the F distribution with m-1 numerator and n-m denominator degrees of freedom (commonly provided in table form in introductory statistics texts), the correction model is needed.

To use the correction model: A new peak angle x* and a new relative intensity y* are measured on an unknown sample. If $\hat{r}^*$ is the predicted value of the fitted model at x*, the corrected relative intensity for the new peak angle on the unknown sample is

$$\text{corrected relative intensity} = \frac{y^*}{\hat{r}^*}.$$

Appendix 6
ICDD cards for the identified crystalline phases presented in this report

The following ICDD cards are for the crystalline phases identified in this report.
The ICDD card includes information about the crystal structure, the reference, the X-ray radiation wavelength, the calculated density and the observed density (if published), the strongest lines and the corresponding 2 theta angles. The table presented at the end of each ICDD card includes information about the 2 theta angle positions, interplanar distances at that angle, the intensity of that particular reflection and Miller indices at that particular 2 theta. The compounds identified in this report match the ICDD card peaks, which are the stick figures in the report.

1) Quartz SiO_2
PDF#01-070-3755(RDB)
Radiation=CuKá1 Lambda=1.5406
Ref: Gualtieri, A.F. J. Appl. Crystallogr., v33 p267 (2000)
Hexagonal - Powder Diffraction, P3121 (152) Z=3
CELL: 4.9158 x 4.9158 x 5.4091 <90.0 x 90.0 x 120.0>
Density(c)=2.644
Strong Lines: 3.35/X 4.26/2 1.82/1 2.46/1 1.54/1 2.28/1

2-Theta	d(Å)	I(f)	(h k l)	Theta	1/(2d)	2pi/d	n^2
20.849	4.2572	21.8	(1 0 0)	10.424	0.1174	1.4759	
26.624	3.3454	100.0	(0 1 1)	13.312	0.1495	1.8782	
36.528	2.4579	6.5	(1 1 0)	18.264	0.2034	2.5563	
39.441	2.2828	6.3	(1 0 2)	19.720	0.2190	2.7524	
40.270	2.2377	2.9	(1 1 1)	20.135	0.2234	2.8079	
42.431	2.1286	4.4	(2 0 0)	21.216	0.2349	2.9518	
45.771	1.9808	2.7	(2 0 1)	22.885	0.2524	3.1721	
50.109	1.8190	9.9	(1 1 2)	25.055	0.2749	3.4543	
50.583	1.8030	0.3	(0 0 3)	25.291	0.2773	3.4848	
54.841	1.6727	3.0	(0 2 2)	27.420	0.2989	3.7564	
55.286	1.6603	1.3	(0 1 3)	27.643	0.3012	3.7844	
57.204	1.6091	0.2	(2 1 0)	28.602	0.3107	3.9049	
59.928	1.5423	6.4	(1 2 1)	29.964	0.3242	4.0740	
63.990	1.4538	1.2	(1 1 3)	31.995	0.3439	4.3219	
65.751	1.4191	0.3	(3 0 0)	32.876	0.3523	4.4277	
67.702	1.3828	3.7	(1 2 2)	33.851	0.3616	4.5437	
68.096	1.3758	4.5	(2 0 3)	34.048	0.3634	4.5669	
68.276	1.3726	4.9	(0 3 1)	34.138	0.3643	4.5775	
73.407	1.2888	1.3	(1 0 4)	36.704	0.3880	4.8751	
75.613	1.2566	1.6	(3 0 2)	37.807	0.3979	5.0001	
77.628	1.2290	0.8	(2 2 0)	38.814	0.4069	5.1126	
79.827	1.2005	1.8	(2 1 3)	39.913	0.4165	5.2337	
79.996	1.1984	1.1	(2 2 1)	39.998	0.4172	5.2429	

81.105	1.1848	1.4	(1 1 4)	40.553	0.4220	5.3032
81.443	1.1807	1.7	(3 1 0)	40.722	0.4235	5.3214
83.787	1.1536	0.9	(1 3 1)	41.894	0.4334	5.4467
84.886	1.1414	0.2	(2 0 4)	42.443	0.4381	5.5047
87.017	1.1189	0.1	(2 2 2)	43.509	0.4469	5.6157
87.383	1.1151	0.2	(3 0 3)	43.691	0.4484	5.6345
90.771	1.0821	1.4	(1 3 2)	45.385	0.4621	5.8064
92.731	1.0643	0.2	(4 0 0)	46.366	0.4698	5.9036
94.558	1.0485	0.7	(0 1 5)	47.279	0.4769	5.9925
95.060	1.0443	0.5	(4 0 1)	47.530	0.4788	6.0168
96.159	1.0352	0.7	(2 1 4)	48.080	0.4830	6.0693
98.672	1.0155	0.6	(2 2 3)	49.336	0.4924	6.1873
102.147	0.9902	0.5	(1 1 5)	51.074	0.5050	6.3457
102.147	0.9902	0.5	(0 4 2)	51.074	0.5050	6.3457
102.489	0.9878	0.4	(1 3 3)	51.244	0.5062	6.3609
103.784	0.9790	0.1	(0 3 4)	51.892	0.5107	6.4182
104.128	0.9767	0.4	(3 2 0)	52.064	0.5119	6.4333
106.016	0.9644	0.1	(0 2 5)	53.008	0.5184	6.5150
106.537	0.9611	0.6	(2 3 1)	53.269	0.5202	6.5373
112.027	0.9290	0.1	(4 1 0)	56.013	0.5382	6.7634
113.974	0.9186	0.2	(3 2 2)	56.987	0.5443	6.8399
114.373	0.9165	0.5	(4 0 3)	57.187	0.5455	6.8554
114.557	0.9156	0.6	(4 1 1)	57.278	0.5461	6.8624
115.766	0.9095	0.2	(2 2 4)	57.883	0.5498	6.9085
117.397	0.9015	0.1	(0 0 6)	58.698	0.5546	6.9696
118.186	0.8978	0.4	(1 2 5)	59.093	0.5569	6.9986
120.011	0.8894	0.3	(3 1 4)	60.005	0.5622	7.0644
121.710	0.8820	0.1	(1 0 6)	60.855	0.5669	7.1241
122.498	0.8786	0.2	(1 4 2)	61.249	0.5691	7.1513
127.106	0.8603	0.1	(3 0 5)	63.553	0.5812	7.3032
127.526	0.8588	0.1	(2 3 3)	63.763	0.5822	7.3165
131.040	0.8464	0.1	(1 1 6)	65.520	0.5908	7.4236
132.649	0.8411	0.1	(0 5 1)	66.325	0.5945	7.4703
134.155	0.8363	0.1	(0 4 4)	67.077	0.5978	7.5127
136.225	0.8301	0.4	(0 2 6)	68.112	0.6023	7.5689
137.738	0.8258	0.5	(1 4 3)	68.869	0.6055	7.6084
140.166	0.8193	0.1	(3 3 0)	70.083	0.6103	7.6690
143.052	0.8121	0.6	(5 0 2)	71.526	0.6157	7.7365
143.052	0.8121	0.6	(2 2 5)	71.526	0.6157	7.7365
143.946	0.8101	0.5	(3 3 1)	71.973	0.6172	7.7564
146.447	0.8045	0.3	(4 2 0)	73.224	0.6215	7.8097
149.906	0.7976	0.2	(1 3 5)	74.953	0.6268	7.8772

2) Rutile, syn (Titanium Oxide) Ti O2PDF#01-077-0440(RDB)
Radiation=CuKá1 Lambda=1.5406 Filter=
Calibration= 2T=27.438-143.112 I/Ic(RIR)=3.6
Ref: Seki, H., Ishizawa, N., Mizutani, N., Kato, M. Yogyo Kyokaishi (J. Ceram. Assoc. Jpn.), v92 p219 (1984)
Tetragonal - Single Crystal, P42/mnm (136) Z=2
CELL: 4.5934 x 4.5934 x 2.9575 <90.0 x 90.0 x 90.0> P.S=tP6.00
Density(c)=4.252
Strong Lines: 3.25/X 1.69/5 2.49/4 2.19/2 1.36/2 1.62/1 1.35/1 1.48/1
Calculated Pattern Original Remarks: Stable from 800 to 1800 K (2nd ref., Tomaszewski), 300-800 K: Pbca.
Temperature of Data Collection: 300 K.
Wyckoff Sequence: f a(P42/MNM).
Unit Cell Data Source: Single Crystal.

2-Theta	d(Å)	I(f)	(h k l)	Theta	1/(2d)	2pi/d	n^2
27.438	3.2480	100.0	(1 1 0)	13.719	0.1539	1.9345	
36.091	2.4867	43.6	(1 0 1)	18.046	0.2011	2.5268	
39.193	2.2967	6.4	(2 0 0)	19.596	0.2177	2.7357	
41.250	2.1868	17.0	(1 1 1)	20.625	0.2286	2.8733	
44.046	2.0542	6.0	(2 1 0)	22.023	0.2434	3.0587	
54.331	1.6872	47.8	(2 1 1)	27.165	0.2964	3.7241	
56.630	1.6240	13.7	(2 2 0)	28.315	0.3079	3.8689	
62.787	1.4788	6.5	(0 0 2)	31.393	0.3381	4.2490	
64.052	1.4526	6.3	(3 1 0)	32.026	0.3442	4.3256	
65.520	1.4235	0.5	(2 2 1)	32.760	0.3512	4.4138	
69.014	1.3597	15.1	(3 0 1)	34.507	0.3677	4.6209	
69.829	1.3458	7.7	(1 1 2)	34.915	0.3715	4.6686	
72.429	1.3038	0.8	(3 1 1)	36.214	0.3835	4.8191	
74.405	1.2740	0.2	(3 2 0)	37.203	0.3925	4.9319	
76.565	1.2433	1.6	(2 0 2)	38.283	0.4021	5.0535	
79.858	1.2001	0.9	(2 1 2)	39.929	0.4166	5.2354	
82.348	1.1700	2.9	(3 2 1)	41.174	0.4273	5.3701	
84.255	1.1484	2.0	(4 0 0)	42.128	0.4354	5.4715	
87.487	1.1141	0.7	(4 1 0)	43.743	0.4488	5.6399	
89.579	1.0934	4.8	(2 2 2)	44.789	0.4573	5.7465	
90.710	1.0827	2.5	(3 3 0)	45.355	0.4618	5.8034	
95.268	1.0426	4.1	(4 1 1)	47.634	0.4796	6.0267	
96.035	1.0363	3.3	(3 1 2)	48.017	0.4825	6.0634	
97.173	1.0271	1.8	(4 2 0)	48.587	0.4868	6.1173	
98.515	1.0167	0.1	(3 3 1)	49.257	0.4918	6.1800	
105.103	0.9703	0.4	(4 2 1)	52.551	0.5153	6.4757	
106.099	0.9639	1.3	(1 0 3)	53.050	0.5187	6.5186	
109.484	0.9433	0.4	(1 1 3)	54.742	0.5300	6.6606	
113.960	0.9187	0.1	(4 3 0)	56.980	0.5443	6.8394	

116.270	0.9070	1.9	(4 0 2)	58.135	0.5513	6.9276
117.538	0.9008	2.1	(5 1 0)	58.769	0.5550	6.9748
119.923	0.8898	1.0	(4 1 2)	59.962	0.5619	7.0613
120.151	0.8888	4.2	(2 1 3)	60.075	0.5626	7.0694
122.804	0.8773	3.9	(4 3 1)	61.402	0.5699	7.1617
123.716	0.8736	2.8	(3 3 2)	61.858	0.5724	7.1926
126.728	0.8618	0.1	(5 1 1)	63.364	0.5802	7.2912
129.129	0.8530	0.1	(5 2 0)	64.565	0.5862	7.3662
131.880	0.8436	2.5	(4 2 2)	65.940	0.5927	7.4482
136.657	0.8289	3.0	(3 0 3)	68.328	0.6032	7.5803
140.063	0.8196	5.1	(5 2 1)	70.031	0.6101	7.6665
141.582	0.8157	0.2	(3 1 3)	70.791	0.6130	7.7027
143.112	0.8120	0.7	(4 4 0)	71.556	0.6158	7.7379

3) Corundum (Aluminum Oxide), Al_2O_3, PDF#01-071-6478

Radiation=CuKá1 Lambda=1.5406
Ref: Riello, P., Canton, P., Fagherazzi, G. Powder Diffraction, v12 p160 (1997)
Rhombohedral - Powder Diffraction, R-3c (167) Z=6
CELL: 4.7598 x 4.7598 x 12.9924 <90.0 x 90.0 x 120.0> P.S=hR10.00 (Á)
Density(c)=3.985
Strong Lines: 2.55/X 2.09/X 1.60/9 3.48/7 1.37/5 1.74/5 2.38/5 1.40/3
ICSD Collection Code: 77810.
Calculated Pattern Original Remarks: R(Bragg)=0.03.
Minor Warning: 10%<R factor<15% (for powder).
Wyckoff Sequence: e c(R3-CH)

2-Theta	d(Å)	I(f)	(h k l)	Theta	1/(2d)	2pi/d	n^2
25.573	3.4805	67.0	(0 1 2)	12.786	0.1437	1.8052	
35.147	2.5512	100.0	(1 0 4)	17.574	0.1960	2.4628	
37.770	2.3799	46.0	(1 1 0)	18.885	0.2101	2.6401	
41.676	2.1654	0.5	(0 0 6)	20.838	0.2309	2.9016	
43.347	2.0857	96.5	(1 1 3)	21.674	0.2397	3.0125	
46.170	1.9646	1.5	(2 0 2)	23.085	0.2545	3.1983	
52.544	1.7403	46.2	(0 2 4)	26.272	0.2873	3.6105	
57.494	1.6016	91.0	(1 1 6)	28.747	0.3122	3.9230	
59.729	1.5469	2.2	(2 1 1)	29.865	0.3232	4.0617	
61.118	1.5151	3.4	(1 2 2)	30.559	0.3300	4.1472	
61.299	1.5110	8.2	(0 1 8)	30.650	0.3309	4.1583	
66.507	1.4048	34.4	(2 1 4)	33.254	0.3559	4.4728	
68.195	1.3740	52.6	(3 0 0)	34.098	0.3639	4.5728	
70.405	1.3362	1.1	(1 2 5)	35.203	0.3742	4.7022	
74.294	1.2756	1.3	(2 0 8)	37.147	0.3920	4.9256	
76.871	1.2392	14.7	(1,0,10)	38.435	0.4035	5.0706	
77.230	1.2343	8.6	(1 1 9)	38.615	0.4051	5.0906	
80.407	1.1933	0.7	(2 1 7)	40.204	0.4190	5.2653	

80.682	1.1900	5.7	(2 2 0)	40.341	0.4202	5.2802
83.203	1.1602	0.6	(0 3 6)	41.601	0.4310	5.4157
84.338	1.1474	4.6	(2 2 3)	42.169	0.4358	5.4759
85.121	1.1389	0.3	(1 3 1)	42.560	0.4390	5.5170
86.333	1.1260	3.4	(3 1 2)	43.167	0.4441	5.5803
86.492	1.1243	2.7	(1 2 8)	43.246	0.4447	5.5885
88.990	1.0991	6.3	(0,2,10)	44.495	0.4549	5.7167
90.707	1.0827	1.6	(0,0,12)	45.354	0.4618	5.8033
91.170	1.0784	7.4	(1 3 4)	45.585	0.4636	5.8263
94.800	1.0465	0.4	(3 1 5)	47.400	0.4778	6.0042
95.231	1.0429	14.4	(2 2 6)	47.615	0.4795	6.0250
98.370	1.0178	1.8	(0 4 2)	49.185	0.4913	6.1733
101.064	0.9978	10.3	(2,1,10)	50.532	0.5011	6.2969
102.819	0.9855	0.3	(1,1,12)	51.409	0.5074	6.3756
103.293	0.9823	1.9	(4 0 4)	51.646	0.5090	6.3966
104.620	0.9734	0.1	(1 3 7)	52.310	0.5137	6.4548
109.511	0.9432	0.4	(3 2 1)	54.756	0.5301	6.6617
109.848	0.9412	0.3	(1,2,11)	54.924	0.5312	6.6755
110.968	0.9349	2.9	(3 1 8)	55.484	0.5348	6.7210
114.050	0.9182	2.0	(2 2 9)	57.025	0.5445	6.8428
116.069	0.9080	9.4	(3 2 4)	58.034	0.5507	6.9200
116.600	0.9054	6.5	(0,1,14)	58.300	0.5523	6.9399
117.817	0.8995	5.2	(4 1 0)	58.908	0.5559	6.9851
120.180	0.8887	0.3	(2 3 5)	60.090	0.5626	7.0704
122.000	0.8807	3.2	(4 1 3)	61.000	0.5677	7.1341
124.567	0.8701	1.9	(0 4 8)	62.284	0.5746	7.2209
127.657	0.8583	10.5	(1,3,10)	63.829	0.5826	7.3206
129.859	0.8504	4.2	(0,3,12)	64.930	0.5879	7.3884
131.092	0.8462	4.1	(2,0,14)	65.546	0.5909	7.4251
132.178	0.8426	0.4	(3 2 7)	66.089	0.5934	7.4568
132.608	0.8412	0.3	(2,1,13)	66.304	0.5944	7.4692
136.032	0.8307	15.7	(1 4 6)	68.016	0.6019	7.5638
139.339	0.8215	0.1	(3,1,11)	69.669	0.6087	7.6487
140.977	0.8172	0.4	(2 3 8)	70.489	0.6118	7.6885
142.308	0.8139	2.1	(1,1,15)	71.154	0.6143	7.7196
145.131	0.8074	6.1	(4,0,10)	72.565	0.6193	7.7821
148.258	0.8008	0.2	(2,2,12)	74.129	0.6244	7.8459
149.147	0.7991	4.1	(0 5 4)	74.574	0.6257	7.8630

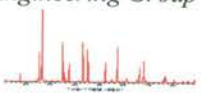
4) Uranium Oxide, U4 O9, PDF#01-075-0944(RDB)

Radiation=CuKá1 Lambda=1.5406
Calibration= 2T=9.963-101.026 I/Ic(RIR)=22.4
Ref: Belbboch, B., Piekarski, C., Perio, P. Bull. Soc. Fr. Mineral. Cristallogr., v83 p206 (1960)
Cubic - Single Crystal, I-43d (220) Z=64
CELL: 21.73 x 21.73 x 21.73 <90.0 x 90.0 x 90.0> P.S=cI832.00
Density(c)=11.353

Strong Lines: 3.14/X 1.92/4 2.72/4 1.64/4 1.25/1 1.11/1 1.21/1 1.57/1
Calculated Pattern Original Remarks: Stable above 338 K (2nd ref., Tomaszewski), below trigonal.
Minor Warning: No e.s.d reported/abstracted on the cell dimension.
No R factors reported/abstracted.
Wyckoff Sequence: e14 d3 c4 b a(I4-3D).
Unit Cell Data Source: Single Crystal.

2-Theta	d(Å)	I(f)	(h k l)	Theta	1/(2d)	2pi/d	n^2
9.963	8.8712	0.2	(2 1 1)	4.981	0.0564	0.7083	6
11.509	7.6827	0.1	(2 2 0)	5.754	0.0651	0.8178	8
12.873	6.8716	0.1	(3 1 0)	6.436	0.0728	0.9144	10
15.244	5.8076	0.1	(3 2 1)	7.622	0.0861	1.0819	14
16.303	5.4325	0.1	(4 0 0)	8.152	0.0920	1.1566	16
19.142	4.6329	0.1	(3 3 2)	9.571	0.1079	1.3562	22
20.002	4.4356	0.1	(4 2 2)	10.001	0.1127	1.4165	24
20.827	4.2616	0.1	(4 3 1)	10.414	0.1173	1.4744	26
22.391	3.9673	0.1	(5 2 1)	11.196	0.1260	1.5837	30
25.244	3.5251	0.1	(6 1 1)	12.622	0.1418	1.7824	38
28.434	3.1365	100.0	(4 4 4)	14.217	0.1594	2.0033	48
30.198	2.9571	0.1	(7 2 1)	15.099	0.1691	2.1248	54
32.949	2.7163	38.3	(8 0 0)	16.474	0.1841	2.3132	64
34.505	2.5972	0.1	(6 5 3)	17.253	0.1925	2.4192	70
35.010	2.5609	0.1	(8 2 2)	17.505	0.1952	2.4535	72
35.509	2.5261	0.1	(7 4 3)	17.755	0.1979	2.4873	74
36.489	2.4604	0.1	(7 5 2)	18.244	0.2032	2.5537	78
36.970	2.4295	0.1	(8 4 0)	18.485	0.2058	2.5862	80
38.384	2.3432	0.1	(7 6 1)	19.192	0.2134	2.6814	86
39.302	2.2905	0.1	(8 5 1)	19.651	0.2183	2.7431	90
40.203	2.2413	0.1	(7 6 3)	20.102	0.2231	2.8034	94
40.647	2.2178	0.1	(8 4 4)	20.324	0.2254	2.8331	96
41.956	2.1516	0.1	(10,1,1)	20.978	0.2324	2.9203	102
42.385	2.1308	0.1	(8 6 2)	21.193	0.2347	2.9487	104
42.811	2.1106	0.1	(9 5 0)	21.405	0.2369	2.9770	106
43.652	2.0719	0.1	(7 6 5)	21.826	0.2413	3.0326	110
45.296	2.0004	0.1	(9 6 1)	22.648	0.2499	3.1409	118

47.288	1.9207	44.0	(8 8 0)	23.644	0.2603	3.2713	128
48.453	1.8772	0.1	(9 7 2)	24.226	0.2664	3.3471	134
49.975	1.8235	0.1	(9 6 5)	24.987	0.2742	3.4456	142
50.350	1.8108	0.1	(12,0,0)	25.175	0.2761	3.4698	144
51.463	1.7743	0.1	(10,7,1)	25.731	0.2818	3.5413	150
52.196	1.7511	0.1	(12,3,1)	26.098	0.2855	3.5882	154
52.921	1.7288	0.1	(11,6,1)	26.460	0.2892	3.6345	158
53.640	1.7073	0.1	(11,5,4)	26.820	0.2929	3.6803	162
54.352	1.6866	0.1	(9 7 6)	27.176	0.2965	3.7254	166
56.105	1.6380	35.0	(12,4,4)	28.052	0.3053	3.8360	176
58.837	1.5682	8.2	(8 8 8)	29.419	0.3188	4.0065	192
59.842	1.5443	0.1	(13,5,2)	29.921	0.3238	4.0687	198
61.165	1.5140	0.1	(14,3,1)	30.583	0.3303	4.1501	206
62.472	1.4854	0.1	(13,6,3)	31.236	0.3366	4.2299	214
62.797	1.4785	0.1	(10,10,4)	31.398	0.3382	4.2496	216
63.120	1.4717	0.1	(12,7,5)	31.560	0.3397	4.2692	218
63.764	1.4584	0.1	(14,5,1)	31.882	0.3428	4.3082	222
65.041	1.4328	0.1	(13,6,5)	32.521	0.3490	4.3852	230
65.675	1.4205	0.1	(11,8,7)	32.838	0.3520	4.4231	234
66.305	1.4086	0.1	(15,3,2)	33.153	0.3550	4.4607	238
67.558	1.3855	0.1	(14,7,1)	33.779	0.3609	4.5351	246
69.107	1.3581	5.4	(16,0,0)	34.554	0.3682	4.6264	256
71.250	1.3225	0.1	(14,7,5)	35.625	0.3781	4.7512	270
72.462	1.3033	0.1	(15,7,2)	36.231	0.3836	4.8211	278
73.065	1.2940	0.1	(16,5,1)	36.533	0.3864	4.8556	282
73.667	1.2849	0.1	(14,9,3)	36.833	0.3891	4.8899	286
73.966	1.2805	0.1	(16,4,4)	36.983	0.3905	4.9070	288
74.863	1.2673	0.1	(13,10,5)	37.432	0.3945	4.9579	294
76.350	1.2463	11.6	(12,12,4)	38.175	0.4012	5.0415	304
76.646	1.2422	6.0	(17,4,1)	38.323	0.4025	5.0580	306
78.710	1.2147	8.9	(16,8,0)	39.355	0.4116	5.1725	320
79.003	1.2110	4.6	(13,12,3)	39.501	0.4129	5.1886	322
81.924	1.1750	0.1	(15,9,6)	40.962	0.4255	5.3473	342
82.215	1.1716	0.1	(18,4,2)	41.107	0.4268	5.3629	344
82.505	1.1682	0.1	(15,11,0)	41.253	0.4280	5.3785	346
83.085	1.1615	0.1	(17,6,5)	41.543	0.4305	5.4095	350
83.376	1.1582	0.1	(12,12,8)	41.688	0.4317	5.4249	352
83.665	1.1549	0.1	(17,8,1)	41.833	0.4329	5.4403	354
84.244	1.1485	0.1	(18,5,3)	42.122	0.4354	5.4709	358
84.534	1.1453	0.1	(16,10,2)	42.267	0.4366	5.4862	360
84.824	1.1421	0.1	(15,11,4)	42.412	0.4378	5.5014	362
85.113	1.1390	0.1	(18,6,2)	42.556	0.4390	5.5166	364
85.401	1.1359	0.1	(19,2,1)	42.701	0.4402	5.5317	366
86.556	1.1236	0.1	(14,13,3)	43.278	0.4450	5.5919	374
87.998	1.1089	9.1	(16,8,8)	43.999	0.4509	5.6661	384



88.286	1.1060	4.7	(19,4,3)	44.143	0.4521	5.6808	386
91.166	1.0784	0.1	(15,10,9)	45.583	0.4636	5.8262	406
91.454	1.0758	0.1	(20,2,2)	45.727	0.4648	5.8405	408
91.742	1.0732	0.1	(19,7,0)	45.871	0.4659	5.8548	410
92.319	1.0680	0.1	(17,11,2)	46.159	0.4682	5.8833	414
94.915	1.0455	7.7	(20,4,4)	47.458	0.4782	6.0098	432
95.206	1.0431	4.0	(19,8,3)	47.603	0.4794	6.0237	434
98.105	1.0198	0.1	(21,3,2)	49.052	0.4903	6.1610	454
98.396	1.0176	0.1	(14,14,8)	49.198	0.4914	6.1745	456
98.687	1.0154	0.1	(20,7,3)	49.343	0.4924	6.1880	458
98.978	1.0132	0.1	(18,10,6)	49.489	0.4935	6.2015	460
99.270	1.0110	0.1	(17,13,2)	49.635	0.4946	6.2150	462
99.562	1.0088	0.1	(20,8,0)	49.781	0.4956	6.2284	464
99.855	1.0066	0.1	(21,5,0)	49.927	0.4967	6.2419	466
100.440	1.0023	0.1	(21,5,2)	50.220	0.4988	6.2686	470
100.733	1.0002	0.1	(18,12,2)	50.367	0.4999	6.2819	472
101.026	0.9981	0.1	(17,13,4)	50.513	0.5010	6.2952	474

5) Uraninite (Uranium Oxide) U O2, PDF#01-072-4553(RDB)

Radiation=CuKá1 Lambda=1.5406
Calibration= 2T=28.373-139.625 I/Ic(RIR)=23.7
Ref: Wasserstein, B. Nature (London), v168 p380 (1951)
Cubic - Single Crystal, Fm-3m (225) Z=4
CELL: 5.444 x 5.444 x 5.444 <90.0 x 90.0 x 90.0> P.S=cF12.00 (Á)
Density(c)=11.116

Strong Lines: 3.14/X 1.92/4 2.72/4 1.64/4 1.25/1 1.11/1 1.22/1 0.92/1
Calculated Pattern Original Remarks: Stable above 30 K (2nd ref., Tomaszewski).
Fluorite type.
Minor Warning: No e.s.d reported/abstracted on the cell dimension.
No R factors reported/abstracted.
Wyckoff Sequence: c a(FM3-M).
Unit Cell Data Source: Single Crystal.

2-Theta	d(Å)	I(f)	(h k l)	Theta	1/(2d)	2pi/d	n^2
28.373	3.1431	100.0	(1 1 1)	14.186	0.1591	1.9990	3
32.877	2.7220	35.2	(2 0 0)	16.439	0.1837	2.3083	4
47.182	1.9247	41.1	(2 2 0)	23.591	0.2598	3.2644	8
55.976	1.6414	35.1	(3 1 1)	27.988	0.3046	3.8279	11
58.701	1.5716	7.7	(2 2 2)	29.350	0.3182	3.9981	12
68.940	1.3610	5.1	(4 0 0)	34.470	0.3674	4.6166	16
76.160	1.2489	11.5	(3 3 1)	38.080	0.4003	5.0308	19
78.511	1.2173	8.4	(4 2 0)	39.255	0.4107	5.1615	20
87.765	1.1113	8.5	(4 2 2)	43.882	0.4499	5.6542	24
94.652	1.0477	7.6	(5 1 1)	47.326	0.4772	5.9971	27

106.339	0.9624	2.6	(4 4 0)	53.170	0.5195	6.5289	32
113.669	0.9202	7.9	(5 3 1)	56.834	0.5434	6.8280	35
116.199	0.9073	4.0	(6 0 0)	58.099	0.5511	6.9249	36
126.988	0.8608	4.1	(6 2 0)	63.494	0.5809	7.2995	40
136.201	0.8302	3.3	(5 3 3)	68.101	0.6023	7.5683	43
139.625	0.8207	2.8	(6 2 2)	69.812	0.6092	7.6558	44

RPL K Basin Project 52578	Document Review Record

Title:	XRD Report: KW 210 Container Composite

Please return the completed form to: Doinita Neiner		Comments Due:
Organization/Department:	**Designated Reviewer:**	**Reviewer Signature/Date:** (Upon completion of review)
D9H63	**Mike Urie**	MW Urie 6/29/10

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
See attached "Track Changes" Copy of Report for all Comments/Corrections/Edits.				
1	General	O	Throughout report references are made to specific 2-theta angles. It would be very beneficial for all detected 2-theta angles above a threshold be included for each sample in the appendices.	Reject → we have a PDF card.
2	Page 1	E	Analysis date does not correspond with analysis date on page 2	Done
3	Page 3	E	Lots of spelling errors in "Comment" section. Also, should the "XRD Operating Parameter" section be completed?	
4	Page 5	E	Sample ID(s) TI008-SJ TiO_2: There is inconsistency in terminology, such as Sample ID(s) are called "XRD Mount ID" on page 6 and sample is shown as TI008-SJ_TiO_2. Similar inconsistencies in sample naming are throughout the report. Use of underscore vs hyphen is routine. How sample IDs or XRD Mount IDs are presented in the report need to be consistent so that there is no possibility of confusion of sample names. This DRR form will not address this issue for every inconsistency … attached report highlights many of these sample naming inconsistencies.	Done
5	Page 6	M	Page 9 states "…. 2-theta with a step size of 0.02°/minute. Page 6 under "Other XRD scan parameters" states "2 deg/minute…."	corrected to 0.02 stepsize
6	Page 7,Tab 1	M	Two places: 01-070-3755 shown as ICDD for UO_2 … this should be 01-072-4553	Done
7	Page 7,Tab 1	E	Under Formula, degrees are not displayed consistently.	Done
8	Page 7,Tab 1	E	Table references ICDD Card Number. Appendix 6 references PDF Cards. Need to be consistent.	correct to ICDD
9	Page7, Tab1	E	Title is confusion. Suggest "Summary of Phases identified in KW210 Container Composite"	Done
10	Page 8,1st Para	E	Change "has been" to "was", "XRD slides preparation" to either "XRD slide preparation" or "XRD slide preparations"	Done
11	Page 8,1st Para	E	Suggest adding "prior to samples being submitted for XRD analysis" at end of second sentence.	Done
12	Page 8, 2nd Para	E	Second sentence … "Typically" implies that some other method other than the one described was used on one or more sample preparations. Suggest deleting "Typically".	Reject Typical prep for all slides
13	Page 8,Tab2	E	"Client Sample ID" is shown as "Sample ID" on Request Form	Done

14	Page 8,Tab2	E	Sample Description: In all tables and all text, the sample description should be consistently presented.	Done
15	Page 9	E	"2 theta" or "2-theta" … need to be consistent throughout report	Done
16	Page 9	M	44 kV and 40 mA shown as 40 kV and 44 mA on benchsheet; one is incorrect.	corrected
17	Page 9 and after	E	There is no reason to write out the entire name of the procedure every time that procedure number is referenced. This simply adds superfluous text! This is especially objectionable when the procedure is referenced multiple times in same paragraph.	Done
18	Page 9, 2nd Para	M	SRM 1976 appendix reference is incorrect. It should be "5"	Done
19	Page 9, 2nd Para	E	Aforementioned is one word	Done
20	Page 9, S 3.1	E	If ICP data was available, a statement should be made that all major element identified by ICP were included in the search element list	No; it was not.
21	Page 9, S 3.1	E	"… hydrates; zirconium metal, and zirconia…" Recommend changing to "… hydrates; and zirconium metal and zirconia …" to be consistent with other entries in the sentence.	Done
22	Page 9, S 3.2	E	What does "account for instrument profile" mean in this sentence.	Rejected
23	Page 10, S 4	E	Suggestions: Replace "made" with "comprised". Change "The small fraction contains in addition to quartz, uraninite" to "The small fraction samples contain uraninite in addition to quartz.	Done
24	Page 10, S 4	E	Probably need to insert "uranium" prior to first "oxyhydroxide". Suggestion: Add semicolon before "aluminum" and add "; or" before "zirconium"	Done
25	Page 11, Tab3	E	Sample descriptions need to be consistent throughout report; e.g., use "<500" or "less than 500".	Done
26	Page 12	E	35° - 36°? are there numerous peaks identified in this "range" or should this be "35°, 36°"?	corrected
27	Page 13	E	Suggest changing "and uranium oxide" to "and uranium oxides (UO_2, U_4O_9)"	Done
28	Page 14	E	Suggest changing "…(quartz), uranium oxide" to " … (quartz) and uranium oxides (UO_2, U_4O_9)"	Done
29	Page 19	M	First sentence references Scintag, last sentence references date 02-09-2011. Need to ensure Daily calibration data presented is from Rigaku and need to change system and date references. Figure 15 needs to be Figure 8.	Done
30	Page 19	E	The explanation in paragraph that describes table headings would be better if each heading was in a list (e.g., a) 2T(r) = … b) 2T(o) = … etc.	Done
31	Page 22	M	First sentence references Scintag and there is a reference to Figure 17 (which should be 8).. Need to ensure Quality Control/Calibration data presented is from Rigaku and need to change system and date references. Is 01-06-2011 date correct?	Done
32	Page 22	M	Last sentence reference PDF card being in Appendix 5. Appendix 5 does not contain the 2T(r) displayed in Table 5; these values come from PDF card in Appendix 6. Appendix 5 seems to merely provide acceptance 2T ranges.	Corrected
33	Page 22	E	The explanation in paragraph that describes table headings would be better if each heading was in a list (e.g., a) 2T(r) = … b) 2T(o) = … etc.	Done
34	Page 23	M	Figure 17 should be Figure 8.	Done

Concur with comment resolution, review complete. Michael W Thien 6/29/11 Reviewer Signature Date	Comments Resolved By: D Meiner 6/29/11 Author Signature Date

OPERATOR	DATE	TIME	SAMPLE ID	FILE	RANGE	SETTINGS	PATH	COMMENT
DN	01/06/11	10:00	Si_ddex_010611b	Si_ddex_010	27-29		xrd\dorinita	
DN	01/06/11	11:00	Si-ddex_010611c	Si-ddex_010	5-65		\dorinita	
DN	01/06/11	12:00	misf-1976-ddex_010611d		5-65		xrd\dorinita	
DN	01/12/11	5:20 pm	Si_011211c	Si_011211c	27-29	0.02/4.		
DN	01/12/11	4:00	T1021_G_011211b	T1021_G_011211	5-65			
DN	01/12/11	5:00	T1021_H_011211c	T1021H_011211	5-65	0.02/4	xrd/dorinita/dev2011	
DN	1/19/11	9:30	Si_01/19/11b		10-70	0.02/5	xrd/dorinita/dev.2011	
DN	01/19/11	10:00am	Na3PO4_2hr_70_011911a		10-70	0.02/5	xrd/	
DN	01/19/11	11:00	Na3PO4_2hr_70_011911c		10-70	0.02/1		
DN	01/19/11	17:00	Na2HPO4_2hr_70_011911d		10-70	0.02/1		
DN	2/9/11	11:30	EDBB_020911a		5-80	0.02/1	xrd/dorinita/EDBB	
DN	2/9/11	2:15	TeO2_62026_020911b		10-70	0.02/2	xrd/dorinita/Randy 2011	
DN	2/9/11	4:20	Tb4O7_p80_SB12_020911c		10-70			
DN	2/10/11	4:30	Tb_oxide_lot3_029		10-70		xrd/dorinita ... /cindy	
DN	2/14/11	1:30 pm	AgCl_MOFTH_021411a		10-70			
DN	2/17/11	1:40 pm	AgCl_MOFZ4_021711a		10-60		/cindy	
DN	2/17/11	2:00	M250 tmu oxide_021711b		10-70	0.02/10 S.	/cindy	
DN	2/18/11	8:30	Mn_doped_TiO2_021811a		10-80	0.02/0.1 S.	/cindy	
DN	2/22/11	12:00	MnO2_cachi_SO2_022111a		10-80	0.02/0.1 S	/cindy	
DN	2/24/11	3:20	Si_NIST_640a_022411a		27.4-29.4	0.02/10 S	/dorinita	
DN	2/24/11	3:30 pm	Mn doped FeO_022411b		10-110	0.02/1 S	/cindy	
DN	Something happened(?) computer was restarted and the power was down for 20kV, 2 mA.							
DN	Power up redux OK!							
DN	2/25/11	1:45	Mn doped FeO_022511a		10-110°	0.02/0.1 S.	/cindy	

OPERATOR	DATE	TIME	SAMPLE ID	FILE	RANGE	SETTINGS	PATH	COMMENTS
Si-640								
DN	03/01/11	11:00	Si_standard 640a	030111a	27.4-29.4	0.002/4.5	/dorinda/kenan	
DN	03/01/11	11:15	Gwinnedite	030111b	10-95	0.02/0.15	/dorinda/kenan	
DN	03/07/11	2:00		030411c	10-80	0.02/0.15		
DN	03/07/11	9:30	GWinnedite	030711a	10-80	0.02/15		
DN	03/17/11	4:30	03/7/11a-VO_2_UO_3		5-90	0.02/0.15	/dorinda/carl/	
DN	03/12/11	2:00 pm	04/8/11 a - Si-Nist640		10-110	0.02/15	/dorinda/carl/	
DN	4/13/11	1:30 pm	04/13/11 a - Fe_2O_3_std		10-110	002/15	/dorinda/cindy/	
DN	4/14/11	2:30 pm	04/14/11a - Mn_doped0.1%		10-110	0.02/15		
DN	4/18/11	10:00 am	4/18/11a - Mn_doped0.1%		10-110	0.02/15		
DN	4/22/11	4:10 pm	4/22/11a - Luke 1		5-95	0.02/205	/Luke/Dorinda	
DN	4/29/11	4:20	4/28/11b - Luke 2		5-95	0.02/405	Luke	
DN	5/5/11	2:00	050511a - Si_NIST640a		27.-29	4	Dorinda	
DN	5/5/11	2:30	050511b - Mn7+ PO_4.H_2O		5-65			
DN	5/17/11	4:30	051711a - Si_Nist640a		27-29	SC		
DN	5/17/11	4:45	051711b - Si_Nist640		27-29	Dtex	Dorinda	
DN	5/17/11	4:50	051711c - UO_3-Y12		5-70	Dtex		
DN	5/17/11	5:00	051711d - UO_2-H_2O?		5-90	Dtex	Luke	
DN	5/18/11	9:15	Si_640a - 051811a		27-29	Dtex, 0.02, 40 deg/s	Dorinda	
DN	5/18/11	9:40	RuO_2 - 051811b		10-110	Dtex, 0.02, 0.1 s	Dorinda/Randy	
DN	5/19/11	10:00	Si_nist - 051911a		27-29	Dtex, 0.02, 10 deg/s	Dorinda/Randy	
DN	5/19/11	10:15	RuO_2 - 051911b		10-70	Dtex, 0.02, 20 deg/s	Dorinda/Randy	
DN	5/19/11	10:30	$RuTy$ - 051911c		10	"	"	
DN	5/19/11	10:45	$RuTy$ - 051911d		"	0.02	5 deg/min	
DN	5/19/11	10:10	Mn_2O_3 only - 051911e	051911e	10-110	0.02	1 deg/min	Dorinda/Cindy
DN	5/19/11	1:10	Mn_doped0.3% - A-Ulg	00C		0.02	0.5 deg/min	
DN	5/20/11	10:50	052011a - Si					
DN	5/23/11	12:00	052311a - Si					Luke
DN	5/23/11	12:10	052311b - Atlanta - $RuTy$	one	10-80	0.02/1 deg/min		Dorinda/Cindy
DN	5/24/11	9:30	052411a - Si		27-29	0.02/10 deg/min		Dorinda/Randy
DN	5/25/11	9:45	052511a - Si		5-65	0.02/2 deg/min		Dorinda/Kenny
DN	5/25/11	11:40	T1008_MH_052511b		"	"		Dorinda/Kenan
DN	5/25/11	1:30	T1008_MH_052511c		"	"		
DN	5/25/11	2:30	T1008_SH_052511		"	"		
DN	5/25/11	4:00	T1008_SH_052511		"	"		
DN	5/25/11	4:30	T1008_UO_2-UO_3		"	"		
DN	6/1/11	11:30	Si - 060111c		27-29	0.02/20 deg/min		
		12:00	SRL - 060111b		5-70			
			SRL - 060111c		5-70			

OPERATOR	DATE	TIME	SAMPLE ID	FILENAME	RANGE	SETTINGS	PATH	COMMENTS
DN	6/1/11	12:00	SP3_060111d	SP3_0 60111	5-70	0.02/ 2deg/min	DTEX ,	deionized water
"	"	1:45	sample 1 — 060111e	sample 1_0 60111e	"	"	"	"
"	"	1:30	sample 2 — 060111f	sample 2_060111f	"	"	"	"
"	"	2:00	sample 3 — 060111g	sample 3_060111g	"	"	"	"
"	"	2:15	sample 4 — 060111h	sample 4_060111h	"	"	"	"
"	"	2:30	sample 5 — 060111i	sample 5_060111i	"	"	"	"
"	"	3:00	NaTFPA — 060111j	NaTFPA_060111j	"	"	"	"
"	"		gibbsite_NK — 060111k	gibbsite_NK_060111k	"	"	"	"
DN	6/2/11	2:15	gibbsite_060211a	gibbsite_060211a	5-95	0.02, 0.1 deg/min ,	SC detector	deionized water
LS	6/3/11	10:57	YSmPO4_060211a	YSmPO4_060211a				
LS	6/3/11	12:01	060311b	3A	5-90	3 deg/min	DTEX	As 3 A
LS	6/3/11		060311d		5-90	1 deg/min	DTEX	
"	"	12:40 AM	CC1611a		5-70	45 deg/min	DTEX	* α-UO2(OH)2
"	"	10:36 AM	CC1611d		5-70	3 deg/min	DTEX	AS-03-B
"	6/13/11	11:20 AM	CC1311a — AS-17-B		5-70	3 deg/min	DTEX	AS-17-B
"	6/13/11	12:04 PM	CC1411g — AS-2-THA		5-70	3 deg/min	DTEX	AS-24-A
DN	6/14/11		CC1411b — Sr_UO2H6		23 - 29	2deg/min		Gibbsite (from deionized water)
DN	6/14/11	1:15	CC1411d — Sr_SiO4		10 - 30	2deg/min	DTEX	
"	6/14/11		CC1411e — AS-3-UO2H6		5-70	20 deg/min	DTEX	Nitsr Si Standard
"	6/14/11		CC1411f — AS-3-B		5-70	3 deg/min	DTEX	AS-06-D2
"	6/14/11	3:45 PM	CC1411h — AS-12-A1		5-70	3 deg/min	DTEX	AS-3-B
"	6/15/11	12:03 PM	CC1411i — AS-12-A1		5-70	3 deg/min	DTEX	AS-12A1
"	6/15/11		CC1511a — AS-2-A		5-70	3 deg/min	DTEX	AS-02-A
DN	6/15/11		CC1511b — CaCO3_Ic611c		5-80	10 deg/min		deionized water
DN	6/15/11	5:10	CC1511c — Ca(OH)2		2-50	5 deg/min		
DN	6/16/11	11:30	103.DI_CC1611c		3-80	2 deg/min		
-S	6/17/11	11:12	CC1511g — AS-32		5-70	3 deg/min	DTEX	AS-32-E
-S	6/17/11	12:10	CC1711b — AS-19-A		5-70	3 deg/min	DTEX	AS-19-A
N	6/17/11	2:30	061711c — C2O3_di		3-80	2 deg/min	DTEX	deionized water
J	6/17/11	3:00	061711d — C2O3_Ca(OH)2		3-80	2 deg/min	DTEX	
	6/17/11							
	6/20/11	9:45	062011a — C2O2_CaCO3		3-80	0.02	DTEX	deionized water
V	6/20/11	11:20	062011b — C2O2_Ca(OH)2		3-80	2 deg/min		
N	6/20/11		062011c — C102_CaCO3		3-80			
J	6/20/11	2:45 pm	062011d — synthetic oxysalt		3-80	0.02	DTEX	deionized/ammonia
	6/20/11	3:45 PM	062011e — AS-6-D1		5-90	3 deg/min		
	6/21/11	10:20 AM	062101a — AS-9-B		5-90	3 deg/min	DTEX	AS-6-D1
	6/21/11	2:30 PM	062111b — Si_std		27-29	10 deg/min.	DTEX	AS-8-B
J	6/21/11	2:45	062111c — ammonium 2.		5-80	10 deg/min		
J	6/21/11	3:00	062111d — ammonium 2		0.02			link

OPERATOR	DATE	TIME	SAMPLE ID	FILENAME	RANGE	SETTINGS	PATH	COMMENTS
NV	6/21/11	3:15	062111e_borehole 1	062111e	5-80	0.02 / 20 deg/min	\ hranice	
NV	〃	3:30	062111f_gibbsite peak	062111f	5-80	0.02 / 10 deg/min	\ hranice	
NV	6/21/11	3:39	062111g_borehole 2	062111g	〃	〃	〃	
NV	6/21/11	3:45	062111h2_borehole 3	062111h2				
NV	〃	3:55	062111i_borehole 4	062111i				
NV	〃	5:00	062111j_kaolinite_2	062111j	3-80	0.02 / 0.5 deg/min	〃	\ kaolinite
NV	6/22/11	5:00	062211a_C202_NT	062211a	〃	0.02 /	〃	
NV	6/23/11	10:15	062311a_C403_DT_Ca(OH)2	062311a	3-80	0.02		\ kaolinite
NV	6/30/11	9:48	063011a_Si_640c	063011a	27-29	0.02 / 10 deg/min		\ bohmite
NV	6/30/11	3:00	063011b_1976_Alumina plate	063011b	5-80	0.02 / 10 deg/min		\ gibbsite
NV	6/30/11	3:10	063011c_sodalite 3	063011c	5-80	0.02 / 5 deg/min		\ dolomite / Fraunhofer
NV	6/30/11	3:25	063011d_sodalite 4	063011d	5-80	0.02 / 5 deg/min		\ dolomite / Fraunhofer
NV	6/30/11	10:00	070111a_Si_640c	070111a	27-29	〃	〃	
NV	7/1/11				5-65	0.02 / 0.5°/min		\ dolomite / Kbefin
NV	6/30/11	4:00	063011e_T1040-421D-2x	063011e	27-29	0.02 / 0.5 deg/min		\ dolomite / Kbefin
NV	7/1/11	10:00	070111a_Si_640c	070111a	5-65	0.02 / 0.5 deg/min		\ dolomite / Kbefin
NV	7/1/11	10:20	070111b_T1040-420D-2x-H_2O	070111b	5-90	0.02 / 1.5 deg/min		\ dolomite / Kbefin
LS	7/1/11	2:00	070111c-H_2O-2	070111c	27-29	0.02 / 10 deg/min		$H_2O \cdot 2H_2O$ - pg 122
AN	7/5/11	12:20	070511a_Si640a	070511a	5-85	0.02 / 0.5 deg/min		\ dolomite / Kbefin
SN	7/5/11	12:30	070511b_T1040_420D-2xH_2O	070511b	〃	〃	〃	
SN	7/5/11	12:45	070511c_T1040-419D-2x	070511c	〃	〃	〃	
SN	7/5/11	5:00	070511d_T1040-422D-2x	070511d	〃	〃	〃	
DN	7/6/11	1:10	070611a_Si640a	070611a	27-29	0.02 / 10 deg/min		
DN	7/6/11	1:20	070611b_T1040-420D-2x	070611b	5-65	0.02 / 0.5 deg/min		
DN	7/6/11	3:40	sodalite 5_070611c	070611c	5-85	0.02 / 1 deg/min		\ dolomite / Fraunhofer
DN	7/6/11	3:48	sodalite 6_070611d	070611d	〃	〃	〃	
DN	7/6/11	4:00	kaolin_070611e	070611e	〃	〃	〃	
DN	7/6/11	4:10	Al-hydrate_070611f	070611f	〃	〃	〃	
DN	7/6/11	4:20	070611g_T1040_793D_2x	070611g	5-65	〃	〃	\ dolomite / Kbefin
S	7/7/11	3:44	070711a - alpha-Al_2O_3	070711a	5-90	0.02/16 deg/min		alpha - Al_2O_3 - pg 122

PROJECT 52578 – SURVEILLANCE REPORT

Number: SR-52578-2011-029

PLANNING

Topic/Issue to be Evaluated: Surveillance of K Basin X-Ray Diffraction Analysis Report; KW 210 Container Composites

This surveillance was undertaken to evaluate the draft *X-Ray Diffraction (XRD) Analysis Report, KW 210 Container Composites* for the samples listed in Table 1.

PERFORMANCE

Date Initiated: 6 April 2011	**Location:** RPL
Date Received: 1 July 2011	**Date Reviewed:** 6 & 7 July 2011
Date Resubmitted: 20 July 2011	**Date Closed:** 20 July 2011
Date Completed: 20 July 2011	**DRAFT Report Issued:** 8 July 2011 **FINAL Report Issued**: 20 July 2011
Contacts:, Clark Carlson, Sandy Fiskum, Doinita Neiner, Mike Urie	**Org Code:** PE137, D9H63
Surveillance Team: Lisa Middleton	

PRELIMINARY INFORMATION

Source of Requirement(s):

- KBC-33786, Rev. 2, December 2009; Quality Assurance Project Plan/Sampling and Analysis Plan (QAPjP/SAP): Quality Assurance Project Plan/Sampling and Analysis Plan for Containerized KW Settler Sludge

Other Applicable Documents:

- RPL-OP-001, Rev. 9, *Routine Research Operations*
- PNNL-RPG-268, Rev. 4, *Solids Analysis by X-Ray Diffraction*
- 52578-ADM-001, Rev. 0, *K Basin Requests for Analysis and Guidelines for Data Reporting and Technical, QE, and Spreadsheet Reviews.*

Number: SR-52578-2011-029

Purpose/Scope:

The 52578 Project QE staff are required via the QA Program to perform a review of K Basin reports generated within the Radiochemical Processing Laboratory (RPL) to determine traceability and accuracy of data contributing to the completed report.

The draft report was reviewed to evaluate that the data are what they purport to be, that is, that the reported results reflect what was actually done and the results meet applicable requirements. When deficiencies in the data are identified, then those deficiencies need to be documented as a K Basin Occurrence Report for the data user's review and, when applicable, resolved by corrective action. The XRD analysis under this Analysis Request was performed to identify crystalline structures in room-temperature-dried sludge, to identify hydrate compounds plus any other identifiable compounds. This includes noting the presence (or absence) of aluminum hydrates, uranium hydrates, uranium oxides as well as zirconium metal.

When issues were found, the cognizant staff were contacted to resolve issues prior to completion of the review.

Approach:

The draft report initiated by a K Basin Analysis Request form dated 4/6/11 was provided as the basis for the data quality review. Four (4) samples were submitted on 4/6/11 for analysis as noted in Table 1:

Table 1: XRD Samples IDs & Descriptions

Client Sample ID	Sample Description	XRD Sample ID	Internal Standard	Date Analyzed	Daily Performance Check w/ 640a?
TI008-MH	-2000/+500 µm, air-dried solids	TI008-MH	None	5/25/11	Y
TI008-MJ	-2000/+500 µm, air-dried solids duplicate	TI008-MJ	None	5/25/11	Y
TI008-SH	-500 µm, wet slurry	TI008-SH	None	5/25/11	Y
TI008-SJ	-500 µm, wet slurry duplicate	TI008-SJ	None	5/25/11	Y
		TI008-SJ-TiO2	Rutile	5/25/11	Y

All samples were to be analyzed without internal standard, one sample (TI008-SJ) was to also be run with rutile, creating 5 samples total.

The report contained the following information:

1. Cover page
2. XRD Analysis Request Form/ XRD Mount Preparation Signed by Sandy Fiskum
3. XRD Analysis Bench Sheet – Sample Preparation
4. XRD Analysis Bench Sheet – Diffraction Measurements

PROJECT 52578 – SURVEILLANCE REPORT

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5. Report
    a. Table 1 – Summary of Phases Identified in KW210 Container Composites
    b. Sample preparation
    c. X-Ray powder diffraction data collection
    d. X-Ray powder diffraction data analysis
    e. Summary of findings
    f. Sample Analyses
    g. Appendix 1 – SRM 674 NIST Certificate
    h. Appendix 2 – Daily Performance Checks – 5/25/11
    i. Appendix 3 – SRM 640a NIST Certificate
    j. Appendix 4 – Quality Control/Calibration – 1/6/11
    k. Appendix 5 - -SRM 1976 NIST Certificate
    l. Appendix 6 – PDF cards for the identified crystalline phases presented in report
    m. DRR form signed by the Technical Reviewer, Mike Urie 4/29/11
    n. Copy of XRD run log, 2 pages

M&TE used:

Location	Description	Manufacturer	Model	Unique ID
RPL/409	X-Ray Diffractometer	Rigaku	Ultima IV	GD 3827N /WD59716
	RGD			#105
	SRM	NIST	640a	640a
	SRM	NIST	674	674
	SRM	NIST	1976	1976

Mike Urie was identified as the technical reviewer and this was documented with a DRR. During the QE review, it was found that 11 of the 34 comments from the DRR had not been addressed.

The completed surveillance report will be posted to the SharePoint site and submitted as a project record. Any documentation associated with this surveillance will be available in the Lead Auditor's office, Building 325, 5-G.

RESULTS

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.

Summary of Identified Concerns:

Note: All concerns identified in the review were addressed in a comment resolution meeting on 20 July 2011, Deborah Coffey, Lisa Middleton and Doinita Neiner attended to disposition review comments. There were no outstanding identified findings or observations.

Number: SR-52578-2011-029

1. A copy of the XRD logbook needs to be provided for review to verify calibration runs. Provided. Closed.

2. Analysis dates on cover page includes the sample preparation dates. Analysis was only done on 5/25/11. This was also noted on the DRR (#2), but not addressed. Analysis dates corrected to 5/25/11 only. No further action required. Closed.

3. Page 2: The sample prep days are incorrect according to the bench sheets provided in pages 4 & 5 of the report. The only days listed for sample preparation are 5/19/11 & 5/24/11. There was no sample prep on 5/25/11 so the sample prep dates should be 5/19/11 – 5/24/11, not 5/19/11 – 5/25/11. Sample prep fixed (5/19/11 – 5/24/11). No further action required. Closed.

4. XRD Analysis Request Form: There are multiple typos in the *Comments* section; see comment copy of report. This was also noted on the DRR (#3), but not addressed. Errors fixed by S Fiskum, new version of XRD Analysis Request Form included in report. No further action required. Closed.

5. XRD Analysis Request Form: The XRD Mount Preparation Section & XRD Operating Parameters Section has not been completed. These sections should have been completed before analysis. Bottom section completed; section is redundant with use of new bench sheets. No further action required. Closed.

6. XRD Analysis Bench Sheets, pages 4-6: These are not the correct bench sheets. The format that was agreed upon has been resent in an e-mail dated 7/7/11. The section identifying which standard was used has been omitted. Correct bench sheets were provided, however, they still need to be paginated. Sheets paginated. No further action required. Closed.

7. Page 8: Table 1 is never addressed in the report, it needs an introduction. Figures can't just be put into a report without discussing them. Discussed. No further action required. Closed.

8. Page 8: Typo in 1st paragraph. Change "has been" to "was". This was also noted on the DRR (#10), but not addressed. Changed. No further action required. Closed.

9. Page 8: Add "prior to samples being submitted for XRD analysis" at the end of the second sentence. This was also noted on the DRR (#11), but not addressed. Fixed adequately. No further action required. Closed.

10. Page 8: Typo: "slides" in the 4th sentence of the 1st paragraph should be singular, "slide". This was also noted on the DRR (#10), but not addressed. Corrected. No further action required. Closed.

11. Sample Preparation, page 8: In the second sentence of the second paragraph, it says "Typically, the sample was mixed with approximately 2 mL of amyl acetate in a 20-mL glass scintillation vial to produce a slurry." This statement implies that the above is the general protocol for sample preparation but that maybe it wasn't followed. Either the sample was mixed with amyl acetate to produce a slurry or it wasn't. This ambiguity needs to be cleared up. This was also noted on the

DRR (#12), but not addressed. Removed "typically," sample was mixed with amyl acetate. No further action required. Closed.

12. Page 9, Section 2, 3rd paragraph: Do not write out the name of the procedure after the 1st time, only the procedure # needs to be used. This was also noted on the DRR (#17), but not addressed. Removed. No further action required. Closed.

13. Page 9, Section 2, 3rd paragraph: The acronym ICDD needs to be defined. Defined as "International Center for Diffraction Data". No further action required. Closed.

14. Page 9 section 2, 3rd paragraph: Aforementioned is one word. This was also noted on the DRR (#19), but not addressed. Corrected. No further action required. Closed.

15. Page 9, section 3.1: "…hydrates; zirconium metal, and zirconia…" should be changed to "…hydrates; and zirconium metal and zirconia…" to be consistent with the other entries in the sentence. This was also noted on the DRR (#21), but not addressed. Changed. No further action required. Closed.

16. Page 9, section 3.1: The acronym PDF needs to be defined or changed to ICDD if they are interchangeable. Just using one term eliminates confusion. PDF defined as Powder Diffraction File. Not interchangeable. No further action required. Closed.

17. Page 9, section 3.2: What does "account for instrument profile" mean in this sentence? This was also noted on the DRR (#22), but not addressed. Sentence removed. No further action required. Closed.

18. Page 10, 1st paragraph: Change PDF to ICDD. Discussed, leave as-is. No further action required. Closed.

19. Page 13: Add chemical formulas for uranium oxides as DRR #27 suggests and is noted as "done". Formulas added. No further action required. Closed.

20. Page 22, Appendix 4: "Is" in the second sentence should be changed to "are". "Data" is plural. Changed to "are." No further action required. Closed.

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CORRECTIVE ACTION

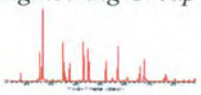
[X] None Required. [ ] Complete [ ] Follow up Corrective Action

Surveillance Performed By:

Lisa Middleton, Quality Engineer LSC 7.20-11

Date: 7/20/11

DISTRIBUTION: Clark Carlson, Sandy Fiskum, Doinita Neiner, Andy Schmidt, Rick Shimskey, Mike Urie, Deborah Coffey, Lisa Middleton



X-Ray Diffraction Analysis Report

Battelle—Pacific Northwest National Laboratory
Radiochemical Sciences and Engineering Group
PO Box 999, Richland, Washington 99352

Project: #52578
Sample ID: KW 210 Undissolved Solids

Procedure: PNNL-RPG-268 Rev. 4, "Solids Analysis by X-Ray Diffraction"

Analyst: Doinita Neiner
Analysis Dates: 06/30/2011-07/06/2011
Instrument: RGD # 105
Property Number: WD 59716
Serial Number: GD 3827N

D Neiner 7/26/11
Prepared By Doinita Neiner /Date

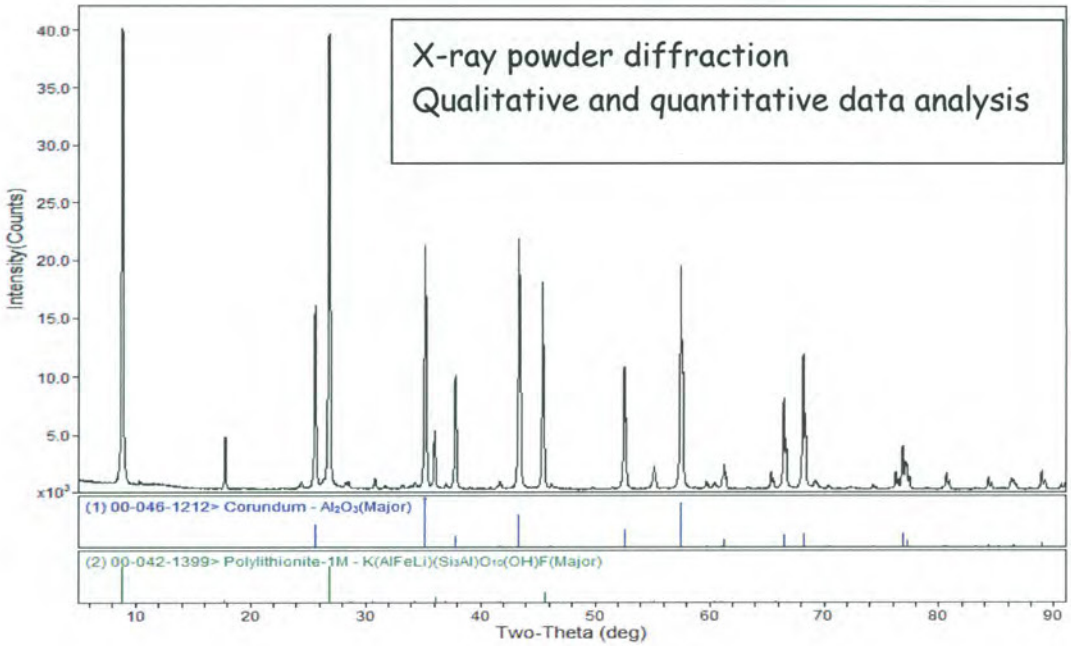
7/26/11
Reviewed By Mike Urie /Date

7/27/11 dsc 7-27-11
QE Reviewed By Deborah Coffey/Date

7-27-2011
Project Management Release Clark Carlson/Date

Battelle - Pacific Northwest National Laboratory *Radiochemical Sciences and Engineering Group*

X-Ray Diffraction Data Analysis

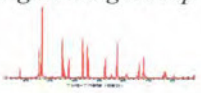


Project Number: 52578
Charge Code: H41223
Client: S. Fiskum
Total Samples: 6 solids (See results for Client IDs)

Analysis Procedure	PNNL-RPG-268 Rev. 4, "Solids Analysis by X-Ray Diffraction"
Prep Procedure	Slurry of amyl acetate and Krylon on a glass slide covered with Kapton
Analyst	Doinita Neiner
Analysis Date	June 30, 2011 through July 6, 2011
Sample Prep. Date	June 23, 2011 through June 30, 2011
Reference Standards	Rutile – SRM 674, PDF 01-077-0440
Software **Reference database**	Jade MDI 9 ICDD PDF-2 2009
M&TE Numbers	XRD System (WD 59716), RPL/317

Battelle - Pacific Northwest National Laboratory *Radiochemical Sciences and Engineering Group*

X-Ray Diffraction Data Analysis



XRD Analysis Request Form

Requester	SK Fiskum	**Work Package**	H41223
Signature	SK Fiskum	**Project Number**	52578
		Subtask	XRD
Date Submitted:	6/16/11	**Date Required:**	7/5/11

Treatability Study? ☑ no ☐ yes; if yes, start date:

Sample ID	Sample Description	Internal Standard Request
TI040-419D-2x	KW210-A1 undissolved solids (UDS)	☐ Rutile ☐ Alumina ☑ None
TI040-420D-2x	KW210-A4 UDS	☑ Rutile ☐ Alumina ☑ None
TI040-421D-2x	KW210-B1 UDS	☐ Rutile ☐ Alumina ☑ None
TI040-422D-2x	KW210-B3 UDS	☐ Rutile ☐ Alumina ☑ None
TI040-792D-2x	KW210-CC, -2000/+500 µm UDS	☐ Rutile ☐ Alumina ☑ None
TI040-793D-2x	KW210-CC, -500 µm UDS	☐ Rutile ☐ Alumina ☑ None

Comments: All samples are undissolved solids (UDS) remaining from the KW210 core samples and container composite samples that had been acid digested according to PNL-ALO-129. The UDS contained resin beads; these beads were manually removed (best effort basis) from the non-resin material.

