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Page 1 of 21. ECN 633345Proj.
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2. ECN Category (mark one) Supplemental ☐ Direct Revision ☒ Change ECN ☐ Temporary ☐ Standby ☐ Supersede ☐ Cancel/Void ☐	3. Originator's Name, Organization, MSIN, and Telephone No. Ruth A. Esch, Production Planning & Control, T6-06, 373-4314	4. USQ Required? ☐ Yes ☒ No	5. Date 04/08/97
	6. Project Title/No./Work Order No. Tank 241-C-106	7. Bldg./Sys./Fac. No. 241-C-106	8. Approval Designator N/A
	9. Document Numbers Changed by this ECN (Includes sheet no. and rev.) HNF-SD-WM-DP-225, Rev. 0 HNF-SD-WM-DP-225, Rev. 0A	10. Related ECN No(s). ECN-633344	11. Related PO No. N/A
12a. Modification Work ☐ Yes (fill out Blk. 12b) ☒ No (NA Blks. 12b, 12c, 12d)	12b. Work Package No. N/A	12c. Modification Work Complete N/A Design Authority/Cog. Engineer Signature & Date	12d. Restored to Original Condition (Temp. or Standby ECN only) N/A Design Authority/Cog. Engineer Signature & Date
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Operating Specification		Interface Control Drawing		Spares Multiple Unit Listing	
Criticality Specification		Calibration Procedure		Test Procedures/Specification	
Conceptual Design Report		Installation Procedure		Component Index	
Equipment Spec.		Maintenance Procedure		ASME Coded Item	
Const. Spec.		Engineering Procedure		Human Factor Consideration	
Procurement Spec.		Operating Instruction		Computer Software	
Vendor Information		Operating Procedure		Electric Circuit Schedule	
OM Manual		Operational Safety Requirement		ICRS Procedure	
FSAR/SAR		IEFD Drawing		Process Control Manual/Plan	
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Environmental Impact Statement		Fac. Proc. Samp. Schedule		Tickler File	
Environmental Report		Inspection Plan			
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Tank Waste Remediation System (TWRS) Privatization Contractor Samples Waste Envelope D Material 241-C-106

Ruth A. Esch

Rust Federal Services of Hanford, Inc., Richland, WA 99352
U.S. Department of Energy Contract DE-AC06-96RL13200

EDT/ECN: ECN-633345 UC: 2070
Org Code: 7576B Charge Code: MD378
B&R Code: EW 3120074 Total Pages: 132

Key Words: Tank, Waste, Remediation, System, TWRS, Privatization,
Contractor Samples, Samples, Waste Envelope, Envelope D, D, Material,
241-C-106, C-106

Abstract: N/A

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**TANK WASTE REMEDIATION SYSTEM (TWRS) PRIVATIZATION
CONTRACTOR SAMPLES**

**WASTE ENVELOPE D MATERIAL
241-C-106**

FINAL ANALYTICAL REPORT

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April 1, 1997

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**TANK WASTE REMEDIATION SYSTEM (TWRS) PRIVATIZATION
CONTRACTOR SAMPLES**

**WASTE ENVELOPE D MATERIAL
C-106**

FINAL ANALYTICAL REPORT

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- Attachment 1: Tank Waste Remediation System Privatization Contractor Samples Analytical Summary Report, Revision 1. (Urie 1997)
- Attachment 2: Tank Waste Remediation System Privatization Contractor Samples Analytical Summary Report, Addendum 1, Revision 1. (Kuhl-Klinger and Urie 1997)
- Attachment 3: Letter Report "PNNL ACL Submittal of Organic and Radionuclide Results from Analysis of C-106 Original Material Supernate" (Urie 1996)
- Attachment 4: Tank Waste Remediation System Privatization Contractor Samples Final Statistical Analysis Report. (Welsh 1997)
- Attachment 5: Tank Waste Remediation System Privatization Contractor Samples -- Primary Analytical Data
- 5A - Analytical Service Requests and Test Instructions
 - 5B - Chain of Custody
 - 5C - Sample Preparation/Distribution Data Sheets
 - 5D - Physical Measurements
 - 5E - Inorganic Analysis (Excluding ICP-AES and ICP-MS)
 - 5F - ICP-AES
 - 5G - ICP-MS
 - 5H - Radiochemistry Analysis

Terms/Acronyms

ACL	Analytical Chemistry Laboratory
AEA	Alpha Energy Analysis
CVAA	Cold Vapor Atomic Absorption
DL ⁽¹⁾	Detection Limit (Instrument Detection Limit)
DSC	Differential Scanning Calorimetry
EQL ⁽¹⁾	Estimated Quantitation Limit
GEA	Gamma Energy Analysis
HLRF	High-Level Radiation Facility
IC	Ion Chromatography
ICP-AES	Inductively Coupled Plasma Atomic Emission Spectrometry
ICP-MS	Inductively Coupled Plasma Mass Spectroscopy
ISE	Ion Specific Electrode
LCS ⁽¹⁾	Laboratory Control Standard
LOI	Letter of Instruction
MDA ⁽¹⁾	Minimum Detectable Activity
PNNL	Pacific Northwest National Laboratory
QC	Quality Control
RPD	Relative Percent Difference
RSD	Relative Standard Deviation
SAL	Shielded Analytical Laboratory
TWRS	Tank Waste Remediation System

(1) For definition of these terms and a definition of QC terms see Analytical Summary Report (Urie 1997).

1.0 INTRODUCTION

This report represents the Final Analytical Report on Tank Waste Remediation System (TWRS) Privatization Contractor Samples for Waste Envelope D. All work was conducted in accordance with "Addendum 1 of the Letter of Instruction (LOI) for TWRS Privatization Contractor Samples Addressing Waste Envelope D Materials - Revision 0, Revision 1, and Revision 2." (Jones 1996, Wiemers 1996a, Wiemers 1996b)

Tank 241-C-106 (C-106) was selected by TWRS Privatization for the Part 1A Envelope D high-level waste demonstration. Twenty bottles of Tank C-106 material were collected by Westinghouse Hanford Company using a grab sampling technique and transferred to the 325 building for processing by the Pacific Northwest National Laboratory (PNNL). At the 325 building, the contents of the twenty bottles were combined into a single Initial Composite Material. This composite was subsampled for the laboratory-scale screening test and characterization testing, and the remainder was transferred to the 324 building for bench-scale preparation of the Privatization Contractor Samples.

1.1 Summary of Characterization Activities

The C-106 material was characterized during five sequential stages of the Envelope D Vendor Product preparation. These stages are summarized as follows:

- 1) Laboratory-Scale Screening Test: Analyses were conducted during and after completing the laboratory-scale washing and leaching screening test. The laboratory-scale screening test was used to establish operational parameters for the bench-scale batch processing. Details of the screening test are provided by Lumetta (1996).
- 2) Original Material Characterization: Analyses were performed on the feed material used for the bench-scale processing. The feed material was a blend of the Initial Composite Material with distilled water (at a 3:2 Initial Composite Material to water ratio). This feed material (i.e., diluted Initial Composite Material) is referred to, in this and previous reports, as "Original Material". The analytical results establish the baseline concentration of the bench-scale processing feed material and are used to establish the effectiveness of the washing and leaching process. The analytical and radionuclide results of the Original Material reported by Urie (1997).
- 3) Vendor Product Characterization: Analyses were performed on the material resulting from the bench-scale batch processing per revision 0 of the LOI. This prepared Envelope D material is referred to, in this and previous reports, as "Vendor Product". The details of the bench-scale operation and the effectiveness of the washing and leaching process are discussed by Brooks (1997). The analytical and radionuclide results for the Vendor Product are reported by Urie (1997).
- 4) Sodium Adjustment Treatment: The Vendor Product did not meet the Envelope D specification for a few analytes/radionuclides, most importantly for sodium. Therefore, the Vendor Product was subjected to a decant/dilute sodium adjustment treatment, per revision 1 of the LOI, to reduce the sodium concentration. Following the decanted/diluted treatment, the Vendor Product is referred to, in this and previous reports, as "Final Vendor Product". Estimates of the Final Vendor Product analyte and radionuclide concentrations were made

based on the quantity of sodium removed and the percent solids of the Final Vendor Product. Kuhl-Klinger and Urie (1997) provide details of the sodium treatment process and the criteria used to estimate the Final Vendor Product concentration.

- 5) Final Vendor Product Characterization: The initial measurement of the weight percent solids and weight percent oxides of the Final Vendor Product were inconsistent with estimated values. Based on this and the variability of the settled solids in each of the Final Vendor Product bottles (ten bottles for each vendor), additional analyses (albeit, a limited set of analyses) were performed following reblending and rebottling per revision 2 of the LOI. These analyses were used to confirm the estimates reported for the Final Vendor Product following the sodium adjustment treatment. The result of the analyses of the Final Vendor Product are reported by Kuhl-Klinger and Urie (1997).

The analytical results for the Original Material and Vendor Product (including Final Vendor Product) have been presented and discussed in previous reports which are included as Attachment 1 (Urie 1997) and Attachment 2 (Kuhl-Klinger and Urie 1997). Also included are the letter report detailing organic and radiochemistry analyses performed on Original Material supernatant as Attachment 3 (Campbell 1996) and the Final Statistical Report (Welsh 1997) for the Vendor Product as Attachment 4. The details of the analytical performance and raw analytical data are presented in Attachment 5 (consisting of Sections 5A through 5H) for all stages of characterization, including the laboratory-scale screening test and bench-scale processing.

The final reports detailing the laboratory-scale screening test and final bench-scale preparation of the Vendor Product are provided by Lumetta (1996) and Brooks (1997), respectively. Although these reports are not included as Attachments, laboratory-scale test and bench-scale processing operations which potentially affected Vendor Product analyte concentrations are included in this report.

1.2 Objective and Report Outline

The overall objective of this report is to evaluate the quality of the analytical data reported, to identify any limitations or discrepancies in the material processing, sampling, or preparation/analysis methods which may impact future characterization efforts, and to provide a basis for the development of the High-Level Waste feed characterization/validation methods. Results from analyses of the Vendor Product and Final Vendor Product are emphasized in this report; however, most of this evaluation also is applicable to the analysis of the Original Material. Each Section in this report is summarized below:

Section 2 provides a summary of the sampling, preparations, and analysis methods used. This includes initial grab sampling, compositing of the grab samples, subsampling for the laboratory-scale screening test, subsampling of the Original Material and Vendor Product during the bench-scale batch processing, and the analytical sampling and analysis methods.

Section 3 evaluates the data quality for the inorganic and radiochemistry results obtained on the Envelope D Privatization Samples. This includes evaluation of the precision and accuracy of the analytical techniques used to characterize the Vendor Product and Final Vendor Product, as well as an evaluation of the analytical sampling methods and the dissolution techniques used to prepare the sample for analysis. A limited set of analyses were performed on the Final Vendor Product following the reblending/rebottling operations, with these analyses providing confirmation of the estimated

(calculated) concentration of the Final Vendor Product following the sodium adjustment treatment. If analyses were performed on the Final Vendor Product and reported by Kuhl-Klinger and Urie (1997), then these results are evaluated for data quality. Otherwise, the Vendor Product data is used for evaluation.

Section 4 evaluates the data quality for the physical measurements, and identifies problems or concerns related to the laboratory-scale screening test and bench-scale batch processing. Although the reports by Lumetta (1996) and Brooks (1997) address most of the issues related to these processes, the key issues are summarized and evaluated for impact on the reported results.

Section 5 provides an evaluation of the analytical processes by comparing the analysis of the Original Material to the summation of results for the centrifuged solids and centrifuged liquid of the Original Material. The objective is to identify discrepancies which might impact approaches to future characterization efforts or that may impact the interpretation of the analytical results. This section also addresses the impact of removing a suspected organic layer from the Original Material prior to characterization.

Section 6 provides a summary of observations, limitations, and recommendations.

2.0 SAMPLE HANDLING

2.1 Initial Grab Sample Composite Preparation

Twenty slurry grab samples from tank 241-C-106 were taken from June 21, 1996 through June 24, 1996 at the East Tank Farms (Conners 1996). All the samples were taken from riser 7 at a sample elevation from 406 to 414 inches. These grab samples were received at the 325 building from June 22, 1996 through June 26, 1996. Due to the dose rates of these samples, the samples were transferred into the High-Level Radiation Facility (HLRF) hot cells where they were homogenized and composited in a series of steps. Each sample was individually homogenized with a high speed mixer, in an ice bath used to control the temperature of the samples during homogenization. The individually homogenized samples were blended together to form a single homogenized C-106 Initial Composite Material. A small aliquot of the Initial Composite Material was transferred to the Shielded Analytical Laboratory (SAL) for the laboratory-scale screening test, while the remainder of the Initial Composite Material was transferred to the 324 building for the bench-scale batch preparation of the Vendor Product.

2.2 Vendor Product Preparation

The laboratory-scale washing and caustic leaching screening test performed on C-106 Initial Composite Material is described by Lumetta (1996). The results from this testing were used (as available) to establish the sludge washing treatment parameters for the bench-scale batch preparation of the Vendor Product. Prior to initiating the bench-scale batch processing, approximately 3000 grams of C-106 Initial Composite Material was blended with approximately 2000 grams of deionized water. At this stage, four 125-ml subsamples identified as Original Material were extracted from the bench-scale processing apparatus and sent to the 325 building for physical and chemical characterization. A fifth sample was removed for archive.

As with the laboratory-scale screening test, two caustic leaches and three inhibited water washes were performed during the bench-scale batch processing. During this processing, each leach or wash step was followed by gravity settling and supernatant removal at 85 °C for leach and ambient temperature for wash to separate the liquid and solid phases. To evaluate the bench-scale processing, the supernatants removed following each leaching and washing operation were transferred to the 325 building SAL for chemical characterization. The analytical results for these supernatants are included in Attachment 5 for completeness; however, no discussion of the analytical results for these supernatants is included in this report.

At the completion of the bench-scale sludge washing/leaching processing, the resulting material (i.e., Vendor Product) was thoroughly mixed and four subsamples were collected and sent to the 325 building for subsequent physical and chemical characterization. A fifth sample was collected for archive. Also, two 1-L containers of Vendor Product (one containing 1624 g and the other 1523 g) were transferred to the 325 building for subsequent bottling and shipping to the two privatization contractors. The details of the bench-scale processing tasks, procedures, and data evaluation are described by Brooks (1997).

2.3 Analytical Sample Preparation

Three of the four subsamples of Original Material and three of the four subsamples of Vendor Product were distributed to the SAL for drying and subsequent analytical sample preparation and/or direct distribution for chemical analysis. The remaining subsamples of the Original Material and Vendor Product were distributed to the HLRF for density, weight loss on drying and firing measurements, and additional physical testing. The preparation methods and analysis procedures used for reporting the results for the Vendor Product and Final Vendor Product are presented in Tables 2.1 - 2.3.

A full explanation of the drying and blending operations performed on the Original Material and Vendor Product chemical and radionuclide characterization samples is detailed in the Analytical Summary Report (Urie 1997). In summary, three subsamples each of Original Material and Vendor Product were dried individually. The dried solids were medium brown in color and the evaporated supernatant left a white coating on top of the dried brown solid layer. No indication of spattering, foaming, etc. during drying was noted. Each dried subsample was manually blended, and then divided into two portions (i.e., a sample and a duplicate) for further sample preparation (e.g., acid digestion) or direct chemical analysis.

For the Original Material subsamples, a clear liquid layer was observed forming on top of each subsample within minutes after transferring the slurries to beakers for drying. The layer was suspected to be organic in nature, because the layer separated rapidly, was distinct from the slurry, and coated the beaker walls when swirled. Due to the concern with drying any material having an organic top layer, the suspected layer was removed. The results of the organic and radiochemical screening of this suspect organic layer is provided in a separate letter report (Campbell 1996), and the effect of the removal of this liquid layer on the Original Material analytical results is discussed in Section 5.2 of this report.

Additional preparations and analyses were performed on the Final Vendor Product. In this case, four bottles of Final Vendor Product were subjected to the same processing as those performed on the three subsamples of the Vendor Product. However, only one of the four Final Vendor Product samples were prepared in duplicate. The average results reported for the Vendor Product and Final Vendor

Product were calculated from six results for the Vendor Product (i.e., sample and duplicate on each subsample) and five results for the Final Vendor Product (i.e., four samples and one duplicate).

Sample preparations conducted in the SAL included potassium hydroxide-potassium nitrate fusion utilizing nickel crucibles (PNL-ALO-115), sodium peroxide-sodium hydroxide-sodium chloride fusion utilizing zirconium crucibles (PNL-ALO-116), nitric acid-hydrochloric acid digestion (PNL-ALO-129), and water leaching (PNL-ALO-103). Throughout this report these preparation procedures are referred to as KOH fusion, Na_2O_2 fusion, acid digestion, and water leaching, respectively.

All samples were prepared for analysis from the dried slurries. Fusions, digestions, leaches, and direct analyses (except the SAL bulk weight percent solids) were performed on the sample and duplicate portions of each Original Material and Vendor Product dried subsample. Quality control (QC) was applied to the analytical preparation batch in accordance with the governing quality assurance plan (Kuhl-Klinger 1996). The batch QC incorporated the use of preparation blanks, spiked blanks or Laboratory Control Standards (LCS), duplicate samples, matrix spiked samples, and radiochemical tracers, where available and appropriate for use. Sections 3.1.1 to 3.1.3 of the Analytical Summary Report (Urie) provide further details on the fusion, digestion, and leaching procedures used.

Limited analytical and radionuclide characterization was performed on centrifuged solids and centrifuged supernatants which were generated during physical characterization operations conducted in the HLRF. The Original Material centrifuged solids were prepared for chemical characterization using the same preparation methods as used for the dried slurry subsamples. Depending on the analysis method, the Original Material and Vendor Product supernatants were either analyzed directly or subjected to a nitric acid-hydrochloric acid digestion (PNL-ALO-128) prior to analysis. The details of the centrifuged solids and supernatant preparation are discussed in Section 4.5 of the Analytical Summary Report (Urie 1997). A comparison of the summation of the concentration of Original Material centrifuged solids and centrifuged supernatant to the dried Original Material subsamples is discussed in Section 5.1 of this report. A similar comparison of the Vendor Product centrifuged solids/supernatant is not available, since the Vendor Product centrifuged solids were not analyzed.

Table 2.1. Analyte Preparation and Analysis Methods for Reported Results

Material	Analyte	Preparation Method	Analysis Method	Procedure
VP	Ag	PNL-ALO-116	ICP-AES	PNL-ALO-211
FVP	Ag	PNL-ALO-115	ICP-AES	PNL-ALO-211
VP/FVP	Al	PNL-ALO-115	ICP-AES	PNL-ALO-211
VP	As	PNL-ALO-115	ICP-MS	PNL-ALO-280
VP	B	PNL-ALO-115	ICP-MS	PNL-ALO-280
VP/FVP	Ba	PNL-ALO-116	ICP-AES	PNL-ALO-211
VP	Be	PNL-ALO-129	ICP-AES	PNL-ALO-211
VP	Bi	PNL-ALO-129	ICP-AES	PNL-ALO-211
FVP	Bi	PNL-ALO-116	ICP-AES	PNL-ALO-211
VP/FVP	Ca	PNL-ALO-129	ICP-AES	PNL-ALO-211
VP	Cd	PNL-ALO-115	ICP-MS	PNL-ALO-280
FVP	Cd	PNL-ALO-129	ICP-AES	PNL-ALO-211
VP	Ce	PNL-ALO-116	ICP-MS	PNL-ALO-280
VP	Co	PNL-ALO-115	ICP-MS	PNL-ALO-280
VP	Cr	PNL-ALO-115	ICP-MS	PNL-ALO-280
FVP	Cr	PNL-ALO-115	ICP-AES	PNL-ALO-211
VP	Cs	PNL-ALO-115	ICP-MS	PNL-ALO-280
VP	Cu	PNL-ALO-115	ICP-MS	PNL-ALO-280
FVP	Cu	PNL-ALO-129	ICP-AES	PNL-ALO-211
VP	Dy	PNL-ALO-115	ICP-MS	PNL-ALO-280
VP	Er	PNL-ALO-115	ICP-MS	PNL-ALO-280
VP	Eu	PNL-ALO-115	ICP-MS	PNL-ALO-280
VP/FVP	Fe	PNL-ALO-115	ICP-AES	PNL-ALO-211
VP	Gd	PNL-ALO-115	ICP-MS	PNL-ALO-280
VP	Hg	Direct	CVAA	PNL-ALO-131/201
VP	Ho	PNL-ALO-115	ICP-MS	PNL-ALO-280
VP	K	PNL-ALO-129	ICP-AES	PNL-ALO-211
VP	La	PNL-ALO-116	ICP-MS	PNL-ALO-280
FVP	La	PNL-ALO-129	ICP-AES	PNL-ALO-211
VP	Li	PNL-ALO-129	ICP-AES	PNL-ALO-211
VP	Mg	PNL-ALO-116	ICP-MS	PNL-ALO-280

Table 2.1. Analyte Preparation and Analysis Methods for Reported Results

Material	Analyte	Preparation Method	Analysis Method	Procedure
FVP	Mg	PNL-ALO-115	ICP-AES	PNL-ALO-211
VP	Mn	PNL-ALO-115	ICP-MS	PNL-ALO-280
VP	Mo	PNL-ALO-115	ICP-MS	PNL-ALO-280
VP/FVP	Na	PNL-ALO-129	ICP-AES	PNL-ALO-211
VP	Nb	PNL-ALO-115	ICP-MS	PNL-ALO-280
VP/FVP	Nd	PNL-ALO-129	ICP-AES	PNL-ALO-211
VP	Ni	PNL-ALO-116	ICP-MS	PNL-ALO-280
FVP	Ni	PNL-ALO-116	ICP-AES	PNL-ALO-211
VP	P	PNL-ALO-115	ICP-AES	PNL-ALO-211
FVP	P	PNL-ALO-116	ICP-AES	PNL-ALO-211
VP	Pb	PNL-ALO-129	ICP-AES	PNL-ALO-211
FVP	Pb	PNL-ALO-116	ICP-AES	PNL-ALO-211
VP	Pd	PNL-ALO-116	ICP-MS	PNL-ALO-280
VP	Pr	PNL-ALO-116	ICP-MS	PNL-ALO-280
VP	Pt	PNL-ALO-115	ICP-MS	PNL-ALO-280
VP	Rb	PNL-ALO-116	ICP-MS	PNL-ALO-280
VP	Re	PNL-ALO-115	ICP-MS	PNL-ALO-280
VP	Rh	PNL-ALO-116	ICP-MS	PNL-ALO-280
VP	Ru	PNL-ALO-115	ICP-MS	PNL-ALO-280
VP	Sb	PNL-ALO-129	ICP-MS	PNL-ALO-280
VP	Se	PNL-ALO-129	ICP-AES	PNL-ALO-211
VP/FVP	Si	PNL-ALO-115	ICP/AES	PNL-ALO-211
VP	Sn	PNL-ALO-116	ICP-MS	PNL-ALO-280
VP	Sr	PNL-ALO-116	ICP-MS	PNL-ALO-280
FVP	Sr	PNL-ALO-115	ICP/AES	PNL-ALO-211
VP	Ta	PNL-ALO-116	ICP-MS	PNL-ALO-280
VP	Tb	PNL-ALO-115	ICP-MS	PNL-ALO-280
VP	Te	PNL-ALO-115	ICP-MS	PNL-ALO-280
FVP	Te	PNL-ALO-129	ICP-AES	PNL-ALO-211
VP/FVP	Th	PNL-ALO-129	ICP-AES	PNL-ALO-211
VP	Ti	PNL-ALO-116	ICP-MS	PNL-ALO-280

Table 2.1. Analyte Preparation and Analysis Methods for Reported Results

Material	Analyte	Preparation Method	Analysis Method	Procedure
VP	Tl	PNL-ALO-129	ICP-AES	PNL-ALO-211
FVP	Tl	PNL-ALO-116	ICP-AES	PNL-ALO-211
VP	Tm	PNL-ALO-115	ICP-MS	PNL-ALO-280
VP	U	PNL-ALO-115	ICP-MS	PNL-ALO-280
FVP	U	PNL-ALO-115	Fluorimetry	PNL-ALO-445
VP	V	PNL-ALO-129	ICP-MS	PNL-ALO-280
VP	W	PNL-ALO-115	ICP-MS	PNL-ALO-280
VP	Y	PNL-ALO-129	ICP-AES	PNL-ALO-211
VP	Yb	PNL-ALO-115	ICP-MS	PNL-ALO-280
VP	Zn	PNL-ALO-115	ICP-MS	PNL-ALO-280
VP/FVP	Zr	PNL-ALO-115	ICP-AES	PNL-ALO-211
VP	NH ₃	PNL-ALO-103	ISE	PNL-ALO-226
VP/FVP	C ₂ O ₄	PNL-ALO-103	IC	PNL-ALO-212
VP/FVP	Cl	PNL-ALO-103	IC	PNL-ALO-212
VP	CN	Direct	Colorimetric	PNL-ALO-285/289
VP/FVP	F	PNL-ALO-103	IC	PNL-ALO-212
VP/FVP	NO ₂	PNL-ALO-103	IC	PNL-ALO-212
VP/FVP	NO ₃	PNL-ALO-103	IC	PNL-ALO-212
VP/FVP	PO ₄	PNL-ALO-103	IC	PNL-ALO-212
VP/FVP	SO ₄	PNL-ALO-103	IC	PNL-ALO-212
VP	TIC/TOC	Direct	Hot Persulfate	PNL-ALO-381
FVP	pH	Direct	Potentiometric	PNL-ALO-225
FVP	OH	Direct	Titration	PNL-ALO-228

CVAA = Cold Vapor Atomic Absorption

FVP = Final Vendor Product

IC = Ion Chromatography

ICP-AES = Inductively Coupled Plasma Atomic Emission Spectrometry

ICP-MS = Inductively Coupled Plasma - Mass Spectroscopy

ISE = Ion Specific Electrode

VP = Vendor Product

Table 2.2. Radionuclide Preparation and Analysis Methods for Reported Results⁽¹⁾

Radionuclide	Prep Method	Analysis Method	Procedure
Total Alpha	PNL-ALO-115	alpha counting	PNL-ALO-420/421
Total Beta	PNL-ALO-115	beta counting	PNL-ALO-430/431
^{110m} Ag	PNL-ALO-115	GEA	PNL-ALO-450
²⁴¹ Am	PNL-ALO-115	separation, AEA	PNL-ALO-417/422
²⁴² Am	PNL-ALO-115	separation, AEA	PNL-ALO-417/422
¹⁴ C	Direct	oxidize, LSC	PNL-ALO-428/474
^{113m} Cd (2)	N/A	Not Measured	N/A
¹⁴⁴ Ce (¹⁴⁴ Pr, ^{144m} Pr)	PNL-ALO-115	GEA	PNL-ALO-450
²⁴² Cm (²⁴² Am, ^{242m} Am)	PNL-ALO-115	separation, AEA	PNL-ALO-417/422
²⁴³⁺²⁴⁴ Cm	PNL-ALO-115	separation, AEA	PNL-ALO-417/422
⁶⁰ Co	PNL-ALO-115	GEA	PNL-ALO-450
¹³⁴ Cs	PNL-ALO-115	GEA	PNL-ALO-450
¹³⁵ Cs	PNL-ALO-115	TIMS	PNL-ALO-452
¹³⁷ Cs (¹³⁷ Ba)	PNL-ALO-115	GEA	PNL-ALO-450
¹⁵² Eu	PNL-ALO-115	GEA	PNL-ALO-450
¹⁵⁴ Eu	PNL-ALO-115	GEA	PNL-ALO-450
¹⁵⁵ Eu	PNL-ALO-115	GEA	PNL-ALO-450
⁵⁵ Fe	PNL-ALO-115	separation, X-ray count	PNL-ALO-435/450
³ H (3)	N/A	Not Measured	N/A
¹²⁹ I	PNL-ALO-129	ICP-MS	PNL-ALO-280
^{93m} Nb	PNL-ALO-115	separation, beta count	PNL-ALO-4003
⁵⁹ Ni	PNL-ALO-116	separation, X-ray count	PNL-ALO-495/450

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Table 2.2. Radionuclide Preparation and Analysis Methods for Reported Results⁽¹⁾

Radionuclide	Prep Method	Analysis Method	Procedure
⁶³ Ni	PNL-ALO-116	separation, LSC	PNL-ALO-495/450
²³⁷ Np	PNL-ALO-116	ICP-MS	PNL-ALO-280
¹⁰⁷ Pd	PNL-ALO-129	ICP-MS	PNL-ALO-280
²³⁸ Pu	PNL-ALO-115	separation, AEA	PNL-ALO-417/422
²³⁹ Pu	PNL-ALO-116	ICP-MS	PNL-ALO-280
²⁴⁰ Pu	PNL-ALO-129	ICP-MS	PNL-ALO-280
²³⁹⁺²⁴⁰ Pu	PNL-ALO-115	separation, AEA	PNL-ALO-417/422
²⁴¹ Pu	PNL-ALO-115	separation, LSC	PNL-ALO-417/474
²⁴² Pu	PNL-ALO-115	separation, AEA	PNL-ALO-417/422
¹⁰⁶ Ru (¹⁰⁶ Rh)	PNL-ALO-115	GEA	PNL-ALO-450
¹²⁵ Sb (^{125m} Te)	PNL-ALO-115	GEA	PNL-ALO-450
⁷⁸ Se	PNL-ALO-115	distillation, LSC	PNL-ALO-440/474
¹⁵¹ Sm+ ¹⁴⁷ Pm	PNL-ALO-115	separation, LSC	PNL-ALO-417/474
¹¹³ Sn (^{113m} In)	PNL-ALO-115	separation, X-ray and gamma count	PNL-ALO-453/450
^{119m} Sn	PNL-ALO-115	separation, X-ray and gamma count	PNL-ALO-453/450
^{121m} Sn	PNL-ALO-115	separation, X-ray and gamma count	PNL-ALO-453/450
¹²⁶ Sn (¹²⁶ Sb, ^{126m} Sb)	PNL-ALO-115	separation, X-ray and gamma count	PNL-ALO-453/450
⁹⁰ Sr (⁹⁰ Y)	PNL-ALO-115	separation, beta count	PNL-ALO-476/431
⁹⁹ Tc	PNL-ALO-129	ICP-MS	PNL-ALO-280/281
²³² Th	PNL-ALO-116	ICP-MS	PNL-ALO-280
²³³ U	PNL-ALO-129	ICP-MS	PNL-ALO-280/282
²³⁴ U	PNL-ALO-129	ICP-MS	PNL-ALO-280/282

Table 2.2. Radionuclide Preparation and Analysis Methods for Reported Results⁽¹⁾

Radionuclide	Prep Method	Analysis Method	Procedure
²³⁵ U	PNL-ALO-129	ICP-MS	PNL-ALO-280/282
²³⁶ U	PNL-ALO-129	ICP-MS	PNL-ALO-280/282
²³⁸ U	PNL-ALO-116	ICP-MS	PNL-ALO-280/282
⁹³ Zr	PNL-ALO-115	separation, LSC	PNL-ALO-4007/474

AEA = Alpha Energy Analysis

VP = Vendor Product

GEA = Gamma Energy Analysis

FVP = Final Vendor Product

LSC = Liquid Scintillation Count

ICP-MS = Inductively Coupled Plasma Mass Spectroscopy

TIMS = Thermal Ionization Mass Spectroscopy

(1) Radiochemistry performed on Vendor Product only, not on Final Vendor Product.

(2) No method available for determining ^{113m}Cd.

(3) Tritium analysis was not performed because all samples were dried, and tritium loss was assumed.

Table 2.3. Physical Measurement Methods for Reported Results

Material	Physical Test	Analysis Method	Procedure
VP	Vol% Solids	Volume/Height Measurement	PNL-ALO-501
VP/FVP	Density	Volume/Mass Measurement	PNL-ALO-501
VP/FVP	Wt% Solids	Gravimetric (Dried at 105 °C)	PNL-ALO-501/504
VP/FVP	Wt% Oxides	Gravimetric (Fired at 1050 °C)	PNL-ALO-501
VP	Shear Strength/Apparent Viscosity/Yield Strength	Viscometer	PNL-ALO-501/502
VP	Heat Capacity	DSC	PNL-ALO-508

DSC = Differential Scanning Calorimetry

FVP = Final Vendor Product

VP = Vendor Product

3.0 SUMMARY OF ANALYTICAL RESULTS AND ASSESSMENT OF DATA QUALITY

3.1 Analytical Results

All analytical results for the Vendor Product and Final Vendor product have been reported previously (Urie 1997, Kuhl-Klinger and Urie 1997). A summary of the best available analytical results and data quality assessment for the inorganic analyses and radionuclide analyses performed on Vendor Product and Final Vendor Product are presented in Table 3.1 and Table 3.2, respectively. The "Method" identifies both the analysis methods and preparation method used to generate the reported analytical results, and "VP or FVP" identifies the material analyzed (i.e., either Vendor Product or Final Vendor Product). Where available, the analytical results for the Final Vendor Product are used for data quality evaluation. Where not available, the Vendor Product results are used for data quality evaluation and comparison to the Envelope D specification. The data quality evaluation is expected to be applicable to the Original Material analysis as well.

The "Average" is the mean analyte or radionuclide concentration for the six analyses on the three subsamples of the Vendor Product, and the five analyses on the four samples of Final Vendor Product. The reported "Average" result uses only results which are above the detection limit; i.e., any results less than the detection limit are not included in the average. This approach has been taken to ensure that averages are not biased low by the less than results, and that the highest (i.e., most conservative) average is used for comparison to the Envelope D specification. If all measurements for an analyte or radionuclide are below the detection limit, the "Average" result is reported as the highest less than value, and for these values the relative standard deviation (RSD) is not applicable ("NA").

Although there may be six sets of analytical data for some analytes resulting from all possible combinations of two analytical techniques (Inductively coupled plasma atomic emission spectrometry [ICP-AES] and inductively coupled plasma mass spectroscopy [ICP-MS]) and three sample preparation techniques (KOH fusion, Na_2O_2 fusion, and acid digestion), only one set of data is selected for reporting and data quality evaluation (e.g., ICP-AES acid digestion). In selecting this set of data, an effort has been made to evaluate the characteristics of the analytical technique and the limitation of the preparation method. In general, where the preparation methods are considered equivalent and the measured result for the analyte (by either ICP-AES or ICP-MS) is above the estimated quantitation limit (EQL), the highest reported average is selected. The exceptions to this are where: a) the relative standard deviation for the analysis set is greater than 10%; b) processing blanks are greater than 5% of the reported results; c) the ICP-AES is near the EQL; and/or d) quality control failures are considered significant (e.g., matrix spike recovery at 30% or LCS recovery at 200%). Where the only reported results for ICP-AES and ICP-MS are less than values (i.e., beryllium, potassium, selenium, thallium, and yttrium), the ICP-AES results are used for evaluation since the ICP-AES results are considered the more reliable and provide the most conservative less than value.

Table 3.1. Analyte Results -- Data Quality Summary

Analyte	Method ⁽¹⁾	VP or FVP	Average µg/g	RSD (%)	Average/ D.Spec.	Result/ DL	Blank/ Sample (%) ⁽²⁾	QC Failures				
								Control Standard (%Rec)	Matrix Spike (%Rec)	Post Spike (%Rec)	Serial Dilution (%Rec)	Not Spiked
Ag	ICP NfF	FVP	1.62E+03	38	0.726	36	nbd					
Al	ICP NfF	FVP	6.68E+04	7	0.462	614	0.5					
As	ICP-MS NfF	VP	2.86E+02	15	0.222	286	4	127		156		
B	ICP-MS NfF	VP	2.29E+02	36	0.022	229	4					X
Ba	ICP ZrF	FVP	5.44E+02	3	0.012	30	nbd					
Be	ICP Acid	VP	<1.00E+00	N/A	<0.002	1	nbd					
Bi	ICP ZrF	FVP	6.60E+02	5	0.019	4	nbd					
Ca	ICP Acid	FVP	4.83E+03	7	0.068	78	1				12.3	
Cd	ICP Acid	FVP	8.40E+01	4	0.003	23	nbd					
Ce	ICP-MS ZrF	VP	2.02E+02	11	0.031	202	nbd	49				
Co	ICP-MS NfF	VP	2.60E+01	24	0.007	26	nbd					
Cr	ICP NfF	FVP	8.06E+02	5	0.143	22	nbd					
Cs	ICP-MS NfF	VP	1.80E+01	22	0.004	18	4					
Cu	ICP Acid	FVP	1.30E+02	6	0.040	21	2					
Dy	ICP-MS NfF	VP	2.02E+01	37	0.098	20	13	193				
Er	ICP-MS NfF	VP	1.31E+01	36	0.509	13	13					X
Eu	ICP-MS NfF	VP	7.86E+00	75	0.061	8	nbd	N/D@1µg/g				
Fe	ICP NfF	FVP	1.58E+05	4	0.614	3493	0.1					
Gd	ICP-MS NfF	VP	2.50E+01	28	0.323	25	nbd					X
Hg	ICP-MS NfF	VP	6.07E+00	25	0.236	6	nbd	395				
K	ICP Acid	VP	<4.92E+02	N/A	<0.047	1	nbd					
La	ICP Acid	FVP	1.12E+02	3	0.004	9	nbd					
Li	ICP Acid	VP	4.60E+01	65	0.042	6	nbd					
Mg	ICP NfF	FVP	1.48E+03	5	0.075	8	nbd					
Mn	ICP ZrF	FVP	3.20E+03	3	0.055	35	nbd					
Mo	ICP-MS NfF	VP	5.70E+01	24	0.011	57	9	258				
Na	ICP Acid	FVP	1.20E+05	6	1.048	3224	0.1				10.9	
Nb	ICP-MS NfF	VP	3.38E+01	77	0.437	34	nbd					X
Nd	ICP Acid	FVP	3.32E+02	4	0.021	13	nbd					
Ni	ICP ZrF	FVP	1.91E+03	6	0.086	35	5					
P	ICP ZrF	FVP	4.81E+03	4	0.239	27	nbd					
Pb	ICP ZrF	FVP	4.12E+03	3	0.392	23	nbd					
Pd	ICP-MS ZrF	VP	2.74E+01	19	0.027	27	nbd					
Pr	ICP-MS ZrF	VP	8.41E+01	6	0.030	84	nbd					X
Pt	ICP-MS NfF	VP	3.07E+01	25	1.192	31	14					
Rb	ICP-MS ZrF	VP	7.68E+00	38	0.005	8	nbd					
Rc	ICP-MS NfF	VP	6.61E+00	31	0.009	7	18					X
Rh	ICP-MS ZrF	VP	1.44E+02	11	0.140	144	nbd					
Ru	ICP-MS NfF	VP	9.57E+01	7	0.034	96	nbd					
S (Not Analyzed)	---	---	---	---	---	---	nbd					

Table 3.1. Analyte Results - Data Quality Summary (Cont.)

Analyte	Method	VP or FVP	Average µg/g	RSD (%)	Average/ D Spec	Result/ DL	Blank/ Sample (%)	QC Failures				
								Control Standard (%Rec)	Matrix Spike (%Rec)	Post Spike (%Rec)	Serial Dilution (%Rec)	Not Spiked
Sb	ICP-MS Acid	VP	7.00E-01	41	0.0001	35	nbd	32				
Se	ICP Acid	FVP	<3.69E+02	N/A	<0.090	1	nbd					
Si	ICP NiF	FVP	8.68E+04	6	0.461	96	nbd					
Sn	ICP-MS ZrF	VP	1.02E+02	28	0.360	102	2					
Sr	ICP NiF	FVP	1.00E+02	4	0.036	4	nbd	129				
Ta	ICP-MS ZrF	VP	2.86E+01	36	0.139	29	nbd					X
Tb	ICP-MS NiF	VP	8.20E+00	19	0.318	8	nbd					
Te	ICP-MS NiF	VP	4.24E+02	20	0.344	424	5					
Th	ICP Acid	FVP	4.28E+02	6	0.847	2	nbd					
Ti	ICP ZrF	FVP	1.24E+03	3	0.127	27	nbd					
Tl	ICP ZrF	VP	<1.23E+02	N/A	<0.034	1	nbd					
Tm	ICP-MS NiF	VP	2.64E+00	52	0.102	3	nbd					X
U	U-Laser	FVP	2.65E+03	0.01	0.018	16	1					
V	ICP-MS Acid	VP	3.72E+01	18	0.144	1860	11					
W	ICP-MS NiF	VP	4.98E-02	15	0.261	498	2	244				
Y	ICP Acid	VP	<8.60E+01	N/A	<0.067	1	nbd					
Yb	ICP-MS NiF	VP	1.26E+01	49	0.489	13	11	1208				
Zn	ICP-MS NiF	VP	3.06E+02	23	0.091	306	2	50				
Zr	ICP NiF	FVP	1.25E+03	38	0.009	14	nbd					
Br	IC water leach	VP	<1.16E+02	N/A	N/A	1	nbd					
F	IC water leach	VP	<2.91E+03	N/A	<0.103	1	nbd		144			
Cl	IC water leach	VP	1.26E+02	21	0.049	1	27					
CN	Direct	VP	9.50E+00	9	0.001	27	4					
Hg	Direct	VP	3.43E+02	5	0.444	7	nbd					
NO ₂	IC water leach	FVP	4.00E+02	0	N/A	2	nbd					
NO ₃	IC water leach	FVP	5.25E+02	10	N/A	2	25					
NO ₂ & NO ₃	IC water leach	FVP	9.25E+02	N/A	0.010	N/A	N/A					
PO ₄	IC water leach	FVP	2.75E+02	16	N/A	1	nbd					
SO ₄	IC water leach	FVP	3.25E+02	14	N/A	2	28					
CO ₃	IC water leach	FVP	8.33E+04	0.4	N/A	31	nbd					
NH ₃	ISE water leach	VP	2.13E+01	43	0.002	3	38					
TTC (CO ₂)	Direct	VP	5.30E+03	11	0.111	589	N/A					
TOC	Direct	VP	4.79E+04	7	0.547	2994	N/A					
TC	Direct	VP	5.22E+04	6	N/A	N/A	N/A					
pH	Direct	FVP	11.7	1	N/A	N/A	N/A					
OH (N)	Direct	FVP	0.021	6	N/A	N/A	N/A					

(1) Method: NiF = KOH Fusion (PNL-ALO-115); ZrF = Na₂O₂ Fusion (PNL-ALO-116);Acid = HNO₃/HCl Digestion (PNL-ALO-129); Water Leach (PNL-ALO-103).

(2) "nbd" = No blank concentration detected in process blank (i.e., blank concentration <DL).

N/A = Not Applicable; N/D = Not Detected

VP = Vendor Product

FVP = Final Vendor Product

Table 3.2. Radionuclide Results - Data Quality Summary

RADIONUCLIDE	Method ¹⁾	VP or FVP	Average µCi/g	RSD (%)	Average/ D Spec	Result/ MDA ²⁾	Blank/ Sample (%) ³⁾	QC Failures				
								Control Std. (%Rec)	Matrix Spikes (%Rec)	Post Spike (%Rec)	Serial Dilution (%Rec)	Not Spiked
Ag-110m	RNIF	VP	<9E-02	N/A	<349.4	1	nbd					
Am-241	RNIF	VP	3.15E+00	23	0.003	7875	nbd					
Am-243	RNIF	VP	<2E-02	N/A	<0.155	1	nbd					
C-14	Direct	VP	2.87E-03	191	0.056	4	N/A					
Cd-113 (not measured)	--	--	--	--	--	--	--					
Ce-144(Pr-144+Pr-144m)	RNIF	VP	<2E+00	N/A	<0.776	1	nbd					
Ce-144(Pr-144+Pr-144m)	RNIF	VP	<2E+00	N/A	<0.776	1	nbd					
Ce-144(Pr-144+Pr-144m)	RNIF	VP	<6E-03	N/A	<2.328	1	nbd					
Cm-242(Am-242+Am-242m)	RNIF	VP	8.07E-03	21	0.008	404	nbd					
Cm-242(Am-242+Am-242m)	RNIF	VP	9.76E-03	21	0.012	404	nbd					
Cm-242(Am-242+Am-242m)	RNIF	VP	9.76E-03	21	0.012	404	nbd					
Cm-243+Cm-244	RNIF	VP	9.86E-02	22	3827	4930	1					
Cm-243+Cm-244	RNIF	VP	9.86E-02	22	0.004	4930	1					
Co-60	RNIF	VP	4.91E-01	8	0.006	123	nbd					
Cs-134	RNIF	VP	<3E-01	N/A	<0.002	1	nbd					
Cs-135	RNIF	VP	2.86E-03	5	0.004	N/A	N/A					
Cs-137(Ba-137)	RNIF	VP	4.96E+02	5	0.006	24800	0.03					
Cs-137(Ba-137)	RNIF	VP	4.69E+02	5	0.006	24800	0.03					
Eu-152	RNIF	VP	<2E+00	N/A	<0.518	1	nbd					
Eu-154	RNIF	VP	3.79E+00	4	0.009	190	nbd					
Eu-155	RNIF	VP	3.13E+00	10	0.013	157	nbd					
Fe-55	RNIF	VP	1.53E-01	5	0.006	17	nbd	115				
Hi-3 (not Measured)	--	--	--	--	--	--	--					
I-129	ICP-MS Acid	VP	2.20E-04	14	0.099	1	nbd					X
Nb-93m	RNIF	VP	2.34E-01	56	0.104	1	nbd	71				
Ni-59	RNIF	VP	2.05E-02	24	0.057	1	nbd					
Ni-63	RNIF	VP	2.40E+00	6	0.058	1200	nbd					
Np-237	ICP-MS ZrF	VP	3.92E-03	20	0.007	6	4					X
Pd-107	ICP-MS Acid	VP	1.10E-03	118	0.011	4	nbd					X
Pu-238	RNIF	VP	6.31E-01	5	0.223	31550	0.2					
Pu-239	ICP-MS ZrF	VP	1.52E+00	7	0.063	25	2					X
Pu-240	ICP-MS Acid	VP	1.30E-01	8	0.019	26	nbd					X
Pu-239+Pu-240	RNIF	VP	2.99E+00	5	N/A	149500	nbd					
Pu-241	RNIF	VP	6.96E+00	6	0.039	17	0.4					
Pu-242	RNIF	VP	<2E-02	N/A	<10.93	1	nbd					
Ra-106(Rh-106)	RNIF	VP	<3E+00	N/A	<0.582	1	nbd					
Ru-106(Rh-106)	RNIF	VP	<3E+00	N/A	<0.582	1	nbd					

Table 3.2. Radionuclide Results - Data Quality Summary (Cont.)

RADIONUCLIDE	Method	VP or FVP	Average B/C ⁽¹⁾	RSD (%)	Average D Spec	Result MDA ⁽²⁾	Blank/ Sample (%)	QC Failures				
								Control Std (%Rec)	Matrix Spike (%Rec)	Post Spike (%Rec)	Serials Dilution (%Rec)	Not Spiked
Sb-125(Te-125m)	RNIF	VP	<2E+00	N/A	<0.008	1	nbd					
Sb-125(Te-125m)	RNIF	VP	<4E-01	N/A	<0.006	1	nbd					
Se-79	RNIF	VP	<2E-04	N/A	<0.019	1	nbd					
Sm-151(Pm-147)	RNIF	VP	9.23E+01	5	0.039	115	0.02					
Sm-151(Pm-147)	RNIF	VP	9.23E+01	5	0.022	115	0.02					
Sn-113(In-113)	RNIF	VP	<1E-02	N/A	<0.206	1	nbd					
Sn-113(In-113)	RNIF	VP	<1E-02	N/A	<0.206	1	nbd					
Sn-119m	RNIF	VP	<4E-02	N/A	<155.3	1	nbd					
Sn-121m	RNIF	VP	1.23E+00	43	5.305	14	nbd					
Sn-126(Sb-126m,Sb-126)	RNIF	VP	1.15E-2	28	0.009	2	nbd					
Sn-126(Sb-126m,Sb-126)	RNIF	VP	1.15E-2	28	0.013	2	nbd					
Sn-126(Sb-126m,Sb-126)	RNIF	VP	1.61E-3	28	0.013	2	nbd					
Sr-90(Y-90)	RNIF	VP	7.30E+02	6	0.009	104	0.01					
Sr-90(Y-90)	RNIF	VP	7.30E+02	6	0.009	104	0.01					
Tc-99	ICP-MS Acid	VP	5.60E-03	17	0.00005	16	nbd					X
Th-232	ICP-MS ZrF	VP	5.48E-05	33	N/A	548	nbd					X
U-233	ICP-MS Acid	VP	1.7E-03	20	65.99	9	nbd					X
U-234	ICP-MS Acid	VP	7.70E-04	18	0.039	8	nbd					X
U-235	ICP-MS Acid	VP	2.80E-05	1	0.034	700	nbd					X
U-236	ICP-MS Acid	VP	1.80E-05	14	0.009	18	nbd					X
U-238	ICP-MS ZrF	VP	6.71E-04	3	0.045	1997	0.01					X
Zr-93	RNIF	VP	<6E-01	N/A	<0.166	1	nbd					
Total Alpha	RNIF	VP	6.78E+00	3	N/A	6780	0.04					
Total Beta	RNIF	VP	2.05E+03	6	N/A	410000	0.01					

(1) The "bolded" radionuclides of the parent/daughter pairs are used to compare to the Envelope D specification; the daughter "averages" have been corrected for the decay fraction.

(2) Method: NIF = KOH Fusion (PNL-ALO-115); ZrF = Na₂O₂ Fusion (PNL-ALO-116); Acid = HNO₃/HCl Digestion (PNL-ALO-129); Water Leach (PNL-ALO-103).

(3) For ICP/MS the ratio is "Results/DL".

(4) "nbd" = No blank concentration detected in process blank (i.e., blank concentration <DL or <MDA).

VP = Vendor Product

FVP = Final Vendor Product

N/A = Not Applicable

The primary objective for the characterization analysis was to demonstrate that the Final Vendor Product complied with the Envelope D specifications (Jones 1996). The specifications are in grams per liter (g/L) at 31 grams of oxide for analytes of interest, and curies per liter (Ci/L) at 31 grams of oxide for radionuclides of interest. The "Average/D Spec." uses the average analyte (or radionuclide) result in $\mu\text{g/g}$ (or $\mu\text{Ci/g}$) converted to g/L (or Ci/L) using the weight percent solids and weight percent oxides. Sections 3.3.1 and 3.3.2 of the Analytical Summary Report Addendum 1 (Kuhl-Klinger and Urie 1997) provide an explanation of the equations used to calculate the g/L (or Ci/L) average results. Values for "Average/D Spec." less than 1.00 indicate that the average measured concentration of the material (either Vendor Product or Final Vendor Product) is within the Envelope D specification.

3.2 Data Quality – Comparison of Analytical Results to Detection Limits

The "Result/DL" or "Result/MDA" is the ratio of the average result to the reported detection limit (DL) or minimum detected activity (MDA). This ratio gives an indication of the reliability of the reported analytical measurement. The magnitude of this ratio has different significance for the various methods used for sample analysis. Typically, the EQL for a method is ten times DL, or equal to the lowest calibration standard used to calibrate the method. Therefore, for analyte concentration less than five to ten times the DL (i.e., $\text{Result/DL} < 5$), the variability of the analytical measurement is anticipated to be quite high. However, because of the high sensitivity (i.e., very low DL) of ICP-MS, a ratio less than 10 to 20 times DL is more reasonable for complex matrices such as C-106 waste. For radiochemistry measurements, the MDA provides an indication of the practical minimum activity which can be measured. Besides the MDA, propagated uncertainties and mean differences are used to used to evaluate radiochemical results near the MDA; see Section 4.4 of the Analytical Summary Report (Urie 1997) for explanation of uncertainty and mean difference evaluations. The variability in analytical results caused by measurements near the DL or MDA is most often reflected in a large RSD and high uncertainties for radiochemistry measurement. As can be seen in Table 3.1 and Table 3.2, over half of the reported ratios are low (i.e., < 20), indicating the reported average results should be used with discretion. Where detection limits are not available for a method, the "Results/DL" or "Result/MDA" is designated as "N/A".

3.3 Data Quality -- Preparation Blank Contribution

The presence of detectable levels of an analyte in the preparation blank is an indication of potential contamination during sample preparation or analysis. The "Blank/Sample" summarizes the contribution from the preparation blank, and is expressed as a percentage of the sample concentration for each analyte or radionuclide. Blank contributions greater than 5% of the sample concentration are considered significant and exceed the limits establish by the governing QA Plan, MCS-033 (Kuhl-Klinger 1996). Dysprosium, erbium, molybdenum, nickel, platinum, rhenium, vanadium, ytterbium, chloride, nitrate, sulfate, and ammonia have blank contributions exceeding 5%. With the exception of molybdenum, nickel, platinum, and vanadium, the analytes and their respective blank concentrations are at or near the DL, which may account for the high (i.e., $> 5\%$) contribution from the blank. With molybdenum, nickel, platinum, and vanadium at concentrations at least 31 times the DL, these blank contaminants contribute significantly to the uncertainty (both precision and accuracy) of the reported results. Nickel is a common, low-level contaminant in the Na_2O_2 fusion preparations; the source of the molybdenum, platinum, and vanadium blank contamination is unknown.

A few analytes (e.g., boron, calcium, copper, iron, sodium, uranium, and zinc) are common contaminants from the fusion and acid digestion preparations performed in the SAL hot cells. The blank concentration for these analytes contribute less than 5% to the sample concentration; therefore, the impact of these blank concentrations is considered to be insignificant.

3.4 Data Quality – Reproducibility

The "RSD" in Table 3.1 and Table 3.2 is the RSD of the mean for the data set reported. For this report, the RSD is used as the measure of reproducibility for the Vendor Product and Final Vendor Product results. With six measurements for the Vendor Product (i.e., three subsamples analyzed in duplicate) and five measurements for the Final Vendor Product (i.e., four samples with one being analyzed in duplicate), RSDs less than 10% are used to indicate high quality results. Subsampling variability (i.e., either bench-scale batch processing subsampling, SAL sample/duplicate splitting, or analysis preparation subsampling) is suggested when the RSD results are high (i.e., >10%) and the "Result/DL" results indicate the measured analyte (or radionuclide) is 10-20 times above the DL (or MDA).

For the reported analyte and radionuclide averages which are above the DL, approximately half of the results have RSD values above 10%. Based on the "Result/DL" and "Result/MDA" values, most of the large RSDs are the result of the analytes being at low concentration (i.e., within 5 to 20 times the DL or MDA). At these concentrations, reproducible results are difficult to obtain due to sample inhomogeneity. There are a few analytes where the results variability can be explained by potential contamination during sample preparation (i.e., vanadium) or from high standard carryover resulting in non-reproducible, biased high results (i.e., ^{14}C).

When the average results with RSD values exceeding 10% are evaluated with the "Results/DL (or MDA)" and "Average/D Spec." values, there are only a few analytes and radionuclides which are impacted by the high RSD values. For those radionuclides which exceed the Envelope D specification by over 5 times (i.e., ^{243}Cm , $^{121\text{m}}\text{Sn}$ and ^{233}U), the effect of large RSD values (e.g., 20% to 40%) is negligible. Regardless of the variability of the results, the results remain significantly over the specification. For analytes with results within 25% to 120% of the Envelope D specification, high RSD values (e.g., 20% to 80%) can have a significant effect if the a 95% confidence limit is used to evaluate the acceptability of the material. Silver, erbium, gadolinium, niobium, platinum (an analyte exceeding the Envelope D specification), tin, and ytterbium fall in this category.

For the Vendor Product (only), Welsh (1997) provides a thorough evaluation of the variability associated with both the sampling and analysis. Welsh addresses all analyses performed on the Vendor Product, and uses the three subsamples of Vendor Product from the bench-scale batch processing, analyzed in duplicate, to evaluate both the subsampling and analytical components of the variability. The differences between the three subsample results are used in determining the sampling variability; whereas, the differences between the duplicate measurements for each subsample are used in determining the analytical variability. The statistical results and evaluation are not reproduced in this report. Since analytical variabilities (and associated analysis problems) were similar for the analysis of the Original Material, Vendor Product, and Final Vendor Product, the quality of the data generated for all materials should be similar and can be used as a base guidance for future analyses of washed sludges.

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3.5 Data Quality -- Accuracy

The accuracy of the reported results is evaluated from laboratory standards or matrix spikes processed and analyzed with the samples, or from post-digestion spiking or serial dilution during the analysis of the samples. In most cases the batch quality control and analytical quality control sample results are within the requirements defined by the governing QA plan, MCS-033 (Kuhl-Klinger 1996). Table 3.1 and Table 3.2 present only those few cases where the LCS, Matrix Spike, Post-Digestion Spike, or Serial Dilution analysis failed to meet the QA Plan requirements. Where the QC sample failures occurred, the measured recovery values or percent difference values (for serial dilution) are reported.

Where no LCS was available and Matrix Spikes (or Post-digestion spikes) were not performed, the accuracy of the reported result can not be determined per the governing QA plan. The analytes which have no preparation QC to support an evaluation of the accuracy are indicated by an "X" under "Not Spiked". For these analytes, the analytical QC samples (e.g., instrument blanks and continuing calibration verifications, interference checks, etc.) were acceptable and met the QA plan requirements. Analytes and radionuclides with only analytical QC to support the reported result are praseodymium, rhenium, thulium, iodine-129, neptunium-237, technetium-99, palladium-107, and plutonium and uranium isotopes determined by ICP-MS.

Most of the LCS, Matrix Spike, or Post-digestion Spike failures resulted from recoveries exceeding the upper control limits for the standards. If the average reported results are corrected for these standard recoveries, the actual average concentration for these analytes is lower. Thus, there is a high probability that the measured average results are biased high. The analytes and radionuclides which may be biased high, based on high standard recoveries, are arsenic, dysprosium, holmium, molybdenum, strontium, tungsten, ytterbium, fluoride, and iron-55. There were some interferences noted for the fluoride measurement, possibly caused by the excess oxalate or co-eluting organic anions. Of more concern are the analytes and radionuclides which demonstrated low standard recoveries, since the measured average value may be significantly lower than the "true" concentration. Those analytes and radionuclides that may be biased low are cerium, antimony, zinc, and niobium-93m. However, the concentrations of these analytes and radionuclides do not exceed 10% of the Envelope D specification. Even considering the potential that the reported average result is biased low, there is little probability of exceeding the specification.

The pH and OH measurements were performed on the Final Vendor Product per procedures PNL-ALO-225 and PNL-ALO-228, respectively. Although both measurements demonstrated good precision, the pH results are a better estimate of the alkaline properties of the Final Vendor Product because the free hydroxide in the samples is too low to measure accurately by titration.

3.6 Preparation and Analysis Comparison for ICP-AES and ICP-MS Results

For a number of analytes, it is possible to compare ICP-AES results to the ICP-MS results; thus providing additional confidence in the results and gaining additional information on the chemistry of the Vendor Product waste material. Table 3.3 summarizes the ICP-AES results for all the preparation methods (i.e., KOH fusion, Na_2O_2 fusion, and acid digestion) and Table 3.4 summarizes the ICP-MS results. Each of the tables identifies those analytes for which there may be significant preparation method differences reported for the specific analysis method. Each table also identifies those analytes for which there may be significant differences between the analysis methods.

Table 3.3. Summary of ICP-AES Results for Vendor Product

ICP-AES/ ICP-MS Difference	Analyte	ICP-AES/ Prep Difference	ICP-AES KOH			ICP-AES Na ₂ O ₂			ICP-AES Acid		
			PB µg/g	Average µg/g	Stddev µg/g	PB µg/g	Average µg/g	Stddev µg/g	PB µg/g	Average µg/g	Stddev µg/g
x	Ag	x	--	1,030	240	--	3,180	680	--	76	5
	Al		[248]	51,200	2300	[218]	49,500	2400	[16]	47,900	1300
	As		--	< 479	n/a	--	< 510	n/a	--	< 123	n/a
	B		[90]	[303]	98	--	[154]	35	[119]	225	62
	Ba		--	407	19	[25]	437	22	--	415	10
x	Be		--	< 9	n/a	--	< 9	n/a	--	< 1	n/a
	Bi		--	[538]	14	--	[582]	58	--	430	11
	Ca	x	[1,120]	6,690	720	[4,180]	9,850	1000	[71]	3,840	93
	Cd		--	[81]	2	--	[81]	5	--	73	13
	Ce		--	< 523	n/a	--	< 1,580	n/a	--	[299]	11
	Co		--	< 87	n/a	--	< 93	n/a	--	[28]	1
	Cr	x	[63]	802	38	[45]	700	46	--	533	12
	Cs-133		n/m	n/m	n/m	n/m	n/m	n/m	n/m	n/m	n/m
	Cs-135		n/m	n/m	n/m	n/m	n/m	n/m	n/m	n/m	n/m
	Cu		[49]	[109]	5	[49]	[109]	15	--	78	2
x	Dy		--	< 87	n/a	--	< 93	n/a	--	< 12	n/a
	Er		n/m	n/m	n/m	n/m	n/m	n/m	n/m	n/m	n/m
	Eu		--	< 174	n/a	--	< 185	n/a	--	< 25	n/a
	Fe		[268]	141,000	7600	[270]	131,000	7800	[9]	137,000	3200
	Gd		n/m	n/m	n/m	n/m	n/m	n/m	n/m	n/m	n/m
	Hg		n/m	n/m	n/m	n/m	n/m	n/m	n/m	n/m	n/m
	Ho		n/m	n/m	n/m	n/m	n/m	n/m	n/m	n/m	n/m
	I-129		n/m	n/m	n/m	n/m	n/m	n/m	n/m	n/m	n/m
	K		n/a	n/a	n/a	[6,930]	[7,590]	1700	--	< 492	n/a
	La		--	[119]	9	--	[164]	26	--	[96]	3
	Li		--	[90]	6	[59]	[94]	25	--	46	30
	Mg	x	--	[1,360]	130	--	[1,310]	99	--	754	23
	Mn		[191]	3,140	140	--	2,590	160	--	2,600	60
	Mo		--	< 87	n/a	--	< 93	n/a	--	[27]	1
x	Na	x	2,990	142,000	9000	n/a	n/a	n/a	[161]	162,000	8000
	Nd		--	[379]	31	[254]	[517]	59	--	282	8
	Ni	x	n/a	n/a	n/a	597	1,660	120	--	1,130	31
	Np-237		n/m	n/m	n/m	n/m	n/m	n/m	n/m	n/m	n/m
	P	x	--	3,330	370	[239]	3,840	670	--	1,340	66
	Pb		--	2,950	510	[203]	3,530	210	--	3,430	78
	Pd		--	< 1,310	n/a	--	< 1,390	n/a	--	< 369	n/a
	Pd-107		n/m	n/m	n/m	n/m	n/m	n/m	n/m	n/m	n/m
	Pr		n/m	n/m	n/m	n/m	n/m	n/m	n/m	n/m	n/m
	Pt		n/m	n/m	n/m	n/m	n/m	n/m	n/m	n/m	n/m
	Pu-239		n/m	n/m	n/m	n/m	n/m	n/m	n/m	n/m	n/m

Table 3.3. Summary of ICP-AES Results for Vendor Product (Cont.)

ICP-AES/ ICP-MS Difference	Analyte	ICP-AES Prep Difference	ICP-AES KOH			ICP-AES Na ₂ O ₂			ICP-AES Acid		
			PB µg/g	Average µg/g	Stddev µg/g	PB µg/g	Average µg/g	Stddev µg/g	PB µg/g	Average µg/g	Stddev µg/g
	Pu-240		n/m	n/m	n/m	n/m	n/m	n/m	n/m	n/m	n/m
	Pu-242		n/m	n/m	n/m	n/m	n/m	n/m	n/m	n/m	n/m
	Rb		n/m	n/m	n/m	n/m	n/m	n/m	n/m	n/m	n/m
	Re		n/m	n/m	n/m	n/m	n/m	n/m	n/m	n/m	n/m
	Rh		--	< 523	n/a	--	< 556	n/a	--	< 74	n/a
	Ru		--	< 1,740	n/a	--	< 1,850	n/a	--	< 738	n/a
	Sb		--	< 871	n/a	--	< 927	n/a	--	< 74	n/a
	Se		--	< 610	n/a	--	< 649	n/a	--	< 369	n/a
	Si	x	--	68,900	4300	--	64,600	3900	[165]	2,800	430
	Sn		--	< 3,920	n/a	--	< 4,170	n/a	--	< 4,060	n/a
	Sr	x	--	[79]	5	[71]	[145]	12	--	54	1
	Ta		n/m	n/m	n/m	n/m	n/m	n/m	n/m	n/m	n/m
	Tb		n/m	n/m	n/m	n/m	n/m	n/m	n/m	n/m	n/m
	Tc-99		n/m	n/m	n/m	n/m	n/m	n/m	n/m	n/m	n/m
x	Te		--	< 2,610	n/a	--	< 2,780	n/a	--	523	12
	Th		--	< 1,740	n/a	--	< 1,850	n/a	--	[349]	5
	Ti	x	--	1,030	79	--	1,020	100	--	350	11
	Tl		--	< 871	n/a	--	< 927	n/a	--	< 123	n/a
	Tm		n/m	n/m	n/m	n/m	n/m	n/m	n/m	n/m	n/m
	U (total)		--	< 4,360	n/a	--	< 6,950	n/a	--	[1,950]	37
	U-233		n/m	n/m	n/m	n/m	n/m	n/m	n/m	n/m	n/m
	U-234		n/m	n/m	n/m	n/m	n/m	n/m	n/m	n/m	n/m
	U-235		n/m	n/m	n/m	n/m	n/m	n/m	n/m	n/m	n/m
	U-236		n/m	n/m	n/m	n/m	n/m	n/m	n/m	n/m	n/m
	U-238		n/m	n/m	n/m	n/m	n/m	n/m	n/m	n/m	n/m
	V		--	< 87	n/a	--	< 93	n/a	--	< 37	n/a
	W		--	< 3,480	n/a	--	< 3,710	n/a	--	< 492	n/a
	Y		--	< 131	n/a	--	< 139	n/a	--	< 86	n/a
	Yb		n/m	n/m	n/m	n/m	n/m	n/m	n/m	n/m	n/m
	Zn		--	[183]	25	--	[161]	14	[16]	[116]	3
x	Zr	x	--	1,110	530	n/a	n/a	n/a	--	661	200

ICP-AES/ICP-MS Difference: "x" = Differences between ICP-AES and ICP-MS results should be evaluated.

ICP-AES Prep Difference: "x" = Differences between KOH/Na₂O₂/Acid prep for ICP-AES should be evaluated.

"n/m" = not measured; "n/a" = not applicable; "n/u" = not used (matrix issue)

"<" = result <IDL; "[]" = results >IDL but <EQL

Table 3.4. Summary of ICP-MS Results for Vendor Product

ICP-MS/ ICP-AES Difference	Analyte	ICP-MS Prep Difference	ICP-MS KOH			ICP-MS Na ₂ O ₂			ICP-MS Acid		
			PB	Average	Stddev	PB	Average	Stddev	PB	Average	Stddev
			mg/g	mg/g	mg/g	mg/g	mg/g	mg/g	mg/g	mg/g	mg/g
X	Ag		n/a	n/a	n/a	n/a	n/a	n/a	n/a	n/a	n/a
	Al		n/a	n/a	n/a	n/a	n/a	n/a	1.22	33,433	2425
	As		12.3	286	44	6.6	256	55	0.97	155	12
	B		8.2	229	83	2.2	101	63	50.3	186	62
	Ba		3.35	412	19	<1	427	14	0.10	362	10
X	Be		n/m	n/m	n/m	n/m	n/m	n/m	n/m	n/m	n/m
	Bi		n/m	n/m	n/m	n/m	n/m	n/m	n/m	n/m	n/m
	Ca	X	65.3	1,723	218	133	3,767	336	84.0	9,573	1124
	Cd		1.54	90.6	11.4	<1	74.1	8.6	0.05	58.1	1.7
	Ce		0.15	160	28	<1	202	23	0.03	160	6
	Co		<1	26	6	<1	21.8	2.3	0.03	15.7	0.5
	Cr	X	11.9	774	84	5.99	739	47	0.67	426	11
	Cs-133		0.70	18.0	4.0	<1	7.18	2.17	<0.02	10.4	0.7
	Cs-135		n/a	n/a	n/a	n/a	n/a	n/a	n/a	n/a	n/a
	Cu	X	<1	21.7	9.6	1.2	156.3	12.7	0.4	110.0	2.0
X	Dy	X	2.68	20.2	7.5	<1	3.60	1.84	0.04	1.47	0.09
	Er	X	1.73	13.1	4.7	<1	<1	n/a	<0.02	0.8	0.0
	Eu		<1	7.86	5.91	<1	7.84	1.26	0.02	6.46	0.24
	Fe		n/a	n/a	n/a	n/a	n/a	n/a	20.2	109,700	8717
	Gd	X	<1	25.0	6.9	<1	12.1	3.4	<0.02	13.6	1.0
	Hg		n/a	n/a	n/a	n/a	n/a	n/a	n/a	n/a	n/a
	Ho	X	<1	6.07	1.53	<1	1.11	n/a	<0.02	0.14	0.04
	I-129		n/a	n/a	n/a	<20	<20	n/a	<1	1.27	0.25
	K		n/m	n/m	n/m	n/m	n/m	n/m	n/m	n/m	n/m
	La		0.63	80.6	12.1	<1	92.1	14.5	<0.02	84.9	3.4
X	Li		n/m	n/m	n/m	n/m	n/m	n/m	n/m	n/m	n/m
	Mg	X	2.6	1,220	156	7.05	1,257	81	2.82	446	17
	Mn		19.3	2,807	206	3.63	2,558	126	4.00	2,043	115
	Mo	X	5.14	57.0	13.7	1.30	27.9	4.5	0.07	9.60	0.60
	Na	X	n/a	n/a	n/a	n/a	n/a	n/a	27.9	181,667	27171
	Nd		5.49	233	34	<1	224	22	0.07	193	4
	Ni	X	n/a	n/a	n/a	30.5	1,605	62	7.40	948	56
	Np-237		n/a	n/a	n/a	<1	5.57	1.12	<0.02	4.48	0.19
	P		n/m	n/m	n/m	n/m	n/m	n/m	n/m	n/m	n/m
	Pb		n/a	n/a	n/a	2.76	2,553	95	8.31	2,853	192
	Pd		<1	23.5	13.1	<1	27.4	5.1	0.1	10.0	0.7
	Pd-107		n/a	n/a	n/a	n/a	n/a	n/a	<0.5	2.20	2.43
	Pr		<1	77.1	10.6	<1	84.1	5.3	0.02	70.5	2.0
	Pt	X	4.38	30.7	7.7	<1	5.19	1.66	0.10	0.2	0.1
	Pu-239	X	n/a	n/a	n/a	<1	24.6	1.7	<0.02	7.34	0.37

Table 3.4. Summary of ICP-MS Results for Vendor Product (Cont.)

ICP-MS/ ICP-AES Difference	Analyte	ICP-MS Prep Difference	ICP-MS KOH			ICP-MS Na ₂ O ₂			ICP-MS Acid		
			PB µg/g	Average µg/g	Stddev µg/g	PB µg/g	Average µg/g	Stddev µg/g	PB µg/g	Average µg/g	Stddev µg/g
	Pu-240		n/u	n/u	n/a	<1	<1	n/a	<0.02	0.56	0.04
	Pu-242		n/u	n/u	n/a	<1	<1	n/a	<0.02	0.02	0.00
	Rb	X	n/a	n/a	n/a	<1	7.68	2.92	0.2	1.10	0.11
	Re	X	1.16	6.61	2.06	<1	1.00	n/a	0.02	0.08	0.03
	Rh	X	<1	36.9	41.0	<1	144	16	<0.02	104.7	3.7
	Ru	X	<1	95.7	6.5	n/a	n/a	n/a	0.1	54.2	1.5
	Sb		<1	<1	n/a	<1	<1	n/a	<0.02	0.73	0.25
	Se		n/a	n/a	n/a	n/a	n/a	n/a	n/a	n/a	n/a
	Si		n/m	n/m	n/m	n/m	n/m	n/m	n/m	n/m	n/m
	Sn	X	<1	29.3	28.5	1.73	102	29	<0.02	34.8	1.5
	Sr	X	<1	59	15	6.87	150	13	0.07	45.1	0.9
	Ta		1.84	21.3	9.9	<1	28.6	10.2	0.09	10.4	2.9
	Tb	X	<1	8.20	1.55	<1	2.34	0.78	0.02	1.46	0.09
	Te-99		<1	<1	n/a	n/a	n/a	n/a	0.048	0.330	0.058
X	Te	X	22.0	424	85	4.4	288	99	1.74	203	8
	Th	X	n/a	n/a	n/a	<1	499	163	<0.2	185	6
	Ti	X	4.4	1,124	121	3.20	1,313	147	0.4	358	12
	Tl		n/m	n/m	n/m	n/m	n/m	n/m	n/m	n/m	n/m
	Tm	X	<1	2.64	1.36	<1	1.17	0.10	<0.02	0.06	0.02
	U (total)		<1	1,897	132	<1	2,010	66	2.12	1,905	72
	U-233		n/a	n/a	n/a	n/a	n/a	n/a	<0.02	0.17	0.03
	U-234		n/a	n/a	n/a	n/a	n/a	n/a	<0.02	0.12	0.02
	U-235		n/a	n/a	n/a	n/a	n/a	n/a	<0.02	13.0	0.8
	U-236		n/a	n/a	n/a	n/a	n/a	n/a	<0.02	0.29	0.04
	U-238		<1	1,887	132	<1	1,998	63	2.11	1,895	72
	V		n/a	n/a	n/a	n/a	n/a	n/a	4	37.2	6.8
	W		11.6	498	74	6.6	357	112	0.9	222	15
	Y		n/m	n/m	n/m	n/m	n/m	n/m	n/m	n/m	n/m
	Yb	X	1.39	12.6	6.2	<1	3.24	1.48	0.02	0.2	0.1
	Zn	X	5.21	306	71	1.50	234	44	5.95	99.3	6.7
X	Zr	X	1.99	433	230	n/a	n/a	n/a	0.25	256	67

ICP-MS/ICP-AES Difference: "X" = Differences between ICP-AES and ICP-MS results should be evaluated

ICP-MS Prep Difference: "X" = Differences between KOH/Na₂O₂/Acid prep for ICP-AES should be evaluated

"n/m" = not measured; "n/a" = not applicable; "n/u" = not used (matrix issue or data unusable)

"<" = results less than EQL

Comparison of the results generated by ICP-AES and ICP-MS (i.e., analysis methods) is presented only for the Vendor Product. A comparison is not possible for the Final Vendor Product, since only ICP-AES analyses were performed. Also, since the ICP-AES and ICP-MS comparative results for the Original Material are similar to the Vendor Product, no comparison of the Original Material ICP-AES and ICP-MS results is presented in this report.

For analytes with concentrations exceeding the ICP-AES EQL, ICP-AES and ICP-MS differences exceeding 20% are considered significant. For analytes with concentrations lower than the ICP-AES EQL, comparison between ICP-AES and ICP-MS are difficult due to the high uncertainty of the ICP-AES result. For analytes exceeding the ICP-AES EQL, the ICP-AES and ICP-MS results from the same preparation method agree quite well, except for calcium and zirconium (and possibly aluminum, iron, and tellurium). Although the difference in the sodium results between ICP-AES and ICP-MS is acceptable per the criteria set forth in this assessment (i.e., ICP-MS is about 12% higher than ICP-AES), the difference is of concern since a major component of the Vendor Product is sodium, and sodium will be used as a metric for privatization contractor service payment. As is expected, for each analysis method, there are significant differences between the results for each of the preparation methods.

3.6.1 Evaluation of Differences Between ICP-AES and ICP-MS Results

As stated above, the differences between the analysis methods (for those analytes detectable by ICP-AES) were for calcium, zirconium, and possibly aluminum, iron, sodium, and tellurium. It is important to note that ICP-AES and ICP-MS Vendor Product analyses were performed on the same dissolution (only different aliquots), as were all batch quality control samples. Table 3.3 (ICP-AES) and Table 3.4 (ICP-MS) identify analytes which show significant difference between the analysis methods. The "ICP-AES/ICP-MS Difference" column contains an "X" if, based on the standard deviation and variability of the results between analysis methods (except for sodium), the difference between average results is considered significant.

Calcium: The first issue is the high (and differing) blanks observed in the ICP-AES analysis and not in the ICP-MS analysis. This has a significant impact on the gross ICP-AES result; however, correcting for the blank (or more probably spectral background) the results should (but do not) compare favorably to the ICP-MS for each specific preparation. The second issue is that the LCS and post-digestion spikes provided excellent recoveries for the ICP-AES analysis; however, the LCS recovered very poorly for the ICP-MS. This is the primary reason for selecting the ICP-AES acid digestion results. Also, no apparent interferences were noted in wavescans for the ICP-AES.

Zirconium: The difference between the ICP-AES and ICP-MS is about a factor of 2.5, with ICP-AES being higher. This ratio is consistent between the preparations (i.e., KOH fusion and acid digestion), suggesting either a high bias for the ICP-AES or a low bias for the ICP-MS. The quality control results for the ICP-MS were unacceptable and the recoveries were all high which would indicate that the reported results for ICP-MS are high. This is inconsistent with the difference between the methods. ICP-AES results were chosen primarily because of the ICP-MS QC sample failures.

Iron: The ICP-MS QC was unacceptable and biased high. However, the single average ICP-MS result (from acid digestion) is significantly lower than the ICP-AES result from the

acid digestion, as well as the ICP-AES iron results from the two fusion preparations. The ICP-AES results are considered more reliable than the ICP-MS results primarily due to the ICP-MS QC failures and the consistency of the ICP-AES iron results between preparation methods (i.e., 131,000 to 141,000 µg/g).

Aluminum: The ICP-AES aluminum analyses are consistent between the three different preparations (i.e., 47,900 to 51,200 µg/g), and all the QC associated with the ICP-AES aluminum analysis were within acceptable limits. The ICP-MS reported only results for the acid digestion and the QC related to the aluminum analysis had poorer recoveries than the ICP-AES. Based on this, the ICP-AES analysis results were selected for evaluation and reporting. In general, the ICP-AES provides better aluminum results for samples containing very high aluminum concentrations (e.g., >3%) than the ICP-MS.

Tellurium: The ICP-AES reported result from the acid digestion is only slightly higher than the EQL, and based on this and peak wavescans, there is a high probability that the tellurium result by the ICP-AES is biased high by as much as a factor of two. Although post spike recoveries were good for the ICP-AES, these recoveries are based on concentration differences and do not reflect potential background or interelement interferences which may not be adequately corrected for in the reported results. Based on this and the acceptable ICP-MS control standard recoveries, the ICP-MS result from the KOH fusion was selected for evaluation and reporting.

Sodium: Based on the relatively high RSD for the ICP-MS results, there appears to be no statistical difference between the ICP-MS and ICP-AES results. The ICP-AES recovery on the LCS was within acceptable limits, although the recovery was slightly low at 90%. Therefore, there remains an uncertainty as to the "true" concentration of the sodium in the Vendor Product; however, the average sodium concentration by both ICP-AES and ICP-MS exceeded the Envelope D specification.

3.6.2 Evaluation of ICP-AES and ICP-MS Differences Between Preparation Methods

Table 3.3 (ICP-AES) and Table 3.4 (ICP-MS) identify analytes which show significant differences between the preparation methods (within each analysis method). The "Prep Difference" column contains an "X" if, based on the standard deviation and the average concentration (i.e., if above or below the analysis method EQL), the average results between preparation methods differs by 20%. The differences reported for the greatest majority of these analytes can be attributed either to the varying contributions from blanks (e.g., calcium or nickel), the ruggedness of the various preparation methods for specific analytes (e.g., acid digestion may not fully solubilize certain analytes, such as chromium, bismuth, silver, titanium, phosphorus, silicon, zirconium, platinum group metal, etc.) or to the measured analyte concentration (e.g., fusion preparations may dilute an analyte too near to the detection limit). The exceptions to this appear to be differences measured between the two fusion methods for copper, molybdenum, platinum, rhodium, tin, and strontium ICP-MS results. Copper, molybdenum, tin, and strontium ICP-AES results were more consistent than ICP-MS, and an error in the ICP-MS results for one or both of the fusions is suspected.

Although the acid digestion showed fewer interferences than the fusion preparations (for both the ICP-MS and ICP-AES), the acid digestion results were consistently lower than those from the fusion preparations. Based on the preparation observations (i.e., residue remained following the acid

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digestion preparation) and the consistently lower results, the acid digestion results are considered less reliable than those from the fusion preparations. Also, the variability in the calcium results can not be explained. The fusion dissolution method should provide reasonable results for calcium, with the acid digestion less likely to provide full dissolution. The difference between the KOH fusion and acid digestion of over five times, with the acid digestion showing the highest concentration, is inconsistent with other ICP-MS results (i.e., Original Material results) and all ICP-AES results.

For the acid digestion ICP-AES silver analysis, the matrix spike recovery for the acid digestion was only 12%, indicating significant matrix/procedure problems. The additional chloride in the Na_2O_2 fusion procedure may enhance the solubilization of the silver; however, this has not been confirmed in presence of high sodium and aluminum concentrations. The silver results from the Original Material, Vendor Product, and Final Vendor Product suggest that the higher chloride in the Na_2O_2 fusion assisted solubilization, but was still inadequate as evidenced by the high RSD values.

3.7 Analyte/Radionuclide Comparisons Based on Different Methods/Procedures

Besides the analytes measured by ICP-AES and ICP-MS, there are a few additional analytes which have been analyzed (either directly or indirectly) by more than one method. For these cases, it is possible to compare the results from the different methods. Comparing the results from different methods can provide additional confidence in the reported results. A comparison of these methods is summarized in Table 3.5.

The comparison between the methods is very good, except for phosphorus by ICP-AES and IC and the summation of ^{239}Pu and ^{240}Pu by ICP-MS and radiochemistry. Of particular interest is the comparison of the carbon measured by the TOC hot persulfate method and carbon measured as oxalate by IC. The results suggest that nearly all, if not all, of the organic carbon is in the form of oxalate.

The ICP-AES analysis measures the total phosphorus and IC measures only the soluble phosphate present in the water leach of the Vendor Product. These methods will only produce comparable results if all the phosphorus is present as water soluble phosphates. Because the ICP-AES result for phosphorus is significantly greater than the IC result, there is an indication that majority of the phosphorus is present as a non-water soluble species.

The radiochemical $^{239+240}\text{Pu}$ determinations were performed on the samples prepared from the KOH fusion only. The fusion preparation technique is an excellent method for dissolution of refractories (such as plutonium oxides). The subsequent radiochemical separations were performed on an aliquot of the acidified KOH melt using well-established separation procedures. The ^{239}Pu and ^{240}Pu were measured together (along with ^{238}Pu) by alpha energy spectrometry. Inductively coupled plasma mass spectroscopy measured the ^{239}Pu and ^{240}Pu separately on the Na_2O_2 fusion and acid digestion preparation. As expected, due to the uncertainty of the solubilization of plutonium by the acid digestion, the ICP-MS ^{239}Pu and ^{240}Pu concentrations for the acid digested samples were considerably lower than the concentrations measured for the fusion samples. When adjusted for the specific activity, the ICP-MS ^{239}Pu and ^{240}Pu results from the Na_2O_2 fusion preparation should provide similar results to radiochemistry results from the KOH fusion preparation; however, the radiochemistry results are nearly twice those of the ICP-MS. Although the ^{239}Pu and ^{240}Pu results from the radiochemistry analysis are a sum of the two isotopes (i.e., separate isotopes are not available for comparison to the Envelope D specification), the radiochemistry result is considered most accurate.

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To confirm this conclusion, the ICP-MS results and radiochemical results for the Original Material were compared to the Vendor Product results. In general, the concentrations for refractory and/or rare earth analytes analyzed by ICP-MS on the Na_2O_2 fusion preparations (e.g., cadmium, chromium, copper, manganese, cerium, lanthanum, and neodymium) were approximately two times higher in the Vendor Product than in the Original Material. Plutonium should demonstrate a similar increase in concentration. However, the average activity concentration for the ICP-MS $^{239/240}\text{Pu}$ on the Vendor Product is only half the average activity concentration measured on the Original Material (i.e., 1.53 $\mu\text{Ci/g}$ versus 2.37 $\mu\text{Ci/g}$). The radiochemistry average $^{239+240}\text{Pu}$ result for the Vendor Material was twice the result measured on the Original Material (i.e., 2.99 $\mu\text{Ci/g}$ versus 1.55 $\mu\text{Ci/g}$), consistent with expectations.

Table 3.5. Analyte Comparison From Different Methods/Procedure for Vendor Product⁽¹⁾

Analyte	Units	Method/Procedure	Average Result	RSD ⁽²⁾	Compared Results ⁽³⁾	RPD
Carbon	$\mu\text{g/g}$	TOC by Hot Persulfate	47,900	7	47,900	3.2
		Oxalate by IC / 3.66	170,00	8	46,400	
Phosphorus	$\mu\text{g/g}$	Phosphorus by ICP	3,330	11	3,330	194
		Phosphate by IC / 3.07	160	23	52	
Total Alpha ^(4,5)	$\mu\text{Ci/g}$	Alpha Counting	6.78	3	6.78	0.4
		$\sum ^{238,239+240}\text{Pu} + ^{241}\text{Am}$	0.61+2.99+ 3.15	<20	6.75	
Total Beta ⁽⁵⁾	$\mu\text{Ci/g}$	Beta Counting	2050	6	2050	4.7
		$\sum ^{137}\text{Cs} + ^{90}\text{Sr}(^{90}\text{Y})$	496+730+ 730	<6	1956	
$^{239+240}\text{Pu}$	$\mu\text{Ci/g}$	ICP-MS $^{239}\text{Pu} + ^{240}\text{Pu}$	1.52+0.13	<8	1.65	58
		Separations/AEA, $^{239+240}\text{Pu}$	2.99	5	2.99	

- (1) Average reported results for the Vendor Product are summarized in Analytical Summary Report (Urie).
- (2) RSD based on average reported result. Where multiple results are summed, "<" indicates that each RSD value is less than highest RSD, which is the one reported.
- (3) For comparison, the reported results are converted to the analyte/radionuclide of interest by either adjusting for stoichiometry or summing the appropriate radionuclides.
- (4) Only the radiochemistry results for the Vendor Product are compared (i.e., the $^{239+240}\text{Pu}$ is from separations/AEA not from ICP-MS).
- (5) Only alpha-emitters and beta-emitters with significant activity are summed for comparison.

4.0 VENDOR PRODUCT PROCESSING AND PHYSICAL MEASUREMENT

From the Vendor Product processing and physical measurement activities, numerous issues have been identified which require further evaluation and clarification. Issues addressed in this report are the difference in the sodium concentration of the laboratory-scale product and the bench-scale processing Vendor Product, the adequacy of the subsampling of the Vendor Product (and Original Material) from the bench-scale processing apparatus, the transferring/bottling procedure used to prepare the initial Vendor Product bottles for shipping, the weight percent solids discrepancies between the HLRF, and SAL, and the apparent loss of about 230 grams of Vendor Product.

4.1 Discrepancies Between Laboratory-Scale and Bench-Scale Results

The laboratory-scale screening test was conducted to provide operational parameters for the bench-scale batch preparation of the Vendor Product. Since the bench-scale processing was initiated prior to completion of the laboratory-scale screening test, these parameters were used by the bench-scale processing, but only as available. The laboratory-scale screening test prepared a product which met the Envelope D Specifications for selected analytes measured, including sodium. However, following the bench-scale processing, the sodium concentration in the Vendor Product was determined to exceed the Envelope D Specification (and was about 50% higher than the laboratory-scale product). The reason for the discrepancy is hypothesized as follows:

Lower Weight Percent Solids: The water wash steps during the laboratory screening test were performed at a lower weight percent solids than those used during the bench-scale processing. Therefore, more water was used in the laboratory-scale screening test to wash out the sodium from a diluted Initial Composite Material than in the bench-scale processing. The goal of the "baseline" enhanced sludge washing is to obtain five weight percent solids during the washes. For the laboratory-scale screening test, this requires estimating the fraction of solids that will dissolve (usually assumed to be 30% of the water insoluble sludge). The bench-scale processing then uses the actual dissolution results of the laboratory-scale screening test to determine the quantity of inhibited water to use for the water washes.

The bench-scale processing used the calculated laboratory-scale screening test initial solids concentrations and final solids concentration to determine the required inhibited water addition to obtain five weight percent solids. However, results of measurements, following the bench-scale processing, determined the weight percent solids of the bench-scale processing feed material (Original Material) to be about 20% higher than expected. Thus, instead of obtaining five weight percent solids during washing, the slurry was estimated to be between six to seven weight percent solids. This is about two times the solids concentration used during the laboratory-scale testing, and it is anticipated that the washing efficiency was reduced.

On evaluation of the laboratory-scale data following in bench-scale processing, it was determined that the laboratory-scale screening test underestimated the solids dissolution and obtained a three weight percent (instead of five weight percent) slurry during water washes. Using this data resulted in the higher bench-scale processing weight percent solids and reduced washing efficiency.

High Sodium Oxalate Concentration: The Original Material (feed material to the bench-scale processing) appears to contain a significant quantity of sodium oxalate. Oxalate ions represented approximately 20% (by weight) of the Original Material (on a dry weight basis), and a significant fraction of this oxalate appears to be sparingly soluble sodium oxalate. During the caustic leach, the high sodium concentration from the NaOH results in the equilibrium shifting toward the solid phase, thus preventing the sodium from being washed from the sludge during these steps. Once the sodium concentration is decreased during the water washes, the sodium oxalate began to dissolve, but only as its solubility equilibrium allows. The amount of sodium oxalate removed is a function of the total volume of water used in washing.

In summary, it appears that a proportionally smaller quantity of water was used in the bench-scale processing than in the laboratory-scale screening test. Therefore, a smaller fraction of the sparingly soluble sodium oxalate was removed in the washing process, resulting in a higher concentration of sodium in the Vendor Product.

4.2 Subsampling Adequacy of the Bench-Scale Processing Apparatus

The bench-scale batch processing apparatus was not validated to insure that the sampling method used to obtain the Original Material and Vendor Product subsamples for characterization and physical measurement provided subsamples representative of the bulk material being processed. The corrective actions related to changes in the bench-scale apparatus or operating parameters is beyond the scope of this report. However, the data from the Original Material, the Vendor Product, and the Final Vendor Product can be used to determine whether the subsampling method provides consistent and representative subsamples. Based on the data available, the subsampling appears very adequate for producing representative samples for the C-106 material.

Three subsamples each of the Original Material and Vendor Product were characterized in duplicate, thus providing analytical data to evaluate the bench-scale processing subsampling uncertainties (i.e., comparison of the analytical results for the three subsamples) and to evaluate the analytical uncertainties associated with the sample preparation (comparison of duplicates). In general, there is very good agreement between the analytes and radionuclides when comparing the results for the three subsamples for both the Original Material and Vendor Product. This is evident by the RSDs (Table 3.1 and Table 3.2) for concentrations significantly above the DL or MDA. Also, for the analytes and radionuclides considered to be insoluble during the washing and leaching processing, the Vendor Product results are consistently about twice the Original Material results (which is expected due to removal of soluble components from the solids). Additional supporting data detailing the sampling variability is provided by the Final Statistical Analysis Report (Welsh 1997). However, this only provides an indication that the bench-scale processing apparatus can provide "reproducible" subsamples, but not necessarily "representative" subsamples.

The analysis of the Final Vendor Product provides a further indication that the subsamples from the bench-scale processing apparatus are representative. In order to perform the decant/dilute sodium treatment operations, all Vendor Product settled solids were transferred to a single container, diluted with water, and blended to obtain a homogeneous composite. After the recompositing and rebottling operation performed per LOI revision 2, additional characterization was performed on the Final Vendor Product to confirm the estimated Final Vendor Product concentrations calculated from the Vendor Product results and decant/dilute parameters. The analysis of the Final Vendor Product

produced results consistent with expectations (i.e., the results agreed with the Vendor Product adjusted for decanting the Vendor Product supernatant and diluting with water) as described by Kuhl-Klinger and Urie (1997). The Final Vendor Product analyses were performed on subsamples initially pipetted in HLRF from the homogenized recomposite. Since this HLRF "stir-pipet" subsampling operation is independent of the bench-scale "pumping" subsampling operation, and since the result from both subsampling operations are analytically consistent, both subsampling methods are considered to provide representative subsamples.

4.3 Problems Resulting in the Need for Revision 2 to LOI

The initial bottling of the Final Vendor Product was accomplished by pouring the composited Final Vendor Product into twenty-two 125-mL sample bottles (twenty bottles for the two privatization contractors and two bottles for physical testing). The composited Final Vendor Product was stirred between pouring into each bottle. After the material in these twenty-two bottles had settled (approximately 3 days was required for the material to settle sufficiently such that no further settling was observed), the amount of solids in each bottle varied by as much as 30% (by volume). Also, the weight percent solids determined on the material collected for physical testing indicated a significantly higher solids content than anticipated (i.e., 21.5% versus an estimated 11.5% based on the decant/dilute sodium adjustment treatment data). Based on the discrepancy of the weight percent solids and the variability of the solids content in each bottle, the Final Vendor Product was recomposited and rebottled per revision 2 of the LOI. Section 4.4 provides additional details on the weight percent solids discrepancy.

In order to ensure that each vendor bottle contained approximately equal quantities of solids and to obtain more representative weight percent solids data than previously measured, the twenty-two bottles of Final Vendor Product and all additional Final Vendor Product were recomposited (i.e., transferred to a single container). Each of the bottles was rinsed with the settled supernatant to obtain essentially all of the solids. The recomposited Final Vendor Product was stirred with a magnetic stirrer and a stir bar to obtain a homogeneous composite. The Final Vendor Product was transferred back into the twenty-two 125-mL bottles by pipetting the samples while stirring the composite. The Final Vendor Product remaining after filling the vendor bottles was transferred to a 1-L bottle for archiving. After settling, each of the twenty vendor bottles contained approximately the same volume of settled solids (i.e., no visual difference in the volume of settled solids in these bottles was observed after three days). The ratio of the volume of settled solids to total volume for two bottles (C106-FP-V1-3 and C106-FP-V1-4) were measured. This ratio was 0.26 for both bottles. The qualitative visual inspection of the settled solids volume provided an indication that the contents (i.e., Final Vendor Product) of each privatization contractor bottle were considered representative of the bulk composite material.

The stir-pour, stir-pour, etc. transferring method appeared to be inadequate for ensuring that representative samples are transferred, particularly for materials with a similar ratio of settled solids to total volume. The inadequacy of this transfer method was primarily due to the settling behavior of the Final Vendor Product. It appeared that the heavier solids settled quickly; however, due to the opaqueness of the material, visual observation did not indicate that any settling had occurred. Visually, it appeared that the composite remained homogenous during the pouring process, whereas significant quantities of solids had settled. The continuous stirring and pipetting transfer method appeared to maintain a more consistent composite throughout the transferring operations.

4.4 Discrepancies Between SAL and HLRF Weight Percent Solids Results

Table 4.1 provides a summary of the physical measurements (excluding rheology and heat capacity) performed on the Vendor Product and Final Vendor Product. Details and evaluation of the physical measurement results (including rheology and heat capacity) are described by Urie (1997). Based on the RSD and RPD values in Table 4.1, the reproducibility of the physical measurements is very good; however, due to the unavailability of a control standard, the accuracy of these results is unknown. All physical measurements met the quality control requirements of the governing QA plan, MCS-033 (Kuhl-Klinger 1996).

Per revision 0 of the LOI, 100 grams of Envelope D material (dry weight basis) was to be available for each privatization contractor. Since the material was being shipped/transferred to each vendor as a slurry, accurate weight percent solids results were required to ensure that each vendor received at least 100 grams. Based on limited weight percent solids results, there appeared to be some inconsistencies between the results measured by the HLRF and those measured by the SAL. In general, the physical measurements performed in the HLRF use relatively small aliquots (1-3 grams) of the product slurries. Although the results are consistent, as evidenced by the good RPDs or RSDs, the use of these small sample aliquots may bias the results because of the difficulty in obtaining representative subsamples from small subaliquots of slurry. Based on the measured weight percent solids of the Vendor Product (14.4% HLRF and 14.8% SAL) and the estimated weight percent solids of Final Vendor Product (11.5%) following the decant/dilute sodium adjustment treatment (LOI Revision 1) and recompositing/rebottling operation (LOI Revision 2), the HLRF weight percent solids of 15.2% on the Final Vendor Product is inconsistent with expectations. However, the SAL weight percent solids for the Vendor Product and Final Vendor Product appear to be consistent with the expected results based on the decant supernatant analysis and decant/dilution data available. The SAL weight percent solids were obtained from drying the entire contents of the subsample (i.e., 30 to 100 grams of slurry). Although the drying times are considerably longer, and there is more opportunity for loss due to spattering, etc., the weight percent solids results obtained on the "bulk" 30 to 100 gram subsamples were considered the most reliable and were more conservative, and were used to determine the quantity of dry-equivalent Final Vendor Product supplied to each privatization contractor.

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Table 4.1. Summary of Physical Measurement for LOI Revisions 0, 1 and 2

LOI Revision	Measurement	Analysis Location ⁽¹⁾	VP or FVP ⁽²⁾	Average	RSD or RPD ⁽³⁾	n
Revision 0	Density (Slurry) (g/ml)	HLRF	VP	1.10	0.4	4
	Volume Percent Settled Solids (%)	HLRF	VP	18.5	10.8	2
	Weight Percent Solids (%) ^(4,5)	HLRF	VP	14.4	2.5	2
	Weight Percent Solids (%) ^(4,5)	SAL (Bulk)	VP	14.8	12	3
	Weight Percent Oxide (%) ⁽⁶⁾	HLRF	VP	11.4	1.7	2
Revision 1	Density (Slurry) (g/ml)	HLRF	FVP	1.13	2.7	2
	Volume Percent Settled Solids (%)	HLRF	FVP	39.2	0.8	2
	Weight Percent Solids (%) ^(4,5)	HLRF	FVP	21.5	2.3	2
	Weight Percent Oxide (%) ⁽⁶⁾	HLRF	FVP	18.6	0.3	2
Revision 2	Density (Slurry) (g/ml)	HLRF	FVP	1.05	1	4
	Weight Percent Solids (%) ^(4,5)	HLRF	FVP	15.2	7.9	4
	Weight Percent Solids (%) ^(4,5)	SAL (Bulk)	FVP	11.9	4.8	7
	Weight Percent Oxide (%) ⁽⁶⁾	HLRF	FVP	12.7	8.7	4

(1) HLRF = High-Level Radiation Facility; SAL = Shielded Analytical Laboratory

(2) VP = Vendor Product; FVP = Final Vendor Product

(3) RPD if n = 2; RSD if n > 2

(4) Sample size 1-3 g for HLRF measurements, 30-100 g for bulk SAL measurements

(5) Weight Percent Solids at 105 °C

(6) Weight Percent Oxides at 1050 °C

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4.5 Evaluation of Mass Loss of Vendor Product

Following the bench-scale batch processing, the Vendor Product was transferred from the 324 building to the HLRF. The contents of the two of the Vendor Product containers (SPD1-C106-051 and SPD1-C106-052) were to provide the material for shipment/transfer to the privatization contractors. There was 1624 grams of Vendor Product in container SPD1-C106-051 and 1523 grams in container SPD1-C106-052 (a total of 3147 g) when the containers were transferred from the 324 building. However, following the decant/dilute sodium adjustment treatment, only 2914 grams of Vendor Product was accounted for (a discrepancy of 233 g, 30 g of which can be attributed to viscosity sample). The Vendor Product accounted for following the decant/dilute sodium treatment is the sum of the SPD1-C106-051 Supernatant (1044.7 g), SPD1-C106-052 Supernatant (957.6 g) and the SPD1-C106-051+052 Settled Solids (911.6 g). Figure 4.1 provides the details of the handling of the contents of the SPD1-C106-051 and SPD1-C106-052 containers, and provides an indication as to where potential material losses may occur. All weight presented in Figure 4.1 are measured, except for the viscosity and shear strength samples which are estimates based on the quantity of material needed for analysis. Kuhl-Klinger and Urie (1997) provide the details of the decant/dilute sodium adjustment treatment.

In investigating the possible losses (or subsampling) of the Vendor Product, a detailed evaluation of the handling, testing, and sodium adjustment operations was performed. The losses fall into three categories as follows:

Subsampling Losses: Of the 3147 grams of Vendor Product, approximately 30 grams of slurry from SPD1-C106-051 was used for viscosity analysis and not returned to the container. The sample for shear strength analysis (approximately 150 grams of settled solids) was also removed from this container; however, this sample material was recombined following the analysis. There are small losses (2-3 grams) associated with the shear strength analysis (e.g., some material remains on the shear vane).

Holdup/Transfer Losses: For the decant/dilute sodium adjustment treatment, the supernatant from the settled Vendor Product remaining in SPD1-C106-051 and SPD1-C106-052, after removal of the viscosity and shear strength subsamples, was decanted and combined with the supernatant removed from the shear strength subsample. The losses associated with residue holdup in the two initial containers (SPD1-C106-051 and SPD1-C106-052) and other container surfaces (e.g. pipets) are considered to be about 0.5% to 1% of the quantity of material handled, or potentially about 30 grams. Previous experience also indicates that significant quantities of material are lost during material transfer operations. Since the Vendor Product is a fluid slurry, the loss is minimized. For this type of slurry, the estimated mass lost during transfers is about 0.5% to 1% of the material mass transferred, or potentially another 30 grams.

HNF-SD-WM-DP-225, REV. 1

Losses

- A) 1) Evaporation; 2) Residue Left on Bottles SPD1-C106-051 and SPD1-C106-052
- B) 1) Transfers; holdup on pipets, minor spill, etc.; 2) Evaporation Losses
- C) 1) Evaporation; 2) Residue Left on Shear Strength Container; 3) Residue Left on Shear Vane

Figure 4.1. Material Losses During Sodium Adjustment Treatment (LOI Revision 1)

Evaporation Losses: The majority of the loss is most likely due to evaporation occurring primarily during handling of the material (e.g., open containers during decanting and testing expose high surface areas for evaporation). Due to the hot cell temperatures, it is estimated that as much as 2-3% of the material evaporated (as water) during open container handling. Also, considerable evaporative losses from closed containers have been observed during storage. A mass loss of approximately 2% was observed over a month period while the Final Vendor Product bottles (capped 125-ml bottles) were stored in the HLRF hot cell while awaiting shipping/transferring to the privatization contractors (See data in Appendix 5D, Final Vendor Product Masses). The Vendor Product container, SPD1-C106-051 and SPD1-C106-052, were stored in the HLRF hot cells for approximately two months prior to opening and beginning the decant/dilute sodium treatment. Therefore, similar evaporation losses are anticipated (i.e., from 120 to 180 gram of water).

On further evaluation, the impact of this loss is considered insignificant on the data reported for the Final Vendor Product. The residual Vendor Product slurry which was left on pipettes, bottles, and working surfaces during transfers and handling only impacts the amount of Final Vendor Product available, and sufficient Final Vendor Product was available to meet the needs of the privatization contractors. The evaporation of water from the Vendor Product does increase the weight percent solids (and weight percent oxides) in the Vendor Product and the analyte concentration of the slurry. However, this evaporation does not impact the concentration of the analytes based on a dry weight basis. If all 203 grams of the unaccounted Vendor Product (233 grams - 30 gram Viscosity subsample) was due to the evaporation of the water, the weight percent solids would be 6.5% higher (15.3 weight percent solids instead of 14.4 weight percent solids [HLRF data]). The weight percent solids (and weight percent oxides) were measured on the Final Vendor Product following the recomposting and rebottling per revision 2 of the LOI; therefore, the weight percent solids measurements on the Vendor Product do not impact the quantity of solids provided to the privatization contractors.

5.0 ORIGINAL MATERIAL

5.1 Comparison of Original Material (Dried Samples to Centrifuged Solid+Liquid)

To provide additional information on the reliability of the analytical measurements, the Original Material slurry was analyzed as a slurry (after drying) and as solids and liquid fractions (after centrifuging). The dried slurry samples consist of all the solids plus the dissolved salts in the slurry liquid. The solids and liquid fraction samples were obtained by centrifuging the Original Material slurry and separating the solids and supernatant liquid fractions by decanting/pipetting the supernatant liquid.

The results for the dried Original Material have been presented by Urie (1997), and consist of duplicate analyses on three subsamples (SPDL1-C106-003, -004, and -005) extracted from the bench-scale processing apparatus just prior to beginning the sludge washing operations. A fourth subsample (SPDL1-C106-002) was collected for physical measurements testing, and was the source of the solid and liquid fractions which were analyzed in duplicate for a limited set of analyses. An aliquot of the fourth subsample was centrifuged and the solids and liquid fractions separated. The solid fraction was analyzed in duplicate by ICP-AES following KOH (PNL-ALO-115) and Na_2O_2 (PNL-ALO-116) fusions and by IC following water leaching (PNL-ALO-103). The liquid fraction was analyzed in duplicate by ICP-AES following an acid digestion (PNL-ALO-128) and directly by IC following filtering and dilution. To provide the information necessary to recombine the analyzed fractions,

weight percent solids (wet) of the centrifuged aliquot (43.4%), weight percent solids (dried at 105 °C) of the centrifuged solids fraction (51.7%), and density of the centrifuged liquid fraction (1.07 g/ml) were measured. To allow all results to be normalized to slurry concentration, the weight percent solids (dried at 105 °C) of the Original Material slurry (29.2%) was determined.

The averaged ICP-AES fusion results (KOH and Na₂O₂) and the IC analyses results from the dried Original Material slurry were compared to the summation of concentrations of the Original Material centrifuged solids and centrifuged liquid. Table 5.1 presents the comparison of the Original Material slurry to the summation of the centrifuged solids/liquid fractions. Although not shown on the table, the uncertainty of the analytical results between the duplicates (and particularly between the two fusion results) is approximately 20% for many analytes, and the uncertainties in the physical measurements used to combine and normalize the results are about 3% for each measurement. Also, some measurements used very small sub-aliquots (e.g. centrifuged solids weight percent solids dried at 105 °C used only 1 gram); this adds additional subsampling uncertainty to the results.

Except for a few analytes, the average concentrations for the slurry and centrifuged solid/liquid fractions are within $\pm 30\%$. Silver, barium, chromium, phosphorus, lead, uranium, nitrate, and phosphate are near or below the EQL and are presented for information only due to high uncertainties. Considering that both the phosphorus and phosphate in the centrifuged liquid were near the EQL, the ICP-AES and IC results compared reasonably well (i.e., 0.020% PO₄ as P from ICP and 0.013% PO₄ from IC). For the other analytes, the comparison may be considered bounding, considering the uncertainties associated with each analysis/measurement, the centrifuged fractions were extracted from a different subsample than the subsamples used for determining the slurry concentration, and the analytical work on the slurry and centrifuged fractions were performed two months apart.

Of particular interest is the sodium and oxalate of the centrifuged liquid fraction, since the analysis is very high in sodium (2.38 wt%) and low in oxalate and other measured anions (total anion = 1.17 wt%). Assuming all the anions are from sodium compounds, only about 30% of the total sodium (i.e., 0.7 wt%) can be attributed to these compounds; the remainder most probably being from sodium hydroxide. The oxalate is considerably lower than would be expected from the quantity of oxalate soluble in the water leaches of both the centrifuged solids fraction and the dried Original Material slurry. However, when compared to the oxalate concentration in the Original Material (5.9 wt%), the oxalate concentration in the centrifuged liquid fraction is similar to the concentration of oxalate determined in the suspected organic layer (Campbell 1996); i.e., 0.34 wt% versus about 0.6 wt%. Based on other work (Russell, et. al 1996; Smith, et.al 1996) the solubility of various oxalates appears to decrease as the pH increases. At a lower pH, the solubility of the various oxalates (e.g. sodium, iron, aluminum) increase, resulting in the higher oxalate concentration.

Although the pH of the centrifuged solids water leach samples was not measured, the pH is expected to be lower than the pH of the centrifuged liquid because the majority of the centrifuged liquid fraction (with a pH near 12) was removed prior to water leaching of the centrifuged solids. At this lower pH, the concentration of oxalates should increase, as is observed in the centrifuged solids leachates. An ICP-AES analysis on the water leach samples would identify the associated cations; however, no ICP-AES analysis was performed on the water leaches. Also, based on the data available, it is not possible to determine if all of the oxalate present in the Original Material slurry or the centrifuged solids was extracted by the water leaching (i.e., the oxalate may be in solubility equilibrium).

Table 5.1. Comparison of Original Material Slurry and Centrifuged Fractions

Analyte ⁽¹⁾	Centrifuged Orig. Mat'l SPDL1-C106-002			Original Material SPDL1-C106-003/005		Orig. Mat'l Compared to (CS+DL) % Diff
	Centrifuged Solids	Decant Liquid	Total (CS+DL) ⁽²⁾	Uncorr'd	Corr'd	
	Wt% Slurry ⁽³⁾	Wt% Slurry ⁽⁴⁾	Wt% Slurry	Wt% ⁽⁵⁾	Wt% ⁽⁶⁾	
Ag	0.011	<0.001	0.011	0.046	0.046	76
Al	1.197	<0.001	1.197	1.079	1.079	-11
Ba	0.007	<0.001	0.007	0.006	0.006	-20
Ca ^{KOH}	0.037	<0.001	0.037	0.074	0.074	50
Cr	0.019	<0.001	0.019	0.017	0.017	-15
Fe	2.429	<0.001	2.429	1.942	1.942	-25
Mn	0.053	<0.001	0.053	0.044	0.044	-20
Na	3.797	2.376	6.173	6.472	6.786	9
Ni	0.018	n/d	0.018	0.016	0.016	-13
P	0.100	0.007	0.107	0.101	0.102	-4
Pb	0.062	<0.001	0.062	0.052	0.052	-20
Si	0.664	<0.001	0.664	0.988	0.988	33
Ti	0.009	<0.001	0.009	0.013	0.013	29
U	0.013	0.032	0.045	0.057	0.062	29
Zr	0.013	0.005	0.018	0.017	0.017	-2
	ICP-AES Total (CS)	ICP-AES Total (DL)	ICP-AES Total (CS+DL)	ICP-AES Total (Orig. Mat'l)		
	8.43	2.42	10.85	10.93	11.25	
Cl	<0.001	<0.001	n/d	0.006	0.006	n/d
NO ₂	0.247	0.627	0.874	0.691	0.756	-16
NO ₃	<0.001	0.024	0.024	0.031	0.031	23
PO ₄	0.045	0.013	0.058	0.033	0.034	-70
SO ₄	0.087	0.169	0.256	0.202	0.219	-17
C ₂ O ₄	4.071	0.336	4.407	5.849	5.903	25
	IC Total (CS)	IC Total (DL)	IC Total (CS+DL)	IC Total (Orig. Mat'l)		
	4.45	1.17	5.62	6.81	6.95	

(1) For all CS, DL and OM analytical/physical results see Analytical Summary Report.

Averaged ICP results from KOH + Na2O2 fusions used for comparison purposes.

All result have been blank corrected for comparison; U on OM is from acid digestion.

(2) CS = Centrifuged Solids; DL = Decant Liquid; OM = Original Material

(3) CS wt% = $\mu\text{g/g result} * (\text{g}/\mu\text{g} * 100) [0.0001] * (\text{wt}\% \text{ CS wet}/100) [0.434] * (\text{wt}\% \text{ CS dry}/100) [0.517]$ (4) DL wt% = $\mu\text{g/ml result} * (\text{g}/\mu\text{g} * 100) [0.0001] * (1 - \text{wt}\% \text{ CS wet}) [0.566] / \text{DL density} [1.07]$ (5) OM wt% = $\mu\text{g/g result} * (\text{g}/\mu\text{g} * 100) [0.0001] * (\text{OM wt}\% \text{ solids on drying}/100) [0.292]$

(6) See Section 5.2 for Corr'd Wt% calculation

(7) n/d = measured analyte not detected above 0.0005%

5.2 Evaluation of Effect of Removal of Suspect Organic Layer

The three Original Material slurry subsamples (SPDL1-C106-003, -004, and -005) were processed for chemical analysis in the SAL. Each of these subsamples, when transferred to beakers for drying, developed a clear layer on top of the slurry material. This clear layer was suspected to be an organic layer, and comprised about 10% of the total volume of each sample. The details of the removal of the layer and subsequent organic and radiochemistry screening are reported by Urie (1997), and the results of the organic screening are reported by Campbell (1996). Since this layer was removed from the subsample prior to drying, the effect of the removal of 10% of the Original Material slurry supernatant on the reported Original Material results has been evaluated.

Based on an initial volume of the Original Material slurry samples (100 ml each), the density of the slurry (1.25 g/ml), the volume of layer removed (10% of the total volume), and the density of the layer removed (1.07 g/ml), the reduction in the quantity of supernatant is estimated to be about 12%. Based on the analytical results from the centrifuged liquid (See Table 5.1), the only analyte with any significant concentration in the supernatant is sodium (and possibly nitrite and oxalate).

Since 12% of the supernatant was removed from the Original Material samples prior to drying, the Original Material results are biased low for those analytes measured in the supernatant. This bias can estimated by reducing (by 12%) the concentration determined in the centrifuged liquid and calculating a new total weight percent slurry [e.g., $(\text{CS Na } 3.797) + (\text{DL Na } 2.376) - ((\text{DL Na } 2.376) * 0.12) = 5.89$], and then ratioing this total to the original total (e.g., $6.17/5.89 = 1.05$). Based on this rough estimate, the reported sodium value for the Original Material is low by 5%. Following the same logic, the reported nitrite result is about 10% low and the reported oxalate is about 1-2% low. Although an estimate, the "corrected" Original Material slurry results have been used for the direct comparison in Table 5.1. Only the chemical and radiochemical results are affected by the removal of the suspected organic layer, since the physical measurements (including weight percent solids) were performed on an independent subsample.

At the same time the organic screening was performed on the layer removed from the Original Material, total alpha activity and total beta activity were also performed. The total alpha activity for the supernatant layer was about 0.2 $\mu\text{Ci/g}$, and the total beta activity was 33 $\mu\text{Ci/g}$. Again, the removal of about 12% of the supernatant has little effect on the final reported alpha and beta activities for the Original Material (3.2 $\mu\text{Ci/g}$ and 1220 $\mu\text{Ci/g}$, respectively), since the majority of the activity appears to be in the solids fraction.

6.0 OBSERVATIONS, LIMITATIONS, and RECOMMENDATIONS

6.1 Data Acceptability

In general, the analytical, radiochemical, and physical measurement results are fundamentally sound and provide a solid basis for full characterization of the Privatization Contractor Samples. As such, the analytical results should be accepted as representing the nominal concentration of the Final Vendor Product.

6.2 Laboratory-Scale and Bench-Scale Operations

Observation: Due to programmatic schedule requirements, the bench-scale processing was initiated prior to completion of the laboratory-scale screening test. Every effort was made to evaluate the laboratory-scale results for each washing/leaching step prior to beginning these steps in the bench-scale processing. However, the failure to recognize that the laboratory-scale test washing/leaching steps were conducted at a weight percent solids lower than target (i.e., 3 wt% versus a target of 5 wt%) resulted in selecting bench-scale processing operating parameters that reduced the washing/leaching efficiency. Because of the decrease in washing efficiency for the bench-scale processing operation, more insoluble sodium oxalate than anticipated remained in the solids and contributed to the sodium concentration which exceeded the Envelope D specification.

Recommendation: Fully evaluate laboratory-scale screening test parameters for acceptability and assess the results against the material specification prior to initiating bench-scale processing activities.

Observation: There were only a selected set of analyses performed on the laboratory-scale screening test product. These analyses were primarily selected based on previous history of similar sludge washing tests, and did not include analytical screening for major components other than those measured by a simultaneous ICP-AES procedure and a specific IC procedure. For the C-106 material, a measurement of the oxalate concentration may have increase awareness of solubility issues and allowed adjustments to the laboratory-scale screening test and bench-scale processing operating parameters to mitigate the high sodium problem.

Recommendation: To better understand potential washing/leaching limitations, characterization of the initial feed material should include measurement of oxalate and complexing metal ions.

Observation: The bench-scale processing apparatus was not evaluated for the ability to provide representative subsamples for chemical/radiochemical analysis and physical measurements. Although the results from the Vendor Product and Final Vendor Product can be used to verify the apparatus provided representative samples for the C-106 material, other material types (e.g., materials with significantly different settling rates) may not behave the same in the apparatus.

Recommendation: It is important to establish a bench-scale apparatus test which can evaluate or demonstrate subsampling reproducibility prior to beginning new (and different) material processing activities.

6.3 Chemical and Physical Measurement Analysis

General Observations:

- The spike recoveries for arsenic, dysprosium, holmium, molybdenum, strontium, tungsten, and yttrium were high; therefore, the reported results may be biased high. Also, the spike recoveries for cerium, antimony, zinc, and niobium-93m were low, indicating a potential that the reported results may be biased low. All of these analytes are present at less than 30% of the Envelope D specification. Therefore, for the Final Vendor Product (C-106), the accuracy resulting from evaluation of these spike recoveries is not of concern.
- Beryllium, potassium, selenium, thorium and yttrium concentrations were below the detection limit for all analyses conducted.
- Total alpha activity and total beta activity measurement provide greater than 95% accountability for the major alpha and beta emitting radionuclides.
- Preparation blank concentrations exceeding 5% of the measured sample concentration were observed for nickel, molybdenum, platinum, and vanadium. The source of the molybdenum, platinum, and vanadium contamination is unknown at this time. The platinum concentration exceeded the Envelope D specification by 20%, and the contribution from the blank adds to the uncertainty of the reported result.
- Major differences between the ICP-MS and ICP-AES results were reported for calcium, zirconium, iron, aluminum, tellurium, and sodium. Inductively coupled plasma mass spectroscopy differences were reported between the two fusion preparations (KOH and Na_2O_2) for copper, molybdenum, platinum, rhenium, tin, and strontium. These differences can not be explained at this time. The unexplained difference in sodium concentration is of particular concern, as it is the metric for service payment.
- Comparison of analysis of the Original Material slurry with "centrifuged solids plus centrifuged supernatant" resulted in agreement of $\pm 30\%$, except for a few trace and minor analytes near the EQL: silver, barium, chromium, phosphorus, lead, uranium, nitrate, and phosphate.
- Hot cell sample handling (e.g., shear strength analysis, compositing, blending, etc.) may result in evaporative losses as high as 10%. Evaporation losses of 2% were observed for closed Final Vendor Product bottles following storage of the bottles for one month in the HLRF hot cells.