Identify crystalline structures in room-temperature-dried sludge. Identify hydrate compounds plus any other identifiable compounds. This includes noting the presence (or absence) of aluminum hydrates, uranium hydrides, uranium oxides as well as zirconium metal. The analytical composition (ICP-OES analytes) will be provided upon availability. Uranium is expected to be a dominant component.

All samples are to be analyzed without internal standard; one sample (selected choice above can be altered depending on quantity of sample available) is to also be run with internal standard rutile (TiO_2). Rutile X-ray diffraction angles and relative peak magnitudes will be used to help demonstrate acceptable system performance within the sample matrix and analysis process.

Please use the new Rigaku system for powder diffraction analysis if at all possible. Please use a bench sheet you have developed to support sample processing and analysis and provide this original bench sheet as part of the data package.

Notes for Pattern Analyst:

XRD Mount preparation

Standard Reference Materials ID's: TiO_2: SRM 674 Al_2O_3: SRM676 Other: _____

Fixative: ☐ Collodion ☒ Krylon ☐ Other _____

Mount: ☒ Glass Slide, ☒ Kapton covered ☐ PIP Holder ☐ Other _____

Prepared by: DKeiser (Signature) 07/01/11 (Date)

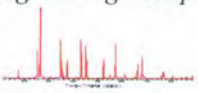
XRD Operating Parameters	X-RAY 2-theta range: 5 – 65 degrees (5-65 default)	
	Step size: 0.02 degrees (0.02 default)	
	Count time: — second(s) (2 default)	☐ Multi-Scans

(Shaded areas to be filled out by the XRD analysis staff.)

Rev. 7 date 09/29/08

Battelle - Pacific Northwest National Laboratory *Radiochemical Sciences and Engineering Group*

X-Ray Diffraction Data Analysis



XRD Analysis Bench Sheet

For Procedure PNNL-RPG-268, Rev. 4,

Solids Analysis by X-Ray Diffraction

Sample Preparation

Print and complete pages as needed in order to document sample preparation for all samples submitted under one Analysis Request.

Sample ID(s) T1040-419D-2X

Sample preparation date (grinding and mounting) 6/23/11

Sample dried air-dried ☐

Internal standard: None ☒ Rutile ☐ Alumina ☐ Other ☐ describe _____

Standard ID: _____

Grinding method: Mortar & Pestle ☒ Mill ☐ Other ☐ describe _____

Fixative: ☒ Krylon ☐ Other

Mount type: Slide ☒ Other ☐ describe _____

Notes: _____

XRD Mount ID(s) T1040-419D-2X

Sample ID(s) T1040-420D-2X

Sample preparation date (grinding and mounting) 6/23/11

Sample dried air-dried ☐

Internal standard: None ☒ Rutile ☐ Alumina ☐ Other ☐ describe _____

Standard ID: _____

Grinding method: Mortar & Pestle ☒ Mill ☐ Other ☐ describe _____

Fixative: ☒ Krylon ☐ Other

Mount type: Slide ☒ Other ☐ describe _____

Notes: _____

XRD Mount ID(s) T1040-420D-2X

Sample ID(s) T1040-421D-2X

Sample preparation date (grinding and mounting) 6/23/11

Sample dried air-dried ☐

Internal standard: None ☒ Rutile ☐ Alumina ☐ Other ☐ describe _____

Standard ID: _____

Grinding method: Mortar & Pestle ☒ Mill ☐ Other ☐ describe _____

Fixative: ☒ Krylon ☐ Other

Mount type: Slide ☐ Other ☐ describe _____

Notes: _____

XRD Mount ID(s) T1040-421D-2X

D Weiner / 6/23/11
Analyst Signature/Date

7/21/11
Reviewer Signature/Date

Battelle - Pacific Northwest National Laboratory *Radiochemical Sciences and Engineering Group*

X-Ray Diffraction Data Analysis

Sample ID(s) T1040-420D-2X
Sample preparation date (grinding and mounting) 6/23/11
Sample dried air-dried ☐
Internal standard: None ☐ Rutile ☒ Alumina ☐ Other ☐ describe _____
Standard ID: TiO_2
Grinding method: Mortar & Pestle ☒ Mill ☐ Other ☐ describe _____
Fixative: ☒ Krylon ☐ Other
Mount type: Slide ☒ Other ☐ describe _____
Notes: _____
XRD Mount ID(s) T1040-420D-2X-TiO_2

Sample ID(s) T1040-422D-2X
Sample preparation date (grinding and mounting) 6/30/11
Sample dried air-dried ☐
Internal standard: None ☒ Rutile ☐ Alumina ☐ Other ☐ describe _____
Standard ID: ~~TiO2~~ DN
Grinding method: Mortar & Pestle ☒ Mill ☐ Other ☐ describe _____
Fixative: ☒ Krylon ☐ Other
Mount type: Slide ☒ Other ☐ describe _____
Notes: _____
XRD Mount ID(s) T1040-422D-2X

Sample ID(s) T1040-792D-2X
Sample preparation date (grinding and mounting) 6/30/11
Sample dried air-dried ☐
Internal standard: None ☒ Rutile ☐ Alumina ☐ Other ☐ describe _____
Standard ID: _____
Grinding method: Mortar & Pestle ☒ Mill ☐ Other ☐ describe _____
Fixative: ☒ Krylon ☐ Other
Mount type: Slide ☒ Other ☐ describe _____
Notes: _____
XRD Mount ID(s) T1040-792-2X

Sample ID(s) T1040-793D-2X
Sample preparation date (grinding and mounting) 6/30/11
Sample dried air-dried ~~☒~~ DN
Internal standard: None ☒ Rutile ☐ Alumina ☐ Other ☐ describe _____
Standard ID: _____
Grinding method: Mortar & Pestle ☒ Mill ☐ Other ☐ describe _____
Fixative: ☒ Krylon ☐ Other
Mount type: Slide ☒ Other ☐ describe _____
Notes: _____
XRD Mount ID(s) T1040-793-2X

D Meiner 6/30/11
Analyst Signature/Date

MW Wilkerson 7/25/11
Reviewer Signature/Date

XRD Analysis Bench Sheet

For Procedure PNNL-RPG-268, Rev. 4,

Solids Analysis by X-Ray Diffraction

Diffraction Measurements

Print and complete pages as needed in order to document sample analysis and daily calibration verification for all samples submitted under one Analysis Request.

Today's date: 06/30/11
XRD daily performance checks with NIST standard run today? ☒ NIST standard: Si 640a
Associated daily calibration check filename 063011a_Si640a
Logbook updated? ☒

XRD mount ID	XRD filename	Notes	Analyst initials
T1040-4210-2x	063011E_T1040_4210_2x		DV

XRD Operating Parameters	
	X-RAY 2-theta range: 5 - 65 degrees (5-65 default)
	Step size: 0.02 degrees (0.02 default)
	Other XRD scan parameters: 0.5°/min DtéX, 40KV, 44mA

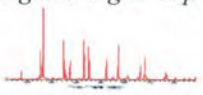
Today's date: 07/01/11
XRD daily performance checks with NIST standard run today? ☒ NIST standard: Si 640a
Associated daily calibration check filename 070111a_Si640a
Logbook updated? ☒

XRD mount ID	XRD filename	Notes	Analyst initials
T1040-4200-2x-TiO2	070111b_T1040_4200-2x-TiO2		DV

XRD Operating Parameters	
	X-RAY 2-theta range: 5 - 65 degrees (5-65 default)
	Step size: 0.02 degrees (0.02 default)
	Other XRD scan parameters: 0.5°/min, DtéX, 40kV, 44mA

DVeinese 07/01/11
Analyst Signature/Date

MLO Thom 7/21/11
Reviewer Signature/Date



Today's date: 07/05/11
XRD daily performance checks with NIST standard run today? ☒ NIST standard: Si 640a
Associated daily calibration check filename 070511a - Si640a
Logbook updated? ☒

XRD mount ID	XRD filename	Notes	Analyst initials
T1040-420D-2X	070511b-T1040-420D-2x		DN
T1040-419D-2x	070511C_T1040-419D-2x		DN
T1040-422D-2X	070511D_T1040-422D-2x		DN

XRD Operating Parameters	
	X-RAY 2-theta range: 5 - 65 degrees (5-65 default)
	Step size: 0.02 degrees (0.02 default)
	Other XRD scan parameters: 0.5°/min, Dtex, 40kV, 44 mA

Today's date: 07/06/11
XRD daily performance checks with NIST standard run today? ☒ NIST standard: Si 640a
Associated daily calibration check filename 070611a - Si640a
Logbook updated? ☒

XRD mount ID	XRD filename	Notes	Analyst initials
T1040-792D-2x	070611B_T1040_792D_2x		DN
T1040-793D-2x	070611G_T1040_793D-2x		DN

XRD Operating Parameters	
	X-RAY 2-theta range: 5 - 65 degrees (5-65 default)
	Step size: 0.02 degrees (0.02 default)
	Other XRD scan parameters: 0.5°/min, Dtex, 40kV, 44 mA

DWeiner / 07/06/11
Analyst Signature/Date

MW Thm 7/21/11
Reviewer Signature/Date

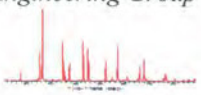
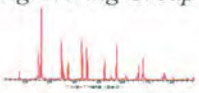


Table 1. Summary of Phases Identified in KW210 Undissolved Solids

Sample ID	PDF Card Number	Relative Intensity[(a)]	Formula	Common Phase Name
TI040-419D-2X	01-070-3755	Major	SiO_2	Quartz
	01-089-1460	Major	$Ca(Al_2Si_2O_8)$	Anorthite
	--	Unassigned		
TI040-420D-2X	01-070-3755	Major	SiO_2	Quartz
	01-089-1460	Major	$Ca(Al_2Si_2O_8)$	Anorthite
	01-071-1403	Minor	$Ca_{0.466}Fe_{0.5185}Al_{0.815}Na_{0.534}O_6Si_2$	Aegerine
	01-070-0909	Minor	$CaSO_4$	Calcium sulfate
	--	Unassigned		
TI040-421D-2X	01-070-3755	Major	SiO_2	Quartz
	01-089-1460	Major	$Ca(Al_2Si_2O_8)$	Anorthite
	--	Unassigned		
TI040-422D-2X	01-070-3755	Major	SiO_2	Quartz
	01-089-1460	Major	$Ca(Al_2Si_2O_8)$	Anorthite
	01-071-1403	Minor	$Ca_{0.466}Fe_{0.5185}Al_{0.815}Na_{0.534}O_6Si_2$	Aegerine
	--	Unassigned		
TI040-792D-2X	01-070-3755	Major	SiO_2	Quartz
	01-089-1460	Major	$Ca(Al_2Si_2O_8)$	Anorthite
	01-071-1403	Minor	$Ca_{0.466}Fe_{0.5185}Al_{0.815}Na_{0.534}O_6Si_2$	Aegerine
	--	Unassigned		
TI040-793D-2X	01-070-3755	Major	SiO_2	Quartz
	01-089-1460	Major	$Ca(Al_2Si_2O_8)$	Anorthite
	01-071-1403	Minor	$Ca_{0.466}Fe_{0.5185}Al_{0.815}Na_{0.534}O_6Si_2$	Aegerine
	--	Unassigned		

(a) Crystalline components above 10 wt.% are classified as Major phases, between 1-10 wt.% as Minor phases, and under 1 wt.% as Possible phases.



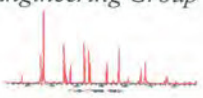
1. Sample preparation

X-ray powder diffraction (XRD) data from the samples identified in Table 2 have been examined for identification of crystalline phases present. The samples were undisolved solids and they were used as received.

All the samples were processed and analyzed according to procedure, Solids Analysis by X-Ray Diffraction, PNNL-RPG-268, Rev. 4. Each sample was mixed with approximately 2 mL of amyl acetate in a 20-mL glass scintillation vial to produce a slurry. A small aliquot of the slurry was transferred into a mortar and ground with a pestle (all tungsten carbide components). After grinding, a small fraction of the sample was pipetted onto a Krylon-covered glass slide and allowed to dry. The dried samples were then covered with Kapton. One client sample, TI040-420D-2X was run in duplicate (one without internal standard and one with 3-4 mg TiO_2, rutile as internal standard). Titania (rutile structure) internal standard SRM 674 was used; the standard certificate is provided in Appendix 1.

Table 2. Sample Identification

Sample ID	XRD Sample ID	Internal Standard
TI040-419D-2X	TI040-419D-2X	None
TI040-420D-2X	TI040-420D-2X	None
	TI040-420D-2X -TiO_2	TiO_2
TI040-421D-2X	TI040-421D-2X	None
TI040-422D-2X	TI040-422D-2X	None
TI040-792D-2X	TI040-792D-2X	None
TI040-793D-2X	TI040-793D-2X	None



2. X-ray powder diffraction data collection

Each sample was analyzed using a Rigaku Ultima IV diffractometer XRD unit equipped with a high speed position sensitive detector system D/teX and a copper X-ray tube (Cu Kα radiation, λ = 1.5418 Å). Data were collected in a step scan mode between 5 and 65° 2-theta with a step size of 0.02°, a speed of 0.5°/minute. The X-ray tube operating conditions were 40 kV and 44 mA.

The daily performance checks associated with the XRD sample files are presented in Appendix 2. These performance checks meet the performance criteria (i.e., 111 Si 2-theta = 28.44°±0.06°) specified in the XRD procedure PNNL-RPG-268 Rev. 4. The NIST certificate for the standard used for the daily performance check (SRM 640a) is presented in Appendix 3.

The quality control/calibration of the XRD instrument results are provided in Appendix 4 and the certificate for the standard used, SRM 1976, is presented in Appendix 5. The requirement for the semiannual calibration of the diffractometer (as stated in the PNNL-RPG-268 Rev. 4, is that observed d-interplanar distance for any given crystal lattice Miller indices be within ±0.02 Å (Del-d value – difference between the calculated and the observed interplanar distance) of the d-values from the International Centre for Diffraction Data (ICDD) card for alumina (corundum structure). For this quality control semiannual calibration, all Del-d values are well within the aforementioned criterion (±0.02 Å Del-d value).

3. X-ray powder diffraction data analysis

3.1. Database Search Details

Phase identification was conducted using JADE search match routines. The search was done including the following elements: C, Na, K, Mg, Ca, Al, Sr, Si, O, H, Cd, Cr, Ba, Cu, Ni, Pb, Mn, Fe, Zn, Zr, Ti and U. All diffraction patterns have been compared against International Centre for Diffraction Data Powder Diffraction File (ICDD PDF-2) 2009 database. All of the XRD analyses included the search for uranium oxides, oxyhydroxides, hydrates and hydroxides; aluminum oxides, oxyhydroxides and hydrates; and zirconium metal and zirconia (from cladding materials). The PDF cards of the crystalline phases identified in the samples analyzed in this report are given in Appendix 6.

3.2. Rietveld Refinement Details

The Rietveld method effectively separates overlapping data, thereby allowing an accurate determination of the structure/composition. Rietveld refinement on powder materials is a widely used application in determining the components of chemical mixtures. The refinement takes into account the PDF cards of the identified phases, and compares those with the calculated peaks from the observed powder pattern. The refinement generates a difference plot between an observed and a calculated powder pattern. In addition, because it calculates a powder pattern from the PDF cards of the phases taken into consideration it gives a goodness of fit for that particular phase, which is a reflection of how good the fit is between the observed and the calculated powder patterns. The refined parameters are the unit cell parameters, background, and

in the observed powder pattern, by comparing all peak positions and intensities with calculated values (from the PDF card). The quality of the refinement can be judged based on the R factors (e.g., R_w) and the difference map (i.e., the difference between an observed and a calculated powder pattern). Typically, the better the Rietveld fit the lower the R_w factor; R_w <20% suggests a good fit, R_w <10% suggests an excellent fit. R_w values <20% have to be supported by a small difference between the calculated and observed powder patterns (i.e., minor deviations within the difference map).

4. Summary of findings

The analysis samples represent the undissolved solids from the KW210 core and container composite after gross resin bead removal. The XRD data on these samples are presented in Figure 1. The samples contain primarily quartz. The samples do not contain uranium oxyhydroxides, hydrates and hydroxides; aluminum oxides, oxyhydroxides and hydrates; or zirconium metal and zirconia as crystalline phases. A summary of phases is presented in Table 1. Per request the powder patterns of the core samples and the container composite samples were presented in two different figures: Figures 15 and 16 (see Appendix 7).

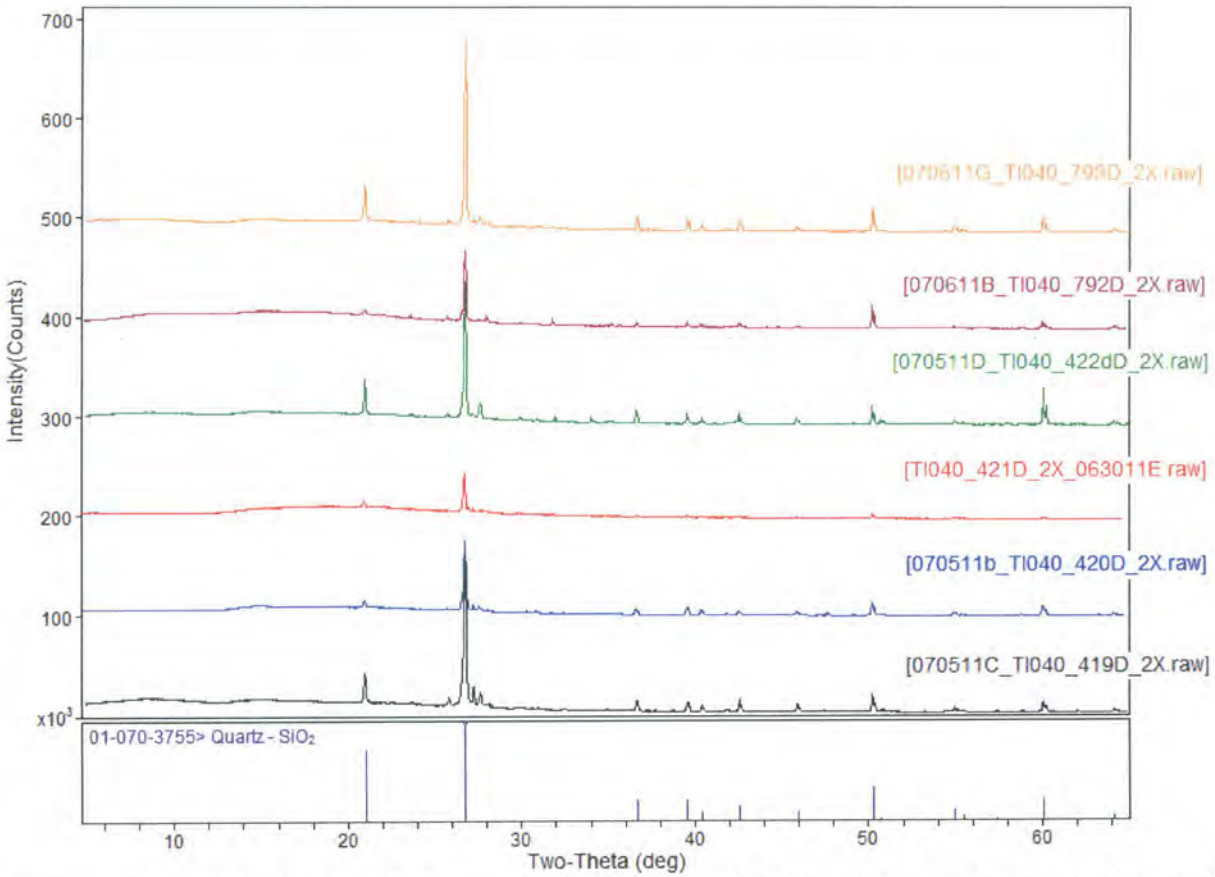
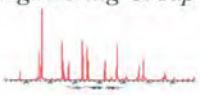


Figure 1. Raw XRD data for all the samples analyzed in this report. No background subtracted.



The following table summarizes the sample analysis-related figures present in this report.

Table 3. Summary of Figures

Client Sample ID	Figures
TI040-419D-2X	2, 3, 15
TI040-420D-2X	4, 5, 15
TI040-421D-2X	6, 15
TI040-422D-2X	7, 15
TI040-792D-2X	8, 16
TI040-793D-2X	9, 16

5. XRD Sample Analysis

5.1. XRD Sample TI040-419D-2X

Figure 2 presents the raw XRD data for sample TI040-419D-2X along with the PDF cards of the crystalline phases identified based on the Rietveld refinement (R_w = 13%). This sample is composed of silica (quartz) and anorthite as major phases. This composition has been previously observed in Hanford site sand. Figure 3 presents a plot of the Hanford site sand and sample TI040-419D-2X for comparison.

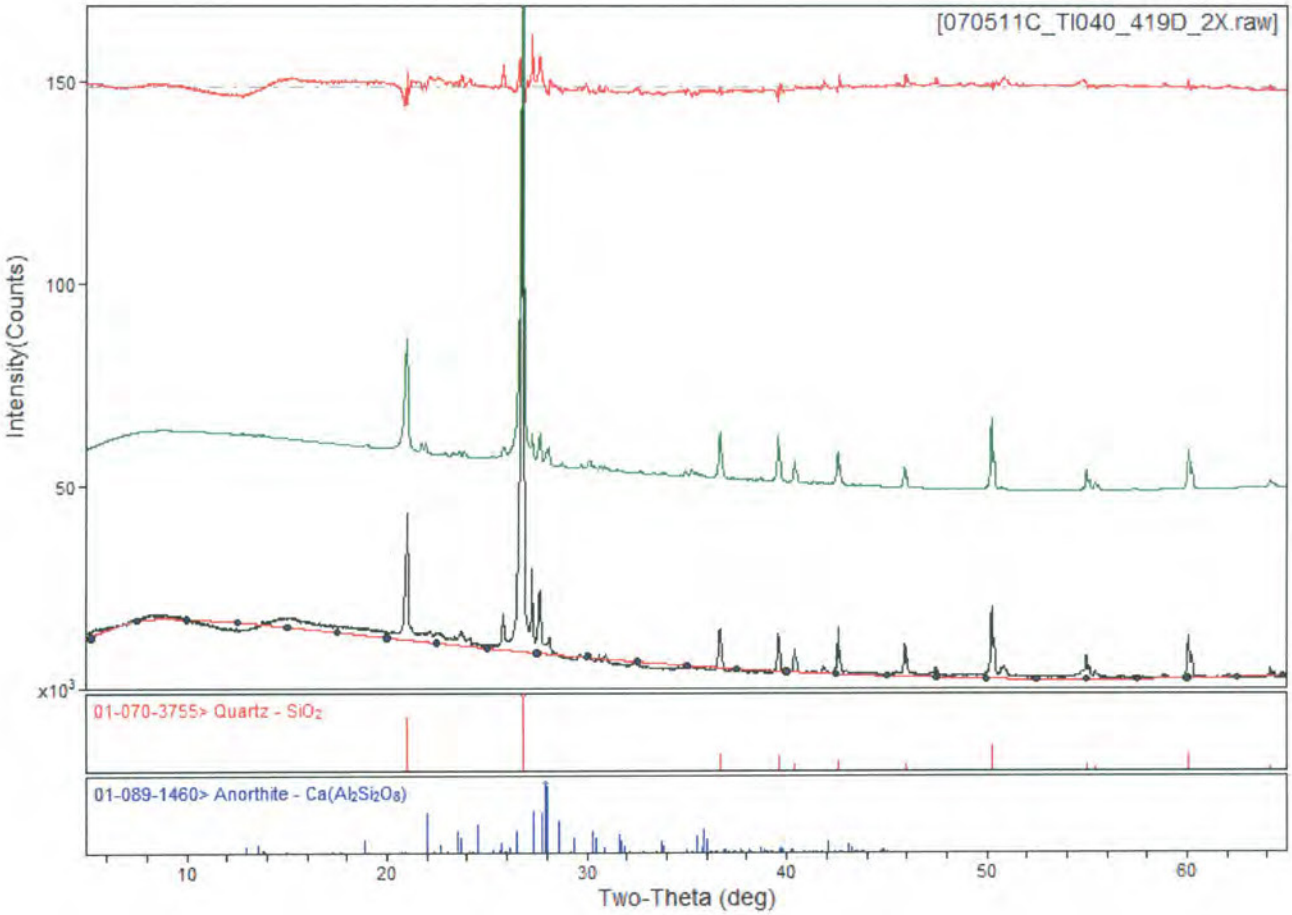


Figure 2. Rietveld refinement of the XRD data for TI040-419D-2X. The black line is the observed powder pattern (the red line underneath the black line is the background), the green line is the simulated powder pattern based on the model, and the top red line is the difference between the calculated and the observed powder patterns. R_w = 13% (See Section 3.2).

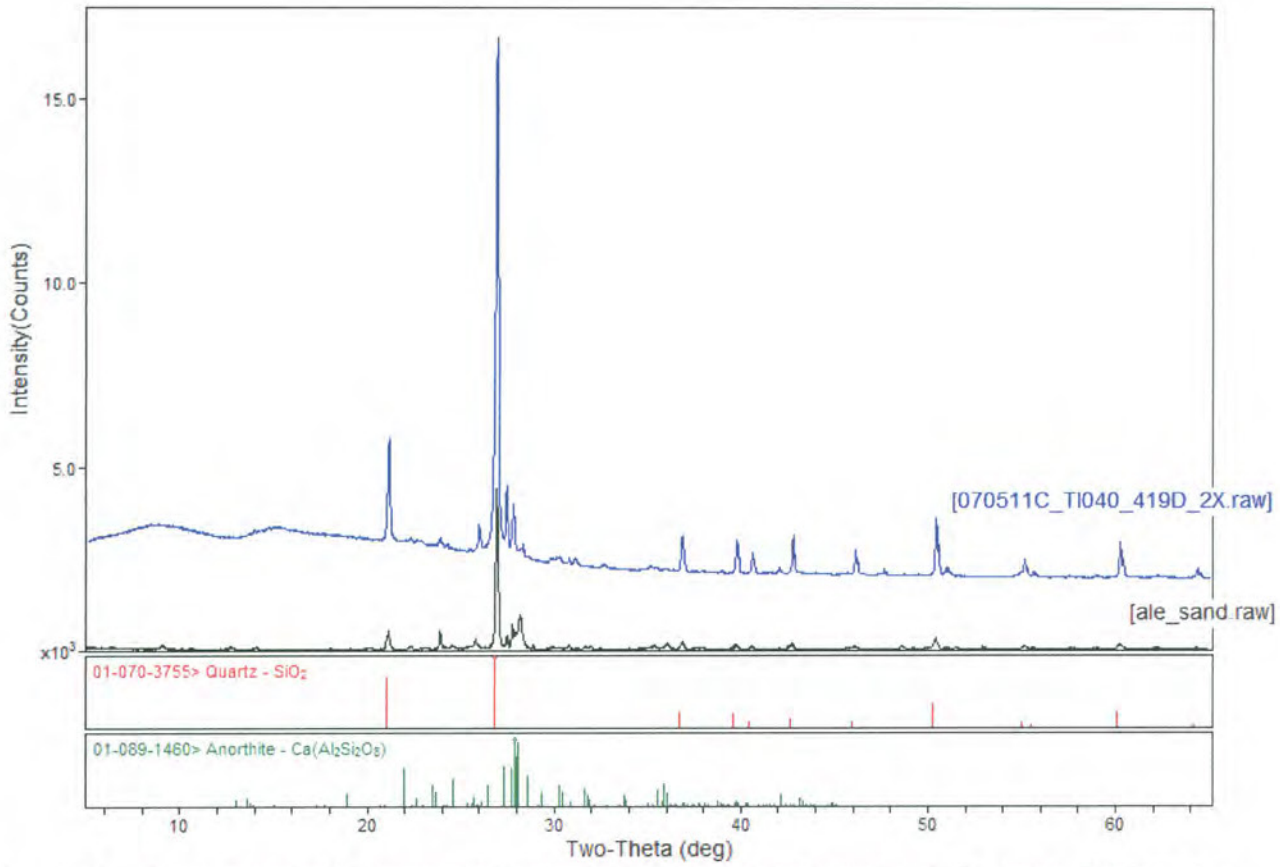


Figure 3. X-ray powder diffraction data for Hanford site sand and sample TI040-419D-2X. No background subtracted spectra are shown.

5.2. XRD Sample TI040-420D-2X and TI040-420D-2X-TiO_2

Figure 4 presents the raw XRD data for samples TI040-420D-2X along with the PDF card of the crystalline phase identified based on the Rietveld refinement (R_w = 10%). This sample is composed of silica (quartz) and anorthite as major phases. In addition the sample contains aegerine and calcium sulfate as minor crystalline phases. Figure 5 presents the background subtracted XRD data for TI040-420D-2X and TI040-420D-2X-TiO_2. Rutile internal standard peaks, SRM 674, are consistent with the ones reported in the PDF card 01-077-0440, which means that the peak positions in the analyzed samples are reliable. The two duplicate samples confirm the diffraction patterns given the overall analysis method uncertainty.

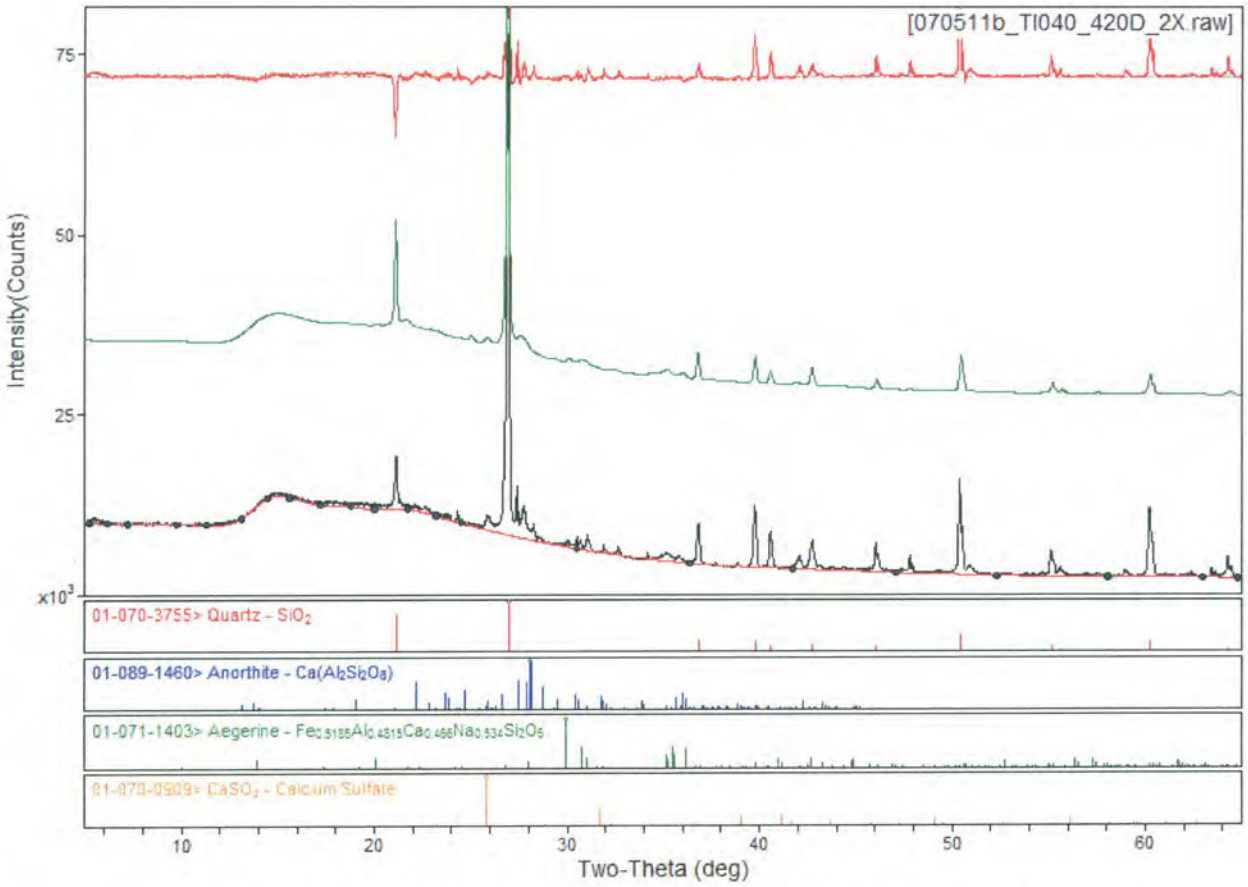


Figure 4. Rietveld refinement of the XRD data for TI040-420D-2X. The black line is the observed powder pattern (the red line underneath the black line is the background), the green line is the simulated powder pattern based on the model, and the red line is the difference between the calculated and the observed powder patterns. R_w = 10% (See Section 3.2).

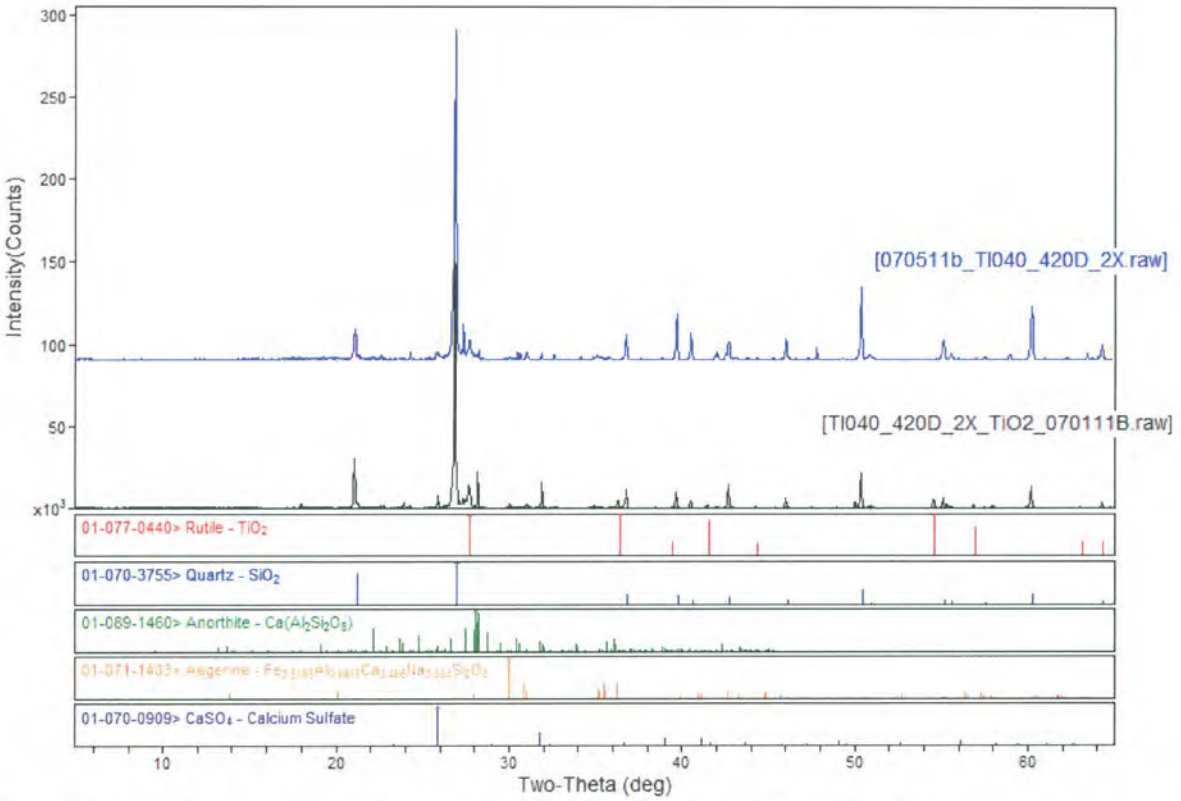


Figure 5. XRD data for TI040-420D-2X and TI040-420D-2X-TiO_2. Background subtracted spectra are shown.

5.3. XRD Sample TI040-421D-2X

Figure 6 presents the raw XRD data for sample TI040-421D-2X along with the PDF cards of the crystalline phases identified based on the Rietveld refinement (R_w = 6%). This sample is composed of silica (quartz) and anorthite as major crystalline phases.

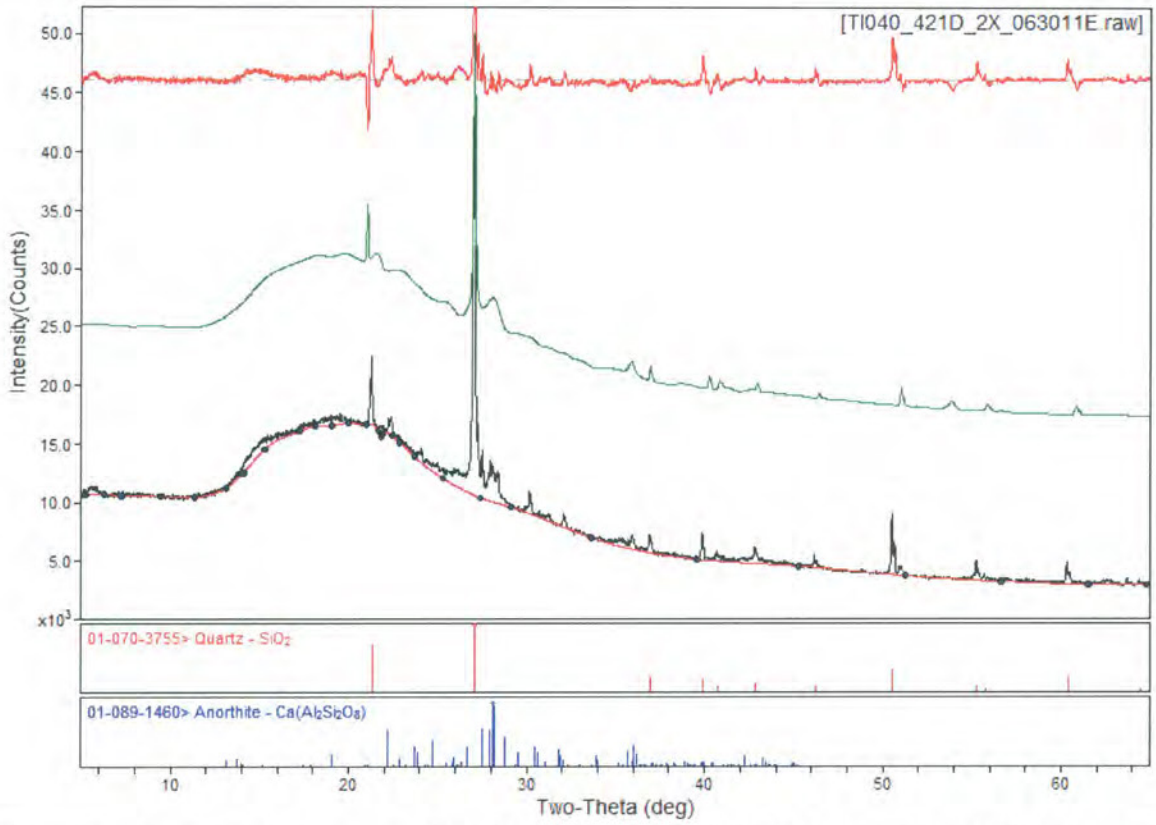


Figure 6. Rietveld refinement of the XRD data for TI040-421D-2X. The black line is the observed powder pattern, the green line is the simulated powder pattern based on the model, and the red line is the difference between the calculated and the observed powder patterns. R_w = 6% (See Section 3.2). No background subtracted spectra shown.

5.4. XRD Sample TI040-422D-2X

Figure 7 presents the Rietveld refinement (R_w = 11%) of the XRD data for sample TI040-422D-2X with the PDF cards of the corresponding crystalline phases. The major phases present in this sample are silica (quartz) and anorthite. In addition the sample contains aegerine as a minor crystalline phase.

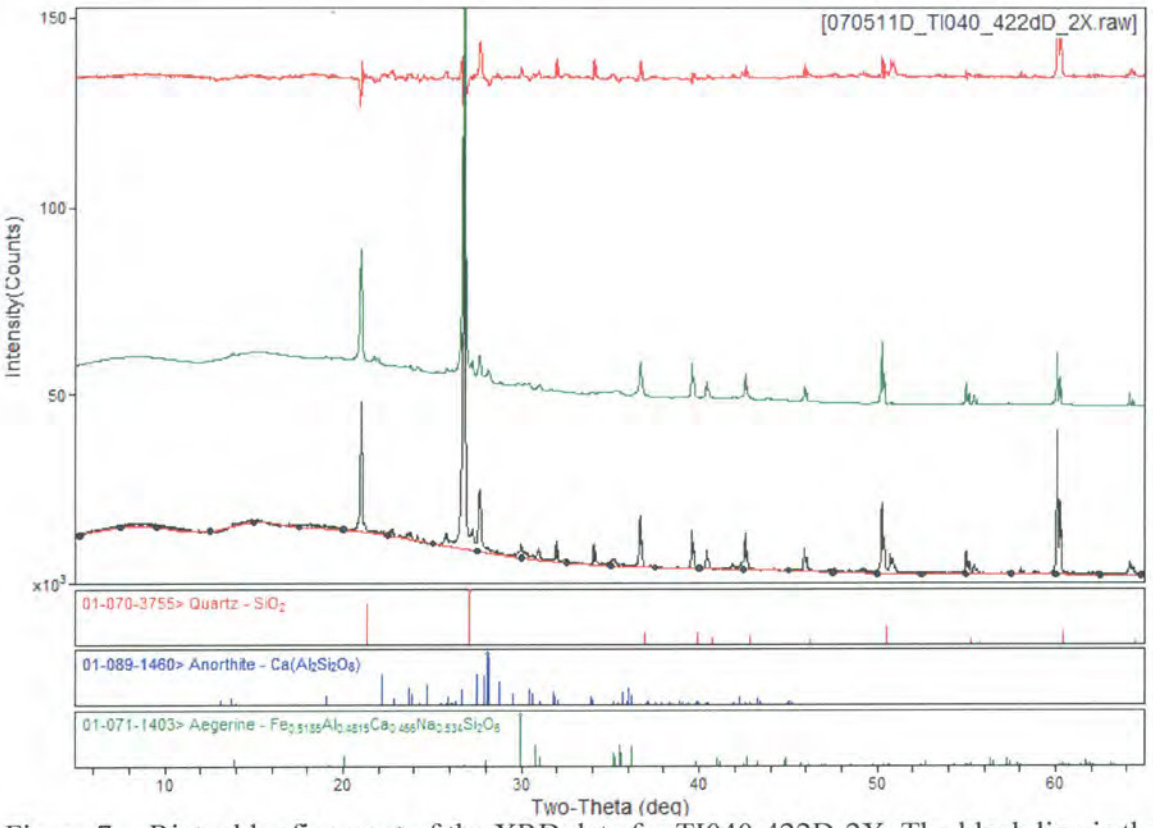


Figure 7. Rietveld refinement of the XRD data for TI040-422D-2X. The black line is the observed powder pattern, the green line is the simulated powder pattern based on the model, and the red line is the difference between the calculated and the observed powder patterns. R_w = 11% (See Section 3.2). No background subtracted spectra shown.

5.5. XRD Sample TI040-792D-2X

Figure 8 presents the Rietveld refinement (R_w = 7 %) of the XRD data for sample TI040-792D-2X with the PDF cards of the corresponding crystalline phases. The major phases present in this sample are silica (quartz) and anorthite. In addition the sample contains aegerine as a minor crystalline contribution.

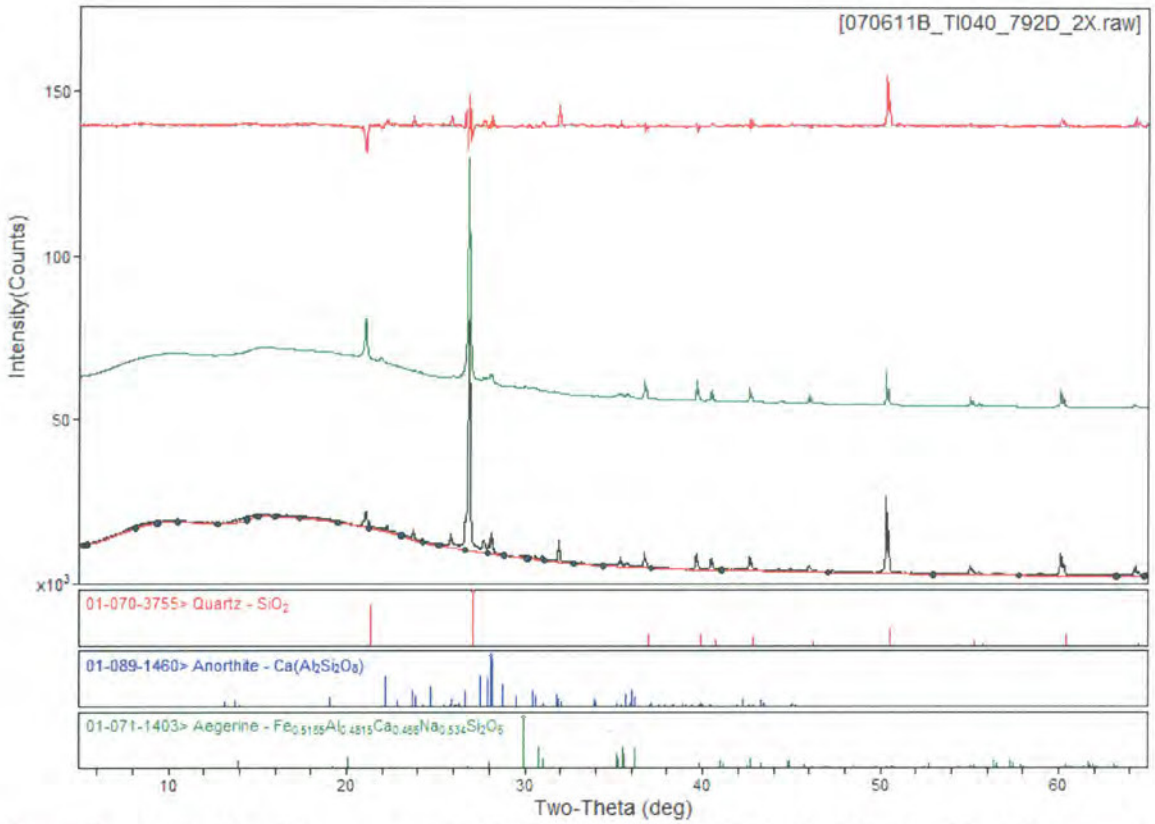


Figure 8. Rietveld refinement of the XRD data for TI040-792D-2X. The black line is the observed powder pattern, the green line is the simulated powder pattern based on the model, and the red line is the difference between the calculated and the observed powder patterns. R_w = 7 % (See Section 3.2). No background subtracted spectra shown.

5.6. XRD Sample TI040-793D-2X

Figure 9 presents the Rietveld refinement (R_w = 7%) of the XRD data for sample TI040-793D-2X with the PDF cards of the corresponding crystalline phases. The major phase present in this sample is silica (quartz). In addition the sample contains anorthite and aegerine as minor crystalline phases.

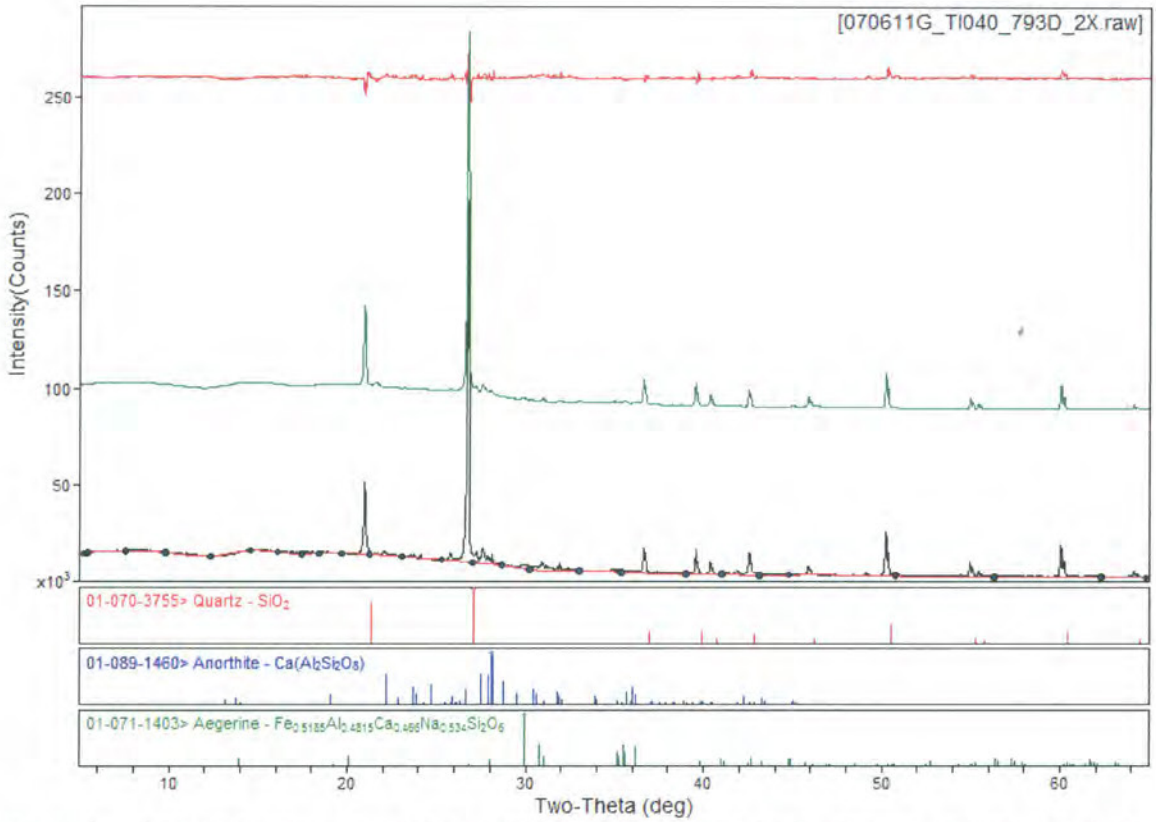
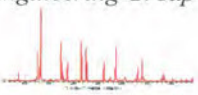


Figure 9. Rietveld refinement of the XRD data for TI040-793D-2X. The black line is the observed powder pattern, the green line is the simulated powder pattern based on the model, and the red line is the difference between the calculated and the observed powder patterns. R_w = 7% (See Section 3.2). No background subtracted spectra shown.



Appendix 1
SRM 674 NIST Certificate

National Bureau of Standards

Certificate

Standard Reference Material 674

X-Ray Powder Diffraction
Intensity Set

C. R. Hubbard

This Standard Reference Material (SRM) consists of five different phases. The phases, separately bottled, are α-Al_2O_3 (corundum structure), ZnO (wurtzite structure), TiO_2 (rutile structure), Cr_2O_3 (corundum structure), and CeO_2 (fluorite structure). These phases can be used as internal standards for quantitative analysis and as external standards for checking the intensity response of x-ray diffraction instruments. The five phases cover the range of linear absorption coefficient from 100 to 1000 cm^{-1} for CuKα radiation. The intensity values are for a constant diffracting volume (fixed divergent slit and infinitely thick mounting or equivalent).

The certified relative intensity values (CuKα radiation), for the major diffraction lines below 70° 2(θ), for each phase are given in Table 1. The certified reference intensity ratios (I/Ic) for ZnO, TiO_2, Cr_2O_3, and CeO_2, relative to α-Al_2O_3 and the lattice parameters are given in Tables 2 and 3. The uncertainties of the relative intensity and lattice parameter values are the standard deviations derived from multiple measurements. The uncertainties of the reference intensity ratio values were determined from the value of the internal inconsistency derived from all possible binary mixtures.

The average crystallite size for each of the five phases is near 2.0 μm. Crystallite size broadening of the diffraction peaks is small but present. This is particularly apparent for α-Al_2O_3, which has a nominal crystallite size less than 1.0 μm. The lattice parameters, uncorrected for refraction, were determined with a relative uncertainty of about three parts in 10^5 using an internal standard. This uncertainty in the lattice parameter must be increased by a factor of two or three if one wishes to determine absolute d-spacings as the uncertainties due to the internal standard 2θ's and to thermal expansion would need to be added.

The technical and support aspects concerning the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by R.K. Kirby.

Washington, D.C. 20234
June 1, 1983

(over)

Stanley D. Rasberry, Chief
Office of Standard Reference Materials

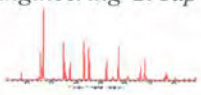


Table 1. Relative Intensities of Major Lines

CuKα Radiation (μ = 124.1 cm^{-1})

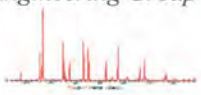
hkl	2θ	I^{rel}	hkl	2θ	I^{rel}	
α-Al_2O_3[a]	(Corundum Structure)		TiO_2	(cont.)		
012	25.58°	55.4 ± 2.4	111	41.20°	21.4 ± 1.4	
104	35.13	87.4 ± 1.9	211	54.27	56.9 ± 2.8	
110	37.78	36.5 ± 1.4	220	56.51	16.9 ± 1.4	
113	43.36	100.0	002	62.76	8.2 ± 0.7	
024	52.55	45.5 ± 1.3	301	68.93	(21.1)	approximate due to
116	57.52	92.5 ± 2.6	112	69.79	(11.1)	partial overlap
214	66.54	34.7 ± 1.0				
300	68.19	55.5 ± 2.2				
ZnO	(Wurtzite Structure)		Cr_2O_3[c]	(Corundum Structure)		
100	31.75°	57.6 ± 1.1	012	24.52°	69.2 ± 1.6	
002	34.44	40.2 ± 1.4	104	33.59	100.0	
101	36.25	100.0	110	36.20	87.1 ± 2.3	
102	47.54	22.8 ± 0.5	113	41.47	32.3 ± 1.0	
110	56.55	34.4 ± 0.8	024	50.21	39.4 ± 0.8	
103	62.87	31.0 ± 0.9	116	54.81	94.5 ± 2.2	
200	66.40	4.7 ± 0.2	214	63.43	30.8 ± 1.3	
112	67.91	25.9 ± 0.6	300	65.09	40.6 ± 1.0	
TiO[b]	(Rutile Structure)		CeO_2[d]	(Fluorite Structure)		
110	27.42°	100.0	111	28.55°	100.0	
101	36.04	44.0 ± 1.7	200	33.08	28.0 ± 0.8	
			220	47.49	53.5 ± 2.0	
			311	57.32	43.4 ± 2.3	

[a] Two broad, very weak peaks (32.80 and 45.5° 2θ) can be detected. These lines are possibly due to γ-Al_2O_3 (PDF 29-63).

[b] Anatase was observed at ~1.0 wt. % level. Unexplained very weak peaks at 13.9 and 18.0° 2θ were also detected.

[c] Small unexplained intensity deviations from smooth background.

[d] Three unexplained, broad, very weak diffraction peaks were observed at 14.40, 16.64, and 23.75° 2θ.



Appendix 2
Daily Performance Checks

The daily performance check shows that the Rigaku Ultima IV diffractometer is within calibration per PNNL-RPG-268 Rev. 4, "Solids Analysis by X-Ray Diffraction" so no adjustments have been made. Figure 10, 11, 12 and 13 present the XRD data obtained on the silicon SRM 640a standard. In Figures 10-13, the black line represents the observed XRD powder pattern, and the green line represents a simulated Si pattern using the PDF card 01-071-3770. Tables 4, 5, 6, 7 contain summarized results of the daily performance check with this standard.

The columns in Tables 4-7 have the following significance:

1) 2T(r) gives the 2-theta of the reference powder pattern (PDF card)
2) 2T(o) is the observed 2-theta
3) Delta is the difference between the observed and the reference 2-theta
4) d(r) is the interplanar distance d for the reference pattern (in Å)
5) d(o) is the observed d
6) Del-d is the difference between the observed and the reference d
7) I % is the intensity in percent (the most intense peak is always 100% and all other peaks are normalized)

Per procedure the measured 2-theta of the performance check standard (i.e., the 111 Si reference line) must be 28.44°±0.06° 2-theta; the observed 2-theta difference (Delta) was 0.014-0.015 and met this requirement. Thus the performance checks met this criterion.

Daily performance check (27° to 29° 2-theta) – Date 06-30-2011

Theta Calibration Report
[063011a_si_640a.raw] - Full Chart
Reference Lines = 01-071-3770> Silicon - Si
Two-Theta Range of Fit = 27.0/29.0(deg)
Zero Offset = -0.019(deg)

Table 4. Daily performance check report summary

2T(r)	2T(o)	Delta	d(r)	d(o)	Del-d	I%
28.443	28.462	-0.019	3.1355	3.1334	0.0020	100.0

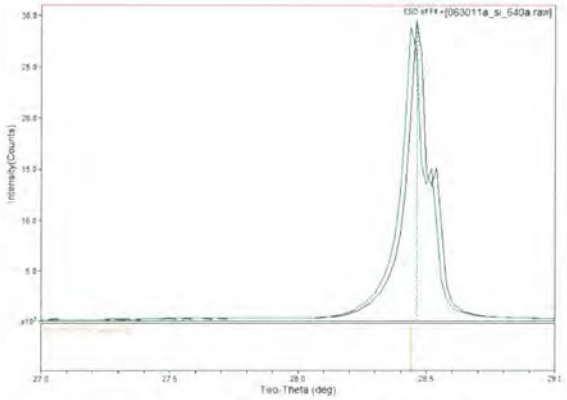


Figure 10. The raw XRD data on SRM 640a against the corresponding PDF card.

Daily performance check (27° to 29° 2-theta) – Date 07-01-2011

Theta Calibration Report
[070111A_SI_640A.raw] STD - Full Chart
Reference Lines = 01-071-3770> Silicon - Si
Two-Theta Range of Fit = 27.0/29.0(deg)
Zero Offset = -0.019(deg)

Table 5. Daily performance check report summary

2T(r)	2T(o)	Delta	d(r)	d(o)	Del-d	I%
28.443	28.462	-0.019	3.1355	3.1334	0.0020	100.0

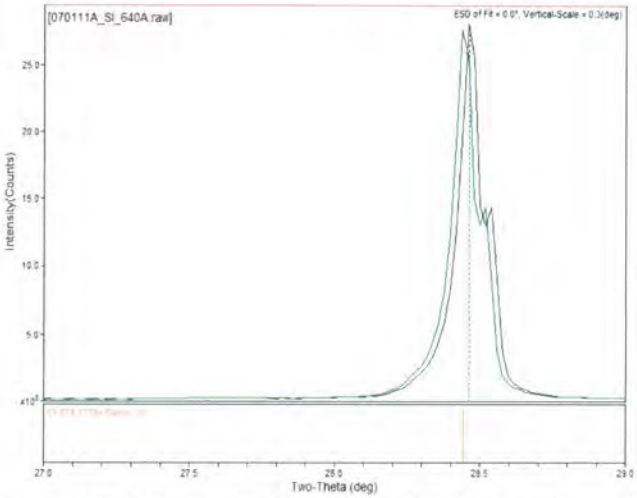


Figure 11. The raw XRD data on SRM 640a against the corresponding PDF card.

Daily performance check (27° to 29° 2-theta) – Date 07-05-2011

Theta Calibration Report
[070511a_si_640a.raw] standard si - Full Chart
Reference Lines = 01-071-3770> Silicon - Si
Two-Theta Range of Fit = 27.0/29.0(deg)
Zero Offset = -0.018(deg)

Table 6. Daily performance check report summary

2T(r)	2T(o)	Delta	d(r)	d(o)	Del-d	I%
28.443	28.461	-0.018	3.1355	3.1335	0.0020	100.0

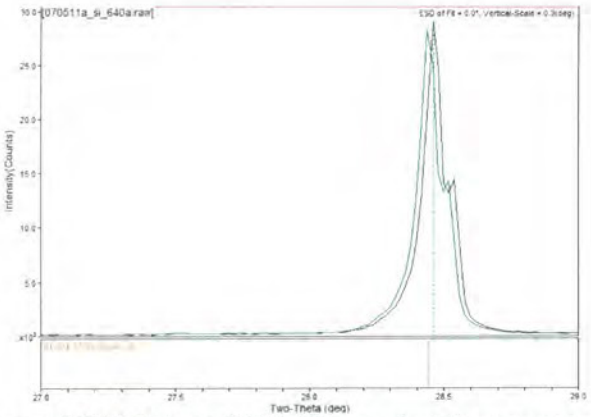
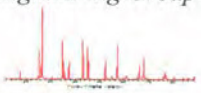


Figure 12. The raw XRD data on SRM 640a against the corresponding PDF card.

Daily performance check (27° to 29° 2-theta) – Date 07-06-2011

Theta Calibration Report
[070611A SI640A.raw] - Full Chart



Reference Lines = 01-071-3770> Silicon - Si
Two-Theta Range of Fit = 27.0/29.0(deg)
Zero Offset = -0.018(deg)

Table 7. Daily performance check report summary

2T(r)	2T(o)	Delta	d(r)	d(o)	Del-d	I%
28.443	28.461	-0.018	3.1355	3.1335	0.0019	100.0

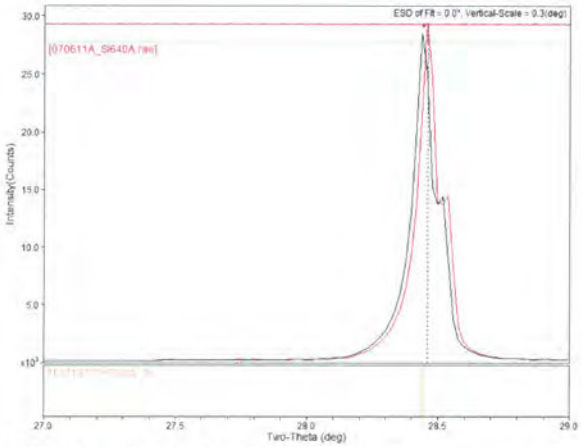
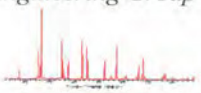
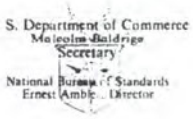


Figure 13. The raw XRD data on SRM 640a against the corresponding structure PDF card.



Appendix 3
SRM 640a NIST Certificate



National Bureau of Standards

Certificate

Standard Reference Material 640a

Silicon Powder 2θ/d-Spacing Standard for X-Ray Diffraction

Camden R. Hubbard

This Standard Reference Material (SRM) was prepared for use as an external or internal 2θ/d-spacing calibration standard for powder diffractometry.

Electronic grade float-zone prepared silicon boules, 99.999 +% pure, were ground to pass a 75 μm (200 mesh) sieve. This powder, which was identical to SRM 640, was then jet-milled to reduce the mean particle size to about 2 μm.

A total of twelve samples, mixed with tungsten and silver internal standards [1], were measured using a high angle goniometer controlled by a minicomputer. The CuKα_1 peak position was determined by profile fitting procedures and then corrected for sample, instrumental, and physical aberrations (except refraction) through use of the internal standard lines [2]. Silicon peak positions were then corrected for effects of thermal expansion. Each corrected set was refined by a least-square routine that minimized $\Sigma(\theta_{obs}-\theta_{calc})^2$ to obtain estimates of a_i and their estimated standard errors, s_i. The weighted average of the twelve lattice parameters, uncorrected for refraction, at 25 °C is

$$\langle a \rangle = 5.430825 \pm 0.000036 \text{ Å}$$

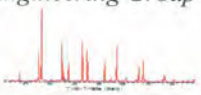
where $\lambda(CuK\alpha_1) = 1.5405981$ Å [3]. The estimated total uncertainty given above includes contributions from three sources (listed in decreasing importance): (1) uncertainty of the lattice parameters of the tungsten and silver standards; (2) random errors of the measurements; and (3) the uncertainty in $\lambda(CuK\alpha_1)$.

The technical and support aspects concerning the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by R.K. Kirby.

Washington, D.C. 20234
February 19, 1982

George A. Uriano, Chief
Office of Standard Reference Materials

(over)



The 2θ values given in Table 1 were calculated from the certified value of the lattice parameter. The relative intensities [4], I (rel), also given in Table 1 can be used as an aid in identifying the silicon lines when SRM 640a is mixed with test materials. The uncertainty of each value of I (rel) may be as large as $\pm$ 3. Suggested methods for use of this SRM are given in references [2], [5], and [6].