Observation/Limitation: Based on the results from the Original Material and Vendor Product, the ICP-MS experienced significant interferences when analyzing dissolutions from either fusion procedure. This was most dramatic on the Vendor Product for the very low concentration analytes, but also apparent in some major constituents. When the interference was severe, the data had to be flagged as "not used". When the ICP-MS results are within 20 times the DL, the results need to be considered for information only. Also, when very large differences exist between the KOH and Na_2O_2 fusion, the results are suspect.

Observation/Limitation: The ICP-MS results for the Original Material are considered more reliable than the results for the Vendor Product. The Vendor Product analyses were the more important (i.e., comparison to the Envelope D specification), and the ICP-MS analyses were performed first on the Vendor Product due to schedule milestones. The ICP-MS analysis and data handling protocols were established on the Vendor Product, and then used for analyzing the Original Material. A number of analysis problems were encountered in the "initial" (i.e., Vendor Product) ICP-MS analysis (Urie 1997, Appendix A) which were related to the C-106 matrix and the large number of analytes and radionuclides measured. Many of analytes and radionuclides measured in the Vendor Product had to be flagged as unusable or not used (i.e., n/u in data tables) because of these analysis problems. The analysis of the Original Material took considerably less time and the QC sample results were significantly better than for the Vendor Product.

Observation/Limitation: Silver, erbium, gadolinium, niobium, platinum, tin, and ytterbium reported results are at 40% to 120% of the Envelope D specification. Validation of these analytes may be subjected to higher uncertainty since their measurement had high relative standard deviations.

Observation/Limitation: The phosphorus analysis by IC measures only soluble phosphates. The total phosphorus concentration is obtained by ICP-AES. Like phosphorus, sulfur analysis by IC measures only soluble sulfates. The PNNL Analytical Chemistry Laboratory currently has no ICP-AES capability for measuring total sulfur.

Observation/Limitation: Although not discussed in this report, the shear strength analysis and heat capacity analysis did not meet analysis expectations. As reported by Urie (1997), the shear strength instrumentation/procedure used by PNNL was unable to measure the low shear strengths required, and the heat capacity was estimated but could not be measured.

Observation: The acid digestion procedure used for the C-106 analyses was a well-established nitric acid-hydrochloric acid dissolution procedure. When the results from this acid digestion are compared to the results from the two fusion procedures used, the acid digestion results are consistently lower. Typically, acid digestion methods provide solutions with less blank contamination, less analyte dilution (i.e., higher sensitivity), and fewer matrix interferences than the fusion methods. Unfortunately, the acid digestion procedure used apparently provides incomplete dissolution for the complex C-106 material.

Recommendation: To gain the benefits of acid digestion dissolution (i.e., less contamination, higher sensitivity, and fewer interferences), a more rugged acid digestion procedure needs to be developed which will provide better dissolution for complex waste material.

Observation: Spike recovery information was not available for ICP-MS measurements of praseodymium, thulium, iodine-129, neptunium-237, technetium-99, palladium-107, and plutonium and uranium isotopes. Also, there was no other appropriate standard information which could be used to assess the accuracy of the reported results.

Recommendation: In the absence of spiking information which is used to evaluate the accuracy of the measured result, other acceptable methods for evaluating accuracy should be implemented and reported.

Observation: The accuracy of the measured analytical results is obtained from evaluating the recovery of standards: LCSs, Blank Spikes, Matrix Spikes, or Post-Digestion Spikes. Each of these standards provides information to estimate the accuracy of the analytical measurement. However, there are no LCSs available which match the matrix of the C-106 material (or any other tank waste). The Blank Spike, Matrix Spike, and Post-Digestion Spike standards are merely solution standards added to the sample matrix (or blank) at different points in the preparation and may not reflect the dissolution characteristics of the sample matrix material.

Recommendation: To better evaluate the accuracy of the measured concentrations in tank sludge waste material, control standards more representative of tank waste material need to be "certified" and used as the LCS by laboratories performing analyses on tank sludge waste.

Observation: The average ICP-MS result for sodium was 12% higher than the ICP-AES result for sodium. Based on an evaluation of all ICP-AES and ICP-MS data, it is difficult to determine the "true" sodium concentration.

Recommendation: In order to ensure accurate sodium concentrations, improvements should be made in ICP-AES and/or ICP-MS sodium analysis or an alternate method identified.

Observation: From the results for the anion measurements performed on water leaches of the dried Original Material, Vendor Product, and Final Vendor Product, it is apparent that the reported results represent only the anions soluble at a 1:100 leaching ratio. There is data to suggest that the oxalate did not completely dissolve at this 1:100 leaching ratio.

Recommendation: To better establish the concentration of anions which may be in excess at these leaching ratios, multiple leaching ratios (e.g., 1:100, 1:200, 1:500) should be used to establish the "true" concentration of these soluble anions. Also, ICP-AES analyses may be performed on all water leached sample to assist in establishing the soluble compound.

Observation: The difference observed between the weight percent solids determinations made in the SAL and in the HLRF can be attributed to sample size differences (i.e., 1-3 g in HLRF and 30-100 g in SAL) and the ability to obtain representative samples when subsampling (particularly, for remote subsampling in hot cells). Although many times it is necessary to use small subsample to conserve material, these small subsamples can inject biases into the final result. This is particularly true when subsampling slurries containing reasonably high levels of solids (e.g., 10-20 wt%). The typical subsampling method used is to vigorously stir the slurry while extracting a subsample by pipet. If the solids are not uniformly distributed throughout the bulk sample by this technique, the location of the pipet tip potentially can alter the measured results. In such cases, the samples withdrawn from the bulk may be very reproducible, but not representative of the bulk material. Worse, different waste materials may behave differently using the same stirring/extracting procedure.

Recommendation: Subsampling and subaliquotting techniques should be evaluated for appropriateness prior to use, and whenever possible, large subsamples should be used for more accurate weight percent solids measurements.

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**TANK WASTE REMEDIATION SYSTEM (TWRS)
PRIVATIZATION CONTRACTOR**

**ENVELOPE D SAMPLES
(C-106)**

**ATTACHMENT 1
ANALYTICAL SUMMARY REPORT**

March 1997

Pacific Northwest National Laboratory

**TANK WASTE REMEDIATION SYSTEM (TWRS) PRIVATIZATION CONTRACTOR
SAMPLES**

**WASTE ENVELOPE D MATERIAL
241-C-106**

ANALYTICAL SUMMARY REPORT

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January 28, 1997

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**TANK WASTE REMEDIATION SYSTEM (TWRS) PRIVATIZATION CONTRACTOR
SAMPLES**

**WASTE ENVELOPE D MATERIAL
C-106**

EXECUTIVE SUMMARY

This report represents the Analytical Summary Report on TWRS Privatization Contractor Samples conducted in support of the Tank Waste Remediation System (TWRS) Privatization Contractor Samples, Waste Envelope D, C-106 characterization testing. All work performed was conducted in accordance with "Addendum 1 of the Letter of Instruction (LOI) for TWRS Privatization Contractor Samples Addressing Waste Envelope D Materials - Revision 0," (Jones, 1996).

Tank 241-C-106 (C-106) was selected by TWRS Privatization for the Part 1A Envelope D high-level waste demonstration. Twenty grab samples of C-106 were composited and characterized during three sequential stages of Envelope D sample preparation: 1) after dilution with water prior to bench-scale processing ("Original Material"); 2) after the bench-scale treatment per revision 0 of the LOI ("Vendor Product"); and 3) after decant/dilute treatment per revision 1 of the LOI ("Final Vendor Product"). This report includes only analytical results for the Vendor Product (Envelope D, Privatization Contractor Samples) and the Original Material.

The primary purpose of this report is to provide the chemical and radiochemical results for the Vendor Product for comparison to the Envelope D specification. A summary of the "most reasonable" averaged results compared to the Envelope D specification is presented in Table ES-1. The results are presented in units of g/L and Ci/L (at 31 gram waste oxide) for comparison with the envelope specification and in $\mu\text{g/g}$ and $\mu\text{Ci/g}$ dried solids for direct comparison to the analytical results.

The Vendor Product resulting from the treatment defined in the LOI Revision 0 exceeds the Envelope D specifications for Na and $^{121\text{m}}\text{Sn}$. Additionally, a trace component (Pt) and a trace radionuclide (^{235}U) were found to exceed the envelope specification, and three radionuclides ($^{110\text{m}}\text{Ag}$, ^{242}Pu , and $^{119\text{m}}\text{Sn}$) could not be verified due to analytical detection limitations. Also, the results for $^{243}\text{Cm}/^{244}\text{Cm}$ and $^{144}\text{Ce}/^{144}\text{Pr}/^{144\text{m}}\text{Pr}$ exceeded the specification; but only if the results are compared to the Envelope D specifications for ^{243}Cm (as a trace radionuclide) and for $^{144\text{m}}\text{Pr}$, respectively. Based on the total activity measured, the specifications for ^{244}Cm , ^{144}Ce , and ^{144}Pr were not exceeded.

Viscosity and shear strength did not meet the LOI Revision 0 specifications. The viscosity was lower than the specification, and the shear strength measured detection limit was higher than the specification

limit. Compliance with these physical properties specifications are not required for shipping the Vendor Product to the vendors.

Inductively coupled plasma-atomic emission spectroscopy (ICP-AES) and inductively coupled plasma-mass spectroscopy (ICP-MS) were performed on multiple sample preparations for many of the analytes of interest. Generally, the highest reliable averaged result (considering the average, the standard deviation, and preparation blank performance) was reported. Table ES-1 indicates which analytical method and preparative technique were selected for comparison. When results fell below the estimated quantitation limit (EQL) or the instrument detection limit (IDL), the lowest reported "less than" value was used. Results preceded by a less than (" $<$ ") indicate that the analyte was not detected. In general, when one average was above the EQL and other averages were below the EQL, the result above the EQL was reported.

Additional processing was performed per "Addendum 1 of the Letter of Instruction (LOI) for TWRS Privatization Contractor Samples Addressing Waste Envelope D Materials - Revision 1," (Wiemers 1996). This additional processing and the adjustments made to establish the concentration of the Final Vendor Product are detailed in Addendum 1 to this report (Kuhl-Klinger 1997).

A summary of Original Material "most reasonable" average results is presented in Table ES-2. The average results are compared to the Envelope D specification for direct comparison to the Vendor Product. The results are presented in units of g/L and Ci/L (at 31 gram waste oxide) for comparison with the envelope specification and in $\mu\text{g/g}$ and $\mu\text{Ci/g}$ for direct comparison to the analytical results.

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Table ES-1. Comparison of Vendor Product Results with Envelope D Specification

Analyte	Envelope D Spec	Envelope D Spec	n	Vendor Product					Analytical Method	Notes
				Average (ppm)	Average (ppm) @ 500 Dis (1)	Std. Dev.	Avg. Spec	>Spec Flag		
Ag	1.7E-01	4.35E+03	6	3.18E+03	1.23E-01	2.9E-02	7.29E-01	N	ICPZIF	See Radionuclide High Uncertainty
Al	4.3E+00	1.11E+05	6	5.12E+04	1.99E+00	9.0E-02	4.62E-01	N	ICPNIIF	
Am	2E-02	---	---	---	---	---	---	---	---	
As	5E-02	1.29E+03	6	2.89E+02	1.11E-02	1.7E-03	2.22E-01	N	ICPMS ZIF	
B	4E-01	1.03E+04	6	2.29E+02	8.69E+03	3.2E-03	2.22E-02	N	ICPMS NIF	
Ba	1.4E+00	3.61E+04	6	4.37E+02	1.70E-02	8.6E-04	1.21E-02	N	ICPZIF	See Radionuclide High Uncertainty
Be	2E-02	5.15E+03	6	4.30E+02	3.89E+05	N/A	+1.94E+03	N	ICP Acid	
Bi	8.6E-01	2.22E+04	6	4.30E+02	1.67E-02	4.4E-04	1.94E-02	N	ICP Acid	
Ca	2.2E+00	5.67E+04	6	3.84E+03	1.49E-01	3.6E-03	6.78E-02	N	ICP Acid	
Cd	1.4E+00	3.61E+04	6	9.06E+01	3.52E+03	4.4E-04	2.51E-03	N	ICPMS NIF	
Ce	2.5E-01	6.44E+03	6	2.02E+02	7.64E+03	8.9E-04	3.14E-02	N	ICPMS ZIF	TRACE Component
Co	1.4E-01	3.61E+03	6	2.60E+01	1.01E-03	2.4E-04	7.21E-03	N	ICPMS NIF	
Cr	2.1E-01	5.41E+03	6	7.74E+02	3.00E-02	3.2E-03	1.43E-01	N	ICPMS NIF	
Cs	1.8E-01	4.64E+03	6	1.80E+01	6.69E-04	1.6E-04	3.86E-03	N	ICPMS NIF	
Cu	1.5E-01	3.65E+03	6	1.56E+02	6.07E+03	5.1E-04	4.04E-02	N	ICPMS ZIF	
Dy	8E-03	2.06E+02	6	2.02E+01	7.84E-04	2.9E-04	9.80E-02	N	ICPMS NIF	TRACE Component
Er	1E-03	2.58E+01	6	1.31E+01	5.09E-04	1.8E-04	5.06E-01	N	ICPMS NIF	
Eu	8E-03	1.29E+02	6	7.88E+00	3.05E-04	2.3E-04	6.10E-02	N	ICPMS NIF	
Fe	8.9E+00	2.29E+05	6	1.41E+05	5.48E+00	2.9E-01	6.14E-01	N	ICP NIF	
Gd	3E-03	7.73E+01	6	2.50E+01	9.70E-04	2.7E-04	3.23E-01	N	ICPMS NIF	
Hg	1E-03	2.58E+01	6	6.07E+00	2.36E-04	5.8E-05	2.36E-01	N	ICPMS NIF	TRACE Component
K	4.1E-01	1.06E+04	6	4.82E+02	+1.91E-02	N/A	+4.66E-02	N	ICP Acid	
La	8E-01	2.06E+04	6	9.21E+01	3.58E+03	5.6E-04	4.47E-03	N	ICPMS ZIF	
Li	4.3E-02	1.11E+03	6	6.60E+01	1.79E+03	1.2E-03	0.042	N	ICP Acid	
Mg	6.5E-01	1.67E+04	6	1.26E+03	4.89E+02	3.1E-03	7.52E-02	N	ICPMS ZIF	
Mn	2.0E+00	5.15E+04	6	2.81E+03	1.09E-01	8.0E-03	5.45E-02	N	ICPMS NIF	Zr Contribution/High Bias
Mo	2.0E-01	5.15E+03	6	5.70E+01	2.21E+03	5.3E-04	1.11E-02	N	ICPMS NIF	
Na	6.0E+00	1.55E+05	6	1.62E+05	6.29E+00	3.1E-01	1.05E+00	Y	ICP Acid	
Nb	3.0E-03	7.73E+01	6	3.38E+01	1.31E+03	1.0E-03	4.37E-01	N	ICPMS NIF	
Nd	5.3E-01	1.37E+04	6	2.82E+02	1.08E+02	3.1E-04	2.07E-02	N	ICP Acid	
Ni	7.3E-01	1.68E+04	6	1.61E+03	6.25E-02	2.4E-03	6.56E-02	N	ICPMS ZIF	See Radionuclide
Np	3E-02	---	---	---	---	---	---	---	---	
P	5.4E-01	1.39E+04	6	3.33E+03	1.29E-01	1.4E-02	2.29E-01	N	ICP NIF	
Pb	3.4E-01	8.76E+03	6	3.43E+03	1.33E-01	3.0E-03	3.92E-01	N	ICP Acid	
Pd	4E-02	1.03E+03	6	2.74E+01	1.05E+03	2.0E-04	2.66E-02	N	ICPMS ZIF	
Pm	3E-02	---	---	---	---	---	---	---	---	See Radionuclide
Pr	1.1E-01	2.63E+03	6	8.41E+01	3.26E+03	2.1E-04	2.97E-02	N	ICPMS ZIF	
Pt	1E-03	2.58E+01	6	3.07E+01	1.19E+03	3.0E-04	1.19E+00	Y	ICPMS NIF	
Pu	1.8E-02	---	---	---	---	---	---	---	---	
Rb	5E-02	1.55E+03	6	7.68E+00	2.86E-04	1.1E-04	4.97E-03	N	ICPMS ZIF	
Re	3E-02	7.73E+02	6	6.61E+00	2.57E-04	8.0E-05	6.55E-03	N	ICPMS ZIF	See Radionuclide High Uncertainty
Rh	4E-02	1.03E+03	6	1.44E+02	5.59E+03	6.2E-04	1.40E-01	N	ICPMS ZIF	
Ru	1.1E-01	2.83E+03	3	6.57E+01	3.71E+03	2.5E-04	3.38E-02	N	ICPMS NIF	
S	2E-01	---	---	---	---	---	---	---	---	
Sb	2.6E-01	6.70E+03	3	7E-01	2.83E+05	1.1E+05	1.09E-04	N	ICPMS Acid	
Se	1.6E-01	4.12E+03	6	3.69E+02	+1.43E-02	N/A	+8.95E-02	N	ICP Acid	See Radionuclide
Si	5.8E+00	1.49E+05	6	8.89E+04	2.67E+00	1.7E-01	4.81E-01	N	ICP NIF	
Sm	5.3E-02	---	---	---	---	---	---	---	---	
Sn	1.1E-02	2.83E+02	6	1.02E+02	3.95E+03	1.1E+03	3.80E-01	N	ICPMS ZIF	
Sr	1.6E-01	4.12E+03	6	1.50E+02	5.62E+03	5.2E-04	3.64E-02	N	ICPMS ZIF	
Ta	8E-03	2.06E+02	6	2.88E+01	1.11E+03	4.0E-04	1.39E-01	N	ICPMS ZIF	TRACE Component
Tb	1E-03	2.58E+01	6	8.20E+00	3.18E-04	6.0E-05	3.18E-01	N	ICPMS NIF	
Tc	6E-02	---	---	---	---	---	---	---	---	
Te	4E-02	1.03E+03	6	4.24E+02	1.65E+02	3.3E+03	4.11E-01	N	ICPMS NIF	
Th	1.6E-02	4.12E+02	6	3.48E+02	1.35E+02	1.9E-04	8.47E-01	N	ICP Acid	
Ti	4E-01	1.03E+04	6	1.31E+03	5.10E+02	5.7E+03	1.27E-01	N	ICPMS ZIF	TRACE Component
Tl	1.4E-01	3.61E+03	6	+1.23E+02	+4.77E-03	N/A	+3.41E-02	N	ICP Acid	
Tm	1E-03	2.58E+01	6	2.64E+00	1.02E-04	5.3E-05	1.02E-01	N	ICPMS NIF	
U	4.2E+00	1.08E+05	6	1.90E+03	7.36E+02	5.1E+03	1.75E-02	N	ICPMS NIF	
V	1E-02	2.58E+02	6	3.72E+03	1.44E+03	2.6E-04	1.44E-01	N	ICPMS NIF	
W	7.4E-02	1.91E+03	6	4.95E+02	1.93E+02	2.9E+03	2.61E-01	N	ICPMS NIF	TRACE Component
Y	5E-02	1.29E+03	6	+8.60E+01	+3.34E-03	N/A	+8.98E-02	N	ICP Acid	
Yb	1E-03	2.58E+01	6	1.26E+01	4.89E-04	2.4E-04	4.89E-01	N	ICPMS NIF	
Zn	1.3E-01	3.35E+03	6	3.06E+02	1.19E+02	2.7E+03	9.14E-02	N	ICPMS NIF	
Zr	4.0E+00	1.19E+05	6	1.11E+03	4.29E+02	2.0E+02	9.33E-03	N	ICP NIF	
Br	None	---	---	---	---	---	---	---	---	Spec includes both
Cl	1.1E+00	2.83E+04	6	1.76E+02	4.50E-03	N/A	+1.03E-01	N	IC water leach	
Co	1E-01	2.58E+03	6	1.26E+02	4.89E+03	1.0E+03	4.89E-02	N	IC water leach	
CN Total	5E-01	1.29E+04	6	9.50E+00	3.69E+04	3.3E+05	7.38E-04	N	Direct	
Hg	3E-02	7.73E-02	6	3.43E-02	1.33E-02	6.3E-04	4.44E-01	N	Direct	
NO2	NO2 + NO3	N/A	6	2.07E+03	8.02E-02	1.5E-02	N/A	N/A	IC water leach	Spec includes both
NO3	NO2 + NO3	N/A	6	8.86E+02	3.44E-02	1.7E-02	N/A	N/A	IC water leach	
NO2/NO3	1.12E+1	2.89E+05	6	2.89E+05	1.15E+01	3.2E-02	1.02E-02	N	IC water leach	
PO4	None	N/A	6	1.80E+02	6.21E+03	1.4E-03	N/A	N/A	IC water leach	
SO4	None	N/A	6	4.70E+02	1.82E-02	3.1E-03	N/A	N/A	IC water leach	

Table ES-1. Comparison of Vendor Product Results with Envelope D Specification

Analyte	Envelope D Spec g/L	Envelope D Spec µg/g	n	Vendor Product						Analytical Method Prep	Notes
				Average µg/L	Average - g/L @ 31g Ox (1)	g/L	Avg Spec	Flag	>Spec		
CO24	None	N/A	6	1.70E-05	6.59E-00	5.1E-01	N/A	N/A	N	IC water leach	N * (17/14) - High Blank
NH3	5E-01	1.29E-04	6	2.13E-01	6.25E-04	3.5E-04	1.65E-03	N	ISE	Direct	
TiC (CO3)	1.68E+0	4.79E-04	6	5.30E-03	2.06E-01	2.2E-02	1.11E-01	N	Direct	Direct	
TOC	3.4E+00	8.76E-04	6	4.79E-04	1.88E+00	1.3E-01	5.47E-01	N	Direct	Direct	
TDC	None	N/A	6	5.22E-04	2.03E+00	1.2E-01	N/A	N/A	N/A	Direct	Sum of TOC & TiC
Radionuclides:											
Ag-110m	1.03E-03	2.54E-04	6	2.87E-03	<3.49E-06	N/A	<3.49E-02	-Y-	N	RNIF	
Am-241	4.30E-02	1.11E+03	6	3.15E+00	1.22E-04	2.8E-05	2.84E-03	N	N	RNIF	
Am-243	5.00E-06	1.29E-01	6	<2E-02	<7.76E-07	N/A	<1.55E-01	N	N	RNIF	
C-14	2.00E-06	5.15E-02	6	2.87E-03	1.11E-07	2.1E-07	5.57E-02	N	Direct	Direct	Poor Analysis Precision
Ce-144(Pr-144+144m)	1.00E-04	2.58E+00	6	<2E+00	<7.76E-05	N/A	<7.76E-01	N	RNIF	RNIF	Ce-144 Specification
Ce-144(Pr-144+144m)	1.00E-04	2.58E+00	6	<2E+00	<7.76E-05	N/A	<7.76E-01	N	RNIF	RNIF	Pr-144m Specification
Ce-144(Pr-144+144m)	1.00E-07	2.58E-03	6	<2E+00	<7.76E-05	N/A	<7.76E-02	-Y-	N	RNIF	Pr-144m Specification
Cm-242(Am-242+242m)	3.70E-05	9.53E-01	6	8.07E-03	3.13E-07	6.5E-08	8.47E-03	N	RNIF	RNIF	Am-242 Specification
Cm-242(Am-242+242m)	3.10E-05	7.99E-01	6	8.07E-03	3.13E-07	6.5E-08	1.01E-02	N	RNIF	RNIF	Am-242m Specification
Cm-242(Am-242+242m)	3.20E-05	8.24E-01	6	8.07E-03	3.13E-07	6.5E-08	9.79E-03	N	RNIF	RNIF	Am-242m Specification
Cm-243+244	1.00E-09	2.58E-05	6	9.89E-02	3.83E-06	8.5E-07	3.83E-03	-Y-	N	RNIF	Cm-243 Specification
Cm-243+244	9.30E-04	2.40E-01	6	9.89E-02	3.83E-06	8.5E-07	4.12E-03	N	RNIF	RNIF	Cm-244 Specification
Co-60	3.00E-03	7.73E+01	6	4.91E-01	1.91E-05	1.5E-06	6.35E-03	N	RNIF	RNIF	
Cs-134	6.80E-03	1.75E-02	6	<3E-01	<1.16E-05	N/A	<1.71E-03	N	RNIF	RNIF	
Cs-135	3.00E-05	7.73E-01	6	2.86E-03	1.11E-07	5.8E-09	3.70E-03	N	RNIF	RNIF	
Cs-137(Ba-137)	3.00E+00	7.73E-04	6	4.86E-02	1.93E-02	1.0E-03	6.42E-03	N	RNIF	RNIF	Cs-137 Specification
Cs-137(Ba-137)	3.00E+00	7.73E-04	6	4.86E-02	1.93E-02	1.0E-03	6.42E-03	N	RNIF	RNIF	Ba-137 Specification
Eu-152	1.50E-04	3.86E+00	6	<2E+00	<7.76E-05	N/A	<5.18E-01	N	RNIF	RNIF	
Eu-154	1.60E-02	4.12E+02	6	3.79E+00	1.47E-04	5.2E-06	9.19E-03	N	RNIF	RNIF	
Eu-155	9.00E-03	2.32E-02	6	3.13E+00	1.21E-04	1.2E-05	1.35E-02	N	RNIF	RNIF	
Fe-55	1.00E-03	2.58E-01	6	1.53E-01	5.84E-06	3.1E-07	5.94E-03	N	RNIF	RNIF	
I-129	8.00E-08	2.32E-03	6	2.20E-04	6.84E-09	2.3E-09	9.49E-02	N	RNIF	ICPMS Acid	Potential Prep Losses
Ir-93m	4.20E-05	2.24E+00	6	2.34E-01	9.08E-06	9.1E-06	1.04E-01	N	RNIF	RNIF	
Ni-59	1.40E-05	3.61E-01	6	2.05E-02	7.99E-07	8.0E-07	5.68E-02	N	RNIF	RNIF	
Ni-63	1.60E-03	4.12E+01	6	2.40E+00	9.32E-05	9.3E-05	5.82E-02	N	RNIF	RNIF	
Np-237	2.30E-05	5.93E-01	6	3.93E-03	1.53E-07	3.0E-08	6.63E-03	N	RNIF	ICPMS ZrF	
Pd-107	4.00E-06	1.03E-01	6	1.10E-03	4.27E-08	5.0E-08	1.07E-02	N	RNIF	ICPMS Acid	
Pu-238	2.10E-04	2.83E-00	6	2.45E-05	2.45E-05	1.1E-06	2.23E-01	N	RNIF	RNIF	
Pu-239	7.50E-04	2.43E-01	6	1.53E-03	5.94E-05	4.1E-06	6.53E-02	N	RNIF	ICPMS ZrF	
Pu-240	2.60E-04	6.70E+00	6	1.27E-01	4.93E-06	3.6E-07	1.90E-02	N	RNIF	ICPMS ZrF	
Pu-241	6.90E-03	1.78E+02	6	6.69E+00	2.70E-04	1.6E-05	3.92E-02	N	RNIF	RNIF	
Pu-242	7.10E-08	1.83E-03	6	<2E-02	<7.76E-07	N/A	<1.00E+01	-Y-	N	RNIF	
Ru-106(Rh-106)	2.00E-04	5.15E+00	6	<3E+00	<1.16E-04	N/A	<5.82E-01	N	RNIF	RNIF	Ru-106 Specification
Ru-106(Rh-106)	2.00E-04	5.15E+00	6	<3E+00	<1.16E-04	N/A	<5.82E-01	N	RNIF	RNIF	Rh-106 Specification
Sb-125(Te-125m)	1.00E-02	2.58E-02	6	<2E+00	<7.76E-05	N/A	<7.76E-03	N	RNIF	RNIF	Sb-125 Specification
Sb-125(Te-125m)	3.00E-03	7.73E-01	6	<2E+00	<7.76E-05	N/A	<2.59E-02	N	RNIF	RNIF	Te-125m Specification
Se-78	4.20E-07	1.08E-02	6	<2E-04	<8.15E-09	N/A	<1.94E-02	N	RNIF	RNIF	
Sm-151(Pm-147)	1.60E-02	2.40E-03	6	9.23E-01	3.55E-03	1.9E-04	3.85E-02	N	RNIF	RNIF	Sm-151 Specification
Sm-151(Pm-147)	1.60E-01	4.12E+03	6	9.23E+01	3.55E-03	1.9E-04	2.24E-02	N	RNIF	RNIF	Pm-147 Specification
Sn-113(In-113)	1.88E-08	4.84E-02	6	<1E-02	<3.88E-07	N/A	<2.06E-01	N	RNIF	RNIF	Sn-113 Specification
Sn-113(In-113)	1.88E-08	4.84E-02	6	<1E-02	<3.88E-07	N/A	<2.06E-01	N	RNIF	RNIF	In-113 Specification
Sn-119m	1.00E-08	2.58E-04	6	<4E-02	<1.55E-06	N/A	<1.55E-02	-Y-	N	RNIF	
Sn-121m	9.00E-08	2.32E-01	6	1.23E+00	4.77E-05	2.1E-05	5.31E-00	-Y-	N	RNIF	
Sn-126(Sb-126m, Sb-126)	4.80E-05	1.24E+00	6	1.15E-02	4.46E-07	1.2E-07	9.30E-03	N	RNIF	RNIF	Sn-126 Specification
Sn-126(Sb-126m, Sb-126)	3.43E-05	8.84E-01	6	1.15E-02	4.46E-07	1.2E-07	1.30E-02	N	RNIF	RNIF	Sb-126m Specification
Sn-126(Sb-126m, Sb-126)	4.83E-05	1.24E+01	6	1.15E-02	4.46E-07	1.2E-07	9.24E-02	N	RNIF	RNIF	Sb-126 Specification
Sn-90(Y-90)	3.10E+00	7.99E-04	6	7.30E-02	2.83E-02	1.7E-03	9.14E-03	N	RNIF	RNIF	Sn-90 Specification
Sn-90(Y-90)	3.10E+00	7.99E-04	6	7.30E-02	2.83E-02	1.7E-03	9.14E-03	N	RNIF	RNIF	Y-90 Specification
Ti-99	4.50E-03	1.16E-02	6	1.37E-01	2.53E-07	3.0E-08	1.14E-04	N	RNIF	ICPMS NIF	
Th-232	See "Th"	See "Th"	6	5.48E-05	2.13E-09	7.0E-10	N/A	N/A	ICPMS ZrF	ICPMS ZrF	
U-233	1.00E-09	2.58E-05	6	1.7E-03	6.6E-08	1.3E-08	6.60E-01	-Y-	N	ICPMS Acid	TRACE Comp - Th Interfers
U-234	7.70E-07	1.98E-02	6	7.70E-04	2.89E-08	4.9E-09	3.88E-02	N	ICPMS Acid	ICPMS Acid	
U-235	3.20E-08	8.24E-04	6	2.80E-05	1.09E-09	9.7E-12	3.40E-02	N	ICPMS Acid	ICPMS Acid	
U-236	8.20E-08	2.11E-03	6	1.80E-05	6.69E-10	7.6E-11	8.52E-03	N	ICPMS Acid	ICPMS Acid	
U-238	5.60E-07	1.46E-02	6	6.37E-04	2.47E-08	1.7E-09	4.20E-02	N	ICPMS ZrF	ICPMS ZrF	
Zr-93	1.40E-04	3.61E+00	6	<6E-01	<2.32E-05	N/A	<1.66E-01	N	RNIF	RNIF	
Total Alpha	none	N/A	6	6.78E-00	2.63E-04	8.7E-06	N/A	N/A	RNIF	RNIF	
Total Beta	none	N/A	6	2.05E-03	7.99E-02	4.6E-03	N/A	N/A	RNIF	RNIF	
Physical Properties											
				Vendor Product							
				HLRF		SAL					
Wt% Solids @105C - (%)	2.5 - 13			14.4	14.8						
Wt% Oxide @1050C - (%)				11.5							
Total Oxide - (g/L)	25 - 100			126.5							
Slurry Density - (g/ml)	1.02 - 1.10			1.10							
Settled Solids - (Vol%)	7 - 95			18.50							
pH	>10										

HLRF=High Level Radiation Facility; SAL=Shielded Analytical Laboratory; TBD=to be determined; "N/A"=not measured; N/A=not applicable

(1) g/L = (µg analyte/g dry solids)/(wt% solids/wt% oxides)/(31g oxide/L)/(1E-06µg). µg/L = (µCi analyte/g dry solids)/(wt% solids/wt% oxides)/(31g oxide/L)/(1E-06µg/L)

(2) HLRF wt% solids performed in duplicate on 3 g sample aliquots. SAL wt% solids performed on single >100 g bulk samples

Table ES-2. Original Material Result Summary

Analyte	Envelope D Spec g/L	Envelope D Spec g/L	n	Average g/L	Original Material				Analytical Method Ref	Notes
					Avg. g/L ± 31g K (1)	Std. Dev. g/L	Avg. Spec g/L	± Spec g/L		
Ag	1.7E-01	4.11E-03	6	2.00E-01	6.64E-02	8.3E-01	6.08E-01	N	ICP ZrF	See Radionuclide High Uncertainty
Al	4.3E+00	1.04E-05	6	3.73E-04	1.54E+00	1.9E+03	3.59E-01	N	ICP NIF	
Am	2E-02	---	---	---	---	---	---	N	---	
As	5E-02	1.21E-03	6	3.49E-01	1.44E-03	1.3E+01	2.88E-02	N	ICPMS ZrF	
B	4E-01	9.88E-03	6	1.08E+02	4.48E-03	2.8E+01	1.12E-02	N	ICPMS ZrF	High Uncertainty - Below EGL
Ba	1.4E+00	3.39E-04	6	2.17E+02	8.97E-03	8.0E+00	6.41E-03	N	ICP ZrF	
Be	2E-02	4.84E-02	6	<2.00E-01	<8.27E-04	N/A	<4.10E-04	N	ICP ZrF	
Bi	8.6E-01	1.08E-04	6	1.62E+02	7.70E-03	2.0E+01	7.79E-03	N	ICP Acid	
Cs	2.2E+00	5.32E-04	6	1.70E+03	7.03E-02	1.2E+02	3.19E-02	N	ICP Acid	High Uncertainty - Below EGL
Cd	1.4E+00	3.39E-04	6	2.81E+01	1.20E-03	3.0E+00	6.59E-04	N	ICPMS Acid	
Ce	2.5E-01	6.05E-03	6	9.49E-01	3.91E-03	6.1E+00	1.56E-02	N	ICPMS Acid	
Co	1.4E-01	3.38E-03	6	4.10E-01	1.69E-03	3.0E+00	1.21E-02	N	ICPMS NIF	
Cr	2.1E-01	5.08E-03	6	6.37E-02	2.63E-02	2.9E+01	1.25E-01	N	ICPMS NIF	TRACE Component
Cs	1.8E-01	4.35E-03	6	8.10E+00	3.35E-04	8.0E-01	1.86E-03	N	ICPMS Acid	
Cy	1.5E-01	3.83E-03	6	7.75E-01	3.21E-03	4.1E+00	2.14E-02	N	ICPMS ZrF	
Er	8E-03	1.94E-02	6	<5.00E+00	<2.07E-04	N/A	<2.54E-02	N	ICPMS Acid	
Eu	1E-03	2.42E-01	6	<5.00E+00	<2.07E-04	N/A	<2.07E-01	N	ICPMS Acid	TRACE Component
Fe	5E-03	1.21E+02	6	<5.00E+00	<2.07E-04	N/A	<4.13E-02	N	ICPMS Acid	
Fe	8.9E+00	2.15E+05	6	6.78E-04	2.80E+00	2.7E+03	3.15E-01	N	ICP NIF	
Gd	3E-03	7.26E-01	6	1.15E-01	5.41E-04	3.6E+00	1.80E-01	N	ICPMS ZrF	
Hg	1E-03	2.42E-01	6	<5.00E+00	<2.07E-04	N/A	<2.07E-01	N	ICPMS Acid	TRACE Component High Uncertainty - Below EGL
K	4.1E-01	9.82E-03	6	8.11E+02	3.35E-02	N/A	8.18E-02	N	ICP Acid	
La	8E-01	1.94E-04	6	5.12E-01	2.19E-03	1.5E+00	2.55E-03	N	ICPMS Acid	
Li	4.3E-02	1.04E-03	6	8.00E-00	3.31E-04	1.0E+00	0.00E	N	ICP Acid	
Mg	6.5E-01	1.57E-04	6	3.31E+02	1.37E-02	1.1E+01	2.10E-02	N	ICP Acid	High Uncertainty - Below EGL
Mn	2.0E+00	4.84E-04	6	1.27E+03	5.25E-02	5.5E+01	2.62E-02	N	ICP ZrF	
Mo	2.0E-01	4.84E-03	6	8.50E-01	2.89E-03	1.3E+01	1.34E-02	N	ICPMS NIF	
Na	6.0E+00	1.45E-05	6	2.25E+05	9.35E+00	6.3E+03	1.55E+00	-Y-	ICP Acid	
Nb	3.0E-03	7.29E-01	6	2.27E-01	1.33E-03	1.3E+01	4.44E-01	N	ICPMS Acid	Zr Contribution/High Bias
Nd	5.3E-01	1.29E-04	6	1.24E+02	5.13E-03	1.7E+01	6.67E-03	N	ICPMS Acid	
Ni	7.3E-01	1.77E-04	6	5.15E+02	2.13E-02	2.4E+01	2.92E-02	N	ICP Acid	
Np	3E-02	---	---	---	---	---	---	N	---	
P	5.4E-01	1.31E-04	6	3.71E+03	1.53E-01	1.3E+02	2.84E-01	N	ICP NIF	See Radionuclide
Pb	3.4E-01	8.23E-03	6	6.18E+03	7.48E-02	8.2E+01	2.20E-01	N	ICP Acid	
Pd	4E-02	9.68E-02	6	3.37E-01	1.29E-03	5.2E+00	3.48E-02	N	ICPMS ZrF	
Pm	3E-02	---	---	---	---	---	---	N	---	
Pr	1.1E-01	2.66E-03	6	3.66E-01	1.51E-03	3.5E+00	1.38E-02	N	ICPMS ZrF	See Radionuclide TRACE Component
Re	1E-03	2.42E+01	6	<5.00E+00	<2.07E-04	N/A	<2.07E-01	N	ICPMS ZrF	
Pu	1.6E-02	---	---	---	---	---	---	N	---	
Rb	6E-02	1.45E-03	6	4.60E+00	1.93E-04	1.4E+00	3.17E-03	N	ICPMS ZrF	
Re	3E-02	7.28E-02	6	---	---	---	---	N	---	Not Analyzed
Rh	4E-02	9.68E-02	6	1.59E-01	6.57E-04	1.2E+00	1.64E-02	N	ICPMS Acid	
Ru	1.1E-01	2.66E-03	6	1.05E-02	4.34E-03	6.0E+00	3.95E-02	N	ICPMS NIF	
S	2E-01	---	---	---	---	---	---	N	---	
Sb	2.6E-01	5.29E-03	6	6.7E+00	2.77E-04	4.0E-01	1.07E-03	N	ICPMS ZrF	No Analysis Performed
Se	1.6E-01	3.87E-03	6	2.10E+02	8.66E-03	6.0E+00	5.43E-02	N	ICP Acid	
Si	5.8E+00	1.40E+05	6	3.33E-04	1.38E+00	1.2E+03	2.37E-01	N	ICP ZrF	
Sm	5.3E-02	---	---	---	---	---	---	N	---	
Sn	1.1E-02	2.66E+02	6	2.60E-01	1.07E-03	2.3E+00	9.77E-02	N	ICPMS Acid	See Radionuclide
Str	1.6E-01	3.87E-03	6	1.24E-01	5.13E-04	1.1E+00	3.20E-03	N	ICPMS Acid	
Ta	8E-03	1.64E-02	6	1.27E-01	5.25E-04	3.0E+00	6.58E-02	N	ICPMS Acid	
Tb	1E-03	2.42E+01	6	<5.00E+00	<2.07E-04	N/A	<2.07E-01	N	ICPMS Acid	
Tc	8E-02	---	---	---	---	---	---	N	---	TRACE Component See Radionuclide
Te	4E-02	9.68E-02	6	2.64E-01	1.09E-03	6.1E+00	2.73E-02	N	ICPMS Acid	
Th	1.6E-02	3.87E+02	6	<4.69E+01	<1.64E-03	N/A	<1.21E-01	N	ICP Acid	
Ti	4E-01	9.68E-03	6	5.64E+02	2.33E-02	5.3E-01	5.83E-02	N	ICPMS NIF	
Tl	1.4E-01	3.39E-03	6	<2.35E-01	<9.71E-04	N/A	<6.46E-03	N	ICP Acid	TRACE Component
Tm	1E-03	2.42E+01	6	<5.00E+00	<2.07E-04	N/A	<2.07E-01	N	ICPMS Acid	
V	1.4E+00	1.02E-05	6	1.13E-03	4.89E-02	1.4E+02	3.3E-02	N	ICPMS Acid	
W	1E-02	2.42E-02	6	9.90E-01	4.09E-03	3.0E+00	4.09E-01	N	ICPMS Acid	
X	7.4E-02	1.79E-03	6	3.89E+02	1.65E-02	1.1E+01	2.23E-01	N	ICPMS Acid	High Blank
Y	5E-02	1.21E-03	6	3.90E-01	1.61E-03	2.0E+00	3.22E-02	N	ICP Acid	
Yb	1E-03	2.42E+01	6	<5.00E+00	<2.07E-04	N/A	<2.07E-01	N	ICPMS Acid	
Zn	1.3E-01	3.15E-03	6	1.67E+02	7.73E-03	1.9E-01	5.95E-02	N	ICPMS NIF	
Zr	4.6E+00	1.15E-05	6	3.95E-02	1.51E-02	7.9E-01	3.29E-03	N	ICP Acid	High Blank
Br	None	N/A	6	<1.31E-02	<5.41E-03	N/A	N/A	N	IC water leach	
Cl	1.1E+00	2.86E-04	6	<3.27E-03	<1.35E-01	N/A	<1.23E-01	N	IC water leach	
F	1E-01	2.42E-03	6	2.26E-02	9.34E-03	1.7E+01	9.34E-02	N	IC water leach	
CN Total	5E-01	1.21E-04	6	6.23E-01	2.58E-03	5.3E+00	5.15E-03	N	Direct	Spec includes both
Hg	3E-02	7.26E-02	6	1.60E+02	6.81E-03	1.1E+01	2.20E-01	N	Direct	
NO2	NO2 + NO3	N/A	6	2.37E-04	9.80E-01	2.6E-03	N/A	N/A	IC water leach	
NO3	NO2 + NO3	N/A	6	1.17E-03	4.84E-02	8.8E-01	N/A	N/A	IC water leach	
NO2/NO3	1.17E+1	2.71E-05	6	2.49E-04	1.03E+00	3.3E-02	9.15E-02	N	IC water leach	Spec includes both
PC4	None	N/A	6	1.13E-03	4.67E-02	9.9E-01	N/A	N/A	IC water leach	
SO4	None	N/A	6	6.99E-03	2.89E-01	1.3E-03	N/A	N/A	IC water leach	

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Table ES-2. Original Material Result Summary

Analyte	Envelope	Envelope	n	Original Material					pSpec	Analytical Method	Notes
	D Spec g/L	O Spec g/g		Average μg/g	Average - @ 31g Ox (t)	Std.Dev. g/L	Avg Spec				
C204	None	N/A	6	2.00E+05	8.27E+00	1.0E+04	N/A	N/A	N	IC water leach	N * (17/14) - High Blank
NH3	5E-01	1.21E+04	6	2.12E+01	8.76E-04	6.5E+00	1.75E-03	N	N	ISE water leach	
TIC (CO3)	1.86E+0	4.50E+04	6	2.77E+01	1.14E+00	1.8E+03	6.16E-01	N	N	Direct	
TOC	3.4E+00	8.23E+04	6	5.54E+04	2.29E+00	2.2E+03	6.73E-01	N	N	Direct	
TIC	None	N/A	6	8.31E+04	3.43E+00	2.9E+03	N/A	N/A	N	Direct	
Sum of TOC & TIC											
Radiocesium											
Ag-110m	1.00E-08	2.42E-04	6	<7E-02	<2.89E-08	N/A	<2.86E-02	—	Y	RNIF	GEA
Am-241	4.30E-02	1.04E+03	6	1.55E+00	6.41E-05	3.0E-01	1.49E-03	N	N	RNIF	
Am-243	5.00E-06	1.21E-01	6	<2E-02	<8.27E-07	N/A	<1.65E-01	N	N	RNIF	
C-14	2.00E-06	4.84E-02	6	6.81E-03	2.81E-07	9.1E-03	1.41E-01	N	N	Direct	
Ce-144(Pr-144+144m)	1.00E-04	2.42E+00	6	<2E+00	<8.27E-05	N/A	<8.27E-01	N	N	RNIF	
Ce-144(Pr-144+144m)	1.00E-04	2.42E+00	6	<2E+00	<8.27E-05	N/A	<8.27E-01	N	N	RNIF	Ce-144 Specification
Ce-144(Pr-144+144m)	1.00E-07	2.42E-03	6	<2E+00	<8.27E-05	N/A	<8.27E+02	—	Y	RNIF	Pr-144m Specification
Cm-242(Am-242+242m)	3.10E-05	6.95E-01	6	3.27E-03	1.35E-07	1.1E-03	3.65E-03	N	N	RNIF	Cm-242 Specification
Cm-242(Am-242+242m)	3.10E-05	7.50E-01	6	3.27E-03	1.35E-07	1.1E-03	4.35E-03	N	N	RNIF	Am-242 Specification
Cm-242(Am-242+242m)	3.20E-05	7.74E-01	6	3.27E-03	1.35E-07	1.1E-03	4.22E-03	N	N	RNIF	Am-242m Specification
Cm-243+244	1.00E-09	2.42E-05	6	2.84E-02	1.17E-06	8.6E-03	1.17E-03	—	Y	RNIF	Cm-243 Specification
Cm-243+244	9.30E-04	2.25E-01	6	2.84E-02	1.17E-06	8.6E-03	1.28E-03	N	N	RNIF	Cm-244 Specification
Co-60	3.00E-03	7.26E+01	6	2.36E-01	9.75E-06	1.7E-02	3.25E-03	N	N	RNIF	
Co-134	6.80E-03	1.65E+02	6	<3E-01	<1.24E-05	N/A	<1.82E-03	N	N	RNIF	
Co-135	3.00E-05	7.26E-01	6	2.96E-03	1.22E-07	7.5E-05	4.08E-03	N	N	RNIF	
Co-137(Ba-137)	3.00E+00	7.26E+04	6	5.15E+02	2.13E-02	1.4E+01	7.10E-03	N	N	RNIF	Co-137 Specification
Co-137(Ba-137)	3.00E+00	7.26E+04	6	5.15E+02	2.13E-02	1.4E+01	7.10E-03	N	N	RNIF	Ba-137 Specification
Eu-152	1.50E-04	3.63E+00	6	<2E+00	<8.27E-05	N/A	<5.51E-01	N	N	RNIF	
Eu-154	1.80E-02	3.87E+02	6	1.56E+00	6.45E-05	1.1E-01	4.03E-03	N	N	RNIF	
Eu-155	9.00E-03	2.18E+02	6	1.23E+00	5.08E-05	3.0E-01	5.65E-03	N	N	RNIF	
Fe-55	1.00E-03	2.42E-01	6	6.25E-02	2.56E-06	1.7E-02	2.58E-03	N	N	RNIF	
I-129	8.00E-08	2.18E-03	6	1.90E-03	7.65E-08	4.2E-04	8.73E-01	N	N	ICP/MS Acid	Potential Prep Losses
Nb-93m	8.70E-05	2.10E+00	6	—	—	—	—	—	—	—	Not Analyzed
Ni-59	1.40E-05	3.39E-01	6	<3.00E-02	<1.16E-06	N/A	<6.32E-02	N	N	RNIF	
Ni-63	1.60E-03	3.87E+01	6	1.17E+00	4.64E-05	8.2E-02	3.02E-02	N	N	RNIF	
Np-237	2.30E-05	5.56E-01	6	1.50E-03	6.20E-08	2.1E-04	2.70E-03	N	N	ICP/MS Acid	
Pd-107	4.00E-06	9.68E-02	6	5.80E-03	2.40E-07	3.8E-03	5.99E-02	N	N	ICP/MS Acid	
Pu-238	1.10E-04	2.65E+00	6	3.47E-01	1.43E-05	1.7E-02	1.30E-01	N	N	RNIF	
Pu-239	~5.0E-04	2.30E+01	6	1.30E+00	5.37E-05	2.6E-01	5.66E-02	N	N	ICP/MS NIF	
Pu-240	2.60E-04	6.29E+00	6	1.70E-01	7.03E-06	3.7E-02	2.70E-02	N	N	ICP/MS Acid	
Pu-241	6.90E-03	1.67E+02	6	3.68E+00	1.52E-04	1.1E+00	2.20E-02	N	N	RNIF	
Pu-242	7.10E-08	1.72E-03	6	<2E-02	<8.27E-07	N/A	<1.16E-01	—	Y	RNIF	
Ru-106(Rh-106)	2.00E-04	4.84E+00	6	<3E+00	<1.24E-04	N/A	<6.20E-01	N	N	RNIF	Ru-106 Specification
Ru-106(Rh-106)	2.00E-04	4.84E+00	6	<3E+00	<1.24E-04	N/A	<6.20E-01	N	N	RNIF	Rh-106 Specification
Sb-125(Te-125m)	1.00E-02	2.42E+02	6	<2E+00	<8.27E-05	N/A	<8.27E-03	N	N	RNIF	Sb-125 Specification
Sb-125(Te-125m)	3.00E-03	7.26E-01	6	<2E+00	<8.27E-05	N/A	<2.76E-02	N	N	RNIF	Te-125m Specification
Se-79	4.20E-07	1.02E-02	6	<2E-03	<6.92E-08	N/A	<2.35E-01	N	N	RNIF	
Sm-151(Pm-147)	9.30E-02	2.25E+03	6	4.52E+01	1.87E-03	1.0E+01	2.01E-02	N	N	RNIF	Sm-151 Specification
Sm-151(Pm-147)	1.60E-01	3.67E+03	6	4.52E+01	1.87E-03	1.0E+01	1.17E-02	N	N	RNIF	Pm-147 Specification
Sn-113(In-113)	1.88E-06	4.55E-02	6	<3E-02	<1.24E-06	N/A	<6.60E-01	N	N	RNIF	Sn-113 Specification
Sn-113(In-113)	1.88E-06	4.55E-02	6	<3E-02	<1.24E-06	N/A	<6.60E-01	N	N	RNIF	In-113 Specification
Sn-115m	1.00E-08	2.42E-04	6	<4E-01	<1.65E-05	N/A	<1.65E+03	—	Y	RNIF	
Sn-121m	9.00E-06	2.18E-01	6	<3E+00	<1.24E-04	N/A	<1.38E-01	—	Y	RNIF	
Sn-126(Sb-126m, Sb-126)	4.80E-05	1.16E+00	6	<3E-02	<1.24E-06	N/A	<2.58E-02	N	N	RNIF	Sn-126 Specification
Sn-126(Sb-126m, Sb-126)	3.43E-05	8.30E-01	6	<3E-02	<1.24E-06	N/A	<3.62E-02	N	N	RNIF	Sb-126m Specification
Sn-126(Sb-126m, Sb-126)	4.83E-06	1.17E-01	6	<3E-02	<1.24E-06	N/A	<2.57E-01	N	N	RNIF	Sb-126 Specification
Sr-90(Y-90)	3.10E+00	7.50E+04	6	3.54E+02	1.46E-02	1.1E+01	4.72E-03	N	N	RNIF	Sr-90 Specification
Sr-90(Y-90)	3.10E+00	7.50E+04	6	3.54E+02	1.46E-02	1.1E+01	4.72E-03	N	N	RNIF	Y-90 Specification
Tc-99	4.50E-03	1.09E+02	6	3.00E-02	1.24E-06	3.2E-03	2.76E-04	N	N	ICP/MS Acid	
Th-232	See "Th"	See "Th"	6	3.90E-06	1.61E-10	9.7E-07	N/A	N/A	N	ICP/MS NIF	
U-233	1.00E-09	2.42E-05	6	<4E-03	<1.65E-07	N/A	<1.65E-02	—	Y	N	TRACE Comp - Th Interferes
U-234	7.70E-07	1.86E-02	6	<1E-03	<4.13E-08	N/A	<5.37E-02	N	N	ICP/MS Acid	
U-235	3.20E-08	7.74E-04	6	1.60E-05	6.61E-10	1.5E-06	2.07E-02	N	N	ICP/MS Acid	
U-236	8.20E-08	1.88E-03	1	2.60E-05	1.07E-09	N/A	1.31E-02	N	N	ICP/MS Acid	Five of Six Results <EQL
U-238	5.83E-07	1.40E-02	6	3.88E-04	1.51E-06	3.9E-05	2.81E-02	N	N	ICP/MS Acid	
Zr-93	1.40E-04	3.39E+00	6	4.72E-02	1.95E-06	1.8E-02	1.39E-02	N	N	RNIF	
Total Alpha	none	N/A	6	3.21E+00	1.33E-04	4.5E-01	N/A	N/A	N	RNIF	
Total Beta	none	N/A	6	1.22E+03	5.04E-02	4.4E+01	N/A	N/A	N	RNIF	
Physical Properties											
				Original Material							
				HLRF	SAL						
Wt% Solids @105C - (%)	2.5 - 13			29.2	N/A						
Wt% Oxide @1050C - (%)				21.9	—						
Total Oxide - (g/L)	25 - 100			—	—						
Slurry Density - (g/ml)	1.02 - 1.10			1.25	—						
Settled Solids - (Vol%)	7 - 95			41.80	—						
pH	>10			—	—						
See Note 2											

HLRF/High Level Radiation Facility; SAL/Shielded Analytical Laboratory; "—"= not measured; N/A=not applicable

(1) g/L = (ug analyte/ dry solids)/(wt% solids/wt% oxides)/(31g oxide/L)/(1E-06gug). C.A. = (uCi analyte/ dry solids)/(wt% solids/wt% oxides)/(31g oxide/L)/(1E-06gug).

(2) HLRF Wt% solids performed in duplicate on 2-3 gram sample aliquots of Original Material.

Original Material: Initial Composite Material + Deionized Water @ 3:2 Ratio, with 22.5% Supernatant Removed Before Drying [See Sections 2.2 and 3.1]

Definition of Terms used in Table ES-1

Avg/Spec	Average result divided by Envelope D specification.
Flag > Spec	N means result does not exceed Envelope D specification, Y indicates result exceeds specification.
n	Number of subsamples (including duplicates) used to generate the average and standard deviation.
N/A	Not Applicable
<	Analyte/Radionuclide of interest was not detected above the instrument detection limit
ICPZrF	Na ₂ O ₂ fusions (Zr crucible) analyzed by ICP-AES
ICP/MS ZrF	Na ₂ O ₂ fusions (Zr crucible) analyzed by ICP-MS
RZrF	Na ₂ O ₂ fusions (Zr crucible) analyzed for radionuclide
ICPNiF	KOH fusions (Ni crucible) analyzed by ICP-AES
ICP/MS NiF	KOH fusions (Ni crucible) analyzed by ICP-MS
RNiF	KOH fusions (Ni crucible) analyzed for radionuclide
ICP Acid	Acid digestion analyzed by ICP-AES
ICP/MS Acid	Acid digestion analyzed by ICP-MS
IC Water Leach	Water leach preparation analyzed by IC
ISE Water Leach	Water leach preparation analyzed by ISE
Direct	Analysis performed on direct aliquots of the material

TERMS/ACRONYMS/DEFINITIONS

ACL	Analytical Chemistry Laboratory
AEA	Alpha Energy Analysis
CVAA	Cold Vapor Atomic Absorption
GEA	Gamma Energy Analysis
HLRF	High Level Radiochemistry Facility
IC	Ion Chromatography
ICP-AES	Inductively Coupled Plasma - Atomic Emission Spectrometry
ICP-MS	Inductively Coupled Plasma - Mass Spectroscopy
ISE	Ion Specific Electrode
LEP	Low-Energy Photon (counting)
LOI	Letter of Instruction
LSC	Liquid Scintillation counting
m/z	mass-to-charge ratio (ICP-MS)
PNNL	Pacific Northwest National Laboratory
QC	Quality Control
SAL	Shielded Analytical Laboratory
TIMS	Thermal Ionization Mass Spectroscopy
TWRS	Tank Waste Remediation System

Quality Control Definitions:

Estimated Quantitation Level (EQL): The EQL is the lowest measurement that can be reliably achieved within specific limits of precision and accuracy during routine operating conditions. Typically, the EQL is either ten times the IDL or is the lowest calibration standard, whichever is appropriate for a specific method. Results reported to the EQL have been adjusted for sample preparation and analytical dilution factors.

Instrument Detection Limit (IDL): The IDL is the minimum concentration that can be measured and reported within 99% confidence that the value is above zero. Generally, the IDL is determined by the repeated analysis of standards with concentrations at or near the estimated IDL. Results reported to the IDL have been adjusted for sample preparation and analytical dilution factors.

Mean Difference (MD): The MD determines if two results are statistically different at the 95% confidence level. MD results greater or equal to 1.96 indicate there is a 95% confidence that the two results are not equal. The MD is typically used to evaluate radionuclide results.

Minimum Detectable Activity (MDA): The MDA is applied to nuclear counting and is defined as the level of activity that is practically achievable by a measurement system using a specified counting time for a specific sample quantity and sample dilution factor.

Relative Percent Difference (RPD): The RPD expresses the precision of duplicates. Generally, RPD values less than 20% indicate acceptable precision.

Quality Control Sample Type Definitions:

Duplicate (D): A second subsample (or aliquot) of the "sample to be analyzed" that is treated/processed the same as the initial subsample in order to determine the precision of the method.

Laboratory Blank: An analytical control used by the laboratory to monitor specific processes for potential contamination.

Laboratory Control Sample (LCS): A control sample of known composition. The laboratory control sample is analyzed using the same sample preparation, reagents, and analytical methods that are used for samples.