Table 1

Calculated Diffraction Angles and Relative Intensities

(T = 25.0 °C)

These Values Are Not Certified

hkℓ	I (rel)	2θpeak	hkℓ	I (rel)	2θpeak
111	100	28.443°	511/333	6	94.955°
220	55	47.304	440	3	106.712
311	30	56.124	531	7	114.096
400	6	69.132	620	8	127.550
331	11	76.378	533	3	136.900
422	12	88.033	444	*	158.644

*Not measured

[1] Swanson, H.E.; McMurdie, H.F; Morris, M.C.; and Evans, E.H. (1966), Standard X-Ray Diffraction Powder Patterns, National Bureau of Standards Monograph 25, Section 4, NBS, Washington, D.C. 20234.

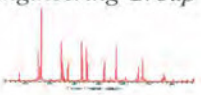
[2] Hubbard, C.R. (1982), submitted for publication in J. Appl. Cryst.

[3] Deslattes, R.D. and Henins, A. (1973), Phys. Rev. Letters, 31, 972-975.

[4] Morris, M.C.; McMurdie, H.F.; Evans, E.H.; Paretzkin, B.; deGroot, J.H.; Hubbard, C.R.; and Carmel, S.J. (1976) Standard X-Ray Diffraction Powder Patterns, National Bureau of Standards Monograph 25, Section 13, NBS, Washington, D.C. 20234.

[5] Snyder, R.L.; Hubbard, C.R.; and Panagiotopoulos, N.C., Advances in X-Ray Analysis, Vol. 25, (in press).

[6] Hubbard, C.R. (1980) Accuracy in Powder Diffraction, NBS Special Publication 567, p 489-502.



Appendix 4
Quality Control/Calibration

This control calibration measurement shows that the Rigaku Ultima IV diffractometer is within calibration per PNNL-RPG-268 Rev. 4, "Solids Analysis by X-Ray Diffraction" so no adjustments have been made. The XRD data obtained on this standard are represented in Figure 14. Table 8 contains summarized results of the quality control/calibration results obtained using NIST SRM 1976 (Corundum).

The columns in Table 8 have the following significance:

1) 2T(r) gives the 2-theta of the reference powder pattern (PDF card)
2) 2T(o) is the observed 2-theta
3) Delta is the difference between the observed and the reference 2-theta
4) d(r) is the interplanar distance d for the reference pattern (in Å)
5) d(o) is the observed d
6) Del-d is the difference between the observed and the reference d
7) I % is the intensity in percent (the most intense peak is always 100% and all other peaks are normalized)

The requirement for this quality check (semiannual calibration verification of the diffractometer as stated in the PNNL-RPG-268 Rev. 4, is that observed d-interplanar distance for any given crystal lattice Miller indices be within ±0.02 Å of the NIST SRM 1976 PDF card d-values. For this quality performance check, all Del-d values are well within this criterion. The PDF card for alumina (corundum structure) is provided in Appendix 6.

Control Calibration Report (10° to 60° 2 theta) – Date 06-30-2011

Theta Calibration Report
[063011b_1976_alumina_plate.raw] - Full Chart
Reference Lines = 01-071-6478> Corundum - Al2O3
Two-Theta Range of Fit = 5.0/80.0(deg)
Zero Offset = 0.026(deg)
ESD of Fit = 0.00647°, |Delta 2-Theta| = 0.026(deg)

Table 8. Summary of the semiannual calibration report

2T(r)	2T(o)	Delta	d(r)	d(o)	Del-d	I%
25.573	25.543	0.03	3.4805	3.4845	0.004	31.1
35.147	35.118	0.029	2.5512	2.5533	0.002	100
37.77	37.743	0.027	2.3799	2.3815	0.0016	12.6
41.676	41.642	0.034	2.1654	2.1671	0.0017	2.7
43.347	43.32	0.027	2.0857	2.087	0.0013	50.2
46.17	46.147	0.023	1.9646	1.9655	0.0009	0.4
52.544	52.519	0.025	1.7403	1.741	0.0008	26
57.494	57.463	0.031	1.6016	1.6024	0.0008	76.1
59.729	59.71	0.019	1.5469	1.5474	0.0005	0.8
61.118	61.103	0.015	1.5151	1.5154	0.0003	1.4
61.299	61.264	0.036	1.511	1.5118	0.0008	13.2

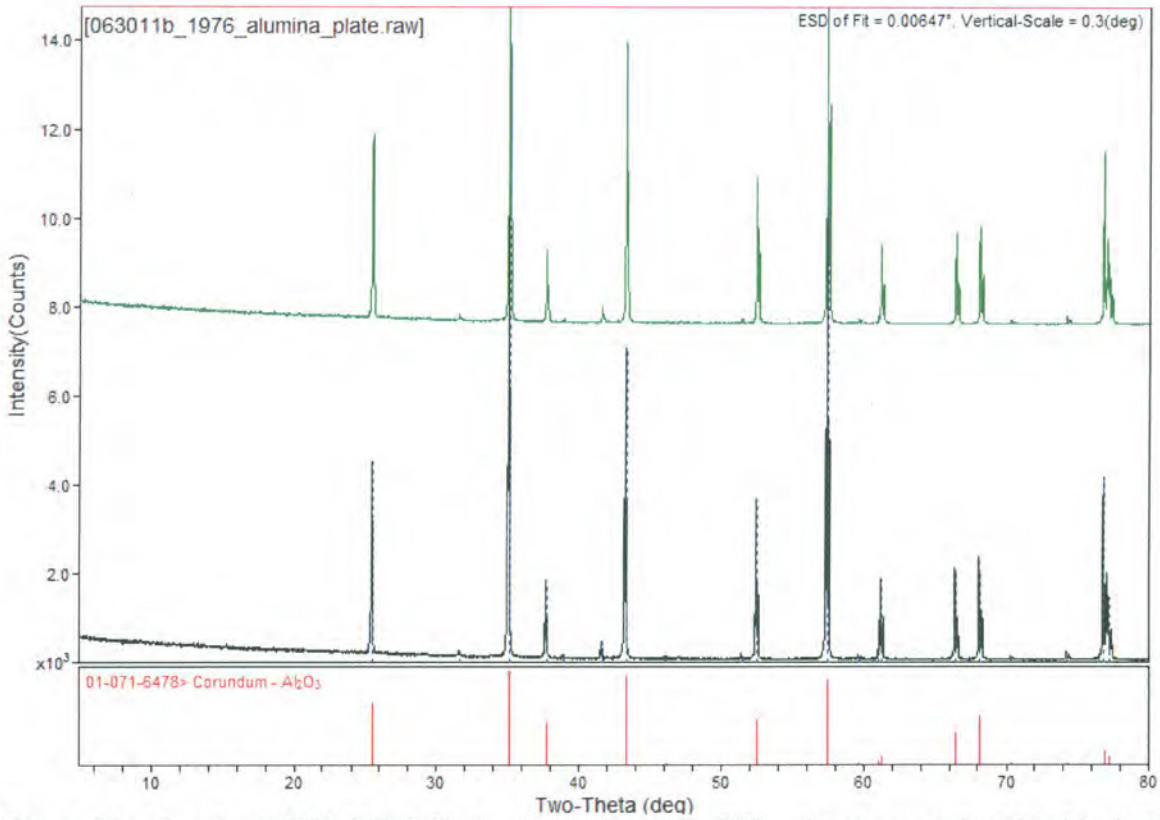
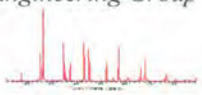


Figure 14. Alumina NIST SRM 1976 plate, observed diffraction pattern is with black (middle image) the simulated reference diffraction pattern is with green (top image) and the PDF card is with red (bottom image).



Appendix 5
SRM 1976 NIST Certificate

National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material 1976

Instrument Sensitivity Standard for X-ray Powder Diffraction

This Standard Reference Material (SRM) consists of a sintered alumina plate, approximately 45 mm on a side by 1.6 mm in thickness, intended for use in calibration of powder X-ray equipment for diffraction intensity as a function of 2Θ angle (instrument sensitivity). The form of the SRM serves to eliminate the variability of sample loading procedure from intensity measurements. This SRM material was chosen for consistency of microstructure with respect to grain size, shape, micro-strain and orientation. The platelets comprising this microstructure are 3 to 7 μm in diameter by 1 to 2 μm in thickness and are highly oriented with the basal plane parallel to the surface of the plate. Some amount of amorphous phase is present though no crystalline impurities could be detected.

The proper use of this SRM requires the measurement of intensity values from test equipment in a manner analogous to either of the two measurement methods used for certification. The certified parameters consist of absolute variation in intensity, 12 relative intensity values calculated from integrated intensity and peak height measurements from 25.5 to 145 degrees 2Θ, and the lattice parameters. Graphical evaluation of the ratio of data from the test equipment to the certified values will allow for an appropriate judgment as to the condition of the test equipment. This may be followed by the calculation of a correction curve, yielding standardization of instrument sensitivity and thus more accurate inter-laboratory comparisons of data involving X-ray diffraction intensity measurements.

The overall coordination of the preparation and the technical measurements leading to certification was performed by J.P. Cline of the NIST Ceramics Division. The project was the result of a round robin study carried out by R. Jenkins and W. Schreiner of the International Centre for Diffraction Data, Swarthmore, Pennsylvania.

The alumina substrates for this SRM were donated to NIST by International Business Machines Corporation, East Fishkill, New York, through the efforts of R. Anderson.

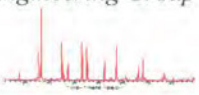
Statistical analysis was provided by S.B. Schiller and K.R. Eberhardt of the NIST Statistical Engineering Division.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Standard Reference Materials Program by N.M. Trahey.

Gaithersburg, MD 20899
November 8, 1991

William P. Reed, Chief
Standard Reference Materials Program

(over)



Certified intensity data were collected from 25 randomly selected samples on a Philips[1] diffractometer using an AEG[1] long fine-focus Cu X-ray tube operated at 1800 watts with a constant potential generator. The diffractometer was equipped with incident and diffracted beam sollar slits, graphite diffracted beam monochromator in anti-parallel geometry, and scintillation detector. The incident beam slits were of the theta compensating type, locked such that the illumination area was 12.5 mm wide at 25 degrees 2Θ. This corresponds to a slit size of 0.93 degrees; the receiving slit was 0.066 degrees. The take-off angle was 4 degrees. Samples were spun at approximately 52 rpm during data collection.

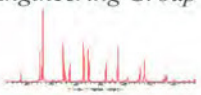
Data were collected and processed with the Siemens Diffrac500[1] version of NBS*Quant. This algorithim scans specific peaks for an integrated intensity measurement, with extended count times for each of ten points on either side of the scan range. Scan angles are indicated in Table 1; the step width was 0.005 degrees. Scan time ranged from 30 to 90 minutes per peak, for a total of 16 hours per sample. The seventh and eleventh scan ranges actually include two peaks, as noted by the two sets of miller indices listed for them in Table 1. The use of two peaks in these scan ranges ensured that intensity data were reported evenly through the desired 2Θ range.

Peak height values were manually determined, with a background subtraction, from the same scans as were the integrated intensity data. Five background data points from each end of the scan range were averaged to determine the background value. Peak height values were determined from an average of the three most intense points for data below 55 degrees 2Θ; four data points were used for the remainder of the data. Due to the proximity of the two reflections used in the seventh and eleventh relative intensity values, peak height values for these data reflect the superposition of the Cu $K\alpha_1$ and $K\alpha_2$ peaks. Thus, peak height data for these two pairs of reflections will be valid only with the use of Cu $K\alpha$ radiation.

Lattice parameters were determined from the 25 samples by means of least squares refinement from 47 peak positions determined with the spline method, ranging from 22 to 152 degrees 2Θ. Peak positions were corrected for optical aberrations with the use of SRM 640b silicon powder, to generate a 2nd order polynomial external standard correction curve which was applied to peak location determinations. Measurements were made on a Siemens D500[1] diffractometer equipped with a focusing Ge incident beam monochromator, sample spinner, and a position sensitive proportional detector. Copper $K\alpha$ radiation was used. Measurements were made within a period of 48 hours during which operating conditions were maintained. The machine was stabilized under such conditions for several days prior to measurements.

The intensity measurements from the SRM material are expected to show heterogeneity due to variations in preferred orientation. The heterogeneous nature of the samples requires a certification of a population of true relative intensities rather than the average relative intensity for each peak. The preferred orientation will uniformly effect the variability of all the peaks so analysis of the data assumed the intensity measurements have the same true relative variance. This allowed for pooling of the variances for both absolute and relative intensities. The exception to this assumption is the (300) peak; the crystallites in the diffracting position for this reflection must be perpendicular to the preferred orientation direction. Increased variability of intensity from this reflection is thus expected. The nominal value of the relative intensity is reported for information only; it was not used in subsequent analysis.

[1] Certain manufacturers are mentioned for informative purposes only and do not represent an endorsement by NIST.



ABSOLUTE VARIATION IN INTENSITY

Analysis of the raw integrated intensity values indicated a drift in the instrument. This drift was modeled with a quadratic equation and removed from subsequent analyses. The relative standard deviation of the raw counts, pooled from the 13 peaks is:

RSD (raw counts) = 2.21%

This value incorporates both material variability and machine imprecision. This parameter can be used to determine differences in absolute sensitivity with different SRM samples.

RELATIVE INTENSITY VARIATION

The relative intensity values displayed no drift. The pooled relative standard deviation from integrated intensity measurements of the 12 peaks is:

RSD (integrated area, relative intensity) = 2.06%

The pooled relative standard deviation of the relative intensity values determined from peak height measurements is:

RSD (peak height, relative intensity) = 2.62%

The total relative uncertainty is a multiple of the relative standard deviation. For each relative intensity, this value is:

Total Uncertainty (integrated intensity) = 6.12%
Total Uncertainty (peak height) = 7.85%

This uncertainty is the half-width of a 90% confidence, 95% coverage tolerance interval. With 90% confidence, 95% of the true relative intensities for all of the peaks of this SRM should fall within the uncertainty of the certified values.

Each SRM plate is sintered to a relatively low density allowing it to be scored and broken easily into a desired size. Care should be taken to ensure that the side opposite the label is illuminated; otherwise, results are invalid. Each plate is cylindrically symmetrical, thus orientation of the plate is not of concern. However, errors due to particle counting statistics will be reduced if a sample spinner is used. Consistency in the degree of size and micro-strain induced peak broadening of the SRM allow for the use of peak heights as a substitution for integrated intensity measurements, though precision is seen to suffer as a result. The ratio of experimental data to the certified values should be plotted as a function of 2Θ angle. If this data is patternless, and if all of the ratios fall within the total uncertainty (0.0612/0.0785) of 1, then the test equipment is in control. However, if there is a clear pattern in the data, or if the ratio for at least one peak is more than 0.0612/0.0785 from 1, then the system is out of control (see Appendix).

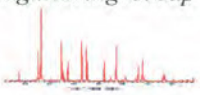


Table 1

Reflection(s) (hkl)	Scan Angles (low/high) 2Θ		Relative Intensity Integrated Area	Peak Height
(012)	24.7	26.2	32.34	33.31
(104)	34.0	36.2	100.0	100.0
(113)	42.4	44.2	51.06	49.87
(024)	51.8	53.3	26.69	25.17
(116)	56.0	59.0	92.13	83.6
(300)	67.4	69.0	19.13	16.89
(1.0.10) (119)	75.7	78.2	55.57	34.61
(0.2.10)	88.1	89.7	11.76	8.99
(226)	94.3	96.0	10.14	7.25
(2.1.10)	100.1	102.0	16.13	10.94
(324) (0.1.14)	115.4	117.4	20.86	10.09
(1.3.10)	126.8	128.95	15.58	7.56
(146)	135.2	137.4	15.47	6.55
(4.0.10)	144.3	146.7	11.29	4.06

LATTICE PARAMETERS

The certified lattice parameters, from the mean of the 25 samples are:

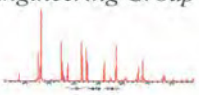
$a = 0.4758846$ nm (4.758846 Å)

$\sigma = 0.0000109$ (0.000109)

$c = 1.299306$ nm (12.99306 Å)

$\sigma = 0.0000238$ (0.000238)

where λ ($CuK\alpha_1$) = 0.1540629 nm (1.540629Å)



Appendix: How to assess and correct instrument bias using this SRM

When looking for instrumental problems, the peaks should be considered jointly, not individually. First, plot control bands as $1 \pm U$ over the range of 2Θ, where U is the relative uncertainty (U = 0.0612 if peak areas were used, or 0.0785 if peak heights were used). Next, plot the ratio of each new measurement to the certified value on the vertical axis against the 2Θ peak location on the horizontal axis. As long as these ratios fall within the control bands and are patternless, the system cannot be labelled "out of control."

However, if there is a pattern in the ratios, then the device is introducing a systematic error, even if all of the ratios fall within the control bands. This systematic error should be corrected by modelling the ratios as a function of peak location and using the model to correct future observations on unknowns.

Let the measured relative intensities on the 12 peaks be y_i and the certified values be c_i, so the ratios to model are

$$r_i = \frac{y_i}{c_i}.$$

Fit a simple model, using ordinary least squares regression, which describes these ratios adequately as a function of peak location (x_i). The fitted model has m parameters: m=1 for a horizontal straight line, m=2 for a slanted straight line, m=3 for a quadratic, etc. The residual sum of squares from the fit, plus an allowance for the uncertainty in the certified values on the certificate, is:

$$RSS_{full} = \sum_{1}^{n} (r_i - \hat{r}_i)^2 + (n-m)\frac{(RSD)^2}{25}$$

and the residual degrees of freedom for this "full" model are n - m. The RSD = 0.0206 if peak areas were used, or 0.0262 if peak heights were used. The residual sum of squares for the horizontal line at 1 (which describes "ideal" data), plus an allowance for the uncertainty in the certified values on the certificate, is:

$$RSS_1 = \sum_{1}^{n} (r_i - 1)^2 + (n-1)\frac{(RSD)^2}{25}$$

and the residual degrees of freedom are n - 1. Then if the test statistic

$$F = \frac{\left\{RSS_1 - RSS_{full} + (m-1)\frac{(RSD)^2}{25}\right\} / (m-1)}{\left\{RSS_{full} + (n-m)\frac{(RSD)^2}{25}\right\} / (n-m)}$$

is larger than the upper 0.05 percentage point from the F distribution with m-1 numerator and n-m denominator degrees of freedom (commonly provided in table form in introductory statistics texts), the correction model is needed.

To use the correction model: A new peak angle x* and a new relative intensity y* are measured on an unknown sample. If $\hat{r}^*$ is the predicted value of the fitted model at x*, the corrected relative intensity for the new peak angle on the unknown sample is

$$\text{corrected relative intensity} = \frac{y^*}{\hat{r}^*}.$$

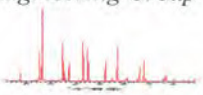
Appendix 6
PDF cards for the identified crystalline phases presented in this report

The following PDF cards are for the crystalline phases identified in this report.
The PDF card includes information about the crystal structure, the reference, the X-ray radiation wavelength, the calculated density and the observed density (if published), the strongest lines and the corresponding 2 theta angles. The table presented at the end of each PDF card includes information about the 2 theta angle positions, interplanar distances at that angle, the intensity of that particular reflection and Miller indices at that particular 2 theta. The compounds identified in this report match the PDF card peaks, which are the stick figures in the report.

1) Quartz SiO_2
PDF#01-070-3755(RDB)
Radiation=CuKá1 Lambda=1.5406
Ref: Gualtieri, A.F. J. Appl. Crystallogr., v33 p267 (2000)
Hexagonal - Powder Diffraction, P3121 (152) Z=3
CELL: 4.9158 x 4.9158 x 5.4091 <90.0 x 90.0 x 120.0>
Density(c)=2.644
Strong Lines: 3.35/X 4.26/2 1.82/1 2.46/1 1.54/1 2.28/1

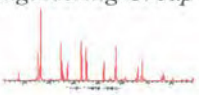
2-Theta	d(Å)	I(f)	(h k l)	Theta	1/(2d)	2pi/d	n^2
20.849	4.2572	21.8	(1 0 0)	10.424	0.1174	1.4759	
26.624	3.3454	100.0	(0 1 1)	13.312	0.1495	1.8782	
36.528	2.4579	6.5	(1 1 0)	18.264	0.2034	2.5563	
39.441	2.2828	6.3	(1 0 2)	19.720	0.2190	2.7524	
40.270	2.2377	2.9	(1 1 1)	20.135	0.2234	2.8079	
42.431	2.1286	4.4	(2 0 0)	21.216	0.2349	2.9518	
45.771	1.9808	2.7	(2 0 1)	22.885	0.2524	3.1721	
50.109	1.8190	9.9	(1 1 2)	25.055	0.2749	3.4543	
50.583	1.8030	0.3	(0 0 3)	25.291	0.2773	3.4848	
54.841	1.6727	3.0	(0 2 2)	27.420	0.2989	3.7564	
55.286	1.6603	1.3	(0 1 3)	27.643	0.3012	3.7844	
57.204	1.6091	0.2	(2 1 0)	28.602	0.3107	3.9049	
59.928	1.5423	6.4	(1 2 1)	29.964	0.3242	4.0740	
63.990	1.4538	1.2	(1 1 3)	31.995	0.3439	4.3219	
65.751	1.4191	0.3	(3 0 0)	32.876	0.3523	4.4277	
67.702	1.3828	3.7	(1 2 2)	33.851	0.3616	4.5437	
68.096	1.3758	4.5	(2 0 3)	34.048	0.3634	4.5669	
68.276	1.3726	4.9	(0 3 1)	34.138	0.3643	4.5775	
73.407	1.2888	1.3	(1 0 4)	36.704	0.3880	4.8751	
75.613	1.2566	1.6	(3 0 2)	37.807	0.3979	5.0001	
77.628	1.2290	0.8	(2 2 0)	38.814	0.4069	5.1126	
79.827	1.2005	1.8	(2 1 3)	39.913	0.4165	5.2337	
79.996	1.1984	1.1	(2 2 1)	39.998	0.4172	5.2429	

81.105	1.1848	1.4	(1 1 4)	40.553	0.4220	5.3032
81.443	1.1807	1.7	(3 1 0)	40.722	0.4235	5.3214
83.787	1.1536	0.9	(1 3 1)	41.894	0.4334	5.4467
84.886	1.1414	0.2	(2 0 4)	42.443	0.4381	5.5047
87.017	1.1189	0.1	(2 2 2)	43.509	0.4469	5.6157
87.383	1.1151	0.2	(3 0 3)	43.691	0.4484	5.6345
90.771	1.0821	1.4	(1 3 2)	45.385	0.4621	5.8064
92.731	1.0643	0.2	(4 0 0)	46.366	0.4698	5.9036
94.558	1.0485	0.7	(0 1 5)	47.279	0.4769	5.9925
95.060	1.0443	0.5	(4 0 1)	47.530	0.4788	6.0168
96.159	1.0352	0.7	(2 1 4)	48.080	0.4830	6.0693
98.672	1.0155	0.6	(2 2 3)	49.336	0.4924	6.1873
102.147	0.9902	0.5	(1 1 5)	51.074	0.5050	6.3457
102.147	0.9902	0.5	(0 4 2)	51.074	0.5050	6.3457
102.489	0.9878	0.4	(1 3 3)	51.244	0.5062	6.3609
103.784	0.9790	0.1	(0 3 4)	51.892	0.5107	6.4182
104.128	0.9767	0.4	(3 2 0)	52.064	0.5119	6.4333
106.016	0.9644	0.1	(0 2 5)	53.008	0.5184	6.5150
106.537	0.9611	0.6	(2 3 1)	53.269	0.5202	6.5373
112.027	0.9290	0.1	(4 1 0)	56.013	0.5382	6.7634
113.974	0.9186	0.2	(3 2 2)	56.987	0.5443	6.8399
114.373	0.9165	0.5	(4 0 3)	57.187	0.5455	6.8554
114.557	0.9156	0.6	(4 1 1)	57.278	0.5461	6.8624
115.766	0.9095	0.2	(2 2 4)	57.883	0.5498	6.9085
117.397	0.9015	0.1	(0 0 6)	58.698	0.5546	6.9696
118.186	0.8978	0.4	(1 2 5)	59.093	0.5569	6.9986
120.011	0.8894	0.3	(3 1 4)	60.005	0.5622	7.0644
121.710	0.8820	0.1	(1 0 6)	60.855	0.5669	7.1241
122.498	0.8786	0.2	(1 4 2)	61.249	0.5691	7.1513
127.106	0.8603	0.1	(3 0 5)	63.553	0.5812	7.3032
127.526	0.8588	0.1	(2 3 3)	63.763	0.5822	7.3165
131.040	0.8464	0.1	(1 1 6)	65.520	0.5908	7.4236
132.649	0.8411	0.1	(0 5 1)	66.325	0.5945	7.4703
134.155	0.8363	0.1	(0 4 4)	67.077	0.5978	7.5127
136.225	0.8301	0.4	(0 2 6)	68.112	0.6023	7.5689
137.738	0.8258	0.5	(1 4 3)	68.869	0.6055	7.6084
140.166	0.8193	0.1	(3 3 0)	70.083	0.6103	7.6690
143.052	0.8121	0.6	(5 0 2)	71.526	0.6157	7.7365
143.052	0.8121	0.6	(2 2 5)	71.526	0.6157	7.7365
143.946	0.8101	0.5	(3 3 1)	71.973	0.6172	7.7564
146.447	0.8045	0.3	(4 2 0)	73.224	0.6215	7.8097
149.906	0.7976	0.2	(1 3 5)	74.953	0.6268	7.8772



2) Rutile, syn (Titanium Oxide) Ti O2PDF#01-077-0440(RDB)
Radiation=CuKá1 Lambda=1.5406 Filter=
Calibration= 2T=27.438-143.112 I/Ic(RIR)=3.6
Ref: Seki, H., Ishizawa, N., Mizutani, N., Kato, M. Yogyo Kyokaishi (J. Ceram. Assoc. Jpn.), v92 p219 (1984)
Tetragonal - Single Crystal, P42/mnm (136) Z=2
CELL: 4.5934 x 4.5934 x 2.9575 <90.0 x 90.0 x 90.0> P.S=tP6.00
Density(c)=4.252
Strong Lines: 3.25/X 1.69/5 2.49/4 2.19/2 1.36/2 1.62/1 1.35/1 1.48/1
Calculated Pattern Original Remarks: Stable from 800 to 1800 K (2nd ref., Tomaszewski), 300-800 K: Pbca.
Temperature of Data Collection: 300 K.
Wyckoff Sequence: f a(P42/MNM).
Unit Cell Data Source: Single Crystal.

2-Theta	d(Å)	I(f)	(h k l)	Theta	1/(2d)	2pi/d	n^2
27.438	3.2480	100.0	(1 1 0)	13.719	0.1539	1.9345	
36.091	2.4867	43.6	(1 0 1)	18.046	0.2011	2.5268	
39.193	2.2967	6.4	(2 0 0)	19.596	0.2177	2.7357	
41.250	2.1868	17.0	(1 1 1)	20.625	0.2286	2.8733	
44.046	2.0542	6.0	(2 1 0)	22.023	0.2434	3.0587	
54.331	1.6872	47.8	(2 1 1)	27.165	0.2964	3.7241	
56.630	1.6240	13.7	(2 2 0)	28.315	0.3079	3.8689	
62.787	1.4788	6.5	(0 0 2)	31.393	0.3381	4.2490	
64.052	1.4526	6.3	(3 1 0)	32.026	0.3442	4.3256	
65.520	1.4235	0.5	(2 2 1)	32.760	0.3512	4.4138	
69.014	1.3597	15.1	(3 0 1)	34.507	0.3677	4.6209	
69.829	1.3458	7.7	(1 1 2)	34.915	0.3715	4.6686	
72.429	1.3038	0.8	(3 1 1)	36.214	0.3835	4.8191	
74.405	1.2740	0.2	(3 2 0)	37.203	0.3925	4.9319	
76.565	1.2433	1.6	(2 0 2)	38.283	0.4021	5.0535	
79.858	1.2001	0.9	(2 1 2)	39.929	0.4166	5.2354	
82.348	1.1700	2.9	(3 2 1)	41.174	0.4273	5.3701	
84.255	1.1484	2.0	(4 0 0)	42.128	0.4354	5.4715	
87.487	1.1141	0.7	(4 1 0)	43.743	0.4488	5.6399	
89.579	1.0934	4.8	(2 2 2)	44.789	0.4573	5.7465	
90.710	1.0827	2.5	(3 3 0)	45.355	0.4618	5.8034	
95.268	1.0426	4.1	(4 1 1)	47.634	0.4796	6.0267	
96.035	1.0363	3.3	(3 1 2)	48.017	0.4825	6.0634	
97.173	1.0271	1.8	(4 2 0)	48.587	0.4868	6.1173	
98.515	1.0167	0.1	(3 3 1)	49.257	0.4918	6.1800	
105.103	0.9703	0.4	(4 2 1)	52.551	0.5153	6.4757	
106.099	0.9639	1.3	(1 0 3)	53.050	0.5187	6.5186	
109.484	0.9433	0.4	(1 1 3)	54.742	0.5300	6.6606	
113.960	0.9187	0.1	(4 3 0)	56.980	0.5443	6.8394	

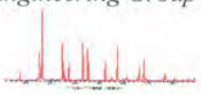


116.270	0.9070	1.9	(4 0 2)	58.135	0.5513	6.9276
117.538	0.9008	2.1	(5 1 0)	58.769	0.5550	6.9748
119.923	0.8898	1.0	(4 1 2)	59.962	0.5619	7.0613
120.151	0.8888	4.2	(2 1 3)	60.075	0.5626	7.0694
122.804	0.8773	3.9	(4 3 1)	61.402	0.5699	7.1617
123.716	0.8736	2.8	(3 3 2)	61.858	0.5724	7.1926
126.728	0.8618	0.1	(5 1 1)	63.364	0.5802	7.2912
129.129	0.8530	0.1	(5 2 0)	64.565	0.5862	7.3662
131.880	0.8436	2.5	(4 2 2)	65.940	0.5927	7.4482
136.657	0.8289	3.0	(3 0 3)	68.328	0.6032	7.5803
140.063	0.8196	5.1	(5 2 1)	70.031	0.6101	7.6665
141.582	0.8157	0.2	(3 1 3)	70.791	0.6130	7.7027
143.112	0.8120	0.7	(4 4 0)	71.556	0.6158	7.7379

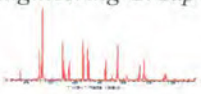
3) Corundum (Aluminum Oxide), Al_2O_3, PDF#01-071-6478

Radiation=CuKá1 Lambda=1.5406
Ref: Riello, P., Canton, P., Fagherazzi, G. Powder Diffraction, v12 p160 (1997)
Rhombohedral - Powder Diffraction, R-3c (167) Z=6
CELL: 4.7598 x 4.7598 x 12.9924 <90.0 x 90.0 x 120.0> P.S=hR10.00 (Á)
Density(c)=3.985
Strong Lines: 2.55/X 2.09/X 1.60/9 3.48/7 1.37/5 1.74/5 2.38/5 1.40/3
ICSD Collection Code: 77810.
Calculated Pattern Original Remarks: R(Bragg)=0.03.
Minor Warning: 10%<R factor<15% (for powder).
Wyckoff Sequence: e c(R3-CH)

2-Theta	d(Å)	I(f)	(h k l)	Theta	1/(2d)	2pi/d	n^2
25.573	3.4805	67.0	(0 1 2)	12.786	0.1437	1.8052	
35.147	2.5512	100.0	(1 0 4)	17.574	0.1960	2.4628	
37.770	2.3799	46.0	(1 1 0)	18.885	0.2101	2.6401	
41.676	2.1654	0.5	(0 0 6)	20.838	0.2309	2.9016	
43.347	2.0857	96.5	(1 1 3)	21.674	0.2397	3.0125	
46.170	1.9646	1.5	(2 0 2)	23.085	0.2545	3.1983	
52.544	1.7403	46.2	(0 2 4)	26.272	0.2873	3.6105	
57.494	1.6016	91.0	(1 1 6)	28.747	0.3122	3.9230	
59.729	1.5469	2.2	(2 1 1)	29.865	0.3232	4.0617	
61.118	1.5151	3.4	(1 2 2)	30.559	0.3300	4.1472	
61.299	1.5110	8.2	(0 1 8)	30.650	0.3309	4.1583	
66.507	1.4048	34.4	(2 1 4)	33.254	0.3559	4.4728	
68.195	1.3740	52.6	(3 0 0)	34.098	0.3639	4.5728	
70.405	1.3362	1.1	(1 2 5)	35.203	0.3742	4.7022	
74.294	1.2756	1.3	(2 0 8)	37.147	0.3920	4.9256	
76.871	1.2392	14.7	(1,0,10)	38.435	0.4035	5.0706	
77.230	1.2343	8.6	(1 1 9)	38.615	0.4051	5.0906	
80.407	1.1933	0.7	(2 1 7)	40.204	0.4190	5.2653	

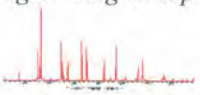


80.682	1.1900	5.7	(2 2 0)	40.341	0.4202	5.2802
83.203	1.1602	0.6	(0 3 6)	41.601	0.4310	5.4157
84.338	1.1474	4.6	(2 2 3)	42.169	0.4358	5.4759
85.121	1.1389	0.3	(1 3 1)	42.560	0.4390	5.5170
86.333	1.1260	3.4	(3 1 2)	43.167	0.4441	5.5803
86.492	1.1243	2.7	(1 2 8)	43.246	0.4447	5.5885
88.990	1.0991	6.3	(0,2,10)	44.495	0.4549	5.7167
90.707	1.0827	1.6	(0,0,12)	45.354	0.4618	5.8033
91.170	1.0784	7.4	(1 3 4)	45.585	0.4636	5.8263
94.800	1.0465	0.4	(3 1 5)	47.400	0.4778	6.0042
95.231	1.0429	14.4	(2 2 6)	47.615	0.4795	6.0250
98.370	1.0178	1.8	(0 4 2)	49.185	0.4913	6.1733
101.064	0.9978	10.3	(2,1,10)	50.532	0.5011	6.2969
102.819	0.9855	0.3	(1,1,12)	51.409	0.5074	6.3756
103.293	0.9823	1.9	(4 0 4)	51.646	0.5090	6.3966
104.620	0.9734	0.1	(1 3 7)	52.310	0.5137	6.4548
109.511	0.9432	0.4	(3 2 1)	54.756	0.5301	6.6617
109.848	0.9412	0.3	(1,2,11)	54.924	0.5312	6.6755
110.968	0.9349	2.9	(3 1 8)	55.484	0.5348	6.7210
114.050	0.9182	2.0	(2 2 9)	57.025	0.5445	6.8428
116.069	0.9080	9.4	(3 2 4)	58.034	0.5507	6.9200
116.600	0.9054	6.5	(0,1,14)	58.300	0.5523	6.9399
117.817	0.8995	5.2	(4 1 0)	58.908	0.5559	6.9851
120.180	0.8887	0.3	(2 3 5)	60.090	0.5626	7.0704
122.000	0.8807	3.2	(4 1 3)	61.000	0.5677	7.1341
124.567	0.8701	1.9	(0 4 8)	62.284	0.5746	7.2209
127.657	0.8583	10.5	(1,3,10)	63.829	0.5826	7.3206
129.859	0.8504	4.2	(0,3,12)	64.930	0.5879	7.3884
131.092	0.8462	4.1	(2,0,14)	65.546	0.5909	7.4251
132.178	0.8426	0.4	(3 2 7)	66.089	0.5934	7.4568
132.608	0.8412	0.3	(2,1,13)	66.304	0.5944	7.4692
136.032	0.8307	15.7	(1 4 6)	68.016	0.6019	7.5638
139.339	0.8215	0.1	(3,1,11)	69.669	0.6087	7.6487
140.977	0.8172	0.4	(2 3 8)	70.489	0.6118	7.6885
142.308	0.8139	2.1	(1,1,15)	71.154	0.6143	7.7196
145.131	0.8074	6.1	(4,0,10)	72.565	0.6193	7.7821
148.258	0.8008	0.2	(2,2,12)	74.129	0.6244	7.8459
149.147	0.7991	4.1	(0 5 4)	74.574	0.6257	7.8630

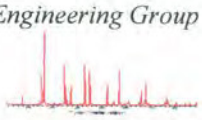


4) PDF#01-070-0909(RDB): Calcium Sulfate, Ca S O4
Radiation=CuKá1 Lambda=1.5406
Ref: Morikawa, H., Tomita, T., Minato, I., Iwai, S. Acta Crystallogr., Sec. B: Struct. Crystallogr. Cryst. Chem., v31 p2164 (1975)
Orthorhombic - Single Crystal, Bmmb (63) Z=4
CELL: 6.992 x 6.999 x 6.24 <90.0 x 90.0 x 90.0> P.S=oC24.00
Density(c)=2.961
Strong Lines: 3.50/X 3.50/X 2.85/3 2.21/2 2.33/2 2.33/2 1.87/1 1.65/1

2-Theta	d(Å)	I(f)	(h k l)	Theta	1/(2d)	2pi/d	n^2
19.047	4.6556	0.1	(1 0 1)	9.524	0.1074	1.3496	
22.924	3.8764	5.1	(1 1 1)	11.462	0.1290	1.6209	
25.458	3.4960	100.0	(0 2 0)	12.729	0.1430	1.7973	
25.458	3.4960	100.0	(2 0 0)	12.729	0.1430	1.7973	
28.587	3.1200	2.2	(0 0 2)	14.294	0.1603	2.0138	
31.365	2.8497	31.8	(0 1 2)	15.683	0.1755	2.2049	
31.968	2.7974	3.1	(1 2 1)	15.984	0.1787	2.2461	
36.293	2.4733	7.2	(2 2 0)	18.146	0.2022	2.5404	
38.648	2.3278	17.9	(2 0 2)	19.324	0.2148	2.6992	
38.648	2.3278	17.9	(0 2 2)	19.324	0.2148	2.6992	
40.820	2.2088	19.8	(2 1 2)	20.410	0.2264	2.8446	
41.318	2.1833	6.1	(3 0 1)	20.659	0.2290	2.8778	
43.346	2.0858	6.0	(3 1 1)	21.673	0.2397	3.0124	
43.346	2.0858	6.0	(1 3 1)	21.673	0.2397	3.0124	
45.458	1.9937	4.3	(1 0 3)	22.729	0.2508	3.1516	
46.836	1.9382	2.9	(2 2 2)	23.418	0.2580	3.2418	
47.374	1.9174	0.4	(1 1 3)	23.687	0.2608	3.2770	
48.695	1.8684	12.9	(0 3 2)	24.348	0.2676	3.3629	
49.144	1.8524	2.9	(3 2 1)	24.572	0.2699	3.3919	
52.294	1.7480	6.7	(0 4 0)	26.147	0.2860	3.5945	
52.294	1.7480	6.7	(4 0 0)	26.147	0.2860	3.5945	
52.805	1.7323	0.3	(1 2 3)	26.402	0.2886	3.6271	
55.739	1.6478	12.6	(2 3 2)	27.869	0.3034	3.8130	
57.790	1.5941	2.1	(3 3 1)	28.895	0.3136	3.9414	
59.022	1.5638	2.9	(4 2 0)	29.511	0.3197	4.0180	
59.022	1.5638	2.9	(2 4 0)	29.511	0.3197	4.0180	
59.179	1.5600	1.7	(0 0 4)	29.589	0.3205	4.0277	
60.679	1.5250	2.5	(0 4 2)	30.339	0.3279	4.1202	
60.679	1.5250	2.5	(4 0 2)	30.339	0.3279	4.1202	
60.781	1.5226	1.5	(0 1 4)	30.391	0.3284	4.1265	
61.118	1.5151	0.9	(3 1 3)	30.559	0.3300	4.1471	
61.118	1.5151	0.9	(1 3 3)	30.559	0.3300	4.1471	
62.258	1.4900	4.3	(4 1 2)	31.129	0.3356	4.2168	
65.464	1.4246	2.5	(0 2 4)	32.732	0.3510	4.4105	
65.464	1.4246	2.5	(2 0 4)	32.732	0.3510	4.4105	

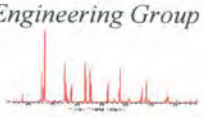


65.774	1.4186	0.9	(3 2 3)	32.887	0.3525	4.4291
66.835	1.3987	1.3	(2 4 2)	33.417	0.3575	4.4923
66.835	1.3987	1.3	(4 2 2)	33.417	0.3575	4.4923
66.981	1.3960	2.1	(2 1 4)	33.490	0.3582	4.5009
68.688	1.3654	0.5	(5 0 1)	34.344	0.3662	4.6018
68.688	1.3654	0.5	(3 4 1)	34.344	0.3662	4.6018
70.147	1.3405	0.1	(1 5 1)	35.073	0.3730	4.6871
70.218	1.3393	0.1	(5 1 1)	35.109	0.3733	4.6913
71.436	1.3195	3.9	(2 2 4)	35.718	0.3789	4.7619
71.710	1.3151	2.1	(1 4 3)	35.855	0.3802	4.7778
72.882	1.2968	1.4	(0 3 4)	36.441	0.3856	4.8451
74.236	1.2765	4.8	(0 5 2)	37.118	0.3917	4.9223
74.236	1.2765	4.8	(4 3 2)	37.118	0.3917	4.9223
77.055	1.2366	0.8	(4 4 0)	38.528	0.4043	5.0809
77.655	1.2286	0.1	(1 0 5)	38.828	0.4070	5.1142
78.624	1.2159	3.0	(2 3 4)	39.312	0.4112	5.1677
79.072	1.2101	0.2	(1 1 5)	39.536	0.4132	5.1924
79.900	1.1996	1.7	(2 5 2)	39.950	0.4168	5.2377
81.684	1.1779	0.3	(3 5 1)	40.842	0.4245	5.3344
81.684	1.1779	0.3	(5 3 1)	40.842	0.4245	5.3344
82.653	1.1665	1.7	(0 6 0)	41.326	0.4286	5.3864
82.754	1.1653	2.0	(6 0 0)	41.377	0.4291	5.3918
82.878	1.1639	1.8	(4 0 4)	41.439	0.4296	5.3984
82.878	1.1639	1.8	(0 4 4)	41.439	0.4296	5.3984
83.174	1.1605	1.0	(5 0 3)	41.587	0.4308	5.4142
83.174	1.1605	1.0	(3 4 3)	41.587	0.4308	5.4142
83.287	1.1592	0.4	(1 2 5)	41.644	0.4313	5.4202
84.140	1.1496	0.4	(4 4 2)	42.070	0.4349	5.4654
84.275	1.1481	0.8	(4 1 4)	42.138	0.4355	5.4725
84.503	1.1456	0.4	(1 5 3)	42.252	0.4364	5.4846
84.503	1.1456	0.4	(5 1 3)	42.252	0.4364	5.4846
85.806	1.1315	0.1	(1 6 1)	42.903	0.4419	5.5529
88.236	1.1065	1.8	(2 6 0)	44.118	0.4519	5.6783
88.415	1.1048	3.6	(4 2 4)	44.207	0.4526	5.6874
88.415	1.1048	3.6	(2 4 4)	44.207	0.4526	5.6874
88.742	1.1015	1.8	(5 2 3)	44.371	0.4539	5.7041
88.877	1.1002	0.5	(3 0 5)	44.438	0.4545	5.7109
89.658	1.0926	0.2	(0 6 2)	44.829	0.4576	5.7505
89.758	1.0917	0.1	(6 0 2)	44.879	0.4580	5.7556
90.243	1.0871	0.1	(3 1 5)	45.121	0.4600	5.7800
90.243	1.0871	0.1	(1 3 5)	45.121	0.4600	5.7800
91.146	1.0786	1.4	(6 1 2)	45.573	0.4636	5.8252
91.429	1.0760	0.8	(5 4 1)	45.714	0.4647	5.8392
94.434	1.0496	0.1	(3 2 5)	47.217	0.4764	5.9866
95.228	1.0429	0.9	(2 6 2)	47.614	0.4794	6.0248



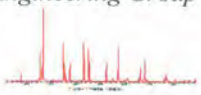
95.396	1.0415	1.7	(4 3 4)	47.698	0.4801	6.0329
95.396	1.0415	1.7	(0 5 4)	47.698	0.4801	6.0329
95.577	1.0400	1.1	(3 5 3)	47.788	0.4808	6.0415
95.577	1.0400	1.1	(0 0 6)	47.788	0.4808	6.0415
95.691	1.0391	1.0	(5 3 3)	47.846	0.4812	6.0470
96.657	1.0312	1.0	(4 5 2)	48.328	0.4849	6.0929
96.954	1.0289	0.9	(0 1 6)	48.477	0.4860	6.1069
96.954	1.0289	0.9	(3 6 1)	48.477	0.4860	6.1069
99.828	1.0068	0.2	(1 6 3)	49.914	0.4966	6.2406
100.009	1.0055	0.1	(1 4 5)	50.005	0.4973	6.2489
100.974	0.9985	0.5	(2 5 4)	50.487	0.5008	6.2929
101.191	0.9969	1.4	(2 0 6)	50.596	0.5016	6.3027
101.191	0.9969	1.4	(0 2 6)	50.596	0.5016	6.3027
101.445	0.9951	0.7	(3 3 5)	50.722	0.5025	6.3141
102.345	0.9888	1.7	(6 3 2)	51.173	0.5057	6.3545
102.621	0.9869	1.1	(2 1 6)	51.311	0.5067	6.3668
102.621	0.9869	1.1	(7 0 1)	51.311	0.5067	6.3668
104.062	0.9771	0.1	(5 5 1)	52.031	0.5117	6.4304
104.062	0.9771	0.1	(7 1 1)	52.031	0.5117	6.4304
105.100	0.9703	1.4	(6 4 0)	52.550	0.5153	6.4756
105.100	0.9703	1.4	(4 6 0)	52.550	0.5153	6.4756
105.285	0.9691	1.2	(4 4 4)	52.643	0.5159	6.4836
105.590	0.9671	0.8	(5 4 3)	52.795	0.5170	6.4968
106.929	0.9587	0.7	(2 2 6)	53.464	0.5215	6.5539
107.996	0.9522	0.5	(0 7 2)	53.998	0.5251	6.5989
108.470	0.9493	0.2	(0 3 6)	54.235	0.5267	6.6186
108.470	0.9493	0.2	(7 2 1)	54.235	0.5267	6.6186
111.085	0.9342	0.1	(0 6 4)	55.542	0.5352	6.7257
111.192	0.9336	0.1	(6 0 4)	55.596	0.5356	6.7300
111.400	0.9324	0.1	(3 6 3)	55.700	0.5362	6.7384
111.640	0.9311	0.1	(3 4 5)	55.820	0.5370	6.7480
111.640	0.9311	0.1	(5 0 5)	55.820	0.5370	6.7480
112.483	0.9265	0.5	(6 4 2)	56.241	0.5397	6.7815
112.483	0.9265	0.5	(4 6 2)	56.241	0.5397	6.7815
112.689	0.9254	0.3	(6 1 4)	56.344	0.5403	6.7896
113.142	0.9230	0.2	(1 5 5)	56.571	0.5417	6.8074
113.142	0.9230	0.2	(5 1 5)	56.571	0.5417	6.8074
113.957	0.9187	0.8	(2 7 2)	56.979	0.5443	6.8393
114.350	0.9167	0.4	(2 3 6)	57.175	0.5455	6.8544
115.972	0.9085	0.1	(7 3 1)	57.986	0.5504	6.9164
115.972	0.9085	0.1	(3 7 1)	57.986	0.5504	6.9164
117.184	0.9025	0.7	(2 6 4)	58.592	0.5540	6.9617
117.184	0.9025	0.7	(6 2 4)	58.592	0.5540	6.9617
117.754	0.8998	0.5	(5 2 5)	58.877	0.5557	6.9828
117.754	0.8998	0.5	(7 0 3)	58.877	0.5557	6.9828

118.794	0.8949	0.4	(4 5 4)	59.397	0.5587	7.0207
118.999	0.8940	1.0	(0 4 6)	59.499	0.5593	7.0281
118.999	0.8940	1.0	(4 0 6)	59.499	0.5593	7.0281
120.650	0.8866	0.5	(5 6 1)	60.325	0.5640	7.0871
120.650	0.8866	0.5	(4 1 6)	60.325	0.5640	7.0871
121.175	0.8843	0.3	(1 0 7)	60.588	0.5654	7.1055
122.812	0.8773	0.1	(1 1 7)	61.406	0.5699	7.1620
123.397	0.8749	0.3	(0 8 0)	61.698	0.5715	7.1818
123.610	0.8740	0.3	(8 0 0)	61.805	0.5721	7.1890
124.099	0.8720	0.2	(7 2 3)	62.049	0.5734	7.2054
125.417	0.8668	0.8	(6 3 4)	62.709	0.5768	7.2489
125.624	0.8660	1.3	(4 2 6)	62.812	0.5774	7.2556
125.624	0.8660	1.3	(2 4 6)	62.812	0.5774	7.2556
125.931	0.8648	0.5	(3 5 5)	62.965	0.5782	7.2656
125.931	0.8648	0.5	(5 3 5)	62.965	0.5782	7.2656
126.971	0.8608	0.8	(6 5 2)	63.485	0.5808	7.2989
127.410	0.8592	0.4	(7 4 1)	63.705	0.5819	7.3128
127.920	0.8573	0.1	(1 2 7)	63.960	0.5832	7.3288
130.355	0.8487	0.3	(2 8 0)	65.178	0.5891	7.4033
130.575	0.8480	0.2	(8 2 0)	65.287	0.5897	7.4098
131.172	0.8459	0.1	(1 6 5)	65.586	0.5911	7.4275
132.489	0.8416	0.4	(8 0 2)	66.244	0.5941	7.4657
132.489	0.8416	0.4	(0 7 4)	66.244	0.5941	7.4657
132.829	0.8405	0.1	(3 7 3)	66.414	0.5949	7.4755
132.981	0.8400	0.2	(7 3 3)	66.490	0.5952	7.4798
134.214	0.8362	0.8	(4 7 2)	67.107	0.5980	7.5144
134.401	0.8356	0.8	(8 1 2)	67.200	0.5984	7.5195
134.653	0.8348	0.3	(0 5 6)	67.327	0.5989	7.5265
134.653	0.8348	0.3	(4 3 6)	67.327	0.5989	7.5265
135.385	0.8326	0.1	(3 0 7)	67.693	0.6005	7.5464
138.244	0.8244	1.1	(6 6 0)	69.122	0.6065	7.6213
138.429	0.8239	0.9	(4 6 4)	69.215	0.6069	7.6260
138.429	0.8239	0.9	(6 4 4)	69.215	0.6069	7.6260
138.874	0.8227	0.7	(5 6 3)	69.437	0.6077	7.6371
139.147	0.8220	0.5	(5 4 5)	69.573	0.6083	7.6440
140.514	0.8184	0.5	(8 2 2)	70.257	0.6109	7.6774
140.514	0.8184	0.5	(2 7 4)	70.257	0.6109	7.6774
143.122	0.8120	0.5	(2 5 6)	71.561	0.6158	7.7381
143.122	0.8120	0.5	(3 8 1)	71.561	0.6158	7.7381
143.974	0.8100	0.4	(3 2 7)	71.987	0.6173	7.7571
145.643	0.8063	0.1	(5 7 1)	72.822	0.6201	7.7930
145.643	0.8063	0.1	(7 5 1)	72.822	0.6201	7.7930
148.486	0.8004	0.2	(3 6 5)	74.243	0.6247	7.8503
148.486	0.8004	0.2	(7 4 3)	74.243	0.6247	7.8503

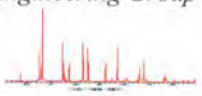


5) PDF#01-089-1460(RDB): Anorthite (Calcium Aluminum Silicate)
Ca (Al2 Si2 O8)
Radiation=CuKá1 Lambda=1.5406 Filter=
Ref: Angel, R.J., Carpenter, M.A., Finger, L.W. Am. Mineral., v75 p150 (1990)
Triclinic - Single Crystal, P-1 (2) Z=8
CELL: 8.1796 x 12.8747 x 14.172 <93.134 x 115.885 x 91.236> P.S=aP104.00
Density(c)=2.76
Strong Lines: 3.20/X 3.18/9 3.18/9 3.18/7 3.26/6 3.26/6 4.04/6 4.04/6

2-Theta	d(Å)	I(f)	(h k l)	Theta	1/(2d)	2pi/d	n^2
6.945	12.7177	0.5	(0 1 0)	3.472	0.0393	0.4941	
6.945	12.7177	0.5	(0 0 1)	3.472	0.0393	0.4941	
9.425	9.3755	0.8	(0-1 1)	4.713	0.0533	0.6702	
10.125	8.7297	0.1	(0 1 1)	5.062	0.0573	0.7197	
10.940	8.0811	0.1	(-1 0 1)	5.470	0.0619	0.7775	
12.994	6.8075	9.6	(-1-1 1)	6.497	0.0734	0.9230	
13.565	6.5222	11.8	(-1 1 0)	6.783	0.0767	0.9633	
13.783	6.4195	4.0	(0 2 0)	6.892	0.0779	0.9788	
13.783	6.4195	4.0	(-1 0 2)	6.892	0.0779	0.9788	
13.915	6.3589	1.4	(0 0 2)	6.958	0.0786	0.9881	
14.997	5.9025	1.3	(0-2 1)	7.499	0.0847	1.0645	
15.301	5.7859	0.5	(-1-1 2)	7.651	0.0864	1.0859	
15.645	5.6597	0.2	(-1 1 2)	7.822	0.0883	1.1102	
15.889	5.5733	0.8	(0 1 2)	7.944	0.0897	1.1274	
15.889	5.5733	0.8	(0 2 1)	7.944	0.0897	1.1274	
16.350	5.4170	0.1	(1 0 1)	8.175	0.0923	1.1599	
17.318	5.1164	2.6	(1-1 1)	8.659	0.0977	1.2280	
17.540	5.0520	0.4	(-1 2 1)	8.770	0.0990	1.2437	
17.719	5.0014	1.6	(-1-2 1)	8.860	0.1000	1.2563	
18.185	4.8743	0.1	(1 1 1)	9.093	0.1026	1.2890	
18.915	4.6878	18.0	(0-2 2)	9.458	0.1067	1.3403	
19.308	4.5933	0.7	(-1-2 2)	9.654	0.1089	1.3679	
19.942	4.4486	0.8	(-1-1 3)	9.971	0.1124	1.4124	
20.329	4.3649	0.3	(0 2 2)	10.165	0.1146	1.4395	
20.550	4.3185	0.9	(-1 1 3)	10.275	0.1158	1.4550	
20.711	4.2853	1.2	(0 3 0)	10.355	0.1167	1.4662	
20.711	4.2853	1.2	(1-2 1)	10.355	0.1167	1.4662	
20.938	4.2393	2.1	(0 0 3)	10.469	0.1179	1.4821	
21.412	4.1465	1.0	(0-3 1)	10.706	0.1206	1.5153	
21.585	4.1137	0.8	(0-1 3)	10.792	0.1215	1.5274	
21.980	4.0405	56.1	(-2 0 1)	10.990	0.1237	1.5550	
21.980	4.0405	56.1	(-2 0 2)	10.990	0.1237	1.5550	
22.367	3.9715	1.3	(0 3 1)	11.184	0.1259	1.5821	
22.697	3.9145	12.4	(1-1 2)	11.349	0.1277	1.6051	
23.034	3.8581	1.6	(-2 1 2)	11.517	0.1296	1.6286	

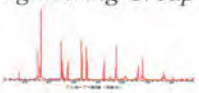


23.034	3.8581	1.6	(-1-2 3)	11.517	0.1296	1.6286
23.501	3.7825	31.0	(-1 3 0)	11.750	0.1322	1.6611
23.670	3.7558	21.8	(1 1 2)	11.835	0.1331	1.6730
24.091	3.6911	2.9	(-2 0 3)	12.046	0.1355	1.7023
24.091	3.6911	2.9	(-1 2 3)	12.046	0.1355	1.7023
24.303	3.6594	2.4	(0-2 3)	12.151	0.1366	1.7170
24.575	3.6196	37.9	(1 3 0)	12.287	0.1381	1.7359
25.007	3.5580	1.0	(-2-1 3)	12.503	0.1405	1.7659
25.159	3.5368	0.8	(-2 1 3)	12.579	0.1414	1.7765
25.159	3.5368	0.8	(-1 0 4)	12.579	0.1414	1.7765
25.343	3.5115	3.9	(-1 3 2)	12.672	0.1424	1.7893
25.619	3.4743	5.3	(1-3 1)	12.809	0.1439	1.8085
25.730	3.4595	14.9	(-1-1 4)	12.865	0.1445	1.8162
25.730	3.4595	14.9	(-2 2 1)	12.865	0.1445	1.8162
25.913	3.4356	3.2	(-2 2 2)	12.956	0.1455	1.8288
25.979	3.4270	3.1	(0 2 3)	12.989	0.1459	1.8334
26.160	3.4037	7.6	(-2-2 2)	13.080	0.1469	1.8460
26.478	3.3635	29.7	(-1 1 4)	13.239	0.1487	1.8680
26.478	3.3635	29.7	(-2-2 1)	13.239	0.1487	1.8680
27.325	3.2611	57.2	(1 3 1)	13.663	0.1533	1.9267
27.325	3.2611	57.2	(-2 2 0)	13.663	0.1533	1.9267
27.771	3.2098	54.8	(0 4 0)	13.886	0.1558	1.9575
27.771	3.2098	54.8	(-2-2 3)	13.886	0.1558	1.9575
27.895	3.1958	100.0	(-2 0 4)	13.948	0.1565	1.9661
27.997	3.1844	71.6	(-2 2 3)	13.998	0.1570	1.9731
28.042	3.1794	92.4	(2 0 1)	14.021	0.1573	1.9762
28.042	3.1794	92.4	(0 0 4)	14.021	0.1573	1.9762
28.570	3.1219	44.4	(-2-1 4)	14.285	0.1602	2.0126
28.570	3.1219	44.4	(2 2 0)	14.285	0.1602	2.0126
29.152	3.0608	2.0	(0 4 1)	14.576	0.1634	2.0528
29.349	3.0407	20.2	(0 1 4)	14.674	0.1644	2.0663
29.349	3.0407	20.2	(1-3 2)	14.674	0.1644	2.0663
29.781	2.9976	0.9	(-1 4 0)	14.890	0.1668	2.0961
29.781	2.9976	0.9	(-1 4 1)	14.890	0.1668	2.0961
29.913	2.9847	1.0	(-2 3 1)	14.956	0.1675	2.1051
29.913	2.9847	1.0	(1 1 3)	14.956	0.1675	2.1051
30.260	2.9512	30.0	(-2 3 2)	15.130	0.1694	2.1290
30.260	2.9512	30.0	(0-4 2)	15.130	0.1694	2.1290
30.447	2.9335	20.0	(0-2 4)	15.224	0.1704	2.1419
30.884	2.8930	7.4	(-2-3 1)	15.442	0.1728	2.1719
30.884	2.8930	7.4	(-2-2 4)	15.442	0.1728	2.1719
31.615	2.8277	24.6	(1 3 2)	15.808	0.1768	2.2220
31.615	2.8277	24.6	(-2 2 4)	15.808	0.1768	2.2220
31.758	2.8153	15.4	(-1 0 5)	15.879	0.1776	2.2318
31.898	2.8033	10.0	(-1-3 4)	15.949	0.1784	2.2413



32.161	2.7810	2.1	(-2 3 3)	16.080	0.1798	2.2593
32.161	2.7810	2.1	(2 2 1)	16.080	0.1798	2.2593
32.856	2.7238	1.5	(-3 0 2)	16.428	0.1836	2.3068
32.856	2.7238	1.5	(-2 0 5)	16.428	0.1836	2.3068
32.947	2.7164	1.1	(-1 1 5)	16.474	0.1841	2.3131
33.046	2.7085	0.6	(2 0 2)	16.523	0.1846	2.3198
33.046	2.7085	0.6	(-1-4 3)	16.523	0.1846	2.3198
33.335	2.6857	0.9	(1 4 1)	16.667	0.1862	2.3395
33.335	2.6857	0.9	(-2-1 5)	16.667	0.1862	2.3395
33.459	2.6760	1.9	(-3 1 2)	16.730	0.1868	2.3480
33.719	2.6560	15.2	(0-4 3)	16.859	0.1883	2.3657
33.719	2.6560	15.2	(-1 3 4)	16.859	0.1883	2.3657
33.820	2.6482	10.3	(0-3 4)	16.910	0.1888	2.3726
33.820	2.6482	10.3	(-3-1 2)	16.910	0.1888	2.3726
34.215	2.6186	0.8	(2 1 2)	17.107	0.1909	2.3994
34.215	2.6186	0.8	(1-3 3)	17.107	0.1909	2.3994
34.532	2.5952	0.4	(-2-3 4)	17.266	0.1927	2.4210
34.532	2.5952	0.4	(-3 1 1)	17.266	0.1927	2.4210
34.626	2.5884	0.4	(-1 4 3)	17.313	0.1932	2.4274
35.048	2.5582	5.7	(2-2 2)	17.524	0.1954	2.4561
35.048	2.5582	5.7	(-3-1 1)	17.524	0.1954	2.4561
35.263	2.5431	3.2	(-2-2 5)	17.631	0.1966	2.4707
35.263	2.5431	3.2	(1 0 4)	17.631	0.1966	2.4707
35.510	2.5260	23.5	(1-1 4)	17.755	0.1979	2.4874
35.510	2.5260	23.5	(-2 4 2)	17.755	0.1979	2.4874
35.745	2.5099	7.3	(-3-1 4)	17.873	0.1992	2.5034
35.881	2.5007	32.2	(-2-4 2)	17.941	0.1999	2.5126
35.881	2.5007	32.2	(-3 1 4)	17.941	0.1999	2.5126
35.994	2.4931	18.4	(-3 2 3)	17.997	0.2006	2.5202
36.185	2.4804	2.0	(-2 4 0)	18.093	0.2016	2.5331
36.185	2.4804	2.0	(-3-2 2)	18.093	0.2016	2.5331
36.295	2.4731	2.3	(-2 2 5)	18.148	0.2022	2.5406
36.295	2.4731	2.3	(-3-2 3)	18.148	0.2022	2.5406
36.446	2.4632	1.4	(-1 5 0)	18.223	0.2030	2.5508
36.446	2.4632	1.4	(0 1 5)	18.223	0.2030	2.5508
36.536	2.4574	1.3	(1 1 4)	18.268	0.2035	2.5569
36.536	2.4574	1.3	(-1 5 1)	18.268	0.2035	2.5569
36.850	2.4371	5.0	(2 2 2)	18.425	0.2052	2.5781
36.850	2.4371	5.0	(-1-5 1)	18.425	0.2052	2.5781
36.976	2.4292	5.4	(0-2 5)	18.488	0.2058	2.5866
36.976	2.4292	5.4	(-3 1 0)	18.488	0.2058	2.5866
37.136	2.4190	2.7	(1 4 2)	18.568	0.2067	2.5974
37.136	2.4190	2.7	(-1-3 5)	18.568	0.2067	2.5974
37.348	2.4058	3.6	(-1-5 2)	18.674	0.2078	2.6117
37.348	2.4058	3.6	(-2 4 3)	18.674	0.2078	2.6117