Matrix Spike/Matrix Spike Duplicate (MS/MSD): A subsample (or aliquot) of a sample spiked with a known quantity of a specific analyte of interest and subjected to the entire analytical procedure in order to indicate the appropriateness of the method to the sample matrix by measuring recovery.

Preparation Blank (PB): An analytical control that contains processing reagents only, which is carried through the entire analytical procedure and used to identify potential processing contamination.

Replicate: A replicate is a second analysis of a single sample by the same method, and usually applies to solutions. For solids, a single subsample analyzed twice may be defined as a replicate analysis; in contrast to a duplicate analysis, where two subsamples are analyzed.

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1.0 INTRODUCTION

This report represents the Analytical Summary Report on Tank Waste Remediation System (TWRS) Privatization Contractor Samples conducted in support of the TWRS Privatization Contractor Samples, Waste Envelope D, C-106 characterization testing. All work performed was conducted in accordance with "Addendum 1 of the Letter of Instruction (LOI) for TWRS Privatization Contractor Samples Addressing Waste Envelope D Materials - Revision 0," (Jones, 1996). All work was conducted under the direction of the assigned Pacific Northwest National Laboratory (PNNL) Analytical Chemistry Laboratory (ACL) Project Manager.

Tank 241-C-106 (C-106) was selected by TWRS Privatization for the Part 1A Envelope D high-level waste demonstration. Twenty bottles of Tank C-106 material were collected using the grab sampling technique and transferred to the 325 building. The contents of the twenty bottles were composited and subsampled for the laboratory-scale screening test and characterization testing. Envelope D material bench-scale batch preparation was performed in the 324 building, followed by characterization testing in the 325 building.

The C-106 composite batch material was characterized during three sequential stages of Envelope D sample preparation: 1) after initial dilution with water prior to beginning the bench-scale processing ("Original Material"); 2) after bench-scale caustic leach and inhibited water wash treatment per revision 0 of the LOI ("Vendor Product"); and 3) after decant and dilute treatment per revision 1 of the LOI ("Final Vendor Product").

This report is one of a series of reports addressing the C-106 vendor sample preparation and characterization. This summary report includes analytical results on the Envelope D Privatization Vendor Product and the Original Material. The final vendor product results are provided in Addendum 1 to this report (Urie 1997). A subsequent Analytical Final Report (in progress) will include an assessment of the preparation and analytical methods, all raw data, and an evaluation of lessons learned. The final reports detailing the laboratory-scale screening and final bench-scale preparation are provided by Lumetta (1996) and Brooks (1997), respectively. Statistical reports for the C-106 analytical data will be prepared by TL Welsh (in progress).

The Envelope D analytes of interest (non-radionuclide and radionuclide), recommended methods, and estimated quantitation levels are defined in revision 0 of the LOI. Table 1.1 summarizes the LOI's analytical requirements for the Vendor Product. In many cases the recommended method for non-radionuclides is ICP-AES. Due to the low concentrations of many of the analytes of interest, ICP-MS results have been reported.

The sample handling, preparation, and analyses are described in Sections 2, 3, and 4, respectively.

Table 1.1 Analytical Requirements for Envelope D Analytes/Radionuclides

Method	Analyte	Procedure	EQL $\mu\text{g/g}$
ICP-AES	Ag	PNL-ALO-211	60
ICP-AES	Al	PNL-ALO-211	240
ICP-AES	B	PNL-ALO-211	80
ICP-AES	Ba	PNL-ALO-211	40
ICP-AES	Be	PNL-ALO-211	20
ICP-AES	Bi	PNL-ALO-211	400
ICP-AES	Ca	PNL-ALO-211	200
ICP-AES	Cd	PNL-ALO-211	60
ICP-AES	Ce	PNL-ALO-211	400
ICP-AES	Co	PNL-ALO-211	40
ICP-AES	Cr	PNL-ALO-211	80
ICP-AES	Cu	PNL-ALO-211	40
ICP-AES	Dy	PNL-ALO-211	200
ICP-AES	Eu	PNL-ALO-211	400
ICP-AES	Fe	PNL-ALO-211	80
ICP-AES	K	PNL-ALO-211	8000
ICP-AES	La	PNL-ALO-211	200
ICP-AES	Li	PNL-ALO-211	120
ICP-AES	Mg	PNL-ALO-211	400
ICP-AES	Mn	PNL-ALO-211	20
ICP-AES	Mo	PNL-ALO-211	120
ICP-AES	Na	PNL-ALO-211	320
ICP-AES	Nd	PNL-ALO-211	200
ICP-AES	Ni	PNL-ALO-211	120
ICP-AES	P	PNL-ALO-211	400
ICP-AES	Pd	PNL-ALO-211	1200
ICP-AES	Rh	PNL-ALO-211	1200
ICP-AES	Ru	PNL-ALO-211	400
ICP-AES	Si	PNL-ALO-211	2000
ICP-AES	Sn	PNL-ALO-211	4000
ICP-AES	Sr	PNL-ALO-211	20

Method	Analyte	Procedure	EQL µg/g
ICP-AES	Te	PNL-ALO-211	2000
ICP-AES	Th	PNL-ALO-211	3200
ICP-AES	Ti	PNL-ALO-211	20
ICP-AES	U	PNL-ALO-211	8000
ICP-AES	V	PNL-ALO-211	40
ICP-AES	W	PNL-ALO-211	2000
ICP-AES	Y	PNL-ALO-211	40
ICP-AES	Zn	PNL-ALO-211	80
ICP-AES	Zr	PNL-ALO-211	40
ICP-MS	Cs	PNL-ALO-280	0.2
ICP-MS	Gd	PNL-ALO-280	0.2
ICP-MS	Nb	PNL-ALO-280	0.2
ICP-MS	Pr	PNL-ALO-280	0.2
ICP-MS	Rb	PNL-ALO-280	0.2
ICP-MS	Re	PNL-ALO-280	0.2
ICP-MS	Ta	PNL-ALO-280	0.2
ICP-AES / ICP-MS	As	PNL-ALO-211 / 280	4800 / 20
ICP-AES / ICP-MS	Cd	PNL-ALO-211 / 280	300 / 6
ICP-AES / ICP-MS	Pb	PNL-ALO-211 / 280	1200 / 2
ICP-AES / ICP-MS	Sb	PNL-ALO-211 / 280	9000 / 4
ICP-AES / ICP-MS	Se	PNL-ALO-211 / 280	6000 / 100
ICP-AES / ICP-MS	Tl	PNL-ALO-211 / 280	9000 / (n/a)
CN	CN	PNL-ALO-287/289/285	0.1
CVAA	Hg	PNL-ALO-131/201	0.05
IC	Cl	PNL-ALO-212	50
IC	F	PNL-ALO-212	50
IC	NO ₂	PNL-ALO-212	100
IC	NO ₃	PNL-ALO-212	100
IC	SO ₄	PNL-ALO-212	100
IC	PO ₄	PNL-ALO-212	100
Hot Persulfate	TIC/TOC	PNL-ALO-381/382/383	1
Ion Specific Electrode	NH ₄ -N	PNL-ALO-226	0.1

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Method	Radionuclide	Procedure	EQL $\mu\text{Ci/g}$
GEA	^{60}Co	PNL-ALO-450	200
GEA	^{106}Ru (^{106}Rh)	PNL-ALO-450	2000
GEA	^{110m}Ag	PNL-ALO-450	200
GEA	^{125}Sb (^{125m}Te)	PNL-ALO-450	500
GEA	^{134}Cs	PNL-ALO-450	200
GEA	^{137}Cs (^{137}Ba)	PNL-ALO-450	200
GEA	^{144}Ce (^{144}Pr , ^{144m}Pr)	PNL-ALO-450	200
GEA	^{152}Eu	PNL-ALO-450	1000
GEA	^{154}Eu	PNL-ALO-450	1000
GEA	^{155}Eu	PNL-ALO-450	1000
separation-->beta count	^{90}Sr (^{90}Y)	PNL-ALO-476/431	100
separation-->beta count	^{93}Zr (^{93m}Nb)	PNL-ALO-478 (modified)	10
separation-->beta count	^{147}Pm + ^{151}Sm	PNL-ALO-474	TBD
separation-->AEA	^{239}Pu	PNL-ALO-417/422	100
separation-->AEA	$^{239-240}\text{Pu}$	PNL-ALO-417/422	100
separation-->AEA	^{242}Pu	PNL-ALO-417/422	100
separation-->AEA	^{241}Am	PNL-ALO-417/422	100
separation-->AEA	^{243}Am	PNL-ALO-417/422	100
separation-->AEA	^{242}Cm (^{242}Am , ^{242m}Am)	PNL-ALO-417/422	100
separation-->AEA	$^{243-244}\text{Cm}$	PNL-ALO-417/422	100
separation-->X-ray and gamma count	^{113}Sn (^{113m}In)	PNL-ALO-453	200
separation-->X-ray and gamma count	^{119m}Sn	PNL-ALO-453	500
separation-->X-ray and gamma count	^{121m}Sn	PNL-ALO-453	800
separation-->X-ray and gamma count	^{126}Sn (^{126}Sb , ^{126m}Sb)	PNL-ALO-453	200
separation-->X-ray count	^{55}Fe	PNL-ALO-435	100
separation-->X-ray count	^{59}Ni	PNL-ALO-495/450	100
separation-->liquid scintillation count	^{63}Ni	PNL-ALO-495/450	10
separation -->liquid scintillation count	^{241}Pu	PNL-ALO-417/4741	100
oxidize-->liquid scintillation	^{14}C	PNL-ALO-428/474	10

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Method	Radionuclide	Procedure	EQL $\mu\text{Ci/g}$
distillation-->liquid scintillation	^{76}Se	PNL-ALO-440/474	10
thermal ionization mass spectrometry	^{137}Cs	PNL-ALO-452	TBD
ICP-MS	^{99}Tc	PNL-ALO-280/281	0.2
ICP-MS	^{107}Pd	PNL-ALO-280	0.2
ICP-MS	^{129}I	PNL-ALO-280	4
ICP-MS	^{237}Np	PNL-ALO-280	0.2
ICP-MS	^{239}Pu	PNL-ALO-280	0.2
ICP-MS	^{240}Pu	PNL-ALO-280	0.2
ICP-MS	$^{241}\text{Pu} + ^{241}\text{Am}$	PNL-ALO-280	0.2
ICP-MS	^{242}Pu	PNL-ALO-280	0.2
ICP-MS	^{232}Th	PNL-ALO-280	0.2
ICP-MS	^{235}U	PNL-ALO-280/282	0.2
ICP-MS	^{238}U	PNL-ALO-280/282	0.2
ICP-MS	^{234}U	PNL-ALO-280/282	0.2
ICP-MS	^{236}U	PNL-ALO-280/282	0.2
ICP-MS	^{238}U	PNL-ALO-280/282	0.2
	$^{113\text{m}}\text{Cd}$ *	Test Plan	TBD

- * Currently there is no method for $^{113\text{m}}\text{Cd}$, and ORIGEN must be used to estimate the concentration. However, a method has recently been proposed, and would be analyzed under a test plan, if approved.

2.0 PRELIMINARY SAMPLE HANDLING

Tank C-106 material was sampled 6/21/96 through 6/24/96 and transferred to the 325 building High Level Radiochemistry Facility (HLRF) 6/22/96 through 6/26/96. Sampling information from chain of custody data is presented in Table 2.1.

Twenty grab samples from C-106 were received from the East Tank Farms. Each grab sample was transported to the 325 building in a 125 mL jar shielded by a B Plant shipping container. Due to the dose rates of these samples, the samples were transferred into a hot cell where they were homogenized and composited in a series of steps. Each sample was individually homogenized with a high speed mixer (OMNI Mixer) according to PNNL technical procedure PNL-ALO-135, "Laboratory Procedure for Homogenization of Solutions, Slurries and Sludges", section 3.0. An ice bath was used to control the temperature of the samples during homogenization.

The homogenized samples were transferred to a 2 liter carboy until the carboy was approximately 3/4 full (≈ 1.5 Liters or 12 samples). The homogenized samples were composited by stirring for approximately 15 minutes with a mechanical stirring motor and a Teflon stirring rod. Approximately 750 mL of this sample was transferred to a 1 liter jar, and the remaining homogenized samples were transferred to the carboy where they were composited by the same procedure as the previous samples. In order to prepare a homogeneous composite, a series of cuts were made between the 1 liter jar and the 2 liter carboy with stirring between each cut. The homogenized composite was then split between the 1 liter jar and 2 liter carboy. The homogenized composite represents the Initial Composite Material. A 15 gram aliquot of the Initial Composite Material was taken and transferred to the Shielded Analytical Laboratory (SAL) for the laboratory-scale screening test.

2.1 Laboratory-Scale Screening Test

The screening test was conducted in accordance with PNNL technical procedure PCS-TP-511-1, Rev. 3. This screening test was designed to test whether washing with dilute hydroxide solution or a combination of caustic leaching and dilute hydroxide washing would yield a solids product (i.e., Vendor Product) which conforms to Envelope D specifications. In the screening test, approximately 15 grams of Initial Composite Material was mixed with approximately 29 grams of water. This test material was divided into five portions; for "simple" sludge washing, "enhanced" sludge washing, chemical analysis, particle size, and microscopy studies.

To determine the chemical composition of the untreated, Initial Composite Material, one 2.2 gram slurry portion was dried for chemical analysis, while two small portions (i.e., 0.7 grams and 0.5 grams slurry) were set aside for particle-size analysis and microscopy studies. A 4.5 gram slurry portion of the test material was washed three successive times with 0.01 M NaOH to determine the effects of "simple" sludge washing; for each wash, approximately 17 grams of wash solution was used per gram of dry sludge solids. Each wash consisted of mixing the sludge/0.01 M NaOH mixture for 1 hour at 100°C, allowing to cool to ambient temperature, centrifuging, then decanting the wash liquor. The washed solids were dried to constant weight at 105°C and analyzed.

Table 2.1 241-C-106 Sample Receipt Information¹

Sample #	Tank/ Riser	Date Sampled	Sampling Depth (inches)	Dose @ Contact (R/hr)	Expected Liquid Content	Expected Solid Content	Date Received In 325	Shipment #
6C-96-18	C106/7	6/21/96	406	1.5	30%	70%	6/22/96	37406
6C-96-19	C106/7	6/21/96	406	1.0	20%	80%	6/22/96	37406
6C-96-20	C106/7	6/21/96	407	2.4	20%	80%	6/22/96	37405
6C-96-21	C106/7	6/21/96	408	1.5	20%	80%	6/22/96	37408
6C-96-22	C106/7	6/21/96	408	2.0	20%	80%	6/22/96	37408
6C-96-23	C106/7	6/21/96	409	1.1	20%	80%	6/22/96	37407
6C-96-24	C106/7	6/21/96	409	2.0	30%	70%	6/22/96	37410
6C-96-25	C106/7	6/21/96	411	1.0	20%	80%	6/22/96	37410
6C-96-26	C106/7	6/21/96	411	1.0	20%	80%	6/22/96	37409
6C-96-27	C106/7	6/21/96	411	2.0	20%	80%	6/22/96	37411
6C-96-28	C106/7	6/21/96	413	2.3	20%	80%	6/22/96	37411
6C-96-29	C106/7	6/21/96	413	1.8	20%	80%	6/22/96	37412
6C-96-30	C106/7	6/23/96	413	2.5	50%	50%	6/24/96	37414
6C-96-31	C106/7	6/23/96	413	2.3	50%	50%	6/24/96	37413
6C-96-32	C106/7	6/23/96	414	1.9	50%	50%	6/24/96	37413
6C-96-33	C106/7	6/23/96	414	1.5	50%	50%	6/25/96	37415
6C-96-34	C106/7	6/24/96	414	1.4	50%	50%	6/25/96	37415
6C-96-35	C106/7	6/24/96	414	1.5	50%	50%	6/25/96	37416
6C-96-36	C106/7	6/24/96	414	1.7	50%	50%	6/26/96	37417
6C-96-37	C106/7	6/24/96	414	2.0	50%	50%	6/26/96	37417

¹Reference: Process Memo, RG Brown to KD Wiemers, *Request for Sludge Sample From Tank 241-C-106*. June 17, 1996.

The remainder of the test material was subjected to an "enhanced" sludge washing test; two caustic and three inhibited water washes. The first caustic leach step consisted of mixing the sludge/NaOH mixture (slurry contained 3.3 weight percent solids and the final free hydroxide concentration was 2.6 M) for 5 hours at 100°C, allowing the mixture to cool to ambient temperature, allowing the sludge

solids to settle by gravity, then decanting the liquid. The second caustic leach was done in a similar manner, but the slurry contained 3.9 weight percent solids and the final free hydroxide concentration was 3.0 M. The leached solids were washed three successive times with inhibited water (i.e., 0.01 M NaOH/0.01 M NaNO₂); these washes consisted of mixing the sludge/wash mixture for 0.5 hours at ambient temperature, allowing the solids to settle by gravity, then removing the wash liquor. The leached solids were dried to constant weight at 105°C and analyzed.

The laboratory-scale washing and caustic leaching tests performed on C-106 sludge material are detailed in report PNNL-11381 (Lumetta 1996). Although no analytical results associated with the laboratory-scale testing are presented in this report, the results from this testing were used (as available) to establish the sludge washing treatment for the bench-scale batch preparation.

2.2 Bench-Scale Vendor Product Batch Preparation

After removing the material for the laboratory-scale testing, all of the remaining composited C-106 material was transferred to the 324 building for Envelope D final batch processing (Enhanced Sludge Washing). Batch processing followed Test Plan Number TWRS-95-3.1A which was modified for C-106 batch preparation based upon performance of the laboratory-scale screening test. Approximately 3000 grams of C-106 Initial Composite Material was blended with approximately 2000 grams of deionized water prior to initiating the enhanced sludge washing final batch processing. At this stage, four subsamples identified as Original Batch Material (hereafter referred to as Original Material unless otherwise noted; see Table 2.2) were extracted from the process and sent to the 325 building for physical and chemical characterization. A fifth subsample was collected for archive.

As with the laboratory-scale testing, two caustic leaches and 3 inhibited water washes were performed. Each leach or wash step was followed by gravity settling and supernatant removal to separate the liquid and solid phases. The first caustic leach added 10 M NaOH and deionized water to the sludge and obtained a 2.74 M free hydroxide solution with 5.7 weight percent caustic-insoluble solids. The slurry was mixed at 100°C for 5 hours and allowed to settle at 85°C. The supernatant was then decanted in preparation for the second caustic leach. The second caustic leach utilized 3 M NaOH to obtain 5.8 weight percent caustic-insoluble solids with 3.22 M free hydroxide; the same mixing and settling temperatures were used as in the first caustic leach. Each of the subsequent three water washes utilized inhibited water (i.e., 0.01 M NaOH/0.01 M NaNO₂) added to obtain approximately 6 weight percent solids concentration. For each wash, the sludge was mixed, settled at ambient temperature, and the supernatant decanted. The bench-scale processing tasks, procedures, and data evaluation are presented in report PNNL-11432 (Brooks 1997).

Following final batch processing, the resulting material (Vendor Product) was thoroughly mixed and five subsamples were collected for subsequent physical and chemical characterization and archive. The four Vendor Product subsamples and all additional pumpable Vendor Product material (two 1L containers) were collected and delivered to the 325 building (See Table 2.2); the remaining subsample was archived in the 324 building. Besides the Vendor Product material, the remaining Initial Composite Material (approximately 800 g for archiving) and samples of the supernatants from each caustic leach and water wash (for analysis) were transferred to the 325 building.

Table 2.2 C-106 Material Transfer from Building 324 to 325

324 Sample ID	Net Weight (g)	Description
SPDL1-C106-003/005 and SPDL1-C106-002	507	Original "Batch" Material ^(a)
SPD1-C106-010	16.4	Supernatant from First Caustic Leach
SPD1-C106-015	22.8	Supernatant from Second Caustic Leach
SPD1-C106-020	22.4	Supernatant from First Water Wash
SPD1-C106-024	13.0	Supernatant from Second Water Wash
SPD1-C106-029	23.2	Supernatant from Third Water Wash
SPD1-C106-046	90.7	Vendor Product (Leached & Washed Sludge)
SPD1-C106-047	70.3	Vendor Product (Leached & Washed Sludge)
SPD1-C106-048	84.7	Vendor Product (Leached & Washed Sludge)
SPD1-C106-049	62.8	Vendor Product (Leached & Washed Sludge)
C-106	800	Initial Composite Material ^(b)
SPD1-C106-051	1624	Vendor Product (Leached & Washed Sludge)
SPD1-C106-052	1523	Vendor Product (Leached & Washed Sludge)

^(a) Original Batch Material: Initial Composite Material and Water mixed at 3:2 ratio to provide initial pumpable material for bench-scale testing.

^(b) Initial Composite Material: Composited material from twenty C-106 grab samples; no additional water added.

2.3 Chemical, Radiochemical, and Physical Characterization

Table 2.3 provides a summary list of all the samples processed and analyzed per revision 0 of the LOI. Additional details of the characterization schemes are provided in Section 3.

Table 2.3 Sample Laboratory Numbers

ACL Lab Number	Sample ID	Material Description and Purpose
96-5892	SPDL1-C106-003	Original Batch Material - Chemical Analysis
96-5893	SPDL1-C106-004	Original Batch Material - Chemical Analysis
96-5894	SPDL1-C106-005	Original Batch Material - Chemical Analysis
96-5945	SPDL1-C106-002	Original Batch Material - Physical Testing
96-5946	C106-002-CS	Centrifuged Solids from SPDL1-C106-002 - Chemical Analysis
96-5947	C106-002-DL	Decant Liquid from SPDL1-C106-002 - Chemical Analysis
96-6103	SPD1-C106-047	Vendor Product Material - Chemical Analysis
96-6104	SPD1-C106-048	Vendor Product Material - Chemical Analysis
96-6105	SPD1-C106-049	Vendor Product Material - Chemical Analysis
96-6396	SPD1-C106-046	Vendor Product Material - Physical Testing
96-6653	SPD1-C106-046	Vendor Product Material - Additional Physical Testing
96-6654	C106-046-DL	Decant Liquid from SPD1-C106-046 (96-6653) - Chemical Analysis
97-0108	SPD1-C106-046	Vendor Product Material - Physical Testing (Wt% Solids/Oxide)
97-0109	SPD1-C106-002	Original Batch Material - Physical Testing (Wt% Solids/Oxide)

3.0 SAMPLE PREPARATION

Four subsamples were collected by the Chemical Separations & Batch Processing Group in the 324 building for physical and chemical analyses from both the C-106 Original Material and from the bench-scale batch preparation of the C-106 Vendor Product. Three subsamples each from the Original Material and Vendor Product were distributed to the SAL for drying and subsequent analytical sample preparation and/or direct distribution for chemical analysis. The remaining subsamples of the Original Material and Vendor Product were distributed to the HLRF for density, weight loss on drying and firing measurements, and additional physical testing.

3.1 Preparations for Chemical Analyses

Vendor Product (See Figure 3.1): The SAL received three containers of Vendor Product for chemical analysis. Each of the three Vendor Product slurries were placed into glass beakers for mixing and drying. The mixed slurries were dried in an oven at 105°C until successive weighings yielded less than 1 percent weight loss between successive dryings. The observed volume reduction of the Vendor Product slurries was approximately 80 percent after drying. In all cases, the remaining dried solids were medium brown in color and the evaporated supernatant left a white coating on the beaker sides and on top of the dried brown solid layer. No indication of spattering, foaming, etc. during drying was noted as evidenced by clean beaker walls above the original fill lines. After drying, each of the three dried slurries were homogenized, and then divided into two halves; forming a sample and a sample duplicate. Homogenization was accomplished by mechanically stirring the beaker contents with a metallic spatula until the contents of each beaker appeared uniform. Analytical sample preparations were conducted on the Vendor Product dried slurries. In addition, analytical sample preparation of a decant supernatant was performed. This decant liquid sample (96-6654) was generated during physical characterization conducted in the HLRF.

Original Material (See Figure 3.2): The SAL received three containers of Original Material for chemical analysis. Each of the three Original Material slurries were placed into glass beakers for mixing and drying. Within minutes after transferring the slurries to beakers, a clear liquid layer was observed forming on top of each slurry material. The layer was suspected to be organic in nature, because the layer separated rapidly, was distinct from the slurry, and coated the beaker walls when swirled. Due to the concern with drying any material having an organic top layer, the suspected layer was removed from the slurries by pipet and placed back into the original containers.

[Note: For each of samples, the clear layer was estimated to be about 10% of the total volume of the samples (i.e., 10 ml out of 100 ml), based on the graduates on the beakers. Approximately 8 to 9 ml of the clear layer was removed; the additional 1 to 2 ml could not be removed from the samples without removing solids. The specific gravity of the clear layer was determined to be 1.06 g/mL, and investigative organic and radchem screening tests were performed. The major organic components identified were bis(2-ethylhexyl)phosphate and oxalate; organics such as EDTA, HEDTA, TBP and HPH were not observed. This layering was not observed in the preparation of material from the laboratory-scale testing, which may be attributed to the additional water added prior to bench-scale processing. A report detailing the results of the organic and radiochemical screening is provided in a separate letter report (Urie 1996). Based on limited analysis of the remaining supernatant and the volume percent

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solids, the removal of the 8 to 9 ml of clear layer is estimated to lower the concentration of the soluble analytes by about 20%, and have no effect on the concentration of the insoluble solids analytes.]

Figure 3.1 Vendor Product Sample Preparation

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Figure 3.2 Original Material Sample Preparation

The remainders of the Original Material slurries were dried in an oven at 105°C until successive weighings yielded less than 1 percent weight loss between successive dryings. The observed volume reduction of the Original Material slurries was approximately 50% after drying; however, this result does not include correction for the clear layer removed prior to drying. In all cases, the remaining dried solids were medium brown in color and the evaporated supernatant left a white coating on the beaker sides and on top of the dried brown solid layer. No indication of spattering, foaming, etc. during drying was noted as evidenced by clean beaker walls above the original fill lines. After drying, each of the three dried slurries was homogenized, and then divided into two halves; forming a sample and a sample duplicate. Homogenization was accomplished by mechanically stirring the beaker

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contents with a metallic spatula until the contents of each beaker appeared uniform. Analytical sample preparations were conducted on the Original Material dried slurries, as well as a centrifuged solid and decanted supernatant. The centrifuged solid (96-5946) and decanted liquid (96-5947) samples were generated during physical characterization conducted in the HLRP.

Original Material and Vendor Product: Sample preparations conducted in the SAL included potassium hydroxide fusion utilizing nickel crucibles, sodium peroxide-sodium chloride fusion utilizing zirconium crucibles, nitric acid-hydrochloric acid digestion, and water leaching. All samples were prepared for analysis in duplicate from the dried slurries, based on the subsampling discussed above. Direct analyses were performed in duplicate wherever practical. All results are reported in $\mu\text{g/g}$ or $\mu\text{Ci/g}$ of dried solids. Quality control (QC) was applied to analytical batch processing techniques in accordance with the governing quality assurance plan (Kuhl-Klinger 1996). Full QC incorporates the use of preparation blanks, spiked blanks (or laboratory control standards), duplicate samples, matrix spiked samples, and radiochemical tracers, where available and appropriate for use. Figure 3.3 (Vendor Product) and Figure 3.4 (Original Material) identify each of the analytical preparations performed and show the analyses performed on each preparation.

3.1.1 Acid Digestion Preparation

Each of the dried samples and sample duplicates were acid digested by method PNL-ALO-129 "HNO₃-HCl Acid Extraction of Solids Using a Dry-Block Heater," and distributed for ICP-AES, ICP-MS and Radiochemical analyses. Generally, one-half gram samples are digested in about 25% Nitric/Hydrochloric acid, and then diluted to a 25 mL final volume, producing approximately 50 fold analytical processing factors. Full QC, including matrix spikes, was applied with this technique.

3.1.2 Potassium Hydroxide and Sodium Peroxide Fusion Preparations

Two fusion techniques, PNL-ALO-115 "Solubilization of Metals from Solids Using KOH-KNO₃ Fusion" and PNL-ALO-116 "Solubilization of Platinum Group Metals using a Na₂O₂-NaCl Fusion," were performed and aliquots distributed for ICP-AES, ICP-MS and Radiochemical analyses. The fusion techniques are usually applied where total solubilization of the sample material is required. Typically, one hundred milligram samples are subjected to molten salt and the resulting mixture dissolved with nitric/hydrochloric acid. The final volume for this preparation is 100 mL, thus creating 1000 fold analytical processing factors. The QC is limited to sample duplicates, a preparation blank, and post fusion spiking (i.e., spiking being performed during analysis) for selective analytes.

3.1.3 Water Leaching Preparation

Each dried sample and sample duplicate were water leached (PNL-ALO-103 "Water Leach of Sludges, Soils and Other Solid Samples"), filtered, and aliquots distributed for anion analysis by Ion Chromatography (IC) and ammonia determination by Ion Selective Electrode (ISE). The water leaching technique produces a 100 fold analytical processing factor. Full QC was applied for all analytes of interest. Oxalate was noted by IC during analysis of the Vendor Product and Original Material, but is not defined as an analyte of interest in the LOI. Since oxalate was not an analyte of interest, no matrix spiking was performed as per the governing QA plan requirements.

Figure 3.3 Vendor Product Preparations and Analyses

* IC anions include: F, Cl, Br, C₂O₄, NO₃, NO₂, PO₄, SO₄.

** ICP-MS isotopes include: Cs-135, I-129, Np-237, Pd-107, Pu-239, Pu-240, Pu-242, Tc-99, Th-232, U-233, U-234, U-235, U-236, U-238.

*** Radionuclides include: Ag-110m, Am-241 (AEA), Am-241 (GEA), Am-243, Ce-144 (Pr-144), Cm-243 & Cm-244, Cm-242 (Am-242, Am-242m), Co-60, Cs-137 (Ba-137), Cs-134, Eu-152, Eu-154, Eu-155, Fe-55, Nb-93m, Pu-238, Pu-239 & Pu-240, Pu-241, Pu-242, Ru-106 (Rh-106), Sb-125 (Te-125m), Se-79, Sn-113 (In-113m), Sn-119m, Sn-121m, Sn-126 (Sb-126m, Sb-126), Sr-90 (Y-90), Zr-93 (Nb-93m).

Figure 3.3 Vendor Product Preparations and Analyses (Cont.)

* IC anions include: F, Cl, Br, C₂O₄, NO₂, NO₃, PO₄, SO₄.

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Figure 3.4 Original Material Preparations and Analyses

* IC anions include: F, Cl, Br, C₂O₄, NO₂, NO₃, PO₄, SO₄.

** ICP-MS isotopes include: Cs-135, I-129, Np-237, Pd-107, Pu-239, Pu-240, Pu-242, Tc-99, Th-232, U-233, U-234, U-235, U-236, U-238.

*** Radionuclides include: Ag-110m, Am-241 (AEA), Am-241 (GEA), Am-243, Ce-144 (Pr-144), Cm-243 & Cm-244, Cm-242 (Am-242, Am-242m), Co-60, Cs-137 (Ba-137), Cs-134, Eu-152, Eu-154, Eu-155, Fe-55, Nb-93m, Pu-238, Pu-239 & Pu-240, Pu-241, Pu-242, Ru-106 (Rh-106), Sb-125 (Te-125m), Se-79, Sn-113 (In-113m), Sn-119m, Sn-121m, Sn-126 (Sb-126m, Sb-126), Sr-90 (Y-90), Zr-93 (Nb-93m).

Figure 3.4 Original Material Preparations and Analyses (Cont.)

* IC anions include: F, Cl, Br, C₂O₄, NO₂, NO₃, PO₄, SO₄.

4.0 INORGANIC, RADIOCHEMICAL, AND PHYSICAL ANALYSIS

The results of the chemical, radionuclide and physical measurement performed on the Vendor Product and Original Material are provided in Tables 4.1 through 4.26. In nearly every case, the analytical results for the sample and duplicate for three samples (either Original Material or Vendor Product), a preparation blank, the average (with standard deviation), and a comparison to the Envelope D specification is reported. The tables are presented in the following order: Physical Measurements, ICP-AES, ICP-MS, Wet Chemistry, Radiochemistry, and Centrifuged Solids/Decant Liquid results. The ICP-AES and ICP-MS results are presented for multiple sample preparations (specifically, potassium hydroxide-potassium nitrate fusion, sodium peroxide-sodium chloride fusion, and nitric/hydrochloric acid digestion). Except where specifically noted, the sample/duplicate results have not been adjusted for the contribution of the preparation blank.

4.1 Evaluation Vendor Product to Envelope D Specification

For comparison of the Vendor Product to the Envelope D specification, the result selected is the "most reasonable" averaged result. For many analytes of interest, obtaining this averaged result required selecting a result from either ICP-AES or ICP-MS for one of three different analytical preparations. In selecting the most reasonable result, an effort has been made to evaluate the results based on the limitations of the analytical technique and the preparation method considering quality control performance.

Where the preparation methods are considered equivalent (for this matrix) and the measured value for the analyte is above the EQL, typically the highest reported result is used for comparison to the envelope specification. There are some instances where high preparation blanks may bias the results (e.g., calcium blank in fusion preparations), and in these cases, results from another preparation are selected for comparison. Where the ICP-AES reports results near the EQL, the results from the ICP-MS (which has better sensitivity than ICP-AES) may be selected since the precision is expected to be better. Attempts have been made to select appropriate results for comparison. However, for all analytes the average result from each preparation is compared to the Envelope D specification, and a "greater than specification" flag is provided in the ICP-AES, ICP-MS, Wet Chemistry, and Radiochemistry results tables to indicate when the specification has been exceeded (for the specific method and specific preparation).

4.1.1 Analytes/Radionuclides/Physical Properties Exceeding Specification

ICP-AES/ICP-MS (Sodium): As can be seen in the "most reasonable" ICP-AES results from acid digestion, the sodium concentration in the Vendor Product exceeds the Envelope D specification by about five percent. The ICP-MS sodium results from acid digestion is considerably high than the ICP-AES result; exceeding the Envelope D specification by 18%. The ICP-AES sodium results from the KOH fusion is at approximately 90% of the specification limit. Based on these analytical results, the Vendor Product can not be verified to be within the specification limit.

ICP-MS (Platinum and ²³³U): Based on the ICP-MS results, platinum (KOH fusion only) and ²³³U (acid digestion only) exceeded the Envelope D specification by factors of 1.2 and 66, respectively. Platinum is considered a trace component and ²³³U a trace radionuclide, and the specifications are near

the EQL/MDA. It should be noted that there was a large variation in the platinum results between the two fusion and acid digestion preparations. For platinum, the preparation blank exhibited some contamination.

Radiochemistry (^{121m}Sn): The measured value of ^{121m}Sn exceeds the specification limit by a factor of 6.

Physical Measurements: The weight percent solids and total oxide content exceed the LOI specification. The weight percent solids (14.4%) exceeds the requirement by 10% and the total oxide (126.5 g/L based on a slurry density of 1.10 g/ml) exceeds the LOI requirement by 26%. Also, the viscosity is below the lower limit at low shear rates. Compliance with these specifications was not a requirement for shipment of the Vendor Product material to the vendors.

4.1.2 Radionuclides/Analytes/Properties Unable to be Evaluated Against Specification

^{110m}Ag , ^{242}Pu , ^{119m}Sn : There are three cases where no activity was measured; however, the MDA (Minimum Detectable Activity) value exceeded the Envelope D limit; ^{110m}Ag by a factor of 349, ^{242}Pu by a factor of 10.9, and ^{119m}Sn by a factor of 155.

^{243}Cm , ^{144m}Pr : Since ^{243}Cm can not be resolved from ^{244}Cm (total activity of $3.13\text{E-}07\text{ Ci/L}$) and ^{144m}Pr can not be resolved from $^{144}\text{Ce}/^{144}\text{Pr}$ (MDA value of $<7.75\text{E-}5\text{ Ci/L}$), there exists some possibility that these radionuclides exceed the Envelope D specification. No estimate of the actual activities (other than that generated from ORIGEN) for these radionuclides can be made at this time.

^{113m}Cd : Although a method has been proposed, there is currently no approved PNNL method for determining this radionuclide. Therefore, per the Addendum 1 LOI Rev 0 (Jones 1996) no results are reported for this radionuclide. The ^{113m}Cd (half-life of 45 days) is expected to make up less than 0.0002% of the total activity.

^3H : Per the Addendum 1 LOI Rev 0 (Jones 1996) no attempt was made to measure tritium on the dried solids. Tritium (half-life of 12 years) is expected to make up less than 0.0002% of the total activity.

Sulfur: There is currently no PNNL analytical method for this analyte; therefore, no results are available.

Shear Strength: The shear strength is below detection limit, and the detection limit is above the specification.

4.1.3 Radionuclides/Analytes within 50% of the Specification

Five analytes have concentrations exceeding 50% of the Envelope D specification, and three radionuclides have MDA level exceeding 50% of the specification. These analytes/radionuclides are silver, erbium, iron, thorium, TOC, ^{144}Ce ($^{144}\text{Pr}/^{144\text{m}}\text{Pr}$), ^{152}Eu , and ^{106}Ru (^{106}Rh). Except for iron and TOC results, the reported concentrations or MDA values may not be sufficiently reliable as to validate that the analyte/radionuclide is not be present above the specification limit. However, the reported concentrations/MDAs are the "most reasonable" values available.

4.2 Physical Measurements — Vendor Product and Original Material

With the exception of SAL bulk weight percent solids determination on Vendor Product, all physical testing was performed by the Process Chemistry Group. Physical measurements on both the Original Material and Vendor Product samples included volume percent (Vol%) settled solids, volume percent centrifuged solids, weight percent (Wt%) centrifuged solids, slurry density (Original and Vendor), centrifuged solids density, centrifuged supernatant density, weight percent solids on drying and weight percent oxides on firing (Original Material, Vendor Product, and both centrifuged solids), and apparent viscosity as a function of shear rate, yield stress, settled solids shear strength after 2 days, and heat capacity. All analyses were performed in duplicate except for the Original Material and Vendor Product densities which were performed in quadruplicate. The bulk weight percent solids measured in the SAL are presented in Table 4.1 and the physical measurement (percent solids, percent oxides, and density) results from the HLRF are presented in Table 4.2. It is worth noting that there is good agreement between the Vendor Product weight percent solids (on drying) obtained in the HLRF and those obtained in the SAL.

Bulk Weight Percent Solids (Vendor Product Only): Table 4.1 presents the weight percent solids determinations performed on the Vendor Product subsample slurries by SAL at the time of drying the samples for subsequent chemical and radiochemical analysis. Weight percent solids were determined on each of the three Vendor Product subsamples prior to any analytical preparations. Since each sample was taken in its entirety for drying, only one weight percent solids determination could be made for each subsample. Weight percent solids determinations were not performed in the SAL on the Original Material subsample slurries.

Table 4.1 Vendor Product Bulk Weight Percent Solids Measurements

ACL Number	324 Identifier	Weight Percent Solids
96-06103	SPD1-C106-047	13.9
96-06104	SPD1-C106-048	16.9
96-06105	SPD1-C106-049	13.7
Average Bulk Wt% Solids		14.8
Standard Deviation		1.8

Table 4.2. Physical Measurements – Vendor Product and Original Material

Vendor Product Test	ACL Lab #	Sample	Duplicate	Average	RPD
Vol% Settled Solids	96-6653	17.5	19.5	18.5	10.8
Vol% Centrifuged Solids	96-6653	12.9	11.9	12.4	8.1
Wt% Centrifuged Solids	96-6653	20.3	19.5	19.9	4.0
Density (g/ml) Slurry Composite	96-6653	1.10 & 1.09	1.10 & 1.10	1.10	0.4 ⁽¹⁾
Density (g/ml) Centrifuged Solids	96-6653	1.72	1.81	1.77	5.1
Density (g/ml) Centrifuged Liquid	96-6653	0.99	1.00	1.00	0.6
Wt% Solids on Drying Slurry Composite	97-0108	14.2	14.6	14.4	2.5
Wt% Oxides on Firing Slurry Composite	97-0108	11.3	11.5	11.4	1.7
Wt% Solids on Drying Centrifuged Solids	96-6653	58.3	58.6	58.5	0.6
Wt% Oxides on Firing Centrifuged Solids	96-6653	49.5	50.0	49.8	1.0
Original Material Test	ACL Lab #	Sample	Duplicate	Average	RPD
Vol% Settled Solids	96-5945	40.5	43.0	41.8	6.0
Vol% Centrifuged Solids	96-5945	31.6	30.9	31.3	2.2
Wt% Centrifuged Solids	96-5945	43.1	43.6	43.4	1.2
Density (g/ml) Slurry Composite	95-5945	1.25 & 1.26	1.24 & 1.25	1.25	0.3 ⁽¹⁾
Density (g/ml) Centrifuged Solids	96-5945	1.71	1.76	1.74	2.9
Density (g/ml) Centrifuged Liquid	96-5945	1.07	1.06	1.07	0.9
Wt% Solids on Drying Slurry Composite	97-0109	28.8	29.6	29.2	2.9
Wt% Oxides on Firing Slurry Composite	97-0109	21.6	22.1	21.9	2.1
Wt% Solids on Drying Centrifuged Solids	96-5945	51.7	51.7	51.7	0.1
Wt% Oxides on Firing Centrifuged Solids	96-5945	33.7	34.9	34.3	3.5

⁽¹⁾Densities analyzed in quadruplicate; relative standard deviation provided instead of RPD.

Vol% Settled Solids, Vol% Centrifuged Solids, Wt% Centrifuged Solids: The volume percent (vol%) settled solids, volume percent (vol%) centrifuged solids, and the weight percent (wt%) centrifuged solids of the Vendor Product and Original Material reported in Table 4.2 were measured in duplicate according to technical procedure PNL-ALO-501 (with no deviations). The mass of the samples was measured on a calibrated balance and balance checks were performed during each batch. Volumes were measured using graduated centrifuge cones. These tests were conducted in conjunction with the density measurements for the slurries, centrifuged solids, and centrifuged supernatants.

Subsamples of the Vendor Product and Original Material slurries were centrifuged at high speed in the clinical centrifuge in the HLRF, and the centrifuged supernatant was removed to produce the centrifuged solids samples. At the appropriate times throughout the procedure, the volume percent and weight percent measurements were performed. The data is recorded in Test Instruction C106-2 and the settling of these samples was videotaped.

Densities: The density measurements were performed on the Vendor Product and Original Material slurry samples, as well as all associated centrifuged solids and supernatants, according to technical procedure PNL-ALO-501 (with no deviations). Subsamples of the Vendor Product and Original Material slurries were centrifuged at high speed in the clinical centrifuge in the HLRF, and the centrifuged supernatant was removed to produce the centrifuged solids and supernatant samples.

The densities of the Vendor Product and Original Material slurries were measured in quadruplicate, and the densities of the centrifuged solids and supernatant were measured in duplicate. The mass of the samples was measured on a calibrated balance and balance checks were performed during each batch. The volume was measured in graduated centrifuge cones. The accuracy of the graduations on these centrifuge cones was measured on a random sampling of this lot of centrifuge cones. The data is recorded in Test Instruction C106-2.

Wt% Solids & Wt% Oxides: Weight loss on drying (to obtain Wt% Solids) and firing (to obtain Wt% Oxides) was performed on the Vendor Product and Original Material slurry samples (97-0108 and 97-0109, respectively), as well as the associated centrifuged solids (96-6653 and 96-5945, respectively). Subsamples of the Vendor Product and Original Material slurries were centrifuged at high speed in the clinical centrifuge in the HLRF, and the centrifuged supernatant was removed to produce the centrifuged solid sample.

The weight percent solids and weight percent oxides were measured in duplicate according to technical procedure PNL-ALO-501 (with no deviation). The mass of each sample was measured on a calibrated balance and balance checks were performed during each batch. The data is recorded in Test Instruction C106-2.

The average solids loading for the Vendor Product is 14.4 weight percent, which exceeds the LOI requirement of 2.5 to 13 weight percent. The oxides loading of the Vendor Product based on the measured slurry density of 1.10 g/ml is 126.5 g/L, which is also above the LOI requirement of 25 to 100 g/L. Compliance with these specifications was not required for shipment of the sample to the vendor. However, attempts to lower the solids loading during the sodium-reduction treatment of the Vendor Product are detailed in Addendum 1 to this report (Urie 1997).

Shear Strength: The shear strength was performed on the settled solids resulting from the combination of the two 1L Vendor Product containers, SPD1-C106-051 and SPD1-C106-052, because there were insufficient solids in the sample originally assigned for this analysis (SPD1-C106-046). Sufficient quantity of solids from these jars were settled, separated from the overlying liquid, and transferred to a single 125 ml I-Chem jar.

Shear strength, τ_s , is a semiquantitative measure of the force required to move the sample. The sample was analyzed for shear strength using a Haake Rotovisco® viscometer modified for Hot-Cell operation with an M5 head and a shear vane manufactured at PNNL. The shear vane had a height of $H_v=1.588$ cm and a diameter of $D_v=0.800$ cm. The shear strength was then calculated according to Equation 1, where 4.9×10^5 dyne/cm is the maximum torque of the M5 head and $\% \tau/100$ is the ratio of the total torque which is recorded as full scale on the plot of the shear stress. The instrumental detection limit of 1000 dyne/cm² exceeds the technical specification of 20-200 dyne/cm². Due to the relatively high detection limit, it is not possible to determine whether the sample met the technical specification. The analysis was performed in case a measureable shear strength could be detected, thus validating that the specification was exceeded.

$$\tau_s = \frac{\frac{\% \tau}{100} \times S_r \times 4.90 \times 10^5}{\frac{\pi \times H_v \times D_v^2}{2} + \frac{\pi \times D_v^3}{6}} \quad (1)$$

The settled solids composite was analyzed in duplicate for shear strength at 29°C according to procedure number PNL-ALO-501. These solids were left undisturbed for two days prior to analysis. Following the shear strength analysis the settled solids from the shear strength analysis and remaining settled solids in bottle SPD1-C106-051 were combined, and subsequently subjected to further processing for sodium reduction (Wiemers 1996, Urie 1997). The decanted supernatant was bottled for archiving. A 51cP standard was analyzed prior to this sample and met the QC requirement of $\pm 10\%$. Both the sample and duplicate results were below the instrumental detection limit of 1000 dyne/cm².

Apparent Viscosity and Yield Stress: A 10 g subsample of the slurry from bottle SPD1-C106-051 was removed for shear stress verses shear rate analysis, as insufficient sample was available from the originally assigned sample, SPD1-C106-046. The subsample was analyzed in replicate (i.e., two individual subsamples were not obtained for analysis) for shear stress as a function of shear rate in at 25°C. Following the analysis, this material was discarded. At shear rates between 10 and 320 s⁻¹, the material displayed near Newtonian behavior. The viscosity of the material remained nearly constant at 1 to 3 cP in the range of 10 and 320 s⁻¹.

The samples were stirred with a spatula to combine the separated liquid and solid layers, and analyzed for shear stress as a function of shear rate using a Bohlin CS viscometer modified for glove box operation. Concentric cylinders with a 25 mm diameter inner cylinder and a 2.5 mm gap between the cylinders were the measuring geometry used with a C25 measuring sensor. Shear stress as a function of shear rate data was obtained by measuring the shear stress produced at a specific shear rate at 25°C.

The shear rate was gradually increased from 0 to 320 s^{-1} generating the increasing shear rate curve, and then back down to 0 s^{-1} generating the decreasing curve. The viscosity was then calculated by dividing the shear stress by the shear rate. Calibration checks were made with a certified 10 cP viscosity standard to ensure that the viscometer was operating properly. Technical Procedure PNL-ALO-501 was used to perform the data acquisition. During data interpretation a deviation was made to Technical Procedure PNL-ALO-501 in determination of the yield stress, the force sufficient to produce fluid behavior in the material. The Bohlin CS viscometer is a controlled stress instrument (versus as controlled rate instrument), therefore the yield stress was determined by plotting the shear stress versus shear rate for the increasing shear rate curve and extrapolating to zero shear rate.

The LOI specification for this material was a viscosity between 6 and 94 cP, 3 and 50 cP, and 1 and 50 cP at shear rates of 10, 25 and 183 s^{-1} , respectively, and a yield stress of between 0.1 and 15 Pa. At 10 s^{-1} , both sample and duplicate had a measured viscosity of 2.1 cP, which is below the minimum specification. At 25 s^{-1} , the sample results were 1.3 and 1.6 cP, which is also below the minimum specification. At 183 s^{-1} , the sample results were 1.1 and 1.0 cP, meeting the low end of the specification. A yield stress was observed, but was well below the quantification limit of 0.1 Pa, which is below the minimum specification. Compliance with these specifications was not required for shipment of the Vendor Product to the vendor. No attempt was made to adjust these properties within the specification.

Heat Capacity: A measure of the heat capacity of the Vendor Product slurry (SPD1-C106-046) was attempted using Differential Scanning Calorimetry (DSC). Heat capacity is a measure of the amount of energy required to produce a given rise in the temperature of the system. Due to several factors (described below), the measurement of the heat capacity of the slurry could not be performed by the DSC method. However, a measurement of the heat capacity of the dried Vendor Product slurry was obtained by this method.

The value for the heat capacity of the dried Vendor Product slurry (heated at 105°C until no further mass is lost) was 0.22 cal/g·°C from 50 to 105°C. The heat capacity of the dried sample is comparable with the heat capacity of NaNO_3 (0.26 cal/g·°C) and NaNO_2 (0.24 cal/g·°C). The dried vendor sample may retain water in other than "free" forms. Water will be removed in different amounts from Hanford tank waste samples from ambient temperatures to roughly 325°C. After drying at 105°C no free water will remain, but some adsorbed water and most of the hydrate water is still present. As long as no further loss of water occurs during the DSC run, the heat-capacity (C_p) value measured is valid since the heat capacity for many solid hydrates (expressed per gram) is not greatly different (commonly 10 to 20%) than for the anhydrous material. The heat capacities of solid hydrates and anhydrous materials are generally much smaller than the heat capacity for water.

A good estimate of the heat capacity of the Vendor Product slurry can be made by averaging the heat capacities per gram of the dried solid and water based on the wt% of each. This estimate provides a heat capacity of 0.89 cal/g·°C which is within the RFP specifications listed in Table TS-8.4 (0.79 - 0.97 cal/g·°C). This calculation is based on 14.4 wt% solids (See Table 4.1) and the reported heat capacity for water 1 cal/g·°C.

The heat capacity was measured on a Seiko Instruments DSC220 with the liquid nitrogen cooling capability. The heat capacity of the sample (C_{p_s}) is calculated according to equation 2 from the difference between the DSC curves of the empty container and sample (Y_s), the difference between the DSC curves of the container and the reference (Y_r), mass of the reference (M_r), mass of the sample (M_s), and heat capacity of the reference (C_{p_r}). The Specific Heat Capacity Calculation Software "DSCCP" supplied by Seiko Instruments Inc. was used to calculate the heat capacity of the sample over the range of the analysis.

$$C_{p_s} = \frac{Y_s}{Y_r} \times \frac{M_r}{M_s} \times C_{p_r} \quad (2)$$

The analysis was performed by performing a DSC analysis on the empty pan (baseline), placing the sapphire standard in that same pan and performing an equivalent analysis (reference), and finally replacing the sapphire with the sample at approximately the same mass of the reference and performing a third equivalent analysis (sample). The baseline is subtracted from both the reference and sample analyses, and the subtracted data is used to calculate the heat capacity.

Several attempts were made to measure the heat capacity of the Vendor Product slurry from 10°C above ambient temperature to 105°C. Because the mass of the sample must be known throughout the analysis and the sample contains water which vaporizes over the temperature range of the analysis, the sample must be placed in a sealed sample pan. A large endotherm from the vaporization of the water in the sample makes it difficult to measure the heat capacity of the sample, because the endothermic transition requires energy to vaporize the water which is not indicative of the heat capacity of the sample. The contribution from the vaporization of water is 540 times the estimated sample heat capacity. When the sample is sealed and water vaporizes, the pressure of the system increases. The heat required to produce a given rise in temperature is dependent upon the conditions under which the system is heated; therefore, heat capacity is generally reported at constant pressure. As the pressure is increasing the measured value of heat capacity is not the heat capacity at constant pressure which is generally reported; therefore, corrections need to be made based on the pressure in the system which is unknown for these samples.

The sample also generates its own heat which could introduce error into the measurement of heat capacity. A calculation of the energy generated (watts) from the sample indicates that this error is minimal for this sample (1×10^{-5} W/g of slurry or 2×10^{-7} W for a 20 mg slurry sample and 1×10^{-5} W for the 16.5 mg dried sample). The heat capacity was not determined for the Initial Composite Material (i.e., prior to diluting).

4.3 Chemical Analysis

4.3.1 Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES)

The three Vendor Product and three Original Material samples prepared in duplicate by three different sample preparation procedures (See Section 3) were analyzed by ICP-AES following method PNL-ALO-211 "Determination of Elements by Inductively Coupled Plasma Atomic Emission Spectrometry. Both the Vendor Product and Original Material were analyzed in the same manner. For instance, post digestion spikes were performed on fusion preparations, matrix spikes are included with the acid digestions, and all instrument QC results (e.g., continuing calibration verifications) were within acceptable limits for all reported results. The results from the ICP-AES analysis are reported for any analyte exceeding the instrument detection limit (IDL). Those results above the IDL but below the estimated quantitation limit (EQL) are reported in brackets "[]". All reported results have been adjusted for preparation and analytical dilution factors.

In general, the consistency of the results between the various dissolution techniques is very good, except for those analytes which exhibit poor dissolution by specific techniques (e.g., Si/Ti by acid digestion). One notable exception is silver, where the sodium peroxide fusion produced significantly higher results than the potassium hydroxide fusion or acid digestion preparation. However, the precision of the silver analysis was very poor.

4.3.1.1 ICP-AES - Vendor Product (Table 4.3 to Table 4.5)

The analyte concentrations found in the samples above 0.1% by weight are aluminum, iron, sodium, silicon, silver, calcium, magnesium, manganese, nickel, phosphorus, lead, titanium, and zirconium, and possibly potassium and uranium. For the fusions preparations, the relative percent difference (RPD) between duplicates are less than 20%, except for silver, calcium, lead, phosphorus, and zirconium. Whereas in the acid digestions, only silver, cadmium, and silicon demonstrate RPD values greater than 20%.

The contribution of the preparation blank to the potassium hydroxide fusion and acid digestion samples is negligible for all analytes, or the blank concentration is insignificant relative to the sample concentration. The exceptions are boron and copper, where the blank accounts for 20-30% of the sample concentration. However, the preparation blank for the sodium peroxide fusion demonstrated considerable contamination for calcium, copper, potassium, lithium, neodymium, nickel, phosphorus, and lead. This contamination is most likely caused from impurities in the sodium peroxide reagent or cross contamination during sample preparation. The ICP-AES analysis for these analytes from the sodium peroxide fusion should be used with caution. However, since these analytes did not exceed the specification, no additional actions (e.g., reanalysis) were taken.

From the acid digestion preparations, the silver matrix spike and blank spike recoveries are 12% and 47%, respectively; whereas all other spiked analytes recovered within acceptable limits (75% to 125%). Post spike recoveries for all preparations and all analytes are within acceptable limits (i.e., 75% to 125%). The reported silver value from the acid digestion is considered unreliable, and likely bias low because of precipitation of the silver with chloride present in the sample or from the hydrochloric acid used during preparation.

4.3.1.2 ICP-AES - Original Material (Table 4.6 to Table 4.8)

The analyte concentrations found in the Original Material above 0.1% by weight are aluminum, iron, sodium, silicon, silver, calcium, manganese, phosphorus, lead, and zirconium, and possibly tin and uranium. For the fusions preparations, the RPD between duplicates are less than 20%, except for silver. Whereas in the acid digestions, silver, boron, silicon, and zirconium demonstrate very poor precision (i.e., RPD values greater than 20%).

The contribution of the preparation blank for the potassium hydroxide fusion and acid digestion samples is negligible for all analytes, or the blank concentration is insignificant relative to the sample concentration. However, the preparation blank for the sodium peroxide fusion demonstrated considerable contamination for bismuth, calcium, cerium, copper, potassium, lithium, magnesium, neodymium, nickel, phosphorus, and lead. The ICP-AES analysis for these analytes from the sodium peroxide fusion are most probably bias high and should be used with caution.

Both the post spike recoveries and matrix spike recoveries for all analytes are within acceptable limits, except for silver. The silver matrix spike recovery is typically about 50% for the acid digestion process used. The silver matrix spike for the Original Material recovered at only 13% and, therefore, the silver results from the acid digestion are probably bias very low and are considered unreliable.

4.3.2 Inductively Coupled Plasma Mass Spectrometry (ICP-MS)

The three Vendor Product and three Original Material samples prepared in duplicate by three different sample preparation procedures (See Section 3) were analyzed by ICP-MS following methods PNL-ALO-280 "Inductively Coupled Plasma-Mass Spectrometer Analysis," PNL-ALO-281 "ICP-MS Determination of ^{99}Tc ," and PNL-ALO-282 "Determination of Uranium Concentration/Isotopic Composition Using ICP-MS." Analysis by ICP-MS were performed on one or more of the three preparations. Unlike ICP-AES, the ICP-MS reports only those results which are above the EQL.

4.3.2.1 ICP-MS - Vendor Product (Table 4.9 to Table 4.14)

The Vendor Product samples were analyzed before the Original Material samples. Multiple analyses of the Vendor Product samples were required for some analytes/isotopes of interest in order to obtain acceptable quality control results. Less repeat analyses were required for the Original Material after having increased knowledge based on the Vendor Product results. Analytes that were used for internal standards (lithium, beryllium, scandium, indium, and thallium) were confirmed not to be present in the samples in measurable concentrations. Isotope values that were calculated from sensitivities of other analytes include ^{240}Pu , ^{242}Pu , ^{135}Cs , ^{107}Pd , and ^{232}Th . As such, there is no calibration verification for these isotopes.

Quality control data indicate high uncertainties for ICP-MS analysis of calcium, sodium, selenium, zirconium, m/z 93 (niobium/zirconium), arsenic, rhodium, barium, and antimony. Matrix spike results for sodium, iron, aluminum, and calcium were unusable due to the relatively high sample concentrations (greater than five times that of spike) present for these analytes.

The m/z 93 results (the most contribution expected from niobium) may also include a contribution from zirconium. Although ^{93}Zr is not naturally occurring, for these samples there may be an interference from zirconium at m/z 93. Resolution of the niobium and zirconium is not complete. The same interference problem is experienced also at m/z 241 with plutonium and americium. Therefore, only one value is reported for m/z 93 and m/z 241; possible/probable isotopes contributing to the response observed at these masses are zirconium/niobium and plutonium/americium, respectively.

There does appear to be a small peak at m/z 233 which indicates the presence of ^{233}U . However, this peak may be attributed to a hydride of ^{232}Th , which is present in a relatively high concentration.

No modifications were performed to either the fusions or acid digestions to assure the ^{129}I remains in solution and not lost as I_2 . Since iodine was not spiked prior to initiating the preparative process, the reported ICP-MS results for ^{129}I may be biased.