37.441	2.4000	2.3	(-3-2 1)	18.720	0.2083	2.6180
37.697	2.3843	4.5	(3 1 0)	18.848	0.2097	2.6352
37.697	2.3843	4.5	(1-5 1)	18.848	0.2097	2.6352
37.782	2.3792	2.7	(-3-2 4)	18.891	0.2102	2.6409
38.096	2.3603	5.0	(-1 5 2)	19.048	0.2118	2.6621
38.096	2.3603	5.0	(2 4 0)	19.048	0.2118	2.6621
38.199	2.3541	3.0	(-3 0 5)	19.100	0.2124	2.6690
38.723	2.3235	8.1	(-3-1 5)	19.362	0.2152	2.7042
38.723	2.3235	8.1	(-3 3 2)	19.362	0.2152	2.7042
38.881	2.3144	5.2	(-1 4 4)	19.441	0.2160	2.7149
38.881	2.3144	5.2	(-1-1 6)	19.441	0.2160	2.7149
38.975	2.3090	2.8	(0 2 5)	19.488	0.2165	2.7211
38.975	2.3090	2.8	(-3 1 5)	19.488	0.2165	2.7211
39.061	2.3041	1.5	(1 2 4)	19.531	0.2170	2.7269
39.232	2.2945	3.0	(-1-5 3)	19.616	0.2179	2.7384
39.232	2.2945	3.0	(-2-4 4)	19.616	0.2179	2.7384
39.305	2.2904	2.0	(-1 3 5)	19.652	0.2183	2.7433
39.305	2.2904	2.0	(-3 3 3)	19.652	0.2183	2.7433
39.476	2.2809	1.1	(-3 3 1)	19.738	0.2192	2.7547
39.673	2.2700	5.3	(-3-3 2)	19.836	0.2203	2.7679
39.673	2.2700	5.3	(-3-3 3)	19.836	0.2203	2.7679
39.775	2.2644	6.5	(1 5 1)	19.888	0.2208	2.7748
39.775	2.2644	6.5	(-1 1 6)	19.888	0.2208	2.7748
39.866	2.2595	3.8	(1-3 4)	19.933	0.2213	2.7808
39.866	2.2595	3.8	(0-3 5)	19.933	0.2213	2.7808
40.257	2.2384	4.1	(1-5 2)	20.129	0.2234	2.8070
40.339	2.2340	4.6	(-1-2 6)	20.170	0.2238	2.8125
40.339	2.2340	4.6	(-2 4 4)	20.170	0.2238	2.8125
40.608	2.2199	0.9	(3-1 1)	20.304	0.2252	2.8304
40.608	2.2199	0.9	(2 3 2)	20.304	0.2252	2.8304
40.957	2.2018	1.4	(-3-3 4)	20.478	0.2271	2.8537
40.957	2.2018	1.4	(-3-3 1)	20.478	0.2271	2.8537
41.173	2.1907	1.9	(2 4 1)	20.587	0.2282	2.8681
41.173	2.1907	1.9	(-3 3 4)	20.587	0.2282	2.8681
41.336	2.1824	1.6	(0 4 4)	20.668	0.2291	2.8790
41.336	2.1824	1.6	(-1-4 5)	20.668	0.2291	2.8790
41.502	2.1741	2.2	(-3 3 0)	20.751	0.2300	2.8900
41.502	2.1741	2.2	(3 1 1)	20.751	0.2300	2.8900
41.801	2.1592	3.0	(-2 2 6)	20.900	0.2316	2.9099
41.801	2.1592	3.0	(-2-5 2)	20.900	0.2316	2.9099
42.140	2.1427	16.6	(2-4 2)	21.070	0.2334	2.9324
42.140	2.1427	16.6	(0 6 0)	21.070	0.2334	2.9324
42.423	2.1290	3.0	(1-1 5)	21.212	0.2349	2.9513
42.423	2.1290	3.0	(0 5 3)	21.212	0.2349	2.9513
42.660	2.1177	3.6	(1 3 4)	21.330	0.2361	2.9670

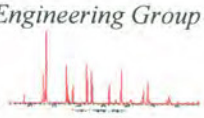


42.660	2.1177	3.6	(-2-4 5)	21.330	0.2361	2.9670
42.797	2.1112	2.1	(-3-1 6)	21.399	0.2368	2.9761
43.138	2.0954	11.1	(1 5 2)	21.569	0.2386	2.9986
43.138	2.0954	11.1	(-3 1 6)	21.569	0.2386	2.9986
43.296	2.0881	6.6	(-2-3 6)	21.648	0.2395	3.0091
43.445	2.0812	1.4	(-1 6 0)	21.723	0.2402	3.0190
43.445	2.0812	1.4	(3 3 0)	21.723	0.2402	3.0190
43.518	2.0779	1.2	(-3 4 1)	21.759	0.2406	3.0238
43.518	2.0779	1.2	(-3-3 5)	21.759	0.2406	3.0238
43.622	2.0732	1.0	(0-6 2)	21.811	0.2412	3.0306
43.622	2.0732	1.0	(0-5 4)	21.811	0.2412	3.0306
43.772	2.0665	2.4	(0 1 6)	21.886	0.2420	3.0406
43.772	2.0665	2.4	(0-4 5)	21.886	0.2420	3.0406
43.903	2.0606	2.2	(2-5 1)	21.952	0.2427	3.0492
43.903	2.0606	2.2	(1-5 3)	21.952	0.2427	3.0492
44.075	2.0529	0.9	(2 5 0)	22.038	0.2436	3.0606
44.075	2.0529	0.9	(-3-4 3)	22.038	0.2436	3.0606
44.194	2.0477	0.9	(-3 3 5)	22.097	0.2442	3.0684
44.194	2.0477	0.9	(-1-6 2)	22.097	0.2442	3.0684
44.269	2.0444	0.7	(-4 0 3)	22.135	0.2446	3.0734
44.419	2.0378	0.6	(1-6 1)	22.210	0.2454	3.0833
44.419	2.0378	0.6	(-2 4 5)	22.210	0.2454	3.0833
44.696	2.0258	2.8	(1 6 0)	22.348	0.2468	3.1015
44.696	2.0258	2.8	(-1 5 4)	22.348	0.2468	3.1015
44.826	2.0203	4.1	(-4 0 4)	22.413	0.2475	3.1101
44.910	2.0167	5.5	(-2 0 7)	22.455	0.2479	3.1156
44.910	2.0167	5.5	(-4 0 2)	22.455	0.2479	3.1156
45.090	2.0091	3.1	(-2-1 7)	22.545	0.2489	3.1274
45.090	2.0091	3.1	(-4-1 3)	22.545	0.2489	3.1274

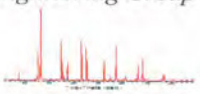
6) PDF#01-071-1403(RDB): Aegerine, calcian, aluminian (Sodium Calcium Iron Aluminum Silicate), Fe0.5185 Al0.4815 Ca0.466 Na0.534 Si2 O6

Radiation=CuKá1 Lambda=1.5406 Filter=
Ref: Curtis, L., Gittins, J., Kocman, V., Rucklidge, J.C., Hawthorne, F.C., Ferguson, R.B. Can. Mineral., v13 p62 (1975)
Monoclinic - Single Crystal, P2/n (13) Z=4
CELL: 9.622 x 8.826 x 5.279 <90.0 x 106.92 x 90.0> P.S=mP40.00
Density(c)=3.485
Strong Lines: 2.98/X 2.53/4 2.53/4 2.90/4 2.48/4 2.48/4 2.55/3 2.55/3

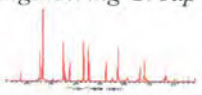
2-Theta	d(Å)	I(f)	(h k l)	Theta	1/(2d)	2pi/d	n^2
10.014	8.8260	0.7	(0 1 0)	5.007	0.0567	0.7119	
13.889	6.3709	15.4	(1 1 0)	6.945	0.0785	0.9862	
17.383	5.0975	0.1	(-1 0 1)	8.691	0.0981	1.2326	



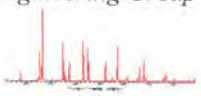
19.268	4.6027	1.7	(2 0 0)	9.634	0.1086	1.3651
20.105	4.4130	18.5	(0 2 0)	10.053	0.1133	1.4238
20.105	4.4130	18.5	(-1 1 1)	10.053	0.1133	1.4238
21.759	4.0811	0.9	(2 1 0)	10.880	0.1225	1.5396
22.323	3.9794	0.3	(1 2 0)	11.161	0.1256	1.5789
22.323	3.9794	0.3	(1 0 1)	11.161	0.1256	1.5789
24.225	3.6710	0.8	(-2 1 1)	12.113	0.1362	1.7116
24.580	3.6188	1.1	(1 1 1)	12.290	0.1382	1.7363
26.806	3.3231	3.0	(0 2 1)	13.403	0.1505	1.8907
27.988	3.1854	11.3	(2 2 0)	13.994	0.1570	1.9725
29.976	2.9786	100.0	(-2 2 1)	14.988	0.1679	2.1095
30.826	2.8983	39.3	(3 1 0)	15.413	0.1725	2.1679
31.043	2.8785	17.7	(-3 1 1)	15.522	0.1737	2.1828
31.514	2.8366	0.8	(2 1 1)	15.757	0.1763	2.2151
31.909	2.8024	0.2	(1 3 0)	15.954	0.1784	2.2421
35.192	2.5481	25.1	(-2 0 2)	17.596	0.1962	2.4659
35.192	2.5481	25.1	(-1 3 1)	17.596	0.1962	2.4659
35.277	2.5421	15.9	(0 3 1)	17.639	0.1967	2.4716
35.521	2.5252	40.4	(-1 1 2)	17.760	0.1980	2.4882
35.521	2.5252	40.4	(0 0 2)	17.760	0.1980	2.4882
35.607	2.5193	23.9	(3 2 0)	17.804	0.1985	2.4940
36.215	2.4784	37.6	(2 2 1)	18.107	0.2017	2.5351
36.215	2.4784	37.6	(2 3 0)	18.107	0.2017	2.5351
36.671	2.4487	0.8	(-2 1 2)	18.335	0.2042	2.5660
36.997	2.4278	0.6	(0 1 2)	18.498	0.2059	2.5880
37.808	2.3776	0.1	(-2 3 1)	18.904	0.2103	2.6427
38.047	2.3632	0.2	(1 3 1)	19.023	0.2116	2.6588
38.476	2.3378	0.1	(3 0 1)	19.238	0.2139	2.6876
39.110	2.3014	0.7	(4 0 0)	19.555	0.2173	2.7302
39.321	2.2895	0.5	(-4 1 1)	19.661	0.2184	2.7443
39.858	2.2599	8.1	(3 1 1)	19.929	0.2213	2.7803
40.379	2.2319	0.4	(-3 1 2)	20.189	0.2240	2.8151
40.474	2.2269	0.7	(4 1 0)	20.237	0.2245	2.8215
40.979	2.2006	13.7	(1 1 2)	20.489	0.2272	2.8552
41.152	2.1918	8.6	(0 2 2)	20.576	0.2281	2.8667
42.077	2.1457	0.9	(1 4 0)	21.038	0.2330	2.9282
42.536	2.1236	4.9	(3 3 0)	21.268	0.2354	2.9587
42.701	2.1158	17.1	(-3 3 1)	21.350	0.2363	2.9697
43.289	2.0884	8.5	(-4 2 1)	21.644	0.2394	3.0086
43.786	2.0658	0.5	(3 2 1)	21.893	0.2420	3.0415
44.357	2.0406	0.5	(4 2 0)	22.178	0.2450	3.0791
44.787	2.0220	12.4	(0 4 1)	22.394	0.2473	3.1075
44.787	2.0220	12.4	(1 2 2)	22.394	0.2473	3.1075
44.871	2.0184	14.0	(-4 0 2)	22.435	0.2477	3.1130
45.697	1.9838	5.2	(2 0 2)	22.848	0.2520	3.1672



46.171	1.9645	2.6	(-4 1 2)	23.086	0.2545	3.1983
46.171	1.9645	2.6	(-1 3 2)	23.086	0.2545	3.1983
46.888	1.9361	3.2	(2 1 2)	23.444	0.2582	3.2452
46.888	1.9361	3.2	(-2 4 1)	23.444	0.2582	3.2452
47.088	1.9284	1.8	(1 4 1)	23.544	0.2593	3.2583
47.406	1.9162	0.4	(-5 0 1)	23.703	0.2609	3.2790
47.406	1.9162	0.4	(0 3 2)	23.703	0.2609	3.2790
48.515	1.8750	1.6	(-5 1 1)	24.257	0.2667	3.3511
49.099	1.8540	0.1	(4 1 1)	24.550	0.2697	3.3890
49.627	1.8355	1.4	(-4 2 2)	24.813	0.2724	3.4231
49.777	1.8303	2.9	(3 3 1)	24.889	0.2732	3.4329
50.215	1.8154	0.7	(-3 3 2)	25.108	0.2754	3.4611
50.393	1.8094	1.5	(2 2 2)	25.196	0.2763	3.4725
50.605	1.8023	3.0	(5 1 0)	25.302	0.2774	3.4862
50.722	1.7984	3.1	(1 3 2)	25.361	0.2780	3.4938
51.078	1.7867	0.2	(-3 4 1)	25.539	0.2798	3.5166
51.391	1.7766	1.1	(2 4 1)	25.696	0.2814	3.5367
51.746	1.7652	0.3	(0 5 0)	25.873	0.2833	3.5595
51.922	1.7596	0.3	(-5 2 1)	25.961	0.2842	3.5708
52.479	1.7423	1.6	(4 2 1)	26.240	0.2870	3.6064
52.761	1.7336	9.9	(1 5 0)	26.380	0.2884	3.6243
53.189	1.7207	0.5	(-2 1 3)	26.595	0.2906	3.6516
53.334	1.7163	0.7	(-5 1 2)	26.667	0.2913	3.6608
53.334	1.7163	0.7	(-1 1 3)	26.667	0.2913	3.6608
53.917	1.6992	0.3	(5 2 0)	26.958	0.2943	3.6978
53.917	1.6992	0.3	(-3 0 3)	26.958	0.2943	3.6978
54.136	1.6928	0.3	(-1 4 2)	27.068	0.2954	3.7117
54.304	1.6880	2.0	(3 1 2)	27.152	0.2962	3.7224
54.989	1.6685	3.5	(-1 5 1)	27.495	0.2997	3.7657
54.989	1.6685	3.5	(-3 1 3)	27.495	0.2997	3.7657
55.139	1.6643	2.5	(-4 3 2)	27.569	0.3004	3.7752
55.239	1.6616	4.9	(0 4 2)	27.619	0.3009	3.7815
55.851	1.6448	0.2	(2 3 2)	27.925	0.3040	3.8200
56.394	1.6302	13.3	(-2 2 3)	28.197	0.3067	3.8542
56.570	1.6256	7.3	(-5 2 2)	28.285	0.3076	3.8652
56.570	1.6256	7.3	(-1 2 3)	28.285	0.3076	3.8652
57.278	1.6072	13.1	(-5 3 1)	28.639	0.3111	3.9095
57.467	1.6023	7.1	(3 2 2)	28.733	0.3120	3.9213
57.847	1.5927	5.6	(4 4 0)	28.923	0.3139	3.9450
57.847	1.5927	5.6	(4 3 1)	28.923	0.3139	3.9450
58.056	1.5875	3.0	(5 0 1)	29.028	0.3150	3.9580
58.235	1.5830	0.4	(1 4 2)	29.117	0.3159	3.9691
58.450	1.5777	0.2	(1 0 3)	29.225	0.3169	3.9825
58.450	1.5777	0.2	(-6 1 1)	29.225	0.3169	3.9825
58.644	1.5729	0.4	(-4 1 3)	29.322	0.3179	3.9946



58.644	1.5729	0.4	(0 2 3)	29.322	0.3179	3.9946
59.079	1.5624	0.6	(5 1 1)	29.540	0.3200	4.0215
59.150	1.5607	0.6	(5 3 0)	29.575	0.3204	4.0259
59.504	1.5522	0.3	(1 1 3)	29.752	0.3221	4.0478
60.274	1.5343	3.2	(6 0 0)	30.137	0.3259	4.0953
60.455	1.5301	6.1	(3 5 0)	30.227	0.3268	4.1064
60.583	1.5272	2.9	(-3 5 1)	30.292	0.3274	4.1143
60.788	1.5225	4.1	(-6 0 2)	30.394	0.3284	4.1269
60.788	1.5225	4.1	(2 5 1)	30.394	0.3284	4.1269
61.475	1.5071	2.6	(-6 2 1)	30.738	0.3318	4.1690
61.475	1.5071	2.6	(-2 3 3)	30.738	0.3318	4.1690
61.657	1.5031	12.0	(-1 3 3)	30.829	0.3326	4.1802
61.657	1.5031	12.0	(-4 2 3)	30.829	0.3326	4.1802
61.783	1.5003	7.0	(-6 1 2)	30.891	0.3333	4.1878
61.902	1.4977	6.4	(4 0 2)	30.951	0.3338	4.1951
62.086	1.4938	2.8	(5 2 1)	31.043	0.3347	4.2063
62.293	1.4893	0.6	(-4 4 2)	31.146	0.3357	4.2189
62.508	1.4847	0.4	(1 2 3)	31.254	0.3368	4.2321
62.508	1.4847	0.4	(3 3 2)	31.254	0.3368	4.2321
62.953	1.4752	0.6	(-5 0 3)	31.477	0.3389	4.2591
62.953	1.4752	0.6	(2 4 2)	31.477	0.3389	4.2591
63.155	1.4710	4.8	(0 6 0)	31.578	0.3399	4.2714
63.155	1.4710	4.8	(-3 3 3)	31.578	0.3399	4.2714
63.336	1.4672	2.8	(-1 5 2)	31.668	0.3408	4.2823
63.972	1.4542	1.0	(-5 1 3)	31.986	0.3438	4.3208
63.972	1.4542	1.0	(1 6 0)	31.986	0.3438	4.3208
64.220	1.4492	0.6	(-2 5 2)	32.110	0.3450	4.3357
64.220	1.4492	0.6	(6 2 0)	32.110	0.3450	4.3357
64.339	1.4468	0.4	(0 5 2)	32.169	0.3456	4.3429
64.339	1.4468	0.4	(-5 4 1)	32.169	0.3456	4.3429
64.716	1.4393	1.0	(4 4 1)	32.358	0.3474	4.3656
64.716	1.4393	1.0	(-6 2 2)	32.358	0.3474	4.3656
65.792	1.4183	1.7	(4 2 2)	32.896	0.3525	4.4301
65.921	1.4158	1.0	(-4 5 1)	32.961	0.3532	4.4378
65.921	1.4158	1.0	(-1 6 1)	32.961	0.3532	4.4378
66.106	1.4123	0.6	(0 6 1)	33.053	0.3540	4.4489
66.296	1.4087	0.8	(-6 3 1)	33.148	0.3549	4.4602
66.296	1.4087	0.8	(3 5 1)	33.148	0.3549	4.4602
66.662	1.4019	8.6	(2 6 0)	33.331	0.3567	4.4820
66.662	1.4019	8.6	(-3 5 2)	33.331	0.3567	4.4820
66.922	1.3971	13.9	(5 3 1)	33.461	0.3579	4.4974
66.922	1.3971	13.9	(-5 2 3)	33.461	0.3579	4.4974
67.087	1.3940	8.6	(1 5 2)	33.544	0.3587	4.5072
67.319	1.3898	1.9	(1 3 3)	33.660	0.3598	4.5210
67.744	1.3821	0.4	(-2 6 1)	33.872	0.3618	4.5461



67.964	1.3782	4.2	(2 2 3)	33.982	0.3628	4.5591
67.964	1.3782	4.2	(1 6 1)	33.982	0.3628	4.5591
68.205	1.3739	2.6	(-7 0 1)	34.103	0.3639	4.5733
68.243	1.3732	2.3	(-2 4 3)	34.122	0.3641	4.5756
68.401	1.3704	1.0	(-1 4 3)	34.201	0.3649	4.5849
68.401	1.3704	1.0	(-5 4 2)	34.201	0.3649	4.5849
68.977	1.3604	0.1	(6 3 0)	34.488	0.3675	4.6187
69.142	1.3575	0.2	(-7 1 1)	34.571	0.3683	4.6284
69.142	1.3575	0.2	(3 4 2)	34.571	0.3683	4.6284
69.821	1.3460	0.1	(-3 4 3)	34.911	0.3715	4.6681
69.821	1.3460	0.1	(6 1 1)	34.911	0.3715	4.6681
70.273	1.3384	0.6	(0 4 3)	35.136	0.3736	4.6945
70.496	1.3347	0.3	(4 3 2)	35.248	0.3746	4.7075
70.771	1.3302	0.1	(-6 1 3)	35.386	0.3759	4.7235
71.308	1.3215	6.0	(2 6 1)	35.654	0.3784	4.7546
71.308	1.3215	6.0	(-7 1 2)	35.654	0.3784	4.7546
71.522	1.3181	6.0	(2 5 2)	35.761	0.3793	4.7669
71.522	1.3181	6.0	(-5 3 3)	35.761	0.3793	4.7669
72.164	1.3079	0.4	(3 1 3)	36.082	0.3823	4.8039
72.349	1.3050	0.4	(-2 1 4)	36.174	0.3831	4.8146
72.587	1.3013	3.7	(6 2 1)	36.294	0.3842	4.8282
72.587	1.3013	3.7	(7 1 0)	36.294	0.3842	4.8282
72.734	1.2991	2.2	(-5 5 1)	36.367	0.3849	4.8366
72.734	1.2991	2.2	(-6 4 1)	36.367	0.3849	4.8366
72.984	1.2952	4.1	(-4 4 3)	36.492	0.3860	4.8510
72.984	1.2952	4.1	(-3 1 4)	36.492	0.3860	4.8510
73.194	1.2920	2.1	(4 5 1)	36.597	0.3870	4.8630
73.393	1.2890	0.8	(-1 1 4)	36.697	0.3879	4.8744
73.393	1.2890	0.8	(5 4 1)	36.697	0.3879	4.8744
73.523	1.2871	0.4	(-6 2 3)	36.761	0.3885	4.8817
73.669	1.2849	0.4	(-1 6 2)	36.835	0.3891	4.8901
74.402	1.2740	2.7	(-2 6 2)	37.201	0.3925	4.9317
74.402	1.2740	2.7	(5 5 0)	37.201	0.3925	4.9317
74.605	1.2711	6.8	(0 6 2)	37.302	0.3934	4.9432
74.894	1.2669	3.0	(3 2 3)	37.447	0.3947	4.9596
75.190	1.2626	3.1	(0 0 4)	37.595	0.3960	4.9763
75.190	1.2626	3.1	(-4 1 4)	37.595	0.3960	4.9763
75.398	1.2597	1.7	(6 4 0)	37.699	0.3969	4.9880
75.398	1.2597	1.7	(7 2 0)	37.699	0.3969	4.9880
75.859	1.2531	0.4	(-6 4 2)	37.930	0.3990	5.0140
76.089	1.2499	0.4	(1 7 0)	38.045	0.4000	5.0269
76.089	1.2499	0.4	(-4 6 1)	38.045	0.4000	5.0269
76.457	1.2448	0.7	(-7 3 1)	38.229	0.4017	5.0475
76.457	1.2448	0.7	(-2 5 3)	38.229	0.4017	5.0475
76.613	1.2427	0.5	(-1 5 3)	38.307	0.4024	5.0562

76.613	1.2427	0.5	(-5 5 2)	38.307	0.4024	5.0562
76.866	1.2392	1.3	(4 6 0)	38.433	0.4035	5.0703
76.866	1.2392	1.3	(4 4 2)	38.433	0.4035	5.0703
77.110	1.2359	0.8	(6 3 1)	38.555	0.4046	5.0839
77.416	1.2318	5.3	(3 5 2)	38.708	0.4059	5.1009
78.003	1.2240	1.6	(-3 5 3)	39.002	0.4085	5.1334
78.003	1.2240	1.6	(-1 7 1)	39.002	0.4085	5.1334
78.545	1.2169	0.2	(-7 3 2)	39.272	0.4109	5.1634
78.545	1.2169	0.2	(2 7 0)	39.272	0.4109	5.1634
78.774	1.2139	0.3	(0 2 4)	39.387	0.4119	5.1760
78.939	1.2118	0.3	(2 4 3)	39.469	0.4126	5.1850
78.939	1.2118	0.3	(-7 1 3)	39.469	0.4126	5.1850
79.198	1.2085	0.2	(-5 1 4)	39.599	0.4137	5.1992
79.372	1.2063	0.1	(3 3 3)	39.686	0.4145	5.2088
79.589	1.2035	0.4	(-2 3 4)	39.794	0.4154	5.2207
79.589	1.2035	0.4	(-2 7 1)	39.794	0.4154	5.2207

7) PDF#01-071-3770, Silicon, syn, Si

Radiation=CuKá1 Lambda=1.5406

Ref: Jette, E.R., Foote, F. J. Chem. Phys., v1 p753 (1933)

Cubic - Fd-3m (227) Z=8

CELL: 5.43077 x 5.43077 x 5.43077 <90.0 x 90.0 x 90.0> P.S=cF8.00

Density(c)=2.329

Strong Lines: 3.14/X 1.92/6 1.64/3 1.11/1 1.25/1 1.36/1 1.05/1 0.92/1

2-Theta	d(Å)	I(f)	(h k l)	Theta	1/(2d)	2pi/d	n^2
28.443	3.1355	100.0	(1 1 1)	14.222	0.1595	2.0039	3
47.304	1.9201	55.2	(2 2 0)	23.652	0.2604	3.2724	8
56.124	1.6374	30.0	(3 1 1)	28.062	0.3054	3.8372	11
58.858	1.5677	0.1	(2 2 2)	29.429	0.3189	4.0078	12
69.132	1.3577	6.9	(4 0 0)	34.566	0.3683	4.6279	16
76.379	1.2459	9.6	(3 3 1)	38.189	0.4013	5.0431	19
88.033	1.1086	11.6	(4 2 2)	44.017	0.4510	5.6679	24
94.956	1.0452	6.1	(5 1 1)	47.478	0.4784	6.0118	27
106.713	0.9600	3.4	(4 4 0)	53.356	0.5208	6.5448	32
114.097	0.9180	5.9	(5 3 1)	57.048	0.5447	6.8447	35
127.551	0.8587	5.1	(6 2 0)	63.775	0.5823	7.3173	40
136.901	0.8282	2.4	(5 3 3)	68.450	0.6037	7.5867	43

Appendix 7
Supplemental XRD powder patterns

Figure 15 presents the XRD powder patterns for UDS core samples: TI040-419D-2X, TI040-420D-2X, TI040-421D-2X, and TI040-422D-2X along with the corresponding PDF cards. Figure 16 presents the XRD powder patterns for UDS container composites: TI040-792D-2X and TI040-793D-2X.

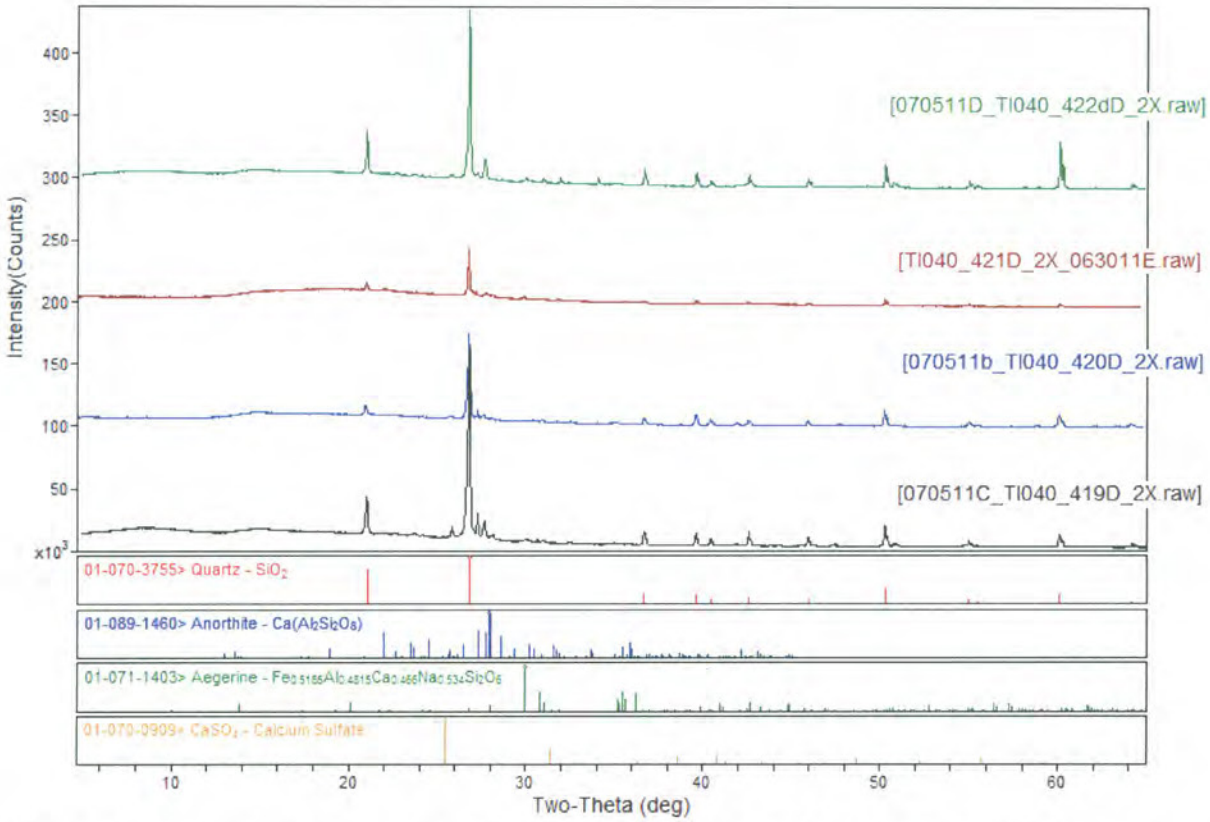


Figure 15. XRD powder patterns for TI040-419D-2X, TI040-420D-2X, TI040-421D-2X, and TI040-422D-2X along with the corresponding PDF cards.

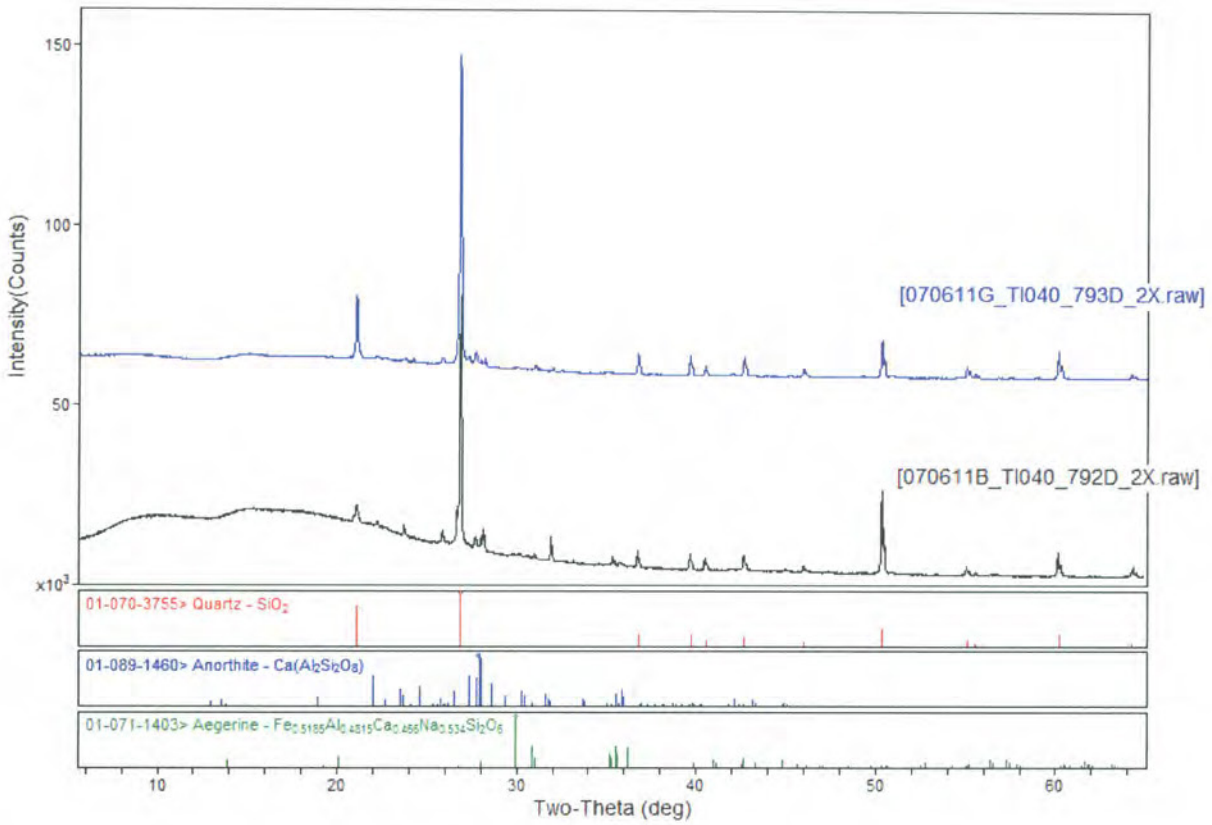


Figure 16. XRD powder patterns for TI040-792D-2X and TI040-793D-2X along with the corresponding PDF cards.

RPL K Basin Project 52578	Document Review Record

Title:	XRD Report for KW 210 Undissolved Solids

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Organization/Department:	**Designated Reviewer:**	**Reviewer Signature/Date:** (Upon completion of review)
D9H63	**Mike Urie**	7/21/11

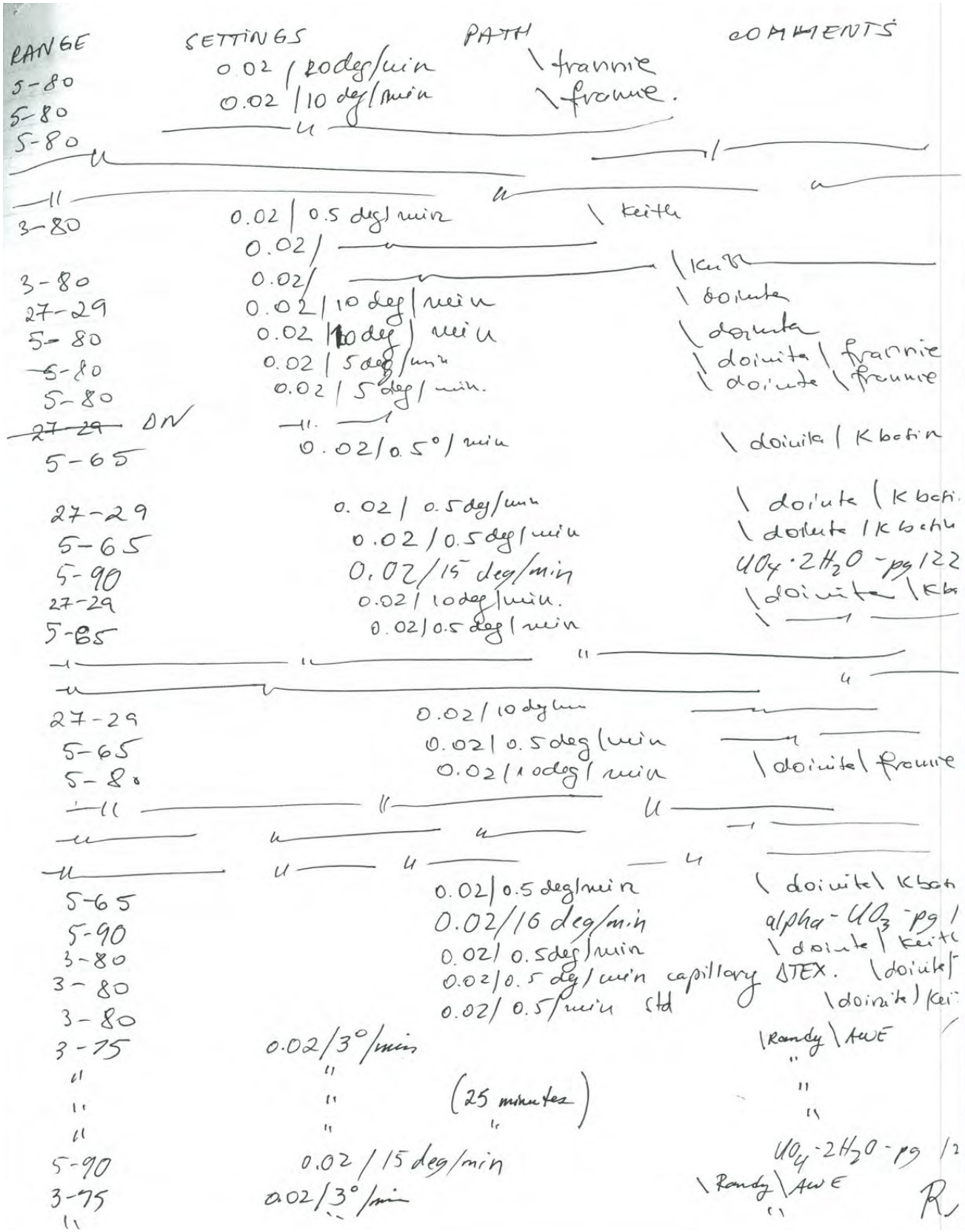
It is optional, but helpful when comments are identified as to type or if the reviewer wishes to specify mandatory comments requiring resolution. In this case, the following conventions apply:

***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	Figure 5	E	Change "show" to "shown" or "is shown"	DONE
2	Figures 3,6,7,8,9	E	Change "spectrum" to "spectra".	DONE

Concur with comment resolution, review complete.	Comments Resolved By:
7/21/11	7/21/11
Reviewer Signature Date	Author Signature Date

OPERATOR	DATE	TIME	SAMPLE ID	FILENAME
DN	6/21/11	3:15	062111e_boehmite 1	062111e
DN	—11	3:30	062111f_gibbsite paste	062111f.
DN	6/21/11	3:39	062111g_bohmite 2	062111g
DN	6/21/11	3:45	062111h_bohmite 3	062111h
DN	—11—	3:55	062111i_bohmite 4	i
DN	—11—	5:00	062111j_hydroxysodalite_2	j
DN	6/22/11	5:00	062211a_C202_~~DI~~ DN ~~Ca(OH)2~~	
DN	6/23/11	10:15	062311a_C203_DI_Ca(OH)2	
DN	6/30/11	2:48	063011a_Si_640a	
DN	6/30/11	3:00	063011b_1976_Alewine plate	
DN	6/30/11	3:10	063011C_Sodalite 3	
DN	6/30/11	3:25	063011d_Sodalite 4	
~~DN~~	~~7/1/11~~	~~10:00~~	~~070111a_Si_640a~~ DN	
~~DN~~	7/1 "			
DN	6/30/11	4:00	063011E_T1040-421D-2x	
DN	7/1/11	10:00	070111a_Si_640a	
DN	7/1/11	10:20	070111b_T1040-420D-2x-T	
LS	7/1/11	2:00	070111c-UO_4-$2H_2O$	
DN	7/5/11	12:20	070511a_Si640a	
DN	7/5/11	12:30	070511b_T1040_420D-2x	
DN	7/5/11	2:45	070511C_T1040-419D-2x	
DN	7/5/11	5:00	070511D_T1040-422D-2x	
DN	7/6/11	1:10	070611A_Si640a	
DN	7/6/11	1:20	070611B_T1040-~~7~~920-2x	
DN	7/6/11	3:40	sodalite 5_070611C	
DN	7/6/11	3:48	sodalite 6_070611D	
DN	7/6/11	4:00	Kaolin_070611E	
DN	7/6/11	4:10	Al-silicate_070611F	
DN	7/6/11	4:30	070611G_T1040_793D_2x	
LS	7/7/11	3:44	070711a-~~UO3~~ alpha-UO_3	
DN	7/11/11	11:00	C202-Ca(OH)2_071111a	
DN	7/12/11	9:30	DADB1_071211a	
DN	7/12/11	11:50	C203_Ca(OH)2_071211b	
AK	7/12/11	4:41	070~~7~~11c AWE Std	
AK	7/13/11	10:46	071311a AWE Std	
AK	7/13/11	11:20	071311b AWE III Pu239-L-1	
AK	7/13/11	12:01	071311c AWE III Pu239-L-2	
LS	7/13/11	3:40 PM	071311-UO_4-$2H_2O$	
AK	7/14/11	10:30	071411a AWE Std	
AK	7/14/11	11:20	071411b AWE III Pu238-L-1	


RANGE
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COMMENTS
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\ frannie
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0.02 / 10 deg/min
\ frannie.
5-80
3-80
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UO4 · 2H2O - pg 122
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\ Randy \ AWE
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(25 minutes)
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3-75
0.02/15 deg/min
0.02/3°/min
UO4 · 2H2O - pg /2
\ Randy \ AWE
R

PROJECT 52578 – SURVEILLANCE REPORT

Number: SR-52578-2011-034

PLANNING

Topic/Issue to be Evaluated: Surveillance of K Basin X-Ray Diffraction Analysis Report; KW 210 Undissolved Solids

This surveillance was undertaken to evaluate the draft *X-Ray Diffraction (XRD) Analysis Report, KW 210 Undissolved Solids* for the samples listed in Table 1 collected during the implementation of test instruction, TI040, Processing of the Undissolved Solids Samples from the K West Basin Core Samples Collected from SCS-CON-210 for Additional Analysis.

PERFORMANCE

Date Initiated: 16 June 2011	**Location:** RPL
Date Received: 25 July 2011	**Date Reviewed:** 25 July 2011
Date Resubmitted: 27 July 2011	**Date Closed:** 27 July 2011
Date Completed: 27 July 2011	**DRAFT Report Issued:** 25 July 2011 **FINAL Report Issued**: 27 July 2011
Contacts:, Clark Carlson, Sandy Fiskum, Doinita Neiner, Mike Urie	**Org Code:** PE137, D9H63
Surveillance Team: Lisa Middleton	

PRELIMINARY INFORMATION

Source of Requirement(s):

- KBC-33786, Rev. 2, December 2009; Quality Assurance Project Plan/Sampling and Analysis Plan (QAPjP/SAP): Quality Assurance Project Plan/Sampling and Analysis Plan for Containerized KW Settler Sludge

Other Applicable Documents:

- RPL-OP-001, Rev. 9, *Routine Research Operations*
- PNNL-RPG-268, Rev. 4, *Solids Analysis by X-Ray Diffraction*
- 52578-ADM-001, Rev. 0, *K Basin Requests for Analysis and Guidelines for Data Reporting and Technical, QE, and Spreadsheet Reviews.*

PROJECT 52578 – SURVEILLANCE REPORT

Number: SR-52578-2011-034

Purpose/Scope:

The 52578 Project QE staff are required via the QA Program to perform a review of K Basin reports generated within the Radiochemical Processing Laboratory (RPL) to determine traceability and accuracy of data contributing to the completed report.

The draft report was reviewed to evaluate that the data are what they purport to be, that is, that the reported results reflect what was actually done and the results meet applicable requirements. When deficiencies in the data are identified, then those deficiencies need to be documented as a K Basin Occurrence Report for the data user's review and, when applicable, resolved by corrective action. The XRD analysis under this Analysis Request was performed to identify crystalline structures in room-temperature-dried sludge, to identify hydrate compounds plus any other identifiable compounds. This includes noting the presence (or absence) of aluminum hydrates, uranium hydrates, uranium oxides as well as zirconium metal.

When issues were found, the cognizant staff were contacted to resolve issues prior to completion of the review.

Approach:

The draft report initiated by a K Basin Analysis Request form dated 6/16/11 was provided as the basis for the data quality review. Six (6) samples were submitted on 6/16/11 for analysis as noted in Table 1:

Table 1: XRD Samples IDs & Descriptions

Client Sample ID	Sample Description	XRD Sample ID	Internal Standard	Date Analyzed	Daily Performance Check w/ NIST SRM 640a?
TI040-419D-2X	KW210-A1 undissolved solids (UDS)	TI040-419D-2X	None	7/5/11	Y 0705011a_Si640a
TI040-420D-2X	KW210-A4 UDS	TI040-420D-2X	None	7/5/11	Y 0705011a_Si640a
		TI040-420D-2X-TiO2	Rutile	7/1/11	Y 0701011a_Si_640a
TI040-421D-2X	KW210-B1 UDS	TI040-421D-2X	None	6/30/11	Y 063011a_Si_640a
TI040-422D-2X	KW210-B3 UDS	TI040-422D-2X	None	7/5/11	Y 0705011a_Si640a
TI040-792D-2X	KW210-CC, -2000/+500 µm UDS	TI040-792D-2X	None	7/6/11	Y 0706011a_Si640a
TI040-793D-2X	KW210-CC, -500 µm UDS	TI040-793D-2X	None	7/6/11	Y 0706011a_Si640a

PROJECT 52578 – SURVEILLANCE REPORT

Number: SR-52578-2011-034

Requirements:

50-100% completeness is specified in KBC-33786, Rev. 2 with the specification that if significant gamma radioactivity is detected in the residue, then 100% completeness is required; otherwise 50% completeness is required. Data quality objectives for MDL, precision and accuracy were not specified in KBC-33786, Rev. 2 for XRD analysis.

Method requirements were that a semi-annual calibration using NIST SRM 1976 and a daily calibration using NIST SRM 640a were performed.

Semi-annual calibration using NIST SRM 1976, Corundum, was performed on 6/30/11; file name 063011b_1976_Aluminaplate. Daily calibrations were performed as noted above in Table 1. All samples were to be analyzed without internal standard, one sample (TI040-420D-2X) was to also be run with rutile as an internal standard, creating 7 samples total.

The report contained the following information:

1. Cover page
2. XRD Analysis Request Form/ XRD Mount Preparation Signed by Sandy Fiskum 6/16/11
3. XRD Analysis Bench Sheet – Sample Preparation (pp.2)
4. XRD Analysis Bench Sheet – Diffraction Measurements (pp.2)
5. Narrative Report and data tables
    a. Table 1 – Summary of Phases Identified in KW210 Undissolved Solids
    b. Sample preparation
    c. X-Ray powder diffraction data collection
    d. X-Ray powder diffraction data analysis
    e. Summary of findings
    f. Sample Analyses
    g. Appendix 1 – SRM 674 NIST Certificate
    h. Appendix 2 – Daily Performance Checks – 5/25/11
    i. Appendix 3 – SRM 640a NIST Certificate
    j. Appendix 4 – Quality Control/Calibration – 1/6/11
    k. Appendix 5 - -SRM 1976 NIST Certificate
    l. Appendix 6 – PDF cards for the identified crystalline phases presented in report
    m. Appendix 7 – Supplemental XRD Powder Patterns
    n. DRR form signed by the Technical Reviewer, Mike Uric 7/21/11
    o. Copy of XRD run log (6/21/11 – 7/14/11), 2 pages

Procedure:

PNNL-RPG-268, Rev. 4, *Solids Analysis by X-Ray Diffraction*

PROJECT 52578 – SURVEILLANCE REPORT

Number: SR-52578-2011-034

M&TE used:

Location	Description	Manufacturer	Model	Unique ID
RPL/409	X-Ray Diffractometer	Rigaku	Ultima IV	GD 3827N /WD59716
	RGD			#105
	SRM	NIST	640a	640a
	SRM	NIST	674	674
	SRM	NIST	1976	1976

The completed surveillance report will be posted to the SharePoint site and submitted as a project record. Any documentation associated with this surveillance will be available in the Lead Auditor's office, Building 325, 5-G.

RESULTS

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.

DQO Compliance:
100% completeness was obtained. Semi-annual and monthly calibrations were performed as required. Sample preparation and analysis was documented using a checklist and therefore not resulting in a recurrence of the deficiency noted in D59205-2-211.

Summary of Identified Concerns:

Note: All concerns identified in the review were addressed in a comment resolution meeting on 26 July 2011, Deborah Coffey, Lisa Middleton and Doinita Neiner attended to disposition review comments. There were no outstanding identified findings or observations.

1. Page 9, 2nd paragraph, 2nd sentence: The sentence should begin "Each sample was mixed…" instead of "A sample was mixed…" This clarifies that identical sample prep occurred for each sample. Sentence changed for clarification. No further action required, closed.

2. Page 10, Section 3.1: The acronym PDF needs to be defined in its first occurrence. The definition can then be removed from the second occurrence later in the paragraph. As a rule – when defining an acronym, the full name is given first, followed by the acronym in parenthesis, such as: Powder Diffraction File (PDF). Acronym correctly defined. No further action required, closed.

3. Page 10, Section 3.1: The acronym RDB-ICSD needs to be defined. Acronym removed, not pertinent to the report. No further action required, closed.

PROJECT 52578 – SURVEILLANCE REPORT

Number: SR-52578-2011-034

4. Page 15: The last sentence should read "The two duplicate samples confirm the diffraction patterns given the overall analysis method uncertainty." Not "The two duplicate samples reasonably reproduce the diffraction patterns given the overall experimental uncertainty." Sentence changed for clarification. No further action required, closed.

5. Page 16, Figure 5: The figure is too large for the page, exceeding the margin, so the right side of the figure is not visible. Figure resized to fit page. No further action required, closed.

6. Page 17, 1st sentence "samples" should be singular, "sample" since you are only talking about TI040-421D-2X. Plurality of "sample" changed. No further action required, closed.

7. Appendix 2, page 24: Where do the reference values in tables 4-7 come from? The value doesn't match values from either the standard certificate or the given PDF cards? The reference value now reflects the value given by the SRM. The PDF card for the SRM (01-071-3770) is now included in Appendix 6 as well. No further action required, closed.

CORRECTIVE ACTION

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

Surveillance Performed By:

Lisa Middleton, Quality Engineer lsc 7-27-11 Date: 7/27/11

DISTRIBUTION: Clark Carlson, Sandy Fiskum, Matt Fountain, Doinita Neiner, Andy Schmidt, Rick Shimskey, Mike Urie, Deborah Coffey, Lisa Middleton

Appendix G

Particle Density

Appendix G

Particle Density

Appendix G contains the particle density characterization report. Particle density was conducted on the size-fractionated samples collected from the Engineered Container SCS-CON-210 container composite. Any reference to an appendix ID *within* the particle density report refers to the self-contained appendices.

The following table of contents shows that pagination specific to Appendix G was applied to the analysis request form, particle density report (inclusive of self-contained appendices), pycnometer analysis data sheets document review record, and surveillance report. This pagination is sequential and consistent (e.g., page G.#).

Appendix G Table of Contents

Request for Particle Density Analysis G.3
Particle Density Report G.5
Pycnometer Analysis Data Sheets G.16
Document Review Record G.24
Surveillance of K Basin Particle Density Report SR-52578-2011-032 ..... G.25

Request for Particle Density Analysis by Gas Pycnometry

Requester	SK Fiskum	**Work Package**	H41224
Signature	SK Fisk	**Project Number**	52578
		Subtask	Particle Density
Date Submitted:	4/6/11	**Date Required:**	5/6/11

Sample ID	**Sample Description**
TI008-L ✓	+2000 µm, air-dried solids
TI008-MK	-2000/+500 µm, air-dried solids
TI008-ML	-2000/+500 µm, air-dried solids duplicate
TI008-SK ✓	-500 µm wet slurry
TI008-SL	-500 µm wet slurry duplicate

Special Instructions, notes:
All samples are from the KW210 container composite that had been wet-sieved into three size fractions. A large quantity of resin beads have been observed in the -2000/+500 µm fraction. The -500 µm sample fractions are wet slurries that will require air drying.

1. Air-dry the wet slurry subsamples at room temperature to constant mass.
2. Analyze the entire subsample provided if at all possible as a single sample (-2000/+500 µm and -500 µm sieve fractions).
3. Split sample TI008-L (+2000 µm sieve fraction) into like morphological pieces, photograph the splits, and conduct the density analysis.
4. Conduct density measurement on each subsample according to procedure, APEL-PIP-4, Rev. 2, *Gas Pycnometry Method for Apparent Specific Gravity Determinations of Consolidated Solids.*
5. The following QC parameters apply the sample and sample duplicate (not sample TI008-L splits):

Precision (RPD between duplicates)	Accuracy (Standard)
±5%	±1%

6. Generate a report in accordance to the requirements defined in 52578-Admin-001 K Basin *Requests for Analysis and Guidelines for Data Reporting and Technical, QE, and Spreadsheet Reviews*. Include the following elements in the summary report:
   a. Signed cover page
   b. Narrative describing the analysis, observations and results
   c. Net sample mass analyzed, sample volume, sample photograph, sample density and estimated uncertainty
   d. Calculated relative percent difference between sample and duplicate
   e. Calibration
   f. Results of standards analysis
   g. Summary of QC sample results and discussion of QC failures (if any)
   h. Completed analysis bench sheets

Special Instructions, notes, continued:

7. Submit report and data for technical review and obtain a Document Review Record (DRR) form; resolve comments, complete form showing resolution of comments and acceptance by Technical Reviewer. Have Technical Reviewer sign cover page
8. Submit report and data for QE review. Resolve draft surveillance report comments. Have QE Reviewer sign cover page

Particle Density Report

Battelle—Pacific Northwest National Laboratory
Radiochemical Sciences and Engineering Group
PO Box 999, Richland, Washington 99352

Project: #52578
Sample ID's: TI008-L, TI008-MK, TI008-ML, TI008-SK, TI008-SL

Procedure: APEL-PIP-4, Rev 2, *Gas Pycnometry Method for Apparent Specific Gravity Determinations of Consolidated Solids*

Analyst: Matthew Edwards
Analysis Dates: 06/14/2011, 06/15/2011, 6/23/11
Instrument: AccuPyc 1330, Serial #3955
Balance: Mettler AB104-S, Serial #1129263167

MEdwards 7/12/11
Prepared By/Date

7/12/2011
Technical Review/Date

7/14/11
QE Review/Date DSC 7-14-11

7/15/11
Project Management Release/Date

1.0 Introduction

This report addresses the particle density measurements of sieved fractions of the KW210 container material (+2000, +500/–2000, and –500 micron sieve fractions).

2.0 Instrumentation

Gas expansion pycnometer

The AccuPyc 1300 gas expansion pycnometer measures the volume of displaced gas (ultra-high purity [UHP] helium) in a volume pre-calibrated sample chamber. The instrument consists of a sample chamber and an expansion chamber separated by an expansion valve. After inserting a sample in the sample chamber, the sample chamber is filled with helium and gas molecules that fill the pores of the sample. The helium is then discharged into the expansion chamber, which has a known internal volume. The pressure difference between the isolated sample chamber and that of the sample chamber and expansion chamber combined is used to calculate volume displacement using Boyle's Law (1)

$$pV = k \tag{1}$$

Where: p denotes the pressure of the system, V is the volume of the gas, and k is a constant value representative of the pressure and volume of the system. Rearranging to fit our system gives (2)(Lowell, 2004).

$$V_s = V_c + \frac{V_r}{1-\frac{P_1}{P_2}} \tag{2}$$

Where V_s is the sample volume, V_c is the volume of the empty sample chamber, V_r is the reference volume, P_1 is the first pressure (in the sample chamber only) and P_2 is the second pressure after expansion of the gas into the combined volumes of sample chamber and reference chamber. V_s is then used with the pre-determined mass of the sample (m) to obtain particle density (ρ) (3).

$$\rho = m/V_s \tag{3}$$

3.0 Samples

Five samples were submitted for analysis. Using the tare weight of the vial that was recorded in 52578-TI008, the dry mass provided was obtained and recorded in Table 1. The –L and -M samples had particles of varying shapes, sizes, and colors and the –M samples also had visible resin beads. Also of note, the –M samples appeared to have a significant charge as can be seen in Figure 1. The –S samples were a dark brown powder. Pictures of the sample materials were taken and can be seen in Figure 2 through Figure 8.

Table 1 Sample Masses

Sub divided fraction identification	Physical description	Mass of subdivision (g)[1]
TI008-L	Particles of varying shapes, sizes and colors.	0.7588
TI008-MK	Particles of varying shapes, sizes and colors. Resin beads evident.	0.5231
TI008-ML	Particles of varying shapes, sizes and colors. Resin beads evident.	0.5074
TI008-SK	Dark Brown powder	0.6755
TI008-SL	Dark Brown powder	0.3462

[1]The tare mass of the vial was recorded in Test Instructions 52578-TI008. A gross weight of the sample was obtained. Mass of subdivision is the difference between the gross and tare masses.

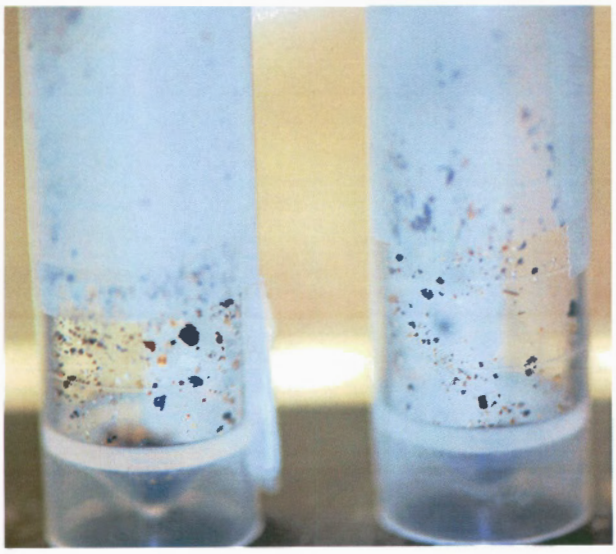

Figure 1 Sample Vials of TI008-MK & ML

Figure 2 TI008-L

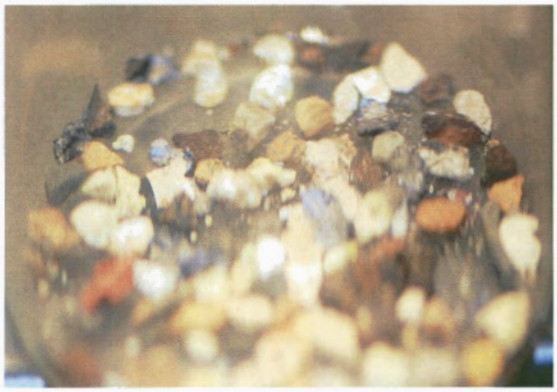

Figure 3 TI008-L

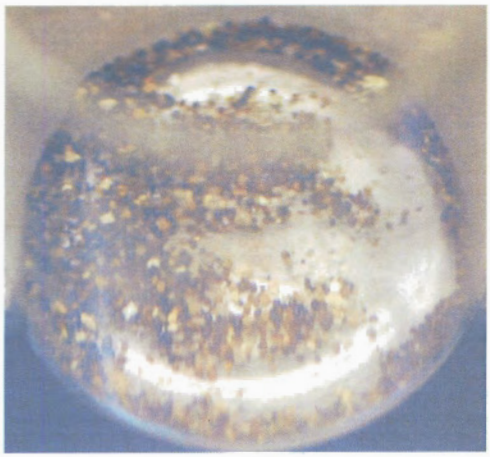

Figure 4 TI008-MK

Figure 5 TI008-MK

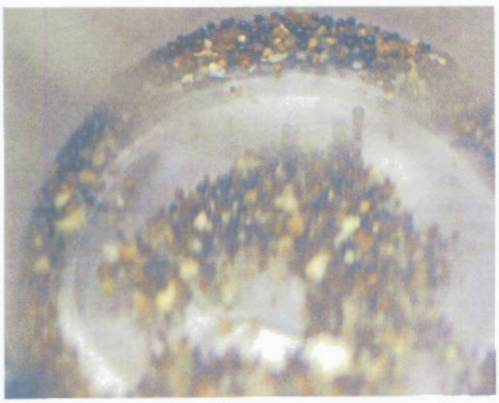

Figure 6 TI008-ML

Figure 7 TI008-ML

Figure 8 TI008-SK and TI008-SL

4.0 Experimental

As required by the analysis procedure APEL-PIP-4, *Gas Pycnometry Method for Apparent Specific Gravity Determinations of Consolidated Solids*, the AccuPyc 1330 Pycnometer was calibrated per the manufacturer's instructions using a verified calibration standard (#3955, verified volume 0.718566 cm^3) and confirmed with a second verified standard (#1500, verified volume 0.7185465 cm^3, expected density 14.9599 g/cm^3) at the beginning of each series of analyses, or at a minimum, daily. The calibration reports, provided from the instrument manufacture, are located in Appendix A.

During the course of K-Basin supported work, the calibration standard has been run multiple times. A summary of calibration data and a record of the pycnometer accuracy are provided in Appendix B. The calibration was considered acceptable if the measured volume of standard #1500 was within ± 1.0% of the certified value. The results of the standard measurements are shown in Table 2 and were within tolerance.

Table 2 Standard #1500 Measurements

Standard Measurement, g/cm^3	% deviation from standard density value of 14.9599 g/cm^3[(1)]	Within ± 1% DQO?, Y/N 15.1094 g/cm^3 > X > 14.8102 g/cm^3
14.9815	0.15	Y
14.9760	0.11	Y
15.0014	0.28	Y
14.9585	0.01	Y

(1) based on measured weight of 10.7494 g divided by certified volume of 0.7185465 cc

The balance (ID #1129263167) calibration was verified by weighing 1, 10, and 20 g standard weights and the checks were considered acceptable when the measured values were within ± 1.0% of the standard values.

The samples were placed in a tared 1-cm^3 pycnometer sample cup and weighed. The pycnometer cup and sample were placed in the AccuPyc 1330 sample chamber and the chamber cap was fastened securely. The sample was analyzed five times using UHP helium, the results were averaged and the density with standard deviation was recorded. To the extent possible, the whole sample aliquot provided was used for measurements.

5.0 Sample Results

The samples were transferred to the analysis cup with the intent of using the entire sample provided. Table 3 shows the success of this effort as well as the density result in g/cm^3 along with %RSD and %RPD where applicable.

Table 3 Mass Provided and Analyzed and Results

Sample	Mass of subdivision (g)(1)	Mass analyzed (g)(2)	Difference (g)	Density, g/cm^3	% RSD	% RPD
TI008-L	0.7588	0.6780	0.0808	2.60	0.15	NA
TI008-MK	0.5231	0.4142	0.1089	1.62	0.04	0
TI008-ML	0.5074	0.3420	0.1654	1.62	0.03	
TI008-SK	0.6755	0.6355	0.0400	2.57	0.05	3
TI008-SL	0.3462	0.2962	0.0500	2.49	0.07	

(1)The tare masses of the vials were recorded in Test Instruction 52578-TI008. Mass of subdivision is the difference between the gross and tare masses.
(2)Mass of the sample removed from vial and placed in analysis sample cup.

6.0 Data Quality Objectives

The accuracy DQO of ±1% was attained for balance and volume measures (refer to summary of calibration checks in Attachment B). The precision DQO of ±5% RPD was met for all duplicate samples.

7.0 Works Cited

Lowell, J. (2004). *Characterization of Porous Solids and Powders:Surface Area, Pore Size, and Density.* Springer.

Appendix A

Calibration Reports

micromeritics®

Calibration Data Report
AccuPyc 1330

Serial Number: 3955

Volume of Calibration Standard: 0.718566 cubic centimeters

Average Cell Volume: 1.2796 cubic centimeters

Average Expansion Volume: 0.7286 cubic centimeters

Temperature Offset: 1.45

Technician: Deane Bratches

Date: Sept. 8, 2005

170-662-36666

micromeritics

Calibration Data Report
AccuPyc 1330

Serial Number: 1500

Volume of Calibration Standard:	.7185465	cubic centimeters
Average Cell Volume:	1.2767	cubic centimeters
Average Expansion Volume:	.6891	cubic centimeters
Temperature Offset:	2.25	

Technician: Jeff Mack

Date: 10-10-96

Appendix B

Summary of Calibration Data

Appendix B

Pycnometer

Calibration Standard (#3955) date	Verification Standard (#1500) Density (g/cm^3)	Volume (cm^3)
5/28/2009	14.9802	0.7176
6/2/2009	14.9502	0.7190
9/4/2009	14.9596	0.7186
9/4/2009	14.9585	0.7186
9/16/2009	14.9733	0.7179
9/17/2009	14.9585	0.7186
9/30/2009	15.0215	0.7156
5/25/2010	14.9607	0.7185
6/10/2010	14.9607	0.7185
2/7/2011	14.9997	0.7166
2/8/2011	14.9686	0.7181
2/9/2011	14.9428	0.7194
2/10/2011	14.9603	0.7185
2/10/2011	14.9561	0.7187
2/10/2011	14.9555	0.7188
2/10/2011	14.9580	0.7186
2/11/2011	14.9602	0.7185
2/14/2011	14.9598	0.7185
2/14/2011	14.9566	0.7187
3/15/2011	14.9663	0.7182
6/13/2011	14.9818	0.7175
6/14/2011	14.9815	0.7175
6/15/2011	14.9760	0.7178
6/20/2011	15.0014	0.7166
6/23/2011	14.9585	0.7186
Average	**14.9682**	**0.7181**
Standard Deviation	**0.0179**	**0.0009**
***Accuracy**	**100.06%**	**99.94%**

*Accuracy calulation for Volume (0.7186 cc certified): 1-((0.7186-(0.7181))/0.7186)

*Accuracy calulation for Density (14.9599 g/cc◊◊): 1-((14.9599-(14.9682))/14.9599)

◊◊ based on measured weight of 10.7494 g divided by certified volume of 0.7186 cc

Pycnometer Analysis Sheet

APEL-PIP-4, Rev. 2

Pycnometer Information:

AccuPyc 1330
Serial Number: 3955
Gas used, Purity: Helium, UHP

*dev (1-5) indicate specific measurement deviation from the analyzed mean
Date analysis was performed: 6/14/2011

Standard Measurement:

Std ID: 1500
Std density: 14.9599 g/cm³
Std weight: 10.7494 g
Temperature: 22 °C

Density (1)	14.9770 g/cm³	dev (1)	0.0039 g/cm³
Density (2)	14.9768 g/cm³	dev (2)	0.0040 → 0.0041 g/cm³ MKE 6/27/11
Density (3)	14.9818 g/cm³	dev (3)	0.0010 → 0.0019 g/cm³
Density (4)	14.9856 g/cm³	dev (4)	0.0047 g/cm³
Density (5)	14.9831 g/cm³	dev (5)	0.0022 g/cm³
Average:			
Density	14.9809 g/cm³	std dev	0.0039 g/cm³

Std measurement +/- 1% certified value (Y/N): Y

Balance Check:

measured +/- 0.5% (Y/N)

Balance ID:	1129263167	
Cal Date:	8/16/2010	
Cal exp. Date:	8/1/2011	Y
1 g Wt check	1.0001 g	Y
10g Wt check	10.0002 g	Y
20g Wt check	20.0002 g	Y

std dev % 0.03%

Sample Measurements:

Sample ID: TI008-L
Weight: 0.6780 g
Temperature: 24.3 °C

Density (1)	2.5992 g/cm³	dev (1)	0.0024 g/cm³
Density (2)	2.6064 g/cm³	dev (2)	0.0048 g/cm³
Density (3)	2.5963 g/cm³	dev (3)	0.0053 g/cm³
Density (4)	2.6040 g/cm³	dev (4)	0.0024 g/cm³
Density (5)	2.6022 g/cm³	dev (5)	0.0005 → 0.0006 g/cm³ MKE 6/27/11
Average:			
Density	**2.6016 g/cm³**	**std dev**	**0.0040 g/cm³**

Analyzed 6/23/11

std dev % 0.15%

Sample ID: TI008-MK
Weight: 0.4142 g
Temperature: 22.8 °C

Density (1)	1.6208 g/cm³	dev (1)	0.0010 g/cm³
Density (2)	1.6215 g/cm³	dev (2)	0.0003 g/cm³
Density (3)	1.6223 g/cm³	dev (3)	0.0005 g/cm³
Density (4)	1.6220 g/cm³	dev (4)	0.0002 g/cm³
Density (5)	1.6223 g/cm³	dev (5)	0.0005 g/cm³
Average:			
Density	**1.6218 g/cm³**	**std dev**	**0.0006 g/cm³**

very statically charged
Analyzed 6/14/11

std dev % 0.04%

Analyst MKE Date 6/27/11
Reviewer [signature] Date 07/12/2011

1

Pycnometer Analysis Sheet APEL-PIP-4, Rev. 2

(Page 2)

Sample Measurements: | Sample Preparation/Observations:

Sample ID: TI008-ML
Weight: 0.3420 g Temperature: 23.0 °C

Density (1)	1.6208 g/cm³	dev (1)	0.0001 g/cm³	
Density (2)	1.6212 g/cm³	dev (2)	0.0006 g/cm³	MKE 6/27/11
Density (3)	1.6210 g/cm³	dev (3)	0.0004 g/cm³	very statically charged
Density (4)	1.6199 g/cm³	dev (4)	0.0008 g/cm³	lost some sample
Density (5)	1.6204 g/cm³	dev (5)	0.0002 g/cm³	Analyzed 6/14/11

Average:

Density	**1.6207 g/cm³**	**std dev**	**0.0005 g/cm³**	std dev %	0.03%
					0.1%

Sample ID: TI008-SK
Weight: 0.6355 g Temperature: 22.3 °C

Density (1)	2.5692 g/cm³	dev (1)	0.0012 g/cm³	MKE 6/27/11
Density (2)	2.5690 g/cm³	dev (2)	0.0014 g/cm³	extra drying time needed,
Density (3)	2.5717 g/cm³	dev (3)	0.0013 g/cm³	sample still damp
Density (4)	2.5699 g/cm³	dev (4)	0.0005 g/cm³	dried 6/13/11-
Density (5)	2.5720 g/cm³	dev (5)	0.0016 g/cm³	Analyzed 6/15/11

Density	**2.5704 g/cm³**	**std dev**	**0.0014 g/cm³**	std dev %	0.05%

Sample ID: TI008-SL
Weight: 0.2962 g Temperature: 22.5 °C

Density (1)	2.4916 g/cm³	dev (1)	0.0020 g/cm³	
Density (2)	2.4939 g/cm³	dev (2)	0.0003 g/cm³	extra drying time needed,
Density (3)	2.4922 g/cm³	dev (3)	0.0014 g/cm³	sample still damp
Density (4)	2.4950 g/cm³	dev (4)	0.0014 g/cm³	dried 6/13/11-
Density (5)	2.4954 g/cm³	dev (5)	0.0018 g/cm³	Analyzed 6/15/11

Average:

Density	**2.4936 g/cm³**	**std dev**	**0.0017 g/cm³**	std dev %	0.07%
					3.0%

Analyst MKE Date 6/27/11
Reviewer [signature] Date 07/12/11

Pycnometer Analysis Sheet

APEL-PIP-4, Rev. 2

(Page 3)

**Tares from: TI008 and measured on balance N04143
**For Gross Weights:

Balance Check:

Date Performed: 6/13/2011

			measured +/- 0.5% (Y/N)
Balance ID:	1.129E+09		
Cal Date:	8/16/2010		
Cal exp. Date:	8/1/2011	√	Y
1 g Wt check	1.0000 g	√	Y
10g Wt check	10.0002 g	√	Y
20g Wt check	20.0003 g	√	Y

Masses Provided:

Sample	Tare wt (g)	Gross wt (g)	Mass Provided:
TI008-L	√ 17.7301	18.4889	√ 0.7588
TI008-MK	√ 4.0141	4.5372	√ 0.5231
TI008-ML	√ 3.9887	4.4961	√ 0.5074

Date Performed: 6/15/2011 measured +/- 0.5%

Standard Measurement:

Std ID:	1500	Std weight:	10.7494 g
Std density:	14.9599 g/cm^3	Temperature:	√ 22.3 °C
Density (1)	√ 14.9777 g/cm^3	dev (1)	√ 0.0017 g/cm^3
Density (2)	√ 14.9741 g/cm^3	dev (2)	√ 0.0019 g/cm^3
Density (3)	√ 14.9750 g/cm^3	dev (3)	√ 0.0010 g/cm^3
Density (4)	√ 14.9759 g/cm^3	dev (4)	√ 0.0001 g/cm^3
Density (5)	√ 14.9772 g/cm^3	dev (5)	√ 0.0012 g/cm^3
Average:			
Density	√ 14.9760 g/cm^3	std dev	√ 0.0015 g/cm^3

Std measurement +/- 1% certified value (Y/N): Y

Balance Check:

			measured +/- 0.5% (Y/N)
Balance ID:	1129263167		
Cal Date:	8/16/2010		
Cal exp. Date:	8/1/2011		Y
1 g Wt check	√ 1.0004 g		Y
10g Wt check	√ 10.0002 g		Y
20g Wt check	√ 20.0006 g		Y
std dev %	0.01%		

Masses Provided:

TI008-SK	√ 17.4061	18.0816	√ 0.6755
TI008-SL	√ 17.681	18.0272	√ 0.3462

extra drying time needed, sample still damp

Analyst MKE
Reviewer

Date 6/27/11
Date 07/12/11

Pycnometer Analysis Sheet

APEL-PIP-4, Rev. 2

(Page 4)

Date Performed: 6/20/2011 measured +/- 0.5%

Standard Measurement:

Std ID:	1500	Std weight:	10.7494 g
Std density:	14.9599 g/cm^3	Temperature:	23.5 °C

Density (1)	15.0006 g/cm^3	dev (1)	0.0008 g/cm^3
Density (2)	15.0010 g/cm^3	dev (2)	0.0005 g/cm^3
Density (3)	15.0003 g/cm^3	dev (3)	0.0011 g/cm^3
Density (4)	15.0015 g/cm^3	dev (4)	0.0001 g/cm^3
Density (5)	15.0037 g/cm^3	dev (5)	0.0023 g/cm^3
Average:			
Density	15.0014 g/cm^3	std dev	0.0014 g/cm^3

Std measurement +/- 1% certified value (Y/N): Y

Balance Check:

		measured +/- 0.5% (Y/N)
Balance ID:	1129263167	
Cal Date:	8/16/2010	
Cal exp. Date:	8/1/2011	Y
1 g Wt check	1.0006 g	Y
10g Wt check	10.0002 g	Y
20g Wt check	20.0006 g	Y
std dev %	0.01%	

Date Performed: 6/23/2011 measured +/- 0.5%

Standard Measurement:

Std ID:	1500	Std weight:	10.7494 g
Std density:	14.9599 g/cm^3	Temperature:	√ 24.3 °C

Density (1)	√ 14.9586 g/cm^3	dev (1)	0.0001 g/cm^3
Density (2)	√ 14.9579 g/cm^3	dev (2)	0.0006 g/cm^3
Density (3)	√ 14.9588 g/cm^3	dev (3)	0.0003 g/cm^3
Density (4)	√ 14.9591 g/cm^3	dev (4)	0.0006 g/cm^3
Density (5)	√ 14.9581 g/cm^3	dev (5)	0.0004 g/cm^3
Average:			
Density	√ 14.9585 g/cm^3	√ std dev	√ 0.0005 g/cm^3

Std measurement +/- 1% certified value (Y/N): Y

Balance Check:

		measured +/- 0.5% (Y/N)
Balance ID:	1129263167	
Cal Date:	8/16/2010	
Cal exp. Date:	8/1/2011	Y
1 g Wt check	√ 1.0003 g	Y
10g Wt check	√ 10.0002 g	Y
20g Wt check	√ 20.0000 g	Y
√ std dev %	0.00%	

Analyst MKE Date 6/27/11

Reviewer [signature] Date 07/12/11

Pycnometer Analysis Sheet

APEL-PIP-4, Rev. 2

Pycnometer Information:

AccuPyc 1330
Serial Number: 3955
Gas used, Purity: Helium, UHP

*dev (1-5) indicate specific measurement deviation from the analyzed mean
Date analysis was performed: 6/14/11

Standard Measurement:

Std ID: 1500
Std density: 0.0000 g/cm³
Std weight: √ 10.7494 g
Temperature: √ 22.0 °C

Density (1)	√ 14.9770 g/cm³	√ dev (1)	0.0039 g/cm³
Density (2)	√ 14.9768 g/cm³	√ dev (2)	0.0040 g/cm³
Density (3)	√ 14.9818 g/cm³	√ dev (3)	0.0010 g/cm³
Density (4)	14.9856 g/cm³	√ dev (4)	0.0047 g/cm³
Density (5)	14.9831 g/cm³	√ dev (5)	0.0022 g/cm³
Average Density	14.9815 g/cm³	std dev	0.0045 g/cm³

14.9809

Std measurement +/- 1% certified value (Y/N): Y

Balance Check:

measured +/- 0.5% (Y/N)

Balance ID: 1129263167
Cal Date: 8/16/2010
Cal exp. Date: 8/1/2011 Y
1 g Wt check √ 1.0001 g
10g Wt check √ 10.0002 g
20g Wt check √ 20.0002 g

std dev %

Sample Measurements:

Sample ID: TI008-L
Weight: ~~0.7013~~ 0.6780 g MKE
Temperature: √ 24.3 °C

Density (1)	2.5942 g/cm³	√ dev (1)	0.0024 g/cm³
Density (2)	2.6064 g/cm³	√ dev (2)	0.0048 g/cm³
Density (3)	2.5983 g/cm³	√ dev (3)	0.0053 g/cm³
Density (4)	2.6040 g/cm³	√ dev (4)	0.0024 g/cm³
Density (5)	2.6022 g/cm³	√ dev (5)	0.0005 g/cm³
Average Density	2.6016 g/cm³	std dev	0.0040 g/cm³

std dev % 0.15

0.6827

Sample Preparation/Observations:

~~split sample in two because of continued instrument error.~~

#1 0.3764 #2 ~~0.3034~~

MKE

Analyzed 6/23/11

Sample iD: TI008-MK
Weight: √ 0.4142 g
Temperature: 22.8 °C

Density (1)	1.6208 g/cm³	√ dev (1)	0.0010 g/cm³
Density (2)	1.6215 g/cm³	√ dev (2)	0.0003 g/cm³
Density (3)	1.6223 g/cm³	√ dev (3)	0.0005 g/cm³
Density (4)	1.6220 g/cm³	√ dev (4)	0.0002 g/cm³
Density (5)	1.6223 g/cm³	√ dev (5)	0.0005 g/cm³
Average Density	1.6218 g/cm³	std dev	0.0006 g/cm³

std dev % 0.04

Very statically charged.

Analyst MKE
Reviewer

Date 6/27/11
Date 07/12/11

Pycnometer Analysis Sheet

APEL-PIP-4, Re

(Page 2) Date analysis was performed: 6/14/11 6/15/11

Sample Measurements:

Sample ID: TI008-ML MLE
Weight: 0.3469 g 0.3420 Temperature: 23.0 °C

Density (1)	1.6208 g/cm³	dev (1) 0.0001 g/cm³
Density (2)	1.6212 g/cm³	dev (2) 0.0006 g/cm³
Density (3)	1.6210 g/cm³	dev (3) 0.0004 g/cm³
Density (4)	1.6199 g/cm³	dev (4) 0.0008 g/cm³
Density (5)	1.6204 g/cm³	dev (5) 0.0002 g/cm³

Average:

Density 1.6207 g/cm³ **std dev** 0.0005 g/cm³ std dev % 0.03%

very statically charged lost some Sam

0.1% RPD

Sample ID: TI008-SK
Weight: 0.6355 g Temperature: 22.3 °C

Density (1)	2.5692 g/cm³	dev (1) 0.0011 g/cm³
Density (2)	2.5690 g/cm³	dev (2) 0.0014 g/cm³
Density (3)	2.5717 g/cm³	dev (3) 0.0013 g/cm³
Density (4)	2.5699 g/cm³	dev (4) 0.0005 g/cm³
Density (5)	2.5720 g/cm³	dev (5) 0.0016 g/cm³

Average:

Density 2.5704 g/cm³ **std dev** 0.0014 g/cm³ std dev % 0.05%

Sample wet dried 6-13-11 to analyzed 6/15/11

Sample ID: TI008-SL
Weight: 0.2962g Temperature: 22.5 °C

Density (1)	2.4916 g/cm³	dev (1) 0.0020 g/cm³
Density (2)	2.4939 g/cm³	dev (2) 0.0003 g/cm³
Density (3)	2.4922 g/cm³	dev (3) 0.0014 g/cm³
Density (4)	2.4950 g/cm³	dev (4) 0.0014 g/cm³
Density (5)	2.4954 g/cm³	dev (5) 0.0018 g/cm³

Average:

Density 2.4936 g/cm³ **std dev** 0.0017 g/cm³ std dev % 0.07

Sample wet dried 6-13-11 to analyzed 6/15

3.0 RPD

Analyst MLE Date 6/27/11
Reviewer [signature] Date 07/12/11

'age 3) Date analysis was performed: 6/14/11 6/15/11

*Tares from: TI008 measured on balance N04143

*For Gross Weights:

alance Check:

ate Performed: 6/13/11 measured +/- 0.5% (Y/N)

alance ID:	1.129E+09		
al Date:	8/16/2010		
al exp. Date:	8/1/2011		
g Wt check	1.0000 g	√	Y
)g Wt check	10.0002 g	√	↓
)g Wt check	20.0003 g	√	↓

lasses Provided:

Sample	Tare wt (g)	Gross wt (g)	Mass Provided:
I008-L	17.7301	18.4889	
I008-MK	4.0141	4.5372	
I008-ML	3.9887	4.4961	MKE
I008-SK	17.4061	~~17.4907~~	18.0816
I008-SL	17.6810	18.0272	

} Samples needed to dry - still damp. weighed 6/15/11

6/15/11

1g Wt. CK. 1.0004 √

10g wt. CK 10.0002 √

20g wt CK 20.0006 √

6/20/11

1g 1.0006

10g 10.0002

20g 20.0006

Std CK 6/15/11 Temp 22.3 C

√ 14.9777	0.0017	
√ 14.9741	0.0019	
√ 14.9750	0.0010	
√ 14.9759	0.0001	
√ 14.9772	0.0012	
√ 14.9760	0.0015	0.01%
15.0006	0.0008	23.5
15.0010	0.0005	
15.0003	0.0011	
15.0015	0.0001	
15.0037	0.0023	
15.0014	0.0014	0.01%

nalyst MKE Date 6/27/11

eviewer [signature] Date 07/12/11

3

6/23/11

1g V 1.0003

10g V 10.0002

20g V 20.0000

Std ck 6/23/11

V	14.9586	0.0001
V	14.9579	0.0006
V	14.9588	0.0003
V	14.9591	0.0006
V	14.9581	0.0004
	14.9585	0.0005

24.3C

0.00%

Analyst MLG 6/27/11

Reviewer [signature] 07/12/11

RPL K Basin Project 52578	Document Review Record MKE 7/12/11

Title:	Particle Density Report ~~Long Term Archive~~ TI008

Please return the completed form to: Matthew Edwards	Comments Due:

Organization/Department:	Designated Reviewer:	Reviewer Signature/Date: (Upon completion of review)
D9H63	Sergey Sinkov	07/12/2011

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	2 of 7	M	V_T in formula 2 does not match text in paragraph below	Changed V_T to V_r in formula 2 to match text below
2	Analysis Sheet	M	Density (5) FOR THE Std measurement on the completed analysis sheet does not match the hard copy (14.9831 and 14.9861) This also changes the Average density and std dev.	Transcription error. Changed value to 14.9831
3	Analysis Sheet	M	Standard dev for the TI008-MK sample is not filled in on the hard copy.	Added the std. dev. Value to the hard copy

Concur with comment resolution, review complete.	Comments Resolved By:
07/12/2011	7/12/11
Reviewer Signature Date	Author Signature Date

PROJECT 52578 – SURVEILLANCE REPORT

Number: SR-52578-2011-032

PLANNING

Topic/Issue to be evaluated: QE Review of Particle Density Report for TI008-L, TI008-MK, TI008-ML, TI008-SK and TI008-SL

This surveillance was undertaken to evaluate the draft Particle Density Report for five KW210 container material samples (+2000, +500/-2000 and -500 micron sieve fractions and supporting data for the samples listed in Table 1.

PERFORMANCE

Date Initiated: 6 April 2011	**Location:** RPL
Date Received: 12 July 2011 **Date Reviewed:** 13July 2011 **Date Completed:** 14 July 2011	**DRAFT Report Issued:** 13 July 2011 **FINAL Report Issued**: 14 July 2011
Contacts: Matt Edwards and Sergey Sinkov	**Org Code:** PE 137, D9H63
Surveillance Team: Lisa Middleton	

PRELIMINARY INFORMATION

Source of Requirement(s):

- KBC-33786, Rev. 2, December 2009; Quality Assurance Project Plan/Sampling and Analysis Plan (QAPjP/SAP): Quality Assurance Project Plan/Sampling and Analysis Plan for Containerized KW Settler Sludge

Other Applicable Documents:

- RPL-OP-001, Rev. 9, *Routine Research Operations*
- APEL-PIP-4, Rev. 2, *Gas Pycnometry Method for Apparent Specific Gravity Determinations of Consolidated Solids*
- 52578-ADM-001, Rev. 0, *K Basin Requests for Analysis and Guidelines for Data Reporting and Technical, QE, and Spreadsheet Reviews.*

Purpose/Scope:

The 52578 Project QE staff are required via the QA Program to perform a review of K Basin reports generated within the Radiochemical Processing Laboratory (RPL) to determine traceability and accuracy of data contributing to the completed report.

Number: SR-52578-2011-032

The draft report was reviewed to evaluate that the data are what they purport to be, that the reported results reflect what was actually done and the results meet applicable requirements. When deficiencies in the data are identified, then those deficiencies need to be documented as a K Basin Occurrence Report for the data user's review and, when applicable, resolved by corrective action.

Requirements:

The requirements for this work are defined in Table 3-10, KBC-33786 Rev 2; as well as the Analysis Request, applicable DQOs are:

- Precision $\pm$ 5%
- Accuracy $\pm$ 1%

The draft report itself was the basis for the data quality review.

When issues were found, the cognizant staff were contacted to resolve issues prior to completion of the review.

APPROACH

The Particle Density Report submitted for review consisted of the following for the five samples listed below in Table 1:

- Particle Density Report for five (5) KW210 samples (pp.7) including Figures 1-8, photographs of submitted samples
- Appendix A - Calibration Reports; Serial Numbers 1550 on 10/10/96 and 3955 on 9/8/05
- Appendix B - Summary of Calibration Data from 5/28/09 to 6/15/11
- Request for Particle Density Analysis by Gas Pycnometry (submitted 4/6/11)
- Pycnometer Analysis Sheet (pp.4) including M&TE, standard 1550 density results (η=5) on 6/14/11; Pycnometer Analysis Sheet with hand-written entries included (pp.4) – (bench sheets)
- Document Review Record, Sergey Sinkov, for Particle Density Report Long Term Archive, signed 7/12/11

Sample ID	Sample Description
TI008-L	Particles of varying shapes, sizes & colors.
TI008-MK	Particles of varying shapes, sizes and colors. Resin beads evident.
TI008-ML	Particles of varying shapes, sizes and colors. Resin beads evident.
TI008-SK	Dark Brown powder
TI008-SL	Dark Brown powder

Table 1. Subsample sieve fractions of KW210.

Approach:

The issues identified in the QE review of the report are noted below and a copy of the draft report with comments was provided to the author.

Before the completed report was signed off for QE review, it was determined that all comments/concerns from the technical review were addressed. There were no outstanding identified findings or observations.

Procedure Used:

APEL-PIP-4, Rev 2, *Gas Pycnometry Method for Apparent Specific Gravity Determinations of Consolidated Solids*

Sample Preparation:

Five (5) samples were submitted for analysis; one sample (TI008-L) was to be split for duplicate analysis. Tare weights are recorded in completed K Basin test Instruction (TI), TI008.

Samples Analyzed:

Five samples were submitted for analysis as noted above. The samples were analyzed five times and the results averaged and mass and density reported.

M&TE Used:

Location	Manufacturer	Model	Unique ID	RPL Calibration Control Tracking Number
RPL/506	Mettler	AB104-S	1129263167 Used on 6/14/11, 6/15/11, 6/20/11 & 6/23/11	1129263167
	AccuPyc	1330	3955	NA

RESULTS

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.

Summary of Identified Concerns:

DQO Compliance:

- Required duplicates; precision ± 5%; Yes
- Accuracy ± 1%; Yes

Note: Concerns identified in the review are expected to be addressed in a comment resolution meeting to be scheduled by the report author when the comments have been addressed. All concerns identified in the review were addressed in a comment resolution meeting on 14 July 2011. Deborah Coffey, Lisa Middleton and Matt Edwards attended. There were no outstanding identified findings or observations.

Identified issues of concern were the following:

1. Page 4 of the bench sheets (hand-written) is dated 6/23/11. The typed version is dated 6/20/11. If the analysis was indeed done on 6/20/11, the cover page of the report needs to be changed to reflect this. There is a second standard check below the 6/20 on the typed version that is dated 6/23. Standard checks were done on both days, but the analysis of the sample was done on 6/23. Discussed, no further action required. Closed

2. Page 5 of report: Is the expected density of the secondary standard supposed to be 14.9598 instead of 14.9599? Should be 14.9599, changed. Steel ball used for standard weighed 10.7494 instead of previous weight used in other analyses (10.7483), giving a different density (14.9599). Values for weight and density given in Table 2 of the report were corrected to reflect values attained in this analysis. No further action required. Closed

3. Can the content of page 7 fit onto page 6 so there isn't a large gap in the report? Yes. No further action required. Closed

4. The first formula in Appendix B should say density instead of volume. Please fix the template you use as well, so this won't come up again. Template was already fixed from last time ☺ I had just printed this out before I made the template change. Fixed, no further action required. Closed

5. The values listed in the volume & density formulas of Appendix B are incorrect. They should be 0.7181 & 14.9682 instead of 0.7183 & 14.9654 changed. Fixed, no further action required. Closed

6. Pycnometer Analysis Sheet: There are 8 deviations that the machine calculated wrong. Standard on page 1 #s 2 &3. TI008-L #5. TI008-ML #s 2, 3 & 5. TI008-SK # 1. 1st standard on page 4 # 2. See comment copy. Changed on sheets with pen and initialed and dated. Also changed future spreadsheets to calculate the number instead of an input from the instrument (since it has this issue occasionally). Fixed, no further action required. Closed

7. Pycnometer Analysis Sheets: It appears that the incorrect standard density was entered into the bench sheets. Shouldn't it be 14.9598 instead of 14.9599? No. 14.9599 is right. It is a calculation based on the standard weight and the calibrated volume. No further action required. Closed

PROJECT 52578 – SURVEILLANCE REPORT

Number: SR-52578-2011-032 Page 5 of 5

CORRECTIVE ACTION

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

Surveillance Performed By:

Lisa Middleton, Quality Engineer 7-14-11 Date: 7/14/11

DISTRIBUTION: Clark Carlson, Matt Edwards, Sandy Fiskum, Andy Schmidt, Rick Shimskey, Sergey Sinkov, Deborah Coffey, Lisa Middleton

Appendix H

Rheology

Appendix H

Rheology

Appendix H contains the rheology characterization report for the KW210 container composite samples and supporting documentation and calculations. The supporting documentation includes data reduction, calculation and graphing sheets, performance verification standard certificates, the referenced Nonconformance Report and the Surveillance Report .

The following table of contents shows that pagination specific to Appendix H was applied to the rheology report and supporting attachments. This pagination is sequential and consistent (e.g., page H.#).

Appendix H Table of Contents

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Rheological Measurements for K West Basin Consolidated Container Composites SCS-CON-210

Battelle—Pacific Northwest National Laboratory
Radiochemical Sciences and Engineering Group
PO Box 999, Richland, Washington 99352

Project: #52578

Sample ID: KW210-CC-A

Supporting Test Instruction: 52578-TI039

Procedure: RPL-COLLOID-02, Rev. 2, *Measurement of Physical and Rheological Properties of Solutions, Slurries and Sludges*

Analyst: Carolyn Burns
Analysis Dates: 05/26/2011 through 06/21/2011
Instrument: Haake RV20-M5, SN# 902398, with MV, MV2P and Vane Geometries

Supporting Electronic Report File Name: "TI-039 - 001 (Reviewed).xlsm"
File location: SharePoint site under TI-039 folder

08/02/11
Prepared By C Burns/Date

8/2/11
Reviewed By RC Daniel/Date

8/2/11
QE Reviewed By DS Coffey/Date DSC 8-2-11

8/3/11
Project Management Release/Date

1. Introduction

The sludge from the K Basins Pits and Floors (referred to as Container Sludge) has been consolidated into five engineered containers located under water in the K-West (KW) Basin. The container sludge originating from the K-East (KE) Basin resides in containers SCS-CON-240, -CON-250, and -CON-260. The container sludge originating from the KW Basin resides in SCS-CON-210 and -CON-220.

Two to four top-to-bottom core samples have been collected from each container and shipped to RPL. Previous test instructions have been implemented to reconstitute the sludge in a given core into a core composite sample. Sub-samples from each core composite within one Engineered Container have been consolidated into a single sample in an effort to represent the entire container (called a Container Composite). The Container Composite has been wet-sieved into +2000 micron, -2000/+500 micron, and -500 micron particle size fractions. Subsamples of the -500 micron fraction have been collected to support rheological characterization of this material. In 2009, rheological measurements were performed on all of the container composites with the exception of SCS-CON-210. This report addresses the rheological characterization for subsamples of the container composites for SCS-CON-210.

Figure 1 summarizes the processing steps for the preparation of the rheology composite sample. Samples were collected in equal volumes of dewatered sludge according to 52578-TI001 and –TI002. The volumes were not re-equalized after settling or further compaction upon standing. The four reserved "-A" subsamples were combined into one bottle, the settled solids volume was measured and the settled solids density calculated. The "A" bottle composite was then submitted for rheology testing.[(a)] Thus rheology (shear strength only) was tested on the full container composite (unseived) as opposed to the -500 micron fraction. Viscosity could not be measured on the unsieved material because of issues associated with the large particles. This approach, which differs from that defined in the QAPjP/SAP, was directed by the Buyer's Technical Representative (BTR) based on sample volume limitations and evolving Sludge Treatment Project priorities.[(b)]

This data package summarizes the test approach and results of rheological measurements performed under Test Instruction 52578-TI039*, Rheological Measurements for K West Basin Consolidated Container Composite SCS-CON-210.* Shear strength measurements were performed on one KW210 container composite sample: KW210-CC-A. Flow curve measurements were not performed as previously discussed.

2. Sample

Table 1 identifies the sample to be subjected to shear strength measurements. The shear strength measurements were conducted in the Shielded Analytical Laboratory (SAL).

(a) This material is expected to be used for Treat/Transfer testing according to scope defined in PRC-STP-TP-00261, Rev. 1 (*Test Plan for Polymer Testing at the Pacific Northwest National Laboratory on Actual K Basin Sludge Incorporating Treat and Transfer While Performing Solids Recirculation Assessment,* B Manning, 2/3/11) following testing for rheology.

(b) E-mail from WW Rutherford to SK Fiskum 2/14/11, "Concurrence requested for alternative subsampling and analysis of KW210 core samples."

Table 1. Container Composite KW210 Sample for Shear Strength Testing

Sample ID	Sample Purpose.	Vessel Description	Approximate Sample Size, wet sludge basis
Container Composite KW210, Subsample Collected under Test Instruction 52578-TI 008			
KW210-CC-A	Shear Strength	240 mL glass Qorpak	~180 mL

The general rheological characterization approach presented in this report was followed/cold tested for the characterization of K Basin sludge simulants in May 2009 (PNNL-18408). Key findings and lessons learned from this simulant characterization effort have been incorporated into this test instruction.

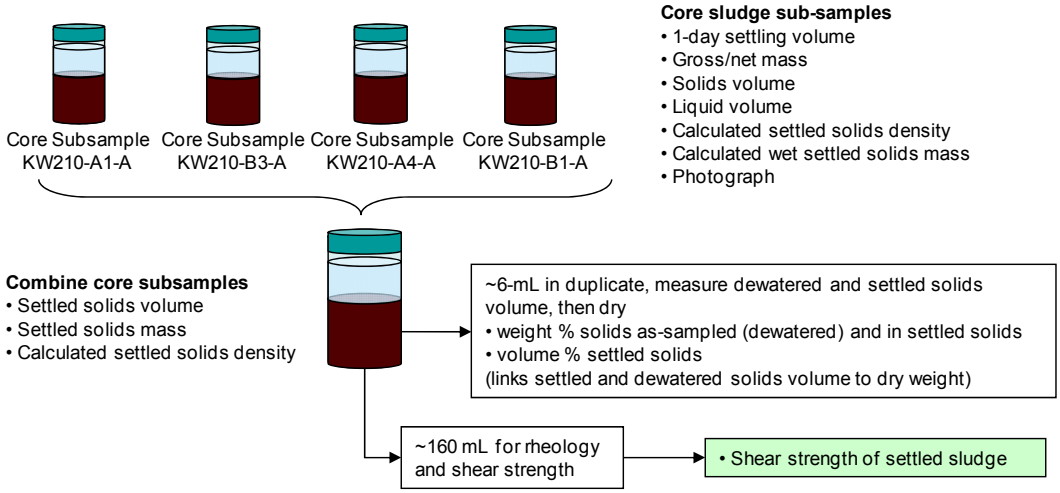

Figure 1. Overall Processing Schematic for the "A" Container Composite (shear strength sample preparation)

3. Shear Strength of Settled Sludge

Test Equipment Description

Shear strength was measured on the settled sludge sample in accordance with PNNL technical procedure, RPL-COLLOID-02, Rev. 2, "*Measurement of Physical and Rheological Properties of Solutions Slurries and Sludges*". The measurements were performed in SAL with a Haake RV20-M5 Viscometer. The RV20-M5 viscometer is capable of evaluating fluid rheological properties using Couette flow devices or shear strength using shear vanes. A shear vane tool was used for shear strength measurements. Table 2 summarizes the M5 measuring system information.

Table 2. Summary of Haake RV20 system with M5 measuring head

Analyzer:	Rotovisco® RV20 Measuring System M with M5 Measuring Head
Measurement principle:	Controlled Rate
Serial Number:	902398
Torque Sensor Range	0.49 to 49 mN·m
Rotational Rate Range	0.05 to 500 RPM
Software	RheoWin Pro, Version 2.96, 1996

Background

Shear strength is a semi-quantitative measurement of the stress required to yield a sample in shear such that it ceases to deform like a solid but instead flows like a viscous material with a finite viscosity. The shear strength of a sample may be dependent on sample history. The shear strength can be obtained by directly measuring resulting torque as a function of time when an immersed vane is slowly rotated in the sample material. The measured torque is converted to a shear stress by equations 1 and 2.

$$\tau = \frac{T}{K} \tag{1}$$

where

$$K = \frac{\pi D^3}{2}\left(\frac{H}{D} + \frac{1}{3}\right) \tag{2}$$

where τ = calculated shear stress in Pascal
T = measured torque in Newton-meters
K = shear vane constant in cubic meters
D = shear vane diameter in meters
H = shear vane height in meters.

This equation is based on the premise that the diameter of the vane shaft is much smaller than the diameter of the vane itself. A typical stress/time profile is shown in Figure 2. The profile shows an initial linear region (τ_y) followed by a nonlinear region, a stress maximum (τ_s), and a stress decay region. The stress maximum is typically associated with the transition between the visco-elastic and fully viscous flow. The shear strength is defined as the transition between these two flows and is associated with the stress maximum. The shear strength of the material is denoted as τ_S.

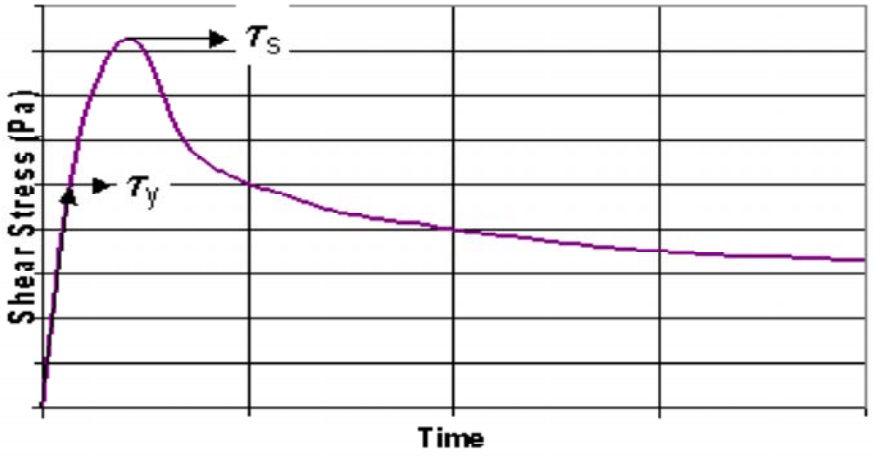

Figure 2. Typical Stress-Versus-Time Profile for a Shear Vane at Constant Shear Rate

Sample Test Approach

As shown in Table 1, the identified sample for shear strength was initially contained in a 240 mL Qorpak jar. The dimensions of this jar has been evaluated against PNNL technical procedure RPL-COLLOID-02, Rev. 2, to confirm that it meets the geometry requirements for a valid shear strength measurement; therefore shear strength measurements were performed *in situ.* Table 3 summarizes the matrix for the shear strength measurements. For the sample, up to 4 shear strength measurements were to be performed (depending on result reproducibility):

- One measurement on the "as received" sample where settling time is dependent on when the subsample was prepared and delivered to SAL for rheological measurements
- Two measurements at a consistent settling time ranging from 48 to 72 hrs
- A single replicate measurement of the two above was to be made should the second measurement of shear strength at 48-72 hrs differ from the first measurement by 25% or greater.

The first measurement was performed on the as-received sample/as sampled material. A 6×6 mm vane tool was implemented for the initial measurement, if the material strength was sufficiently low a second measurement may be possible. A larger, and hence more sensitive vane, would be implemented if the sample was undisturbed. Depending on the vane tool used for the remainder of the shear strength measurements, (determined after the initial measurement is performed) the sample size may only permit one sample measurement of the material at a point in time. After the initial measurement, the sample had been disturbed; therefore, following each measurement, the sample was remixed, and allowed to settle for a specified time, before the next measurement was taken.

As shown in Table 3, following the initial measurement, two measurements were performed on the sample after 48 to 72 hrs of settling and under near identical conditions. These measurements were to be performed over a period of 6 to 9 days (to allow mixing/setting between each measurement).

Table 3. Test Matrix for Shear Strength Measurements

Sample Identification (Container ID)	Sample Settling Time			
	As-received[(a)]	48 to 72 hr[(b)]	48 to 72 h[(b)]	48 to 72 hr[(b)]
KW210-CC-A (210)	x	x	x	x[(c)]

(a) As-received measurement to be performed on sample which has not been disturbed since parent sample aliquot.
(b) After a measurement is made, sample is to be remixed and allowed to settle. For good mixing, additional water may need to be added to the sample. It is understood that this mixing and settling could result in some segregation of sample components.
(c) If the first two measurements at 48-72 hours differ by more than 25% the third 48-72 hr measurement will be taken.

4. Results

Three shear strength measurements were performed on KW210-CC-A: one measurement of the "as-received" settled solids strength and two measurements of the settled solids strength ~48-hrs after solids re-suspension. Shear strength measurements employed either a 6×6 mm or a 16×16 mm (diameter by height) vane tool. The sequence for the three shear strength measurements and mixing/resuspension techniques employed for sample KW210-CC-A are described in Table 4.

Table 4. Test sequence for shear strength measurements reported for sample KW210-CC-A.

Sequence	Test	Description	Post-Test Mixing
1	"As-Received"	Shear strength measurement of the "as-received" settled solids. Approximately 2-weeks of time between current test and time of last solids disruption.	Sample homogenized and transferred to a 120 mL jar for subsequent measurements.
2	First Mix, ~48 -hr Settled	Shear strength measurement ~48-hrs following post-test mixing operations associated with the "as-received" measurements	Mixed with a 16×16 mm vane impeller for 2 minutes. Sample inverted to inspect jar bottom for complete mixing.
3	Second Mix, ~48 hr Settled	Shear strength measurement 48-hrs following post-test mixing operations associated with the first 48-hr settled test.	Mixed with a 16×16 mm vane impeller for 2 minutes. Sample inverted to inspect jar bottom for complete mixing.

Table 5. Settled solids and total slurry height inventories taken during testing associated with this data package.

Sample ID	Test	Settled Solids Height, cm	Total Slurry Height, cm	Temperature °C
KW210-CC-A	"As-received"	3.2	7.5	27.3
	First Mix	4.7	5.8	29.5
	Second Mix	5.2	6	29.8

Both before and during the course of the series of measurements outlined in Table 4, samples were monitored for loss of water. The total level of slurry (settled solids plus water) was adjusted as deemed necessary by the lead rheologist (Carolyn Burns) or cognizant scientist (Rick Shimskey). Water loss due to evaporation was offset in most cases by the DI water used to rinse down the vane tool after each measurement. Efforts were made to keep the total slurry volume of the sample constant, small volumes of water were used to wash down the vane tool after each measurement to minimize sample loss. Table 5 provides a summary of the sample volumes.

The results of shear strength testing are presented in Table 6. Sample test and mixing times are given in Table 7. All measurements were made at ambient cell temperature. "As-received" measurements corresponded to an overall settling time of ~1824 hrs (~11 weeks); all other measurements corresponded to a settling period ~48 hrs following sample mixing.

The "as-received" sample, settling time of approximately 11 weeks, was measured using a 6×6 mm vane tool to avoid exceeding the maximum operating torque of the RV20-M5 measuring system. This is consistent with guidance from CHPRC directing PNNL to use a 6×6 mm vane tool (on October 11, 2010) for initial shear strength measurements. The initial strength of the sample was established to be within the working stress range of the 16×16 mm vane and all subsequent measurements were made with this vane tool. The sample was transferred to a smaller container so that the 16×16 mm vane tool could be used, as there was insufficient settled solids height to use this vane in the original container. Table 8 provides a summary of shear strength measurements. An average shear strength for sample KW210-CC-A is given; the average was determined using the first measurement from the first and second settle-and-mix operations. For each test, the vane was immersed into the center of jar at the indicated depth.

Table 6. Summary of shear strength measurement results.

Sample ID	Test	Vane Tool [D×H], mm	Settling Time, hrs	Immersion Depth, mm	Shear Strength, Pa
KW210-CC-A	"As-received"	6×6	1824	6	281
	First Mix	16×16	50	16	63
	Second Mix	16×16	47	16	58
	Second Mix	16×16		26	113

Table 7. Summary of Test and Mixing Times

Sample ID	Test	Last Mixed	Sample Test	Estimated Settling Time, hrs
KW210-CC-A	"As-received"	2011/03/17 15:19	2011/06/01 15:00	1824
	First Mix	2011/06/10 15:00	2011/06/12 16:48	52
	Second Mix	2011/06/12 16:55	2011/06/14 15:38	47

Table 8. Shear Strength Summary Table for KW210-CC-A
All measurements were taken at ambient cell temperature (~29° C)

Sample ID	Measurement	Settling Time (hr)	Immersion Depth (mm)	Shear Strength (Pa)
KW210-CC-A (KW210-SCC)	As-Received[(c)]	1824	6	~281
	1st Mix	50	16	63[(a)]
	2nd Mix	47	16	58[(a)]
	2nd Mix	47	26	113
	Average			**60 ± 5[(b)]**

(a) Used in average calculation.
(b) Reported uncertainty is twice the standard error of the mean (which approximated the lower bound of the 95% confidence limits).
(c) Initial "as-received" strength measurement using a 6×6 mm vane. Subsequent measurements taken with the 16×16 mm vane.

References

HNF-SP-1201: Makenas, BJ, TL Welsh, RB Baker, GR Golcar, PR Bredt, AJ Schmidt and JM Tingey. 1997. *Analysis of Sludge From Hanford K East Basin Canisters*, HNF-SP-1201, Westinghouse Hanford Company, Richland, Washington.

PNNL-18408, Rev. 1: Burns, CA; M Luna, AJ Schmidt, 2011, *Characterization of Settler Tank, KW Container and KE Container Sludge Simulants*, PNNL-18408, Rev. 1., Pacific Northwest National Laboratory, Richland, Washington.

PNNL-19035: Fiskum SK, JM Billing, SJ Bos, CA Burns, CD Carlson, DS Coffey, JV Crum, RC Daniel, CH Delegard, MK Edwards, OT Farmer, LR Greenwood, SA Jones, D Neiner, BM Oliver, KN Pool, AJ Schmidt, RW Shimskey, CZ Soderquist, CJ Thompson, ML Thomas, T Trang-Le, and MW Urie. 2009. *Characterization Data Package for Containerized Sludge Samples collected from Engineered Containers SCS-CON-240, 250, 260, and 220*. PNNL-19035, Rev. 1, Pacific Northwest National Laboratory, Richland, Washington.

52578-TI039-Rheology Data Package - Coverpage

Description:
This Excel file data package documents measurement results (using Microsoft® Office Excel) for rheology data collected under Test Instruction (TI) 52578-TI039, Rheological Measurements for K West Basin Consolidated Container Composites SCS-CON-210. Results for sample KW210-CC-A are given.

Computer Employed:
MHP Dell Latitude D830 Laptop Computer (Property # WE14110)

Operating Environment:
Microsoft Windows 7 Enterprise

Base Software Program:
Microsoft Office Professional Plus 2010 Excel Version 14.0.5128.5000 (32-bit)

Package Contents:
Package consists of this coverpage, a calculation review page, print-outs for one Microsoft® Office Excel workbook, and all other relevant documentation.

Description	File Date	File Size	Number of Pages
Data package coverpage	n/a	n/a	1
Calculation review worksheet	n/a	n/a	1
Excel printouts: "TI-039 - 001 (Reviewed).xlsm"	8/3/2011 11:45	45 KB	5
Standard certificate: "Certificate 092710 (332587).pdf"	5/13/2011 13:20	578 KB	1
K-Basin Non-Conformance Report - NCR 52578-11-08-10	5/13/2011 13:22	853 KB	2
K-Basin Surveillance Report - SR-52578-2011-035	n/a	n/a	5

Package Generator Signature:

Generator: Carolyn Burns

08/03/11
Date

Calculation Review Sheet

Reviewer Name: Richard C. Daniel
Reviewer Title: S/E
Review Date: 7/26/2011

Title of Spreadsheet Calculation Reviewed: One spreadsheet and a calibration certificate are included in this review package:
1) "TI-039 - 001 (Reviewed).xlsm"
2) "Certificate 092710 (332587).pdf"

Revision Number: n/a
Date Prepared: 7/26/2011

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

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

CHECK LIST

Spreadsheet/Calculation Identification	**Yes**	**No**	**NA**
Spreadsheet/Calculation Title:			X
Revision Number:	X		
Date Prepared	X		
Prepared by:	X		
General Statement of Purpose:	X		
General Description of Approach (note: specific approaches may be described for each section/subsection of spreadsheet/calculation)			X

Comments: Standard rheology spreadsheet, approach per RPL-COLLOID-02, Rev. 2

Assumptions	**Yes**	**No**	**NA**
Are assumptions clearly stated?	X		
Are assumptions supported/justified?	X		
Are assumptions reasonable?	X		

Comments: No 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		

Comments: No comments

Equations/Approach	**Yes**	**No**	**NA**
Are equations adequately defined?	X		
Are equations properly referenced?			X
Are limitations of approach/equations identified?			X
Are equations appropriate?	X		
Are units consistent?	X		

Comments: No comments

Results/Conclusions	**Yes**	**No**	**NA**
Are formulas consistent in spreadsheet cells?	X		
Are calculations correct?	X		
Are conclusions consistent with results?			X
Are conclusions consistent with applicable limits?			X

Comments: No comments

Reviewer Sign/Date: 7/26/2011
Richard C. Daniel

	A	B	C	D	E	F	G	H	I	J
1	**Summary of Shear Strength Testing**									
2										
3	**Sample ID**	**State**	**Last Mixed**	**Tested**	**Gel Time**	**Settled Solids Height**	**Total Sample Height**	**Temperature**	**Measurement**	**Vane tool**
4					**[hrs]**	**[cm]**	**[cm]**	**[°C]**		**[D×H] mm**
5	KW210-CC-A	"As-Received"	2011/03/17 15:19	2011/06/01 15:00	1824	3.2	7.5	27.3	1 of 1	6×6
6		Mix 1	2011/06/10 15:00	2011/06/12 16:48	50	4.7	5.8	29.5	1 of 1	16×16
7		Mix 2	2011/06/12 16:55	2011/06/14 15:38	47	5.2	6	29.8	1 of 2	16×16
8			2011/06/12 16:55	2011/06/14 15:56	47	5.2	6	29.8	2 of 2	16×16
9										
10	All measurements taken in the centre of the sample container.									

	K	L	M	N	O
1					
2					
3	**Immersion Depth**	**Measurement File**	**Shear Strength**	**Post-Test Mixing**	**Comments**
4	**[mm]**		**[Pa]**		
5	6	KW210-CC-A-Ar-110601-1.1	280.9	Sample homoginized and transferred	Transferred sample to 120 mL container for subsequent measurments.
6	16	KW210-CC-A-Mix 1-110612-1.1	62.77	Mixed ~ 2 min with 16 mm vane	N/A
7	16	KW210-CC-A-Mix 2-110614-1.1	57.83	Used for Treat and Transfer sample	N/A
8	26	KW210-CC-A-Mix 2-110614-1.2	112.80	N/A	Mix 1 and 2 values differed by ~ 8%: third mix not needed
9					
10					

Filename: TI-039 - 001 (Reviewed).xlsm
Worksheet: Shear Strength Table

	A	B	C	D	E	F	G
1	**Shear Strength Summary Table (For Report)**						
2							
3	**Sample ID**	**Test**	**Temperature**	**Settling Time**	**Location**	**Immersion Depth**	**Shear Strength**
4			**(°C)**	**(hr)**		**(mm)**	**(Pa)**
5	KW210-CC-A	As received	27.3	1824	center	6	280.9
6		Mix 1	29.5	50	center	16	63
7		Mix 2	29.8	47	center	16	58
8		Mix 2	29.8	47	center	26	113
9							
10	**Average Shear Strength for KW210-CC-A at ~48 hrs Settling Time**						
11	Based on 16 mm measurements (Mix 1 and 2)						
12	**Average**	60.3	Pa				
13	**Standard Deviation**	3.5	Pa				
14	**Standard Error**	2.5	Pa				
15	**Formatted Result**	60 ± 5	Pa				

	A	B	C	D	E	F	G	H	I
1	**Performance Check of M5-RV20 (SAL Cell 5)**								
2	Date:	2011-05-26							
3	Effective to:	2011-06-25							
4									
5	In-Cell (SAL) Temperature Measurement								
6	**Device**	**ID**	**Expires**						
7	Thermocouple	28534	2011-11-02						
8	Thermocouple Display - Dickson	28537	2011-11-01						
9									
10	Cold Rheometer Temperature Measurements for Cold-Reference Measurements (Performed in February)								
11	**Device**	**ID**	**Expires**						
12	Thermocouple	22483	2011-03-01						
13	Thermocouple Display	22793	2011-02-19						
14									
15	Viscosity Standard								
16	**Description**	**Lot**	**List Viscosity**	**Temperature**	**Expires**	**Low**	**High**		
17			**[cP]**	**[°C]**		**[cP]**	**[cP]**		
18	Brookfield Fluid 100 (SAL)	092710	96.0	25	2012-04-27	86.4	105.6		
19	Brookfield Fluid 100 (APEL)	092710	96.0	25	2012-05-02	86.4	105.6		
20	Viscosity Measurements								
21	**Description**	**Instrument**	**Serial Number**	**Temperature**	**Filename**	**Viscosity**	**E_r**	**Acceptable**	**Sensor**
22				**[°C]**		**[cP]**	**[%]**		
23	Cold-Reference Measurement 1	RS600	1200202352004	Control (20)	110222_112_b	107.3	n/a	n/a	Z41
24	Cold-Reference Measurement 2	RS600	1200202352004	Control (25)	110222_112_a	98.41	2.5	Yes	Z41
25	Cold-Reference Measurement 3	RS600	1200202352004	Control (30)	110222_112_c	89.68	n/a	n/a	Z41
26	Predicted Reference Viscosity	n/a	n/a	24.4	n/a	99.42	n/a	n/a	n/a
27	SAL Measurement 1	M5-RV20	SAL Cell 5	Ambient (24.4)	110526-Performance check -24.4	102.5	3.1	Yes	MV2P
28	SAL Measurement 2	M5-RV20	SAL Cell 5	Control (25)	110526-Performance check -25	101.7	5.9	Yes	MV2P
29	SAL Measurement 3	M5-RV20	SAL Cell 5	Control (24.4)	110526-Performance check -24.4b	102.5	n/a	n/a	MV2P
30									
31	Reference Data Analysis								
32	**Measurement**	**Temperature**	**Viscosity**	**1/T**					
33		**[°C]**	**[cP]**	**[K^{-1}]**					
34	1	20	107.3	0.003411223					
35	2	25	98.41	0.003354016					
36	3	30	89.68	0.003298697					
37									
38	Slope	156579.4303							
39	Intercept	-426.8051413							
40	R^2	0.999980293							

H16

	A	B	C	D	E	F	G	H	I
1	**Performance Check of M5-RV20 (SAL Cell 5)**								
2	Date:	2011-06-21							
3	Effective to:	2011-07-21							
4									
5	In-Cell (SAL) Temperature Measurement								
6	**Device**	**ID**	**Expires**						
7	Thermocouple	28534	2011-11-02						
8	Thermocouple Display - Dickson	28535	2011-11-01						
9									
10	Cold Rheometer Temperature Measurements for Cold-Reference Measurements (Performed in February)								
11	**Device**	**ID**	**Expires**						
12	Thermocouple	22483	2011-03-01						
13	Thermocouple Display	22793	2011-02-19						
14									
15	Viscosity Standard								
16	**Description**	**Lot**	**List Viscosity**	**Temperature**	**Expires**	**Low**	**High**		
17			**[cP]**	**[°C]**		**[cP]**	**[cP]**		
18	Brookfield Fluid 100 (SAL)	092710	96.0	25	2012-04-27	86.4	105.6		
19	Brookfield Fluid 100 (APEL)	092710	96.0	25	2012-05-02	86.4	105.6		
20	Viscosity Measurements								
21	**Description**	**Instrument**	**Serial Number**	**Temperature**	**Filename**	**Viscosity**	**E_r**	**Acceptable**	**Sensor**
22				**[°C]**		**[cP]**	**[%]**		
23	Cold-Reference Measurement 1	RS600	1200202352004	Control (20)	110222_112_b	107.3	n/a	n/a	Z41
24	Cold-Reference Measurement 2	RS600	1200202352004	Control (25)	110222_112_a	98.41	2.5	Yes	Z41
25	Cold-Reference Measurement 3	RS600	1200202352004	Control (30)	110222_112_c	89.68	n/a	n/a	Z41
26	Predicted Reference Viscosity	n/a	n/a	30.2	n/a	89.36	n/a	n/a	n/a
27	SAL Measurement 1	M5-RV20	SAL Cell 5	Ambient (30.2)	110621-Performance check-T30-a	90.28	1.0	Yes	MV1
28	SAL Measurement 2	M5-RV20	SAL Cell 5	Control (25)	110621-Performance check-T25-b	96.71	0.7	Yes	MV1
29	SAL Measurement 3	M5-RV20	SAL Cell 5	Control (30.3)	110621-Performance check-T30.3Controlled-c	88.56	n/a	n/a	MV1
30									
31	Reference Data Analysis								
32	**Measurement**	**Temperature**	**Viscosity**	**1/T**					
33		**[°C]**	**[cP]**	**[K^{-1}]**					
34	1	20	107.3	0.003411223					
35	2	25	98.41	0.003354016					
36	3	30	89.68	0.003298697					
37									
38	Slope	156579.4303							
39	Intercept	-426.8051413							
40	R^2	0.999980293							

ISO 9001 CERTIFIED

PO #136236
BC# 332587

BROOKFIELD

CERTIFICATE DATE

September 29, 2010

CERTIFICATE OF VISCOSITY STANDARD CALIBRATION

FLUID	VISCOSITY	TEMPERATURE	LOT NUMBER
100	96.0	25.0	092710
	(CENTIPOISE)	(°C)	

rec'd 11/5/10

Accepted as per QA Clause 18 as cert'd 12-2-10 NCR 52578-11-08-10 use-as-is

TEST INSTRUMENT

MODEL	SERIAL NUMBER
LVDV-II+	6529213

TEMPERATURE SENSOR

MODEL	SERIAL NUMBER
1529	A33422

CALIBRATION OF THE ABOVE-MENTIONED BROOKFIELD VISCOSITY STANDARD IS ACCURATE TO WITHIN 1% OF ITS STATED VALUE.

THE VISCOSITY STANDARD OF THIS LOT NUMBER WAS MEASURED WITH A BROOKFIELD VISCOMETER THAT HAS BEEN CALIBRATED AGAINST A PRIMARY STANDARD, TRACEABLE TO THE NATIONAL INSTITUTE OF STANDARDS & TECHNOLOGY.

BROOKFIELD ENGINEERING LABORATORIES, INC.

David J. Moonay

DAVID J. MOONAY, Ph.D - RHEOLOGY LABORATORY SUPERVISOR

BROOKFIELD ENGINEERING LABORATORIES, INC.
11 Commerce Blvd., Middleboro, MA 02346-1031 U.S.A.
TEL 800-628-8139 or 508-946-6200 FAX 508-946-6262
www.brookfieldengineering.com

NONCONFORMANCE REPORT Project Number: 52578	Page 1 of 2	NCR No.: 52578-11-08-10 Rev. No.: 0

Item Name/Description:
Item No. 100 CPS; 500 mL Silicon Oil, 100 cP Viscosity Standard , Lot # 332587, Certificate of Analysis received from Brookfield Engineering Laboratories, Inc. for PO#136236.

Hold Location: In RPL/302 on the bench across from the particle size analyzer.
Quantity of Tags: 1 hold tag applied for 2 containers; 1 "Accepted" green tag applied for 2 containers

Supplier Name: Brookfield Engineering Laboratories, Inc.	**Purchase Order No.:** PO 136236

Description of Nonconformance (As-Required vs. As-Found)

During the QA Clause 188 review by ^PNNL Acquisition Quality Support for Item No. 100 CPS; 500 mL Silicon Oil, 100 cP Viscosity Standard , Lot # 332587, Certificate of Analysis received from Brookfield Engineering Laboratories, Inc. for PO#136236, the following issues were identified:

The requirements of Quality Assurance Clause 188 were not met at the project-level for the following reasons:
The NIST traceability although stated on the certificate is not explicitly identified to a NIST standard or standards and therefore, a basis for traceability was not established; Brookfield states that this information is available upon request, but when requested it was not forthcoming.

1. Further, the certificate does not link to the item (part) number.
2. An uncertainty is not provided for the viscosity value.
3. No expiration date for the standard was provided.

Note: The contract specialist was contacted as well as David A. DiCorpo, General Manager, Laboratory Sales, Brookfield Engineering Laboratories, Inc. and we attempted to resolve the issues. A custom certificate was requested (11/11/2010), but as of 2 December 2010 it had not been received. As the supplier was deemed non-responsive, the Project Manager directed the NCR to be dispositioned as noted on page 2.

Originator's Signature: DS Coffey — **Date:** 2 Dec 2010
DS Coffey

Send copy of NCR to Project 52578 Quality Engineer. **Date:** 2 Dec 2010

NONCONFORMANCE REPORT Project Number: 52578	Page 2 of 2	NCR No.: 52578-11-08-10 Rev. No.: 0

Disposition, Justification, and Instructions: [ ] **Repair** [ ] **Rework** [X] **Use As-Is** [ ] **Reject**

Procurement of equivalent standards from a supplier different than Brookfield Engineering Laboratories, Inc. will be investigated to determine if a verified NIST –traceable standard can be obtained.

Design Document Change Required? **No [X] Yes [ ]** **If Yes, Document No.:** **Rev. No.:**	
Designated Technical Staff Member: (Clark Carlson, K Basin Project Manager) **Date:** 12/2/2010	**Quality Engineer:** DS Coffey (DS Coffey, Concurrence) **Date:** 2 Dec 2010
Disposition Completed as Directed DS Coffey **Verification and Close-out** (DS Coffey, Quality Engineer) **Date:** 2 Dec 10	

Distribution:
Technical Staff: Richard Daniel
Originator: : DS Coffey
Contract Specialist: : Leandra Peterson
Quality Engineer: : DS Coffey

PROJECT 52578 – SURVEILLANCE REPORT

Number: SR-52578-2011-035

PLANNING

Topic/Issue to be evaluated: Surveillance of Completed K Basin TI, 52578-TI039, Rev. 0, Test Instruction for *Rheological Measurements for K West Basin Consolidated Container Composite SCS-CON-210.*

This surveillance was undertaken to evaluate completed test instruction (TI), 52578-TI039, Test Instruction for *Rheological Measurements for K West Basin Consolidated Container Composite SCS-CON-210,* associated supporting information and spreadsheets, and the completed report, *Rheological Measurements for K West Basin Consolidated Container Composites SCS-CON-210.* The report was reviewed concurrently with TI039, but the report review will be documented via a Document Review Record (DRR) form. TI039 was used between 5/26/2011 and 7/26/2011. TI039 directs the performance of rheological measures for one sample using procedure, RPL-COLLOID-02, Rev. 2, *Measurement of Physical and Rheological Properties of Solutions Slurries and Sludges.* Carolyn Burns signed off TI steps. Richard Daniel performed a Technical Review of the TI.

PERFORMANCE

Date Initiated: 2 February 2011 **Date Completed**: 26July 2011	**Location:** RPL
Date Received: 27 July 2011 **Date Reviewed:** 28 July 2011	**DRAFT Report Issued:** 29 July 2011 **FINAL Report Issued**: 2 August 2011
Contacts: Carolyn Burns, Clark Carlson, Richard Daniel and Sandy Fiskum	**Org Code:** PE 137, D9H63
Surveillance Team: Lisa Middleton	

Source of Requirement(s):

- KBC-33786, Rev. 2, Quality Assurance Project Plan/Sampling and Analysis Plan (QAPjP/SAP): Quality Assurance Project Plan/Sampling and Analysis Plan for Containerized KW Settler Sludge; December 2009.
- CH2M Hill Plateau Remediation, Contract 41108, Statement of Work, Technical Support for Planning, Preparing, and Analyzing KW Settler Sludge (February 11, 2010).
- HNF-36985, Rev. 3, Data Quality Objectives for Sampling and Analysis of K Basin Sludge; July 2009

Other Applicable Documents:

- RPL-OP-001, Rev. 9, *Routine Research Operations*
- ADM-RSEG-BALANCES, Rev. 0, *Balance Calibration Verification*

Number: SR-52578-2011-035

Purpose/Scope:

The 52578 Project QE staff are required via the TI to perform a review of completed TIs. The document was reviewed to evaluate completeness, that all entries were recorded properly, the errors were corrected appropriately and when needed, that justification was present to describe data corrections, that any issues affecting data quality are documented as Occurrence Reports and that the attachments to the TI were at the stage to become a project record. Additionally, TI's are reviewed to assure that the technical reviews were performed and issues were dispositioned and that data are traceable to point of use, generally Excel spreadsheets, which also require technical review and comment/issue resolution. When issues were found, the cognizant staff were contacted to resolve issues prior to completion of the review.

Approach:

TI, 52578-TI039, required the rheology analysis of one sample as noted below:

Sample ID	Sample Purpose.	Vessel Description	Approximate Sample Size, wet sludge basis
Container Composite KW210, Subsample Collected under Test Instruction 52578-TI 008			
KW210-CC-A	Shear Strength	240 mL glass Qorpak	~180 mL

The TI package submitted for review consisted of the following:

1. Completed TI, 52578-TI039, Rev. 0, Test Instruction for *Rheological Measurements for K West Basin Consolidation Container Composite SCS-CON-210* (pp. 1-21) With the following inserted: Performance checks dated 5/26/11 & 6/21/11 & Flow curves/ rheograms dated 6/1/11, 6/12/11 & 6/14/11
2. Brookfield certificate of viscosity standard calibration (lot # 092710)
3. Nonconformance Report – NCR 52578-11-08-10, related to QA Clause 188 review by ^PNNL Acquisition Quality Support for Item No. 100 CPS; 500 mL Silicon Oil, 100 cP Viscosity Standard , Lot # 332587, Certificate of Analysis received from Brookfield Engineering Laboratories, Inc. for PO#136236, the following issues were identified:
   - Basis for NIST traceability was not demonstrated.
   - Further, the certificate does not link to the item (part) number.
   - An uncertainty was not provided for the viscosity value.
   - No expiration date for the standard was provided.

   Disposition was to use as-is, and identify for a different vendor for this standard
4. M&TE including BPCL forms for Ohaus balance AV4101C (8031221019): 6/14/11 through 7/8/11& Sartorius balance MSG234 (40023610/WD68444): 7/6/11 &7/7/11 IS&T Records of Calibration: duel element temperature display (28535) & thermocouple (28534))
5. Calculation Review Sheet signed by Technical Reviewer Richard Daniel,7/26/11 for TI039 – 001 (Reviewed).xlsm
6. Spreadsheet named TI-039 – 001 (Reviewed).xlsm with the following worksheets:
   - *Summary*
   - *Shear Strength Table*
   - *PCheck-Open*

- *PCheck-Close*

7. DRR for completed TI039 & report *Rheological Measurements for K West Basin Consolidated Container Composites SCS-CON-210*, signed by Richard Daniel 7/27/11

M&E Used:

M&TE	Description	ID #/Serial Number	Cal. Exp.	Dates Used
Balance	Ohaus AV4101C	8031221019	2/12	6/10/11
Balance	Sartorius MSG234	40023610/WD68444	8/11	6/10/11
Thermocouple	KMQSS-062U-6/Omega type K	28534	11/2/11	6/1/11, 6/12/11 & 6/14/11
Dual Element Temperature Display	FT350/Dickson	10217201/28535	11/1/11	6/1/11, 6/12/11 & 6/14/11

Procedure Used:

RPL-COLLOID-02, Rev. 2, Measurement *of Physical and Rheological Properties of Solutions Slurries and Sludges*

RESULTS

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.