Additional information on general observations made during ICP-MS analyses and analyte/isotope data qualifications is included in Appendix A.

4.3.2.2 ICP-MS - Original Material (Table 4.15 to Table 4.20)

In general, analyses of the Original Material met with fewer problems, required fewer re-runs, and exhibited more consistency in QC performance than that of the Vendor Product. This was primarily due to the experience gained in analyzing the Vendor Product material. Each sample preparation (KOH fusion, acid digestion, and Na_2O_2 fusion) was analyzed separately. The samples were analyzed at three different dilutions to obtain valid data for the wide range of analyte concentrations observed. In general, the same data qualifications apply to the Original Material results as to the Vendor Product results (See Section 4.3.2.1 and Appendix A).

4.3.3 Ion Chromatography (IC) for Anions (Table 4.21 and Table 4.22)

The IC analyses were performed in duplicate on each of the three Vendor Product and three Original Material samples following leaching with water. The Vendor Product and Original Material were analyzed as a single batch following method PNL-ALO-212 "Determination of Inorganic Anions by Ion Chromatography." Besides the routine suite of anions (i.e., F, Cl, Br, NO_2^- , NO_3^- , SO_4^{2-} , and PO_4^{3-}), oxalate was also determined on both materials by this method.

In general, the RPD values were within acceptable limits, except for those results at or near the lowest calibration standard concentration. However, the excessively high oxalate is suspected of contributing to the higher than normal RPDs. Most anions were measured near the lowest calibration standard due to the high dilutions required because of the 15% to 20% oxalate present. The preparation blank exhibited significant concentrations of chloride, nitrate and sulfate; with the chloride and nitrate being well above 5% of the sample concentrations. However, since these analytes were below the specification, no further actions were taken.

The blank spikes and matrix spikes associated with the Original Material water leach preparation recovered within the acceptance limits of $\pm 20\%$. The blank spike associated the Vendor Product water

leach preparation was also within acceptance limits; however, fluoride, nitrite, and phosphate spike recoveries failed for the Vendor Product matrix spike. The fluoride recovery (at 144%) appears to be bias high due to matrix interferences; these interferences also result in higher reportable detection limits. The nitrite and phosphate spiking levels were less than 25% of the sample concentration, thus the calculated recoveries are not meaningful. Since these analytes were below the specification, no further actions were taken.

4.3.4 Ion Specific Electrode for Ammonia (Table 4.21 and Table 4.22)

Ammonia analyses were performed in duplicate on each of the three Vendor Product and three Original Material samples following leaching with water. The Vendor Product and Original Material were analyzed as separate batches following method PNL-ALO-226 "Ammonia (Nitrogen) in aqueous Samples." Immediately following the water leach preparation, the leached samples were acidified to stabilize the ammonia, then removed from the SAL and analyzed in the laboratory. For all samples analyzed, the ammonia level was below the linear portion of the calibration curve, thus the results are anticipated to have a high uncertainty. The preparation blank concentrations are basically the same as the sample concentration, thus indicating that there is little if any ammonia in the water leached samples.

The RPDs for the sample/duplicate sets were poor; however, this is expected at the low concentrations measured. Also, except for one laboratory double spiking problem, the blank spikes and matrix spikes recovered within acceptable limits. The ammonia method requires post spiking of all samples; the post spikes for the Vendor Product and Original Material samples ranged from 87% to 119%.

4.3.5 Cold Vapor Atomic Absorption for Mercury (Table 4.21 and Table 4.22)

To obtain mercury results on the dried slurries, small (0.04g) subsamples of the dried materials were weighed in the SAL and distributed to the Inorganic Group for cold vapor atomic absorption (CVAA) analysis. Due to analytical instrumentation problems during the analysis of the initial set of samples, an additional set of samples were weighed in the SAL and distributed to the laboratory. Provisions were made for full QC to be applied at the time of analysis (e.g., samples weighed for subsequent laboratory spike additions). The CVAA Mercury analyses were performed in duplicate on each of the three Vendor Product and three Original Material samples. The Vendor Product and Original Material were analyzed as a single batch following Method PNL-ALO-131 for digestion and PNL-ALO-201 for analysis.

Due to the high concentration of mercury in the samples (relative to other Hanford tank materials), the matrix spike could not be recovered (i.e., the matrix spike concentration was less than 20% of the sample concentration). The RPD values for all the samples were within acceptance limits of 20%, and no mercury was detected in the preparation blank. It should be noted, however, that the continuing calibration verification standards recovered at 82% to 85% which is just within the lower acceptance limit of 80%. This suggests that the instrument results may be bias slightly low for all the sample results. However, if the results are adjusted for recovery of both the Vendor Product and the Original Material, the results are well within the Envelope D specification.

4.3.6 Cyanide (Table 4.21 and Table 4.22)

Total cyanide analyses were performed in duplicate on each of the three Vendor Product and three Original Material samples. The Vendor Product and Original Material were analyzed as a single batch. An attempt was made to perform a remote cyanide analysis (PNL-ALO-287 "Midi and Micro Distillation of Cyanide in Liquid and Solid Samples") on the dried slurry materials. The samples became very effervescent with the addition of digesting acid, $2M$ H_2SO_4 . Enough gas was generated to breach the membrane separating the digesting sample from the NaOH trapping solution (HCN distillate). Additionally the samples turned black in color when the acid was applied, and the batch QC failed. Sample sizes were reduced to about 40 milligrams and subsamples distributed to the Inorganic Group for a repeat of the same technique in a fume hood environment; the results were similar. A third set of samples were then weighed and distributed to the Inorganic Group, and an alternate CN method utilizing a predissolution step and larger distillation tubes (to allow more room for gas expansion during digestion) was performed. This analysis yielded acceptable QC and sample performance. The samples were predigested in an EDTA and ethylenediamine solution prior to distillation, following procedure PNL-ALO-285 "Total Cyanide by Remote Microdistillation and Argentometric Titration." After the predigestion, the distillates were analyzed following method PNL-ALO-289 "Total Cyanide Determination with Spectrophotometry (Manual or Automated)." All batch QC and instrument QC are within acceptable limits. The matrix spike ranged in recovery from 98% to 122%, and the RPD between duplicates ranged from 1% to 19%.

4.2.7 Total Carbon - Inorganic and Organic Carbon (Table 4.21 and Table 4.22)

Total carbon analyses were performed in duplicate in the SAL directly on each of the three Vendor Product and three Original Material samples by the hot persulfate wet oxidation method PNL-ALO-381 "Determination of TC, TOC, and TIC in Radioactive Liquids, Soils, and Sludges." The dried samples and duplicates were acid digested, swept with a pure oxygen stream and the resulting carbon dioxide routed through the coulometry unit of the analyzer. Per the procedure, all sample results have been corrected for the carbon system blank and the control standard recovery. The TIC and TOC are measured directly and the TC is determined by summing the TIC and TOC. Quality Control for the method involves instrumental blanks for TIC and TOC, inorganic and organic standards analysis, and matrix spiked samples.

For the Vendor Product analysis, all batch QC and analytical QC were acceptable, except for one RPD which calculated at 25%; slightly outside the acceptance limit of 20%. For the Original Material, all analytical QC and batch QC were acceptable. However, the range demonstrated by the control standard (i.e., 82% to 111%) is considerably greater than typically experienced. Although the control standards varied more than normal, the sample RPD range (0.5% to 25%) is typical for the matrix type. The oxalate determined by IC analysis accounts for nearly 100% of the TOC.

4.4 Radiochemistry Analysis

4.4.1 Radiochemistry - Vendor Product (Table 4.23)

The Vendor Product radiochemistry analyses were performed on the KOH fusion preparations, unless otherwise noted. Small aliquots of these prepared solutions were then used for the radiochemical analyses. All of the Vendor Product radiochemical results are reported in $\mu\text{Ci/g}$ dried solids.

Gamma Energy Analyses (GEA): Aliquots of the prepared samples were directly gamma counted following procedure PNL-ALO-450. ^{137}Cs was the dominant gamma emitter. However, ^{60}Co , ^{154}Eu , ^{155}Eu , and ^{241}Am were also detected. None of the other gamma emitters of interest (i.e., ^{106}Ru (^{106}Rh), $^{110\text{m}}\text{Ag}$, ^{125}Sb ($^{125\text{m}}\text{Te}$), ^{134}Cs , ^{144}Ce ($^{144\text{m}}\text{Pr}$), and ^{152}Eu) were detected and MDA values are reported. In some cases, the measured MDA values are orders of magnitude higher than requested. The requested MDA values cannot be obtained due to the very strong interference of Compton background from ^{137}Cs . Hence, it would be necessary to chemically separate out the cesium in order to achieve lower detection limits.

The RPD values show good agreement between the samples and duplicates in cases where reasonable counting statistics were achieved. It should also be noted that the three Vendor Product sample splits also show good agreement. There are three cases where the RPD values exceeded the required 20% limits; ^{241}Am on sample 96-6103 and ^{241}Am and ^{155}Eu on sample 96-6104. In these cases, the counting uncertainties for both the sample and the duplicate are very high; however, the mean difference (MD) values indicate with high confidence that the results are statistically the same. The preparation blank showed only weak ^{137}Cs activity.

Total Alpha: The total alpha activities were determined following procedures PNL-ALO-420/421. All of the sample analyses give similar results with RPD values below 10%. Weak alpha activity was detected in the preparation blank; however, the value is a factor of about 1,000 less than the samples. No alpha activity was detected in the laboratory blank. Standard and sample spike recoveries were 106% and 113%, respectively.

Total Beta: The total beta activities were determined following procedures PNL-ALO-430/431. All of the sample analyses give similar results with RPD values below 11%. Weak beta activity was detected in the preparation blank; however, the value is a factor of 10,000 less than the samples. No beta activity was detected in the lab blank. Standard and sample spike recoveries were 97% and 101%, respectively.

Americium/Curium: The americium and curium isotopes were determined together because they have identical chemical behavior. Analysis proceeded according to procedures PNL-ALO-417/496/422. Sample-specific radiochemical yields/counting efficiencies were determined relative to ^{243}Am tracer.

In order to determine the ^{243}Am activity, a separate aliquot was processed and the indigenous ^{241}Am was used to determine radiochemical yield/counting efficiency, applying the ^{241}Am concentration measured initially. No ^{243}Am was found in the samples so the original premise of using the ^{243}Am to trace yields is valid. The sample-specific ^{243}Am detection limit is driven by the tailing of the ^{241}Am alpha peak into the ^{243}Am alpha energy region.

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The radiochemical yields for the sample preparations were nominally 85%. The RPD values for ^{241}Am activity were below 4%. The $^{243,244}\text{Cm}$ reproducibility was not as tight as that of ^{241}Am with sample 96-6104 at 20.7% and sample 96-6105 at 25.6% RPD. The ^{242}Cm RPD values were also rather high at 31.9% (96-6103) and 23.7% (96-6104) however, the errors associated with the results were also high at nominally 20%. In all of these cases, the MD is less than 1.5 indicating that the results are consistent within the analytical uncertainties. The preparation blank activity for the americium/curium isotopes was well below that of the samples. The highest preparation blank activity is for $^{243,244}\text{Cm}$ which represents 2% of the sample activity for sample 96-6104. The matrix spike and blank spike yield-corrected recoveries were 93% and 100% respectively.

Carbon-14: Radiocarbon determinations were performed in the SAL directly on Vendor Product sample per method PNL-ALO-482 "Determination of Carbon-14 in Radioactive Liquids, Soils, and Sludges." The in-cell UICTM analyzer module was used to digest the samples and sweep the carbon dioxide to a caustic trap located in a fume hood outside the hot cell. The individual sample trap solutions were then combined with scintillation cocktail and counted using a Liquid Scintillation Counter. The QC for this method involves instrumental blanks, analysis of a ^{14}C standard and matrix spiked with the ^{14}C standard material. The reproducibility of the ^{14}C analyses between samples and duplicate was very poor, due to carryover from previously analyzed high standards. Although the matrix spike recoveries are good, the extremely poor sample reproducibility makes the reported results suspect. Even at the highest ^{14}C result, the ^{14}C concentration is only about 25% of the Envelope D specification; therefore, no additional actions were taken. However, the reason for the carryover from preceding standards is being evaluated.

Cesium Isotopes: The ^{134}Cs and ^{137}Cs activities were measured by GEA. In order to further obtain the activities of ^{135}Cs , which has a very long half-life of 2.3E6 years, the cesium was chemically separated, and then measured by thermal ionization mass spectrometry (TIMS) following procedure PNL-ALO-452. The activities of ^{135}Cs were then calculated from the ^{137}Cs activities, measured by GEA, and the $^{135}\text{Cs}/^{137}\text{Cs}$ mass ratios, measured by TIMS. The results indicate nearly the same activity of ^{135}Cs in all of the samples and RPD values are all less than 5%. The ^{134}Cs could not be detected either by GEA or TIMS measurements and MDA values were reported for the GEA results. The TIMS measurements also detected stable ^{133}Cs . Although this isotope was not requested, concentration values are provided for information. Since cesium only has one stable isotope (^{133}Cs), no appropriate TIMS standards are available. A rubidium standard was thus analyzed instead. The measured ratio of $^{85}\text{Rb}/^{87}\text{Rb}$ was 2.59, in excellent agreement with the natural isotope ratio of 2.59.

Fe-55: An iron chemical separation was performed following procedures PNL-ALO-435/450. Stable iron was used to trace the yield. A minor deviation from procedure PNL-ALO-435 was performed to obtain radiochemical yields from stable iron. The iron hydroxide precipitate was dissolved and an aliquot of solution was analyzed by ICP-AES for the iron content. The radiochemical yields averaged 70%.

No ^{55}Fe was detected in either the hot cell preparation blank or the lab blank. The blank spike and sample matrix spike yield-corrected recoveries were 115% and 86%, respectively. The samples demonstrated good agreement with RPD values below 8%.

Ni-59 and Ni-63: The three Vendor Product samples (including duplicates) from the sodium peroxide fusion process were analyzed for ^{59}Ni and ^{63}Ni . Procedure PNL-ALO-495 was used for the nickel purification, PNL-ALO-450 was used for the low-energy photon (LEP) counting, and PNL-ALO-474 was used for beta counting (liquid scintillation). Minor changes were made to the nickel separation procedure. A nickel-specific column from Eichrom Industries was used in place of the house-made columns. The Eichrom column functions with the same chemical principals and would not affect data quality. Another change is the treatment of the nickel-dimethylglyoxime precipitate on the filter. This precipitate was dissolved using 5 mL of 3 M HCl and an aliquot taken for yield determination; another aliquot was taken to dryness, 0.5 mL water added and 10-mL Ultima Gold added for subsequent liquid scintillation counting. This is a quality improvement in that there is no potential for nickel to be lost due to potential volatilization of the $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$.

Radiochemical yields were generally high, nominally 90%. The hot cell preparation blank resulted in no detectable activity for either ^{59}Ni or ^{63}Ni . The ^{59}Ni LEP spectra and the ^{63}Ni beta spectra were visually clean from other interferences. The ^{59}Ni activity is barely detectable in some of the samples, at about $2\text{E}-2 \mu\text{Ci/g}$. This is virtually at the MDA. The ^{63}Ni concentration is consistent throughout the sample set at nominally $2.4 \mu\text{Ci/g}$. The matrix spike samples were spiked with ^{63}Ni , since ^{59}Ni is not commercially available. The blank spike, blank spike duplicate, and matrix spike recoveries were excellent at 95%, 96% and 101%, respectively.

Nb-93m: The niobium radiochemical separation from other fission products was conducted according to PNL-ALO-4003. (This procedure is currently in the review/approval process.) The final sample preparations were counted by both GEA and LEP according to PNL-ALO-450. Radiochemical yields were traced with ^{93}Nb and averaged about 70%. It appears that ^{125}Sb carried through with the niobium fraction, however this isotope did not adversely interfere with the 16.6 keV photon of ^{93}Nb . The blank spike recovery is low at 71%; the reason for the low recovery is not known. The matrix spike recovery is acceptable at 84%. The RPD values are high at 30% to 79%; however, the MD shows that within the error of the measurement, the results are consistent.

Plutonium: Plutonium isotopes were separated in sequence after the americium/curium elution according to PNL-ALO-417. One minor modification was made to the procedure to effect this separation/elution. The plutonium was reduced to a +3 oxidation state using TiCl_3 solution in 4M HCl. This is a more effective and faster way to elute the plutonium fraction. The Pu fraction then was precipitation-plated directly according to PNL-ALO-496. Alpha spectrometry proceeded according to PNL-ALO-422. The plutonium radiochemical yield/counting efficiency was traced with ^{242}Pu .

In order to determine the ^{242}Pu activity, a separate aliquot was processed without added tracer. The indigenous ^{239}Pu in each sample was used to trace the yield. The ^{239}Pu activities were determined using the results of the ^{242}Pu -traced aliquot. No ^{242}Pu was found in the samples so the original premise of using the ^{242}Pu to trace yields is valid. The sample-specific ^{242}Pu detection limit is driven by the tailing of the ^{239}Pu alpha peak into the ^{242}Pu alpha energy region.

The radiochemical yields for the plutonium fractions were nominally 85%. The sample precision for $^{239+240}\text{Pu}$ and ^{238}Pu , as determined by the RPD, is 9.6% or better. The preparation blank $^{239+240}\text{Pu}$

activity is less than a factor of 1000 relative to the sample activity. The preparation blank ^{238}Pu activity represents less than 0.5% of the sample activity.

Overall, the sum of the plutonium and americium/curium data is in excellent agreement with the total alpha results.

Plutonium-241: The sample preparations for plutonium were also used for the subsequent beta analysis of ^{241}Pu ; a draft procedure was used to perform the ^{241}Pu analysis. The precipitation-plated filter samples were placed in liquid scintillation vials, 10 ml of Ultima Gold were added and the samples were counted by liquid scintillation counting (LSC) following procedure PNL-ALO-474.

The LSC spectra showed both the alpha-peak activity (^{238}Pu , $^{239+240}\text{Pu}$, and ^{242}Pu) and a well-separated beta peak at low energies due to ^{241}Pu . The samples and duplicates showed good agreement at a nominal activity level of 7 $\mu\text{Ci/g}$. The preparation blank and laboratory blank activities were well below this level; i.e., at 0.03 $\mu\text{Ci/g}$ or less. The blank spike and matrix spike samples were spiked at the time the filter was placed in the LSC vial. The spike recoveries are high at 146% and 182%, respectively. The blank spike preparation was repeated in duplicate with spike recoveries of 111 and 94%. The reason for the high spike recoveries is not clear; no chemistry was involved in the matrix spike sample preparation.

The ^{241}Pu activities were also calculated independently by using the plutonium alpha activities as an internal tracer. The ^{241}Pu activities calculated by this method are in good agreement with the beta calculations and the mean activity values agree within 15%.

Sm-151 and Pm-147: The $^{151}\text{Sm}/^{147}\text{Pm}$ screen/determinations were made on the final sample preparations for ^{241}Am analysis. As rare earths, the samarium and promethium radiochemically follow the other rare earths and the Am^{-3} using the TRU-Spec column chemistry (PNL-ALO-417). The samarium and promethium would also be precipitation-plated (PNL-ALO-496), presumably as efficiently as the americium. Thus the radiochemical yield determined for Am is used to correct for the radiochemical yield of Sm/Pm. It would have been better to utilize blank and matrix spikes to verify the theory, however the request for ^{151}Sm analysis was made too late (after the Am separation) to utilize the ^{151}Sm spike in these analyses.

The precipitation-plated americium preparation was simply placed into LSC cocktail and counted according to PNL-ALO-474 by liquid scintillation. For instrument calibration, ^{63}Ni was used because its endpoint energy is similar to that of the ^{151}Sm . The ^{147}Pm has a 224 keV E_{max} which tails under the ^{151}Sm peak at 76 keV E_{max} ; thus a summation of ^{151}Sm and ^{147}Pm is provided. The resulting beta spectra indicated a large peak due to the ^{151}Sm ; however, another peak (most probably ^{147}Pm) tailed under the samarium peak. The alpha peak associated with americium/curium was also evident. The hot cell blank and laboratory blank resulted in a small amount of beta activity relative to the calibration blank; however, their activities are about 3000 times lower than that of the samples. The propagated error associated with these measurements is nominally 4%. A measure of bias cannot be determined without blank spike/matrix spike data.

Along with the rare earths samarium and promethium, europium should also appear in this fraction. The samples were counted by GEA to evaluate the ^{154}Eu and ^{155}Eu activities; however, none was

detected. The absence of the europium isotopes is not understood at this time. The ^{154}Eu and ^{155}Eu concentrations in the Am-preparation were determined based on the GEA, and the dilution and yield chemistries applied to the samples should have resulted in a detectable quantity of both ^{154}Eu and ^{155}Eu . Because they were not detected, no beta interference for ^{154}Eu and ^{155}Eu was present. Had europium been present at the anticipated levels, its contribution would have amounted to nominally 5% of the beta activity in the 2-90 keV region.

This method must be considered a screening method for $^{151}\text{Sm}/^{147}\text{Pm}$ because the radiochemical separation from europium had not been performed and the radiochemical recovery is based solely on the americium recovery. Europium did not recover as well as the americium, indicating that samarium/promethium may have low recovery also.

Se-79: The ^{79}Se activities were determined following procedures PNL-ALO-440/474. The separation procedure involves an anion/cation exchange to remove most radiochemical interferences followed by a selenium bromide distillation. Since ^{79}Se is not available as a standard, it was not possible to measure standard or sample spike recoveries. Natural selenium metal was added to each sample and the recovered selenium precipitate was used to determine the gravimetric yields.

Acid Digestion: Radiochemical yields were nominally 60% and all known contaminants were successfully removed, as determined by beta spectral analysis. No ^{79}Se beta activity was seen in either the samples or the blanks above the calibration blank background counting rate. Hence, MDA values are reported.

KOH Fusion: The radiochemical yields of Se averaged about 50%. No ^{79}Se beta activity was seen in either the samples or the blanks above the calibration blank background counting rate. Hence, MDA values are reported.

Sr-90: The ^{90}Sr activities were determined following procedure PNL-ALO-476/431. Radiochemical yields, traced with ^{85}Sr , were excellent averaging 90%. All of the sample analyses gave similar results with RPD values below 19%. Weak ^{90}Sr activity was detected in the preparation blank and the lab blank; however, the values are a factor of about 10,000 less than the activities in the samples. Standard and sample spike recoveries were 93% and 92%, respectively.

Sn Isotopes: Analyses for ^{113}Sn , $^{119\text{m}}\text{Sn}$, $^{121\text{m}}\text{Sn}$, and ^{126}Sn were conducted according to procedure PNL-ALO-453/450. A modification was made to the separation procedure to accommodate $^{119\text{m}}\text{Sn}$ and $^{121\text{m}}\text{Sn}$ analysis. The final sample mount was a thin $\text{Sn}(\text{OH})_4$ precipitate enabling the low-energy photons to be analyzed. The stable tin addition had the additional advantage to be used as a radiochemical yield monitor.

Radiochemical yields were nominally 50%. Purity with respect to other gamma emitters was excellent. The ^{126}Sn is reported in conjunction with ^{126}Sb and $^{126\text{m}}\text{Sb}$ because these isotopes quickly obtain secular equilibrium. The same reasoning applies to ^{113}Sn and $^{113\text{m}}\text{In}$. Only ^{126}Sn (and $^{126}/^{126\text{m}}\text{Sb}$) and $^{121\text{m}}\text{Sn}$ were found in the samples. The sample and duplicate values were significantly different in some cases, resulting in high RPD values. However, except for one sample pair, the MD test shows that, within the error of the method, the results are consistent. Sample 96-6105-Dup (KOH fusion) appears to be very different from the other samples in the set. The most likely explanation is that the

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yield determination failed with respect to this sample. A replicate analysis was performed on this sample as part of the analytical batch and its results are consistent with the other samples. The result for the replicate sample (96-6105D) is reported in Table 4.21.

At the time of analysis, a tin isotope was not available for spiking a blank spike or matrix spike. A ^{113}Sn isotope was ordered and used for the subsequent analysis of the Original Material. The reagent blank and hot cell preparation blank resulted in no detectable activity for these isotopes.

Zr-93: The ^{93}Zr analyses were performed according to draft procedure PNL-ALO-4007. No ^{93}Zr activity was detected in the Vendor Product samples, and MDA values are reported. The samples were analyzed using a separation procedure modified to improve the zirconium carrier recovery, increase the zirconium/niobium separation, and to utilize beta-counting for the final ^{93}Zr determination. In summary, stable zirconium was added as a yield monitor and the samples were passed through two anion exchange columns under varying conditions to separate impurities. The carrier recovery on these samples was reasonably high, but gave only about 80% decontamination from plutonium, and consequently, the ^{241}Pu beta caused the ^{93}Zr count rate to be biased considerably high. Probably over half the total counts in the ^{93}Zr region are from ^{241}Pu . No contaminant other than plutonium is discernable in the ^{93}Zr beta spectrum. The actual ^{93}Zr activity in each of these samples is probably less than half the MDA value reported. The MDA values were conservatively estimated from the total beta count rates in the region of the ^{93}Zr activity.

4.4.2 Radiochemistry - Original Material (Table 4.24)

The Original Material radiochemistry analyses were performed on KOH fusion preparations, unless otherwise noted. Small aliquots of these prepared solutions were then used for the radiochemical analyses. All of the radiochemical results are reported in $\mu\text{Ci/g}$ dried solids.

Gamma Energy Analyses: Aliquots of the prepared sample were directly gamma counted following procedure PNL-ALO-450. ^{137}Cs was the dominant gamma emitter. However, ^{60}Co , ^{154}Eu , ^{155}Eu , and ^{241}Am were also detected. None of the other gamma emitters of interest (i.e., ^{106}Ru (^{106}Rh), ^{110m}Ag , ^{125}Sb (^{125m}Te), ^{134}Cs , ^{144}Ce (^{144}Pr , ^{144m}Pr), and ^{152}Eu) were detected and MDA values are reported. In some cases, the measured MDA values are orders of magnitude higher than requested. The requested MDA values cannot be obtained due to the very strong interference of Compton background from ^{137}Cs . Hence, it would be necessary to chemically separate out the cesium in order to achieve lower detection limits.

The RPD values show good agreement between the samples and duplicates in cases where reasonable counting statistics were achieved. It should also be noted that the three Original Material splits also show good agreement. There are three cases where the RPD values exceeded the required 20% limits; ^{155}Eu on sample 96-5892, ^{241}Am on sample 96-5893, and ^{155}Eu on sample 96-5894. In these cases, the counting uncertainties are so high that MD values indicate high confidence that the results are statistically acceptable. The preparation blank showed only weak ^{137}Cs activity.

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Total Alpha: The total alpha activities were determined following procedures PNL-ALO-420/421. All of the sample analyses gave similar results with RPD values below 6%. No alpha activity was detected in either the preparation blank or the laboratory blank. Standard and sample spike recoveries were 110% and 113%, respectively.

Total Beta: The total beta activities were determined following procedures PNL-ALO-430/431. All of the sample analyses give similar results with RPD values below 10%. Weak beta activity was detected in the preparation blank; however, the value is a factor of 10,000 less than the samples. No beta activity was detected in the laboratory blank. Standard and sample spike recoveries were 96% and 94%, respectively.

Americium/Curium: The americium and curium isotopes were determined together because they chemically behave identically. Analytical separation proceeded according to PNL-ALO-417/496/422. Sample-specific radiochemical yields/counting efficiency were determined relative to ^{243}Am tracer.

In order to determine the ^{243}Am activity, a separate aliquot was processed and the indigenous ^{241}Am was used to determine radiochemical yield/counting efficiency, applying the ^{241}Am concentration after the initial determination. No ^{243}Am was found in the samples so the original premise of using the ^{243}Am to trace yields is valid. The sample-specific ^{243}Am detection limit is driven by the tailing of the ^{241}Am alpha peak into the ^{243}Am alpha energy region.

The radiochemical yields for the sample preparations were good at nominally 75%. The RPD values in the calculated ^{241}Am activity were better than 13%. The $^{243-244}\text{Cm}$ and ^{242}Cm reproducibilities were not as tight as that of ^{241}Am with some RPD values at 93% and 46%. Sample 96-5894 resulted in 1.6 times the concentration of $^{243/244}\text{Cm}$ found in the rest of the sample set. This higher activity was confirmed with the untraced americium sample. The errors associated with the ^{242}Cm results were high, reaching to 41% due to poor counting statistics. Although the RPD values were not below 20% on all samples, the MD is below the threshold of 1.96. The preparation blank activity for the Am/Cm isotopes was well below that of the samples. The worst-case effect from the preparation blank $^{243-244}\text{Cm}$ activity is for sample 96-5893 where it represents 7% of the sample activity. The matrix spike and blank spike yield-corrected recoveries ranged from 95% to 106%.

Carbon-14: Radiocarbon determinations were performed in the SAL directly on Original Material sample per method PNL-ALO-482 "Determination of Carbon-14 in Radioactive Liquids, Soils, and Sludges." The in-cell UICTM analyzer module was used to digest the samples and sweep the carbon dioxide to a caustic trap located in a fume hood outside the hot cell. The individual sample trap solutions were then combined with scintillation cocktail and counted using a Liquid Scintillation Counter. The QC for this method involves instrumental blanks, analysis of a ^{14}C standard and matrix spiked with the ^{14}C standard material. The reproducibility of the ^{14}C analyses between samples and duplicate was very poor, due to carryover from previously analyzed high standards. Although the matrix spike recoveries are good, the extremely poor sample reproducibility makes the reported results suspect. The reason for the carryover from high standards is being evaluated.

Cesium Isotopes: The ^{134}Cs and ^{137}Cs activities were measured by GEA. In order to further obtain the activities of ^{135}Cs , which has a very long half-life of 2.3E6 years, the cesium was chemically separated and then measured by TIMS, following procedure PNL-ALO-452. The activities of ^{135}Cs were then

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calculated from the ^{137}Cs activities, measured by GEA, and the $^{135}\text{Cs}/^{137}\text{Cs}$ mass ratios, measured by TIMS. The results indicate nearly the same activity of ^{135}Cs in all of the samples and RPD values are all less than 8%. Cesium-134 could not be detected either by GEA or TIMS, and the GEA MDA values are reported for ^{134}Cs . The TIMS measurements also detected stable ^{133}Cs . Although this isotope was not requested, concentration values are provided for information only. Since cesium only has one stable isotope, no appropriate mass spectrometry standards are available. A rubidium standard was thus analyzed instead. The measured ratio of $^{85}\text{Rb}/^{87}\text{Rb}$ was 2.61, in good agreement with the natural isotope ratio of 2.59.

Fe-55: The iron separations were conducted according to PNL-ALO-435/450. Stable iron was used to trace yield. A minor deviation from procedure PNL-ALO-435 was performed to obtain radiochemical yields from stable iron. The iron hydroxide precipitate was dissolved and an aliquot of solution was analyzed by ICP-AES for the iron content. The radiochemical yields averaged 77%.

No ^{55}Fe was detected in either the hot cell preparation blank or the lab preparation blank. The blank spike and sample matrix spike yield-corrected ^{55}Fe recoveries are 92% and 97%, respectively. The samples demonstrated a fair consistency between the three Original Material samples. One sample, 96-5894/96-5894Dup, resulted in an RPD of 30%; however, the MD is 0.98 indicating these samples are statistically equal, given the error of the measurements.

Ni-59 and Ni-63: The three Original Material samples and duplicates from the sodium peroxide fusion process were analyzed for ^{59}Ni and ^{63}Ni . Procedure PNL-ALO-495 was used for the nickel purification, PNL-ALO-450 was used for the LEP counting, and PNL-ALO-474 was used for beta counting (i.e., liquid scintillation). Minor changes were made to procedure PNL-ALO-495. A nickel-specific column from Eichrom Industries was used in place of the house-made columns. The Eichrom column functions with the same chemical principals and would not affect data quality. Another change is the treatment of the nickel-dimethylglyoxime precipitate on the filter. This precipitate was dissolved using 5mL of 3 M HCl and an aliquot taken for yield determination; another aliquot was taken to dryness, 0.5mL water added and 10mL Ultima Gold added for subsequent liquid scintillation counting. This is a quality improvement in that there is no potential for nickel to be lost due to potential volatilization of the $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$.

Radiochemical yields were generally high, nominally 90%. The hot cell preparation blank resulted in no detectable activity for either ^{59}Ni or ^{63}Ni . The ^{59}Ni LEP spectra and the ^{63}Ni beta spectra were visually clean from other interferences. No ^{59}Ni activity is detectable in the samples. The ^{63}Ni concentration is consistent throughout the sample; nominally 1.2 $\mu\text{Ci/g}$. The matrix spiked samples were spiked only with ^{63}Ni , as ^{59}Ni is not commercially available. The blank spike, blank spike duplicate, and matrix spike recoveries were excellent at 101%, 98% and 98%, respectively.

Plutonium: Plutonium isotopes were separated in sequence with americium/curium according to procedure PNL-ALO-417, the plutonium was eluted from the column following elution of americium. One change was made to the procedure to effect this separation/elution. The plutonium was reduced to a +3 oxidation state using TiCl_3 solution in 4M HCl. This is a more effective and faster way to elute the plutonium fraction. The plutonium fraction then was precipitation-plated directly according to PNL-ALO-496, and the plutonium measured by alpha energy analysis according to PNL-ALO-422. The plutonium radiochemical yield/counting efficiency was traced with ^{242}Pu .

In order to determine the ^{242}Pu activity, a separate aliquot was processed without added tracer. The indigenous ^{239}Pu was used to trace the yield/count efficiency. The ^{239}Pu activity was determined based on the results of the ^{242}Pu -traced aliquot. No ^{242}Pu was found in the samples so the original premise of using the ^{242}Pu to trace yields is valid. The sample-specific ^{242}Pu detection limit is driven by the tailing of the ^{239}Pu alpha peak into the ^{242}Pu alpha energy region.

The radiochemical yields for the plutonium fractions were good (nominally 70%). The precision for $^{239+240}\text{Pu}$, as determined by the RPD, is 12.2% or better. The RPD associated with ^{238}Pu is high on one sample pair 96-5894/5894dup. Sample 96-5894 had a higher ^{238}Pu concentration than that of the rest of the set. This higher concentration is confirmed on the untraced sample analysis. It may be worthy to note that this same sample also had a higher $^{243/244}\text{Cm}$ activity. However, the MD for sample and duplicate is below 1.96, indicating that the results are statistically equal. For the preparation blank, the $^{239+240}\text{Pu}$ activity represent less than 0.1% of the sample activity, and the ^{238}Pu activity represents less than 0.4% of the sample activity. The blank spike recoveries were 96% or better; the matrix spike recoveries were 95% or better.

Pu-241: The sample preparations for $^{239+240}\text{Pu}$ and ^{238}Pu were used for the subsequent ^{241}Pu determination. The precipitation-plated samples were placed in LSC vials, 10 mL Ultima Gold were added and the samples counted by liquid scintillation according to PNL-ALO-474. Yield was determined by counting the ^{242}Pu by alpha spectrometry.

The LSC spectra showed both the alpha-peak activity ($^{239+240}\text{Pu}$, ^{242}Pu , ^{238}Pu) and the beta peak from the ^{241}Pu . Spectral resolution was excellent. The samples showed good agreement at a nominal $3\mu\text{Ci/g}$. Two samples were higher than this nominal activity concentration: 96-5892 ($5.7\mu\text{Ci/g}$) and 96-5894 ($4.23\mu\text{Ci/g}$). However, because of the relatively high uncertainties, the MD test indicates the results are not statistically different. The mean ^{241}Pu sample activity is 2.4 times that of the $^{239+240}\text{Pu}$. This is the same trend identified for the Vendor Product, where the ratio is 2.3.

The preparation blank activity (i.e., $0.07\mu\text{Ci/g}$) is about 50 times lower than the sample activity. There is not detectable activity in the laboratory blanks. The blank spike and matrix spike recoveries are high at 141% ($\pm 20\%$) and 110% ($\pm 26\%$), respectively. This indicates a possible high bias to the technique, however, the error associated with the spike recoveries are high. These errors may have been lower had the sample been spiked with higher ^{241}Pu activity.

Sm-151 and Pm-147: The $^{151}\text{Sm}/^{147}\text{Pm}$ screen/determinations were made on the final sample preparations for ^{241}Am analysis. The precipitation-plated americium preparation was placed into LSC cocktail and counted according to PNL-ALO-474 by liquid scintillation. For instrument calibration, ^{63}Ni was used because its endpoint energy is similar to that of ^{151}Sm . The ^{147}Pm has a $224\text{ keV } E_{\text{max}}$ which tails under the ^{151}Sm peak at $76\text{ keV } E_{\text{max}}$; thus a summation of ^{151}Sm and ^{147}Pm is provided. The resulting beta spectra indicated a large peak due to the ^{151}Sm ; however, another peak (most probably ^{147}Pm) tailed under the samarium peak. The alpha peak associated with americium/curium was also evident. The preparation blank and laboratory blank resulted in a small amount of beta activity relative to the calibration blank; however, their activities are about 3000 times lower than that of the samples. The propagated error associated with these measurements is nominally 4%. A measure of bias cannot be determined without the blank spike/matrix spike.

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As rare earths, the samarium and promethium radiochemically follow the other rare earths and the Am³ using the TRU-Spec column chemistry (PNL-ALO-417). The samarium and promethium would also be precipitation-plated (PNL-ALO-496), presumably as efficiently as the americium. Thus the radiochemical yield determined for americium is used to correct for the radiochemical yield of samarium/promethium. A better approach would be to utilize blank and matrix spikes to verify the yield. [Note: This was not done, since the request for the ¹⁵¹Sm analysis was received after the americium separation processing and the preparation additional samples was not approved.]

Along with the rare earths samarium and promethium, europium should also appear in this fraction. The samples were counted by GEA to evaluate the ¹⁵⁴Eu and ¹⁵⁵Eu activities; however, none was detected. The absence of the europium isotopes is not understood at this time. The ¹⁵⁴Eu and ¹⁵⁵Eu concentrations in the Am-preparation were determined based on the GEA, and the dilution and yield chemistries applied to the samples should have resulted in a detectable quantities of both ¹⁵⁴Eu and ¹⁵⁵Eu. Because they were not detected, no beta interference for ¹⁵⁴Eu and ¹⁵⁵Eu was present. Had europium been present at the anticipated levels, its contribution would have amounted to nominally 5% of the beta activity in the 2-90 keV region.

This method must be considered a screening method for ¹⁵¹Sm/¹⁴⁷Pm because the radiochemical separation from europium had not been performed and the radiochemical recovery is based solely on the americium recovery. Europium did not recover as well as the americium, indicating that samarium/promethium may have low recovery also.

Se-79: The ⁷⁹Se activities were determined following procedures PNL-ALO-440/474. The separation procedure involves an anion/cation exchange to remove most radiochemical interferences followed by a selenium bromide distillation. Since ⁷⁹Se is not available as a standard, it was not possible to measure standard or sample spike recoveries. Natural selenium metal was added to each sample and the recovered selenium precipitate was used to determine the gravimetric yields.

Acid Digestion Radiochemical yields were nominally 50% and all known beta spectral interferences were removed. No ⁷⁹Se beta activity was seen in either the samples or the blanks above the calibration blank background counting rate. Hence, MDA values are reported.

KOH Fusion Radiochemical yields averaged about 50%. No ⁷⁹Se beta activity was seen in either the samples or the blanks above the calibration blank background counting rate. Hence, MDA values are reported.

Sr-90: The ⁹⁰Sr activities were determined following procedures PNL-ALO-476/431. Radiochemical yields, traced with ⁸⁴Sr, were excellent averaging 90%. The sample analyses gave similar results with RPD values below 10%. Weak ⁹⁰Sr activity was detected in the preparation blank and the lab blank; however, the values are a factor of about 10,000 less than the activities in the samples. Standard and sample spike recoveries were 93% and 88%, respectively.

Sn Isotopes: Radiochemical separation of tin was conducted according to procedure PNL-ALO-453/450, with modifications to accommodate the low-energy photons of ^{119m}Sn and ^{121m}Sn. Modifications include use of tin carrier, and final analytical product mounting on a 47mm filter, allowing for counting of both low and high energy photons. The carrier recovery was measured by

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dissolving the tin off the filter after the counting was completed, then analyzing the solution for elemental tin by ICP-AES. All the analytical results were below the observed detection limits.

A ^{113}Sn matrix spike was analyzed with this set. Its yield-corrected recovery was about 50%, which is lower than the known sources of uncertainty can explain. One possible explanation is that the chemical form of the spike was different from that of the carrier (e.g., a fluoride versus a chloride complex) with different distribution coefficients on the anion exchange resin.

Zr-93: The ^{93}Zr analyses were performed according to draft procedure PNL-ALO-4007. The ^{93}Zr was analyzed using an analytical procedure modified to improve the carrier recovery and provide complete decontamination from plutonium. In summary, the procedure utilizes zirconium carrier to establish radiochemical yield, the sample is passed through three anion exchange columns under varying conditions to separate the impurities. On these samples, the procedure gave carrier recoveries in the 91-99% range and beta spectra with very little contamination. The ^{93}Zr results are about 40 times above the MDA.

Because no ^{93}Zr standard is available, no matrix spikes or blank spikes are possible. The ^{93}Zr counting efficiency was estimated from ^{63}Ni counting efficiency under similar counting conditions. ^{93}Zr and ^{63}Ni have very similar beta energies, with no gamma emitters.

The samples gave RPD values of 28%, 24%, and 45%. These RPD values are greater than the normal 20% limit, but in all three cases the MD shows that the duplicates are statistically the same at 95% confidence. Additionally, two samples were run in replicate; they agree within 2%.

The ^{93}Zr beta spectra obtained from these samples appear to show an alpha emitter — presumably plutonium, the most abundant alpha emitter in the samples. If plutonium alpha is present (that is, $^{238,239,240}\text{Pu}$), ^{241}Pu must also be present, buried in the ^{93}Zr peak. To correct the ^{93}Zr count rate for this bias, the ^{241}Pu count rate in each sample was estimated from the alpha count rate and subtracted from the ^{93}Zr count rate. This correction reduced the ^{93}Zr count rate by roughly 6% in each sample.

The ^{241}Pu correction has considerable uncertainty. If other alpha emitters besides plutonium are contributing to the alpha activity, then the ^{241}Pu correction is too big. If the true ^{241}Pu counting efficiency is much lower than the estimate used in the calculations, then the ^{241}Pu correction is too small. However, because relatively little ^{241}Pu is present in any case, its influence on the overall uncertainty is minor.

4.5 Additional Sample Analyses — Centrifuged Solids and Decanted Liquids

The results of the various analyses performed on the centrifuged solids and decant liquids are presented in Table 4.24 and Table 4.25 for information only. No attempt has been made to correlate the results with the Original Material and Vendor Product analyses. An evaluation of these results will be presented in the Analytical Final Report (In progress).

4.5.1 Preparation of Vendor Product (Decant Liquid)

The HLRF conducted physical testing on Vendor Product sample SPDL-C106-046. Following the conclusion of physical testing activities, samples from the decanted liquid (obtained following centrifuging) were sent to the SAL for limited chemical characterization. The decanted liquid was subsampled for TOC/TIC/TC and IC, and a portion was acid digested (PNL-ALO-128 "HNO₃-HCl Acid Extraction of Liquids for Metals Analysis Using a Dry-Block Heater") and distributed for ICP-AES and limited radiochemical analyses.

4.5.2 Preparation of Original Material (Centrifuged Solids and Decanted Liquid)

The HLRF conducted physical testing on Original Material sample SPDL1-C106-002. Following the conclusion of the physical testing activities, samples from the centrifuged solids and decanted liquid were sent to the SAL for limited chemical characterization. Two fusions techniques, PNL-ALO-115 "Solubilization of Metals from Solids Using KOH-KNO₃ Fusion"; PNL-ALO-116 "Solubilization of Platinum Group Metals using a Na₂O₂-NaCl Fusion," were performed on the centrifuged solids sample and aliquots distributed for ICP-AES. A portion of the centrifuged solids was water leached per PNL-ALO-103 "Water Leach of Sludges, Soils and Other Solid Samples," filtered, and aliquots distributed for anion analysis by Ion Chromatography. The decanted liquid was subsampled for anion determinations by Ion Chromatography, and a portion was acid digested (PNL-ALO-128 "HNO₃-HCl Acid Extraction of Liquids for Metals Analysis Using a Dry-Block Heater") and distributed for ICP-AES.

4.5.3 Chemical Analysis

The analyses of the Vendor Product decanted liquid (following centrifuging) are presented in Table 4.24. The IC (anions) and TOC analyses were performed in duplicate directly on the decanted liquid. The Vendor Product decanted liquid was subjected to an acid digestion or acid dilution prior to ICP-AES (or radiochemical) analysis.

The analysis of the Original Material centrifuged solids and decanted liquid (following centrifuging) are presented in Table 4.25. The IC analyses were performed in duplicate directly on the decanted liquid, as well as on water leaches from the centrifuged solids. The centrifuged solid sample was prepared in duplicate by potassium hydroxide fusion and sodium peroxide fusion technique prior to ICP-AES analysis.

4.5.4 Radiochemical Analysis

The Vendor Product decant liquid (following centrifuging) was prepared by acid digestion, and analyzed for total alpha, ⁹⁰Sr, GEA, and ⁹⁹Tc (by ICP-MS). Weak ⁹⁰Sr activity (0.004 μCi/g of liquid), about 20,000 times less than that measured in the Vendor Product (on a per gram basis), was detected. Cesium-137 was the only gamma emitter detected, and the activity (2.3 μCi/g of liquid) is about 200 times less than the activity measured in the Vendor Product.

5.0 REFERENCES

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Table 4.3 ICP-AES KOH Fusion-Vendor Product
C106 Privatization Contractor Sample

Analyte	KOH Fusion Utilizing Ni Crucibles										Envelope D Specification	Ave/Spec	Flag
	Process Blank 96-6103-PB	SPD1-C106-047 96-6103	SPD1-C106-048 96-6104	SPD1-C106-049 96-6105	SPD1-C106-049 96-6105 Dup	Average		Sliver					
	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g				
Ag		817	1,010	1,370	920	803	1,260	1,030	240	4.38E+03	2.35E-01		
Al	[248]	52,800	50,200	54,400	52,400	48,600	48,900	51,200	2300	1.11E+05	4.65E-01		
As								< 479	N/A	1.29E+03	3.72E-01		
Ba	[90]	[345]	[478]	[193]	[259]	[278]	[267]	[303]	98	1.03E+04	2.84E-02		
Be		405	395	436	422	384	397	407	19	3.61E+04	1.13E-02		
Bi								< 9	N/A	5.15E+02	1.69E-02		
Br		[552]	[551]	[527]	[548]	[526]	[524]	[538]	14	2.22E+04	2.43E-02		
Ca	[1,120]	7,890	8,250	8,950	8,690	8,580	9,770	8,890	720	5.67E+04	1.18E-01		
Cd		[83]	[84]	[80]	[80]	[81]	[78]	[81]	2	3.61E+04	2.25E-03		
Ce								< 523	N/A	8.44E+03	8.12E-02		
Co								< 87	N/A	3.61E+03	2.42E-02		
Cr	[63]	784	800	863	825	790	752	802	38	5.41E+03	1.48E-01		
Cu	[49]	[106]	[111]	[113]	[109]	[115]	[102]	[109]	5	3.88E+03	2.83E-02		
Dy								< 87	N/A	2.06E+02	4.23E-01		
Eu								< 174	N/A	1.25E+02	1.35E+00		
Fe	[268]	142,000	138,000	152,000	146,000	132,000	134,000	141,000	7500	2.28E+05	6.14E-01		
K	N/U	N/U	N/U	N/U	N/U	N/U	N/U	N/U	N/U	1.06E+04	N/U	N/U	
La		[123]	[130]	[118]	[114]	[105]	[121]	[119]	9	2.06E+04	5.75E-03		
Li		[96]	[94]					[90]	6	1.11E+03	8.08E-02		
Mg		[1,370]	[1,440]	[1,500]	[1,450]	[1,180]	[1,210]	[1,260]	130	1.87E+04	8.11E-02		
Mn	[181]	3,070	3,260	3,330	3,170	3,040	2,860	3,140	140	5.15E+04	6.09E-02		
Mo								< 87	N/A	5.15E+03	1.69E-02		
Na	2,990	137,000	145,000	131,000	133,000	153,000	151,000	142,000	9000	1.55E+05	9.19E-01		
Nd		[402]	[427]	[373]	[347]	[347]	[375]	[379]	31	1.37E+04	2.77E-02		
Ni	N/U	N/U	N/U	N/U	N/U	N/U	N/U	N/U	N/U	1.88E+04	N/U	N/U	
P		3,230	3,880	3,620	2,890	3,180	3,550	3,330	370	1.39E+04	2.39E-01		
Pb		2,840	2,060	2,860	3,510	3,200	3,250	2,950	510	8.76E+03	3.37E-01		
Pd								< 1,310	N/A	1.03E+03	1.27E+00		
Rh								< 523	N/A	1.03E+03	5.07E-01		
Ru								< 1,740	N/A	2.63E+03	6.15E-01		
Sb								< 871	N/A	6.70E+03	1.30E-01		
Se								< 610	N/A	4.12E+03	1.48E-01		
Si		69,100	67,300	75,100	72,500	64,200	64,900	68,900	4300	1.49E+05	4.61E-01		
Sn								< 9,920	N/A	2.63E+02	1.38E-01		
Sr		[79]	[78]	[87]	[84]	[73]	[74]	[79]	5	4.12E+03	1.92E-02		
Te								< 2,810	N/A	1.03E+03	2.54E+00		
Th								< 1,740	N/A	4.12E+03	4.23E-01		
Ti		1,010	965	1,100	1,150	959	984	1,050	78	1.03E+04	9.89E-02		
Tl								< 871	N/A	3.61E+03	2.42E-01		
U								< 4,360	N/A	1.06E+05	4.03E-02		
V								< 87	N/A	2.84E+02	3.35E-01		
W								< 3,480	N/A	1.91E+03	1.83E+00		
Y								< 131	N/A	1.29E+03	1.01E-01		
Zn		[203]	[188]	[174]	[181]	[210]	[141]	[183]	25	3.35E+03	5.46E-02		
Zr		1,090	1,500	1,260	[515]	[478]	1,790	1,110	530	1.19E+05	9.33E-03		

-- Analyte not detected above the IDL; only average IDL reported as less than (<) value.

N/A Not Applicable

N/U Not Usable

Results in brackets are qualified but they are included in the calculation of the average.

Table 4.4 ICP-AES Na2O2 Fusion-Vendor Product
C106 Privatization Contractor Sample

Analyte	Na2O2 Fusion: Utilizing Zr Crucibles										Envelope D Specification	Avg/Spec	Flag
	Process Blank 96-6103-PB	SPD1-C106-047 96-6103		SPD1-C106-048 96-6104 Dup		SPD1-C106-049 96-6105 Dup		Average		Slide			
	Upp/L	Upp/L	Upp/L	Upp/L	Upp/L	Upp/L	Upp/L	Upp/L	Slide				
Ag	[50]	1,890	3,880	3,450	3,080	3,350	3,420	3,180	680	4.38E+03	7.28E-01		
Al	[218]	48,000	47,300	51,400	53,200	49,800	47,800	49,500	2400	1.11E+05	4.47E-01		
As	—	—	—	—	—	—	—	< 910	N/A	1.29E+03	3.96E-01		
B	—	[186]	[159]	—	—	—	—	[116]	[154]	35	1.03E+04	1.49E-02	
Ba	[25]	414	430	457	470	431	418	437	22	3.81E+04	1.21E-02		
Be	—	—	—	—	—	—	—	< 9	N/A	5.15E+02	1.80E-02		
Bi	—	[506]	[839]	[661]	[569]	[560]	[554]	[582]	58	2.22E+04	2.82E-02		
Cd	[4,180]	8,350	11,000	10,300	9,800	8,970	10,700	9,850	1000	5.67E+04	1.74E-01		
Ca	—	[73]	[91]	[80]	[87]	[91]	[81]	[91]	5	3.81E+04	2.24E-03		
Ce	—	—	—	—	—	—	—	< 1,580	N/A	6.44E+03	2.45E-01		
Co	—	—	—	—	—	—	—	< 93	N/A	3.61E+03	2.57E-02		
Cr	[45]	647	673	737	771	895	675	700	48	5.41E+03	1.29E-01		
Cu	[49]	[84]	[110]	[126]	[119]	[101]	[111]	[109]	15	3.86E+03	2.81E-02		
Dy	—	—	—	—	—	—	—	< 93	N/A	2.06E+02	4.50E-01		
Eu	—	—	—	—	—	—	—	< 185	N/A	1.29E+02	1.44E+00		
Fe	[270]	124,000	126,000	138,000	142,000	130,000	123,000	131,000	7800	2.29E+05	5.69E-01		
K	[6,930]	[4,840]	[8,790]	[8,260]	[8,630]	[7,580]	[8,420]	[7,590]	1700	1.06E+04	7.18E-01		
La	—	[118]	[176]	[196]	[171]	[166]	[157]	[164]	26	2.06E+04	7.96E-03		
Li	[59]	[105]	[138]	[84]	[71]	[76]	[89]	[94]	25	1.11E+03	8.47E-02		
Mg	—	[1,210]	[1,380]	[1,380]	[1,410]	[1,250]	[1,190]	[1,310]	99	1.67E+04	7.79E-02		
Mn	—	2,470	2,500	2,720	2,820	2,590	2,410	2,590	160	5.15E+04	5.02E-02		
Mo	—	—	—	—	—	—	—	< 93	N/A	5.15E+03	1.80E-02		
Na	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	1.55E+05	N/A	N/A	
Nd	[254]	[400]	[544]	[562]	[525]	[518]	[550]	[517]	59	1.37E+04	3.78E-02		
Ni	597	1,470	1,580	1,780	1,790	1,830	1,700	1,660	120	1.88E+04	8.82E-02		
P	[239]	3,850	3,920	4,570	4,560	3,520	2,800	3,640	870	1.39E+04	2.78E-01		
Pb	[203]	3,230	3,450	3,760	3,760	3,580	3,390	3,530	210	8.76E+03	4.03E-01		
Pd	—	—	—	—	—	—	—	< 1,390	N/A	1.03E+03	1.35E+00		
Rh	—	—	—	—	—	—	—	< 356	N/A	1.03E+03	5.40E-01		
Ru	—	—	—	—	—	—	—	< 1,850	N/A	2.83E+03	6.54E-01		
Sb	—	—	—	—	—	—	—	< 927	N/A	6.70E+03	1.38E-01		
Se	—	—	—	—	—	—	—	< 649	N/A	4.12E+03	1.57E-01		
Si	—	82,000	82,000	67,600	71,200	63,500	61,300	64,600	3900	1.49E+05	4.33E-01		
Sn	—	—	—	—	—	—	—	< 4,170	N/A	2.83E+02	1.47E+01		
Sr	[71]	[127]	[156]	[146]	[147]	[134]	[159]	[145]	12	4.12E+03	3.51E-02		
Te	—	—	—	—	—	—	—	< 2,780	N/A	1.03E+03	2.70E+00		
Th	—	—	—	—	—	—	—	< 1,850	N/A	4.12E+03	4.50E-01		
Ti	—	960	979	1,130	1,170	987	922	1,020	100	1.03E+04	9.94E-02		
Tl	—	—	—	—	—	—	—	< 927	N/A	3.61E+03	2.57E-01		
U	—	—	—	—	—	—	—	< 6,950	N/A	1.08E+05	6.42E-02		
V	—	—	—	—	—	—	—	< 93	N/A	2.58E+02	3.60E-01		
W	—	—	—	—	—	—	—	< 3,710	N/A	1.91E+03	1.94E+00		
Y	—	—	—	—	—	—	—	< 139	N/A	1.29E+03	1.08E-01		
Zn	—	[150]	[159]	[151]	[187]	[162]	[155]	[161]	14	3.35E+03	4.80E-02		
Zr	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	1.19E+05	N/A	N/A	

— Analyte not detected above the IDL; only average IDL reported as less than (<) value.

N/A Not Applicable

N/A Not Usable

Results in brackets are qualified but they are included in the calculation of the average.

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Table 4.5 ICP-AES Acid Digestion-Vendor Product
C106 Privatization Contractor Sample

Analyte	Process Blank 96-6103-PB	Acid Digestion										Average	Stddev	Envelope D Specification	Comparison	
		SPD1-C106-047 96-6103 Dup		SPD1-C106-048 96-6104 Dup		SPD1-C106-049 96-6105 Dup										
		ug/g	ug/g	ug/g	ug/g	ug/g	ug/g									
Ag		75.9	75.5	78.1	78.1	66.4	61.3	76	5	4.35E+03	1.73E-02					
Al	[16]	46,900	46,500	48,100	48,100	47,900	50,100	47,900	1300	1.11E+05	4.33E-01					
As		--	--	--	--	--	--	< 123	--N/A--	1.29E+03	9.55E-02					
B	[119]	310	292	153	183	201	211	225	62	1.03E+04	2.16E-02					
Ba		408	403	417	416	411	433	415	10	3.61E+04	1.15E-02					
Be		--	--	--	--	--	--	< 1	--N/A--	5.15E+02	2.38E-03					
Bi		426	414	438	429	426	447	430	11	2.22E+04	1.94E-02					
Ca	[71]	3,760	3,740	3,900	3,910	3,760	3,960	3,840	93	5.67E+04	6.78E-02					
Cd		66.2	66.3	66.7	66.4	66.5	69.7	73	13	3.61E+04	2.02E-03					
Ce		[307]	[297]	[290]	[288]	[295]	[318]	[299]	11	6.44E+03	4.65E-02					
Co		[28]	[27]	[29]	[28]	[28]	[29]	[29]	1	3.61E+03	7.80E-03					
Cr		527	520	535	533	527	566	533	12	5.41E+03	9.85E-02					
Cu		77.1	76.2	78.6	77.7	74.9	80.3	78	2	3.66E+03	2.00E-02					
Dy		--	--	--	--	--	--	< 12	--N/A--	2.06E+02	5.97E-02					
Eu		--	--	--	--	--	--	< 25	--N/A--	1.29E+02	1.91E-01					
Fe	[8]	135,000	134,000	140,000	139,000	133,000	140,000	137,000	3200	2.29E+05	5.97E-01					
K		--	--	--	--	--	--	< 492	--N/A--	1.06E+04	4.86E-02					
La		[98]	[94]	[95]	[94]	[94]	[102]	[99]	3	2.06E+04	4.86E-03					
Li		85.5	84.2	[26]	[26]	[26]	[27]	46	30	1.11E+03	4.14E-02					
Mg		752	741	783	771	719	760	754	23	1.67E+04	4.50E-02					
Mn		2,560	2,530	2,620	2,600	2,550	2,700	2,600	60	5.15E+04	5.04E-02					
Mo		[27]	[26]	[27]	[26]	[27]	[28]	[27]	1	5.15E+03	5.19E-03					
Na	[161]	168,000	165,000	149,000	156,000	164,000	170,000	162,000	8000	1.55E+05	1.05E+00					
Nd		285	276	279	276	277	297	262	8	1.37E+04	2.06E-02					
Ni		1,130	1,100	1,130	1,120	1,120	1,190	1,130	31	1.88E+04	6.02E-02					
P		1,450	1,380	1,290	1,270	1,310	1,350	1,340	66	1.39E+04	9.64E-02					
Pb		3,370	3,340	3,460	3,450	3,400	3,560	3,430	78	8.76E+03	3.92E-01					
Pd		--	--	--	--	--	--	< 369	--N/A--	1.03E+03	3.58E-01					
Rh		--	--	--	--	--	--	< 74	--N/A--	1.03E+03	7.16E-02					
Ru		--	--	--	--	--	--	< 736	--N/A--	2.83E+03	2.81E-01					
Sb		--	--	--	--	--	--	< 74	--N/A--	6.70E+03	1.10E-02					
Se		--	--	--	--	--	--	< 369	--N/A--	4.12E+03	8.96E-02					
Si	[165]	3,190	2,340	2,290	2,930	2,750	3,310	2,800	430	1.49E+05	1.88E-02					
Sn		--	--	--	--	--	--	< 4,060	--N/A--	2.83E+02	1.43E+01					
Sr		53.2	52.5	54.1	54.0	53.0	56.2	54	1	4.12E+03	1.31E-02					
Te		517	509	526	522	518	545	523	12	1.03E+03	5.07E-01					
Th		[366]	[346]	[349]	[350]	[342]	[350]	[349]	5	4.12E+03	8.46E-02					
Ti		349	337	357	354	338	365	350	11	1.03E+04	3.40E-02					
Tl		--	--	--	--	--	--	< 123	--N/A--	3.61E+03	3.41E-02					
U		[1,940]	[1,920]	[1,930]	[1,940]	[1,920]	[1,930]	[1,930]	37	1.06E+05	1.80E-02					
V		--	--	--	--	--	--	< 37	--N/A--	2.55E+02	1.43E-01					
W		--	--	--	--	--	--	< 492	--N/A--	1.91E+03	2.56E-01					
Y		--	--	--	--	--	--	< 86	--N/A--	1.29E+03	5.69E-02					
Zn	[16]	[116]	[114]	[118]	[118]	[116]	[111]	[116]	3	3.35E+03	3.45E-02					
Zr		837	874	400	455	729	671	651	200	1.19E+05	5.58E-03					

-- Analyte not detected above the IDL; only average IDL reported as less than (<) value.