Note: All concerns identified in the review were addressed in a comment resolution meeting on 2 August 2011. Lisa Middleton, Deborah Coffey, Richard Daniel and Carolyn Burns attended.

Before the completed TI was signed off for QE review, it was determined that all comments/concerns were addressed. There were no outstanding identified findings or observations.

It was noted that there is an entry in the TI, step 3.1, dated before the TI was signed off for approval. The step, a performance check, was performed early due to the urgency of completing the work.

Issues of concern are summarized below:

1. Page 10, Table 6: Need to get initials & signature from Richard Daniel. Completed, no further requirements. Closed.

2. Page 11, ID 1: Add balance model # for Balance 1 (AV4101C). Added, no further action required. Closed

3. Add page #s to the attachments following page 17 (17A, 17B, 17C &17 D). Completed, no further requirements. Closed

4. Page 19: Closing date is listed as 02/22/11; however, analysis was done on 6/1/11, 6/12/11 & 6/14/11. This doesn't make sense. The date of 2/22/11 refers to when performance testing was done at APEL using the cold system, not in SAL. Opening was performed in SAL on 6/1/11; closing on 6/22/11. No further action required. Closed.

5. Add page #s to the attachments following page 19 (19A, 19B, 19C 19D & 19E). Completed, no further requirements. Closed

6. Page 21: A BPCL was not provided for balance #2 (Sartorius MSG234 40023610/WD68444) used on 6/10/11. A completed BPCL was obtained for that time frame; however, a performance check was not performed on 6/10/11. This balance was actually used on 6/7/11. An acceptable performance check was performed. No further action required. Closed.

7. Page 21: A BPCL was not provided for balance #1 (Ohaus AV4101C 8031221019) used on 6/10/11. A completed BPCL was obtained for that time frame; however, a performance check was not performed on 6/10/11. This balance was actually used on 6/1/11. An acceptable performance check was performed. No further action required. Closed.

8. Page 21: The entry for "Balance ID" doesn't make sense. It looks to me like "12#2". Should it be "#1 & #2"? It should be #1 & #2. Corrected, no further action required. Closed.

9. Excel file *TI039 – 001 (Reviewed),* Summary Tab, cell C6: Incorrect time listed. Should be 15:00 not 13:00. See page 17 of TI. Corrected, no further action required. Closed.

10. Excel file *TI039 – 001 (Reviewed),* PCheck-Open Tab: Thermocouple Display ID & expiration date listed differs from the M&TE listed in TI. Shouldn't it be 28535 exp. 11/1/11 instead of 28537 exp. 11/2/11? Corrected, no further action required. Closed.

11. Excel file *TI039 – 001 (Reviewed),* PCheck-Open Tab: Expiration date of viscosity standard listed differs from TI. TI says 4/27/12; spreadsheet says 2/5/12? There are 2 different standards, one for SAL & one for APEL. Spreadsheet was modified to show the dates of the 2 different standards (4/27/12 & 5/2/12). No further action required. Closed.

12. Excel file *TI039 – 001 (Reviewed),* PCheck-Close Tab: Thermocouple Display expiration date listed differs from the M&TE listed in TI. Shouldn't it be 11/1/11 instead of 11/2/11? Corrected, no further action required. Closed.

13. Excel file *TI039 – 001 (Reviewed),* PCheck-Close Tab: Expiration date of viscosity standard listed differs from TI. TI says 4/27/12; spreadsheet says 2/5/12? There are 2 different standards, one for SAL & one for APEL. Spreadsheet was modified to show the dates of the 2 different standards (4/27/12 & 5/2/12). No further action required. Closed.

PROJECT 52578 – SURVEILLANCE REPORT

Number: SR-52578-2011-035

CORRECTIVE ACTION

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

Surveillance Performed By:

Lisa Middleton, Quality Engineer 8-3-11 Date: 8/2/11

DISTRIBUTION: Carolyn Burns, Clark Carlson, Deborah Coffey, Richard Daniel, Sandy Fiskum, Matt Fountain, Rick Shimskey, and Andy Schmidt

Appendix I

Particle Size Distribution

Appendix I

Particle Size Distribution

Appendix I contains the particle size distribution characterization report for the KW210 container composite −500 μm size fraction samples and supporting documentation and calculations. The supporting documentation includes data manipulation, calculations, graphing sheets, performance verification standard certificates, and Surveillance Reports.

The following table of contents shows that pagination specific to Appendix I was applied to the particle size distribution characterization report and supporting attachments. This pagination is sequential and consistent (e.g., page I.#).

Appendix I Table of Contents

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Battelle—Pacific Northwest National Laboratory
Radiochemical Sciences and Engineering Group
PO Box 999, Richland, Washington 99352

Project: #52578

Particle Size Analysis Performed Under Test Instruction 52578-TI-037, *Particle Size Distribution Measurements for K West Basin Consolidated Container Composite SCS-CON-210, Using Minus 500 Micron Particle Size Fraction Samples*

Sample ID's: TI008-SP and TI008-SQ

Procedure: RPL-COLLOID-01, Rev. 1, *Particle Size Analysis Using Malvern MS2000*

Analyst: Carolyn Burns
Analysis Date: 06/15/2011
Instrument: Malvern Mastersizer 2000, SN# MAL 100406, Hydro μP Dispersion Unit, RPL Room 302

Supporting Electronic Report File Name: "52578-TI037 (Reviewed).xlsm"
File location: SharePoint site under TI-037 folder

08/03/2011
Prepared By C A Burns/Date

8/3/2011
Reviewed By R C Daniel/Date

8/3/11
QE Reviewed By DS Coffee/Date
DSC 8-3-11

8/3/11
Project Management Release/Date

1 Introduction

The sludge from the K Basin Pits and Floors (referred to as Container Sludge) has been consolidated into five engineered containers located under water in the K-West (KW) Basin. The container sludge originating from the K-East (KE) Basin resides in containers SCS-CON-240, -CON-250, and -CON-260. The container sludge originating from the KW Basin resides in SCS-CON-210 and -CON-220.

Two to four top-to-bottom core samples have been collected from each container and shipped to RPL. Previous TIs have been implemented to reconstitute the sludge in a given core into a core composite sample. Sub-samples from each core composite within one Engineered Container have been consolidated into a single sample in an effort to represent the entire container (called a Container Composite). The Container Composite has been wet-sieved into +2000 micron, -2000/+500 micron, and -500 micron particle size fractions. Subsamples of the -500 micron fraction have been collected to support particle size distribution (PSD) characterization of this material. The subsamples submitted for particle size analysis were suspended in DI water and removed from the hot cell. In 2009, all of the samples were analyzed with the exception of SCS-CON-210.

This data package summarizes the test approach and results of particle size analysis performed under Test Instruction 52578-TI-037*, Particle Size Distribution Measurements for K West Basin Consolidated Container Composite SCS-CON-210, Using Minus 500 Micron Particle Size Fraction Samples.* Particle size analyses were performed on a primary sample (Sample ID TI008-SP) and a duplicate sample (Sample ID TI008-SQ) from the KW210 container composite generated under 52578-TI008. Figure 1 shows the complete scope of characterization work being conducted on the Container Composite samples. The extent of this data package encompasses the analysis of PSD for the -500 micron sieve fraction of K-Basin sludge from Container Composite sample KW210. The work outlined in this data package is shaded in green in Figure 1.

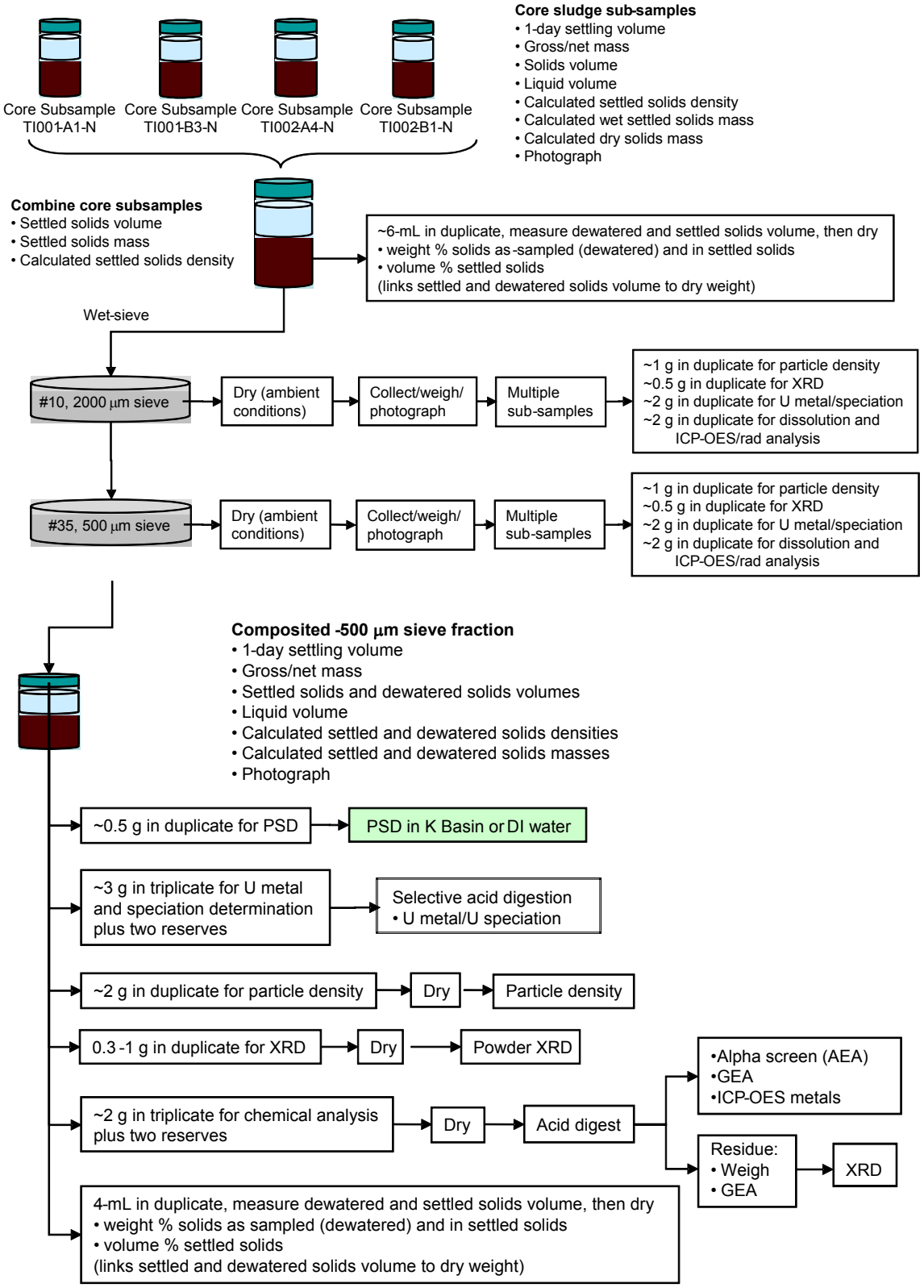


Figure 1. Overall Processing Schematic of Container Composite.
PSD Characterization Scope is Highlighted in Green.

2 Background

PSD describes the size fractionation of solid species in a given powder, dispersion, or slurry sample. PSD is typically described by either cumulative or differential population fraction versus a given particle size indicator. For example, the size distribution of particles in a slurry are often described using a histogram expressing the differential volume of particles falling between two equivalent sphere diameters over a large array of equivalent sphere diameters. PSD measurements can be accomplished using a number of approaches, such as settling experiments, microscopic imaging, and light obscuration and scattering.

The particle size measurements discussed herein are carried out on a Malvern Mastersizer 2000 particle size analyzer (Malvern Instruments, Inc., Southborough, MA). This instrument operates using the principle of laser diffraction (see Figure 2). Here, a monochromatic laser (red and/or blue) is directed through a transparent cell containing a dilute dispersion of the solid particles being analyzed. On the opposite side of the flow cell is a series of ring detectors capable of detecting the intensity of laser light at various scattering angles. If the laser does not strike a particle in the flow cell, it simply passes through the cell undisturbed and strikes the central detector. When the laser interacts with a particle, it is scattered at various angles. The scattered light is picked up across a number of rings of the detector, creating a unique "diffraction pattern" that can be mapped as a function of diffracted light intensity versus ring detector position. Prolonged observation of the light scattered from the dispersion allows a near complete sampling of the particle species contained therein (assuming all particles are uniformly dispersed). Comparison of the time-averaged diffraction signal against a "clean" reference cell signal generates a diffraction pattern unique to the test dispersion. Given the optical properties of the particulate and dispersing phases, mathematical analysis of the averaged "diffraction pattern" allows determination of size fractionation species contained in the dispersion.

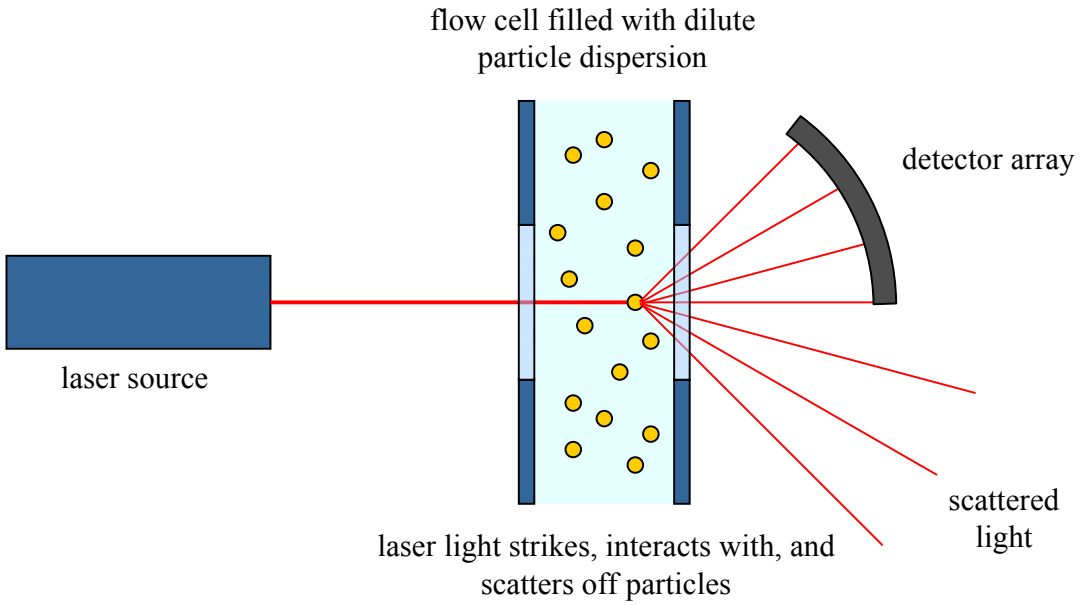


Figure 2. Schematic of a typical laser diffraction particle size analyzer.

It is important to recognize that particle size measurements by laser diffraction are intended to capture the size of a single, well-dispersed particle species. This "true" PSD captures all particles in the solid

dispersion in a fully suspended and un-agglomerated primary particle state. Full dispersion at the primary particle level requires the correct selection of suspending phase chemistry, which is often modified through the use of dispersing agents or surfactants, and sufficient flow to suspend all particles during analysis. Complete suspension of all particle species requires sufficient mixing energy.

When dealing with complex dispersions, which contain multiple particle species and a broad distribution of sizes, finding the correct dispersing medium and measurement conditions may be difficult (if not impossible), as individual particle species in the solids mixture may have contradictory suspending phase chemistry requirements. As such, particle size analysis of complex solids dispersions is generally performed to determine the "apparent" PSD as a function of processing conditions such as flow rate and sonication and suspending phase chemistry such as pH. The apparent PSD differs from the true PSD in two ways: 1) particle agglomerates exist and are treated as single particle species and 2) not all particles may be suspended at the flow conditions selected. Despite these short comings, apparent PSDs provide useful information about how the PSD of the test dispersion exists in the process from which it is derived and can highlight potential difficulties in suspending large/dense particles.

3 Samples

Samples for particle size analysis were collected under Test Instructions 52578-TI-008.

Table 1. Summary of Samples Provided for Particle Size Analysis.

Sample ID	Sample Purpose	Vessel Description	Sample Size (wet sludge basis)
TI008-SP	PSD	~20-mL glass vial	0.5 g
TI008-SQ	PSD duplicate	~20-mL glass vial	0.5 g

4 Instrumentation

Particle size characterization was accomplished using a Mastersizer 2000 (Malvern Instruments, Inc., Southborough, MA 01772 USA) with a Hydro μP wet dispersion accessory. The Mastersizer has a nominal size measurement range of 0.02-2000 μm. The actual range is dependent on the accessory used as well as the properties of the solids being analyzed. When coupled with the Hydro μP wet dispersion accessory, the nominal measuring range is reduced to 0.02-150 μm (also dependent on material density). Although particle sizes above 150 μm can be observed with the Hydro μP, reliable determination of their volume contribution is a challenge for the instrument and is highly dependent on the material density and size. Previous PSD determinations indicate that articles much larger than ~600 μm may "jam" or become stuck in the Hydro μP dispersion unit impeller, preventing their circulation to the flow cell observation window. Given that the container composite samples have been passed through a 500 μm sieve, it was anticipated that there could be a remainder of large (~500 μm) particles that would not be able to pass through the dispersion unit and hence would not be accounted for in the PSD measurements obtained.

The Hydro μP wet dispersion accessory consists of a 20 mL sample flow cell with a continuously variable and independent pump and ultrasound. Both flow and sonication can be controlled and changed during measurement. As such, PSD measurements can be made before, during, and after sonication, allowing determination of the influence of each on the sample's PSD. The primary measurement functions of the Malvern analyzer are controlled through computer software. For the current measurements, Mastersizer 2000 software, Version 5.60 [Malvern Instruments, Ltd. Copyright

© 1998-2009] was employed. Table 2 provides a summary of basic information regarding the analyzer and accessory located in Room 302 in the Radiological Processing Laboratory (RPL).

Table 2. Summary of Malvern Mastersizer 2000 Instrument Information.

Analyzer:	Mastersizer 2000
Measurement principle:	Laser Diffraction (Mie Scattering)
Analyzer Accessory:	Hydro μP
Serial Number:	MAL 100406
Measurement Range:	0.02-2000 μm nominal (0.02-150 μm with accessory)
Type:	Flow cell system with continuously variable and independent pump and ultrasound.
Capacity:	20 mL
Pump Speed Range:	0-5000 RPM (variable)
Ultrasound Power	0-20 W (variable)
Software Version/date	5.60/1998-2009

5 Test Approach

Particle size distribution was determined according to the procedure RPL-COLLOID-01, Rev. 1, *Particle Size Analysis Using Malvern MS2000*. Before conducting particle-size measurements with KW210 samples, the PSD of a particle size standard traceable to the National Institute of Standards and Technology (NIST) was measured to verify instrument performance. The standard consisted of polydispersed silica particles with diameters falling primarily between 15-150 μm. The results from this measurement were compared to the standard's certificate of analysis provided by the supplier to determine if the performance check indicated acceptable instrument performance (as dictated by RPL-COLLOID-01, Rev. 1). After completion of all PSD analyses associated with this test report, a closing performance check of the particle size analyzer was conducted.

For the PSD work described in this report, it was expected that the PSD measurements performed at initial recirculation times, without sonication, would be the most relevant PSD measurements for the sludge treatment project with respect to the current plans to load the sludge in the sludge transport and storage containers. These initial measurements (which correspond to a relatively unsheared slurry) would likely best reflect the expected PSD of the sludge particles in a settle/decant/filtration processing operation.

With this in mind, the following test approach was implemented to study and mitigate potential issues as described below:

- Subsampling – Samples were concentrated and required dilution to obtain a suitable sample for measurement. Efforts were made to withdraw sub-samples that best represent the submitted sample fraction.
- Quantity of Sample Material - when measuring the PSD of test material, the typical approach is to disperse (dilute) small aliquots of the concentrated (i.e., as-received) material in a suitable solvent (such as DI water or the particle suspending phase) contained within a variable-speed recirculator (Hydro μP dispersion unit). The sample quantity requirements are determined by monitoring the amount of light scattered by the diluted particle dispersion—referred to as obscuration. Sufficient sample material to yield obscuration values of 5 to 20% are generally considered acceptable for size measurements, which corresponds to sample sizes in the range of 0.1 g (for fine/less-dense particles) to 2 g for larger/more-dense particles.

- The test approach employed for the concentrated K-Basin PSD samples was as follows. K-Basin water was provided to use as the suspending phase for the analysis of these sample. The water was filtered through a 0.45 μm syringe filter to remove particulate matter. First, the flow cell was filled with the suspending phase (filtered K-Basin water) and "as-received" test material was added drop wise until an acceptable obscuration was reached. For sample TI008-SP one drop of the as received material resulted in an obscuration of 16-20%. For materials containing fine particles, obscuration values of the range 5-10 % are more desirable to minimize the possibility of multiple scattering, which can bias the PSD if present. In contrast, one drop of the duplicate sample, TI008-SQ, resulted in an obscuration in the 5-10% range. Side-by-side comparison of the two PSD sample vials found that the sample volume of TI008-SP was approximately half that of sample TI008-SQ. It was suspected that TI008-SP and may have lost some water due to evaporation resulting in the higher obscuration. Approximately 0.5 mL of filtered KW water was added to sample TI008-SP to obtain an obscuration value similar to that for sample TI008-SQ from one drop.

- Number of PSD Measurements at Each Condition - PSD characterizations at a given set of test conditions consisted of an average of three individual PSD measurements (with each measurement consisting of a 10 second red light observation and a 10 second blue light observation) for two sub-aliquots per sample. Data obtained for each of the sub-aliquots was averaged to obtain one PSD for each measuring condition per sample, i.e. at 1 minute recirculation (unsonicated), 100 % sonication (20 watts) and post sonication.

Table 3 provides the matrix of conditions for each of the PSD samples listed in Table 1. The initial measurement conditions at 1 and 3 minute recirculation times are based on results obtained for the parameter evaluation study performed under Test Instruction, 52578-TI017. An additional data measurement set was collected (a post-sonicated measurement after 5 minutes of total recirculation) to further evaluate the effect of sonication on the measured PSD for the diluted samples.

Table 3. Modified Condition Matrix for PSD Determinations

Recirculation Rate, RPM[(a)]	**Recirculation Time, min[(a)]**	**Sonication[(a,b)] power, time**
2000	1	Pre-sonication
2000	3	120 sec/100 %
2000	5	Post-sonication
Other Measures/Conditions		
Number of PSD measurements/conditions: Each measurement consists of three separate observations (with each observation consisting of a 10 second red light measurement and a 10 second blue light measurement).		
(a) The two initial measurement conditions for Recirculation RPM, recirculation time and sonication power and time are based on results of the parameter evaluation in Test Instruction 52578-TI017. A third measurement condition (at 5 minutes recirculation) was added to further evaluate the impacts of sonication on the measured PSD. (b) The Hydro μP dispersion cell employed for particle size analysis is equipped with variable power sonicator with an ultrasound power of up to 20W (at an instrument sonicator setting of 100%).		

Instrument Performance Check

As required by the procedure RPL-COLLOID-01, Rev. 1, the performance of the Malvern analyzer must be verified at the beginning of each series of analyses (with the period between performance

checks not to exceed 90 days during use). Checks are performed using particle size standards traceable to the National Institute of Standards and Technology (NIST). Checks verify that the particle size analyzer can measure a PSD standard's d(50), the 50% volume/weight fractile and mean particle size, to within 10% of the value specified on the manufacturer's certificate of analysis.

For the Mastersizer 2000 system employed for measurements (MAL100406 at RPL), two performance checks were conducted: 1) an opening performance check before the start of any PSD measurements on 06/15/11 and 2) a closing performance check at the end of measurements on the same day, 06/15/11. Both performance checks employed a Malvern Quality Audit Standard (QAS) QAS3004 supplied by Malvern Instruments, Inc. (Southborough, MA 01772 USA). The standard is traceable back to its certificate of analysis through a unique bottle number identifier.

The particle size standard was supplied as 0.10 g single shot of dry powder. Before measuring the performance check standard, the Hydro μP dispersion unit was filled with DI water. Next, the dry powder shot was added directly to the Hydro μP dispersion unit. A continuous pump speed of 2000 RPM was set to mix the flow cell contents and, after adequate mixing time, the PSD of the resulting dispersion was measured. The performance check size analysis employed a particle refractive index and absorption of 1.52 and 0, respectively, and a suspending phase particle refractive index of 1.33 (for water).

The results of both the opening and closing performance checks are provided in Table 4. An average of three 2000 RPM measurements of the PSD indicated a d(50) of 63.112 μm and 61.997 μm for opening and closing performance checks, respectively, for the MS2000 particle size analyzer situated in RPL. These results deviate less than 2% from the d(50) listed on the standard's certificate of analysis from Malvern Instruments, Inc. In accordance with RPL-COLLOID-01, Rev. 1, an acceptable performance check is defined as: the d(50) value for the standard is within 10% of the certified reference value.

Table 4. Performance check results for opening, and closing checks covering the period of measurement for PSD measurements for both instruments used for sample analysis under this test instruction.

Size Range:	15 to 150 μm		
Catalogue #:	QAS3004		
Bottle #	02/03858 (opening check), 02/03980 (closing check)		
Weight:	0.1 grams		
Opening Check 06/15/11			
PSD Percentile	List	Measured (μm)[a), c)]	E_r (%)[b)]
d(50):	62.362	63.112	1.20 %
Closing Check 06/15/11			
PSD Percentile	List	Measured (μm)[a), c)]	E_r (%) [b)]
d(50):	62.362	61.997	-0.59%

(a) As measured for the period of performance applicable for this report.
(b) Calculated before rounding of significant figures in list and measured percentiles. Defined as: $E_r(\%) = (X_{measured}-X_{certified})/ X_{certified} * 100$
(c) Average of triplicate measurements reported here as per the standard protocol of the QAS supplier.

Particle Size Characterization

All PSD measurements were performed with diluted "as-received" material with 1 drop of original sample diluted in a filled Hydro μP flow cell to achieve target test concentrations. Before each sampling event for PSD, the analyzer was drained, flushed with 20 mL of deionized (DI) water at least three times, filled with filtered K-Basin water, and brought into a measurement ready state. The analyst then examined the test sample to determine the best sampling approach. The dispersion unit flow cell was fully filled with filtered K-Basin water in preparation of sample introduction. Next, the sample was introduced to the Malvern dispersion unit and analyzed as follows:

1. The analyzer flow cell pump was set to the initial circulation rate of 2000 RPM (see Table 3).
2. The sample was prepared for analysis by re-suspending the settled solids. This was accomplished by initially agitating the sample vial to re-suspend the settled solids. The sample was then repeatedly pulsed with a 1 mL disposable plastic pipette until the contents were uniformly dispersed and representatively sub-sampled in the pipette. Each pulse involved drawing off a fraction of the sample into the pipette and immediately jetting the drawn liquid back into the sample vial.
3. Immediately after re-suspension, one drop of sample was introduced into the instrument (while the pump was active). For the current analyses, an obscuration of 5-20% was targeted. The time at which the sample introduction was complete was noted and was associated with the start of sample recirculation.
4. The material (sample) and suspending phase optical properties were set in the analyzer software. The standard optical properties employed were an RI of 2.42 and an absorption index of 1.
5. The sample PSD was measured under the conditions outlined in the sample test matrix (see Table 3).

For each test condition outlined in Table 3, the dispersion PSD was measured three times, with each measurement consisting of a 10 second red light observation and a 10 second blue light observation. An average of the 3 measurements was calculated, yielding one average PSD for each test condition per sample aliquot measured. Condition set points (i.e., pump speed and sonication) were changed at the specified recirculation times. Recirculation times were calculated as the difference between the current measurement time and the time sample dispersion was completed. PSD measurements were started at recirculation times of 1, 3, and 5 minutes

Analysis of the raw particle size data is performed automatically by the Mastersizer software immediately after each measurement. Analysis calculates the particle size distribution based on the scattered light intensity as a function of detection angle and the particle size model selected (e.g., single narrow, multiple narrow, or broad distributions). As indicated in the test steps outlined in the preceding paragraphs, the optical properties such as the refractive index (RI) of the sample and suspending phase must be entered into analyzer at the time of measurement. Table 5 outlines the optical properties employed in the current analysis. Selection of these optical properties is based on the results of the parameter evaluation study performed under Test Instruction, 52578-TI017.

Table 5. Default optical properties employed for PSD analysis of KW210 container composites. These properties were assessed through post-measurement reanalysis of KW240 (TI010-SP) data collected during the initial parameter evaluation study.

Property	Values Employed
Refractive Index (RI)	2.42
Absorption Index	1
Particle Shape	Irregular
Model	General Purpose (Broad Distribution)

6 Quality Assurance

The quality assurance requirements defined in the QAPjP/SAP invoked Hanford Analytical Services Quality Assurance Requirements Document (HASQARD), Rev. 3 Volume 1 and 4 and balance calibration verification controls. These requirements have been implemented for this work as defined in this test instruction and are summarized in Table 6.

Table 6. QA requirements and implementation.

QA Requirement	Implementation
Controlling sample identification	Samples generated during the execution of this TI will be uniquely identified according to instruction in this TI.
Container selection	Containers were made of glass, consistent with the intended storage and follow-on processing.
Preservation	Sample preservation is not applicable for current scope.
Sample chain of custody (COC)	Internal COC is not required. Sample custody is maintained during the sample preparation phase and any extra material is archived in case the remainder is needed for reanalysis. 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 processing steps are documented in TIs and on bench sheets. Processed sample fractions are also uniquely identified.
Sample storage	There are no sample storage requirements for PSD samples; they are stored at room temperature.
Sample handling and transfer	Handling and transfers was conducted in a manner to minimize potential for contamination (clean containers and sampling devices), minimize loss (use secondary containment during process operations—where practical), and minimize tendency for sample to dry out (except where drying is required) where the wetting agent is deionized (DI) water.
Holding time limits	Holding time limits are not applicable to PSD measurement.
Waste disposition	Waste was processed through the normal waste operations protocols and procedures.
Preparing required quality control (QC) samples	The primary sample and duplicate were generated from independent replicate tests, and are not considered true analytical duplicates. Therefore duplicate precision criteria, (i.e., ± 20%) for the PSD was not applied to these samples.
Balance checks	NA; balances are not typically used in PSD measurements.

7 Results

Particle size distribution measurements for samples derived from KW210 container composite sample are presented below. Table 7 summarizes the cumulative percent at 1 minute of recirculation (unsonicated) and with sonication applied for the primary and duplicate samples, TI008-SP and TI008-SQ, respectively, investigated in this study. The cumulative % undersize data given in Table 7 has been plotted along with the volume contribution % PSD and are shown in Figures 3 through 6. The post sonicated data collected have been included on Figures 3 through 6. Only the 1 minute recirculation time and the sonicated data have been included in Table 7; this is consistent with the work scope of this TI and previous reports. Post sonication data were collected as a precautionary measure, given the sample dilutions required to obtain a suitable sample obscuration. Dilute samples sometimes form bubbles and or agglomerate as a result of sonication. Neither was observed for either sample analyzed. The data obtained at 1 minute recirculation time are more representative of the suspended particles present during settling of actual sludge material while the sonicated data better represents the "true" PSD of the fully suspended sample particles. The average particle size distribution of the container composite KW210 – 500 µm size fraction is given in Table 8 and Figures 7 and 8, again only the unsonicated and sonicated data is given in Tables 7 and 8.

For all sample aliquots analyzed, grinding/knocking sounds could be heard inside the dispersion unit; this noise is indicative of large particles getting caught-up in and knocking on the internal parts of the dispersion. This would suggest that there were particles present in both samples provided for analysis with at least one linear dimension greater than 500 µm. Sieving through a 500 µm sieve does not always eliminate elongated particles with one dimension greater than 500 µm. If large particles are held up in the dispersion components, they may not flow into the observation cell and be measured by the instrument. Additionally, hold-up of large particles may block flow of smaller particles, preventing their observation. The grinding/knocking sound was not heard once the instrument was flushed, cleaned and filled with K-Basin water for the background measurement, which suggests that the large particles did not accumulate in the system and effect subsequent measurements.

A summary of a select number of percentiles is given in Table 9; these data provide a brief overview of the particle sizes present, in particular the tails of the PSD (upper and lower limits).

Table 7. Cumulative Percent Undersize measured at 1 minute recirculation time (unsonicated) and during sonication at 3 minutes recirculation time.

Size, μm	[a]TI-008-SP		[a]TI-008-SQ	
	Unsonicated	100 % Sonication	Unsonicated	100 % Sonication
0.05	0	0	0	0
0.5	0.28	0.64	0.00	0.32
1	1.01	2.45	0.34	1.35
2.5	4.51	7.53	2.06	4.12
5	10.2	13.0	4.66	7.07
10	16.1	19.1	7.31	10.5
20	20.8	25.3	9.56	14.3
40	24.9	31.8	11.7	18.8
60	27.8	36.1	13.8	22.4
80	30.1	38.9	15.5	24.9
100	32.0	40.6	16.7	26.4
125	34.0	42.1	18.0	27.5
150	36.1	43.4	19.3	28.5
175	38.5	45.0	21.3	30.1
200	41.4	47.1	24.1	32.3
300	56.7	60.2	41.5	47.8
400	72.7	75.1	61.4	66.2
500	85.2	87.0	77.6	81.1
600	93.4	94.7	88.5	91.0
700	98.0	98.7	95.1	96.7
800	99.9	100	98.5	99.4
1000	100	100	100	100

a) Based on the average of 6 PSD measurements generated from 2 aliquots.

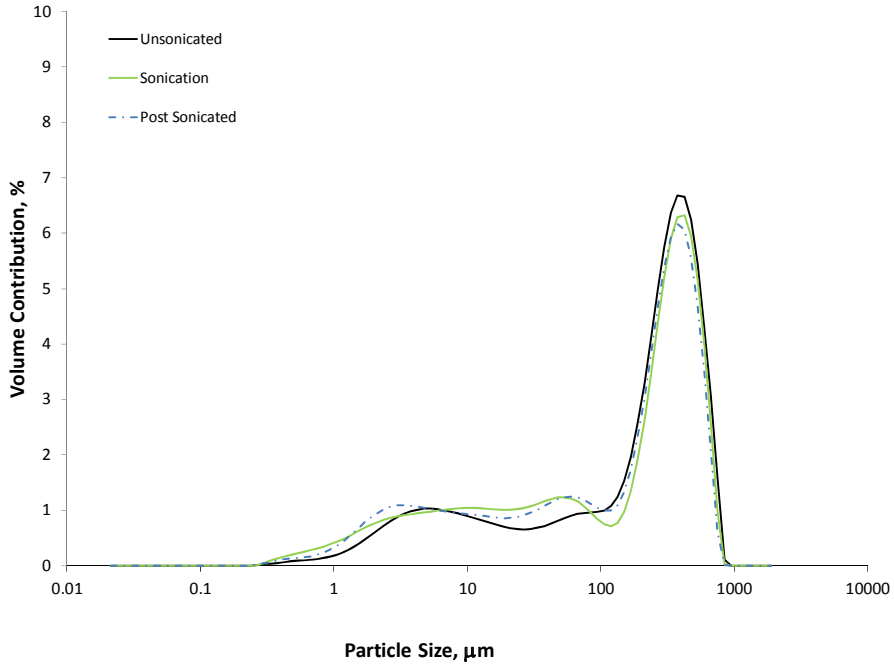


Figure 3. Average volume particle size distribution measured at 1 minute recirculation, 100 % sonication and post sonication for sample TI008-SP. Based on the average of 6 PSD measurements generated from 2 aliquots.

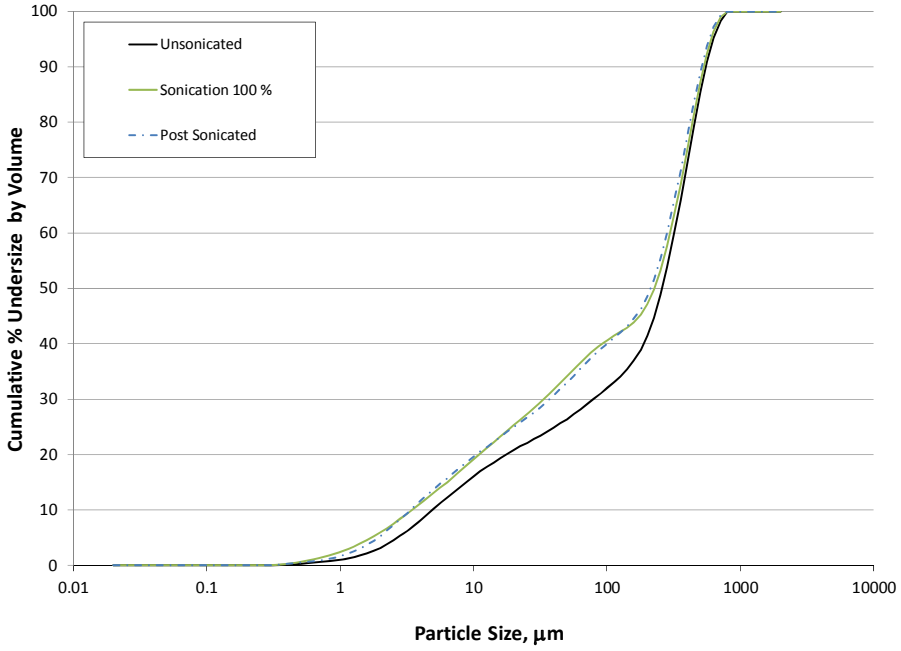


Figure 4. Average Cumulative % undersize plot measured at 1 minute recirculation, 100 % sonication and post sonication for sample TI008-SP. Based on the average of 6 PSD measurements generated from 2 aliquots.

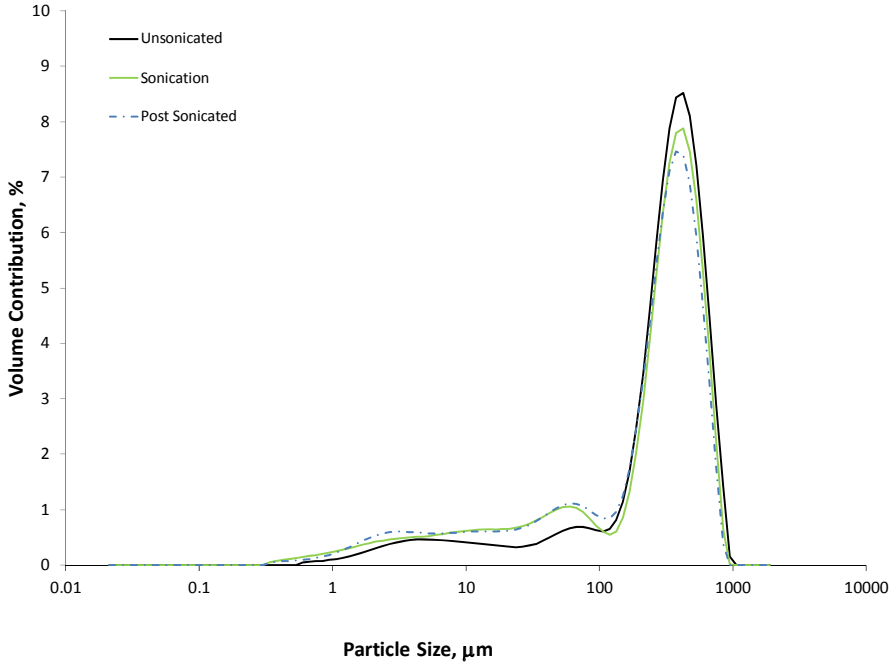


Figure 5. Average volume particle size distribution measured at 1 minute recirculation, 100 % sonication, and post sonication for sample TI008-SQ. Based on the average of 6 PSD measurements generated from 2 aliquots.

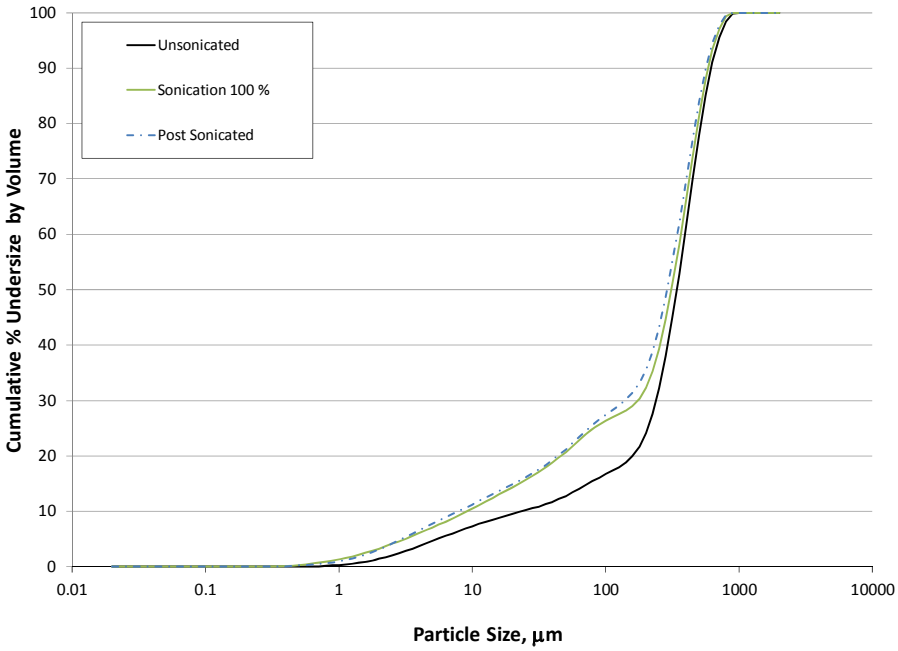


Figure 6. Average Cumulative % undersize plot measured at 1 minute recirculation and 100% sonication for sample TI008-SQ. Based on the average of 6 PSD measurements generated from 2 aliquots.

Table 8. KW210 SCC Average Cumulative Percent Undersize measured at 1 minute recirculation time (unsonicated) and during sonication at 3 minutes recirculation time.

Size, µm	[a]KW210 SCC	
	Unsonicated	100 % Sonication
0.05	0	0
0.5	0.14	0.48
1	0.68	1.90
2.5	3.28	5.83
5	7.41	10.1
10	11.7	14.8
20	15.2	19.8
40	18.3	25.3
60	20.8	29.2
80	22.8	31.9
100	24.4	33.5
125	26.0	34.8
150	27.7	35.9
175	29.9	37.5
200	32.8	39.7
300	49.1	54.0
400	67.1	70.6
500	81.4	84.0
600	90.9	92.9
700	96.5	97.7
800	99.2	99.7
1000	100	100

a) Based on the average of 12 PSD measurements generated from 4 aliquots.

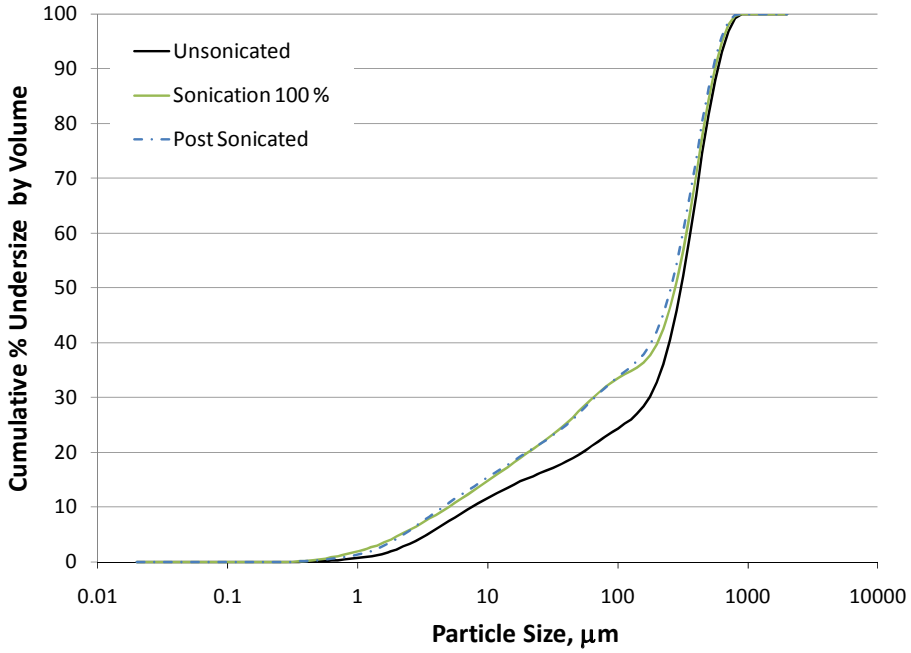


Figure 7. Average Cumulative Volume % Undersize (Less Than 500 µm Size Fraction) Plot of KW210-SCC, based on the average of 12 measurements generated from four aliquots, two aliquots from sample TI008-SP (primary) and two aliquots from TI008-SQ (duplicate)

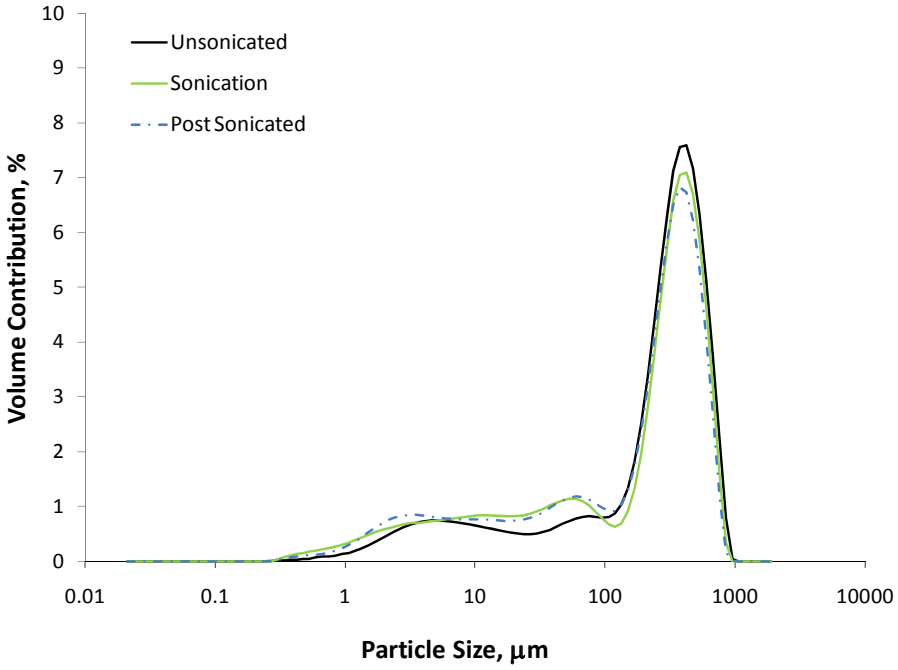


Figure 8. Average Volume % PSD of Sample KW210-SCC (Less Than 500 µm Size Fraction) Plot of KW210-SCC, based on the average of 12 measurements generated from four aliquots, two aliquots from sample TI008-SP (primary) and two aliquots from TI008-SQ (duplicate)

Table 9. Summary table of selected percentile values for measurements taken at 1 minute recirculation time (unsonicated) and during sonication at 3 minutes recirculation time.

	[(a)]TI008-SP		[(a)]TI008-SQ		KW210 SCC Average	
Percentiles	**Unsonicated**	**100 % Sonication**	**Unsonicated**	**100 % Sonication**	**Unsonicated**	**100 % Sonication**
d(0.01)	0.99	0.60	1.65	0.83	1.24	0.68
d(0.05)	2.69	1.72	5.45	3.10	3.42	2.15
d(0.10)	4.92	3.45	23.3	9.12	7.52	4.96
d(0.50)	259	227	342	312	305	276
d(0.90)	552	533	619	587	588	562
d(0.95)	628	605	699	663	666	636
d(0.99)	737	711	823	776	786	750

a) Based on the average of 6 PSD measurements generated from 2 aliquots.
b) Based on the averaged of 12 PSD measurements generated from 4 aliquots, two aliquots from TI008-SP (primary) and two aliquots from TI008-SQ (duplicate).

8 References

RPL-COLLOID-01, Rev. 1, *Particle Size Analysis Using Malvern MS2000.*

KBC-33786, Rev. 2, December 2009; Quality Assurance Project Plan/Sampling and Analysis Plan (QAPjP/SAP): Quality Assurance Project Plan/Sampling and Analysis Plan for Containerized KW Settler Sludge

CH2M Hill Plateau Remediation, Contract 41108, Statement of Work, *Technical Support for Planning, Preparing, and Analyzing KW Settler Sludge* (February 11, 2010).

Data Quality Objectives for Sampling and Analysis of K Basin Sludge HNF-36985, Rev. 3, July 2009

52578-TI037-PSD Data Package - Coverpage

Description:
This Excel file data package documents measurement results (using Microsoft® Office Excel) for particle size distribution (PSD) data collected under Test Instruction (TI) 52578-TI-037, Particle Size Distribution Measurements for K West Basin Consolidated Container Composite SCS-CON-210, Using Minus 500 Micron Particle Size Fraction Samples. Results for samples TI008-SP and TI008-SQ are given.

Computer Employed:
MHP Dell Latitude D830 Laptop Computer (Property # WE14110)

Operating Environment:
Microsoft Windows 7 Enterprise

Base Software Program:
Microsoft Office Professional Plus 2010 Excel Version 14.0.5128.5000 (32-bit)

Package Contents:
Package consists of this coverpage, two calculation review pages, print-outs for two Microsoft® Office Excel workbooks, and all other relevant documentation.

Description	File Date	File Size	Number of Pages
Data package coverpage	n/a	n/a	1
Calculation review worksheet	n/a	n/a	1
Supplemental calculation review worksheet	n/a	n/a	1
Excel printouts: "52578-TI037 (Reviewed).xlsm"	7/7/2011 19:55	100 KB	29
Excel printouts: "TI008 averaged data.xlsm"	8/4/2011 15:48	73 KB	21
Standard certificate: "Malvern Standard - QAS3004.pdf"	5/3/2011 15:04	480 KB	2
K-Basin Surveillance Report - SR-52578-2011-030 (Rev. 0)	n/a	n/a	3
K-Basin Surveillance Report - SR-52578-2011-030 Rev. 1	n/a	n/a	4

Package Generator Signature:

Generator: Carolyn Burns

08/04/2011
Date

Calculation Review Sheet

Reviewer Name: Richard C. Daniel
Reviewer Title: S/E
Review Date: 7/7/2011

Title of Spreadsheet Calculation Reviewed: One spreadsheet and a calibration certificate are included in this review package:
1) "52578-TI037 (Reviewed).xlsm"
2) "Malvern Standard - QAS3004.pdf"

Revision Number: n/a
Date Prepared: 7/7/2011

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

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

CHECK LIST

Spreadsheet/Calculation Identification	**Yes**	**No**	**NA**
Spreadsheet/Calculation Title:			X
Revision Number:	X		
Date Prepared	X		
Prepared by:	X		
General Statement of Purpose:	X		
General Description of Approach (note: specific approaches may be described for each section/subsection of spreadsheet/calculation)			X

Comments: Standard PSD spreadsheet, approach per RPL-COLLOID-01, Rev. 1

Assumptions	**Yes**	**No**	**NA**
Are assumptions clearly stated?	X		
Are assumptions supported/justified?	X		
Are assumptions reasonable?	X		

Comments: No 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		

Comments: No comments

Equations/Approach	**Yes**	**No**	**NA**
Are equations adequately defined?	X		
Are equations properly referenced?			X
Are limitations of approach/equations identified?			X
Are equations appropriate?	X		
Are units consistent?	X		

Comments: No comments

Results/Conclusions	**Yes**	**No**	**NA**
Are formulas consistent in spreadsheet cells?	X		
Are calculations correct?	X		
Are conclusions consistent with results?			X
Are conclusions consistent with applicable limits?			X

Comments: No comments

Reviewer Sign/Date: 7/7/11
Richard C. Daniel

Calculation Review Sheet

Reviewer Name:	Richard C. Daniel
Reviewer Title:	S/E
Review Date:	8/1/2011
Title of Spreadsheet Calculation Reviewed:	This review covers the following supplemental files for the 52578-TI037-PSD data package "TI008 averaged data.xlsm"
Revision Number:	n/a
Date Prepared:	8/1/2011

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

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

CHECK LIST

Spreadsheet/Calculation Identification	**Yes**	**No**	**NA**
Spreadsheet/Calculation Title:			X
Revision Number:	X		
Date Prepared	X		
Prepared by:	X		
General Statement of Purpose:	X		
General Description of Approach (note: specific approaches may be described for each section/subsection of spreadsheet/calculation)			X

Comments: Standard PSD spreadsheet, approach per RPL-COLLOID-01, Rev. 1

Assumptions	**Yes**	**No**	**NA**
Are assumptions clearly stated?	X		
Are assumptions supported/justified?	X		
Are assumptions reasonable?	X		

Comments: No 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		

Comments: No comments

Equations/Approach	**Yes**	**No**	**NA**
Are equations adequately defined?	X		
Are equations properly referenced?			X
Are limitations of approach/equations identified?			X
Are equations appropriate?	X		
Are units consistent?	X		

Comments: No comments

Results/Conclusions	**Yes**	**No**	**NA**
Are formulas consistent in spreadsheet cells?	X		
Are calculations correct?	X		
Are conclusions consistent with results?			X
Are conclusions consistent with applicable limits?			X

Comments: No comments

Reviewer Sign/Date: R. C. D. 8/1/2011

Richard C. Daniel

Filename: 52578-TI037 (Reviewed).xlsm
Worksheet: Summary Page

	A	B	C	D	E	F
1	**Summary of PSD Records**					
2	Source File:	"52578-TI-037.mea"				
3						
4	**Record number**	**Sample Name**	**Average result records**	**Pump speed**	**Stirrer speed**	**Ultrasonic level**
5	61	TI008-SP-Unsonicated	13,14,15,49,50,51""	2000	0	0
6	62	TI008-SP-Sonicated	17,18,19,53,54,55""	2000	0	100
7	65	TI008-SQ-Sonicated	29,30,31,41,42,43""	2000	0	100
8	66	TI008-SQ-Post Sonicated	33,34,35,45,46,47""	2000	0	0
9	67	TI008-SQ-Unsonicated	25,26,27,37,38,39""	2000	0	0
10	68	TI008-SP-Post Sonicated	21,22,23,57,58,59""	2000	0	0

	A	B	C	D	E	F	G	H	I	J	K
1	**Particle Size Distribution - Differential Format**										
2	Source File:	"52578-TI-037.mea"									
3											
4	**Record number**	**Sample Name**	**Sample source type**	**Sample bulk lot ref**	**SOP Name**	**Operator name**	**Measurement date and time**	**Analysis date and time**	**Particle name**	**Particle refractive index**	**Particle absorption index**
5											
6											
7	61	TI008-SP-Unsonicated		52578-TI-037		D3M966	Wednesday, June 15, 2011 1:00:40 PM	Wednesday, June 15, 2011 1:00:41 PM	Iron III Oxide_1	2.42	1
8	62	TI008-SP-Sonicated		52578-TI-037		D3M966	Wednesday, June 15, 2011 1:03:41 PM	Wednesday, June 15, 2011 1:03:43 PM	Iron III Oxide_1	2.42	1
9	65	TI008-SQ-Sonicated		52578-TI-037		D3M966	Wednesday, June 15, 2011 1:17:38 PM	Wednesday, June 15, 2011 1:17:40 PM	Iron III Oxide_1	2.42	1
10	66	TI008-SQ-Post Sonicated		52578-TI-037		D3M966	Wednesday, June 15, 2011 1:20:18 PM	Wednesday, June 15, 2011 1:20:19 PM	Iron III Oxide_1	2.42	1
11	67	TI008-SQ-Unsonicated		52578-TI-037		D3M966	Wednesday, June 15, 2011 1:14:25 PM	Wednesday, June 15, 2011 1:14:27 PM	Iron III Oxide_1	2.42	1
12	68	TI008-SP-Post Sonicated		52578-TI-037		D3M966	Wednesday, June 15, 2011 1:06:07 PM	Wednesday, June 15, 2011 1:06:09 PM	Iron III Oxide_1	2.42	1

	L	M	N	O	P	Q	R	S	T	U	V	W	X	Y	Z	AA	AB
1																	
2																	
3																	
4	**Dispersant name**	**Dispersant refractive index**	**Accessory name**	**Analysis model**	**Start result channel size**	**Last result channel size**	**Result emulation**	**Obscuration**	**Residual**	**Concentration**	**Span**	**Result transform type**	**D [4, 3] - Volume weighted mean**	**Uniformity**	**Specific surface area**	**D [3, 2] - Surface weighted mean**	**d (0.1)**
5																	
6																	
7	Water	1.33	Hydro 2000µP (A)	General purpose	0.02	2000	Off	8.42	1.392	0.033	2.11	Volume	261.569	0.686	0.426	14.079	4.915
8	Water	1.33	Hydro 2000µP (A)	General purpose	0.02	2000	Off	10.79	1.949	0.0254	2.335	Volume	235.001	0.813	0.646	9.288	3.448
9	Water	1.33	Hydro 2000µP (A)	General purpose	0.02	2000	Off	11.46	1.361	0.0409	1.855	Volume	302.964	0.571	0.364	16.49	9.118
10	Water	1.33	Hydro 2000µP (A)	General purpose	0.02	2000	Off	12.17	1.321	0.0463	1.925	Volume	285.523	0.6	0.351	17.117	7.836
11	Water	1.33	Hydro 2000µP (A)	General purpose	0.02	2000	Off	7.67	1.448	0.0547	1.742	Volume	342.915	0.49	0.19	31.654	23.301
12	Water	1.33	Hydro 2000µP (A)	General purpose	0.02	2000	Off	11.17	1.832	0.0304	2.398	Volume	226.058	0.826	0.592	10.132	3.355

	AC	AD	AE	AF	AG	AH	AI	AJ	AK	AL	AM	AN	AO	AP	AQ	AR	AS	AT
1																		
2																		
3																		
4	**d (0.5)**	**d (0.9)**	**Result Between User Sizes (Sizes in um)**															
5			0.02	0.02244	0.025179	0.028251	0.031698	0.035566	0.039905	0.044774	0.050238	0.056368	0.063246	0.070963	0.079621	0.089337	0.100237	0.112468
6			**Average Size, um**	0.02122	0.02381	0.026715	0.029975	0.033632	0.037736	0.04234	0.047506	0.053303	0.059807	0.067105	0.075292	0.084479	0.094787	0.106353
7	259.368	552.063		0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
8	226.637	532.714		0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
9	311.807	587.366		0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
10	289.715	565.474		0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
11	341.755	618.591		0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
12	212.463	512.873		0	0	0	0	0	0	0	0	0	0	0	0	0	0	0

	AU	AV	AW	AX	AY	AZ	BA	BB	BC	BD	BE	BF	BG	BH	BI	BJ	BK	BL	BM
1																			
2																			
3																			
4																			
5	0.126191	0.141589	0.158866	0.17825	0.2	0.224404	0.251785	0.282508	0.316979	0.355656	0.399052	0.447744	0.502377	0.563677	0.632456	0.709627	0.796214	0.893367	1.002374
6	0.11933	0.13389	0.150228	0.16856	0.2	0.212202	0.238095	0.267147	0.299744	0.336318	0.377354	0.423398	0.475061	0.533027	0.598067	0.671042	0.752921	0.844791	0.947871
7	0	0	0	0	0	0	0	0.011295	0.033376	0.041921	0.050012	0.065743	0.085408	0.09506	0.101074	0.108727	0.120624	0.138653	0.16536
8	0	0	0	0	0	0	0	0.010822	0.053131	0.096361	0.12999	0.165724	0.195324	0.224928	0.253439	0.281949	0.313263	0.348482	0.389481
9	0	0	0	0	0	0	0	0	0.006417	0.04762	0.073214	0.091442	0.110433	0.127118	0.144046	0.160791	0.17873	0.198412	0.220672
10	0	0	0	0	0	0	0	0	0	0.030456	0.057619	0.069658	0.07681	0.085624	0.095543	0.108364	0.126272	0.150484	0.182367
11	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0.049062	0.060287	0.067011	0.076298	0.090355
12	0	0	0	0	0	0	0	0.018669	0.051023	0.061201	0.09743	0.11865	0.125501	0.13823	0.151575	0.170359	0.198744	0.239196	0.294564

	BN	BO	BP	BQ	BR	BS	BT	BU	BV	BW	BX	BY	BZ	CA	CB	CC	CD	CE	CF
1																			
2																			
3																			
4																			
5	1.124683	1.261915	1.415892	1.588656	1.782502	2	2.244037	2.517851	2.825075	3.169786	3.556559	3.990525	4.477442	5.023773	5.636766	6.324555	7.096268	7.962143	8.933672
6	1.063529	1.193299	1.338904	1.502274	1.685579	1.891251	2.122019	2.380944	2.671463	2.997431	3.363173	3.773542	4.233984	4.750608	5.33027	5.980661	6.710412	7.529206	8.447908
7	0.202884	0.251632	0.313274	0.384643	0.466106	0.552156	0.640703	0.727085	0.807029	0.878234	0.936253	0.98134	1.011055	1.026873	1.029449	1.02067	1.002379	0.977214	0.946455
8	0.437396	0.491245	0.551294	0.613072	0.675546	0.733493	0.785396	0.829022	0.863848	0.891154	0.911938	0.929103	0.944234	0.959556	0.97558	0.992275	1.008735	1.023171	1.034477
9	0.24606	0.274062	0.304861	0.336243	0.367774	0.396892	0.422887	0.444678	0.462048	0.475726	0.486368	0.495683	0.504773	0.515208	0.527623	0.54234	0.559143	0.576855	0.594837
10	0.222636	0.27002	0.324275	0.380812	0.438018	0.490261	0.535134	0.569523	0.5921	0.603385	0.604892	0.599397	0.590465	0.581202	0.574313	0.571249	0.572467	0.577251	0.58449
11	0.108389	0.130864	0.159122	0.191563	0.228474	0.267302	0.306964	0.345178	0.379839	0.409774	0.43307	0.449898	0.459604	0.4632	0.461549	0.455936	0.447566	0.437776	0.427112
12	0.366411	0.452611	0.552805	0.658513	0.76667	0.866572	0.953484	1.021211	1.066776	1.090612	1.094838	1.083967	1.063962	1.039896	1.016422	0.996001	0.979442	0.966309	0.95478

	CG	CH	CI	CJ	CK	CL	CM	CN	CO	CP	CQ	CR	CS	CT	CU	CV	CW
1																	
2																	
3																	
4																	
5	10.023745	11.246826	12.619147	14.158916	15.886565	17.825019	20	22.440369	25.178508	28.250751	31.697864	35.565588	39.905246	44.774423	50.237729	56.367659	63.245553
6	9.4787085	10.635286	11.932987	13.389032	15.022741	16.855792	18.91251	21.220185	23.809439	26.71463	29.974308	33.631726	37.735417	42.339835	47.506076	53.302694	59.806606
7	0.913048	0.877236	0.840917	0.804056	0.767665	0.733278	0.702374	0.67815	0.662024	0.656597	0.663285	0.683101	0.715604	0.759153	0.808633	0.859423	0.903106
8	1.041091	1.042583	1.038826	1.030654	1.019959	1.009811	1.003981	1.006151	1.018863	1.042584	1.077023	1.118859	1.163339	1.204032	1.231859	1.23824	1.215676
9	0.611158	0.625144	0.635357	0.641665	0.644607	0.645986	0.648918	0.656989	0.674213	0.703052	0.746098	0.802449	0.86885	0.939271	1.002309	1.046896	1.05909
10	0.592386	0.599651	0.604805	0.607437	0.608074	0.608594	0.612317	0.622944	0.644607	0.679469	0.729993	0.794806	0.87042	0.950887	1.025034	1.083226	1.112536
11	0.416416	0.40533	0.393793	0.38116	0.367399	0.353177	0.339795	0.330041	0.326611	0.332875	0.352007	0.385839	0.43416	0.494639	0.559823	0.622334	0.669561
12	0.943679	0.931176	0.916768	0.900323	0.883338	0.868755	0.860592	0.863116	0.879475	0.910518	0.956503	1.014078	1.078459	1.143333	1.199	1.23763	1.250144

	CX	CY	CZ	DA	DB	DC	DD	DE	DF	DG	DH	DI	DJ	DK	DL	DM
1																
2																
3																
4																
5	70.962678	79.621434	89.336718	100.237447	112.468265	126.191469	141.589157	158.865647	178.250188	200	224.403691	251.785082	282.507509	316.978638	355.655882	399.052463
6	67.104116	75.292056	84.479076	94.7870825	106.352856	119.329867	133.890313	150.227402	168.557918	189.1251	212.201846	238.094387	267.146296	299.743074	336.31726	377.354173
7	0.935936	0.954601	0.963145	0.972802	1.002857	1.082853	1.245228	1.532084	1.960859	2.552375	3.28516	4.113116	4.971761	5.749434	6.361355	6.68406
8	1.159563	1.072299	0.962066	0.846749	0.753256	0.7151	0.774328	0.974865	1.341743	1.897142	2.622123	3.469441	4.371953	5.211316	5.897135	6.292074
9	1.030023	0.956415	0.844553	0.714291	0.59968	0.545508	0.609612	0.852773	1.312728	2.022373	2.960597	4.06635	5.25035	6.354996	7.261087	7.794122
10	1.106509	1.063033	0.989844	0.906578	0.845051	0.84762	0.964753	1.251476	1.735202	2.440372	3.337098	4.360245	5.420351	6.370671	7.101659	7.465621
11	0.694278	0.690722	0.662995	0.626694	0.610365	0.656087	0.81421	1.145236	1.681364	2.456581	3.449073	4.599466	5.820247	6.954192	7.883251	8.431035
12	1.232592	1.185529	1.116934	1.044019	0.992869	0.997231	1.096604	1.333762	1.728112	2.296367	3.011228	3.818489	4.645772	5.37711	5.925023	6.170612

	DN	DO	DP	DQ	DR	DS	DT	DU	DV	DW	DX	DY	DZ	EA
1														
2														
3														
4														
5	447.744228	502.377286	563.676586	632.455532	709.626778	796.214341	893.367184	1002.374467	1124.68265	1261.914689	1415.891569	1588.656469	1782.501876	2000
6	423.398346	475.060757	533.026936	598.066059	671.041155	752.92056	844.790763	947.8708255	1063.52856	1193.29867	1338.903129	1502.274019	1685.579173	1891
7	6.654544	6.235928	5.448067	4.299124	3.070077	1.617135	0.107164	0	0	0	0	0	0	0
8	6.327372	5.948176	5.153541	3.994619	2.574332	0.998245	0.025603	0	0	0	0	0	0	0
9	7.877551	7.462613	6.580755	5.307477	3.84805	2.252255	0.638393	0.012436	0	0	0	0	0	0
10	7.401084	6.881454	5.948386	4.686926	3.283186	1.798539	0.382064	0	0	0	0	0	0	0
11	8.521395	8.104943	7.214914	5.946154	4.478477	2.924131	1.408417	0.154616	0	0	0	0	0	0
12	6.059313	5.557623	4.677158	3.513057	2.085217	0.629513	0.012553	0	0	0	0	0	0	0

Filename: 52578-TI037 (Reviewed).xlsm
Worksheet: PSD

	EB
1	
2	
3	
4	**Operator Notes**
5	
6	
7	Average of 6 measurements from 52578-TI-037.mea
8	Average of 6 measurements from 52578-TI-037.mea
9	Average of 6 measurements from 52578-TI-037.mea
10	Average of 6 measurements from 52578-TI-037.mea
11	Average of 6 measurements from 52578-TI-037.mea
12	Average of 6 measurements from 52578-TI-037.mea

	A	B	C	D	E	F	G	H	I	J	K
1	**Particle Size Distribution - Cumulative Format**										
2	Source File:	"52578-TI-037.mea"									
3											
4	**Record number**	**Sample Name**	**Sample source type**	**Sample bulk lot ref**	**SOP Name**	**Operator name**	**Measurement date and time**	**Analysis date and time**	**Particle name**	**Particle refractive index**	**Particle absorption index**
5											
6	61	TI008-SP-Unsonicated		52578-TI-037		D3M966	Wednesday, June 15, 2011 1:00:40 PM	Wednesday, June 15, 2011 1:00:41 PM	Iron III Oxide_1	2.42	1
7	62	TI008-SP-Sonicated		52578-TI-037		D3M966	Wednesday, June 15, 2011 1:03:41 PM	Wednesday, June 15, 2011 1:03:43 PM	Iron III Oxide_1	2.42	1
8	65	TI008-SQ-Sonicated		52578-TI-037		D3M966	Wednesday, June 15, 2011 1:17:38 PM	Wednesday, June 15, 2011 1:17:40 PM	Iron III Oxide_1	2.42	1
9	66	TI008-SQ-Post Sonicated		52578-TI-037		D3M966	Wednesday, June 15, 2011 1:20:18 PM	Wednesday, June 15, 2011 1:20:19 PM	Iron III Oxide_1	2.42	1
10	67	TI008-SQ-Unsonicated		52578-TI-037		D3M966	Wednesday, June 15, 2011 1:14:25 PM	Wednesday, June 15, 2011 1:14:27 PM	Iron III Oxide_1	2.42	1
11	68	TI008-SP-Post Sonicated		52578-TI-037		D3M966	Wednesday, June 15, 2011 1:06:07 PM	Wednesday, June 15, 2011 1:06:09 PM	Iron III Oxide_1	2.42	1

Filename: 52578-TI037 (Reviewed).xlsm
Worksheet: Cumulative

	L	M	N	O	P	Q	R	S	T	U	V	W	X	Y	Z	AA	AB
1																	
2																	
3																	
4	**Dispersant name**	**Dispersant refractive index**	**Accessory name**	**Analysis model**	**Start result channel size**	**Last result channel size**	**Result emulation**	**Obscuration**	**Residual**	**Concentration**	**Span**	**Result transform type**	**D [4, 3] - Volume weighted mean**	**Uniformity**	**Specific surface area**	**D [3, 2] - Surface weighted mean**	**d (0.1)**
5																	
6	Water	1.33	Hydro 2000µP (A)	General purpose	0.02	2000	Off	8.42	1.392	0.033	2.11	Volume	261.569	0.686	0.426	14.079	4.915
7	Water	1.33	Hydro 2000µP (A)	General purpose	0.02	2000	Off	10.79	1.949	0.0254	2.335	Volume	235.001	0.813	0.646	9.288	3.448
8	Water	1.33	Hydro 2000µP (A)	General purpose	0.02	2000	Off	11.46	1.361	0.0409	1.855	Volume	302.964	0.571	0.364	16.49	9.118
9	Water	1.33	Hydro 2000µP (A)	General purpose	0.02	2000	Off	12.17	1.321	0.0463	1.925	Volume	285.523	0.6	0.351	17.117	7.836
10	Water	1.33	Hydro 2000µP (A)	General purpose	0.02	2000	Off	7.67	1.448	0.0547	1.742	Volume	342.915	0.49	0.19	31.654	23.301
11	Water	1.33	Hydro 2000µP (A)	General purpose	0.02	2000	Off	11.17	1.832	0.0304	2.398	Volume	226.058	0.826	0.592	10.132	3.355

	AC	AD	AE	AF	AG	AH	AI	AJ	AK	AL	AM	AN	AO	AP	AQ	AR	AS	AT
1																		
2																		
3																		
4	**d (0.5)**	**d (0.9)**	**Result Below User Sizes (Sizes in um)**															
5			0.02	0.02244	0.025179	0.028251	0.031698	0.035566	0.039905	0.044774	0.050238	0.056368	0.063246	0.070963	0.079621	0.089337	0.100237	0.112468
6	259.368	552.063	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
7	226.637	532.714	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
8	311.807	587.366	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
9	289.715	565.474	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
10	341.755	618.591	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
11	212.463	512.873	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0

	AU	AV	AW	AX	AY	AZ	BA	BB	BC	BD	BE	BF	BG	BH	BI	BJ	BK	BL	BM
1																			
2																			
3																			
4																			
5	0.126191	0.141589	0.158866	0.17825	0.2	0.224404	0.251785	0.282508	0.316979	0.355656	0.399052	0.447744	0.502377	0.563677	0.632456	0.709627	0.796214	0.893367	1.002374
6	0	0	0	0	0	0	0	0.011295	0.044671	0.086592	0.136604	0.202347	0.287756	0.382816	0.48389	0.592617	0.713241	0.851893	1.017253
7	0	0	0	0	0	0	0	0.010822	0.063953	0.160314	0.290305	0.456028	0.651353	0.87628	1.129719	1.411669	1.724931	2.073413	2.462895
8	0	0	0	0	0	0	0	0	0.006417	0.054037	0.127251	0.218693	0.329127	0.456245	0.600291	0.761082	0.939812	1.138224	1.358896
9	0	0	0	0	0	0	0	0	0	0.030456	0.088075	0.157733	0.234543	0.320168	0.415711	0.524076	0.650347	0.800831	0.983199
10	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0.049062	0.109349	0.17636	0.252658	0.343012
11	0	0	0	0	0	0	0	0.018669	0.069692	0.130893	0.228323	0.346973	0.472473	0.610704	0.762279	0.932638	1.131381	1.370578	1.665142

	BN	BO	BP	BQ	BR	BS	BT	BU	BV	BW	BX	BY	BZ	CA	CB	CC	CD	CE
1																		
2																		
3																		
4																		
5	1.124683	1.261915	1.415892	1.588656	1.782502	2	2.244037	2.517851	2.825075	3.169786	3.556559	3.990525	4.477442	5.023773	5.636766	6.324555	7.096268	7.962143
6	1.220138	1.47177	1.785043	2.169686	2.635792	3.187948	3.828651	4.555736	5.362765	6.240999	7.177252	8.158591	9.169647	10.196519	11.225969	12.246639	13.249018	14.226232
7	2.900291	3.391536	3.94283	4.555902	5.231448	5.964941	6.750337	7.579359	8.443207	9.334361	10.246299	11.175402	12.119636	13.079192	14.054772	15.047047	16.055782	17.078953
8	1.604956	1.879019	2.183879	2.520122	2.887896	3.284787	3.707674	4.152352	4.6144	5.090126	5.576494	6.072177	6.576951	7.092159	7.619782	8.162122	8.721264	9.298119
9	1.205835	1.475855	1.800129	2.180942	2.61896	3.109221	3.644355	4.213879	4.805979	5.409364	6.014255	6.613653	7.204118	7.785319	8.359632	8.930881	9.503349	10.0806
10	0.451401	0.582266	0.741388	0.932951	1.161425	1.428727	1.735692	2.08087	2.460708	2.870482	3.303552	3.75345	4.213054	4.676254	5.137803	5.593738	6.041305	6.479081
11	2.031553	2.484164	3.036969	3.695483	4.462153	5.328724	6.282209	7.30342	8.370196	9.460807	10.555646	11.639613	12.703575	13.74347	14.759893	15.755894	16.735337	17.701646

	CF	CG	CH	CI	CJ	CK	CL	CM	CN	CO	CP	CQ	CR	CS	CT	CU	CV
1																	
2																	
3																	
4																	
5	8.933672	10.023745	11.246826	12.619147	14.158916	15.886565	17.825019	20	22.440369	25.178508	28.250751	31.697864	35.565588	39.905246	44.774423	50.237729	56.367659
6	15.172687	16.085735	16.962971	17.803887	18.607944	19.375609	20.108886	20.811261	21.48941	22.151434	22.808031	23.471316	24.154417	24.870021	25.629174	26.437807	27.29723
7	18.113429	19.154521	20.197103	21.23593	22.266583	23.286543	24.296353	25.300334	26.306485	27.325348	28.367932	29.444955	30.563814	31.727153	32.931185	34.163044	35.401284
8	9.892957	10.504115	11.129259	11.764616	12.406281	13.050888	13.696873	14.345792	15.00278	15.676994	16.380046	17.126144	17.928593	18.797444	19.736715	20.739024	21.78592
9	10.66509	11.257476	11.857127	12.461932	13.069369	13.677443	14.286038	14.898355	15.521298	16.165905	16.845374	17.575367	18.370174	19.240593	20.19148	21.216515	22.29974
10	6.906193	7.32261	7.72794	8.121733	8.502893	8.870292	9.223469	9.563265	9.893305	10.219917	10.552792	10.904799	11.290638	11.724798	12.219438	12.779261	13.401595
11	18.656426	19.600105	20.531281	21.448049	22.348372	23.23171	24.100465	24.961057	25.824173	26.703648	27.614166	28.570669	29.584747	30.663206	31.806539	33.00554	34.243169

	CW	CX	CY	CZ	DA	DB	DC	DD	DE	DF	DG	DH	DI	DJ	DK	DL
1																
2																
3																
4																
5	63.245553	70.962678	79.621434	89.336718	100.237447	112.468265	126.191469	141.589157	158.865647	178.250188	200	224.403691	251.785082	282.507509	316.978638	355.655882
6	28.200336	29.136272	30.090873	31.054018	32.026819	33.029677	34.11253	35.357758	36.889842	38.850701	41.403076	44.688236	48.801352	53.773113	59.522546	65.883901
7	36.61696	37.776523	38.848822	39.810888	40.657637	41.410892	42.125992	42.90032	43.875186	45.216929	47.11407	49.736194	53.205635	57.577588	62.788904	68.686039
8	22.84501	23.875033	24.831448	25.676001	26.390292	26.989972	27.53548	28.145091	28.997864	30.310592	32.332965	35.293563	39.359913	44.610263	50.96526	58.226347
9	23.412277	24.518786	25.581819	26.571663	27.478241	28.323292	29.170912	30.135665	31.387141	33.122343	35.562715	38.899813	43.260059	48.680409	55.05108	62.152739
10	14.071157	14.765434	15.456156	16.119152	16.745846	17.356211	18.012298	18.826507	19.971744	21.653107	24.109688	27.558761	32.158228	37.978475	44.932667	52.815918
11	35.493313	36.725906	37.911435	39.028369	40.072388	41.065257	42.062487	43.159091	44.492853	46.220965	48.517332	51.52856	55.34705	59.992821	65.369931	71.294955

	DM	DN	DO	DP	DQ	DR	DS	DT	DU	DV	DW	DX	DY	DZ	EA
1															
2															
3															
4															
5	399.052463	447.744228	502.377286	563.676586	632.455532	709.626778	796.214341	893.367184	1002.374467	1124.68265	1261.914689	1415.891569	1588.656469	1782.501876	2000
6	72.567961	79.222505	85.458433	90.9065	95.205624	98.275701	99.892836	100	100	100	100	100	100	100	100
7	74.978112	81.305484	87.25366	92.407201	96.40182	98.976153	99.974397	100	100	100	100	100	100	100	100
8	66.020469	73.898019	81.360633	87.941388	93.248865	97.096915	99.34917	99.987564	100	100	100	100	100	100	100
9	69.61836	77.019444	83.900898	89.849284	94.536211	97.819397	99.617936	100	100	100	100	100	100	100	100
10	61.246953	69.768348	77.873291	85.088205	91.034359	95.512836	98.436967	99.845384	100	100	100	100	100	100	100
11	77.465566	83.524879	89.082503	93.759661	97.272718	99.357935	99.987447	100	100	100	100	100	100	100	100

Filename: 52578-TI037 (Reviewed).xlsm
Worksheet: Cumulative

	EB
1	
2	
3	
4	**Operator Notes**
5	
6	Average of 6 measurements from 52578-TI-037.mea
7	Average of 6 measurements from 52578-TI-037.mea
8	Average of 6 measurements from 52578-TI-037.mea
9	Average of 6 measurements from 52578-TI-037.mea
10	Average of 6 measurements from 52578-TI-037.mea
11	Average of 6 measurements from 52578-TI-037.mea

Filename: 52578-TI037 (Reviewed).xlsm
Worksheet: Cumulative Tables

	A	B	C	D	E
1	**Particle Size Distribution - Cumulative Table Format**				
2	Source File:	"52578-TI-037.mea"			
3					
4	**Record number**	61	62	67	65
5	**Sample Name**	TI008-SP-Unsonicated	TI008-SP-Sonicated	TI008-SQ-Unsonicated	TI008-SQ-Sonicated
6	**Analysis model**	General purpose	General purpose	General purpose	General purpose
7	**Particle refractive index**	2.42	2.42	2.42	2.42
8	**Pump speed**	2000	2000	2000	2000
9					
10	**Size, µm**	**Percent Cumulative Under Size by Volume, %**			
11	0.05	0	0	0	0
12	0.5	0.28	0.64	0.00	0.32
13	1	1.01	2.45	0.34	1.35
14	2.5	4.51	7.53	2.06	4.12
15	5	10.2	13.0	4.66	7.07
16	10	16.1	19.1	7.31	10.5
17	20	20.8	25.3	9.56	14.3
18	40	24.9	31.8	11.7	18.8
19	60	27.8	36.1	13.8	22.4
20	80	30.1	38.9	15.5	24.9
21	100	32.0	40.6	16.7	26.4
22	125	34.0	42.1	18.0	27.5
23	150	36.1	43.4	19.3	28.5
24	175	38.5	45.0	21.3	30.1
25	200	41.4	47.1	24.1	32.3
26	300	56.7	60.2	41.5	47.8
27	400	72.7	75.1	61.4	66.2
28	500	85.2	87.0	77.6	81.1
29	600	93.4	94.7	88.5	91.0
30	700	98.0	98.7	95.1	96.7
31	800	99.9	100	98.5	99.4
32	1000	100	100	100	100

Filename: 52578-TI037 (Reviewed).xlsm
Worksheet: Percentiles

	A	B	C	D	E
1	**Particle Size Distribution - Percentiles**				
2	Source File:	"52578-TI-037.mea"			
3					
4	**Record number**	61	62	67	65
5	**Sample Name**	TI008-SP-Unsonicated	TI008-SP-Sonicated	TI008-SQ-Unsonicated	TI008-SQ-Sonicated
6	**Pump speed**	2000	2000	2000	2000
7	**Stirrer speed**	0	0	0	0
8	**Ultrasonic level**	0	100	0	100
9	**d(0.010), µm**	0.99	0.60	1.65	0.83
10	**d(0.050), µm**	2.69	1.72	5.45	3.10
11	**d(0.100), µm**	4.92	3.45	23.3	9.12
12	**d(0.500), µm**	259	227	342	312
13	**d(0.900), µm**	552	533	619	587
14	**d(0.950), µm**	628	605	699	663
15	**d(0.990), µm**	737	711	823	776

	A	B	C	D	E	F	G	H	I
1	**Bounding Performance Check Results**								
2	Source File:	"RPL performance check log.mea"							
3									
4	**Opening Check (QAS3004) - 06/15/2011**								
5	Bottle Lot No.	02/03858							
6	Percentile	d(50)							
7	List	62.362							
8	Measured	63.112							
9	Relative Error, E_r, (%)	1.20							
10	[(a)] Acceptable yes/no	y							
11									
12	**Closing Check (QAS3004) - 06/15/11**								
13	Bottle Lot No.	02/03980							
14	Percentile	d(50)							
15	List	62.362							
16	Measured	61.997							
17	Relative Error, E_r, (%)	-0.59							
18	[(a)] Acceptable yes/no	y							
19									
20	**(a) Acceptance criteria as per RPL-COLLOID-01, Rev. 1, E_r less than 10 % from list and measure value for the d(50)**								
21									
22	**Exported Performance Check Results**								
23	**Measurement date and time**	**Sample Name**	**Average result records**	**Record number**	**Sample source type**	**Sample bulk lot type**	**Sample bulk lot ref**	**Operator name**	**Analysis date and time**
24	Wednesday, June 15, 2011 12:29:10 PM	QAS3004 6.15.2011-1.1 - Average	13,14,15""	16	Malvern	02/03858	02/03858	D3M966	Wednesday, June 15, 2011 12:29:11 PM
25	Wednesday, June 15, 2011 1:59:56 PM	QAS3004 6.15.2011-closing-1.1 - Average	17,18,19""	20	Malvern	02/03980	02/03980	D3M966	Wednesday, June 15, 2011 1:59:58 PM

	J	K	L	M	N	O	P	Q	R	S	T	U	V	W	X
1															
2															
3															
4															
5															
6															
7															
8															
9															
10															
11															
12															
13															
14															
15															
16															
17															
18															
19															
20															
21															
22															
23	**Particle name**	**Particle refractive index**	**Particle absorption index**	**Dispersant name**	**Dispersant refractive index**	**Accessory name**	**Analysis model**	**Start result channel size**	**Obscuration**	**Last result channel size**	**Residual**	**Concentration**	**Span**	**Result transform type**	**Uniformity**
24	Glass beads (typical)	1.52	0	Water	1.33	Hydro 2000µP (A)	Single narrow mode (spherical)	0.02	15.25	2000	0.409	0.2353	0.792	Volume	0.253
25	Glass beads (typical)	1.52	0	Water	1.33	Hydro 2000µP (A)	Single narrow mode (spherical)	0.02	17.13	2000	0.361	0.2598	0.824	Volume	0.262

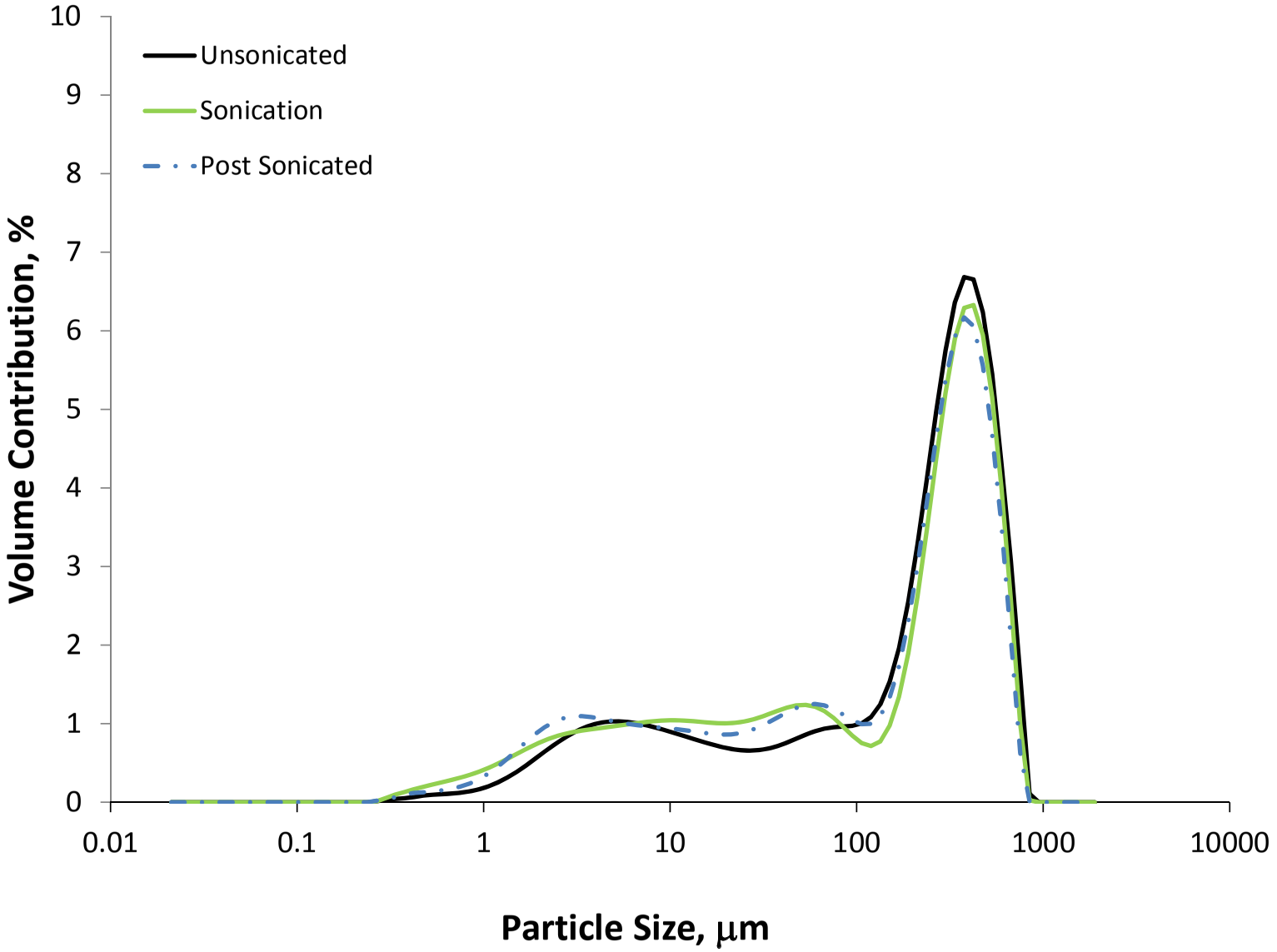
Filename: 52578-TI037 (Reviewed).xlsm
Worksheet: Performance Checks

	Y	Z	AA
1			
2			
3			
4			
5			
6			
7			
8			
9			
10			
11			
12			
13			
14			
15			
16			
17			
18			
19			
20			
21			
22			
23	**d (0.1)**	**d (0.5)**	**d (0.9)**
24	41.303	63.112	91.269
25	39.273	61.997	90.367

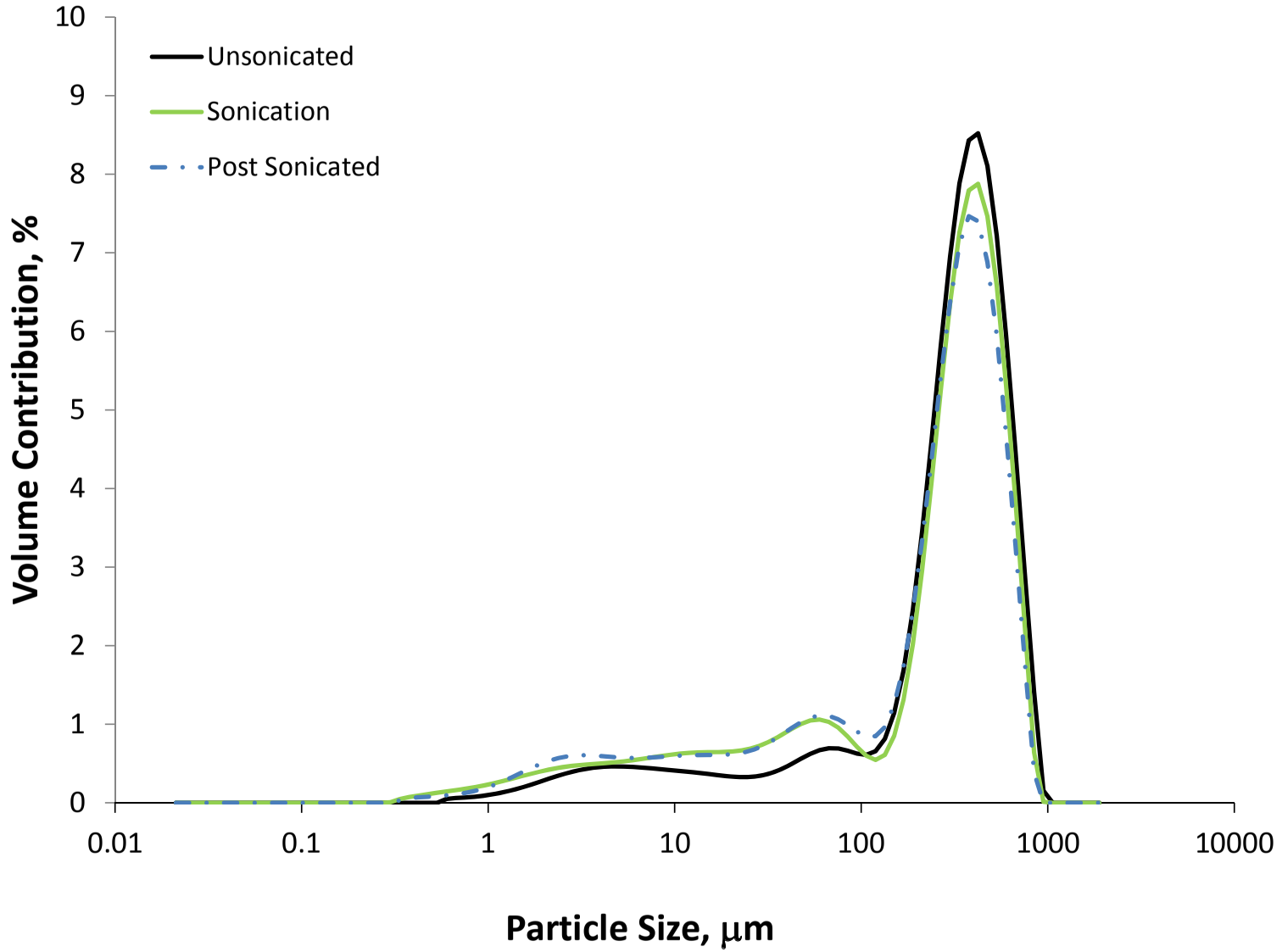
Filename: 52578-TI037 (Reviewed).xlsm
Worksheet: Graph Information

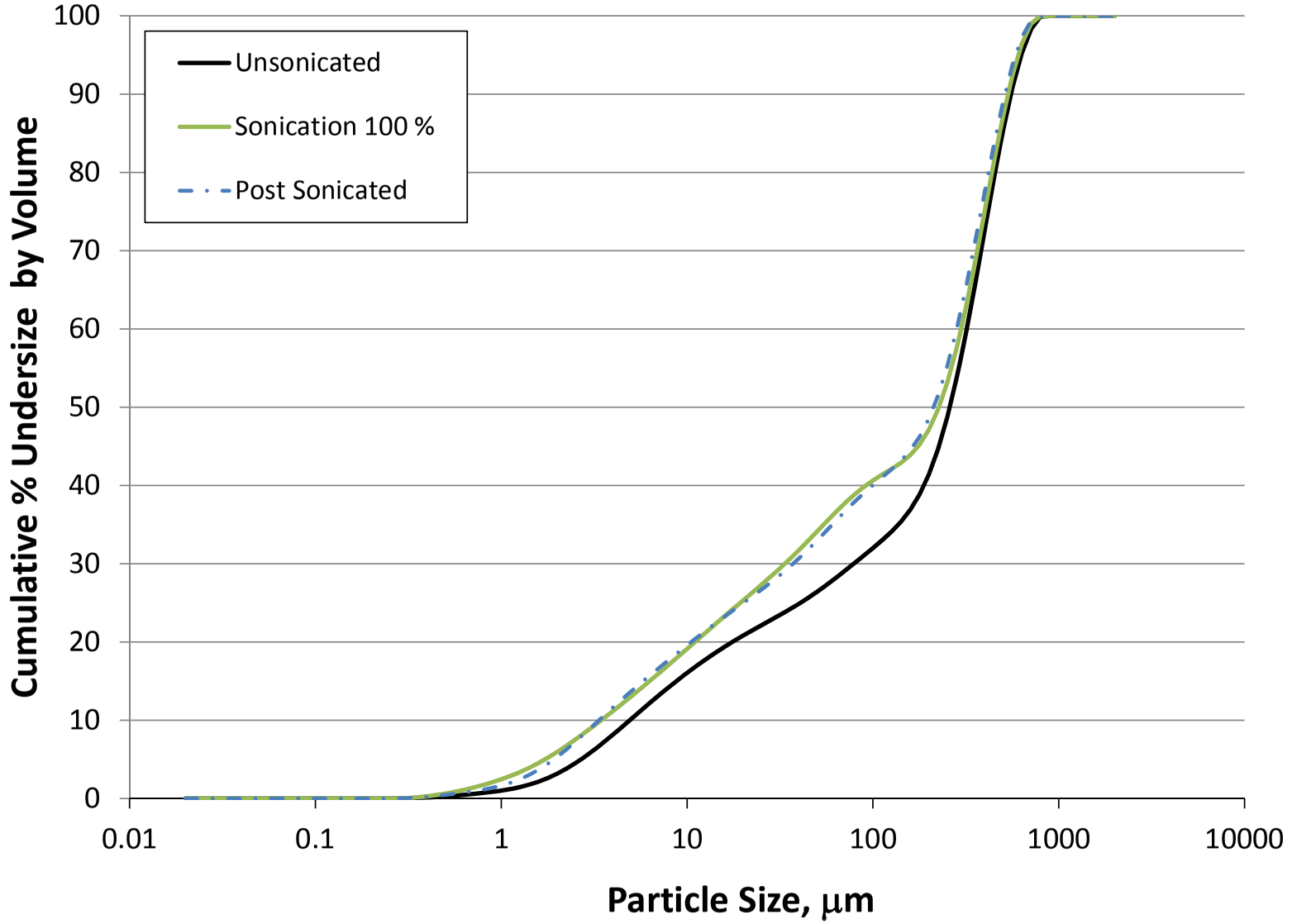
	A	B
1	**Summary of Graphs and Graph Reference Cells**	
2		
3	**Stand Alone Charts**	
4		
5	Graph #:	1
6	Worksheet Label:	TI-008-SP PSD chart
7	Number of Data Series:	3
8	Series 1 Formula:	=SERIES("Unsonicated",PSD!$AF$6:$EA$6,PSD!$AF$7:$EA$7,1)
9	Series 2 Formula:	=SERIES("Sonication",PSD!$AF$6:$EA$6,PSD!$AF$8:$EA$8,2)
10	Series 3 Formula:	=SERIES("Post Sonicated",PSD!$AF$6:$EA$6,PSD!$AF$12:$EA$12,3)
11		
12		
13	Graph #:	2
14	Worksheet Label:	TI008-SQ PSD chart
15	Number of Data Series:	3
16	Series 1 Formula:	=SERIES("Unsonicated",PSD!$AF$6:$EA$6,PSD!$AF$11:$EA$11,1)
17	Series 2 Formula:	=SERIES("Sonication",PSD!$AF$6:$EA$6,PSD!$AF$9:$EA$9,2)
18	Series 3 Formula:	=SERIES("Post Sonicated",PSD!$AF$6:$EA$6,PSD!$AF$10:$EA$10,3)
19		
20		
21	Graph #:	3
22	Worksheet Label:	TI-008-SP CUM chart
23	Number of Data Series:	3
24	Series 1 Formula:	=SERIES("Unsonicated",Cumulative!$AE$5:$EA$5,Cumulative!$AE$6:$EA$6,1)
25	Series 2 Formula:	=SERIES("Sonication 100 %",Cumulative!$AE$5:$EA$5,Cumulative!$AE$7:$EA$7,2)
26	Series 3 Formula:	=SERIES("Post Sonicated",Cumulative!$AE$5:$EA$5,Cumulative!$AE$11:$EA$11,3)
27		
28		
29	Graph #:	4
30	Worksheet Label:	TI-008-SQ CUM chart
31	Number of Data Series:	3
32	Series 1 Formula:	=SERIES("Unsonicated",Cumulative!$AE$5:$EA$5,Cumulative!$AE$10:$EA$10,1)
33	Series 2 Formula:	=SERIES("Sonication 100 %",Cumulative!$AE$5:$EA$5,Cumulative!$AE$8:$EA$8,2)
34	Series 3 Formula:	=SERIES("Post Sonicated",Cumulative!$AE$5:$EA$5,Cumulative!$AE$9:$EA$9,3)
35		

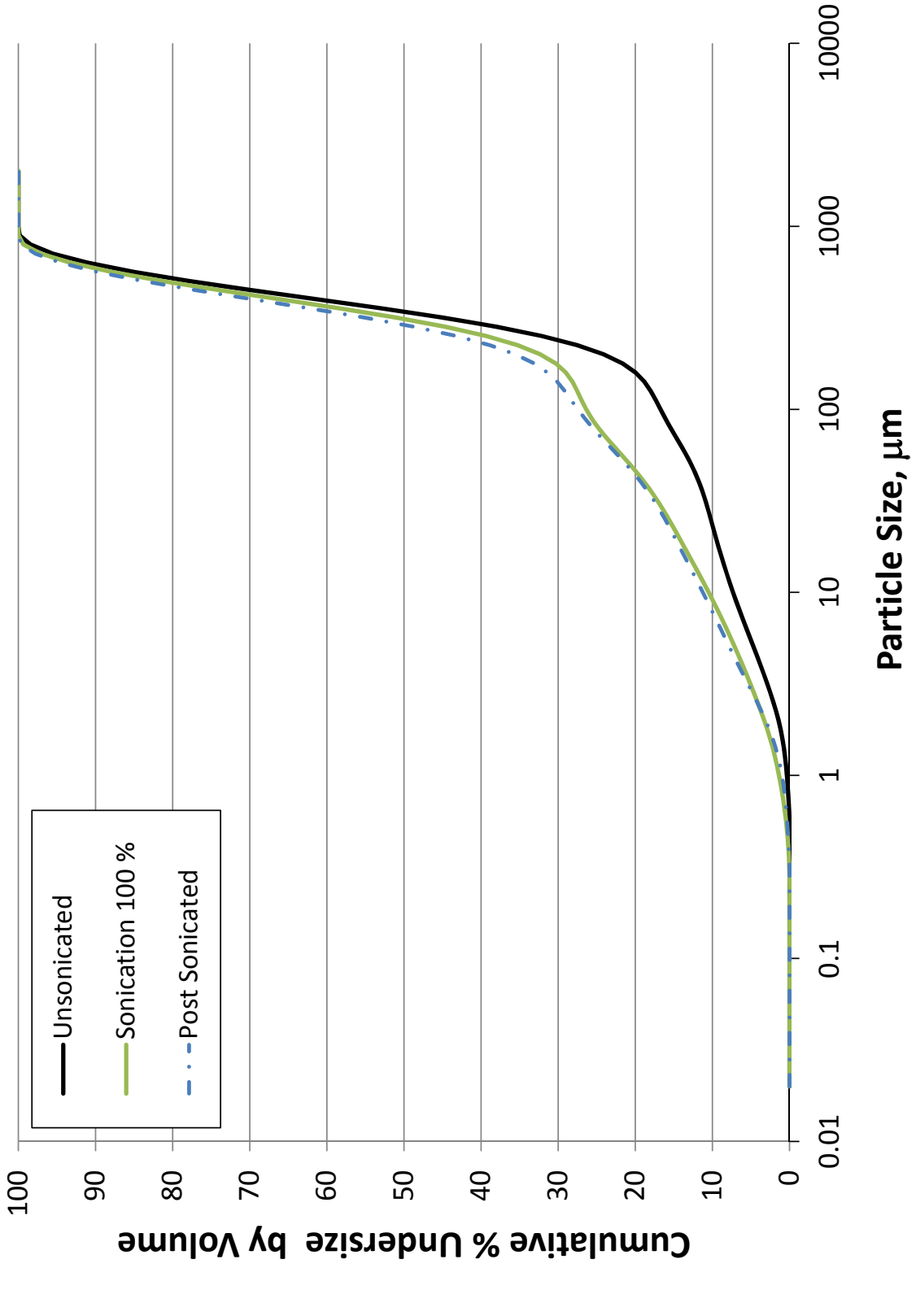
Filename: 52578-TI037 (Reviewed).xlsm
Worksheet: TI-008-SP PSD chart



Filename: 52578-TI037 (Reviewed).xlsm
Worksheet: TI008-SQ PSD chart




Unsonicated
Sonication 100 %
Post Sonicated
Cumulative % Undersize by Volume
Particle Size, µm
0
10
20
30
40
50
60
70
80
90
100
0.01
0.1
1
10
100
1000
10000



Filename: 52578-TI037 (Reviewed).xlsm
Worksheet: TI-008-SQ CUM chart

July 7, 2011

Filename: TI008 averaged data.xlsm
Worksheet: summary

	A	B	C	D	E	F
1	**Summary of Records - Supplemental Data Package**					
2	This data package supplements KW210 PSD data in "52578-TI037 (Reviewed).xlsm"					
3	The source Malvern measurement file for the current data package is "52578-TI-037.mea"					
4						
5	**Record number**	**Sample Name**	**Average result records**	**Pump speed**	**Stirrer speed**	**Ultrasonic level**
6	69	TI008-Unsonicated Average	13,14,15,25,26,27,37,38,39,49,50,51""	2000	0	0
7	70	TI008-Sonicated Average	17,18,19,29,30,31,41,42,43,53,54,55""	2000	0	100
8	71	TI008-Post Sonication Average	21,22,23,33,34,35,45,46,47,57,58,59""	2000	0	0

	A	B	C	D	E	F	G	H	I	J	K	L	M
1	**Differential PSD Data - Supplemental Package**												
2	Source:	"52578-TI-037.mea"											
3													
4	**Record number**	**Sample Name**	**Sample source type**	**Sample bulk lot ref**	**SOP Name**	**Operator name**	**Measurement date and time**	**Analysis date and time**	**Particle name**	**Particle refractive index**	**Particle absorption index**	**Dispersant name**	**Dispersant refractive index**
5													
6													
7	69	TI008-Unsonicated Average		52578-TI-037		D3M966	Wednesday, June 15, 2011 1:00:40 PM	Wednesday, June 15, 2011 1:00:41 PM	Iron III Oxide_1	2.42	1	Water	1.33
8	70	TI008-Sonicated Average		52578-TI-037		D3M966	Wednesday, June 15, 2011 1:03:41 PM	Wednesday, June 15, 2011 1:03:43 PM	Iron III Oxide_1	2.42	1	Water	1.33
9	71	TI008-Post Sonication Average		52578-TI-037		D3M966	Wednesday, June 15, 2011 1:06:07 PM	Wednesday, June 15, 2011 1:06:09 PM	Iron III Oxide_1	2.42	1	Water	1.33

Filename: TI008 averaged data.xlsm
Worksheet: psd

	N	O	P	Q	R	S	T	U	V	W	X	Y	Z	AA	AB	AC	AD
1																	
2																	
3																	
4	**Accessory name**	**Analysis model**	**Start result channel size**	**Last result channel size**	**Result emulation**	**Obscuration**	**Residual**	**Concentration**	**Span**	**Result transform type**	**D [4, 3] - Volume weighted mean**	**Uniformity**	**Specific surface area**	**D [3, 2] - Surface weighted mean**	**d (0.1)**	**d (0.5)**	**d (0.9)**
5																	
6																	
7	Hydro 2000µP (A)	General purpose	0.02	2000	Off	8.05	1.42	0.0438	1.902	Volume	302.242	0.576	0.308	19.489	7.515	304.954	587.612
8	Hydro 2000µP (A)	General purpose	0.02	2000	Off	11.12	1.655	0.0332	2.019	Volume	268.982	0.668	0.505	11.883	4.958	275.765	561.607
9	Hydro 2000µP (A)	General purpose	0.02	2000	Off	11.67	1.576	0.0383	2.093	Volume	255.79	0.689	0.471	12.729	4.507	256.157	540.55

	AE	AF	AG	AH	AI	AJ	AK	AL	AM	AN	AO	AP	AQ	AR	AS	AT	AU	AV	AW	AX
1																				
2																				
3																				
4	**Result Between User Sizes (Sizes in um)**																			
5	0.02	0.02244	0.025179	0.028251	0.031698	0.035566	0.039905	0.044774	0.050238	0.056368	0.063246	0.070963	0.079621	0.089337	0.100237	0.112468	0.126191	0.141589	0.158866	0.17825
6	**Average Size (um)**	0.02122	0.0238095	0.026715	0.0299745	0.033632	0.0377355	0.0423395	0.047506	0.053303	0.059807	0.0671045	0.075292	0.084479	0.094787	0.1063525	0.1193295	0.13389	0.1502275	0.168558
7		0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
8		0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
9		0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0

Filename: TI008 averaged data.xlsm
Worksheet: psd

	AY	AZ	BA	BB	BC	BD	BE	BF	BG	BH	BI	BJ	BK	BL	BM	BN	BO	BP	BQ	BR	BS
1																					
2																					
3																					
4																					
5	0.2	0.224404	0.251785	0.282508	0.316979	0.355656	0.399052	0.447744	0.502377	0.563677	0.632456	0.709627	0.796214	0.893367	1.002374	1.124683	1.261915	1.415892	1.588656	1.782502	2
6	0.189125	0.212202	0.2380945	0.2671465	0.2997435	0.3363175	0.377354	0.423398	0.4750605	0.533027	0.5980665	0.6710415	0.7529205	0.8447905	0.9478705	1.0635285	1.193299	1.3389035	1.502274	1.685579	1.891251
7	0	0	0	0	0.022381	0.020935	0.024605	0.033813	0.041434	0.048281	0.074413	0.085952	0.092505	0.107985	0.127884	0.155551	0.19129	0.236187	0.288103	0.347291	0.409729
8	0	0	0	0	0.035818	0.071623	0.100518	0.129829	0.152347	0.176126	0.198758	0.221347	0.246006	0.273445	0.305077	0.341729	0.382653	0.428077	0.474658	0.52166	0.565192
9	0	0	0	0.008854	0.027006	0.044636	0.078175	0.093458	0.101434	0.111873	0.123551	0.139374	0.162502	0.194841	0.238466	0.294523	0.361316	0.43854	0.519663	0.602344	0.678416

	BT	BU	BV	BW	BX	BY	BZ	CA	CB	CC	CD	CE	CF	CG	CH	CI	CJ	CK	CL
1																			
2																			
3																			
4																			
5	2.244037	2.517851	2.825075	3.169786	3.556559	3.990525	4.477442	5.023773	5.636766	6.324555	7.096268	7.962143	8.933672	10.023745	11.246826	12.619147	14.158916	15.886565	17.825019
6	2.1220185	2.380944	2.671463	2.9974305	3.3631725	3.773542	4.2339835	4.7506075	5.3302695	5.9806605	6.7104115	7.5292055	8.4479075	9.4787085	10.6352855	11.9329865	13.3890315	15.0227405	16.855792
7	0.473834	0.536131	0.593434	0.644004	0.684661	0.715619	0.73533	0.745036	0.745499	0.738303	0.724973	0.707495	0.686783	0.664732	0.641283	0.617355	0.592608	0.567532	0.543228
8	0.604141	0.63685	0.662948	0.68344	0.699153	0.712393	0.724504	0.737382	0.751602	0.767307	0.783939	0.800013	0.814657	0.826125	0.833864	0.837092	0.836159	0.832283	0.827898
9	0.744309	0.795367	0.829438	0.846998	0.849865	0.841682	0.827213	0.810549	0.795368	0.783625	0.775955	0.77178	0.769635	0.768033	0.765414	0.760786	0.75388	0.745706	0.738675

Filename: TI008 averaged data.xlsm
Worksheet: psd

	CM	CN	CO	CP	CQ	CR	CS	CT	CU	CV	CW	CX	CY	CZ	DA	DB	DC	DD
1																		
2																		
3																		
4																		
5	20	22.440369	25.178508	28.250751	31.697864	35.565588	39.905246	44.774423	50.237729	56.367659	63.245553	70.962678	79.621434	89.336718	100.237447	112.468265	126.191469	141.589157
6	18.9125095	21.2201845	23.8094385	26.7146295	29.9743075	33.631726	37.735417	42.3398345	47.506076	53.302694	59.806606	67.1041155	75.292056	84.479076	94.7870825	106.352856	119.329867	133.890313
7	0.521085	0.504095	0.494317	0.494736	0.507646	0.53447	0.574882	0.626896	0.684228	0.740879	0.786334	0.815107	0.822661	0.81307	0.799748	0.806611	0.86947	1.029719
8	0.82645	0.83157	0.846538	0.872818	0.911561	0.960654	1.016095	1.071652	1.117084	1.142568	1.137383	1.094793	1.014357	0.90331	0.78052	0.676468	0.630304	0.69197
9	0.736454	0.74303	0.762041	0.794994	0.843248	0.904442	0.974439	1.04711	1.112017	1.160428	1.18134	1.16955	1.124281	1.053389	0.975298	0.91896	0.922426	1.030679

Filename: TI008 averaged data.xlsm
Worksheet: psd

	DE	DF	DG	DH	DI	DJ	DK	DL	DM	DN	DO	DP	DQ	DR	DS	DT	DU
1																	
2																	
3																	
4																	
5	158.865647	178.250188	200	224.403691	251.785082	282.507509	316.978638	355.655882	399.052463	447.744228	502.377286	563.676586	632.455532	709.626778	796.214341	893.367184	1002.374467
6	150.227402	168.5579175	189.125094	212.2018455	238.0943865	267.1462955	299.7430735	336.31726	377.3541725	423.3983455	475.060757	533.026936	598.066059	671.041155	752.9205595	844.7907625	947.8708255
7	1.33866	1.821112	2.504478	3.367116	4.356294	5.396	6.351806	7.122359	7.557374	7.588242	7.170656	6.328664	5.132295	3.756677	2.268327	0.797891	0.049916
8	0.913819	1.327236	1.959758	2.791359	3.767898	4.811149	5.78315	6.579153	7.042967	7.102667	6.705562	5.865011	4.658346	3.19789	1.623507	0.347821	0
9	1.292619	1.731657	2.36837	3.174164	4.089365	5.033065	5.873897	6.513293	6.818266	6.729963	6.219348	5.315218	4.091636	2.699431	1.216021	0.170246	0.016065

Filename: TI008 averaged data.xlsm
Worksheet: psd

	DV	DW	DX	DY	DZ	EA	EB
1							
2							
3							
4							**Operator Notes**
5	1124.68265	1261.914689	1415.891569	1588.656469	1782.501876	2000	
6	1063.528559	1193.29867	1338.903129	1502.274019	1685.579173	1891.250938	
7	0	0	0	0	0	0	Average of 12 measurements from 52578-TI-037.mea
8	0	0	0	0	0	0	Average of 12 measurements from 52578-TI-037.mea
9	0	0	0	0	0	0	Average of 12 measurements from 52578-TI-037.mea

	A	B	C	D	E	F	G	H	I	J	K	L	M
1	**Cumulative PSD Data - Supplemental Package**												
2	Source:	"52578-TI-037.mea"											
3													
4	**Record number**	**Sample Name**	**Sample source type**	**Sample bulk lot ref**	**SOP Name**	**Operator name**	**Measurement date and time**	**Analysis date and time**	**Particle name**	**Particle refractive index**	**Particle absorption index**	**Dispersant name**	**Dispersant refractive index**
5													
6	69	TI008-Unsonicated Average		52578-TI-037		D3M966	Wednesday, June 15, 2011 1:00:40 PM	Wednesday, June 15, 2011 1:00:41 PM	Iron III Oxide_1	2.42	1	Water	1.33
7	70	TI008-Sonicated Average		52578-TI-037		D3M966	Wednesday, June 15, 2011 1:03:41 PM	Wednesday, June 15, 2011 1:03:43 PM	Iron III Oxide_1	2.42	1	Water	1.33
8	71	TI008-Post Sonication Average		52578-TI-037		D3M966	Wednesday, June 15, 2011 1:06:07 PM	Wednesday, June 15, 2011 1:06:09 PM	Iron III Oxide_1	2.42	1	Water	1.33

Filename: TI008 averaged data.xlsm
Worksheet: cumulative

	N	O	P	Q	R	S	T	U	V	W	X	Y	Z	AA	AB	AC	AD	AE	AF	AG
1																				
2																				
3																				
4	**Accessory name**	**Analysis model**	**Start result channel size**	**Last result channel size**	**Result emulation**	**Obscuration**	**Residual**	**Concentration**	**Span**	**Result transform type**	**D [4, 3] - Volume weighted mean**	**Uniformity**	**Specific surface area**	**D [3, 2] - Surface weighted mean**	**d (0.1)**	**d (0.5)**	**d (0.9)**	**Result Below User Sizes (Sizes in um)**		
5																		0.02	0.02244	0.025179
6	Hydro 2000µP (A)	General purpose	0.02	2000	Off	8.05	1.42	0.0438	1.902	Volume	302.242	0.576	0.308	19.489	7.515	304.954	587.612	0	0	0
7	Hydro 2000µP (A)	General purpose	0.02	2000	Off	11.12	1.655	0.0332	2.019	Volume	268.982	0.668	0.505	11.883	4.958	275.765	561.607	0	0	0
8	Hydro 2000µP (A)	General purpose	0.02	2000	Off	11.67	1.576	0.0383	2.093	Volume	255.79	0.689	0.471	12.729	4.507	256.157	540.55	0	0	0

	AH	AI	AJ	AK	AL	AM	AN	AO	AP	AQ	AR	AS	AT	AU	AV	AW	AX	AY	AZ	BA	BB	BC	BD
1																							
2																							
3																							
4																							
5	0.028251	0.031698	0.035566	0.039905	0.044774	0.050238	0.056368	0.063246	0.070963	0.079621	0.089337	0.100237	0.112468	0.126191	0.141589	0.158866	0.17825	0.2	0.224404	0.251785	0.282508	0.316979	0.355656
6	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0.022381	0.043316
7	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0.035818	0.107442
8	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0.008854	0.03586	0.080496

	BE	BF	BG	BH	BI	BJ	BK	BL	BM	BN	BO	BP	BQ	BR	BS	BT	BU	BV	BW	BX	BY	BZ
1																						
2																						
3																						
4																						
5	0.399052	0.447744	0.502377	0.563677	0.632456	0.709627	0.796214	0.893367	1.002374	1.124683	1.261915	1.415892	1.588656	1.782502	2	2.244037	2.517851	2.825075	3.169786	3.556559	3.990525	4.477442
6	0.067921	0.101734	0.143167	0.191448	0.265861	0.351813	0.444318	0.552303	0.680187	0.835738	1.027028	1.263215	1.551318	1.898609	2.308338	2.782171	3.318303	3.911737	4.55574	5.240402	5.95602	6.69135
7	0.20796	0.33779	0.490137	0.666263	0.865021	1.086367	1.332374	1.605819	1.910895	2.252624	2.635277	3.063355	3.538012	4.059672	4.624864	5.229006	5.865856	6.528804	7.212244	7.911397	8.62379	9.348294
8	0.158671	0.252128	0.353562	0.465435	0.588987	0.728361	0.890863	1.085705	1.32417	1.618694	1.980009	2.418549	2.938212	3.540556	4.218973	4.963282	5.758649	6.588087	7.435086	8.284951	9.126633	9.953846

	CA	CB	CC	CD	CE	CF	CG	CH	CI	CJ	CK	CL	CM	CN	CO	CP	CQ	CR	CS	CT
1																				
2																				
3																				
4																				
5	5.023773	5.636766	6.324555	7.096268	7.962143	8.933672	10.023745	11.246826	12.619147	14.158916	15.886565	17.825019	20	22.440369	25.178508	28.250751	31.697864	35.565588	39.905246	44.774423
6	7.436387	8.181886	8.920189	9.645162	10.352657	11.03944	11.704172	12.345455	12.96281	13.555418	14.12295	14.666178	15.187263	15.691358	16.185675	16.680411	17.188058	17.722528	18.29741	18.924306
7	10.085676	10.837277	11.604585	12.388523	13.188536	14.003194	14.829318	15.663182	16.500273	17.336433	18.168716	18.996614	19.823063	20.654633	21.501172	22.37399	23.28555	24.246204	25.262299	26.333951
8	10.764395	11.559763	12.343388	13.119343	13.891123	14.660758	15.42879	16.194204	16.95499	17.70887	18.454577	19.193252	19.929706	20.672736	21.434777	22.22977	23.073018	23.977461	24.9519	25.99901

	CU	CV	CW	CX	CY	CZ	DA	DB	DC	DD	DE	DF	DG	DH	DI	DJ	DK	DL
1																		
2																		
3																		
4																		
5	50.237729	56.367659	63.245553	70.962678	79.621434	89.336718	100.237447	112.468265	126.191469	141.589157	158.865647	178.250188	200	224.403691	251.785082	282.507509	316.978638	355.655882
6	19.608534	20.349413	21.135746	21.950853	22.773515	23.586585	24.386333	25.192944	26.062414	27.092133	28.430793	30.251904	32.756382	36.123498	40.479792	45.875792	52.227598	59.349957
7	27.451035	28.593603	29.730986	30.825779	31.840136	32.743445	33.523965	34.200433	34.830737	35.522707	36.436526	37.763761	39.723519	42.514878	46.282776	51.093925	56.877075	63.456229
8	27.111027	28.271455	29.452795	30.622346	31.746627	32.800016	33.775314	34.694274	35.6167	36.647378	37.939997	39.671654	42.040024	45.214187	49.303552	54.336617	60.210514	66.723806

Filename: TI008 averaged data.xlsm
Worksheet: cumulative

	DM	DN	DO	DP	DQ	DR	DS	DT	DU	DV	DW	DX	DY	DZ	EA
1															
2															
3															
4															
5	399.052463	447.744228	502.377286	563.676586	632.455532	709.626778	796.214341	893.367184	1002.374467	1124.68265	1261.914689	1415.891569	1588.656469	1782.501876	2000
6	66.907331	74.495573	81.666229	87.994893	93.127188	96.883865	99.152192	99.950084	100	100	100	100	100	100	100
7	70.499196	77.601863	84.307424	90.172436	94.830782	98.028671	99.652179	100	100	100	100	100	100	100	100
8	73.542072	80.272035	86.491383	91.806601	95.898237	98.597668	99.813689	99.983935	100	100	100	100	100	100	100

Filename: TI008 averaged data.xlsm
Worksheet: cumulative

	EB
1	
2	
3	
4	**Operator Notes**
5	
6	Average of 12 measurements from 52578-TI-037.mea
7	Average of 12 measurements from 52578-TI-037.mea
8	Average of 12 measurements from 52578-TI-037.mea

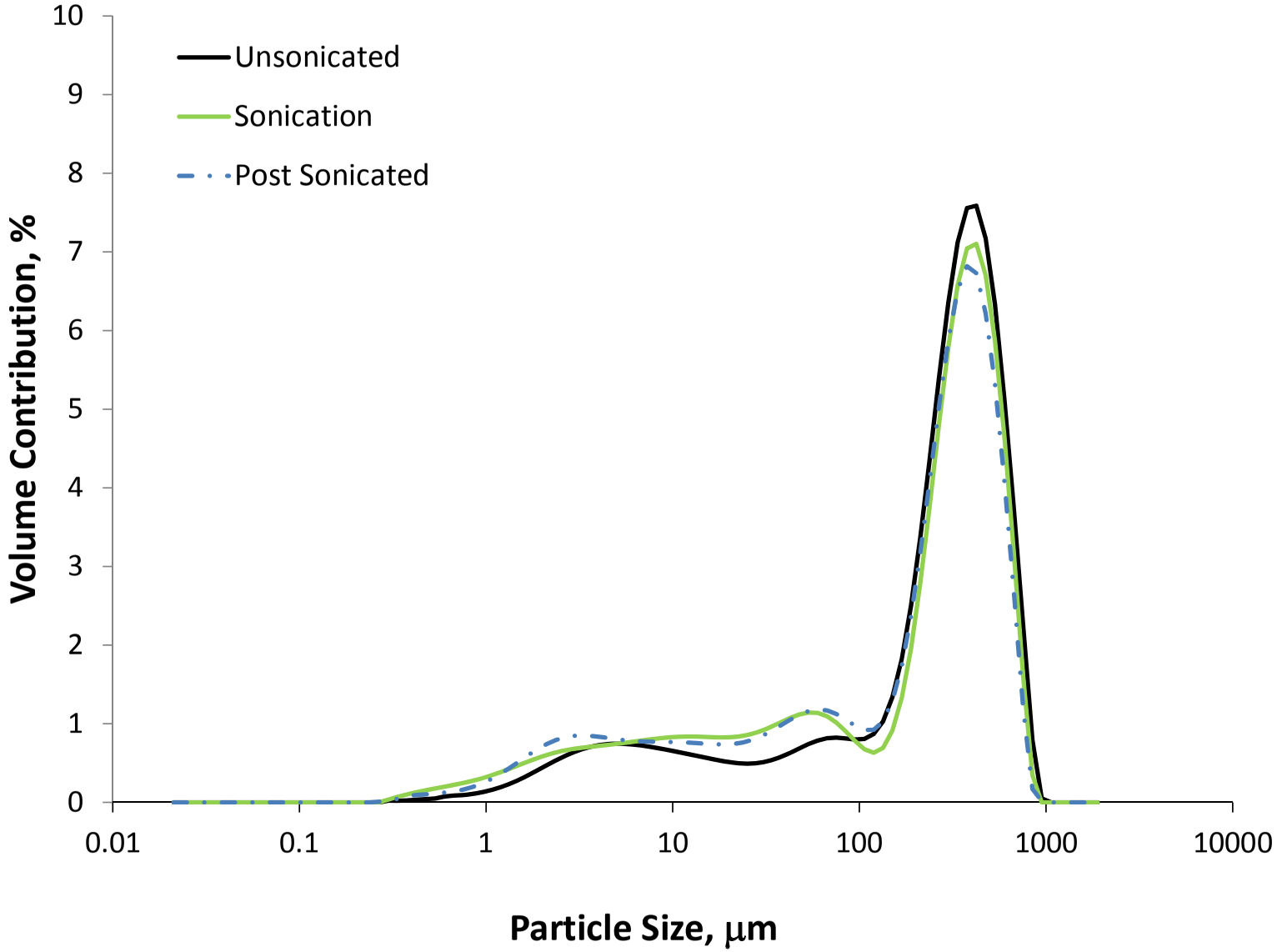
Filename: TI008 averaged data.xlsm
Worksheet: cumulative table

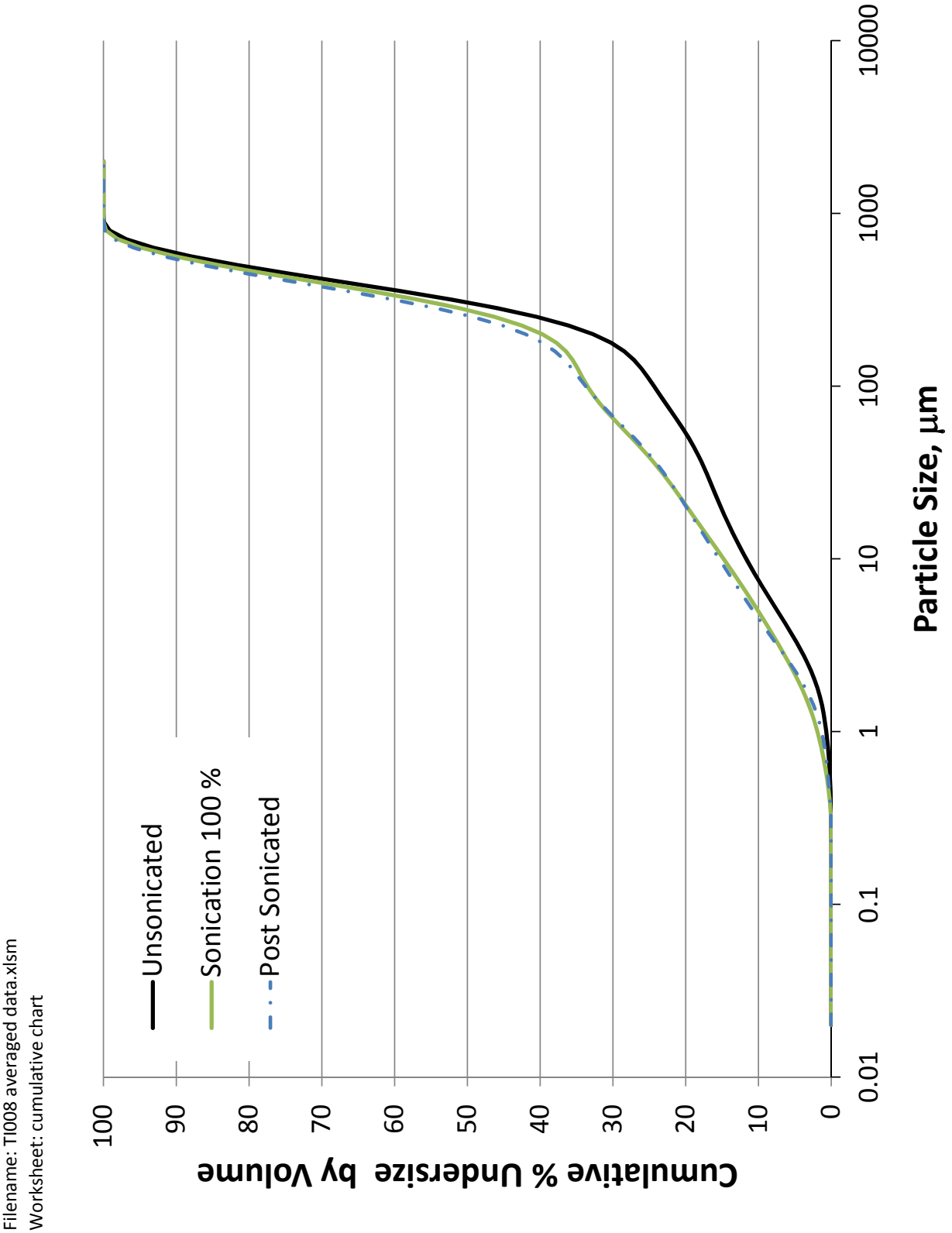
	A	B	C	D
1	**Cumulative PSD Data in Tabular Form - Supplemental Package**			
2	Source:	"52578-TI-037.mea"		
3				
4	**Record number:**	69	70	71
5	**Sample name:**	TI008-Unsonicated Average	TI008-Sonicated Average	TI008-Post Sonication Average
6	**Analysis model:**	General purpose	General purpose	General purpose
7	**Particle refractive index:**	2.42	2.42	2.42
8	**Pump speed, RPM:**	2000	2000	2000
9	**Result Below Size, µm:**	**Cumulative Percent Undersize, % Volume**		
10	0.05	0	0	0
11	0.5	0.141	0.483	0.349
12	1	0.677	1.904	1.319
13	2.5	3.283	5.826	5.708
14	5	7.406	10.055	10.731
15	10	11.691	14.812	15.413
16	20	15.187	19.823	19.93
17	40	18.31	25.284	24.973
18	60	20.771	29.214	28.912
19	80	22.807	31.88	31.791
20	100	24.37	33.509	33.756
21	125	25.986	34.778	35.538
22	150	27.715	35.942	37.253
23	175	29.922	37.516	39.358
24	200	32.756	39.724	42.04
25	300	49.081	54	57.308
26	400	67.065	70.647	73.683
27	500	81.387	84.048	86.253
28	600	90.945	92.868	94.194
29	700	96.507	97.726	98.348
30	800	99.214	99.689	99.833
31	1000	100	100	100

Filename: TI008 averaged data.xlsm
Worksheet: graph information

	A	B
1	**Summary of Graphs and Graph Reference Cells**	
2		
3	**Stand Alone Charts**	
4		
5	Graph #:	1
6	Worksheet Label:	PSD chart
7	Number of Data Series:	3
8	Series 1 Formula:	=SERIES("Unsonicated",psd!$AF$6:$EA$6,psd!$AF$7:$EA$7,1)
9	Series 2 Formula:	=SERIES("Sonication",psd!$AF$6:$EA$6,psd!$AF$8:$EA$8,2)
10	Series 3 Formula:	=SERIES("Post Sonicated",psd!$AF$6:$EA$6,psd!$AF$9:$EA$9,3)
11		
12		
13	Graph #:	2
14	Worksheet Label:	cumulative chart
15	Number of Data Series:	3
16	Series 1 Formula:	=SERIES("Unsonicated",cumulative!$AE$5:$EA$5,cumulative!$AE$6:$EA$6,1)
17	Series 2 Formula:	=SERIES("Sonication 100 %",cumulative!$AE$5:$EA$5,cumulative!$AE$7:$EA$7,2)
18	Series 3 Formula:	=SERIES("Post Sonicated",cumulative!$AE$5:$EA$5,cumulative!$AE$8:$EA$8,3)
19		

Filename: TI008 averaged data.xlsm
Worksheet: PSD chart





Filename: TI008 averaged data.xlsm
Worksheet: cumulative chart

August 3, 2011

Malvern Validation Initiative

Quality Audit Standard for the Hydro 2000µP Small Volume Sample dispersion unit QAS3004

Pack of 10 100mg one-shot bottles of 13 to 130µm polydisperse glass-bead transfer standard, Part No.CRM0020.