N/A Not Applicable

Results in brackets are qualified but they are included in the calculation of the average.

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Table 4.6 ICP-AES KOH Fusion—Original Material
C106 Privatization Contractor Sample

Analyte	KOH Fusion Utilizing Ni Crucibles										Envelope D Specification	Comparison	Flag
	Process Blank 96-5892-PB	SPDL1-C106-003 96-5892-1 96-5892 Dup		SPDL1-C106-004 96-5893-1 96-5893 Dup		SPDL1-C106-005 96-5894-1 96-5894 Dup		Average	Stdev				
	Uppr	Uppr	Uppr	Uppr	Uppr	Uppr	Uppr	Uppr	Uppr				
Ag	-	833	1,190	927	1,410	1,040	1,140	1,090	200	4.38E+03	2.49E-01		
Al	[409]	38,300	34,400	37,200	37,200	38,400	40,000	37,300	1900	1.11E+05	3.36E-01		
As	-	-	-	-	-	-	-	< 279	N/A	1.29E+03	2.17E-01		
B	-	[254]	[243]	[254]	[157]	[145]	[287]	[227]	53	1.03E+04	2.20E-02		
Ba	-	218	[190]	202	203	195	212	203	10	3.61E+04	5.63E-03		
Be	-	-	-	-	-	-	-	< 9	N/A	5.15E+02	1.81E-02		
Bi	-	[284]	[238]	[248]	[253]	[186]	[245]	[248]	34	2.22E+04	1.11E-02		
Ca	[702]	[3,310]	[2,890]	[3,370]	[3,440]	[3,000]	[3,390]	[3,230]	230	5.67E+04	3.70E-02		
Cd	-	[45]	[48]	[45]	[44]	[42]	[48]	[46]	2	3.61E+04	1.26E-03		
Ce	-	-	-	-	-	-	-	< 372	N/A	8.44E+03	5.78E-02		
Co	-	-	-	-	-	-	-	< 93	N/A	3.51E+03	2.58E-02		
Cr	[93]	841	897	843	603	588	658	821	30	5.41E+03	1.15E-01		
Cu	-	[99]	[93]	[83]	[88]	[74]	[93]	[88]	9	3.86E+03	2.29E-02		
Dy	-	-	-	-	-	-	-	< 93	N/A	2.06E+02	4.52E-01		
Eu	-	-	-	-	-	-	-	< 186	N/A	1.29E+02	1.45E+00		
Fe	466	70,300	84,000	66,600	85,800	66,800	71,000	67,800	2700	2.29E+05	2.95E-01		
K	N/U	N/U	N/U	N/U	N/U	N/U	N/U	N/U	N/U	1.06E+04	N/U		
La	-	-	-	-	-	-	-	< 93	N/A	2.06E+04	4.52E-03		
Li	-	-	-	-	-	-	-	< 56	N/A	1.11E+03	5.04E-02		
Mg	-	[656]	[753]	[597]	[531]	[575]	[576]	[615]	79	1.67E+04	3.67E-02		
Mn	[148]	1,850	1,690	1,800	1,710	1,620	1,910	1,780	110	5.15E+04	3.42E-02		
Mo	-	-	-	-	-	-	-	< 93	N/A	5.15E+03	1.81E-02		
Na	3,350	229,000	218,000	224,000	235,000	221,000	221,000	225,000	6300	1.55E+05	1.45E+00		
Nd	-	[290]	[248]	[211]	[195]	[172]	[196]	[217]	44	1.37E+04	1.59E-02		
Ni	N/U	N/U	N/U	N/U	N/U	N/U	N/U	N/U	N/U	1.88E+04	N/U		
P	-	3,940	3,610	3,750	3,720	3,510	3,530	3,710	130	1.39E+04	2.87E-01		
Pb	-	[1,280]	[1,540]	[1,370]	[1,720]	[1,470]	1,920	1,550	240	8.76E+03	1.77E-01		
Pd	-	-	-	-	-	-	-	< 652	N/A	1.03E+03	6.33E-01		
Rh	-	-	-	-	-	-	-	< 359	N/A	1.03E+03	5.42E-01		
Ru	-	-	-	-	-	-	-	< 279	N/A	2.83E+03	9.86E-02		
Sb	-	-	-	-	-	-	-	< 466	N/A	6.70E+03	8.95E-02		
Se	-	-	-	-	-	-	-	< 466	N/A	4.12E+03	1.13E-01		
Si	-	-	-	-	-	-	-	34,400	1600	1.49E+05	2.30E-01		
Sn	[997]	36,100	31,700	34,800	34,100	33,700	35,700	34,400	1600	2.83E+03	6.57E+00		
Sr	-	-	-	-	-	-	-	< 1,680	N/A	4.12E+03	7.77E-03		
Te	-	[35]	[30]	[33]	[32]	[51]	[32]	[31]	2	4.12E+03	7.77E-03		
Th	-	-	-	-	-	-	-	< 1,400	N/A	1.03E+03	1.36E+00		
Ta	-	-	-	-	-	-	-	< 1,490	N/A	4.12E+03	3.81E-01		
Ti	-	[445]	[377]	[448]	[416]	424	[450]	427	28	1.03E+04	4.14E-02		
Tl	-	-	-	-	-	-	-	< 931	N/A	3.61E+03	2.58E-01		
U	-	-	-	-	-	-	-	< 3,720	N/A	1.08E+05	3.44E-02		
V	-	[52]	[48]	[41]	[37]	[34]	[38]	[41]	7	2.58E+02	1.60E-01		
W	-	-	-	-	-	-	-	< 931	N/A	1.91E+03	4.88E-01		
Y	-	-	-	-	-	-	-	< 47	N/A	1.29E+03	3.61E-02		
Zn	[50]	[124]	[106]	[115]	[123]	[100]	[149]	[121]	18	3.35E+03	3.82E-02		
Zr	-	[456]	572	[472]	624	703	606	572	94	1.19E+05	4.83E-03		

— Analyte not detected above the IDL; only average IDL reported as less than (<) value.

N/A Not Applicable

N/U Not Usable

Results in brackets are qualified but they are included in the calculation of the average.

Table 4.7 ICP-AES Na2O2 Fusion--Original Material
C106 Privatization Contractor Sample

Analyte	Na2O2 Fusion Utilizing Zr Crucibles										Envelope D Specification	Avg/Spec	% Flag
	Process Blank 96-5892 Pb	SPDL1-C106-003 96-5892 Zr	SPDL1-C106-003 96-5892 Dup	SPDL1-C106-004 96-5893 Zr	SPDL1-C106-004 96-5893 Dup	SPDL1-C106-005 96-5894 Zr	SPDL1-C106-005 96-5894 Dup	Average	% Stdev				
	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g			
Ag	[49]	2,040	2,050	2,210	2,150	1,960	2,100	2,060	83	4.35E+03	4.77E-01		
Al	[303]	36,900	37,000	37,600	37,000	34,200	36,900	36,800	1200	1.11E+05	3.30E-01		
As	--	--	--	--	--	--	< 254	--	--	1.29E+03	1.97E-01		
B	--	--	--	--	--	--	[92]	--	9	1.03E+04	8.92E-03		
Ba	[22]	219	223	228	217	202	215	217	9	3.61E+04	6.03E-03		
Be	--	--	--	--	--	--	--	< 8	--	5.15E+02	1.64E-02		
Bi	[201]	[318]	[330]	[335]	[280]	[210]	[229]	[284]	54	2.22E+04	1.38E-03		
Ca	4,690	7,210	6,870	6,100	7,400	7,230	7,070	6,960	470	5.67E+04	1.23E-01		
Cd	--	[48]	[45]	[40]	[47]	[42]	[40]	[43]	3	3.61E+04	1.20E-03		
Ce	[454]	[537]	[528]	[1,060]	[420]	[406]	[373]	[554]	260	6.44E+03	8.60E-02		
Co	--	--	--	--	--	--	--	< 85	--	3.61E+03	2.35E-02		
Cr	[43]	531	552	565	528	491	519	531	26	5.41E+03	9.82E-02		
Cu	[66]	[93]	[93]	[86]	[89]	[66]	[64]	[79]	14	3.86E+03	2.03E-02		
Dy	--	--	--	--	--	--	--	< 85	--	2.06E+02	4.10E-01		
Eu	--	--	--	--	--	--	--	< 169	--	1.29E+02	1.31E+00		
Fe	[320]	66,000	66,900	67,900	65,500	61,200	66,400	65,700	2300	2.29E+05	2.86E-01		
K	[9,190]	[7,760]	[7,460]	[5,690]	[4,160]	[4,330]	[3,490]	[5,480]	1800	1.06E+04	5.19E-01		
La	--	[120]	[118]	[114]	[100]	[97]	[64]	[107]	12	2.06E+04	5.19E-03		
Li	[80]	[99]	--	[53]	--	--	[47]	[53]	6	1.11E+03	4.79E-02		
Mg	[170]	[675]	[694]	[573]	[640]	[619]	[667]	[645]	44	1.87E+04	3.85E-02		
Mn	--	1,250	1,280	1,350	1,270	1,180	1,260	1,270	55	5.15E+04	2.48E-02		
Mo	--	--	--	--	--	--	--	< 85	--	5.15E+03	1.64E-02		
Na	N/U	N/U	N/U	N/U	N/U	N/U	N/U	N/U	N/U	1.35E+05	N/U		
Nd	[322]	[386]	[379]	[343]	[301]	[299]	[278]	[331]	45	1.37E+04	2.42E-02		
Ni	515	1,080	1,040	1,080	1,060	1,040	889	1,059	36	1.99E+04	5.59E-02		
P	[222]	3,470	2,940	3,530	3,140	3,300	3,080	3,240	230	1.39E+04	2.33E-01		
Pb	[274]	2,110	2,160	2,060	2,000	1,840	1,960	2,030	110	9.78E+03	2.31E-01		
Pd	--	--	--	--	--	--	--	< 1,270	--	1.03E+03	1.23E+00		
Rh	--	--	--	--	--	--	--	< 508	--	1.03E+03	4.93E-01		
Ru	--	--	--	--	--	--	--	< 1,890	--	2.63E+03	5.97E-01		
Sb	--	--	--	--	--	--	--	< 846	--	6.70E+03	1.26E-01		
Se	--	[431]	--	[432]	--	--	--	[432]	1	4.12E+03	1.05E-01		
Si	--	34,000	34,300	33,000	33,200	31,100	34,000	33,300	1200	1.49E+05	2.23E-01		
Sn	--	--	--	--	--	--	--	< 2,540	--	2.83E+02	8.96E+00		
Sr	[68]	[94]	[95]	[76]	[100]	[95]	[88]	[91]	8	4.12E+03	2.21E-02		
Te	--	--	--	--	--	--	--	< 2,540	--	1.03E+03	2.46E+00		
Th	--	--	--	--	--	--	--	< 1,890	--	4.12E+03	4.10E-01		
Ti	--	456	478	460	[420]	[420]	440	445	450	1.03E+04	4.32E-02		
Tl	--	--	--	--	--	--	--	< 846	--	3.61E+03	2.35E-01		
U	--	--	--	--	--	--	--	< 5,080	--	1.08E+05	4.69E-02		
V	--	--	--	--	--	--	--	< 846	--	2.58E+02	3.28E+00		
W	--	--	--	--	--	--	--	< 3,380	--	1.91E+03	1.78E+00		
Y	--	--	--	--	--	--	--	< 846	--	1.29E+03	6.57E-01		
Zn	--	[107]	[118]	[107]	[113]	[107]	[122]	[112]	7	3.35E+03	3.35E-02		
Zr	N/U	N/U	N/U	N/U	N/U	N/U	N/U	N/U	N/U	1.19E+05	N/U		

-- Analyte not detected above the IDL, only average IDL reported as less than (<) value.

N/A Not Applicable

N/U Not Usable

Results in brackets are qualified but they are included in the calculation of the average.

Analytical Summary Report - January 28, 1997

Table 4.8 ICP-AES Acid Digestion--Original Material
C106 Privatization Contractor Sample

Analyte	Process Blank 96-5892-PB	Acid Digestion										Envelope D Specification	Comparison	Flag
		SPDL1-C106-003 96-5892 96-5892 Dup		SPDL1-C106-004 96-5893 96-5893 Dup		SPDL1-C106-005 96-5894 96-5894 Dup		Average		Stdev				
		ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g			
Ag		175	141	144	149	155	150	152	12	4.38E+03	3.44E-02			
Al	[8]	37,200	33,800	35,400	34,600	33,200	32,500	34,400	1700	1.11E+05	3.11E-01			
As		[74]	[73]	[75]	[79]	[69]	[73]	[74]	4	1.29E+03	5.72E-02			
B	85.9	156	120	129	139	149	135	138	13	1.03E+04	1.34E-02			
Ba	--	208	188	195	194	184	180	192	10	3.61E+04	5.32E-03			
Be	--	--	--	--	--	--	--	--	< 0.2	--N/A--	5.15E+02	4.55E-04		
Bi	--	[180]	[168]	[178]	[175]	[138]	[137]	[162]	20	2.22E+04	7.33E-03			
Ca	[39]	1,700	1,550	1,840	1,640	1,840	1,850	1,700	120	5.67E+04	3.01E-02			
Cd	--	39.0	35.7	37.4	36.8	[38]	[39]	37	2	3.61E+04	1.01E-03			
Ce	--	[193]	[182]	[190]	[190]	[179]	[181]	[186]	8	8.44E+03	2.89E-02			
Co	--	[15]	[14]	[15]	[14]	[14]	[14]	[14]	0	3.61E+03	3.94E-03			
Cr	--	451	408	431	422	402	366	418	21	5.41E+03	7.73E-02			
Cu	--	[39]	[38]	[39]	[38]	[37]	[38]	[38]	1	3.86E+03	9.86E-03			
Dy	--	--	--	--	--	--	--	--	< 2.4	--N/A--	2.06E+02	1.14E-02		
Eu	--	--	--	--	--	--	--	--	< 4.7	--N/A--	1.29E+02	3.64E-02		
Fe	--	71,100	63,400	67,600	65,800	64,100	63,400	65,900	3000	2.23E+06	2.87E-01			
K	--	[813]	[823]	[773]	[855]	[882]	[939]	[811]	110	1.06E+04	7.88E-02			
La	--	[57]	[55]	[57]	[57]	[54]	[54]	[56]	2	2.06E+04	2.69E-03			
Li	--	--	[7]	[8]	[8]	[9]	[8]	[9]	1	1.11E+03	7.40E-03			
Mg	--	345	319	344	331	321	328	331	11	1.67E+04	1.98E-02			
Mn	--	1,370	1,240	1,320	1,290	1,230	1,220	1,280	59	5.15E+04	2.48E-02			
Mo	--	[30]	[30]	[26]	[31]	[32]	[30]	[30]	1	5.15E+03	5.90E-03			
Na	100	228,000	227,000	216,000	229,000	230,000	217,000	225,000	6300	1.55E+05	1.45E+00			
Nd	--	[164]	[157]	[162]	[161]	[153]	[155]	[159]	4	1.37E+04	1.16E-02			
Ni	[2]	549	509	531	524	491	487	515	24	1.88E+04	2.74E-02			
P	--	2,080	1,910	2,020	2,000	1,930	1,880	1,970	76	1.39E+04	1.42E-01			
Pb	--	1,940	1,780	1,870	1,830	1,740	1,720	1,810	83	8.78E+03	2.07E-01			
Pd	--	--	--	--	--	--	--	--	< 35.2	--N/A--	1.03E+03	3.41E-02		
Rh	--	--	--	--	--	--	--	--	< 14.1	--N/A--	1.03E+03	1.37E-02		
Ru	--	--	--	--	--	--	--	--	< 46.9	--N/A--	2.83E+03	1.66E-02		
Sb	--	--	--	--	--	--	--	--	< 23.5	--N/A--	6.70E+03	3.50E-03		
Se	--	[221]	[202]	[217]	[210]	[205]	[205]	[210]	8	4.12E+03	5.06E-02			
Si	[163]	25,200	10,200	23,000	22,900	22,500	20,700	20,800	5400	1.49E+05	1.39E-01			
Sn	--	[1,440]	[1,383]	[1,470]	[1,440]	[1,450]	[1,460]	[1,440]	32	2.83E+02	5.06E+00			
Sr	--	[17]	[16]	[17]	[17]	[16]	[19]	[16]	1	4.12E+03	3.94E-03			
Te	--	--	--	--	--	--	--	--	< 70.4	--N/A--	1.03E+03	6.83E-02		
Th	--	--	--	--	--	--	--	--	< 46.9	--N/A--	4.12E+03	1.14E-02		
Tl	--	154	134	147	144	140	144	144	7	1.03E+04	1.40E-02			
Ti	--	--	--	--	--	--	--	--	< 23.5	--N/A--	3.81E+03	6.50E-03		
U	--	[1,090]	[1,370]	[1,270]	[1,500]	[1,450]	[1,460]	[1,360]	150	1.08E+05	1.25E-02			
V	--	[25]	[24]	[25]	[25]	[24]	[24]	[25]	1	2.58E+02	8.50E-02			
W	--	--	--	--	--	--	--	--	< 93.8	--N/A--	1.91E+03	4.82E-02		
Y	--	[41]	[39]	[39]	[39]	[38]	[37]	[39]	2	1.29E+03	3.02E-02			
Zn	--	[57]	[53]	[59]	[57]	[53]	[57]	[56]	2	3.35E+03	1.87E-02			
Zr	--	469	237	353	423	345	367	368	79	1.19E+05	3.09E-03			

-- Analyte not detected above the IDL, only average IDL reported as less than (<) value.

N/A Not Applicable

Results in brackets are qualified but they are included in the calculation of the average.

Table 4.9 ICP/MS KOH Fusion--Vendor Product
C106 Privatization Contractor Sample

Analyte	KOH Fusion Utilizing Ni Crucibles										Envelope D Specification µg/g	Comparison µg/g Spec Flag
	Process Blank 96-6103-PB12	SPD1-C106-047 96-6103-96-6103 Dup	SPD1-C106-048 96-6104-96-6104 Dup	SPD1-C106-049 96-6105-96-6105 Dup	Average µg/g	Stdev µg/g	Stdev µg/g	Stdev µg/g	Stdev µg/g	Stdev µg/g		
Ag	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	4.38E-03	
Al	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	1.11E-05	N/A
As	12.3	299	347	263	230	258	286	44	1.29E-03	2.22E-01	2.22E-01	
B	8.2	277	181	156	193	193	229	83	1.03E-04	2.23E-02	2.23E-02	
Ba	3.35	381	412	439	402	414	412	19	3.61E-04	1.14E-02	1.14E-02	
Ca	65.3	1550	2070	1910	1570	1560	1723	218	5.67E-04	3.04E-02	3.04E-02	
Cd	1.34	101	87.0	98.8	97.7	71.3	90.6	11.4	3.61E-04	2.51E-03	2.51E-03	
Ce	0.15	145	139	189	172	176	160	28	6.44E-02	2.48E-02	2.48E-02	
Co	<1	17	24	36	26	25	26	6	3.61E-03	7.22E-03	7.22E-03	
Cr	11.9	708	752	925	746	704	774	84	5.41E-03	1.43E-01	1.43E-01	
Cu-133	0.70	17.1	18.6	23.1	17.4	11.2	20.4	18.0	4.0	4.84E-03	3.87E-03	N/A
Cu-135	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
Cu	<1	<1	27.0	27.4	<1	10.6	21.7	9.6	3.86E-03	5.61E-03	5.61E-03	
Dy	2.88	18.9	12.4	30.6	24.6	11.1	20.2	7.5	2.08E-02	9.81E-02	9.81E-02	
Er	1.73	14.2	15.4	16.3	16.1	6.95	13.1	4.7	2.58E-01	5.08E-01	5.08E-01	
Eu	<1	18.6	3.11	5.03	7.15	10.3	7.86	5.91	1.29E-02	6.11E-02	6.11E-02	
Fe	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	2.29E-05	N/A	N/A	N/A
Gd	<1	24.8	24.9	31.1	26.2	30.8	25.0	6.9	7.73E-01	3.23E-01	3.23E-01	
Hg	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	7.73E-02	N/A	N/A	N/A
Hr	<1	6.73	5.30	6.92	4.71	4.37	6.07	1.53	2.59E-01	2.36E-01	2.36E-01	
I-129	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
La	0.63	58.6	79.1	82.1	83.1	95.2	80.6	12.1	2.08E-04	3.91E-03	3.91E-03	
Mo-241	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
Mo-93	<1	8.2	67.1	46.4	3.8	54.6	33.8	26.0	1.67E-04	N/A	N/A	N/A
Mg	2.6	1130	1260	1460	1110	1040	1220	156	5.15E-04	5.45E-02	5.45E-02	
Mn	19.3	2580	3120	2940	2670	2660	2807	206	5.15E-04	5.45E-02	5.45E-02	
Mo	5.14	48.7	44.3	63.5	53.0	50.6	57.0	13.7	5.15E-03	1.11E-02	1.11E-02	
Na	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	1.55E-05	N/A	N/A	N/A
Nd	5.49	194	207	248	257	209	233	34	1.37E-04	1.70E-02	1.70E-02	
Ni	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	1.88E-04	N/A	N/A	N/A
Pb-207	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
Pb	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	8.76E-03	N/A	N/A	N/A
Pd	<1	24.7	6.9	33.8	31.1	7.9	36.7	23.5	1.03E-03	2.28E-02	2.28E-02	
Pd-107	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
Pr	<1	84.5	68.8	85.0	70.1	64.2	89.8	71.1	2.63E-03	2.72E-02	2.72E-02	
Pu	4.38	38.4	41.6	29.0	22.9	23.5	30.7	7.7	2.59E-01	1.19E-01	1.19E-01	
Pu-239	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
Pu-240	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
Pu-242	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A

Table 4.9 ICP/MS KOH Fusion--Vendor Product
C106 Privatization Contractor Sample

Analyte	KOH Fusion Utilizing Ni Crucibles										Envelope D		Comparison	Flag
	Process Blank		SPD1-C106-047		SPD1-C106-048		SPD1-C106-049		Average	Stdev	Specification			
	99-8103-PB1	99-8103	99-8103 Dup	99-8104	99-8104 Dup	99-8105	99-8105 Dup							
	ug/g		ug/g		ug/g		ug/g		ug/g			Ave/Spec		
Rb	NAU	NAU	NAU	NAU	NAU	NAU	NAU	NAU	NAU	N/A	1.55E+03	N/A		
Re	1.16	5.00	8.26	8.03	8.79	5.92	3.68	6.61	2.06	N/A	N/A	N/A		
Rh	<1	<1	<1	15.4	84.3	<1	11.2	36.9	41.0	1.03E+03	3.59E-02	3.59E-02		
Ru	<1	91	103	103	93	93	95.7	95.7	6.5	2.83E+03	3.38E-02	3.38E-02		
Sb	<1	<1	<1	<1	<1	<1	<1	<1	N/A	8.70E+03	1.49E-04	1.49E-04		
Se	NAU	NAU	NAU	NAU	NAU	NAU	NAU	NAU	NAU	N/A	4.12E+03	N/A		
Sn	<1	5.44	31.6	83.2	24.7	24.6	6.1	29.3	28.5	2.83E+02	1.03E-01	1.03E-01		
Sr	<1	45	54	77	78	49	50	59	15	4.12E+03	1.42E-02	1.42E-02		
Ta	1.84	15.3	26.8	32.7	29.6	6.91	16.6	21.3	9.9	2.06E+02	1.03E-01	1.03E-01		
Tb	<1	7.41	8.46	9.75	9.86	5.71	7.99	8.20	1.55	2.58E+01	3.18E-01	3.18E-01		
Tc-99	<1	<1		<1		<1		<1	N/A	N/A	N/A	N/A		
Te	22.0	322	511	472	515	353	372	424	85	1.03E+03	4.11E-01	4.11E-01		
Th-232	NAU	NAU	NAU	NAU	NAU	NAU	NAU	NAU	NAU	N/A	N/A	N/A		
Tl	4.4	1003	1040	1260	1290	1050	1100	1124	121	1.03E+04	1.09E-01	1.09E-01		
Tm	<1	2.41	4.10	<1	1.40	<1	<1	2.64	1.36	2.58E+01	1.02E-01	1.02E-01		
Total U	<1	1760	2010	2060	1810	1770	1970	1897	132	1.08E+05	1.75E-02	1.75E-02		
U-233	NAU	NAU	NAU	NAU	NAU	NAU	NAU	NAU	NAU	N/A	N/A	N/A		
U-234	NAU	NAU	NAU	NAU	NAU	NAU	NAU	NAU	NAU	N/A	N/A	N/A		
U-235	NAU	NAU	NAU	NAU	NAU	NAU	NAU	NAU	NAU	N/A	N/A	N/A		
U-236	NAU	NAU	NAU	NAU	NAU	NAU	NAU	NAU	NAU	N/A	N/A	N/A		
U-238	<1	1750	2000	2050	1800	1760	1960	1887	132	N/A	N/A	N/A		
V	NAU	NAU	NAU	NAU	NAU	NAU	NAU	NAU	N/A	2.58E+02	N/A	N/A		
W	11.6	474	541	618	451	407	500	498	74	1.91E+03	2.61E-01	2.61E-01		
Yb	1.39	14.9	20.4	15.8	13.5	2.69	8.26	12.6	6.2	2.58E+01	4.89E-01	4.89E-01		
Zn	5.21	258	444	281	320	268	268	306	71	3.35E+03	9.15E-02	9.15E-02		
Zr	1.99	260	762	343	621	147	465	433	230	1.19E+05	3.66E-03	3.66E-03		

NAU Not Applicable

NAU Not Usable

-No standards used; response taken from different isotope.

Table 4.10 ICP/MS Isotopes KOH Fusion--Vendor Product
C106 Privatization Contractor Sample

Analyte	KOH Fusion Utilizing Ni Crucibles										Envelope D Specification µCi/g	Comparison Ave/Spec - Flag
	Process Blank 96-6103-PB µCi/g	SPDI-C106-047 96-6103 µCi/g	SPDI-C106-048 96-6104 Dup µCi/g	SPDI-C106-049 96-6105 Dup µCi/g	SPDI-C106-049 96-6105 Dup µCi/g	Average µCi/g	Slide µCi/g					
Cs-135	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	7.73E-01	N/A
I-129											2.32E-03	0.0E+00
Nb-237											5.93E-01	0.0E+00
Pu-107*											1.03E-01	0.0E+00
Pu-239											2.45E+01	0.0E+00
Pu-240*											8.10E+00	0.0E+00
Pu-242*											1.03E-03	0.0E+00
Tc-99	<1.7E-02	<1.7E-02	<1.7E-02	<1.7E-02	<1.7E-02	N/A					1.18E-02	1.5E-04
Th-232*											1.98E-02	0.0E+00
U-233*											2.58E-05	0.0E+00
U-234*											1.98E-02	0.0E+00
U-235*											8.24E-04	0.0E+00
U-236*											2.11E-03	0.0E+00
U-238*	1.78E-07	5.88E-04	6.89E-04	6.05E-04	5.91E-04	6.34E-04	4.44E-05				1.49E-02	4.2E-02

N/A Not Applicable

N/A Not Usable

*No standards used, response taken from different isotopes.

Blank field no data quantified

Table 4.11 ICP/MS Na2O2 Fusion--Vendor Product
C106 Privatization Contractor Sample

Analyte	Na2O2 Fusion Utilizing Zr Crucibles										Envelope D Specification	Comparison	
	Process Blank 96-6103-PB	SPD1-C106-047 96-6103 96-6103 Dup		SPD1-C106-048 96-6104 96-6104 Dup		SPD1-C106-049 96-6105 96-6105 Dup		Average	Stddev				
	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	Ave/Spec	Flag	
Ag	N/U	N/U	N/U	N/U	N/U	N/U	N/U	N/U	N/A	4.38E+03	N/A		
Al	N/U	N/U	N/U	N/U	N/U	N/U	N/U	N/U	N/A	1.11E+05	N/A		
As	6.6	219	192	310	334	228	250	256	55	1.29E+03	1.98E-01		
B	2.2	198	160	87	48	51	63	101	63	1.03E+04	9.82E-03		
Ba	<1	417	446	410	436	420	432	427	14	3.61E+04	1.18E-02		
Ce	133	3340	3820	4210	4080	3500	3650	3767	336	5.67E+04	6.65E-02		
Cd	<1	84.8	78.0	81.0	71.0	67.0	62.6	74.1	8.6	3.61E+04	2.05E-03		
Co	<1	198	216	210	235	181	174	202	23	6.44E+03	3.14E-02		
Cr	<1	18.6	20.0	23.7	23.6	23.9	21.0	21.8	2.3	3.61E+03	6.04E-03		
Cr	5.99	696	705	783	809	735	706	739	47	5.41E+03	1.37E-01		
Cs-133	<1	9.30	6.77	6.50	9.70	7.10	3.70	7.18	2.17	4.64E+03	1.55E-03		
Cs-135	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A		
Cu	1.2	154.0	136.1	160.0	175.8	156.4	155.3	156.3	12.7	3.86E+03	4.04E-02		
Dy	<1	1.70	1.00	4.00	4.30	5.50	5.12	3.60	1.84	2.06E+02	1.75E-02		
Er	<1	<1	<1	<1	0.3	<1	<1	0.3	N/A	2.58E+01	1.16E-02		
Eu	<1	7.30	8.00	7.20	9.60	6.10	8.86	7.84	1.26	1.29E+02	6.09E-02		
Fe	N/U	N/U	N/U	N/U	N/U	N/U	N/U	N/U	N/A	2.29E+05	N/A		
Gd	<1	14.0	14.0	14.0	15.0	8.90	6.90	12.1	3.4	7.73E+01	1.57E-01		
Hg*	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	7.73E+02	N/A		
Ho	<1	<1	1.11	<1	<1	<1	<1	1.11	N/A	2.58E+01	4.31E-02		
I-129	<20	<20	<20	<20	<20	<20	<20	N/A	N/A	N/A	N/A		
La	<1	79.9	88.9	110	109	90.4	74.9	92.1	14.5	2.06E+04	4.47E-03		
m/z-241	<1	1.10	1.84	2.14	1.60	1.53	1.70	1.65	0.34	N/A	N/A		
m/z-93	<1	257	256	297	323	157	117	234	81	N/A	N/A		
Mg	7.05	1230	1330	1260	1370	1190	1160	1257	81	1.67E+04	7.50E-02		
Mn	3.63	2500	2460	2040	2760	2570	2420	2558	128	5.15E+04	4.97E-02		
Mo	1.30	21.0	24.2	32.0	32.0	28.0	30.0	27.9	4.5	5.15E+03	5.41E-03		
Na	N/U	N/U	N/U	N/U	N/U	N/U	N/U	N/U	N/A	1.55E+05	N/A		
Nd	<1	202	241	220	260	210	213	224	22	1.37E+04	1.64E-02		
Ni	30.5	1500	1560	1650	1650	1620	1650	1605	62	1.88E+04	8.53E-02		
Np-237	<1	6.70	4.70	6.92	5.89	5.00	4.18	5.57	1.12	N/A	N/A		
Pb	2.76	2470	2540	2660	2640	2590	2420	2553	95	8.76E+03	2.92E-01		
Pd	<1	22.0	29.0	36.2	23.0	26.0	28.2	27.4	5.1	1.03E+03	2.66E-02		
Pd-107*	N/U	N/U	N/U	N/U	N/U	N/U	N/U	N/U	N/A	N/A	N/A		
Pr	<1	83.6	84.4	87.2	91.9	76.4	81.0	84.1	5.3	2.83E+03	2.97E-02		
Pl	<1	5.21	4.00	3.50	5.27	4.90	8.26	5.19	1.66	2.58E+01	2.01E-01		
Pu-239	<1	22.6	25.0	26.0	26.5	22.4	24.9	24.6	1.7	N/A	N/A		
Pu-240*	<1	<1	<1	<1	<1	<1	<1	<1	N/A	N/A	N/A		
Pu-242*	<1	0.01	<1	0.03	<1	0.03	0.02	0.02	0.01	N/A	N/A		

Table 4.11 ICP/MS Na2O2 Fusion--Vendor Product
C106 Privatization Contractor Sample

Analyte	Na2O2 Fusion Utilizing Zr Crucibles										Envelope D Specification	Comparison	
	Process Blank 96-6103-PB		SPD1-C106-047 96-6103 96-6104 Dup		SPD1-C106-048 96-6104 96-6104 Dup		SPD1-C106-049 96-6105 96-6105 Dup		Average	Stddev			
	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	Ave/Spec	Flag	
Rb	<1	4.90	7.70	13.00	8.00	5.17	7.30	7.68	2.92	1.55E+03	4.97E-03		
Re	<1	<1	1.00	<1	<1	<1	<1	1.00	N/A	N/A	N/A		
Rh	<1	143	120	147	170	144	141	144	16	1.03E+03	1.40E-01		
Ru	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	2.83E+03	N/A		
Sb	<1	<1	<1	<1	<1	<1	<1	<1	N/A	6.70E+03	1.49E-04		
Se	N/U	N/U	N/U	N/U	N/U	N/U	N/U	N/U	N/A	4.12E+03	N/A		
Sn	1.73	75.0	100	130	138	105	64.6	102	29	2.83E+02	3.60E-01		
Sr	6.87	137	160	152	151	134	168	150	13	4.12E+03	3.65E-02		
Ta	<1	28.8	33.0	39.0	38.0	17.70	15.1	28.6	10.2	2.06E+02	1.39E-01		
Tb	<1	1.00	2.00	2.85	2.80	2.23	3.15	2.34	0.78	2.58E+01	9.08E-02		
Tc-99	N/U	N/U	N/U	N/U	N/U	N/U	N/U	N/U	N/A	N/A	N/A		
Te	4.4	340	160	430	340	218	240	288	99	1.03E+03	2.80E-01		
Th-232	<1	461	575	644	685	290	336	499	163	N/A	N/A		
Ti	3.20	1270	1240	1500	1490	1240	1140	1313	147	1.03E+04	1.27E-01		
Tm	<1	<1	<1	1.24	<1	<1	1.10	1.17	0.10	2.58E+01	4.54E-02		
Total U	<1	1940	2030	1990	2120	2030	1950	2010	66	1.08E+05	1.86E-02		
U-233	N/U	N/U	N/U	N/U	N/U	N/U	N/U	N/U	N/U	N/A	N/A		
U-234	N/U	N/U	N/U	N/U	N/U	N/U	N/U	N/U	N/U	N/A	N/A		
U-235	N/U	N/U	N/U	N/U	N/U	N/U	N/U	N/U	N/U	N/A	N/A		
U-236	N/U	N/U	N/U	N/U	N/U	N/U	N/U	N/U	N/U	N/A	N/A		
U-238	<1	1930	2020	1980	2100	2020	1940	1998	63	N/A	N/A		
V	N/U	N/U	N/U	N/U	N/U	N/U	N/U	N/U	N/U	2.58E+02	N/A		
W	6.6	388	371	455	474	268	183	357	112	1.91E+03	1.87E-01		
Yb	<1	2.00	3.50	1.80	5.30	4.61	2.20	3.24	1.48	2.58E+01	1.26E-01		
Zn	1.50	228	251	263	288	164	208	234	44	3.35E+03	6.97E-02		
Zr	N/U	N/U	N/U	N/U	N/U	N/U	N/U	N/U	N/A	1.19E+05	N/A		

N/A Not Applicable

N/U Not Usable

*No standards used; response taken from different isotope.

Table 4.12 ICPIMS Isotopes Na2O2 Fusion--Vendor Product
C106 Privatization Contractor Sample

Analyte	Na2O2 Fusion Utilizing Zr Crucibles										Envelope D. Specification Upper UCLg	Companion AveSpec Flag
	Process Blank 96-4103-PB	SPDI-C106-047 96-6103	SPDI-C106-048 96-6104	SPDI-C106-049 96-6105	SPDI-C106-050 96-6106	SPDI-C106-051 96-6107	SPDI-C106-052 96-6108	SPDI-C106-053 96-6109	SPDI-C106-054 96-6110	SPDI-C106-055 96-6111		
Ca-135	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	7.75E-01	N/A
Ca-136	<4E-03	<4E-03	<4E-03	<4E-03	<4E-03	<4E-03	<4E-03	<4E-03	<4E-03	<4E-03	2.32E-03	1.7E+00
Ca-237	1.4E-04	4.72E-03	4.88E-03	4.15E-03	4.15E-03	4.15E-03	4.15E-03	4.15E-03	4.15E-03	4.15E-03	5.93E-01	6.6E-03
Pu-107	NU	NU	NU	NU	NU	NU	NU	NU	NU	NU	1.03E-01	N/A
Pu-239	3E-02	1.40E+00	1.59E+00	1.61E+00	1.61E+00	1.61E+00	1.61E+00	1.61E+00	1.61E+00	1.61E+00	2.45E-01	6.2E-02
Pu-240	<2E-03	4.1E-02	3.6E-02	4.5E-02	2.3E-02	2.3E-02	2.3E-02	2.3E-02	2.3E-02	2.3E-02	6.70E+00	4.7E-03
Pu-242	<4E-03	<4E-03	<4E-03	<4E-03	<4E-03	<4E-03	<4E-03	<4E-03	<4E-03	<4E-03	1.03E-03	2.2E+00
Ti-99	<1E-07	5.07E-05	6.33E-05	7.08E-05	7.54E-05	7.54E-05	7.54E-05	7.54E-05	7.54E-05	7.54E-05	1.0E+02	0.0E+00
Ti-232											1.98E-02	2.0E-03
U-233											2.98E-05	0.0E+00
U-234											1.98E-02	0.0E+00
U-235											8.24E-04	0.0E+00
U-236											2.11E-03	0.0E+00
U-238	3.80E-08	6.48E-04	6.65E-04	7.08E-04	6.79E-04	6.79E-04	6.79E-04	6.79E-04	6.79E-04	6.79E-04	1.48E-02	4.5E-02

N/A Not Applicable

NU Not Usable

No standards used, response taken from different isotopes

Table 4.13 ICP/MS Acid Digestion--Vendor Product
C106 Privatization Contractor Sample

Analyte	Acid Digestion										Envelope D Specification	Comparison	
	Process Blank 96-6103-PB	SPD1-C106-047		SPD1-C106-048		SPD1-C106-049		Average		Stddev		Ave/Spec	Flag
	ug/g	96-6103	96-6103 Dup	96-6104	96-6104 Dup	96-6105	96-6105 Dup	ug/g	ug/g	ug/g			
Ag	NAJ	NAJ	NAJ	NAJ	NAJ	NAJ	NAJ	NAJ	NAJ	N/A	4.38E-03	N/A	
Al	8.22	32000	32000	32300	34100	32100	38100	33433	2425	1.11E+05	3.02E-01		
As	0.97	172	163	158	138	145	154	155	12	1.29E+03	1.20E-01		
B	50.3	267	259	121	142	158	169	186	62	1.03E+04	1.80E-02		
Ba	0.10	348	362	365	358	359	378	362	10	3.61E+04	1.00E-02		
Ca	84.0	11300	8950	8360	9140	9090	10600	9573	1124	5.67E+04	1.69E-01		
Cd	0.05	58.4	60.0	58.5	55.9	56.0	59.5	58.1	1.7	3.61E+04	1.61E-03		
Ce	0.03	161	164	157	151	156	167	160	6	6.44E+03	2.48E-02		
Co	0.03	15.6	16.0	16.1	15.6	14.8	15.9	15.7	0.5	3.61E+03	4.35E-03		
Cr	0.67	429	433	429	412	414	440	426	11	5.41E+03	7.88E-02		
Cs-133	<0.02	10.9	11.6	10.1	10.0	10.2	9.88	10.4	0.7	4.64E+03	2.25E-03		
Cs-135	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	
Cu	0.4	109.7	109.0	112.3	109.5	107.1	112.3	110.0	2.0	3.86E+03	2.85E-02		
Dy	0.04	1.52	1.40	1.45	1.60	1.34	1.50	1.47	0.09	2.06E+02	7.13E-03		
Er	<0.02	0.7	0.80	0.8	0.7	0.8	0.80	0.8	0.0	2.58E+01	2.99E-02		
Eu	0.02	6.20	6.40	6.45	6.70	6.23	6.77	6.46	0.24	1.29E+02	5.01E-02		
Fe	20.2	96200	110000	115000	114000	103000	120000	109700	8717	2.29E+05	4.78E-01		
Gd	<0.02	14.9	13.3	12.2	13.1	13.3	14.8	13.6	1.0	7.73E+01	1.76E-01		
Hg	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	
Ho	<0.02	0.15	0.14	0.16	0.10	0.20	0.10	0.14	0.04	2.58E+02	5.50E-03		
I-129	<1	1.00	<1	<1	1.20	1.27	1.60	1.27	0.25	N/A	N/A	N/A	
La	<0.02	84.7	86.3	84.2	80.3	83.7	90.5	84.9	3.4	2.06E+04	4.12E-03		
m/z-241	<0.02	0.90	0.77	0.70	0.77	0.74	0.76	0.77	0.07	N/A	N/A	N/A	
m/z-93	<0.02	60.5	65.2	34.3	36.7	52.6	50.4	49.9	12.4	N/A	N/A	N/A	
Mg	2.82	472	452	436	429	428	455	446	17	1.67E+04	2.66E-02		
Mn	4.00	2060	1930	2130	2040	1900	2200	2043	115	5.15E+04	3.97E-02		
Mo	0.07	10.7	9.23	9.29	9.60	8.99	9.78	9.60	0.60	5.15E+03	1.86E-03		
Na	27.9	150000	208000	186000	148000	188000	210000	181667	27171	1.55E+05	1.18E+00		
Nd	0.07	194	199	192	187	191	197	193	4	1.37E+04	1.42E-02		
Ni	7.40	963	912	993	939	864	1020	948	56	1.88E+04	5.04E-02		
Np-237	<0.02	4.22	4.52	4.39	4.78	4.43	4.55	4.48	0.19	N/A	N/A	N/A	
Pb	8.31	2700	2740	2960	2620	3100	3000	2853	192	8.76E+03	3.26E-01		
Pd	0.1	9.12	10.4	10.8	10.0	9.29	10.3	10.0	0.7	1.03E+03	9.71E-03		
Pd-107	<0.5	5.00	1.00	<0.5	<0.5	0.60	<0.5	2.20	2.43	N/A	N/A	N/A	
Pr	0.02	70.8	72.3	69.4	67.9	69.5	73.2	70.5	2.0	2.83E+03	2.49E-02		
Pl	0.10	0.2	0.3	0.2	0.2	0.1	0.4	0.2	0.1	2.58E+01	8.86E-03		
Pu-239	<0.02	7.52	7.65	6.86	6.87	7.52	7.59	7.34	0.37	N/A	N/A	N/A	
Pu-240	<0.02	0.60	0.59	0.48	0.55	0.58	0.55	0.56	0.04	N/A	N/A	N/A	
Pu-242	<0.02	0.02	0.02	<0.02	<0.02	<0.02	<0.02	0.02	0.00	N/A	N/A	N/A	

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Table 4.13 ICP/MS Acid Digestion--Vendor Product
C106 Privatization Contractor Sample

Analyte	Acid Digestion										Envelope D. Specification ¹ ug/g	Comparison	
	Process Blank 96-6103-PB		SPD1-C106-047 96-6103 96-6103 Dup		SPD1-C106-048 96-6104 96-6104 Dup		SPD1-C106-049 96-6105 96-6105 Dup		Average	Stddev			
	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g		Ave/Spec	Flag
Rb	0.2	1.21	1.07	1.25	1.10	1.00	1.00	1.10	0.11	1.55E+03	7.15E-04		
Re	0.02	0.03	0.12	0.08	0.10	0.05	0.09	0.08	0.03	N/A	N/A		
Rh	<0.02	108.6	107.7	100.4	101.9	101.6	107.6	104.7	3.7	1.03E+03	1.02E-01		
Ru	0.1	55.7	56.3	53.3	52.6	53.3	53.8	54.2	1.5	2.83E+03	1.91E-02		
Sb	<0.02	1.00	<0.02	<0.02	<0.02	0.70	0.50	0.73	0.25	6.70E+03	1.09E-04		
Se	N/U	N/U	N/U	N/U	N/U	N/U	N/U	N/U	N/A	4.12E+03	N/A		
Sn	<0.02	36.8	35.5	35.4	33.5	32.8	34.6	34.8	1.5	2.83E+02	1.23E-01		
Sr	0.07	44.9	45.6	45.3	44.6	43.7	46.3	45.1	0.9	4.12E+03	1.09E-02		
Ta	0.09	15.3	9.7	8.2	8.4	8.50	12.4	10.4	2.9	2.06E+02	5.06E-02		
Tb	0.02	1.52	1.46	1.42	1.55	1.50	1.30	1.46	0.09	2.58E+01	5.66E-02		
Tc-99	0.048	0.396	0.399	0.260	0.293	0.333	0.297	0.330	0.058	N/A	N/A		
Te	1.74	206	202	213	193	196	210	203	8	1.03E+03	1.97E-01		
Th-232	<0.2	194	178	185	186	180	185	185	6	N/A	N/A		
Ti	0.4	366	362	365	351	338	368	358	12	1.03E+04	3.48E-02		
Tm	<0.02	0.10	0.05	0.06	0.06	0.05	0.05	0.06	0.02	2.58E+01	2.39E-03		
Total U	2.12	1910	1910	1910	1770	1950	1980	1905	72	1.08E+05	1.76E-02		
U-233	<0.02	0.19	0.18	0.13	0.14	0.18	0.22	0.17	0.03	N/A	N/A		
U-234	<0.02	0.13	0.11	0.11	0.11	0.11	0.17	0.12	0.02	N/A	N/A		
U-235	<0.02	13.1	13.8	12.8	11.7	13.7	12.8	13.0	0.8	N/A	N/A		
U-236	<0.02	0.34	0.28	0.25	0.24	0.33	0.28	0.29	0.04	N/A	N/A		
U-238	2.11	1900	1900	1900	1760	1940	1970	1895	72	N/A	N/A		
V	4	44.4	42.6	38.4	32.4	26.3	39.5	37.2	6.8	2.58E+02	1.45E-01		
W	0.9	237	240	210	205	214	224	222	15	1.91E+03	1.16E-01		
Yb	0.02	0.40	0.30	0.20	0.29	0.20	0.10	0.2	0.1	2.58E+01	9.63E-03		
Zn	5.95	110	100	103	93.4	91.7	97.1	99.3	6.7	3.35E+03	2.96E-02		
Zr	0.25	307	340	171	183	276	258	256	67	1.19E+05	2.16E-03		

N/A Not Applicable

N/U Not Usable

*No standards used; response taken from different isotope.

Table 4.14 ICP/MS Isotopes Acid Digestion--Vendor Product
C106 Privatization Contractor Sample

Analyte	Process Blank		Acid Digestion										Isotope D. Specification		Comparison	
	96-5103-PB	96-5103-PB	SPD1-C106-047	96-5103 Dup	SPD1-C106-048	96-5104 Dup	SPD1-C106-049	96-5105 Dup	SPD1-C106-048	96-5104 Dup	SPD1-C106-049	96-5105 Dup	96-5103-PB	96-5104 Dup	96-5105 Dup	Flag
Cs-133	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	
I-129	<2E-04	2E-04	<2E-04	<2E-04	2.1E-04	2.1E-04	2.2E-04	2.8E-04	3.17E-03	3.21E-03	2.2E-04	3.1E-05	2.32E-03	2.32E-03	9.4E-02	
Nb-237	<7E-06	2.98E-03	3.19E-03	3.09E-03	<3E-04	<3E-04	3.0E-04	<3E-04	4.66E-01	4.71E-01	3.16E-03	1.3E-04	5.93E-01	5.93E-01	5.3E-03	
Pb-207	<3E-04	2.6E-03	5.1E-04	<3E-04	<3E-04	<3E-04	3.0E-04	<3E-04	4.20E-01	4.55E-01	1.1E-03	1.3E-03	1.03E-01	1.03E-01	1.1E-02	
Pu-239	<6E-04	4.66E-01	4.74E-01	4.25E-01	4.25E-01	4.25E-01	4.66E-01	4.71E-01	4.66E-01	4.71E-01	4.55E-01	2.3E-02	2.45E-01	2.45E-01	1.9E-02	
Pu-240	<2E-03	1.4E-01	1.3E-01	1.1E-01	1.2E-01	1.2E-01	1.3E-01	1.2E-01	1.3E-01	1.2E-01	1.3E-01	1.0E-02	6.70E-00	6.70E-00	1.9E-02	
Pu-242	<4E-05	8E-05	8E-05	<4E-05	4E-05	4E-05	4E-05	4E-05	5.69E-03	5.69E-03	6E-05	2.2E-05	1.83E-03	1.83E-03	3.1E-02	
Tc-99	8.20E-04	6.73E-03	6.78E-03	4.42E-03	4.98E-03	4.98E-03	5.69E-03	5.05E-03	5.69E-03	5.05E-03	5.69E-03	9.7E-04	1.16E-02	1.16E-02	4.8E-05	
Th-232	<2E-08	2.13E-05	1.95E-05	2.04E-05	2.05E-05	2.05E-05	1.98E-05	2.04E-05	1.98E-05	2.04E-05	2.03E-05	6.0E-07	1.98E-02	1.98E-02	1.0E-03	
U-233	<1E-04	1.9E-03	1.8E-03	1.2E-03	1.3E-03	1.3E-03	1.7E-03	2.2E-03	1.7E-03	2.2E-03	1.7E-03	3.4E-04	2.98E-05	2.98E-05	6.9E-01	
U-235	<6E-05	8.1E-04	7.1E-04	7.1E-04	6.7E-04	7.0E-04	7.0E-04	1.0E-03	7.7E-04	1.0E-03	7.7E-04	1.4E-04	1.98E-02	1.98E-02	3.9E-02	
U-236	<2E-05	2.8E-05	3.0E-05	2.8E-05	2.5E-05	3.0E-05	3.0E-05	2.8E-05	3.0E-05	2.8E-05	2.8E-05	1.6E-06	8.24E-04	8.24E-04	3.4E-02	
U-238	<6E-07	2.2E-05	1.8E-05	1.8E-05	1.5E-05	2.1E-05	1.8E-05	1.8E-05	2.1E-05	1.8E-05	1.8E-05	2.6E-06	2.11E-03	2.11E-03	8.7E-03	
U-238	7.07E-07	6.38E-04	6.38E-04	6.38E-04	5.91E-04	6.52E-04	6.52E-04	6.62E-04	6.52E-04	6.62E-04	6.4E-04	2.4E-05	1.49E-02	1.49E-02	4.3E-02	

N/A Not Applicable

NU Not Usable

*No standards used; response taken from different isotope.

Table 4.15 ICP/MS KOH Fusion--Original Material
C106 Privatization Contractor Sample

Analyte	Process Blank		SPDL-4-C106-003		SPDL-4-C106-004		SPDL-1-C106-005		Average		Envelope D.		Completion	
	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	Flag
Ag	<50	723	978	645	1200	1030	1100	979	22967	2109	1.11E+03	2.94E-01		
Al	<1000	20800	21100	21900	26200	24500	23300	56	45	22	1.29E+03	4.34E-02		
As	<20	73	<20	83	29	48	45	56	22	63	1.03E+04	2.02E-02		
B	56	266	246	107	156	156	248	208	63	11	3.61E+04	4.11E-03		
Be	<100	137	162	157	148	151	133	684	93	6	5.07E+04	1.22E-02		
Ce	<100	793	535	739	638	724	736	26	33	3	3.01E+03	7.09E-04		
Cl	<20	34	<20	21	24	24	<20	35	41	3	3.01E+03	1.14E-01		
Co	<20	46	38	43	42	37	41	637	29	N/A	4.94E+03	1.08E-02		
Cu	112	647	613	673	613	610	668	<50	<20	N/A	4.94E+03	1.08E-02		
Cr-133	<50	<50	<50	<50	<50	<50	<50	<50	<20	N/A	4.94E+03	1.08E-02		
Cu-135	13	15	<10	10	10	<10	<10	13	4	N/A	4.94E+03	1.08E-02		
Dy	<20	115	122	121	115	126	118	120	4	N/A	4.94E+03	1.08E-02		
Er	<20	<20	<20	<20	<20	<20	<20	<20	<20	N/A	4.94E+03	1.08E-02		
Eu	<20	<20	<20	<20	<20	<20	<20	<20	<20	N/A	4.94E+03	1.08E-02		
Fe	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	4.94E+03	1.08E-02		
Gd	<20	<20	<20	<20	<20	<20	<20	<20	<20	N/A	4.94E+03	1.08E-02		
Hg	20	33	31	26	23	37	32	30	5	N/A	4.94E+03	1.08E-02		
Mo	<20	<20	<20	<20	<20	<20	<20	<20	<20	N/A	4.94E+03	1.08E-02		
La-129	88	<50	<50	73	<50	120	70	88	28	N/A	4.94E+03	1.08E-02		
La	<20	29	21	24	<20	28	<20	25	4	N/A	4.94E+03	1.08E-02		
Mn-241	2.6	<2	2.2	3.3	2.4	2.1	3.2	2.6	0.6	N/A	4.94E+03	1.08E-02		
Mn-93	<20	30	<20	<20	<20	<20	<20	<20	30	N/A	4.94E+03	1.08E-02		
Mg	55	575	581	489	418	484	463	495	55	N/A	4.94E+03	1.08E-02		
Mn	131	1680	1540	1690	1530	1530	1750	1020	98	N/A	4.94E+03	1.08E-02		
Mo	<20	66	78	75	63	66	42	65	13	N/A	4.94E+03	1.08E-02		
Na	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	4.94E+03	1.08E-02		
Nd	32	132	86	105	93	98	100	102	16	N/A	4.94E+03	1.08E-02		
Ne	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	4.94E+03	1.08E-02		
Ne-237	<2	4.6	4.0	4.7	6.5	3.4	2.3	4.3	1.4	N/A	4.94E+03	1.08E-02		
Ph	28	796	1090	981	1110	1410	1410	1103	210	N/A	4.94E+03	1.08E-02		
Pr	<20	<20	<20	<20	<20	<20	<20	<20	<20	N/A	4.94E+03	1.08E-02		
Pu-107	<20	22	21	21	23	<20	<20	<20	<20	N/A	4.94E+03	1.08E-02		
Pu	<20	<20	<20	<20	<20	<20	<20	<20	<20	N/A	4.94E+03	1.08E-02		
Pu-239	3.1	22.2	17.9	23.1	19.6	13.1	20.3	4.5	1.6	N/A	4.94E+03	1.08E-02		
Pu-240	4.1	4.1	3.6	5.4	4.9	7.9	3.8	5.0	0.7	N/A	4.94E+03	1.08E-02		
Pu-242	2.9	3.4	3.2	4.1	2.6	2.0	3.2	3.1	0.7	N/A	4.94E+03	1.08E-02		

Table 4.15 ICP/MS KOH Fusion--Original Material
C106 Privatization Contractor Sample

Analyte	KOH Fusion Utilizing Ni Crucibles										Envelope D Specification	Comparison	
	Process Blank 96-5892-PB		SPDL1-C106-003 96-5892 96-5892 Dup		SPDL1-C106-004 96-5893 96-5893 Dup		SPDL1-C106-005 96-5894 96-5894 Dup		Average	Stddev		Ave/Spec	Flag
	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	
Rb	330	445	405	429	385	389	420	412	24	1.55E+03	2.67E-01		
Re	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A		
Rh	<20	<20	<20	<20	<20	<20	<20	<20	<20	1.03E+03	1.94E-02		
Ru	<20	107	101	113	110	99	100	105	6	2.83E+03	3.70E-02		
Sb	<20	30	29	<20	27	<20	35	30	3	6.70E+03	4.53E-03		
Se	<100	<100	<100	<100	<100	<100	<100	<100	N/A	4.12E+03	2.43E-02		
Sn	<20	<20	<20	<20	<20	<20	<20	<20	N/A	2.83E+02	7.06E-02		
Sr	<20	26	<20	30	22	24	23	25	3	4.12E+03	6.07E-03		
Ta	<20	<20	<20	<20	<20	<20	<20	<20	N/A	2.06E+02	9.70E-02		
Tb	<20	<20	<20	<20	<20	<20	<20	<20	N/A	2.58E+01	7.76E-01		
Tc-99	<5	<5	6.0	<5	5.2	<5	<5	5.6	0.6	N/A	N/A		
Te	<50	<50	<50	<50	<50	66	<50	66	N/A	1.03E+03	6.39E-02		
Th-232	<20	44	36	30	46	23	33	35	9	N/A	N/A		
Ti	43	664	528	532	541	563	539	564	53	1.03E+04	5.48E-02		
Tm	<20	<20	<20	<20	<20	<20	<20	<20	N/A	2.58E+01	7.76E-01		
Total U	<2	1250	1170	1220	1270	1210	856	1163	154	1.08E+05	1.07E-02		
U-233	<2	<2	<2	<2	<2	<2	3	3	N/A	N/A	N/A		
U-234	2.9	2.3	<2	3.8	<2	2.0	<2	2.7	1.0	N/A	N/A		
U-235	<2	10.8	7.5	10.5	9.0	9.3	6.2	8.9	1.8	N/A	N/A		
U-236	2.4	<2	<2	4.2	2.6	2.0	2.2	2.8	1.0	N/A	N/A		
U-238	<2	1240	1160	1210	1260	1200	850	1153	152	N/A	N/A		
V	125	315	415	346	428	397	391	382	43	2.58E+02	1.48E+00		
W	22	432	368	436	420	387	453	416	32	1.91E+03	2.18E-01		
Yb	<20	<20	<20	<20	<20	<20	<20	<20	N/A	2.58E+01	7.76E-01		
Zn	98	195	157	183	202	174	209	187	19	3.35E+03	5.58E-02		
Zr	<20	319	256	230	140	193	130	211	72	1.19E+05	1.78E-03		

N/A Not Applicable

NU Not Usable

*No standards used; response taken from different isotopes.