MRK0823-04
07-2009

Malvern

www.malvern.com

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Introduction

Malvern's QAS3004 Quality Audit Standard (bottle part number CRM0020) has been produced to provide users of Malvern particle size analysers with a reliable, one-shot, polydisperse transfer standard that enables them to check the performance of the Hydro 2000µP sample dispersion unit on a regular basis.

This standard replaces Malvern's QAS2005 standard, which was first launched in 1999. QAS2005 was produced from a master batch of 250kg of glass beads (Batch 01), supplies of which were finally exhausted after 6 years. QAS3004 is produced from a new master batch of 1000kg of glass beads (Batch 02), representing the world's largest batch of particle size standards ever produced. The size of this master batch is designed to give users the assurance that QAS3004 will be available as a sizing reference for even longer than QAS2005.

Compliance with International Standards

As with all Malvern Quality Audit Standards, QAS3004 complies with the recommendations of ISO13320, USP <429> and EP 2.9.31 relating to the validation of laser diffraction systems using certified or standard reference materials. The glass bead particles present within QAS3004 are spherical, cover a decade in size and have precisely defined optical properties. In addition, the standard is used in conjunction with a clear measurement procedure, as outlined in this datasheet. As such, QAS3004 provides a reliable means of checking and documenting the consistent operation of a laser diffraction system, as part of FDA or other international laboratory accreditation schemes (e.g. ISO, NAMAS, and IAF).

Sample Variability

Polydisperse particle sizing standards are prone to segregation during transit, which can lead to sampling errors. To overcome this, Malvern's Quality Audit Standards are produced by one of Europe's leading suppliers of particle size standards, Whitehouse Scientific Ltd., who have used an extremely efficient riffle-splitting process to ensure that each one-shot sample is representative of the entire batch. Random sampling of QAS3004 bottles across the entire batch has shown that the relative standard deviation for the median (Dv50) particle size is of the order of 0.85%. This confirms that, as long as the entire contents of the bottle are used during a measurement in accordance with the instructions included on this datasheet, reproducible results can be obtained.

Shelf Life and Batch Numbering

Malvern's Quality Audit Standards are made of inert glass beads. Since they are also stored in sealed containers, they have an indefinite shelf life. Because of this, it has been possible to provide many years of continuous supply from a single master batch. For this reason, the only batch number for QAS3004 is 02.

Traceability

The pass/fail specifications set for Malvern's Quality Audit Standards have been developed via a fully documented programme of testing using a reference Mastersizer 2000 laser diffraction system. This reference system has been verified using NIST-traceable polystyrene latex standards. As such, although these standards are transfer standards, they are indirectly traceable to NIST.

Dispersant Selection

The Hydro 2000µP dispersion unit was designed primarily for the measurement of small sample quantities in organic dispersants. For this reason, the decision was made to characterise QAS3004 using Propan-2-ol as a dispersant, as this is miscible with most organic dispersants, as well as water. No specifications will be issued for the use of this standard in water.

Establishing Pass/Fail criteria and measurement procedures

An extensive and ongoing programme of dispersion unit testing is carried out by Malvern in order to characterize each Quality Audit Standard and establish the pass/fail criteria referenced on this datasheet. As testing continues, Malvern constantly assesses the average measurement values obtained over the entire population of units. As the population increases, slight adjustments to the pass/fail criteria may be required in order to ensure that these accurately reflect the expected performance of the units currently in field, as well as those leaving Malvern's production facility. Changes may also be made to the measurement procedure in order to ensure robust measurements can be made.

Given the above, it is important that the latest version of this datasheet is used, especially when carrying out an annual system OQ or PV. In case of doubt, the latest version number (MRK823-nn) can be verified by visiting Malvern's website. If there is any disagreement between the datasheet and the latest PV certificate and specification, the PV certificate and specification should be considered to take precedence over the datasheet, since these are subject to strict change control and are automatically updated and distributed to the Malvern support network.

Result Continuity

All the data used to set the original pass/fail criteria for the Batch 02 standards were generated using sample dispersion units which had first been tested using Batch 01 standards. This means that a unit which would have passed the earlier standard should also pass using Batch 02.

Target Value Specifications

In order to bring the specification for QAS3004 into line with ISO13320, USP<429> and EP 2.9.31, the limits for the Dv10 and Dv90 have been adjusted to ±5%, instead of ±6% as previously specified. In addition, the median values and limits for all Quality Audit Standards have been standardised to a precision of three decimal places. In order to achieve this, the source data used in setting the limits was reanalysed. This has caused a fractional change in the target values for QAS3004.

An analysis of the original measurement data used in setting the limits for QAS3004 suggests that the above changes should not cause the number of failed measurements to increase significantly.

Expected Results

The expected limits for the Hydro 2000µP dispersion units are set at ±3% for the Dv50 and ± 5% for the Dv10 and Dv90.

	Dv10 / µm	Dv50 / µm	Dv90 / µm
Lower Limit:	37.244	60.491	85.450
Target Value	39.204	62.362	89.947
Upper Limit:	41.164	64.233	94.444

Malvern Instruments Limited

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PNNL-20650, Rev. 2

Material Safety Data Sheet (MSDS)

1. IDENTIFICATION OF THE SUBSTANCE AND THE COMPANY /UNDERTAKING

Product Name: Malvern Quality Audit Standards
Cas-Number: 65997-17-3
EINECS-Number: 266046O
Product Code: 0390
Synonyms: GLASS BEADS
Use/description of product: Soda Lime Glass
Company: Whitehouse Scientific Ltd. Whitchurch Road, Waverton, Chester, CH3 7PB, England
Tel: +44 (0) 1244 332626
Fax: +44 (0) 1244 335098
email: info@whitehousescientific.com

2. COMPOSITION / INFORMATION ON INGREDIENTS

Hazardous Ingredients: SODA LIME GLASS 100.000%
EINECS: 266046O
CAS: 65997-17-3

3. HAZARDS IDENTIFICATION

Main Hazards: No significant hazard.

4. FIRST AID MEASURES (SYMPTOMS)

Skin contact: There may be mild irritation at the site of contact.
Eye contact: There may be irritation and redness.
Ingestion: No Symptoms.
Inhalation: Exposure may cause coughing or wheezing.

4. FIRST AID MEASURES (ACTIONS)

Skin contact: Wash immediately with plenty of soap and water.
Eye contact: Bathe the eye with running water for at least 15 minutes. Also rinse under the eyelids. If irritation persists, consult a specialist.
Ingestion: Wash out mouth with water.
Inhalation: Remove to fresh air.

5. FIRE FIGHTING MEASURES

Extinguishing Media: Non-Flammable substance, not applicable. Suitable extinguishing media for the surrounding fire
Protection of fire-fighters: Should be used. Wear self-contained breathing apparatus. Wear protective clothing to prevent contact with skin and eyes.

6. ACCIDENTAL RELEASE MEASURES

Personal Precautions: Refer to section 8 below for personal protection details.
Clean-Up Procedure: Transfer to a suitable container. Material can create slippery conditions underfoot. Avoid creating dust.

7. HANDLING AND STORAGE

Handling Requirements: Ensure that there is sufficient ventilation of the area. Avoid direct contact with the substance. Avoid the formation or spread of dust in the air.
Storage Conditions: Store in cool, well ventilated area. Keep bottles tightly closed.

8. EXPOSURE CONTROLS / PERSONAL PROTECTION

Hazardous ingredients: SODA LIME GLASS
TWA (8hr exposure limit): 5mg/m3 (OES)
Engineering Methods: Ensure that there is exhaust ventilation of the area.
Respiratory Protection: Respiratory protective device with particle filter.
Hand protection: Protective gloves.
Eye Protection: Safety glasses. Ensure eye bath is to hand.
Skin Protection: Protective clothing.

9. PHYSICAL AND CHEMICAL PROPERTIES

State: Solid.
Colour: White.
Odour: Odourless.
Melting Point/Range°C: Approximately 730°C
Relative Density: 2.6 g/cm3 (20°C)

10. STABILITY AND REACTIVITY

Stability: Stable under normal conditions.

11. TOXICOLOGICAL INFORMATION

Chronic Toxicity: Overexposure to dust may cause irritation of eyes and throat.
Routes of Exposure: No data available.

12. ECOLOGICAL INFORMATION

Mobility: No data available.
Persistence and degradability: No data available.
Bioaccumulative Potential: No data available.

13. DISPOSAL CONSIDERATIONS

Disposal Operations: Contact waste disposal services.
Disposal of Packaging:Contact waste disposal services.
NB: The user's attention is drawn to the possible existence of regional or national regulations regarding disposal.

14. TRANSPORTATION INFORMATION

ADR / RID
UN No: -
Shipping Name: "NOT SUBJECT TO ADR"
IMDG / IMO
UN No: -
IATA / ICAO
UN No: -

15. REGULATORY INFORMATION

Hazard Symbols: No significant hazard.
Note: The regulatory information given above only indicates the principal regulations specifically applicable to the product described in the safety data sheet. The user's attention is drawn to the possible existence of additional provisions which complete these regulations. Refer to all applicable national, international and local regulations or provisions.

16. OTHER INFORMATION

Other Information: Complies with Directives (2001/58/EC), (1999/45/EC), (91/155/EEC), (67/548/EEC) as amended and Chemicals (hazard information and packaging for supply) 2002 (CHIP3) Regulation, EH40.

Legal Disclaimer: The information contained in this safety Data Sheet is correct to the best of our knowledge, information and belief at the date of its publication. The information given is designed only as guidance for safe handling, use, processing, storage, transportation, disposal and release and it is not to be considered a warranty or quality specification. The information relates only to the specific material designated and may not be valid for such material used in combination with any other materials or in any process unless specified in the text.

Malvern Standard Certificate - Page 2 of 2

Cleaning Routine

1) Set the measurement mode to 'Add Sample' in the manual measurement mode dialogue and note the obscuration level.
2) Stop the pump and open the drain. Inject 20mls of dispersant directly into the dispersion unit via the sample well on the top of the unit.
3) Close the drain and inject 25mls of dispersant into the dispersion unit via the dispersion inlet tube on the front of the unit.
4) Turn on the pump and check the obscuration figure on the screen. It should have halved compared to the previous reading.
5) Repeat steps 2-4 until the obscuration has dropped to zero.

PROJECT 52578 – SURVEILLANCE REPORT

Number: SR-52578-2011-030

PLANNING

Topic/Issue to be evaluated: Surveillance of Completed K Basin TI, 52578-TI037, Rev. 0, Test Instruction for *Particle Size Distribution Measurements for K West Basin Consolidated Container Composite SCS-CON-210, Minus 500 micron Particle Size Fraction Samples.*

This surveillance was undertaken to evaluate completed test instruction (TI), 52578-TI037, Test Instruction for *Particle Size Distribution Measurements for K West Basin Consolidated Container Composite SCS-CON-210, Minus 500 micron Particle Size Fraction Samples,* associated supporting information and spreadsheets, and the completed report, *Particle Size Analysis Performed Under Test Instruction 52578-TI-037, Particle Size Distribution Measurements for K West Basin Consolidated Container Composite SCS-CON-210, Using Minus 500 Micron Particle Size Fraction Samples.* The report was reviewed concurrently with TI037, but the report review will be documented via a Document Review Record (DRR) form. TI037 was used between 6/15/2011 and 7/7/2011. TI037 directs the performance of particle size distribution measurements for 2 samples using procedure, RPL-COLLOID-01, Rev. 1, *Particle Size Analysis Using Malvern MS2000.* Carolyn Burns signed off TI steps. Richard Daniel performed a Technical Review of the TI.

PERFORMANCE

Date Initiated: 15 June 2011 **Date Completed**: 7 July 2011	**Location:** RPL
Date Received: 8 July 2011 **Date Reviewed:** 11 July 2011	**DRAFT Report Issued:** 11 July 2011 **FINAL Report Issued**: 21 July 2011
Contacts: Carolyn Burns, Clark Carlson, Richard Daniel and Sandy Fiskum	**Org Code:** PE 137, D9H63
Surveillance Team: Lisa Middleton	

PRELIMINARY INFORMATION

Source of Requirement(s):

- KBC-33786, Rev. 2, December 2009; Quality Assurance Project Plan/Sampling and Analysis Plan (QAPjP/SAP): Quality Assurance Project Plan/Sampling and Analysis Plan for Containerized KW Settler Sludge
- Data Quality Objectives for Sampling and Analysis of K Basin Sludge HNF-36985, Rev. 3, July 2009

Other Applicable Documents:

- RPL-OP-001, Rev. 9, *Routine Research Operations*
- ADM-RSEG-BALANCES, Rev. 0, *Balance Calibration Verification*

Purpose/Scope:

The 52578 Project QE staff are required via the TI to perform a completeness review. The document was reviewed to evaluate that all entries were recorded properly, the errors were corrected appropriately and when needed, that justification was present to describe data corrections, and that the attachments to the TI were at the stage to become a project record. When issues were found, the cognizant staff were contacted to resolve issues prior to completion of the review.

Approach:

The TI package submitted for review consisted of the following:

1. Completed TI, 52578-TI037, Rev. 0, Test Instruction for *Particle Size Distribution Measurements for K West Basin Consolidated Container Composite SCS-CON-210, Minus 500 micron Particle Size Fraction Samples* (pp. 1-19)
2. DRR & Calculation Review Sheet, both signed by Richard Daniel on 7/7/11
3. Excel File: 52578-TI037 (Reviewed)
4. PDF version of Excel file with cover page & review form
5. Word File: 52578-TI037 Writeup (ITR) Rev B (Report: *Particle Size Analysis Performed Under Test Instruction 52578-TI-037, Particle Size Distribution Measurements for K West Basin Consolidated Container Composite SCS-CON-210, Using Minus 500 Micron Particle Size Fraction Samples)*
6. PDF of Word file listed above with reviewer mark-up.

M&E Used:

Description	ID #/Serial Number	Dates Used
Malvern Mastersizer 2000	MAL100406	6/15/11

Procedure Used:

RPL-COLLOID-01, Rev. 1, *Particle Size Analysis Using Malvern MS2000*

RESULTS

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.

Note: All concerns identified in the review were addressed; a comment resolution meeting was not necessary.

PROJECT 52578 – SURVEILLANCE REPORT

Number: SR-52578-2011-030

Before the completed TI was signed off for QE review, it was determined that all comments/concerns were addressed. There were no outstanding identified findings or observations.

Issues of concern are summarized below:

1. Line-outs need to be initialed & dated on pages 8, 16 & 17. Completed, no further action required; closed.

2. The Result Analysis Reports inserted into the TI need to have page #'s added to them (e.g. 16A, 17A, 17B…). Completed, no further action required; closed.

CORRECTIVE ACTION

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

Surveillance Performed By:

Lisa Middleton, Quality Engineer DSC 9-21-11 Date: 9/21/11

DISTRIBUTION: Carolyn Burns, Clark Carlson, Deborah Coffey, Richard Daniel, Sandy Fiskum, Matt Fountain, Rick Shimskey, and Andy Schmidt

PROJECT 52578 – SURVEILLANCE REPORT

Number: SR-52578-2011-030 **Rev. 1**

PLANNING

Topic/Issue to be evaluated: Surveillance of Completed K Basin TI, 52578-TI037, Rev. 0, Test Instruction for *Particle Size Distribution Measurements for K West Basin Consolidated Container Composite SCS-CON-210, Minus 500 micron Particle Size Fraction Samples.*

This report has been revised to include the addition of a data reduction spreadsheet that was added to the report and was not included in the report when submitted for review.

This surveillance was undertaken to evaluate completed test instruction (TI), 52578-TI037, Test Instruction for *Particle Size Distribution Measurements for K West Basin Consolidated Container Composite SCS-CON-210, Minus 500 micron Particle Size Fraction Samples,* associated supporting information and spreadsheets, and the completed report, *Particle Size Analysis Performed Under Test Instruction 52578-TI-037, Particle Size Distribution Measurements for K West Basin Consolidated Container Composite SCS-CON-210, Using Minus 500 Micron Particle Size Fraction Samples.* The report was reviewed concurrently with TI037, but the report review will be documented via a Document Review Record (DRR) form. TI037 was used between 6/15/2011 and 7/7/2011. TI037 directs the performance of particle size distribution measurements for 2 samples using procedure, RPL-COLLOID-01, Rev. 1, *Particle Size Analysis Using Malvern MS2000.* Carolyn Burns signed off TI steps. Richard Daniel performed a Technical Review of the TI.

PERFORMANCE

Date Initiated: 15 June 2011 **Date Completed**: 7 July 2011	**Location:** RPL
Date Received: 8 July 2011 **Date Reviewed:** 11 July 2011	**DRAFT Report Issued:** 11 July 2011 **FINAL Report Issued**: 21 July 2011 **REVISED Report Issued:** 3 August 2011
Contacts: Carolyn Burns, Clark Carlson, Richard Daniel and Sandy Fiskum	**Org Code:** PE 137, D9H63
Surveillance Team: Lisa Middleton	

PRELIMINARY INFORMATION

Source of Requirement(s):

- KBC-33786, Rev. 2, December 2009; Quality Assurance Project Plan/Sampling and Analysis Plan (QAPjP/SAP): Quality Assurance Project Plan/Sampling and Analysis Plan for Containerized KW Settler Sludge
- Data Quality Objectives for Sampling and Analysis of K Basin Sludge HNF-36985, Rev. 3, July 2009

Other Applicable Documents:

- RPL-OP-001, Rev. 9, *Routine Research Operations*

PROJECT 52578 – SURVEILLANCE REPORT

Number: SR-52578-2011-030 **Rev. 1** Page 2 of 4

- ADM-RSEG-BALANCES, Rev. 0, *Balance Calibration Verification*

Purpose/Scope:

The 52578 Project QE staff are required via the TI to perform a completeness review. The document was reviewed to evaluate that all entries were recorded properly, the errors were corrected appropriately and when needed, that justification was present to describe data corrections, and that the attachments to the TI were at the stage to become a project record. When issues were found, the cognizant staff were contacted to resolve issues prior to completion of the review.

Approach:

The TI package submitted for review consisted of the following:

1. Completed TI, 52578-TI037, Rev. 0, Test Instruction for *Particle Size Distribution Measurements for K West Basin Consolidated Container Composite SCS-CON-210, Minus 500 micron Particle Size Fraction Samples* (pp. 1-19)
2. DRR & Calculation Review Sheet, both signed by Richard Daniel on 7/7/11
3. Excel File: 52578-TI037 (Reviewed).xlsm with the following tabs:
   - Summary Page
   - Performance Checks
   - PSD
   - Cumulative
   - Cumulative Tables
   - Percentiles
   - TI-008-SP PSD Chart
   - TI-008-SQ PSD Chart
   - TI-008-SP CUM Chart
   - TI-008-SQ CUM Chart
4. PDF version of Excel file with cover page & review form
5. Word File: 52578-TI037 Writeup (ITR) Rev B.doc (Report: *Particle Size Analysis Performed Under Test Instruction 52578-TI-037, Particle Size Distribution Measurements for K West Basin Consolidated Container Composite SCS-CON-210, Using Minus 500 Micron Particle Size Fraction Samples)*
6. PDF of Word file listed above with reviewer mark-up.
7. **A new Excel spreadsheet was submitted for review on 2 August 2011 (TI008 average data.xls), containing the following tabs:**
   - **Summary**
   - **PSD**
   - **Cumulative**
   - **Percentiles**
   - **Cumulative Chart**
   - **Cumulative Table**
   - **PSD Chart**

PROJECT 52578 – SURVEILLANCE REPORT

Number: SR-52578-2011-030 **Rev. 1**

M&E Used:

Description	ID #/Serial Number	Dates Used
Malvern Mastersizer 2000	MAL100406	6/15/11

Procedure Used:

RPL-COLLOID-01, Rev. 1, *Particle Size Analysis Using Malvern MS2000*

RESULTS

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.

Note: All concerns identified in the review were addressed; a comment resolution meeting was not necessary.

Before the completed TI was signed off for QE review, it was determined that all comments/concerns were addressed. There were no outstanding identified findings or observations.

Issues of concern are summarized below:

1. Line-outs need to be initialed & dated on pages 8, 16 & 17. Completed, no further action required; closed.

2. The Result Analysis Reports inserted into the TI need to have page #'s added to them (e.g. 16A, 17A, 17B…). Completed, no further action required; closed.

3. **The Excel file: TI008 averaged data.xls was reviewed on 8/3/11 along with a revised copy of the PSD Report. There were no changes identified for the Excel file. Editorial comments in the report were recorded using the "track changes" function. All comments in report were addressed. No further action required, closed.**

PROJECT 52578 – SURVEILLANCE REPORT

Number: SR-52578-2011-030 **Rev. 1** Page 4 of 4

CORRECTIVE ACTION

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

Surveillance Performed By:

Lisa Middleton, Quality Engineer dsc 8-3-11 Date: 8/3/11

DISTRIBUTION: Carolyn Burns, Clark Carlson, Deborah Coffey, Richard Daniel, Sandy Fiskum, Matt Fountain, Rick Shimskey, and Andy Schmidt

Appendix J

Verification Processes and Revision 2 Correction Summary

Appendix J

Verification Processes and Revision 2 Correction Summary

In accordance with the K Basin Quality Assurance Project Plan/Sampling and Analysis Plan (QAPjP/SAP), KBC-33786, Rev. 2 (Baker et al. 2009), the Analytical Support Operations (ASO) procedures, and the quality management system, data verification activities were performed to confirm and document that the reported results are accurate and reflect the characterization analyses that were actually performed and that the quality control (QC) sample results and data met the applicable requirements. Activities associated with sample receipt, consolidation, subsampling, and characterization were complex. The QAPjP/SAP requirements were implemented and controlled by adherence to the ASO Quality Assurance (QA) Plan, sample preparation and analysis procedures, and controlling work documents (e.g., test instructions [TIs]). The ASO QA plan, procedures, and work documents are consistent and compliant with DOE/RL-96-68, HASQARD, Revision 3, Volume 1, Administrative Requirements and Volume 4, Laboratory Technical Requirements. A number of approaches were employed to verify the data from origination through final reporting and these approaches are discussed in this appendix.

A summary of the changes made in developing Revision 2 of this data package is found in Section J.7. These changes are based on responding to general corrections identified during the Sludge Treatment Project (STP) validation and assessment activities (performed after issuance of Revision 1 of this data package) and minor changes directed from PNNL project management.

J.1 Sample Receipt and Preparation

Table J.1 summarizes the verification review requirements corresponding to the documentation of sample receipt with the corresponding record location.

Table J.1. Summary of Verification Records for Sample Receipt

Requirement	Verification Record	Record Location
Completed Chain of Custody (COC) forms	Signed and dated COC forms	Appendix A of this report
Observations of samples/sludge polybottles	Reviewer signature and date in laboratory record book (LRB) and on front cover of completed Tis	DVD-52578-PNNL-20650 Rev. 2, Section III, September 2013
Provided sample identification (ID) nomenclature crosswalks	Complete summary provided in Section 2.0 of this report	Section 2.0 of report

Images of the COC forms submitted with each polybottle pair containing K Basin sludge are provided in Appendix A. The COCs have been reviewed for completeness by the project technical staff and the Quality Engineer (QE).

Sample ID nomenclature used within this characterization effort included client-provided identification (i.e., engineered container IDs [e.g., SCS-CON-210] and core IDs [e.g., A1]), and Pacific Northwest National Laboratory (PNNL) identification (i.e., TI subsample IDs [e.g., TI001-A1-B], ASO laboratory analytical services request (ASR) numbers [e.g., 8798], and Radiochemical Processing Laboratory (RPL) analytical laboratory IDs [e.g., 11-0419]). Section 2.0 of this report includes sample ID cross reference tables to assist the reader in the navigation of both the document and the appendices.

Table J.2 summarizes the approach for verification reviews to evaluate work against requirements corresponding to sample consolidation, subsampling, and other characterization testing controlled via test instructions (e.g., rheology, particle size analyses) along with the corresponding location of the objective review.

Table J.2. Summary of Verification Records for Test Instructions

Requirement	Verification Record	Record Location
Planning		
Work document (TI) includes steps to obtain required samples and data meeting technical compliance.	Technical reviewer signature and date	Front cover of completed TI, DVD-52578-PNNL-20650 Rev. 2, Section III, September 2013
Work document collects data and samples consistent with the QAPjP/SAP.	Project manager signature and date	
Work document meets CH2MHill Plateau Remediation Company (CHRPC) Sludge Treatment Project (STP) requirements, consistent with the QAPjP/SAP.	Buyer's Technical Representative (BTR) signature and date	
Work document appropriately captures measurement and test equipment requirements and documentation.	QE signature and date	
Execution/Completion		
Steps in work document have been completed, changes have been documented, records are logical and understood, collected data are in technical compliance with project requirements.	Technical reviewer signature and date	Front cover of completed TI, DVD-52578-PNNL-20650 Rev. 2, Section III, September 2013
Steps in work document have been conducted in accordance with QA requirements.	QE signature on completed TI	
Transcriptions into data-calculation spreadsheets and data-reduction calculations and measurement uncertainties from completed TI are properly conducted.	Technical reviewer signature and date	Calculation spreadsheet review form, DVD-52578-PNNL-20650 Rev. 2, Section III, September 2013
Overall check for completeness of each TI, data traceability, associated attachments, and spreadsheets including identification and disposition of all issues.	TI surveillance report signed by QE	DVD-52578-PNNL-20650 Rev. 2, Section III, September 2013

The activities associated with sample receipt, consolidation, subsampling, and physical characterization were controlled and documented in detailed TIs.[(a)] The TIs were reviewed by at least one

(a) Completed TIs are provided in digital format; see DVD-52578-PNNL-20650 Rev. 2, Section III, September 2013. A listing of the applicable TIs is also provided within the summary report, just after the table of contents.

technical reviewer, the QE, and the client before issuance. These reviews were performed to verify that all client data needs were addressed during planning for processing activities. After the TIs were completed, the primary user performed a completeness review and the technical reviewer performed a technical review. Calculation spreadsheets were developed for each processing TI to determine various physical parameters (e.g., sample mass, settled sludge density). Spreadsheet reviews included a technical review of data entries from TIs into the calculation spreadsheet as well as calculation algorithms, cross-references to other cells, assignments of uncertainties, error propagation (where applicable), and overall reasonableness and consistency of the data. Spreadsheet reviews were performed by annotating issues and capturing responses to questions raised. The calculation spreadsheets, with documentation of the reviews, are included as part of the completed TIs.

Formal QE reviews of TIs, attachments, and supporting spreadsheets were performed to verify all entries were recorded properly, errors were corrected appropriately, justification was present (when needed) to describe data corrections, data traceability was evident, and TIs and attachments were acceptable to become a project record. Identified issues and concerns were discussed and resolved before the TI review was signed by the QE. QE reviews were documented as formal surveillance reports and are included as part of the completed TI. When necessary, Occurrence Reports were initiated to track discussions with the client on issues of concern and to determine if the occurrence needed to be elevated to a Deficiency Report or Nonconformance Report.

J.2 Sample Analysis and Characterization

Table J.3 summarizes the records documenting the planning for sample preparation and analysis, completed TIs, data forms, summary reports, and supporting information, along with the corresponding location of records indicating technical and QE review.

The data quality objectives (DQOs) defined in the QAPjP /SAP documents identify processes/analytical techniques for each characterization parameter (e.g., ^{137}Cs concentration using gamma energy analyses [GEA] and detection limit, and precision and accuracy acceptance criteria). Applicable procedure DQOs were also considered and evaluated relative to the QC sample results. Whenever there was a discrepancy between the DQOs in the ASR or the ASO procedure/ASO QA Plan, the DQOs in the ASRs (project Special Instructions) took precedence as the Special Instructions conveyed the specific K Basin project requirements. The specific PNNL/ASO procedures used for the various analyses are documented in DVD-52578-PNNL-20650 Rev. 2, Section II, September 2013. Procedures used to measure nuclear material in core samples for nuclear material accountability purposes were provided to the safeguards representative for review before use. The approach and control limits used by the laboratory for balance accuracy and linearity checks were provided to the safeguards representative for review and acceptance before use.

As previously noted, elemental and isotopic analyses and associated QC requirements in the QAPjP/SAP were provided to the ASO in the form of Special Instructions appended to the ASRs. Because the analytical requirements were complex in nature, these Special Instructions were reviewed and approved both internally by PNNL staff (author, technical reviewer, ASO Manager, and QE) and by the contract BTR to help make sure that all requirements were properly rolled out to the ASO. As with the TIs, approval/concurrence was documented by signature on the Special Instructions. The ASRs and the Special Instructions are included in the appendices of this report.

DQOs for non-ASO analyses (X-ray diffraction [XRD], isotopic Pu and U, and particle density) were conveyed to the analysts via Analysis Request forms. Procedures used for non-ASO measures were provided to the project for review before use. These analyses were conducted according to the Analysis Requests and PNNL test procedures. Particle size distribution (PSD) and rheology were conducted according to PNNL procedures with specific processing parameters defined in TIs. These TIs were reviewed and signed before implementation using the same protocol applied to the sample receipt and processing TIs. These completed TIs are also provided in the digital format (DVD-52578-PNNL-20650 Rev. 2, Section III, September 2013).

Table J.3. Summary of Verification Records for Analytical Sample Preparation and Analysis

Requirement	Verification Record	Record Location
Planning		
Work documents (ASR Special Instructions) identify all subsamples and specific analyses to be performed. All method and QAPjP/SAP QC requirements have been identified and properly communicated to the analytical laboratory.	ASR Special Instructions signatures on ASR Special Instructions as follows: • cognizant scientist • project manager • BTR	Appendices B, C, and D
Execution/Completion		
For each ASO analysis, a technical review has been performed, consisting of the following elements: • transcription accuracy • correctness of calculations • evaluation of overall consistency and reasonableness of data.	Technical reviewer signature on the completed ASO analytical report	ASO reports in Appendices B, C, D
For each ASO analysis, a QE review has been performed, consisting of the following elements: • evaluation of project, ASR, and method DQOs against QC sample results • transcription accuracy • correctness of calculations • evaluation of overall consistency and reasonableness of data • implementation of appropriate corrective action, when necessary. • data traceability • documentation of QC sample results not meeting DQOs and other data quality issues.	QE review surveillance reports	QE surveillance reports in ASO ASR files
Non-ASO analysis (such as particle density, isotopic distribution by mass spectrometry, XRD, rheology, PSD, etc.) reports are subjected to a technical review with the following general elements. • evaluation of method and QC performance • evaluation of compliance with technical and QC requirements, as defined in the ASR and Special Instruction • transcription accuracy • correctness of calculations • evaluation of overall consistency and reasonableness of data.	Technical reviewer signature on completed technical report	Appendices E, F, G, H, and I

Table J.3. (contd)

Requirement	Verification Record	Record Location
Non-ASO analysis (particle density, isotopic distribution by thermal ionization mass spectrometry, XRD, rheology, and PSD) reports are subjected to a QE review with the following general elements. • evaluation of project, Analysis Request, and method DQOs against QC sample results • transcription accuracy • correctness of calculations • evaluation of overall consistency and reasonableness of data • data traceability • implementation of appropriate corrective action, when necessary • documentation of QC sample results not meeting DQOs and other data quality issues.	QE reviewer signature on completed technical report	Appendices E, F, G, H, and I
Documentation of occurrences, deficiencies, and nonconformance	Occurrence Reports signed by the project manager and the QE	Appendices B, C, D, E, F, and G and summarized in Table J.4

J.3 Data Reporting

Preliminary ASO data reports received internal technical reviews (by ASO staff) and provided to PNNL project staff and the BTR. This early project review facilitated preliminary data reporting and timely identification of issues and concerns. Technical staff evaluated results received from the ASO for overall consistency between duplicates and processes. Where possible, duplicative analysis results were compared (e.g., ^{241}Am concentrations determined by GEA and alpha energy analysis [AEA] and total U concentrations determined by kinetic phosphorescence analysis and inductively coupled plasma optical emission spectrometry [ICP-OES]) and isotopic ratios (e.g., ^{241}Am:^{154}Eu and ^{238}Pu:$^{239+240}$Pu) were reviewed for consistency.

Each ASO data report includes sections that address each of the analytical QC samples and DQO compliance to the procedure requirements and to requirements identified in the ASR/Special Instructions and the QAPjP/SAP. Each ASO analysis report was reviewed and signed by the preparer and the technical reviewer. The report and supporting data were then forwarded to the ASO QE for review. The QE reviews were documented as surveillance reports and were performed to verify that data were what they purported to be (i.e., the reported results reflected what was actually done and the results meet applicable requirements, particularly procedure and project data quality objectives). Identified issues and concerns were discussed and resolved before the QE signed and issued the surveillance report. All QE review comments were resolved before the analytical report was finalized. The QE review and comment resolution are documented in surveillance reports that are available in the ASO records. The ASO Lead also performed a final review of each ASO report for completeness and provided authorization to release the data.

Individual, non-ASO reports (e.g., XRD, PSD, particle density, rheology, and isotopic analysis by thermal ionization mass spectrometry [TIMS]) contributing to the final K Basin reports are provided in appendices. These reports were subject to a minimum of technical and QE reviews. The QE reviews

were documented as formal surveillance reports and were performed to verify that data were what they purported to be, that is, that the reported results reflected what was actually done and the results meet applicable requirements, particularly procedure and project data quality objectives. Identified issues and concerns were discussed and resolved before the QE signed the surveillance report. Surveillance reports are included in the relevant appendices. When necessary, Occurrence Reports were initiated to track discussions with the client on issues of concern and to determine if the occurrence needed to be elevated to a Deficiency Report or Nonconformance Report.

Tiered reviews of the completed sections of the summary K Basin report were conducted. Reviews were conducted by data contributors (e.g., ASO staff, X-ray spectroscopist), staff tasked with verifying numerical data entries (transcriptions from calculation spreadsheets to report tables) and result discussion text, an independent technical reviewer (for overall reasonableness and accuracy of presentation and conclusions), the RPL project management staff, and the assigned QE.

J.4 Occurrences, Deficiencies, and Nonconformance

The process for documenting issues of concern and determining if significant conditions adverse to quality were indicated, Occurrence Report forms, Deficiency Report forms, and Nonconformance Report forms were generated, as applicable. The project manager and QE evaluated occurrences for conditions adverse to quality to determine if the occurrence was a significant condition adverse to quality that required elevation to a deficiency or nonconformance. These terms are defined as follows:

- Occurrences are defined to be any issue affecting sample integrity or data quality. Occurrences were tracked to document any issue of concern to bring the issue to the attention of project management and the client and when applicable, to define appropriate corrective actions or paths forward.
- Deficiencies are defined as failures to follow work-controlling documents such as test plans, TIs, or procedures. Deficiency Reports may result from Occurrence Reports that are determined by the responsible manager to be a significant condition adverse to quality requiring corrective action and tracking to closure or may be issued without an Occurrence Report when it is clear that a failure to follow work-controlling documents occurred.
- Nonconformances are defined as failures of items to meet specifications or to operate as expected; this includes using out-of-service equipment and measuring and test equipment used outside of a valid calibration interval and other types of calibration-related issues. Nonconformance Reports result from occurrences that are determined by the responsible manager to be a significant condition adverse to quality requiring disposition of the item (e.g., use as-is, reject, repair, re-work), corrective action, and tracking to closure.

Occurrence Reports were uniquely identified and include a description of the issue, affected samples, investigative findings, recommended actions, and an assessment of significant impact to data quality. Occurrence Reports were used to inform the client and establish concurrence with respect to recommended actions. Project management made the determination as to whether the issues needed to be elevated to formal Nonconformance Reports or Deficiency Reports. Table J.4 provides summaries of the issued Occurrence Reports and identifies the location where these records can be found. No Deficiency Reports were generated for this scope of the K Basin work. One Nonconformance Report was generated.

Table J.4. Occurrence Report Summary

ID	Summary	Record Location
OR-52578-04-18-11	ASR 8798, two duplicate pair results for GEA exceed the ±20% relative percent difference (RPD) requirement (^{60}Co RPD for TI001-A1 pair was 48% and ^{155}Eu RPD for TI002-B1 pair was 32%).	Appendix B
OR-52578-04-20-11	ASR 8798, three duplicate pair core sample results on several analytes exceed ±20% RPD requirement; the matrix spike sample results for Sb, Tl, Zn, and Zr exceed ±20% accuracy requirement.	Appendix B
OR-52578-04-29-11	ASR 8824, the MCC sample duplicate pair results exceed ±20% RPD requirement for ICP-OES results from PNL-ALO-129 acid digest and [Na] in preparation blank was greater than 5% present in samples.	Appendix C
OR-52578-05-09-11	Minor isotopes in the U TIMS standard did not meet the ±2% acceptance criterion; all ^{238}Pu sample and duplicate pair precision results exceed ±20% RPD requirement; ^{241}Pu sample TI001-A1 duplicate pair precision result exceeds ±20% RPD requirement.	Appendix E
OR-52578-05-19-11	ASR 8824, U metal duplicate pair results exceed ±20% RPD requirement (RPD for samples TI008-ME and TI008-MF was 86%).	Appendix C
OR-52578-05-26-11	ASR 8798, U metal duplicate pair exceeds ±20% RPD requirement (RPD for TI002-A4 pair was 54%).	Appendix B
OR-52578-05-27-11	ASR 8798, two duplicate pair results for ^{237}Np exceed ±20% RPD requirement (RPD for TI001-A1 pair was 22% and RPD for TI002-B1 pair was 82% RPD)	Appendix B
OR-52578-06-27-11	ASR 8824, GEA and AEA MCC duplicate pair results exceed ±20% RPD requirement.	Appendix C
OR-52578-06-29-11	Four PNNL subsample IDs were the same as the client sample IDs: KW210-A1-A, KW210-A4-A, KW210-B1-A, and KW210-B3-A and did not contain the TI ID. These samples were combined to create one of the container composites.	Section III, DVD-52578-PNNL-20650 Rev. 2, September 2013
OR-52578-06-30-11	ASR 8798, total organic carbon (TOC) samples were dried to constant mass at 105°C before analysis as per the analysis procedure, but there was a QAPjP/SAP statement that this analysis was to be performed on a wet sample.	Appendix B
OR-52578-07-19-11	ASR 8798, TOC/TIC Method QC, duplicate/replicate samples and analytical spike sample did not meet method and project acceptance criteria.	Appendix E
OR-52578-08-19-11	Gelatinous material impact on settled sludge density for sample TI002-A4-N.	52578-TI002, Section III, DVD-52578-PNNL-20650 Rev. 2, September 2013
OR-52578-08-31-11	ASR 8878, detection limit for gross alpha analysis samples was greater than the requested detection limit of 1.0E-03 μCi/g.	Appendix D

Table J.5. Nonconformance Report Summary

ID	Summary	Record Location
NCR-52578-11-08-10	The viscosity standard certificate was not supported for NIST traceability by the vendor and therefore did not meet PNNL Quality Assurance Clause 188 requirements.	Appendix H

J.5 Surveillances

There were six QE surveillances of completed TIs, three surveillances of supporting LRB entries, one surveillance of two data analysis Excel spreadsheets, and four data report surveillances performed. In two cases (rheology and PSD), reviews of completed TIs and draft reports were conducted concurrently and summarized under one surveillance report for each analysis activity. Surveillances for ASRs 8798, 8824, 8878 and 8919 are available in the ASO records system. Table J.6 provides a summary and location of surveillance reports.

Table J.6. Surveillance Report Summary

Number	Subject	Report Issued/ Summary of Concerns	Associated Record
SR-52578-2011-009	QE Surveillance of RPL K Basin LRB 60605, pp. 29 to 60, March 2011, which covered LRB entries from 8/9/10 to 2/2/11	3/18/11 All comments addressed.	LRBs, Section III, DVD-52578-PNNL-20650 Rev. 2, September 2013
SR-52578-2011-021	QE Surveillance of RPL K Basin LRB 60605, pp. 65 to 77, May 2011, which covered LRB entries from 2/25/11 to 5/5/11	6/3/11 All comments addressed.	LRBs, Section III, DVD-52578-PNNL-20650 Rev. 2, September 2013
SR-52578-2011-023	QE Surveillance of Completed 52578-TI001, *K West Basin Sample Receipt and Preparation of Consolidated Core Composites: SCS-CON-210-A1 and SCS-CON-210-B3*	6/14/11 All comments addressed.	TIs, 52578-TI001 Section III, DVD-52578-PNNL-20650 Rev. 2, September 2013
SR-52578-2011-024	QE Surveillance of Completed 52578-TI002, *K West Basin Sample Receipt and Preparation of Consolidated Core Composites: SCS-CON-210-A4 and SCS-CON-210-B1*	6/14/11 All comments addressed.	TIs, 52578-TI002 Section III, DVD-52578-PNNL-20650 Rev. 2, September 2013
SR-52578-2011-025	QE Surveillance of completed 52578-TI008, *Preparation of K West Basin Consolidated Container Composite for SCS-CON-210*	6/23/11 All comments addressed.	TIs, 52578-TI008 Section III, DVD-52578-PNNL-20650 Rev. 2, September 2013
SR-52578-2011-027, Rev. 1	QE Review of *TIMS Report for Isotopic Analysis of KW 210 Core Samples*	8/3/11 All comments addressed and OR 52578-05-09-11 issued.	Appendix E
SR-52578-2011-029	QE Surveillance of *X-Ray Diffraction Analysis Report KW 210 Container Composites*	7/20/11 All comments addressed.	Appendix F
SR-52578-2011-030, Rev. 1	QE Surveillance of Completed 52578-TI037, *Particle Size Distribution Measurements for K West Basin Consolidated Container Composite SCS-CON-210, Using Minus 500 micron Particle Size Fraction Samples*	8/3/11 All comments addressed.	TIs, 52578-TI037 Section III, DVD-52578-PNNL-20650 Rev. 2, September 2013

Table J.6. (contd)

Number	Subject	Report Issued/ Summary of Concerns	Associated Record
SR-52578-2011-032	QE Surveillance of *Particle Density Report for TI008-L, TI008-MK, TI008-ML, TI008-SK & TI008-SL*	7/14/11 All comments addressed.	Appendix G
SR-52578-2011-33	QE Surveillance of Completed 52578-TI-040, *Processing of the Undissolved Solids Samples from the K West Basin Core Samples Collected from SCS-CON-210 for Additional Analysis*	7/28/11 All comments addressed.	TIs, 52578-TI040 Section III, DVD-52578-PNNL-20650 Rev. 2, September 2013
SR-52578-2011-034	QE Surveillance of *XRD Analysis Report for KW210 Undissolved Solids*	7/27/11 All comments addressed.	Appendix F
SR-52578-2011-035	QE Surveillance of completed K Basin TI, 52578-TI039, *Rheological Measurements for K West Basin Consolidated Container Composites SCS-CON-210*	8/2/11 All comments addressed.	Appendix H
SR-52578-2011-041	QE Surveillance of : Surveillance of RPL K Basin LRB 60605, pp. 82 to 92, September 2011, which covered LRB entries from 5/20/11 to 8/5/11	9/13/11 All comments addressed.	LRBs, Section III, DVD-52578-PNNL-20650 Rev. 2, September 2013
SR-52578-2011-042	QE Surveillance of Two Spreadsheets: ASR 8824 Data Reduction Rev1.xlsx and ASR 8798 Data Reduction Rev2.xlsx	9/26/11 All comments addressed.	ASR Data Reductions, Section III, DVD-52578-PNNL-20650 Rev. 2, September 2013

J.6 Technical Reviewers

Table J.7 provides technical reviewers' names, qualifications, and areas of review for this project.

Table J.7. Technical Reviewers Supporting the K Basin Sludge Characterization Activities

General Review Area	Reviewer	Qualifications
TI review before implementation	AJ Schmidt	MS Chemical Engineering; 22 years of experience, including 14 years with K Basin Sludge characterization, testing and evaluation.
TIs, supporting data calculation spreadsheet, and LRB	SK Fiskum	BS Chemistry, 26 years of chemical and radiochemical characterization and reporting experience.
	RW Shimskey	BS Chemical Engineering, PE License; 7 years of radiochemical processing, characterization, and reporting experience at PNNL; 11 years of zirconium/titanium processing experience geared to the nuclear cladding and aerospace industry.
TIs, supporting data calculation spreadsheet, LRB, ASRs, particle density, PSD, rheology, XRD, TIMS, and characterization analyses	DS Coffey	BS Biology/Chemistry, MS Resource Ecology, 2 years as Laboratory Director and 11 years as deputy and Quality Manager EPA ERL laboratory, 5 years as Deputy QM WIPP Sandia National Laboratory, 11 years as QE Battelle, 30 years of analytical data reporting with focus on regulatory work.

Table J.7. (contd)

General Review Area	Reviewer	Qualifications
TIs, supporting data calculation spreadsheet, LRB, PSD, rheology, XRD and characterization analyses	LA Middleton	BS Environmental Science (with a minor in Chemistry), 2 years of experience as QE at PNNL.
TOC reports	KA Pool	BS Chemistry, 24 years of experience in chemical and radiochemical characterization and reporting.
ICP-OES reports	CJ Thompson	BS Chemistry, MS Analytical Chemistry, PhD Analytical Chemistry; 16 years of experience in chemical characterization and reporting.
Radiochemistry reports	CZ Soderquist	MS, Chemistry, 27 years of experience in radioanalytical chemistry.
Mass spectrometry reports	JC Braley	PhD Chemistry, 1 year of post-degree experience with an emphasis on radioanalytical and separations chemistry.
Radiochemistry reports	LR Greenwood	PhD Nuclear Physics, Laboratory Fellow, 40 years of experience with reactor dosimetry, nuclear data measurements, and radiation detector development; team lead for Radiochemistry; project manager on numerous projects
Radiochemistry reports	TT Trang-Le	BS Mathematics-Actuarial Science, 16 years of experience in radioanalytical reporting.
XRD reports	MW Urie	BS Chemistry, BS Electrical Engineering, 43 years of chemical characterization; 20 years of XRF, XRD, Auger, and physical measurements experience.
Particle density and U speciation reports	SI Sinkov	PhD Chemistry (with an emphasis on chemistry of transuranium elements), 26 years of experience, including 6 years with K Basin sludge characterization, testing, and reporting.
Rheology report	CA Burns	PhD Chemistry (with an emphasis on surface and colloid science), 5 years of post-degree experience in the characterization of physical properties of both radiological and non radiological materials.
PSD report	RC Daniel	PhD Chemical Engineering (with an emphasis on surface and colloid science), 5 years of post-degree experience in the size characterization of radiological dispersions.
Mass spectrometry reports and mass measurement reports	CH Delegard	BS Chemistry, 38 years of chemical, radiochemical, and safeguards characterization and reporting experience.
Transcription review of analytical data calculation spreadsheets and ASO reports; data incorporation into summary report; transcription accuracy	M Luna	BS Chemistry/Biology, 4 years of experience in the characterization of physical properties of both radiological and non radiological materials, testing, and reporting, 8 years of experience in Chemical/Biological warfare agents and clinical pathogens decontamination, and 7 years studying molecular process and bioengineering autoimmune and infectious diseases.
Technical review of completed report	JM Tingey	PhD Physical Chemistry, 27 years of experience in the characterization and treatment of nuclear wastes and spent nuclear fuel, basic and applied actinide chemistry, plutonium and spent fuel processing, and colloid chemistry. Several years experience in characterization of K Basin sludges.

J.7 STP Validation and Revision 2 Corrections

The initial report (Revision 1) was delivered to CHPRC for data validation by the STP. The validation activity resulted in identification of errata and provided the basis for Revision 2 to this report. Table J.8 summarizes the errata identified during the STP validation process and the corrections made to the report. Additional editorial changes directed by the project are summarized in Table J.9.

Table J.8. Summary of STP Validation Errata and Changes Made in Revision 2

PNNL-20650, Rev. 1 Report Location	Errata Summary	Correction Made
Page xiii, Definitions section	The given definition of "Dry Sludge: Sludge that has been dried to a constant mass at 105°C." A clarifying definition should be added for the term "As-Sampled" since there is a tendency to equate "as-sampled" and "dewatered" (in-part because Test Instructions have equated these terms in their "Relevant Terms" definition section [e.g., page 11 of 52578-TI008 where sieving was done includes a definition for "Dewatered or As-Sampled"]).	Definition of "as-sampled" added, "The actual state of the sludge at the point of sampling; in most instances this is the "dewatered" sludge state, but in the case of sieved sample material retained on a sieve it is taken as "air-dried" (dried at ambient conditions in the hot cell)." The Definition now appears on page xv of revision 2.
Page 2.3 Figure 2.1	Under icon for "KW & KE 230", in gray square, under "59205-TI023" it is indicated this TI addresses "Cores A4 and B2" the correct cores are "Cores A2 and B4".	Figure 2.1 changed to reflect correct core identifications.
Page 2.10 Table 2.3 Column "Subsample ID" Archive samples; Page 2.11 Table 2.4 Column "Subsample ID" Archive samples; Page 2.12 Table 2.5 Column "Subsample ID" Archive samples; Page 2.13 Table 2.6 Column "Subsample ID" Archive samples	Page 2.10 Table 2.3, page 2.11 Table 2.4, page 2.12 Table 2.5, and page 2.13 Table 2.6: in the column under "Subsample ID" for the archive samples that are the last group of samples shown in this column, the sample prefixes have incorrectly been shown as 'TI001" or "TI002"; the actual sample ID prefix for all these archive samples is "KW210".	Sample identifications corrected. Table 2.4 footnote (d) was similarly amended to reflect nomenclature.
Page 2.20 Table 2.9, Column "As-sampled Mass"	Added footnote (a) to table to clarify the as-sampled mass basis for middle and large sieve fraction samples is based on air-dried sample masses.	Added footnote (a) which states, "The as-sampled mass basis for all large and medium sieve fraction samples (i.e. TI008-LA and TI008-M*, where * is A-L) is "air dried" and thus the sample may contain some residual water." All other existing footnote identities were incremented up by one letter.

Table J.8. (contd)

PNNL-20650, Rev. 1 Report Location	Errata Summary	Correction Made
Page 5.5, Section 5.2, second sentence in first paragraph	"Appendix J contains the detailed rheology including shear strength rheograms." The actual Appendix is "H" instead of indicated "J". Also the ending phrase "…including strength rheograms" should be deleted. The rheograms were not collected; only shear strength measures were collected.	Sentence now reads, "Appendix H contains the detailed rheology report." and the ending phrase was deleted.
Page 5.10, Table 5.5 title	In the table title, sample number should be KW210-CC-N rather than indicated KW210-SS-N.	Sample identification corrected to KW210-CC-N.
Page 5.21, Table 5.15, MCC U Metal, As-sampled basis	For MCC samples, table should include clarification that "as-sampled" basis is "air dried".	Inserted new footnote (b) which states, "The as-sampled mass basis for medium sieve fraction samples (i.e. TI008-ME and TI008-MF) is air dried and thus the sample may contain some residual water. Existing footnotes (b) through (d) were incremented up by one letter.
Page 5.21, Table 5.15, footnote (c)	In the footnote (c), "0.474 $\mathbf{g_{sampled}/g_{dry}}$ should be: 0.474 $\mathbf{g_{dry}/g_{sampled.}}$	Units corrected to $\mathbf{g_{dry}/g_{sampled}}$ in footnote (d) of revision 2.
Page 5.21, Table 5.15, footnote (d)	In the footnote (d) reference, "(Fiskum et al. 2011a, Appendix J)," the "2011a" should be "2011b".	Reference correction to Fiskum et al. 2011b on footnote (e) of revision 2.
Page 5.22, first paragraph, fourth sentence	The reference for "(Fiskum et al. 2011, Appendix J)," the "20**11**" should be "20**11b**".	Reference corrected in text to read Fiskum et al. 2011b.
Page 5.23, Table 5.17, Sample ID column	The three samples identified with "TI009" prefixes should have prefixes of "TI008".	Corrected all TI009 prefixes to TI008.
Page 5.31, Table 5.24, As-measured ^{60}Co concentration for sample TI008-SA	Value of Co-60 concentration should be **1.12E-3** instead of **1.2E-3**. Also under the "Relative Amount" Co-60 should be **0.25**% instead of to **0.27**%. {Related to this: On the PNNL-20650 Rev 1 DVD, under Section III, that includes data analysis MS Excel files, under "ASR 8824 Data Reduction Rev1", tab "Radchem" the spreadsheet cell O43 (the concentration of Co-60 in sample TI008-SA) should be **1.12E-3** μCi/g instead of the indicated **1.2E-3** μCi/g to match the ASO reported value (page C.36). This in-turn changes the relative Co-60 value in cell Q60 to **0.25%** instead of the indicated **0.27%**.}.	As-measured ^{60}Co concentration changed from 1.2E-3 to 1.12E-3 and relative amount changed from 0.27% to 0.25%. Spreadsheet "ASR8824 Data Reduction Rev.1.xlsx, worksheet "radchem", cell O43, updated to 1.12E-3. Spreadsheet worksheet "overview" updated to reflect change and the spreadsheet filename revised to "ASR8824 Data Reduction Rev.2.xlsx. The spreadsheet package was reviewed and added to the DVD data package for this report.
Page 5.33, Table 5.26, "(RPL/ASO Parent ID) Sieve Fraction" column	The sample identifications indicated as "(11-0792-D)" and "(11-0793-D) should be "(11-0792-129-D)" and "(11-0793-129-D)."	Sample identifications changed to 11-0792-129-D and 11-0793-129-D.

Table J.8. (contd)

PNNL-20650, Rev. 1 Report Location	Errata Summary	Correction Made
Appendix C	Revision 2 to the Uranium Metal Concentration in K Basin Sludge Report needs to be issued. It should contain the following edits: a. A new second paragraph should be inserted under "Sample Analysis/Results Discussion" p. C.47 explaining the mass difference of the MCC samples between the value reported in the ASR (also given in Table 2.9) and the values given in Table A.1 (p. C.50). {letter PNNL-52578-20212-L10 includes background on these issues} b. The second sentence in the first paragraph under "Sample Analysis/Results Discussion" p. C.47 should be revised to read “Five samples were delivered for analysis as wet slurries with an overburden of supernatant water and two as dried solids.” (The words “five” and “two” are switched in the Rev. 1 ASO report.) c. Text above Table A.1 (p. C.50) should be revised to indicate the KW-210-SCC samples were dewatered. Currently, it indicates that KW-210-SCC samples were dried.	The following edits were made in Revision 2 to the Uranium Metal Concentration in K Basin Sludge Report: The number of samples delivered as wet slurries from two to five and the number delivered as dried solids from five to two in the first paragraph of the section "Sample Analysis/Results Discussion". The drying method also was further clarified to be "air-dried". A new second paragraph in the "Sample Analysis/Results Discussion" section was added to explain the mass differences for the KW-210-MCC samples between those reported in ASR 8824 and those presented in Table A.l of Attachment 1. Finally, the explanatory paragraph above Attachment 1 table was corrected to describe the KW-210-SCC samples as being dewatered (not dried) and the drying method further clarified to be "air-dried".

Table J.9. Summary of PNNL Directed Changes Made in Revision 2

PNNL-20650, Rev. 1 Report Location	Editorial Change Summary	Correction Made
Disclaimer page	Provide name, address, phone number, and email address of two agencies at which this report will be archived and available at request.	Provided contact information to request a copy of this report.
Page ii, Revision history table	Provide new entry to revision table for report revision 2. The description column explains the corrections are a direct result of the STP validation process.	Inserted new entry in revision history table reflecting the changes made to this report.
Page iii, Certification statement	Include a new certification page for revision 2 and keep the original certification page generated for Revision 1.	Inserted new page for certification for revision 2 prior to the original certification page.
Page ix, x, and xi, Acronyms	Acronym definitions needed for DVD, NARA, and TRIM.	The DVD, NARA, and TRIM acronyms were defined in Rev. 2 of the report.

Table J.9. (contd)

PNNL-20650, Rev. 1 Report Location	Editorial Change Summary	Correction Made
Page xxv, DVD TOC, Section III, ASR Data Reductions	Provide revision 2 spreadsheet name to list of files.	Spreadsheet "ASR 8824 Data Reduction Rev2.xlsx" included in DVD folder file list on page xxvii of revision 2.
Page xxiii, DVD TOC introduction	New report revision and publish date requires DVD title and electronic report name updates.	Revision 1 replaced with Revision 2 and October 2011 replaced with September 2013 on page xxv of revision 2.
Page 2.1, footnote (b)	Reflect change in the name of the DVD in footnote (b) on this page.	Footnote (b) on page 2.1 now shows **DVD-52578-PNNL-20650 Rev. 2, Section III, September 2013**.
Page 2.4, first paragraph, second sentence	Reflect change in the name of the DVD on this page.	Sentence now references D**VD-52578-PNNL-20650 Rev. 2, Section III, September 2013**.
Page 2.27, Section 2.4, fourth paragraph	Reflect change in the name of the DVD on this page.	Sentence now references D**VD-52578-PNNL-20650 Rev. 2, Section II, September 2013.**
Page 3.5, Table 3.1, second table note	Reflect change in the name of the DVD on this page.	Sentence now references D**VD-52578-PNNL-20650 Rev. 2, Section III, September 2013**.
Page 3.6, footnote (b)	Reflect change in the name of the DVD on this page.	Sentence now references D**VD-52578-PNNL-20650 Rev. 2, Section III, September 2013**
Page 3.7, second paragraph, third sentence	Reflect change in the name of the DVD on this page.	Sentence now references D**VD-52578-PNNL-20650 Rev. 2, Section III, September 2013**.
Page 4.2, first paragraph, third sentence	Reflect change in the name of the DVD on this page.	Sentence now references D**VD-52578-PNNL-20650 Rev. 2, Section IV, September 2013**.
Page 4.9, Figure 4.10, bottom-right image	The sample ID prefix has been incorrectly shown as 'TI001," the actual sample ID prefix for all archive samples is "KW210."	The sample ID for the image now reads KW210-B3-Archive-1.
Page 4.12, Table 4.2, table note	Reflect change in the name of the DVD on this page.	Sentence now references D**VD-52578-PNNL-20650 Rev. 2, Section III, September 2013**.
Page 4.17, Table 4.6, third table note	Reflect change in the name of the DVD on this page.	Sentence now references D**VD-52578-PNNL-20650 Rev. 2, Section III, September 2013**.
Page 4.18, Table 4.7, footnote (a)	Reflect change in the name of the DVD on this page.	Sentence now references D**VD-52578-PNNL-20650 Rev. 2, Section III, September 2013**.
Page 4.19, Table 4.8, second table note	Reflect change in the name of the DVD on this page.	Sentence now references D**VD-52578-PNNL-20650 Rev. 2, Section III, September 2013**.
Page 4.20, Table 4.9, second table note	Reflect change in the name of the DVD on this page.	Sentence now references D**VD-52578-PNNL-20650 Rev. 2, Section III, September 2013**.

Table J.9. (contd)

PNNL-20650, Rev. 1 Report Location	Editorial Change Summary	Correction Made
Page 4.34, Table 4.17, second table note	Reflect change in the name of the DVD on this page.	Sentence now references D**VD-52578-PNNL-20650 Rev. 2, Section III, September 2013**.
Page 4.40, Table 4.21, table note	Reflect change in the name of the DVD on this page.	Sentence now references D**VD-52578-PNNL-20650 Rev. 2, Section III, September 2013**.
Page 4.42, Table 4.22, Core Sample column	Correct core sample ID from B4 to B3	Core sample ID corrected to B3.
Page 5.2, footnote (a)	Reflect change in the name of the DVD on this page.	Sentence now references D**VD-52578-PNNL-20650 Rev. 2, Section III, September 2013**.
Page 5.39, Table 5.29, footnote (a)	Reflect change in the name of the DVD on this page.	Sentence now references D**VD-52578-PNNL-20650 Rev. 2, Section III, September 2013**.
Page 6.1, first paragraph, second sentence	Reflect change in the name of the DVD on this page.	Sentence now references D**VD-52578-PNNL-20650 Rev. 2, Section I-IV, September 2013**.
Page 6.1, Introduction	Add statement that references where DVD is stored for potential public inquiry. Add statement how this records storage differs from ASO records storage.	Text now reads: The contents of the DVD are stored in the PNNL records electronic database Total Records Information Management (TRIM) records system. The long-term storage of ASO-specific records are maintained in the National Archives and Records Administration (NARA) Federal Records Center (Seattle, Washington) (hardcopy), which in turn is based on DOE and other Federal records management requirements.
Page 6.1, Section 6.1, end of first paragraph	Reflect the change and addition of NARA.	Text addition in bold: (Note: All records submitted for storage created by PNNL under the Battelle/DOE Operating Contract [DE-ACO5-76RL01830] are transferred to the **NARA** Federal Records Center in Seattle, Washington.)
Page 6.2, Table 6.1, position "PNNL Records Management; Records Storage and Retrieval"	Update PNNL Records Management Contact.	Patricia Eick has been replaced by Terri Mooney; her contact information was added.
Appendix J, page J.1	Add new paragraph describing the genesis of the data package Revision 2 relative to the STP validation.	New paragraph 2 was added to page J.1 describing the reason for issuing Revision 2.

Table J.9. (contd)

PNNL-20650, Rev. 1 Report Location	Editorial Change Summary	Correction Made
Appendix J Throughout	Reflect change in the name of the DVD.	Rev.1 was changed to Rev. 2 and October 2011 was changed to September 2013.
Appendix J	Add new subsection to discuss the STP validation and changes made for Revision 1.	Added subsection J.7 "**STP Validation and Revision 2 corrections.**"