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Table 4.16 ICP/MS Isotopes KOH Fusion--Original Material
C106 Privatization Contractor Sample

Analyte	KOH Fusion Utilizing NI Crucibles										Envelope D Specification uCi/g	Comparison Avg/Spec Flag
	Process Blank 96-5892-PB uCi/g	SPDI-C106-003 96-5892 Dup uCi/g	SPDI-C106-004 96-5893 Dup uCi/g	SPDI-C106-005 96-5894 Dup uCi/g	Average uCi/g	Stddev uCi/g						
Cs-135	1.1E-02	1.3E-02	9E-03	9E-03	9E-03	1.1E-02	1.6E-03	7.7E-01	1.4E-02			
I-129	1.7E-02	<1E-02	1.3E-02	<1E-02	1.2E-02	1.5E-02	4.9E-03	2.32E-03	6.9E+00	*		
Np-237	<1E-04	3.2E-03	2.8E-03	3.3E-03	2.4E-03	3.0E-03	1.0E-03	5.93E-01	5.0E-03			
Pd-107		<1E-02	<1E-02	<1E-02	<1E-02	<1E-02	N/A	1.03E-01	9.7E-02			
Pu-239	2E-01	1.4E+00	1.1E+00	1.6E+00	1.2E+00	1.3E+00	2.6E-01	2.46E+01	5.1E-02			
Pu-240	9E-01	9E-01	1.2E+00	1.1E+00	9E-01	1.1E+00	3.6E-01	6.70E+00	1.7E-01			
Pu-242	1.1E-02	1.3E-02	1.6E-02	1.9E-02	1.3E-02	1.2E-02	2.8E-03	1.03E-03	6.0E+00			
Tc-99	<8.9E-02	<8.9E-02	1.0E-01	8.8E-02	<8.9E-02	9.4E-02	6.5E-03	1.16E+02	8.1E-04			
Th-232	<2.2E-06	4.80E-06	3.30E-06	5.10E-06	2.80E-06	3.90E-06	9.7E-07	1.89E+02	2.0E-04			
U-233	<2E-02	<2E-02	<2E-02	<2E-02	3.0E-02	3.0E-02	N/A	2.58E-05	1.2E+03	*		
U-234	1.8E-02	1.4E-02	2.4E-02	1.2E-02	<1E-02	1.7E-02	6.4E-03	1.98E-02	8.4E-01			
U-235	4.3E-06	2.3E-05	1.6E-05	2.3E-05	1.3E-05	1.9E-05	3.9E-06	8.24E-04	2.3E-02			
U-236	3.0E-02	<2.5E-02	5.3E-02	3.3E-02	2.8E-02	3.5E-02	1.3E-02	2.11E-03	1.6E-01	*		
U-238	6.70E-07	4.17E-04	3.90E-04	4.23E-04	1.31E-04	3.62E-04	1.1E-04	1.49E-02	2.4E-02			

N/A Not Applicable

ND Not Usable

*No standards used; response taken from different isotope.

Table 4.17 ICP/MS Na2O2 Fusion-Original Material
C106 Privatization Contractor Sample

Analyte	Na2O2 Fusion Utilizing Zr Crucibles										Envelope D Specification	Comparison Avg/Spec : Flag
	Process Blank 96-5892-Pg	SPDL1-C106-003 96-5892-Dup	SPDL1-C106-004 96-5893-Dup	SPDL1-C106-005 96-5894-Dup	Average Stddev	Average Stddev	Average Stddev	Average Stddev	Average Stddev	Average Stddev		
Ag	<20	2300	2600	2560	2390	2110	2390	177	4.38E+03	5.6E-01		
Al	NUI	NUI	NUI	NUI	NUI	NUI	NUI	NUI	1.11E+05	N/A		
As	<5	38.9	46.0	36.4	21.3	46.2	34.9	12.5	1.29E+03	2.71E-02		
B	<20	71	112	89	106	148	108	26	1.03E+04	5.0E-02		
Ba	<5	210	195	210	186	195	198	10	3.01E+04	5.0E-03		
Ca	400	1000	890	950	890	840	907	58	5.07E+04	1.00E-02		
Cd	7.7	39.5	38.5	41.7	31.6	35.0	37.0	3.6	3.01E+04	1.00E-03		
Ce	<5	82.5	60.7	86.6	71.5	71.7	77.3	12.2	6.44E+03	1.20E-02		
Co	<5	7.3	5.7	7.4	<5	<5	7.1	0.8	3.81E+03	1.97E-03		
Cr	<20	538	573	596	496	474	530	39	5.41E+03	9.79E-02		
Cu-133	<5	10.8	7.0	6.6	7.1	<5	7.8	1.6	4.04E+03	1.69E-03		
Cu-135	NUI	NUI	NUI	NUI	NUI	NUI	NUI	NUI	N/A	N/A		
Cu	8.8	82.8	78.8	76.0	71.5	81.2	77.6	4.1	3.86E+03	2.01E-02		
Dy	6.1	6.3	5.8	5.6	7.0	6.2	6.7	1.3	2.06E+02	3.33E-02		
Er	6.0	<5	<5	<5	6.0	<5	6.0	N/A	2.98E+01	2.34E-01		
Eu	<5	<5	<5	<5	<5	5.8	5.8	NUI	1.29E+02	4.50E-02		
Fe	NUI	NUI	NUI	NUI	NUI	NUI	NUI	NUI	2.25E+05	N/A		
Gd	<5	16.4	17.0	15.7	10.2	9.3	13.1	3.6	7.73E+01	1.70E-01		
Hf	23	NUI	NUI	NUI	NUI	NUI	NUI	NUI	7.73E+02	N/A		
Ho	<5	<5	<5	<5	<5	<5	<5	NUI	2.98E+01	1.94E-01		
I-129	NUI	NUI	NUI	NUI	NUI	NUI	NUI	NUI	N/A	N/A		
La	<5	52.0	48.5	43.3	47.1	36.1	45.9	5.5	2.06E+04	2.23E-03		
La-241	NUI	NUI	NUI	NUI	NUI	NUI	NUI	NUI	N/A	N/A		
Mn-55	5.4	86.2	45.4	107.1	90.9	95.4	72.3	37.5	N/A	N/A		
Mg	58	610	624	471	555	588	568	55	1.67E+04	3.39E-02		
Mn	<20	1240	1340	1220	1150	1170	1228	67	5.15E+04	2.98E-02		
Mo	9.3	30.2	43.6	34.3	51.2	39.8	38.6	7.8	5.15E+03	7.59E-03		
Na	NUI	NUI	NUI	NUI	NUI	NUI	NUI	NUI	1.55E+05	N/A		
Nd	5	103	106	117	102	94	107	10	1.37E+04	7.86E-03		
Ni	<50	482	338	479	466	445	450	57	1.88E+04	2.98E-02		
Np-237	NUI	NUI	NUI	NUI	NUI	NUI	NUI	NUI	N/A	N/A		
Pb	6.2	1630	1750	1720	1610	1640	1680	60	8.78E+03	1.92E-01		
Pd	30.3	39.8	32.0	40.6	28.7	30.8	33.7	5.2	1.03E+03	3.27E-02		
Pd-107	NUI	NUI	NUI	NUI	NUI	NUI	NUI	NUI	N/A	N/A		
Pt	<5	43.0	34.8	37.1	35.6	32.9	36.6	3.5	2.83E+03	1.29E-02		
Rh	<5	<5	<5	<5	<5	<5	<5	N/A	2.98E+01	1.94E-01		
Pu-239	NUI	NUI	NUI	NUI	NUI	NUI	NUI	NUI	N/A	N/A		
Pu-240	NUI	NUI	NUI	NUI	NUI	NUI	NUI	NUI	N/A	N/A		
Pu-242	NUI	NUI	NUI	NUI	NUI	NUI	NUI	NUI	N/A	N/A		

Table 4.17 ICP/MS Na2O2 Fusion--Original Material
C106 Privatization Contractor Sample

Analyte	Na2O2 Fusion Utilizing Zr Crucibles										Envelope D. Specification		Comparison	
	Process Blank 96-5892-P8		SPDL1-C106-003		SPDL1-C106-004		SPDL1-C106-005		Average	Stddev				
	96-5892	96-5892 Dup	96-5893	96-5893 Dup	96-5894	96-5894 Dup	ug/g	ug/g			ug/g	ug/g	Ave/Spec	Flag
Rb	3.3	6.2	5.7	3.6	5.4	2.9	3.6	4.6	1.4	1.55E+03	2.95E-03			
Re	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A		
Rh	<20	<20	<20	<20	<20	<20	<20	<20	N/A	1.03E+03	1.94E-02			
Ru	N/U	N/U	N/U	N/U	N/U	N/U	N/U	N/U	N/U	2.83E+03	N/A			
Sb	<5	6.1	7.0	7.1	6.4	7.1	6.6	6.7	0.4	6.70E+03	1.00E-03			
Se	<50	<50	<50	<50	<50	56	<50	56	N/A	4.12E+03	1.35E-02			
Sn	144.6	97.2	48.6	99.1	86.9	70.0	71.1	78.8	19.3	2.83E+02	2.78E-01			
Sr	73.9	76.5	88.2	65.7	82.7	83.9	75.0	78.7	8.0	4.12E+03	1.91E-02			
Ta	<5	13.8	5.1	17.6	14.9	13.7	<5	13.0	4.7	2.06E+02	6.32E-02			
Tb	<5	<5	<5	<5	<5	<5	<5	<5	N/A	2.58E+01	1.94E-01			
Tc-99	N/U	N/U	N/U	N/U	N/U	N/U	N/U	N/U	N/U	N/A	N/A			
Te	137.5	102.7	63.6	134.0	77.5	100.6	55.7	89.0	29.1	1.03E+03	8.64E-02			
Th-232	N/U	N/U	N/U	N/U	N/U	N/U	N/U	N/U	N/U	N/A	N/A			
Ti	416	780	800	671	736	734	689	735	50	1.03E+04	7.13E-02			
Tm	<5	<5	<5	<5	<5	<5	<5	<5	N/A	2.58E+01	1.94E-01			
Total U	<5	962	1180	1070	1090	1380	1110	1132	141	1.08E+05	1.05E-02			
U-233	N/U	N/U	N/U	N/U	N/U	N/U	N/U	N/U	N/U	N/A	N/A			
U-234	N/U	N/U	N/U	N/U	N/U	N/U	N/U	N/U	N/U	N/A	N/A			
U-235	N/U	N/U	N/U	N/U	N/U	N/U	N/U	N/U	N/U	N/A	N/A			
U-236	N/U	N/U	N/U	N/U	N/U	N/U	N/U	N/U	N/U	N/A	N/A			
U-238	N/U	N/U	N/U	N/U	N/U	N/U	N/U	N/U	N/U	N/A	N/A			
V	507	646	895	942	941	1030	843	883	131	2.58E+02	3.43E+00			
W	<5	259	260	339	368	403	255	314	65	1.91E+03	1.65E-01			
Yb	<5	<5	<5	<5	<5	<5	<5	<5	N/A	2.58E+01	1.94E-01			
Zn	114	136	105	123	120	163	118	127	20	3.35E+03	3.80E-02			
Zr	N/U	N/U	N/U	N/U	N/U	N/U	N/U	N/U	N/U	1.19E+05	N/A			

N/A Not Applicable

N/U Not Usable

*No standards used; response taken from different isotope.

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Table 4.18 ICP/MS Isotopes Na2O2 Fusion--Original Material
C106 Privatization Contractor Sample

Analyte	Na2O2 Fusion Utilizing Zr Oxidables				Envelope D Specification uCi/g	Comparison Avg/Spec ± Flag
	Process Blank 96-5892-PB1 uCi/g	SPD1-C106-003 96-5892-96-5892 Dup uCi/g	SPD1-C106-004 96-5893-96-5893 Dup uCi/g	SPD1-C106-005 96-5894-96-5894 Dup uCi/g		
Cs-135					7.73E-01	0.0E+00
I-129					2.32E-03	0.0E+00
Nb-237					5.93E-01	0.0E+00
Pd-107					1.03E-01	0.0E+00
Pd-238					2.46E-01	0.0E+00
Pd-240					6.70E+00	0.0E+00
Pu-242					1.83E-03	0.0E+00
Tc-99					1.10E+02	0.0E+00
Th-232					1.99E-02	0.0E+00
U-233					2.58E-05	0.0E+00
U-234					1.94E-02	0.0E+00
U-235					8.24E-04	0.0E+00
U-236					2.11E-03	0.0E+00
U-238					1.46E-02	0.0E+00

N/A Not Applicable

ND Not Usable

-No standards used; response taken from different isotope.
Blank field no data quantified

Table 4.19 ICP/MS Acid Digestion--Original Material
C106 Privatization Contractor Sample

Analyte	Acid digestion										Envelope D. Specification	Comparison	
	Process Blank 96-5892-PB	SPDL1-C106-003 96-5892 96-5892 Dup		SPDL1-C106-004 96-5893 96-5893 Dup		SPDL1-C106-005 96-5894 96-5894 Dup		Average	Stddev			Ave/Spec	Flag
Ag	<5	94.8	71.4	79.6	79.6	70.0	80.4	79.3	8.8	4.38E+03	1.81E-02		
Al	<50	30100	28400	28500	31000	28900	26900	28967	1431	1.11E+05	2.61E-01		
As	15.2	29.6	35.4	32.2	39.0	18.0	22.1	29.4	8.0	1.29E+03	2.28E-02		
B	82	123	106	97	107	126	109	111	11	1.03E+04	1.08E-02		
Ba	<6	199	198	199	185	179	201	194	9	3.61E+04	5.37E-03		
Ca	28	410	310	388	382	408	400	383	38	5.67E+04	6.76E-03		
Cd	<5	31.6	32.0	27.3	26.2	25.8	31.8	29.1	3.0	3.61E+04	8.07E-04		
Ce	<5	103.1	96.8	97.9	85.7	90.7	93.4	94.6	6.1	6.44E+03	1.47E-02		
Co	<5	8.2	7.4	7.7	7.2	8.4	7.3	7.7	0.5	3.61E+03	2.13E-03		
Cr	10	402	393	379	365	369	382	382	14	5.41E+03	7.05E-02		
Cs-133	<5	8.6	9.4	7.5	7.7	7.9	7.5	8.1	0.8	4.64E+03	1.75E-03		
Cs-135	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	N/A	N/A	N/A		
Cu	<5	71.2	70.6	68.9	66.4	67.8	67.5	68.4	2.0	3.86E+03	1.77E-02		
Dy	<5	<5	<5	<5	<5	<5	<5	<5	N/A	2.06E+02	2.43E-02		
Er	<5	<5	<5	<5	<5	<5	<5	<5	N/A	7.58E+01	1.94E-01		
Eu	<5	<5	<5	<5	<5	<5	<5	<5	N/A	1.29E+02	3.80E-02		
Fe	<6000	97000	95700	97800	86800	95600	96500	95033	4148	2.29E+05	4.14E-01		
Gd	<5	11.0	9.1	15.2	8.9	9.5	9.4	10.5	2.4	7.73E+01	1.36E-01		
Hg	<5	56	59	60	57	40	54	56	4	7.73E+02	7.22E-02		
Ho	<5	<5	<5	<5	<5	<5	<5	<5	N/A	2.58E+01	1.94E-01		
I-129	<5	7.8	9.2	11.8	9.8	10.4	14.8	10.6	2.4	N/A	N/A		
La	<5	52.6	51.8	52.2	51.7	47.5	51.5	51.2	1.9	2.06E+04	2.48E-03		
m/z-241	<0.2	0.6	0.5	0.6	<0.5	<0.5	<0.5	0.6	0.1	N/A	N/A		
m/z-93	<5	31.8	15.4	36.0	47.6	42.4	19.8	32.2	12.6	N/A	N/A		
Mg	<5	278	262	277	249	261	273	267	11	1.87E+04	1.59E-02		
Mn	<5	1150	1120	1140	1020	1090	1110	1105	47	5.15E+04	2.14E-02		
Mo	<5	18.8	23.2	19.7	20.6	21.1	21.8	20.9	1.6	5.15E+03	4.05E-03		
Na	<200	178000	185000	169000	199000	181000	167000	179833	11669	1.55E+05	1.16E+00		
Nd	<7	148	108	116	106	128	136	124	17	1.37E+04	9.06E-03		
Ni	31	241	259	222	191	181	199	216	30	1.88E+04	1.15E-02		
Np-237	0.2	1.6	2.2	2.1	2.4	2.0	2.1	2.1	0.3	N/A	N/A		
Pb	6	1217	1191	1176	1100	1105	1137	1154	48	8.76E+03	1.32E-01		
Pd	<5	8.0	9.0	8.6	8.5	8.8	8.7	8.3	0.9	1.03E+03	8.03E-03		
Pd-107	<5	16.5	6.1	<5	<5	<5	<5	11.3	7.4	N/A	N/A		
Pr	<5	43.2	43.6	43.6	35.2	37.1	37.8	40.1	3.8	2.83E+03	1.41E-02		
Pt	<5	<5	<5	<5	<5	<5	<5	<5	N/A	2.58E+01	1.94E-01		
Pu-239	<0.2	4.9	6.5	3.7	4.6	5.3	6.9	5.3	1.2	N/A	N/A		
Pu-240	<0.4	0.9	0.9	<0.4	0.7	0.7	0.5	0.7	0.2	N/A	N/A		
Pu-242	<0.6	<0.6	<0.6	<0.6	<0.6	<0.6	<0.6	<0.6	N/A	N/A	N/A		

Table 4.19 ICP/MS Acid Digestion--Original Material
C106 Privatization Contractor Sample

Analyte	Process Blank 96-5892-PB				Acid digestion				SPOL-1-C106-004				SPOL-1-C106-005				Envelope D Specification				Comparison	
	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	Ave/Spec	Flag
Rb	<5	N/A	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	N/A	N/A	N/A	N/A	3.23E-03	
Rh	<5	N/A	14.7	17.8	15.7	14.8	16.8	15.5	15.9	15.5	15.5	15.5	15.5	15.5	15.5	15.5	1.2	1.03E-03	1.54E-02	1.54E-02	1.54E-02	
Ru	<5	81.9	90.2	78.6	86.0	88.4	87.5	85.4	85.4	85.4	85.4	85.4	85.4	85.4	85.4	85.4	4.4	2.83E-03	3.01E-02	3.01E-02	3.01E-02	
Sb	6	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	N/A	6.70E-03	7.48E-04	7.48E-04	7.48E-04	
Sa	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	N/A	4.12E-03	1.70E-02	1.70E-02	1.70E-02	
Sc	<5	26.0	26.8	25.9	22.0	26.0	28.1	26.0	28.1	26.0	28.1	26.0	28.1	26.0	28.1	26.0	2.3	2.83E-02	9.17E-02	9.17E-02	9.17E-02	
Sr	<5	13.4	13.9	12.1	12.2	11.0	11.6	11.6	11.6	11.6	11.6	11.6	11.6	11.6	11.6	11.6	1.1	4.12E-03	3.00E-03	3.00E-03	3.00E-03	
Ta	<5	14.2	6.9	14.1	12.4	13.5	15.0	12.7	12.7	12.7	12.7	12.7	12.7	12.7	12.7	12.7	3.0	2.06E-02	6.16E-02	6.16E-02	6.16E-02	
Tb	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	N/A	2.58E-01	1.94E-01	1.94E-01	1.94E-01	
Tc-99	<0.5	1.9	1.7	1.6	1.7	1.7	2.1	1.7	2.1	1.7	2.1	1.7	2.1	1.7	2.1	1.7	0.2	N/A	N/A	N/A	N/A	
Te	<5	34.4	31.5	21.0	18.2	25.7	25.7	27.4	26.4	26.4	26.4	26.4	26.4	26.4	26.4	26.4	6.1	1.03E-03	2.56E-02	2.56E-02	2.56E-02	
Th-232	<5	63	115	50	41	57	99	69	69	69	69	69	69	69	69	69	28	N/A	N/A	N/A	N/A	
Ti	11	179	167	153	161	176	176	178	169	169	169	169	169	169	169	169	11	1.03E-04	1.04E-02	1.04E-02	1.04E-02	
Tm	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	N/A	2.58E-01	1.94E-01	1.94E-01	1.94E-01	
Total U	1	920	1186	995	1163	1113	1113	1208	1096	1096	1096	1096	1096	1096	1096	1096	117	1.08E-05	1.01E-02	1.01E-02	1.01E-02	
U-233+	<0.4	<0.4	<0.4	<0.4	<0.4	<0.4	<0.4	<0.4	<0.4	<0.4	<0.4	<0.4	<0.4	<0.4	<0.4	<0.4	N/A	N/A	N/A	N/A	N/A	
U-234+	<0.2	<0.2	0.4	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	N/A	N/A	N/A	N/A	N/A	
U-235+	<0.2	5.8	7.7	7.1	6.8	7.4	8.1	7.2	7.2	7.2	7.2	7.2	7.2	7.2	7.2	7.2	0.8	N/A	N/A	N/A	N/A	
U-236+	<0.2	<0.2	<0.2	0.2	<0.2	<0.2	0.4	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	0.1	N/A	N/A	N/A	N/A	
U-238	1	910	1180	980	1150	1110	1110	1200	1080	1080	1080	1080	1080	1080	1080	1080	117	N/A	N/A	N/A	N/A	
V	43	98	97	97	96	104	104	100	98	98	98	98	98	98	98	98	3	2.58E-02	3.85E-01	3.85E-01	3.85E-01	
W	<10	389	386	304	409	401	416	399	399	399	399	399	399	399	399	399	11	1.31E-02	2.08E-01	2.08E-01	2.08E-01	
Yb	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	N/A	2.58E-01	1.94E-01	1.94E-01	1.94E-01	
Zn	37.1	87.7	67.4	85.8	79.1	79.1	73.3	73.3	78.7	78.7	78.7	78.7	78.7	78.7	78.7	78.7	7.6	3.35E-03	2.35E-02	2.35E-02	2.35E-02	
Zr	<5	313	161	467	574	509	509	261	384	384	384	384	384	384	384	384	155	1.19E-05	3.24E-03	3.24E-03	3.24E-03	

N/A Not Applicable

NDU Not Usable

-No standards used; response taken from different isotope.

Table 4.20 ICP/MS Isotopes Acid Digestion—Original Material
C106 Privatization Contractor Sample

Analyte	Acid digestion											Envelope D Specification		Comparison	
	Process Blank	SPD1-C106-003			SPD1-C106-004			SPD1-C106-005							
	96-5892-PB	96-5892	96-5892 Dup	96-5893	96-5893 Dup	96-5894	96-5894 Dup	Average	Stddev	uCi/g	uCi/g				
	uCi/g	uCi/g	uCi/g	uCi/g	uCi/g	uCi/g	uCi/g	uCi/g	uCi/g	uCi/g	uCi/g				
Cs-135	<5E-04	<5E-04	<5E-04	<5E-04	<5E-04	<5E-04	<5E-04	<5.0E-04	N/A	7.73E-01	6.5E-04				
I-129	<9E-04	1.4E-03	1.6E-03	2.0E-03	1.7E-03	1.8E-03	2.6E-03	1.9E-03	4.2E-04	2.32E-03	8.0E-01				
Np-237	1.4E-05	1.1E-03	1.6E-03	1.5E-03	1.7E-03	1.4E-03	1.5E-03	1.5E-03	2.1E-04	5.93E-01	2.5E-03				
Pd-107	<3E-03	8.5E-03	3.1E-03	<3E-03	<3E-03	<3E-03	<3E-03	5.8E-03	3.8E-03	1.03E-01	5.6E-02				
Pu-239	<1E-02	3.0E-01	4.0E-01	2.3E-01	2.9E-01	3.3E-01	4.3E-01	3.3E-01	7.4E-02	2.45E+01	1.3E-02				
Pu-240	<9E-02	2.0E-01	2.0E-01	<9E-02	1.6E-01	1.6E-01	1.1E-01	1.7E-01	3.7E-02	6.70E+00	2.5E-02				
Pu-242	<2E-03	<2E-03	<2E-03	<2E-03	<2E-03	<2E-03	<2E-03	<2E-03	N/A	1.83E-03	1.1E+00				
Tc-99	<9E-03	3.2E-02	2.9E-02	2.7E-02	2.9E-02	3.6E-02	2.9E-02	3.0E-02	3.2E-03	1.16E+02	2.6E-04				
Th-232	<6E-07	6.9E-06	1.3E-05	5.5E-06	4.5E-06	6.3E-06	9.8E-06	7.7E-06	3.2E-06	1.98E-02	3.9E-04				
U-233	<4E-03	<4E-03	<4E-03	<4E-03	<4E-03	<4E-03	<4E-03	<4E-03	N/A	2.58E-05	1.6E+02				
U-234	<1E-03	<1E-03	<1E-03	<1E-03	<1E-03	<1E-03	<1E-03	<1E-03	N/A	1.98E-02	5.0E-02				
U-235	<4E-07	1.3E-05	1.7E-05	1.5E-05	1.5E-05	1.6E-05	1.7E-05	1.6E-05	1.5E-06	8.24E-04	1.9E-02				
U-236	<1E-05	<1E-05	<1E-05	<1E-05	<1E-05	2.6E-05	<1E-05	2.6E-05	N/A	2.11E-03	1.2E-02				
U-238	3.00E-07	3.06E-04	3.96E-04	3.29E-04	3.06E-04	3.73E-04	4.03E-04	3.66E-04	3.9E-05	1.49E-02	2.4E-02				

N/A Not Applicable

NU Not Usable

-No standards used; response taken from different isotope.

Analytical Summary Report - January 28, 1997

Table 4.21 Wet Chemistry Analysis--Vendor Product
C106 Privatization Contractor Sample

Analyte	Process Blank 96-6103-PB		SPDL-C106-047 96-6103 - 96-6103 Dup		SPDL-C106-048 96-6104 - 96-6104 Dup		SPDL-C106-049 96-6105 - 96-6105 Dup		Average		Standard Dev		Envelope D Specification		Comparison	
	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	Ave/Spec	Flag
Br	<2.6E-01	<7.69E-01	<1.10E+02	<9.30E+01	<9.69E+01	<1.03E+02	<1.04E+02	<1.10E+02	<1.10E+02	<1.10E+02	N/A	N/A	N/A	N/A	N/A	
C2O4	<5.1E-01	1.75E-05	1.89E-05	1.62E-05	1.53E-05	1.63E-05	1.77E-05	1.70E-05	1.70E-05	1.70E-05	2.62E-01	2.59E-03	N/A	4.9E-02	N/A	
Cl	3.39E-01	1.13E-02	1.72E-02	1.12E-02	1.02E-02	1.42E-02	1.15E-02	1.29E-02	1.29E-02	1.29E-02	2.62E-01	2.60E-04	2.60E-04	1.0E-03	N/A	
F	<2.6E-01	<1.92E+03	<2.91E+03	<2.37E+03	<2.72E+03	<2.64E+03	<2.59E+03	<2.59E+03	<2.59E+03	<2.59E+03	7.45E-02	1.00E-04	1.00E-04	1.7E-03	N/A	
NH3-N	8.00E-00	1.70E-01	2.60E-01	9.00E-03	1.26E-01	2.04E-01	2.23E-03	2.07E-03	2.07E-03	2.07E-03	3.93E-02	2.69E-05	2.69E-05	1.0E-02	N/A	
NO2	<5.1E-01	1.26E-03	4.61E-03	8.85E-03	1.60E-02	6.82E-02	6.39E-02	6.06E-02	6.06E-02	6.06E-02	4.29E-02	2.69E-05	2.69E-05	1.0E-02	N/A	
NO3	<5.1E-01	1.26E-03	4.61E-03	8.85E-03	1.60E-02	6.82E-02	6.39E-02	6.06E-02	6.06E-02	6.06E-02	4.29E-02	2.69E-05	2.69E-05	1.0E-02	N/A	
PO4	<5.1E-01	1.11E-02	1.95E-02	1.47E-02	1.50E-02	2.03E-02	1.44E-02	1.60E-02	1.60E-02	1.60E-02	3.51E-01	N/A	N/A	N/A	N/A	
SO4	6.17E-01	4.62E-02	6.95E-02	4.19E-02	3.65E-02	4.80E-02	4.92E-02	4.70E-02	4.70E-02	4.70E-02	8.99E-01	N/A	N/A	N/A	N/A	
Direct																
CN	3.40E-01	9.63E-00	9.00E-03	8.55E-03	8.83E-03	1.04E-01	1.00E-01	9.50E-03	9.50E-03	9.50E-03	8.93E-01	1.29E-04	1.29E-04	7.4E-04	N/A	
Hg (CVAA)	<1.0E-01	3.20E-02	3.60E-02	3.92E-02	3.60E-02	3.40E-02	3.30E-02	3.43E-02	3.43E-02	3.43E-02	1.63E-01	7.73E-02	7.73E-02	4.4E-01	N/A	
TC	5.49E-04	5.74E-04	4.85E-04	5.45E-04	5.18E-04	5.18E-04	5.08E-04	5.27E-04	5.27E-04	5.27E-04	5.27E-04	N/A	N/A	N/A	N/A	
TIC	5.60E-04	5.60E-04	5.60E-04	5.60E-04	5.60E-04	5.25E-03	5.99E-03	5.30E-03	5.30E-03	5.30E-03	5.66E-02	4.79E-04	4.79E-04	1.1E-01	N/A	
TDC	5.30E-04	5.30E-04	5.30E-04	4.3E-04	4.3E-04	4.59E-04	4.69E-04	4.79E-04	4.79E-04	4.79E-04	3.32E-03	8.59E-04	8.59E-04	5.9E-01	N/A	
Wt% Solids	1.39E-01	N/A	1.69E-01	N/A	1.37E-01	N/A	N/A	1.49E-01	1.49E-01	1.49E-01	1.77E-02	N/A	N/A	N/A	N/A	

* Envelope D Specification for NO3+NO2, NO3 Ave/Spec + (Average NO2/Average NO3)/Spec

Table 4.22 Wet Chemistry Analysis--Original Material
C106 Privatization Contractor Sample

Analyte	Process Blank 96-5892-PB	SPDL1-C106-047		SPDL1-C106-048		SPDL1-3016-049		Average	Stddev	Envelope D Specification	Comparison	
	ug/g	96-5892	96-5892 Dup	96-5893	96-5893 Dup	96-5894	96-5894 Dup	ug/g	ug/g	ug/g	Ave/Spec	Flag
Water Leach												
Br	< 2.4E+01	< 1.07E+02	< 9.30E+01	< 8.69E+01	< 1.31E+02	< 9.24E+01	< 9.84E+01	< 1.31E+02	N/A	N/A	N/A	
C2O4	< 4.8E+01	2.07E+05	1.97E+05	1.91E+05	1.90E+05	2.17E+05	1.98E+05	2.00E+05	1.04E+04	N/A	N/A	
Cl	3.47E+01	2.41E+02	2.11E+02	2.14E+02	2.36E+02	2.07E+02	2.45E+02	2.26E+02	1.69E+01	2.58E+03	8.74E-02	
F	< 2.4E+01	< 2.66E+03	< 2.33E+03	< 2.17E+03	< 3.27E+03	< 2.31E+03	< 2.46E+03	< 3.27E+03	N/A	2.83E+04	1.16E-01	
NH3-N	2.60E+01	3.20E+01	1.90E+01	1.80E+01	2.60E+01	1.50E+01	1.70E+01	2.12E+01	6.49E+00	1.06E+04	2.0E-03	
NO2	< 4.8E+01	2.64E+04	2.47E+04	2.43E+04	2.36E+04	1.80E+04	2.51E+04	2.37E+04	2.94E+03			
NO3	1.28E+02	1.21E+03	1.12E+03	1.14E+03	1.22E+03	1.06E+03	1.29E+03	1.17E+03	8.28E+01	2.89E+05	4.1E-03	
PO4	< 4.8E+01	1.13E+03	1.07E+03	1.04E+03	1.29E+03	1.05E+03	1.20E+03	1.13E+03	9.87E+01	N/A	N/A	
SO4	5.75E+01	7.13E+03	7.25E+03	7.57E+03	7.78E+03	4.38E+03	7.80E+03	6.99E+03	1.30E+03	N/A	N/A	
Direct												
CN	3.40E-01	7.08E+01	5.84E+01	6.50E+01	6.29E+01	6.11E+01	5.56E+01	6.23E+01	5.32E+00	1.29E+04	4.8E-03	
Hg (CVAA)	< 1.0E-02	1.80E+02	1.60E+02	1.60E+02	1.60E+02	1.50E+02	1.50E+02	1.60E+02	1.10E+01	7.73E+02	2.07E-01	
ug/g												
TC		8.69E+04	8.20E+04	8.34E+04	7.86E+04	8.29E+04	8.51E+04	8.31E+04	2.85E+03	N/A	N/A	
THC		2.94E+04	2.47E+04	2.75E+04	2.69E+04	2.85E+04	2.92E+04	2.77E+04	1.76E+03	4.79E+04	5.78E-01	
TDC		5.75E+04	5.72E+04	5.59E+04	5.17E+04	5.44E+04	5.59E+04	5.54E+04	2.16E+03	8.56E+04	6.48E-01	
ug/g												
WT% Solids		N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	

* Envelope D Specification for NO3+NO2; NO3 Ave/Spec = (Average NO2+Average NO3)/Spec

Table 4.23 Radiochemical Analysis--Vendor Product
C106 Privatization Contractor Sample

Analyte	Process Blank (106-6103-DP)	KOH Fusion Utilizing Ni Crucibles										Envelope D Specification UC20g	Comparison Ave(Spec) Flag
		SPD-C106-047 96-6103	SPD-C106-048 96-6104	SPD-C106-049 96-6105	SPD-C106-047 96-6103	SPD-C106-048 96-6104	SPD-C106-049 96-6105	Average UC19	Stddev UC19	Average UC19	Stddev UC19		
Ag-110 m	< 5 E-03	< 8 E-02	< 9 E-02	< 9 E-02	< 8 E-02	< 9 E-02	< 9 E-02	< 9 E-02	N/A	< 9 E-02	N/A	2.58E-04	3.0E-02
Am-241(GEA)	< 2 E-02	2.95E+00	2.95E+00	2.95E+00	2.95E+00	2.95E+00	2.95E+00	3.15E+00	7.3E-01	3.15E+00	7.3E-01	1.11E+03	2.86E-03
Am-241 (AEA)	1.37E-03	2.87E+00	2.88E+00	2.87E+00	2.87E+00	2.87E+00	2.87E+00	2.59E+00	2.77E+00	2.59E+00	2.77E+00	1.11E+03	2.50E-03
Am-243	< 8 E-05	< 2 E-02	< 2 E-02	< 2 E-02	< 2 E-02	< 2 E-02	< 2 E-02	< 2 E-02	N/A	< 2 E-02	N/A	1.29E-01	1.55E-01
C-14	N/A	1.40E-02	7.10E-04	3.00E-04	2.50E-04	1.40E-03	5.50E-04	2.07E-03	5.5E-03	2.07E-03	5.5E-03	5.15E-02	5.57E-02
Ca(Pt)-144	< 3 E-02	< 2 E+00	< 2 E+00	< 2 E+00	< 2 E+00	< 2 E+00	< 2 E+00	< 2 E+00	N/A	< 2 E+00	N/A	2.58E+00	7.76E-01
Ca(Pt)-144	< 3 E-02	< 2 E+00	< 2 E+00	< 2 E+00	< 2 E+00	< 2 E+00	< 2 E+00	< 2 E+00	N/A	< 2 E+00	N/A	2.58E+00	7.76E-02
Ca(Pt)-144 (144m)	< 1 E-03	< 2 E-01	1.23E-01	7.15E-02	8.00E-02	1.05E-01	8.12E-02	9.86E-02	2.2E-02	9.86E-02	2.2E-02	2.58E+05	3.03E+03
Cm-(242/244)	< 2 E-05	7.30E-03	1.00E-02	3.80E-03	7.30E-03	7.03E-03	6.90E-03	8.07E-03	1.67E-03	8.07E-03	1.67E-03	9.53E-01	8.46E-03
Cm-242(Am-242/242m)	< 2 E-05	7.30E-03	1.00E-02	3.80E-03	7.30E-03	7.03E-03	6.90E-03	8.07E-03	1.67E-03	8.07E-03	1.67E-03	7.99E-01	1.01E-02
Cm-242(Am-242/242m)	< 2 E-05	7.30E-03	1.00E-02	3.80E-03	7.30E-03	7.03E-03	6.90E-03	8.07E-03	1.67E-03	8.07E-03	1.67E-03	8.24E-01	9.78E-03
Cm-242(Am-242/242m)	< 4 E-03	4.88E-01	4.67E-01	5.30E-01	4.97E-01	4.94E-01	5.24E-01	4.91E-01	3.74E-02	4.91E-01	3.74E-02	7.73E+01	6.36E-03
Co-60	1.37E-01	5.16E-02	5.12E-02	5.20E-02	4.99E-02	4.61E-02	4.65E-02	4.96E-02	2.62E-01	4.96E-02	2.62E-01	7.73E+04	6.41E-03
Co(Ba)-137	< 4 E-03	< 3 E-01	< 3 E-01	< 3 E-01	< 3 E-01	< 3 E-01	< 3 E-01	< 3 E-01	4.96E-02	< 3 E-01	4.96E-02	7.73E+04	6.41E-03
Co-134	N/A	2.98E-03	2.97E-03	3.00E-03	2.86E-03	2.67E-03	2.70E-03	2.86E-03	1.5E-04	2.86E-03	1.5E-04	1.75E-02	1.71E-03
Co-135	< 2 E-02	< 2 E+00	< 2 E+00	< 2 E+00	< 2 E+00	< 2 E+00	< 2 E+00	< 2 E+00	N/A	< 2 E+00	N/A	3.86E+00	5.18E-01
Eu-152	< 2 E-02	3.61E+00	3.65E+00	3.89E+00	3.96E+00	3.96E+00	3.76E+00	3.76E+00	1.3E-01	3.76E+00	1.3E-01	4.17E+02	9.20E-03
Eu-154	< 2 E-02	3.21E+00	3.22E+00	2.68E+00	3.62E+00	3.01E+00	3.02E+00	3.15E+00	3.1E-01	3.15E+00	3.1E-01	2.32E+02	1.35E-02
Fe-55	< 9 E-03	1.50E-01	1.67E-01	1.45E-01	1.54E-01	1.47E-01	1.50E-01	1.53E-01	7.9E-03	1.53E-01	7.9E-03	2.58E+01	5.94E-03
Ni-59 (1)	< 3 E-02	< 2 E-02	1.56E-02	< 3 E-02	1.78E-02	2.15E-02	2.70E-02	2.05E-02	5.0E-03	2.05E-02	5.0E-03	3.61E-01	5.68E-02
Ni-63 (1)	< 2 E-03	2.41E+00	2.32E+00	2.48E+00	2.18E+00	2.44E+00	2.59E+00	2.40E+00	1.4E-01	2.59E+00	1.4E-01	4.12E+01	5.83E-02
Nb-93m	< 2 E-01	8.95E-02	1.48E-01	2.16E-01	4.97E-01	2.10E-01	2.84E-01	2.34E-01	1.3E-01	2.84E-01	1.3E-01	2.24E+00	1.04E-01
Pu-238	1.29E-04	6.01E-01	6.10E-01	6.59E-01	6.59E-01	6.59E-01	6.02E-01	6.31E-01	2.9E-02	6.31E-01	2.9E-02	2.83E+00	2.23E-01
Pu-(238/240)	5.9E-04	2.88E+00	2.92E+00	3.39E+00	2.99E+00	2.95E+00	2.87E+00	2.95E+00	1.6E-01	2.95E+00	1.6E-01	2.45E+01	1.22E-01
Pu-(239/240)	2.02E-02	7.19E+00	6.45E+00	7.61E+00	6.55E+00	6.92E+00	6.97E+00	6.95E+00	4.1E-01	6.95E+00	4.1E-01	6.70E+00	4.46E-01
Pu-242	< 1 E-04	< 2 E-02	< 9 E-03	< 1 E-02	< 1 E-02	< 1 E-02	< 7 E-03	< 2 E-02	N/A	< 2 E-02	N/A	1.78E-02	3.91E-02
Ru(Rh)-108	< 4 E-02	< 3 E-00	< 3 E-00	< 3 E-00	< 3 E-00	< 3 E-00	< 3 E-00	< 3 E-00	N/A	< 3 E-00	N/A	5.15E+00	5.62E-01
Ru(Rh)-108	< 2 E-02	< 2 E+00	< 2 E+00	< 2 E+00	< 2 E+00	< 2 E+00	< 2 E+00	< 2 E+00	N/A	< 2 E+00	N/A	2.58E+02	7.76E-03
Sm(Te)-125m-125	< 1.5E-03	< 1.4E-03	< 2.0E-03	< 2.4E-03	< 1.3E-03	< 1.6E-03	< 1.8E-03	< 2.4E-03	N/A	< 2.4E-03	N/A	1.00E-02	2.22E-01
Sm-70	< 1.5E-03	< 1.4E-03	< 2.0E-03	< 2.4E-03	< 1.3E-03	< 1.6E-03	< 1.8E-03	< 2.4E-03	N/A	< 2.4E-03	N/A	1.00E-02	2.22E-01
Sm-79 (2)	< 1.5E-04	< 1.1E-04	< 1.5E-04	< 1.7E-04	< 1.1E-04	< 1.4E-04	< 1.6E-04	< 2.1E-04	4.9E-00	< 2.1E-04	4.9E-00	2.40E-03	3.95E-02
Sm-151(Pm-147)	1.42E-02	8.77E+01	8.72E+01	9.34E+01	9.95E+01	8.97E+01	9.69E+01	9.25E+01	4.9E-00	9.25E+01	4.9E-00	4.12E+03	2.24E-02
Sm-151(Pm-147)	< 8 E-03	< 4 E-03	< 8 E-03	< 5 E-03	< 4 E-03	< 5 E-03	< 6 E-03	< 1 E-02	N/A	< 6 E-03	< 1 E-02	4.84E-02	2.06E-01

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Table 4.23 Radiochemical Analysis—Vendor Product
C-106 Privatization Contractor Sample

Analyte	KOH Fusion Utilizing Ni Crucibles										Envelope D Specification		Comparison	
	Process Blank 96-6103 PB	SPD1-C106-047 96-6103	SPD1-C106-048 96-6104	SPD1-C106-049 96-6105 Dup	Average	Slidev	Average	Slidev	Average	Slidev	Specification	Ave/Spec	Ave/Spec	Flag
Sn-113 (In-113m)	< 2E-02 < 9E-02	< 3E-02 9.05E-01	< 4E-02 1.55E+00	< 4E-02 2.07E+00	< 4E-02 1.95E+00	< 2E-02 9.90E-01	< 1E-02 1.25E+00	< 4E-02 5.3E-01	< 1E-02 1.25E+00	< 4E-02 5.3E-01	4.84E-02 2.58E-04 2.32E-01	2.06E-01 1.55E-02 5.30E+00	1.55E-02 5.30E+00	.
Sn-126 (Sb-126m)(Sb-126)	< 6E-02	7.97E-03	1.30E-02	1.62E-02	1.34E-02	9.91E-03	1.15E-02	3.2E-03	1.15E-02	3.2E-03	1.24E+00	9.33E-03	1.31E-02	
Sn-126 (Sb-126m)(Sb-126)	4.31E-02	7.44E-02	6.57E-02	7.91E-02	7.17E-02	7.45E-02	7.30E-02	4.4E-01	7.30E-02	4.4E-01	7.99E-04	9.15E-03	9.15E-03	
Sn-90														
Sn-90														
Total Alpha	2.44E-03	6.87E-02	7.15E+00	6.70E+00	6.50E+00	6.50E+00	6.70E+00	2.2E-01	6.70E+00	2.2E-01	N/A	N/A	N/A	N/A
Total Beta	2.20E-01	2.06E+03	2.25E+03	2.03E+03	1.98E+03	1.89E+03	2.05E+03	1.2E+02	2.05E+03	1.2E+02	N/A	N/A	N/A	N/A
Zr-93	< 3E-04	< 2E-01	< 6E-01	< 2E-01	< 5E-01	< 3E-01	< 6E-01	N/A	< 6E-01	N/A	3.61E+00	1.66E-01	1.66E-01	
Ce-133 (Mass Spec)	N/A	1.73E-01	1.53E+01	1.72E+01	1.48E+01	1.30E+01	1.57E+01	1.4E+00	1.57E+01	1.4E+00	N/A	N/A	N/A	

(1) Ni-59 & Ni-63 sodium peroxide fusion utilizing Zr crucibles

(2) Sn-79 Acid Digestion

N/A Not Applicable

Table 4.24 Radiochemical Analysis--Original Material
C106 Privatization Contractor Sample

Analyte	KOH Fusion Utilizing Ni Crucibles										Envelope D Specification	Comparison	
	Process Blank 96-5892-PB	SPDL1-C106-003		SPDL1-C106-004		SPDL1-C106-005		Average	Stdev				
	uCi/g	96-5892	96-5892 Dup	96-5893	96-5893 Dup	96-5894	96-5894 Dup			uCi/g	uCi/g	Ave/Spec	Flag
Ag-110 m	< 1.E-02	< 6.E-02	< 6.E-02	< 7.E-02	< 6.E-02	< 6.E-02	< 7.E-02	< 7.E-02	N/A	2.58E-04	2.72E+02	.	
Am-241(GEA)	< 3.E-02	1.56E+00	1.66E+00	1.08E+00	1.99E+00	1.46E+00	1.53E+00	1.55E+00	3.0E-01	1.11E+03	1.40E+03	.	
Am-241 (AEA)	1.75E-03	1.35E+00	1.19E+00	1.31E+00	1.26E+00	1.27E+00	1.35E+00	1.29E+00	6.2E-02	1.11E+03	1.16E+03	.	
Am-243	< 4E-05	< 9E-03	< 2E-02	< 2E-02	< 9E-03	< 1E-02	< 2E-02	< 2E-02	N/A	1.29E-01	1.55E-01	.	
C-14	N/A	2.40E-02	3.20E-03	1.10E-03	6.30E-04	1.00E-02	1.90E-03	6.81E-03	9.1E-03	5.15E-02	1.32E-01	.	
Ce(Pu-144 +144m)	< 6.E-02	< 2.E+00	< 2.E+00	< 2.E+00	< 2.E+00	< 2.E+00	< 2.E+00	< 2.E+00	N/A	2.58E+00	7.76E-01	.	
Ce(Pu-144 +144m)								< 2.E+00	N/A	2.58E+00	7.76E-01	.	
Ce(Pu-144 +144m)								< 2.E+00	N/A	2.58E-03	7.76E+02	.	
Cm-(243/244)	1.47E-03	2.76E-02	2.32E-02	2.27E-02	2.32E-02	4.53E-02	2.83E-02	2.84E-02	8.6E-03	2.58E-05	1.10E+03	.	
Cm-(243/244)								2.84E-02	8.6E-03	2.40E+01	1.18E-03	.	
Cm-242 (Am-242/242m)	< 6.E-06	3.62E-03	1.32E-03	3.98E-03	2.55E-03	3.82E-03	4.31E-03	3.27E-03	1.1E-03	9.53E-01	3.43E-03	.	
Cm-242(Am-242/242m)								3.27E-03	1.1E-03	7.99E-01	4.09E-03	.	
Cm-242(Am-242/242m)								3.27E-03	1.1E-03	8.24E-01	3.96E-03	.	
Co-60	< 1.E-02	2.12E-01	2.34E-01	2.44E-01	2.39E-01	2.22E-01	2.62E-01	2.36E-01	1.7E-02	7.73E-01	3.05E-03	.	
Cs(Ba)-137	1.80E-01	5.24E+02	4.93E+02	5.11E+02	5.25E+02	5.06E+02	5.31E+02	5.15E+02	1.4E+01	7.73E+04	6.66E-03	.	
Cs(Ba)-137								5.15E+02	1.4E+01	7.73E+04	6.66E-03	.	
Cs-134	< 9.E-03	< 3.E-01	< 3.E-01	< 3.E-01	< 3.E-01	< 3.E-01	< 3.E-01	< 3.E-01	N/A	1.75E+02	1.71E-02	.	
Cs-135	N/A	3.05E-03	2.84E-03	2.91E-03	3.00E-03	2.98E-03	2.99E-03	2.96E-03	7.5E-05	7.73E-01	3.83E-03	.	
Eu-152	< 3.E-02	< 2.E+00	< 2.E+00	< 2.E+00	< 2.E+00	< 2.E+00	< 2.E+00	< 2.E+00	N/A	3.86E+00	5.18E-01	.	
Eu-154	< 2.E-02	1.68E+00	1.46E+00	1.64E+00	1.41E+00	1.64E+00	1.53E+00	1.56E+00	1.1E-01	4.12E+02	3.78E-03	.	
Eu-155	< 3.E-02	1.80E+00	1.13E+00	1.04E+00	1.07E+00	1.30E+00	1.30E+00	1.23E+00	3.0E-01	2.32E+02	5.28E-03	.	
Fe-55	< 1E-02	5.86E-02	6.75E-02	4.94E-02	4.22E-02	6.69E-02	9.03E-02	6.25E-02	1.7E-02	2.58E+01	2.43E-03	.	
Ni-59 (1)	< 3.E-02	< 2.E-02	< 3.E-02	< 2.E-02	< 2.E-02	< 2.E-02	< 3.E-02	< 3.E-02	N/A	3.61E-01	8.32E-02	.	
Ni-63 (1)	< 9.E-04	1.13E+00	1.21E+00	1.28E+00	1.21E+00	1.06E+00	1.10E+00	1.17E+00	8.2E-02	4.12E+01	2.83E-02	.	
Nb-93m				Not Analyzed						2.24E+00	0.00E+00	.	
Pu-238	1.13E-03	3.48E-01	3.16E-01	3.21E-01	3.26E-01	4.41E-01	3.32E-01	3.47E-01	4.7E-02	2.83E+00	1.23E-01	.	
Pu-(239/240)	1.32E-03	1.60E+00	1.51E+00	1.52E+00	1.59E+00	1.56E+00	1.38E+00	1.53E+00	8.0E-02	2.45E+01	6.24E-02	.	
Pu-(239/240)								1.53E+00	8.0E-02	6.70E+00	2.28E-01	.	
Pu-241	6.81E-02	5.70E+00	2.87E+00	2.97E+00	3.24E+00	4.23E+00	3.04E+00	3.68E+00	1.1E+00	1.78E+02	2.07E-02	.	
Pu-242	< 1E-05	< 1E-02	< 3E-03	< 5E-03	< 9E-03	< 9E-03	< 2E-02	< 2.E-02	N/A	1.83E-03	1.09E+01	.	
Ru(Rh)-106	< 8.E-02	< 3.E+00	< 3.E+00	< 3.E+00	< 3.E+00	< 3.E+00	< 3.E+00	< 3.E+00	N/A	5.15E+00	5.82E-01	.	
Ru(Rh)-106								< 3.E+00	N/A	5.15E+00	5.82E-01	.	
Sb(Te-125m)-125	< 3.E-02	< 2.E+00	< 2.E+00	< 2.E+00	< 2.E+00	< 2.E+00	< 2.E+00	< 2.E+00	N/A	2.58E+02	7.76E-03	.	
Sb(Te-125m)-125								< 2.E+00	N/A	7.73E-01	2.59E-02	.	
Se-79	< 2.0E-03	< 1.9E-03	< 1.3E-03	< 1.8E-03	< 2.4E-03	< 2.0E-03	< 1.7E-03	< 2.4E-03	N/A	1.08E-02	2.22E-01	.	
Se-79 (2)	< 1.4E-04	< 2.0E-04	< 1.9E-04	< 1.5E-04	< 1.6E-04	< 1.6E-04	< 1.6E-04	< 2.0E-04	N/A	1.08E-02	1.85E-02	.	
Sm-151(Pm-147)	1.31E-02	4.99E+01	4.19+01	3.77E+01	3.61E+01	5.70E+01	4.94+01	4.52E+01	1.0E+01	2.40E+03	1.89E-02	.	
Sm-151(Pm-147)								4.52E+01	1.0E+01	4.12E+03	1.10E-02	.	
Sn-113 (In-113m)	< 3.E-02	< 3.E-02	< 3.E-02	< 3.E-02	< 3.E-02	< 3.E-02	< 3.E-02	< 3.E-02	N/A	4.84E-02	6.19E-01	.	

Table 4.24 Radiochemical Analysis--Original Material
C106 Privatization Contractor Sample

Analyte	KOH Fusion Utilizing Ni Crucibles								Envelope D Specification uCi/g	Comparison	
	Process Blank 96-5892-PB	SPDL1-C106-003 96-5892 96-5892 Dup		SPDL1-C106-004 96-5893 96-5893 Dup		SPDL1-C106-005 96-5894 96-5894 Dup		Average	Stddev		
	uCi/g	uCi/g		uCi/g		uCi/g		uCi/g		Ave/Spec	Flag
Sn-113 (In-113m)								< 3.E-02	N/A	4.84E-02	6.19E-01
Sn-113m	< 2.E-01	< 4.E-01	< 3.E-01	< 4.E-01	< 3.E-01	< 4.E-01	< 4.E-01	< 4.E-01	N/A	2.50E-04	1.55E+03
Sn-121m	< 2.E+00	< 2.E+00	< 3.E+00	< 3.E+00	< 2.E+00	< 3.E+00	< 3.E+00	< 3.E+0	N/A	2.32E-01	1.29E+01
Sn-126 (Sb-126m)(Sb-126)	< 2.E-02	< 2.E-02	< 2.E-02	< 3.E-02	< 2.E-02	< 2.E-02	< 3.E-02	< 3.E-02	N/A	1.24E-01	2.43E-01
Sn-126 (Sb-126m)(Sb-126)								< 3.E-02	N/A	8.84E-01	3.40E-02
Sn-126 (Sb-126m)(Sb-126)								< 3.E-02	N/A	1.24E-01	2.41E-01
Sr(Y)-90	9.08E-02	3.60E+02	3.35E+02	3.58E+02	3.50E+02	3.65E+02	3.57E+02	3.54E+02	1.1E+01	7.99E+04	4.43E-03
Sr(Y)-90								3.54E+02	1.1E+01	7.99E+04	4.43E-03
Total Alpha	< 5.E-03	2.83E+00	2.80E+00	3.05E+00	3.01E+00	3.87E+00	3.67E+00	3.21E+00	4.5E-01	N/A	N/A
Total Beta	4.34E-01	1.29E+03	1.17E+03	1.19E+03	1.22E+03	1.19E+03	1.24E+03	1.22E+03	4.4E+01	N/A	N/A
Zr-93	< 2.E-03	5.78E-02	7.63E-02	4.15E-02	3.25E-02	4.59E-02	2.91E-02	4.72E-02	1.8E-02	3.61E+00	1.31E-02
	ug/g	ug/g		ug/g		ug/g		ug/g		ug/g	Flag
Ca-133 (Mass Spec)	N/A	1.53E+01	1.39E+01	1.54E+01	1.64E+01	1.53E+01	2.88E+01	1.75E+01	5.6E+00	N/A	N/A

(1) Ni-59 & Ni-63 sodium peroxide fusion utilizing Zr crucibles

(2) Se-79 Acid Digestion

N/A Not Applicable

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Table 4.25 Decant Liquid--Vendor Product
C106 Privatization Contractor Sample

Analyte	Decant Liquid			Analyte	Decant Liquid		
	Process Blank 96-6654-PB	C106-046-DL 96-6654	C106-046-DL 96-6654 Dup		Process Blank 96-6654-PB	C106-046-DL 96-6654	C106-046-DL 96-6654 Dup
	ug/ml	ug/ml	ug/ml		ug/ml	ug/ml	ug/ml
Ag	—	—	—	Am-241	<4E-04	<7E-03	<7E-03
Al	[6.3]	64.6	61.8	Co-60	<2E-04	<7E-04	<5E-04
As	—	—	—	Cs-137	3.62E-03	2.34E+00	2.30E+00
B	62.9	68.4	61.7	Eu-154	<3E-04	<2E-03	<8E-04
Ba	—	—	—	Eu-155	<3E-04	<5E-03	<5E-03
Be	—	—	—	Sr-90	1.91E-03	3.59E-02	4.84E-02
Bi	—	—	—	Total Alpha	4.42E-04	<4E-03	<4E-03
Ca	[47]	[22]	[9]	Tc-99	2.55E-04	4.93E-04	5.36E-04
Cd	—	—	—	Analyte	ug/ml	ug/ml	ug/ml
Ce	—	—	—	TIC	127	559	587
Co	—	—	—	TOC	1.214	6,645	7,134
Cr	[0.87]	[1.8]	[1.8]	TC	N/A	7,204	7,721
Cu	—	—	—	Analyte	ug/ml	ug/ml	ug/ml
Dy	—	—	—	Br	N/A	<10.0	<10.0
Eu	—	—	—	Cl	N/A	20	20
Fe	[2.5]	[3.7]	[2.3]	F	N/A	<250.0	<250.0
K	—	—	—	NO2	N/A	390	400
La	—	—	—	NO3	N/A	60	60
Li	—	—	—	PO4	N/A	20	20
Mg	[8.0]	[5.0]	—	SO4	N/A	80	80
Mn	—	—	—	C2O4	N/A	24,000	25,000
Mo	—	—	—				
Na	86.9	14,200	13,800				
Nd	—	—	—				
Ni	[1.2]	—	—				
P	—	24.8	28.0				
Pb	—	—	—				
Pd	—	—	—				
Rh	—	—	—				
Ru	—	—	—				
Sb	—	—	—				
Se	—	—	—				
Si	263	234	168				
Sn	—	—	—				
Sr	—	—	—				
Te	—	—	—				
Th	—	—	—				
Ti	—	—	—				
Tl	—	—	—				
U	—	—	—				
V	—	—	—				
W	—	—	—				
Y	—	—	—				
Zn	[2.7]	—	—				
Zr	—	—	—				

— Analyte not detected above the IDL

Results in brackets are qualified but they are included in the calculation of the average.

N/A Not Applicable

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Table 4.26 Centrifuged Solids/Decant Liquid-Original Material
C106 Privatization Contractor Sample

Analyte	Centrifuged Solid						Decant Liquid	
	KOH/Ni Fusion			Na2O2/Zr Fusion			Acid Digestion	
	Process Blank 96-5946-PB.3	C106-002-CS 96-5946-PB.3	96-5946-PB.3	Process Blank 96-5946-PB.3	C106-002-CS 96-5946-PB.3	96-5946-PB.3	Process Blank 96-5946-PB.3	C106-002-DL 96-5946-PB.3
Analyte	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/mL	ug/mL
Ag		[84]	1,110	[102]	[233]	454	[6.4]	[5.5]
Al	[979]	46,300	64,200		50,496	52,799	[5.4]	[1.3]
As								
B		[147]	[193]				58.2	53.0
Ba		315	385		289	329		
Be								
Bi		[553]	[560]		[274]	[227]		
Ca	[1,180]	[3,070]	[2,560]	4,342	4,071	5,640		[10]
Cd		[73]	[78]		[83]	[64]		
Ce				[954]	[824]	[1,277]		
Co								
Cr		929	1,010	[38]	713	809		[1.2]
Cu	[84]	[159]	[170]	[57]	[135]	[163]		[2.2]
Dy								
Eu								
Fe	[200]	107,000	127,000	548	94,569	106,207	[0.86]	[2.9]
K	N/U	N/U	N/U	[4,685]	[5,072]	[5,493]		[360]
La		[89]	[97]		[98]	[92]		
Li								
Mg		[521]	[598]		[439]	[431]		[4.8]
Mn	[555]	2,520	2,940		1,888	2,128		
Mo								
Nb	3,500	163,000	183,000	N/U	N/U	N/U	73.4	[9.4]
Ni	[185]	[333]	[345]	[155]	[225]	[251]		
P	N/U	N/U	N/U	490	1,170	1,383	[2.5]	[6.5]
Pb	[208]	4,390	4,700	[192]	4,373	4,368		130
Pd		2,190	2,010		3,124	3,808		[5.5]
Rh								
Ru								
Sb								
Se								
Si		22,700	43,500		27,427	25,007	[79]	[73]
Sn					[1,781]			
Sr		[50]	[37]	[56]	[67]	[88]		
Ta								
Tb								
Ti		[404]	[453]		391	[433]		
Tl								
U		[3,910]		[3,092]	[3,281]	[4,086]		842
V								
W								
Y					[50]			
Zn		[140]	[209]		[89]	[104]		
Zr		[184]	934	N/U	N/U	N/U		90.9
Analyte	Water Leach ug/g						Direct ug/mL	
Br	<20	<1000	<1000				N/A	<25.0
Cl	120	<1000	<1000				N/A	<250
F	40	<3000	<3000				N/A	<250
NO2	<50	8,000	14,000				N/A	12,200
NO3	290	<2000	<2000				N/A	460
PO4	<50	<2000	2,000				N/A	240
SO4	140	3,000	5,000				N/A	3,300
C2O4	80	228,000	137,000				N/A	6,500

- Analyte not detected above the IOL

N/U Not Usable

N/A Not Applicable

Results in brackets are qualified but they are included in the calculation of the average.

APPENDIX A

ICP-MS DATA QUALIFICATIONS AND OBSERVATIONS – VENDOR PRODUCT

Selenium

There were significant disparities observed between the different preparations for nearly every element. One element for which this was especially apparent was selenium. It was not immediately clear if the software misinterpreted the mass spectra when integrating and calculating, but none of the spectra for any of the preparations indicate a measurable quantity of selenium. The isotope used for these calculations (m/z 78) yielded the best calibration curve; however, it is typically interfered with by the $^{38}\text{Ar}^{40}\text{Ar}$ dimer.

Vanadium

There were also disparities observed between vanadium results. Visual inspection of the data indicated ClO^+ interference peaks for both fusion preparations; the presence of this interference in the acid digestion preparation could not be confirmed.

Mass 93 (Zr/Nb) and Mass 241 (Pu/Am)

The ICP-MS m/z 93 (probably Nb) results also include a contribution from zirconium, since there is an interference from zirconium at m/z 93 by ICP-MS. Resolution of the niobium and zirconium is not complete. The same interference problem is experienced at m/z 241 with plutonium and americium. Therefore, only one value is reported for m/z 93 and m/z 241; possible/probable isotopes contributing to the response observed at these masses are zirconium/niobium and plutonium/americium, respectively. However, it should be noted that $^{93\text{m}}\text{Nb}$, ^{93}Zr , ^{241}Pu , and ^{241}Am were measured separately by conventional radiochemistry techniques.

Mass 242 (Pu)

While there appear to be measurable peaks at m/z 240 and m/z 241, the values reported for m/z 242 are estimated quantitation levels. The lowest ^{241}Pu standard analyzed was 0.1 ppb, which would amount to approximately 0.125 ppm in the sample. Because a response was obtained for this standard, 0.1 ppm is a reasonable estimate for a lower limit of detection/quantitation for ^{242}Pu .

Rubidium

The rubidium results on the KOH fusions are questionable. Based on the preparation blank result, it is possible that the KOH was contaminated with rubidium; multiplying by a total dilution factor of 25,000 would amplify this affect. The other two sample preparation results agreed reasonably well, and did not have as high a dilution factor, making those values more reliable.

Palladium-107

The ^{107}Pd results show a questionably high concentration in the Na_2O_2 fusions. These values (in the mid-200 ppm range) may be due to a ZrO^+ interference. Also, the relatively high concentration of Ag in these samples makes for a very large correction factor, necessarily resulting in a large uncertainty. Between 1-5 ppm ^{107}Pd was observed in the acid digestion samples. Because the silver and zirconium concentrations are significantly lower in the acid digested samples, the results are considered more reliable.

Zirconium

Because the reported value for zirconium was calculated from the response at m/z 90, it includes a contribution from ^{90}Sr , likely making the ICP-MS zirconium value bias high.

Isotopic and Fission-product Considerations

- Ba: The barium isotopic abundance appears to be significantly altered. This is most notable for 138, whereas all isotopes below 137 agree with each other. The m/z 138 was used to measure the Ba concentration, which means that the reported values may be bias high. It is unclear whether the 138 peak is barium or perhaps fission-produced cerium.
- Cu: Copper appears to be completely natural, with no apparent contribution from ^{63}Ni . The reported result was obtained from m/z 65.
- Cs: The cesium values reported are for the concentration of ^{133}Cs ONLY, and does not include ^{133}Cs or ^{137}Cs .
- Pd: There seems to be slight enrichment at 106 and 107, which may be fission-product palladium, or may be ZrO^+ . Confirmation is not possible without a separation; however, little change in the result would be anticipated. The m/z 105 peak used agrees well with m/z 108.
- Ru: The ruthenium present appears to be exclusively fission-produced. The concentration was calculated using the response at m/z 102, with an assumed isotopic abundance of ~47% based on the relative abundance observed of m/z 101 and 102.
- Te: The tellurium present in the samples resembles that of fission-product tellurium. The most apparent indication that at most (if not all) of the tellurium present is fission produced is the presence of a prominent m/z 130 peak, which appears to be almost 5 times that of the m/z 128 peak; for natural abundance the responses should be nearly equal. In addition, the responses observed where the natural lower-mass tellurium isotopes should be found (i.e., m/z 122, m/z 123, m/z 124) appear to be due to natural tin and antimony.
- Zr: The m/z 90 was used in calculating the reported zirconium result, which in retrospect was probably not the best choice due to ^{90}Sr . However when the spectra were reviewed, it was observed that the zirconium abundance appeared to be completely natural, with m/z 90 being just slightly low rather than high. It appears that the results reported are representative of the total zirconium concentration, but with a possible low bias.

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ICP-MS DATA QUALIFICATIONS AND OBSERVATIONS -- ORIGINAL MATERIAL

Mass 93 (Zr/Nb) and Mass 241 (Pu/Am)

As in the Vendor Product, only one value is reported for m/z 93 and 241. The possible/probable isotopes contributing to the response observed at these masses are zirconium/niobium, and plutonium/americiuim, respectively. The m/z-93 results from the Na_2O_2 fusion do not appear to be usable due to high levels of zirconium and possible niobium contamination (from the zirconium). There are large disparities observed between the samples and their duplicates.

Mass 240/242

There appears to be a blank problem at m/z 240 and m/z 242 for the KOH fusion; the concentrations found in the samples are virtually identical to those found in the preparation blank. The acid digestion was analyzed at a lower dilution and has a lower detection limit. Based on this, the acid digestion results are considered the most reliable.

Vanadium

As in the Vendor Product analysis, both fusion preparations appear to have a significant ClO^+ response at m/z 51, which interferes with vanadium. This could not be confirmed with the acid digestion preparation.

Rubidium

The rubidium results on the KOH fusions are questionable, due to an inexplicably high blank result. The suspected cause of the high rubidium concentration in this preparation is contamination.

Palladium-107

The ^{107}Pd was not reported for the Na_2O_2 fusions due to problems observed in obtaining reliable results from this preparation. The ^{107}Pd measured relatively high due to a ZrO^+ interference. Also, the relatively high concentration of silver in these samples makes for a very large correction factor, necessarily resulting in a large uncertainty. Because of lower silver and zirconium concentrations, the acid digestion preparation results are considered more reliable.

Zirconium

The reported value for zirconium was calculated from the response at m/z 90 and included a contribution from ^{90}Sr that made the zirconium value bias high. The zirconium results reported were corrected for this ^{90}Sr contribution, which was found to be approximately 10 ppm.

Isotopic and fission-product considerations

- Ba: The isotopic abundance is significantly altered, most notably for 138; however, all isotopes below 137 agree with each other. For calculations, m/z 138 was used, which means that the reported results may be biased high. It is unclear whether the 138 peak is barium or perhaps fission-produced cerium.
- Cu: Copper appears to be completely natural, with no apparent contribution from ^{63}Ni . Result was obtained from m/z 65.
- Pd: There seems to be slight enrichment at 106 and 107, which may be fission-product palladium, or may be ZrO^+ . This cannot be confirmed without a separation, but it should not make a significant difference even if the additional response were included. The m/z 105 peak used agrees well with m/z 108.
- Ru: The ruthenium present appears to be exclusively fission-produced. The concentration was calculated using the response at m/z 102, with an assumed isotopic abundance of ~47% based on the relative abundance observed of m/z 101 and 102.
- Te: The tellurium present in the samples resembles that of fission-product tellurium. The most apparent indication that most (if not all) of the tellurium present is fission produced is the presence of a prominent m/z 130 peak, which appears to be almost 5 times that of the m/z 128 peak; for natural abundance the responses should be nearly equal. In addition, the responses observed where the natural lower-mass tellurium isotopes should be found (i.e., m/z 122, m/z 123, m/z 124) appear to be due to natural tin and antimony.

**TANK WASTE REMEDIATION SYSTEM (TWRS)
PRIVATIZATION CONTRACTOR**

**ENVELOPE D SAMPLES
(C-106)**

ATTACHMENT 2

**ANALYTICAL SUMMARY REPORT
ADDENDUM 1, REVISION 2**

March 1997

Pacific Northwest National Laboratory

**TANK WASTE REMEDIATION SYSTEM (TWRS) PRIVATIZATION CONTRACTOR
SAMPLES**

**WASTE ENVELOPE D MATERIAL
241-C-106**

ANALYTICAL SUMMARY REPORT

ADDENDUM 1 - Revision 2

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Analytical Chemistry Laboratory (ACL)
C-106 Project Manager**

March 7, 1997

1.0 INTRODUCTION

This report represents Addendum 1 to the Analytical Summary Report on TWRS Privatization Contractor Samples conducted in support of the Tank Waste Remediation System (TWRS) Privatization Contractor Samples, Waste Envelope D, C-106 characterization testing. All work performed was conducted in accordance with "Addendum 1 of the Letter of Instruction (LOI) for TWRS Privatization Contractor Samples Addressing Waste Envelope D Materials - Revision 0, Revision 1, and Revision 2." [Jones 1996, Wiemers 1996 (a), Wiemers 1996 (b)]

The Initial Vendor Product¹ resulting from the treatment defined in the LOI Revision 0 exceeded the Envelope D specifications for Na and ^{121m}Sn. Additionally, a trace component (Pt) and a trace radionuclide (²³⁵U) were found to exceed the envelope specification, and some radionuclides (^{110m}Ag, ²⁴²Pu, and ^{119m}Sn) could not be verified due to analytical detection limitations. Also, the result for ²⁴³Cm/²⁴⁴Cm and ¹⁴⁴Ce/¹⁴⁴Pr/^{144m}Pr were not within the specification; but only if the results are compared to the Envelope D specifications for ²⁴³Cm (as a trace radionuclide) and for ^{144m}Pr, respectively. Primarily due to failure of Na to meet the specification, further treatment of the Initial Vendor Product was requested, as defined by the LOI Revision 1. A decant/dilute treatment was performed to bring the sodium concentration within specification limits; the details of this treatment are described in Section 2.

Following the sodium adjustment treatment, the Final Vendor Product was transferred to twenty individual containers for shipping to the privatization vendors. However, the percent solids and percent oxide measurements performed on this material were inconsistent with expected results. Visual observation of the individual containers noted inconsistent levels of settled solids in each container. Based on these measurements and observations, additional blending, sampling/analyses, and packaging were requested, as defined by the LOI Revision 2. The reblending and repackaging are described in Section 2 and the sampling/analysis results are discussed in Section 4. Table A1-1

¹Within this report "Initial Vendor Product" is used to identify product material resulting from the bench-scale processing (i.e., LOI Rev. 0) and the "Final Vendor Product" is used to identify material resulting from the decant/dilute treatment to reduce the Na content (i.e., LOI Rev. 1 and LOI Rev. 2).

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presents the "best" value for analytes measured in the Initial Vendor Product materials and the measured or "adjusted" (i.e., calculated estimates) values for the Final Vendor Product. The analysis and calculations are further described in Section 3 and Section 4.

Following the decant/dilute treatment the C106 Final Vendor Product is within specification (including sodium), except one inorganic analyte of interest (Pt) and two radionuclides (^{121m}Sn and ^{233}U) have measured concentrations above the Envelope D specification, and three radionuclides (^{110m}Ag , ^{119m}Sn , ^{242}Pu) have analytical detection limits above the specification. And, as with the Initial Vendor Product, $^{243}\text{Cm}/^{244}\text{Cm}$ and $^{144}\text{Ce}/^{144}\text{Pr}/^{144m}\text{Pr}$ exceed the specification if the results are compared to the Envelope D specifications for ^{243}Cm (as a trace radionuclide) and ^{144m}Pr , respectively. Statistical analysis of the product analytical results is reported in the "Preliminary Statistical Report" and "Final Statistical Report" (in progress).

Prior to completing all treatment physical measurements (e.g., weight percent oxide on firing), a preliminary estimate of the Final Vendor Product composition was provided to DOE-RL by Wiemers.² The composition reported by Wiemers was estimated based on a calculated adjusted oxide content (i.e., oxide adjusted for loss of sodium by decanting) and preliminary data for the Initial Vendor Product and Initial Vendor Product supernatant composition. The results provided in this report supersede the composition estimates reported by Wiemers.

²Internal memo, K Wiemers to R Gilbert and N Brown, "Composition of Envelope D Private Contractor Sample," dated 8 November 96. This memo was provided to J Dickey, LMAES by R Gilbert, "Transfer of Envelope D Samples - Contract Number DE-RP06-96RL13309", DOE-RL letter #96-WDD-190, dated 8 Nov 96.

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Table A1-1. Comparison of Adjusted and Measured Final Vendor Product Results with Envelope D Specification

Analysis (t)	Envelope D Spec	Initial Product (IP)		Final Product (FP)		C	Result	FP	FP	FP	Notes
		Avg g/g	Avg g/L (2)	Avg g/g	Avg g/L (2)						
Ag	1.7E-01	3.1E+03	1.23E-01	1.40E-01	1.62E+03	6.01E-02	M	6.01E-02	3.54E-01	N	Detected in Supernate
Am	4.3E+00	5.12E+04	1.99E+00	2.23E+00	6.68E+04	2.48E+00	M	2.48E+00	8.76E-01	N	Detected in Supernate
As	2E-02	—	—	—	—	—	M	2.45E-02	2.85E-02	N	Detected in Supernate
As	5E-02	2.86E+02	1.11E-02	1.26E-02	n/d	n/d	C	1.26E-02	2.51E-01	N	Detected in Supernate
B	4E-01	2.29E+02	8.89E-03	4.05E-04	n/d	n/d	C	4.05E-04	1.01E-03	N	Detected in Supernate
Be	1.4E+00	4.37E+02	1.70E-02	1.92E-02	5.44E+02	2.02E-02	M	2.02E-02	1.44E-02	N	Detected in Supernate
Bi	2E-02	1.10E+00	3.88E+03	4.39E+03	n/d	n/d	C	4.39E+03	4.22E-03	N	Detected in Supernate
Bi	8.6E-01	4.30E+02	1.97E-02	1.85E-02	6.60E+02	2.45E-02	M	2.45E-02	2.85E-02	N	Detected in Supernate
Bi	2.2E+00	3.84E+03	1.49E-01	1.55E-01	4.83E+03	1.79E-01	M	1.79E-01	8.14E-02	N	Detected in Supernate
Cd	1.4E+00	8.06E+01	3.52E-03	3.98E-03	8.40E+01	3.12E-03	M	3.12E-03	2.23E-03	N	Detected in Supernate
Ce	2.3E-01	2.02E+02	7.84E-03	8.87E-03	n/d	n/d	C	8.87E-03	3.55E-02	N	Detected in Supernate
Co	1.4E-01	2.60E+01	1.01E-03	1.14E-03	n/d	n/d	C	1.14E-03	8.16E-03	N	Detected in Supernate
Cr	2.1E-01	7.74E+02	3.00E-02	3.40E-02	8.06E+02	2.98E-02	M	2.98E-02	1.42E-01	N	Detected in Supernate
Cs	1.8E-01	1.80E+01	6.99E-04	7.91E-04	n/m	n/m	C	7.91E-04	4.39E-03	N	Detected in Supernate
Cu	1.5E-01	1.56E+02	6.07E-03	6.87E-03	1.30E+02	4.82E-03	M	4.82E-03	3.22E-02	N	Detected in Supernate
Dy	8E-03	2.02E+01	7.84E-04	8.87E-04	n/d	n/d	C	8.87E-04	1.11E-01	N	Detected in Supernate
Er	1E-03	1.31E+01	5.09E-04	5.75E-04	n/m	n/m	C	5.75E-04	5.75E-01	N	Detected in Supernate
Eu	5E-03	7.86E+00	3.05E-04	3.45E-04	n/m	n/m	C	3.45E-04	8.91E-02	N	Detected in Supernate
Fe	8.9E+00	1.41E+06	5.46E+00	6.18E+00	1.58E+06	5.87E+00	M	5.87E+00	6.60E-01	N	Detected in Supernate
Gd	3E-03	2.50E+01	9.70E-04	1.10E-03	n/m	n/m	C	1.10E-03	3.66E-01	N	Detected in Supernate
Hf	1E-03	8.07E+00	2.38E-04	2.67E-04	n/m	n/m	C	2.67E-04	2.67E-01	N	Detected in Supernate
K	4.1E-01	4.92E+02	1.91E-02	2.16E-02	n/d	n/d	C	2.16E-02	4.27E-02	N	Detected in Supernate
La	8E-01	9.21E+01	3.58E-03	4.05E-03	1.12E+02	4.16E-03	M	4.16E-03	1.19E-03	N	Detected in Supernate
Li	4.3E-02	4.80E+01	1.79E-03	2.02E-03	n/d	n/d	C	2.02E-03	4.70E-02	N	Detected in Supernate
Mg	6.5E-01	1.29E+03	4.89E-02	5.53E-02	1.48E+03	5.48E-02	M	5.48E-02	8.44E-02	N	Detected in Supernate
Mo	2.0E+00	2.81E+03	1.33E-01	1.38E-01	3.22E+03	1.19E-01	M	1.19E-01	5.94E-02	N	Detected in Supernate
Nb	2.0E-01	5.70E+01	2.21E-03	2.50E-03	n/d	n/d	C	2.50E-03	1.25E-02	N	Detected in Supernate
Nb	6.0E+00	1.62E+05	6.29E+00	4.09E+00	1.20E+05	4.45E+00	M	4.45E+00	7.42E-01	N	Detected in Supernate
Ni	3.0E-03	3.38E+01	1.31E-03	1.48E-03	n/m	n/m	C	1.48E-03	4.95E-01	N	Detected in Supernate
Nd	6.3E-01	2.82E+02	1.09E-02	1.24E-02	3.30E+02	1.23E-02	M	1.23E-02	2.32E-02	N	Detected in Supernate
Ni	7.3E-01	1.81E+03	6.23E-02	7.07E-02	1.51E+03	7.07E-02	M	7.07E-02	8.85E-02	N	Detected in Supernate
Np	3E-02	—	—	—	—	—	—	—	—	n/a	See Radionuclide
P	5.4E-01	3.33E+03	1.29E-01	1.35E-01	4.81E+03	1.78E-01	M	1.78E-01	3.30E-01	N	Detected in Supernate
Pb	3.4E-01	3.43E+03	1.33E-01	1.51E-01	1.12E+03	1.53E-01	M	1.53E-01	4.49E-01	N	Detected in Supernate
Pd	4E-02	2.74E+01	1.96E-03	1.20E-03	n/d	n/d	C	1.20E-03	3.01E-02	N	Detected in Supernate
Pm	3E-02	—	—	—	—	—	—	—	—	n/a	See Radionuclide
Pr	1.1E-01	8.41E+01	3.26E-03	3.69E-03	n/m	n/m	C	3.69E-03	3.36E-02	N	Detected in Supernate
Pu	1E-03	3.07E+01	1.19E-03	1.35E-03	n/m	n/m	C	1.35E-03	1.35E+00	N	Detected in Supernate
Pu	1.9E-02	—	—	—	—	—	—	—	—	n/a	See Radionuclide
Rb	8E-02	7.68E+00	2.98E-04	3.37E-04	n/m	n/m	C	3.37E-04	5.62E-03	N	Detected in Supernate
Re	3E-02	6.61E+00	2.57E-04	2.90E-04	n/m	n/m	C	2.90E-04	9.68E-03	N	Detected in Supernate
Rh	4E-02	1.44E+02	5.59E-03	6.33E-03	n/d	n/d	C	6.33E-03	1.58E-01	N	Detected in Supernate
Rh	1.1E-01	9.57E+01	3.71E-03	4.20E-03	n/d	n/d	C	4.20E-03	3.82E-02	N	Detected in Supernate
S	2E-01	—	—	—	—	—	—	—	—	n/a	No Analysis Performed
Sb	2.6E-01	7.30E-01	2.83E-05	3.21E-05	n/d	n/d	C	3.21E-05	1.23E-04	N	Detected in Supernate
Se	1.6E-01	3.69E+02	1.43E-02	1.62E-02	n/d	n/d	C	1.62E-02	1.01E-01	N	Detected in Supernate
Si	5.6E+00	6.89E+04	2.67E+00	3.02E+00	6.86E+04	3.22E+00	M	3.22E+00	5.55E-01	N	Detected in Supernate
Sm	9.3E-02	—	—	—	—	—	—	—	—	n/a	See Radionuclide
Sn	1.1E-02	1.02E+02	3.96E-03	4.45E-03	n/d	n/d	C	4.45E-03	4.07E-01	N	Detected in Supernate
Sr	1.6E-01	1.50E+02	5.82E-03	6.59E-03	1.00E+02	3.71E-03	M	3.71E-03	2.32E-02	N	Detected in Supernate
Ta	8E-03	2.86E+01	1.11E-03	1.28E-03	n/m	n/m	C	1.28E-03	1.57E-01	N	Detected in Supernate
Tb	1E-03	8.20E+00	3.18E-04	3.60E-04	n/m	n/m	C	3.60E-04	3.60E-01	N	Detected in Supernate
Tb	8E-02	—	—	—	—	—	—	—	—	n/a	See Radionuclide
Ta	4E-02	4.24E+02	1.65E-02	1.85E-02	5.06E+02	1.88E-02	M	1.88E-02	4.45E-01	N	Detected in Supernate
Th	1.6E-02	3.49E+02	1.35E-02	1.53E-02	4.28E+02	1.59E-02	M	1.59E-02	9.92E-01	N	Detected in Supernate
Ti	4E-01	1.31E+03	5.10E-02	5.77E-02	1.24E+03	4.59E-02	M	4.59E-02	1.15E-01	N	Detected in Supernate
Ti	1.4E-01	1.23E+02	4.77E-03	5.40E-03	n/d	n/d	C	5.40E-03	3.86E-02	N	Detected in Supernate
Tl	1E-03	2.84E+00	1.03E-04	1.16E-04	n/m	n/m	C	1.16E-04	1.16E-01	N	Detected in Supernate
U	4.2E+00	1.50E+03	7.36E-02	8.33E-02	2.65E+03	9.83E-02	M	9.83E-02	1.34E-01	N	Detected in Supernate
V	1E-02	3.72E+01	1.44E-03	1.63E-03	n/d	n/d	C	1.63E-03	1.63E-01	N	Detected in Supernate
W	7.4E-02	4.98E+02	1.53E-02	2.19E-02	n/d	n/d	C	2.19E-02	2.96E-01	N	Detected in Supernate
Y	5E-02	8.60E+01	3.34E-03	3.74E-03	n/d	n/d	C	3.74E-03	7.56E-02	N	Detected in Supernate
Yb	1E-03	1.26E+01	4.89E-04	5.53E-04	n/m	n/m	C	5.53E-04	5.53E-01	N	Detected in Supernate
Zn	1.3E-01	3.06E+02	1.19E-02	1.34E-02	n/d	n/d	C	1.34E-02	1.03E-01	N	Detected in Supernate
Zr	4.6E+00	1.11E+03	4.29E-02	4.86E-02	1.25E+03	4.65E-02	M	4.65E-02	1.01E-02	N	Detected in Supernate
None	None	1.16E+02	4.80E-03	5.10E-03	n/d	n/d	C	5.10E-03	n/a	n/a	Detected in Supernate
None	None	2.91E+02	1.13E-01	1.28E-01	n/d	n/d	C	1.28E-01	4.59E-08	N	Detected in Supernate
None	None	1.01E+02	4.89E-03	5.40E-03	n/d	n/d	C	5.40E-03	1.34E-02	N	Detected in Supernate
None	None	1.60E+02	6.21E-03	2.83E-03	2.75E-02	1.02E-02	M	1.02E-02	n/a	n/a	Detected in Supernate
None	None	4.70E+02	1.82E-02	3.86E-03	3.25E-02	1.21E-02	M	1.21E-02	n/a	n/a	Detected in Supernate

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Table A1-1. Comparison of Adjusted and Measured Final Vendor Product Results with Envelope D Specification

Analyte(s)	Envelope D Spec	Initial Product (IP) Measured		Final Product (FP) Measured		C or M	FP Result	FP (Result) Spec	FP Spec	Notes
		Atx g/L	Avg g/L (3)	Adjusted g/L	Avg g/L (3)					
D204	None	1.70E+05	8.59E+00	2.51E+00	6.33E+04	3.09E+00	n/a	n/a	n/a	Detected in Supernate
H43	5E-01	2.13E+01	8.25E-04	9.33E-04	n/a	n/a	M	9.33E-04	1.87E-03	N
TIC (TCN)	1.86E+00	3.30E+03	2.06E-01	1.13E-01	n/a	n/a	C	1.13E-01	6.05E-02	N
TOC	5E+00	4.75E+01	1.86E+00	6.84E-01	n/a	n/a	C	6.84E-01	1.94E-01	N
TC	None	5.22E+04	2.03E+00	7.27E-01	n/a	n/a	C	7.27E-01	n/a	N
Radioisotopes										
Ag-110m	1.00E-08	<9E-02	<3.45E-06	<3.95E-06	n/a	n/a	C	<3.95E-06	3.95E+00	-Y-
Am-241	4.30E-02	3.15E+00	1.22E-04	1.38E-04	n/a	n/a	C	1.38E-04	3.22E-03	N
Am-243	5.00E-06	<2E-02	<7.76E-07	<8.79E-07	n/a	n/a	C	<8.79E-07	<1.78E-01	N
Ca-144	2.00E-05	2.87E-03	1.11E-07	1.29E-07	n/a	n/a	C	1.29E-07	6.30E-02	N
Ca-144(Pri-144+144m)	1.00E-04	<2E+00	<7.76E-05	<8.79E-05	n/a	n/a	C	<8.79E-05	<8.79E-01	N
Ca-144(Pri-144+144m)	1.00E-04	<2E+00	<7.76E-05	<8.79E-05	n/a	n/a	C	<8.79E-05	<8.79E-01	N
Ca-144(Pri-144+144m)	1.00E-07	<2E+00	<7.76E-05	<8.79E-05	n/a	n/a	C	<8.79E-05	<8.79E-02	-Y-
Ca-242(AM-242+242m)	3.70E-05	8.07E-03	3.13E-07	3.55E-07	n/a	n/a	C	3.55E-07	8.58E-03	N
Ca-242(AM-242+242m)	3.10E-05	8.07E-03	3.13E-07	3.55E-07	n/a	n/a	C	3.55E-07	1.11E-02	N
Ca-242(AM-242+242m)	3.20E-05	8.07E-03	3.13E-07	3.55E-07	n/a	n/a	C	3.55E-07	1.11E-02	N
Ca-243+244	1.00E-08	9.86E-02	3.83E-06	4.33E-06	n/a	n/a	C	4.33E-06	4.33E+03	-Y-
Ca-243+244	9.30E-04	9.86E-02	3.83E-06	4.33E-06	n/a	n/a	C	4.33E-06	4.66E-03	N
Co-60	3.00E-03	4.91E-01	1.91E-05	2.16E-05	n/a	n/a	C	2.16E-05	7.19E-03	N
Co-134	6.80E-03	<3E-01	<1.18E-05	<1.32E-05	n/a	n/a	C	<1.32E-05	<1.94E-03	N
Co-135	3.00E-05	2.88E-03	1.11E-07	1.26E-07	n/a	n/a	C	1.26E-07	4.19E-03	N
Co-137(Ba-137)	3.00E+00	4.98E+02	1.93E-02	2.13E-02	n/a	n/a	C	2.13E-02	7.10E-03	N
Co-137(Ba-137)	3.00E+00	4.98E+02	1.93E-02	2.13E-02	n/a	n/a	C	2.13E-02	7.10E-03	N
Eu-152	1.50E-04	<2E+00	<7.77E-05	<8.79E-05	n/a	n/a	C	<8.79E-05	<3.86E-01	-Y-
Eu-154	1.60E-02	3.77E+00	1.47E-04	1.66E-04	n/a	n/a	C	1.66E-04	1.04E-02	N
Eu-155	9.00E-03	3.13E+00	1.21E-04	1.37E-04	n/a	n/a	C	1.37E-04	1.53E-02	N
Fe-55	1.00E-03	1.53E-01	5.64E-06	6.72E-06	n/a	n/a	C	6.72E-06	6.72E-03	N
Ir-193	9.00E-08	2.20E-04	9.54E-07	9.68E-08	n/a	n/a	C	9.68E-08	1.07E-01	N
Nb-93m	8.70E-05	2.94E-01	9.08E-06	1.03E-05	n/a	n/a	C	1.03E-05	1.18E-01	N
Ni-59	1.40E-05	2.05E-02	7.98E-07	9.01E-07	n/a	n/a	C	9.01E-07	6.43E-02	N
Ni-63	1.60E-03	2.40E+00	9.32E-05	1.05E-04	n/a	n/a	C	1.05E-04	6.59E-02	N
Np-237	2.30E-05	3.33E-03	1.53E-07	1.73E-07	n/a	n/a	C	1.73E-07	7.51E-03	N
Pd-107	4.00E-06	1.10E-03	4.27E-06	4.83E-06	n/a	n/a	C	4.83E-06	1.21E-02	N
Pu-238	1.10E-04	6.31E-01	2.45E-05	2.77E-05	n/a	n/a	C	2.77E-05	2.92E-01	N
Pu-239	6.50E-04	1.53E+00	5.54E-05	6.72E-05	n/a	n/a	C	6.72E-05	7.07E-02	N
Pu-240	2.60E-04	1.27E-01	4.93E-06	5.68E-06	n/a	n/a	C	5.68E-06	2.15E-02	N
Pu-241	6.90E-03	6.98E+00	2.70E-04	3.06E-04	n/a	n/a	C	3.06E-04	4.43E-02	N
Pu-242	7.10E-08	<2E-02	<7.77E-07	<8.79E-07	n/a	n/a	C	<8.79E-07	<1.24E-01	-Y-
Ru-106(Rh-106)	2.00E-04	<3E+00	<1.16E-04	<1.32E-04	n/a	n/a	C	<1.32E-04	<6.59E-01	N
Ru-106(Rh-106)	2.00E-04	<3E+00	<1.16E-04	<1.32E-04	n/a	n/a	C	<1.32E-04	<6.59E-01	N
Ru-106(Rh-106)	1.00E-02	<2E+00	<7.76E-05	<8.79E-05	n/a	n/a	C	<8.79E-05	<8.79E-03	N
Sb-125(Te-125m)	3.00E-03	<2E+00	<7.76E-05	<8.79E-05	n/a	n/a	C	<8.79E-05	<2.93E-02	N
Sb-125(Te-125m)	4.20E-07	<2E-04	<8.15E-09	<9.22E-09	n/a	n/a	C	<9.22E-09	<2.20E-02	N
Sm-151(Pm-147)	9.30E-02	9.23E+01	3.58E-03	4.05E-03	n/a	n/a	C	4.05E-03	4.36E-02	N
Sm-151(Pm-147)	1.60E-01	8.23E+01	3.58E-03	4.05E-03	n/a	n/a	C	4.05E-03	2.63E-02	N
Sn-113(In-113)	1.88E-06	<1E-02	<3.88E-07	<4.39E-07	n/a	n/a	C	<4.39E-07	<2.34E-01	N
Sn-113(In-113)	1.88E-06	<1E-02	<3.88E-07	<4.39E-07	n/a	n/a	C	<4.39E-07	<2.34E-01	N
Sn-119m	1.00E-08	<4E-02	<1.55E-06	<1.75E-06	n/a	n/a	C	<1.75E-06	<1.75E+02	-Y-
Sn-121m	9.00E-06	1.23E+00	4.77E-05	5.40E-05	n/a	n/a	C	5.40E-05	6.00E+00	-Y-
Sn-126(Sb-126m, Sb-126)	4.80E-05	1.15E-02	4.46E-07	5.05E-07	n/a	n/a	C	5.05E-07	1.05E-02	N
Sn-126(Sb-126m, Sb-126)	3.43E-05	1.15E-02	4.46E-07	5.05E-07	n/a	n/a	C	5.05E-07	1.47E-02	N
Sn-126(Sb-126m, Sb-126)	4.83E-06	1.15E-02	4.46E-07	5.05E-07	n/a	n/a	C	5.05E-07	1.05E-01	N
Sn-126(Sb-126m, Sb-126)	3.10E+00	7.30E+02	2.83E-02	3.21E-02	n/a	n/a	C	3.21E-02	1.03E-02	N
Sn-126(Sb-126m, Sb-126)	3.10E+00	7.30E+02	2.83E-02	3.21E-02	n/a	n/a	C	3.21E-02	1.03E-02	N
Sn-126(Sb-126m, Sb-126)	4.50E-03	1.32E-02	5.12E-07	4.70E-07	n/a	n/a	C	4.70E-07	1.05E-04	N
Th-232	8E-07	5.46E-05	2.13E-09	2.41E-09	n/a	n/a	C	2.41E-09	n/a	N
U-233	1.00E-09	1.70E-03	6.65E-08	7.47E-08	n/a	n/a	C	7.47E-08	7.47E-01	-Y-
U-234	7.70E-07	7.70E-04	2.99E-08	3.38E-08	n/a	n/a	C	3.38E-08	4.39E-02	N
U-235	3.20E-08	2.80E-05	1.09E-09	1.23E-09	n/a	n/a	C	1.23E-09	3.84E-02	N
U-238	8.20E-08	1.80E-05	6.99E-10	7.91E-10	n/a	n/a	C	7.91E-10	9.64E-03	N
U-238	5.80E-07	9.37E-04	2.47E-08	2.80E-08	n/a	n/a	C	2.80E-08	4.92E-02	N
Zr-93	1.40E-04	<6E-01	<2.33E-05	<2.64E-05	n/a	n/a	C	<2.64E-05	<1.88E-01	N
Total Alpha	none	6.78E+00	2.63E-04	2.89E-04	n/a	n/a	C	2.89E-04	n/a	N
Total Beta	none	2.05E+03	7.96E-02	9.01E-02	n/a	n/a	C	9.01E-02	n/a	N
Physical Properties										
		Initial Product		Final Product						
		Min	Max	Min	Max					
Wt Solids @105C - (%)	2.5 - 13	14.4	14.8	15.2	11.9					See Anal 4
Wt Oxide @1050C - (%)		11.5	n/a	12.7	n/a					
Total Oxide - (g/L)	25 - 100	126.5	n/a	133.4	n/a					
Slurry Density - (g/mL)	1.02 - 1.10	1.10	n/a	1.05	n/a					
Settled Solids - (Vol%)	7 - 95	18.50	n/a	n/a	n/a					
pH	>10	n/a	n/a	n/a	11.7					

HLRF=High Level Radiation Facility; SAL=Shielded Analytical Laboratory; TBD=to be determined; n/a=not measured; nd=not detected; n/a=not applicable; Y=yes; N=no

(1) Shaded analysis detected in Initial Vendor Product information.

(2) g/L = (g analyte dry solids)/(wt% solids*wt% solids)*(1/1E-06g/g). C/L = (wt% analyte dry solids)/(wt% solids*wt% solids)*(1/1E-06g/g). [See Section 3]

(3) TC: Final results are calculated values based on IP results (See Section 3 for analysis). Y: Final results are measured values from analysis of IP.

(4) HRF wt% solids performed in duplicate on 3 g sample aliquots. SAL wt% solids performed on single 10-50 g bulk samples.

2.0 FINAL VENDOR PRODUCT SODIUM-REDUCTION TREATMENT

A flowchart of the treatment and analysis scheme is presented in Figure A1-1. The Initial Vendor Product was received from the 324 building in two containers, labeled SPD1-C106-051 (1624 g) and SPD1-C106-052 (1523 g) [Brooks 1997]. Since the sodium concentration of the Initial Vendor Product exceeded the Envelope D specification, a sodium-reduction treatment was performed on the material. The treatment as described in LOI Revision 1 consisted of decanting a known quantity of supernatant containing sodium and replacing it with deionized water in excess of the decant supernatant volume. Water was added in excess of the volume of supernatant decanted with the intent to bring the product into specification with respect to weight percent solids and weight percent oxide. However, meeting the weight percent solids/oxides specification is not a requirement for shipment of the product.

2.1 DECANT/DILUTE OPERATION – LOI Revision 1

The supernatant from the containers SPD1-C106-051 and SPD1-C106-052 was decanted after gravity settling, combined, weighed, and poured into two 1 L polyethylene bottles (C106-Supernatant-1, C106-Supernatant-2) for archiving. The total mass of supernatant decanted was 2002.32 grams. A subsample of the combined, decanted supernatant was collected for supernatant density measurement and ICP-AES analysis. The residual solids from each of these two containers of Initial Vendor Product were combined together into a 4 liter plastic beaker. The total mass of the blended solids was 908.60 grams. A known mass of distilled water was used to rinse the Initial Vendor Product from the containers as well as for final dilution of the solids. The total mass of distilled water added to the solids was 2602.85 grams. Table A1-2.1 presents the weights obtained for the decanted supernatant, distilled water and resulting Final Vendor Product. The weight of the Initial Vendor Product is calculated from the weight of the Final Vendor Product adjusted for the weight differences between the decant liquid and added water. This weight is approximately 200 g less than the quantity of material received from the 324 building (i.e., 3147 g). This weight discrepancy is not anticipated to have any significant impact on the final product results; however, this is to be further evaluated and conclusion reported in the "Analytical Final Report" which is to be issued in March 1997.

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Figure A1-1. Initial Product Analysis and Sodium Adjustment Treatment

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Table A1-2.1. Decant/Dilution Weights

Description	Sample ID	Lab Number	Weight (g)
Initial Vendor Product	SPD1-C106-051 and SPD1-C106-052	N/A	2910.92 (Calculated)
Decanted Liquid	C106 Vendor Supernate	97-0180	2002.32
Distilled Water Added	N/A	N/A	2602.85
Final Vendor Product	C106 Vendor Final	97-0179	3511.45

2.1.1 Physical Measurements

The Final Vendor Product was sampled for physical testing measurements by pouring while stirring mechanically just prior to filling a sample bottle (Container ID 97-0179). These measurements consisted of weight percent solids, weight percent oxide, slurry density, and volume percent settled solid, and the results are presented in Table A1-2.2. The subsamples for each of the physical measurements were extracted from the bottle while stirring; however, both the sample and duplicate were extracted from the same bottle. The resulting weight percent solids (21.5%) and weight percent oxide (18.6%) were inconsistent with expectations. Since water in excess of the decanted supernatant volume was added during the decant/dilute treatment, the weight percent solids should have been lower than the Initial Vendor Product result (i.e., <14.4%). Resampling of the material with Lab Number 97-0179 and measurement of the weight percent solids confirmed the high result. The higher than expected percent solids has been attributed to the difficulty in stirring/pouring the slurries. Although there was no immediate visual clue that the subsampling was not representative, after settling it was observed that the settled solids heights were significantly different among the twenty bottles prepared for shipping to the vendors. The physical measurement results from this subsample are included for information only, since these results are superseded by the measurements conducted following reblending, resampling, and rebottling operations [See Section 2.2; Note – The weight percent solids and weight percent oxides presented in Table A1-2.2 are not used to calculate any “adjusted” concentrations.]

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Table A1-2.2. Settled Solids and Density Measurements
 per LOI Revision 1 (for Information Only)

Sample ID	Settled Solids (Vol%)	Solids (Wt%)	Oxides (Wt%)	Oxides (g/L)	Density (g/ml)
97-0179 (Final Vendor Product)	39.1 ^(a)	21.2	18.5	206	1.11
97-0179 Dup (Final Vendor Product)	39.4 ^(a)	21.7	18.6	214	1.15
Final Vendor Product - Average	39.2	21.5	18.6	210	1.13
Final Vendor Product - RPD (%)	0.8	2.3	0.3	3.8	3.6

^(a) Greater than 95% of the solids have settled in less than eight hours.

2.1.2 Initial Bottling of Final Vendor Product

Following the decant/dilute treatment, twenty vendor subsamples (ten for each vendor) were aliquotted into glass bottles; this operation was performed by pouring while stirring mechanically prior to filling each bottle. This initial bottling of the Final Vendor Product resulted in bottles containing variable quantities of solids, as observed from the settled solids height in each bottle. Based on the inconsistency in the settled solids level, as well as the unexpectedly high weight percent solids, additional processing of the Final Vendor Product was necessary in order to establish credible solids and oxides concentrations [See Section 2.2].

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2.2 REBLENDING, RESAMPLING, REBOTTLING OPERATION – LOI Revision 2**2.2.1 Reblending**

All Final Vendor Product material including the material initially bottled for each vendor and the remaining archive material was composited into a 4 L plastic beaker and blended by mechanical stirring. A flowchart detailing the reblending, rebottling, and reanalysis is presented in Figure A1-2. This operation was performed per the instructions provided in the LOI Revision 2.

2.2.2 Bottling of Final Vendor Product

Following compositing and mechanical stirring to thoroughly blend the Final Vendor Product slurry, twenty vendor bottles were prepared. While mechanically stirring the slurry contents of the 4 L beaker, the Final Vendor Product was transferred to the vendor bottles by pipetting. Each vendor bottle was filled with two 50 ml pipet aliquots. Two additional bottles (97-0179-1 and 97-0179-2) were filled to provide material for any future physical testing, if required. Table A1-2.3 provides the vendor container identification numbers, the transferring sequence numbers (i.e., 1 through 20), and the quantities transferred to each container. Observation of the solids level following settling indicate no visual difference in the quantities of solids transferred to each vendor bottle. Four of these bottles, i.e., C106-FP-V1-1, C106-FP-V1-6, C106-FP-V2-3, and C106-FP-V2-8, were selected for further physical and chemical analyses (See Section 2.2.3 and Section 4).

Figure A1-2. Reblending, Rebottling, and Reanalysis of Final Vendor Product

Table A1-2.3 Vendor Subsampling Container ID and Quantity

Sampling #	Container ID	Subsample Weight (g)	Sampling #	Container ID	Subsample Weight (g)
1	C106-FP-V1-1*	99.62	2	C106-FP-V2-1	97.64
3	C106-FP-V1-2	99.02	4	C106-FP-V2-2	102.13
5	C106-FP-V1-3	98.51	6	C106-FP-V2-3*	98.74
7	C106-FP-V1-4	100.31	8	C106-FP-V2-4	102.49
9	C106-FP-V1-5	99.06	10	C106-FP-V2-5	89.93
11	C106-FP-V1-6*	100.02	12	C106-FP-V2-6	96.21
13	C106-FP-V1-7	95.74	14	C106-FP-V2-7	88.13
15	C106-FP-V1-8	99.25	16	C106-FP-V2-8*	97.00
17	C106-FP-V1-9	89.14	18	C106-FP-V2-9	106.12
19	C106-FP-V1-10	103.61	20	C106-FP-V2-10	99.10

* Samples selected for additional physical and chemical analyses

2.2.3 Physical Measurement

The physical measurements conducted on the rebleded Final Vendor Product composite are presented in Table A1-2.4, and consist of weight percent solids, weight percent oxide, and slurry density. The weight percent solids on drying and the oxide concentration on firing of the Final Vendor Product remain slightly higher than the values obtained on the Initial Vendor Product (see Table A1-1). The use of small sample aliquots (i.e., 3 g) collected by pipetting for measuring the percent solids in this material appears to bias the results high relative to the percent solids determined from large quantity,

bulk samples (See Section 4). The percent solids and percent oxides results from Table A1-2.4 (i.e., 15.2% and 12.7%) are used to convert the Final Vendor Product "measured" results from $\mu\text{g/g}$ dried solids to g/L at 31 g oxide.

Table A1-2.4. Wt% Solids and Wt% Oxide Measurements per LOI Revision 2

Lab Number	Sample/Container ID	Density (g/ml)	Solids (Wt%)	Oxides (Wt%)	Oxides (g/L)
97-1112	C106-FP-V1-1	1.06	14.4	12.0	126
97-1113	C106-FP-V1-6	1.04	13.9	11.6	122
97-1114	C106-FP-V2-3	1.06	16.3	13.9	146
97-1115	C106-FP-V2-8	1.06	15.9	13.3	140
Final Vendor Product - Average		1.05	15.2	12.7	133.5
Final Vendor Product - Std Dev		± 0.01	± 1.2	± 1.1	± 12

3.0 ADJUSTED ANALYTE CONCENTRATIONS FOLLOWING SODIUM REDUCTION

The source and type of analysis used to estimate the composition of the Final Vendor Product is shown in Figure A1-1. The Final Vendor Product concentration is calculated from the Initial Vendor Product concentration, the oxide content of the Initial Vendor Product and Final Vendor Product, and the analyte/radionuclide concentrations of the decanted supernatant (where measured and detected).

3.1 INITIAL VENDOR PRODUCT ANALYSIS

The Initial Vendor Product was analyzed per the requirements of LOI Revision 0 and all chemical, radionuclide, and physical measurement results have been presented in the Analytical Summary Report [Urie 1997]. The analytical results are summarized in Table A1-1 (Initial Product Measured).

3.2 INITIAL VENDOR PRODUCT SUPERNATANT ANALYSIS

The Initial Vendor Product supernatant was analyzed twice. The first set of analyses (sample C106-046-DL, 96-6654) were performed on centrifuged decant liquid of the Initial Vendor Product. Limited chemical and radionuclide analyses were performed on this supernatant. Since this supernatant was decanted nearly a month prior to performing the sodium adjustment treatment process, there was concern that the analyses might not accurately reflect the composition of the Initial Vendor Product just prior to the decant/dilute treatment for Final Vendor Product generation. Therefore, following gravity settling of Initial Vendor Product, another sample of supernatant (sample C-106-Vendor Supernate, 97-0180) was taken just prior to decanting. This second sample was analyzed by ICP-AES (only) and compared to the original ICP-AES results.

A summary of the chemical and radionuclide analyses performed on the two Initial Vendor Product supernatant samples is provided in Table A1-3.1. Besides density shown in Table A1-3.2, ICP-AES was the only analysis performed on both supernatant samples. As can be seen from the ICP-AES results, there appears to be little change in the chemical composition of the supernatant with time, which is expected from a saturated supernatant. Sodium is clearly the primary ICP-AES component in both supernatants. Aluminum was present in quantitative levels; whereas, Boron, Calcium, and Phosphorus were present in low, semi-quantitative levels and are subject to large errors. For these analytes (i.e., sodium, aluminum, boron, calcium, and phosphorus), only the ICP-AES results from the second analysis (sample C-106-Vendor Supernate, 97-0180) are used to calculate the quantity of analyte decanted.

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Table A1-3.1. Initial Vendor Product Supernatant Analytical Results

Analyte	First Analysis - Centrifuged			Second Analysis - Gravity Settled		
	Process Blank	C108-046-DL		Diluent Bk	C-108-Vendor Supernate	
	96-6654-PB	96-6654	96-6654 Dup	97-0180	97-0180	97-0180 Dup
	ug/mL	ug/mL	ug/mL	ug/mL	ug/mL	ug/mL
Ag	—	—	—	—	—	—
Al	[6.3]	64.6	61.8	[0.1]	91.9	94.6
As	—	—	—	—	—	—
B	62.9	68.4	61.7	2.84	[44]	51.3
Ba	—	—	—	—	—	—
Be	—	—	—	—	—	—
Bi	—	—	—	—	—	—
Ca	[47]	[22]	[9]	—	[78]	[49]
Cd	—	—	—	—	—	—
Ce	—	—	—	—	—	—
Co	—	—	—	—	—	—
Cr	[0.87]	[1.6]	[1.6]	—	[2.4]	—
Cu	—	—	—	—	—	—
Dy	—	—	—	—	—	—
Eu	—	—	—	—	—	—
Fe	[2.5]	[3.7]	[2.3]	—	—	—
K	—	—	—	—	—	—
La	—	—	—	—	—	—
Li	—	—	—	—	—	—
Mg	[6.0]	[5.0]	—	—	—	—
Mn	—	—	—	—	—	—
Mo	—	—	—	—	—	—
Na	86.9	14,200	13,800	3.29	14,700	15,200
Nd	—	—	—	—	—	—
Ni	[1.2]	—	—	—	—	—
P	—	24.6	28.0	—	[39]	[41]
Pb	—	—	—	—	—	—
Pd	—	—	—	—	—	—
Rh	—	—	—	—	—	—
Ru	—	—	—	—	—	—
Sb	—	—	—	—	—	—
Se	—	—	—	—	—	—
Si	263	234	168	[0.60]	—	—
Sn	—	—	—	—	—	—
Sr	—	—	—	—	—	—
Te	—	—	—	—	—	—
Th	—	—	—	—	—	—
Ti	—	—	—	—	—	—
Tl	—	—	—	—	—	—
U	—	—	—	—	—	—
V	—	—	—	—	—	—
W	—	—	—	—	—	—
Y	—	—	—	—	—	—
Zn	[2.7]	—	—	—	—	—
Zr	—	—	—	—	—	—

*-- Analyte not detected above the Instrument Detection Limit

Results in brackets are below the Estimated Quantitation Limit but above the Instrument Detection Limit.

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Table A1-3.1 (Cont). Initial Vendor Product Supernatant Analytical Results

Analyte	First Analysis - Centrifuged			Second Analysis - Gravity Settled		
	Process Blanks	C-106-046-DL		Diluent Bk	C-106-Vendor Supernate	
	96-6654-PB	96-6654	96-6654 Dup	97-0180	97-0180	97-0180 Dup
Analyte	uCi/g	uCi/g	uCi/g	No Analysis Performed		
Am-241	<4E-04	<7E-03	<7E-03			
Co-60	<2E-04	<7E-04	<5E-04			
Cs-137	3.82E-03	2.34E+00	2.30E+00			
Eu-154	<3E-04	<2E-03	<8E-04			
Eu-155	<3E-04	<5E-03	<5E-03			
Sr-90	1.91E-03	3.59E-02	4.84E-02			
Total Alpha	4.42E-04	<4E-03	<4E-03			
Tc-99	2.55E-04	4.93E-04	5.36E-04			
Analyte	ugC/g	ugC/g	ugC/g			
TIC	127	559	587			
TOC	1214	6645	7134			
TC	N/A	7204	7721			
Analyte	ug/ml	ug/ml	ug/ml			
Br	N/A	<10.0	<10.0			
Cl	N/A	20	20			
F	N/A	<250.0	<250.0			
NO2	N/A	390	400			
NO3	N/A	60	60			
PO4	N/A	20	20			
SO4	N/A	80	80			
C2O4	N/A	24000	25000			

No radiochemistry, TOC, or anion analyses performed on 97-0180.

"<" - Analyte not detected above the Instrument Detection Limit

N/A - Not Applicable

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**Table A1-3.2. Initial Vendor Product Supernatant Density Measurements
per LOI Revision 1**

Laboratory Number (Sample ID)	Density (g/ml)
96-6653 (C106-046-DL, First Analysis Initial Vendor Product Supernatant) -- Centrifuged	1.000
97-0180 (C-106-Vendor Product, Second Analysis Initial Vendor Product Supernatant) -- Gravity Settled	1.036

3.3 CALCULATED "ADJUSTED" FINAL VENDOR PRODUCT CONCENTRATION

Based on the consistency demonstrated by the sodium, and assuming other analytes behave similarly, the Final Vendor Product results have been adjusted for the reduced sodium concentration and loss of analytes/radionuclides measured in the decanted supernatants, where applicable. The "adjusted" concentration is presented in Table A1-1 as the Final Product results, whenever Final Vendor Product measured results (See Section 4) are not available (i.e., either not measured or not detected).

3.3.1 Adjusted Concentrations for Analytes/Radionuclides NOT Detected or Measured in the Initial Vendor Product Supernatant

For analytes/radionuclides not measured or detected in the Initial Vendor Product supernatant, the reported Final Vendor Product concentration is the Initial Vendor Product concentration adjusted for the reduction in the oxide loading following the sodium reduction treatment (Equation 1). For explanation of calculation of Final Vendor Product "total oxide" (FO) contents, see Appendix A.

$$\text{FVP} = (\text{IVP}) * (\text{PS} / \text{PO}) * (\text{IO} / \text{FO}) * (31) * (1\text{e-}6) \quad (\text{Equation 1})$$

Where, FVP = Final Vendor Product concentration in g/L (or Ci/L)

IVP = Initial Vendor Product concentration in $\mu\text{g/g}$ (or $\mu\text{Ci/g}$)--See Table A1-1

PS = Weight percent solids for Initial Vendor Product (14.4%)

PO = Weight percent oxide for Initial Vendor Product (11.5%)

IO = Initial Vendor Product total oxide content (334.76 g)

FO = Final Vendor Product estimated total oxide content (295.81 g)

31 = Gram oxide per 31 g oxide/L

1e-6 = Gram per microgram

3.3.2 Adjusted Concentrations for Analytes/Radionuclides Detected or Measured in the Initial Vendor Product Supernatant

For analytes/radionuclides measured in the Initial Vendor Product supernatant, the reported Final Vendor Product concentration is the Initial Vendor Product concentration adjusted for the reduction in the oxide loading following the sodium reduction treatment and the quantity of analyte/radionuclide removed during the decanting process (Equation 2). For explanation of the calculation of total masses (i.e., dried solids and decant liquid) and final total oxide, see Appendix A.

$$\text{FVP} = [(\text{IVP} * \text{MD}) - (\text{IVS} * \text{MS})] * (1 / \text{FO}) * (31) * (1\text{e-}6) \quad (\text{Equation 2})$$

Where, FVP = Final Vendor Product concentration in g/L (or Ci/L)

IVP = Initial Vendor Product concentration in $\mu\text{g/g}$ (or $\mu\text{Ci/g}$)--See Table A1-1)

MD = Total mass of dried Initial Vendor Product (419.17 g)

IVS = Initial Vendor Supernatant concentration in $\mu\text{g/g}$ (or $\mu\text{Ci/g}$) (See Table A1-3.1, adjusted for supernatant density of 1.036 g/ml or 1.000 g/ml, where appropriate)

MS = Total Mass of decanted Supernatant (2002.3 g)

FO = Final Vendor Product estimated total oxide content (295.81 g)

31 = Gram oxide per 31 g oxide/L

1e-6 = Gram per microgram

4.0 FINAL VENDOR PRODUCT ANALYSES -- LOI Revision 2

Following the compositing and rebottling of the Final Vendor Product, four bottles were selected for additional analyses. The analyses performed were slurry density (See Table A1-2.5), bulk weight percent solids, total organic carbon, pH/OH, anions by IC, uranium by laser fluorimetry, and metals by ICP-AES. The results for the chemical analyses are presented in Tables A1-4.1 through A1-4.5. Due to balance capacity limitations (i.e., weighing container and sample was limited to 200 g) in the Shielded Analytical Laboratory (SAL) and the requirement to perform weight percent solids on the entire contents of the containers, the contents of the four containers were split in High Level Radiation Facility, resulting in eight containers being submitted to the SAL for additional analyses. One split (i.e., C106-FP-V1-6b) was lost during handling. The C106 Final Vendor Product samples received from the HLRF for preparation and analysis consisted of seven, 30-50 g Final Vendor Product slurries and four, 10 ml aliquots for pH or OH. The pH/OH aliquots were allowed to settle and 5 mls of the supernatant from each was taken out of the hot cell and delivered to the ACL Sample Receiving and Prep Laboratory for analysis.

4.1 SAMPLE DRYING AND BLENDING

Each of the seven Final Product slurries was weighed upon receipt and dried in a oven at 105 °C until successive weighings yielded less than 1 percent weight loss between successive dryings. The bulk sample weight percent solids on drying are presented in Table A1-4.6. The observed volume reduction ranged from 55% to 70% after drying. The average reduction was 60% with a relative standard deviation of about 10%. In all cases, the remaining dried solids were medium brown in color and the evaporated supernatant left a white coating on the container sides and on top of the dried brown solid layer. No indication of spattering, foaming, etc. during drying was noted as evidenced by clean container walls above the original fill lines. After drying, all seven dried slurries were individually homogenized by manual stirring, and then sample splits were recombined and blended together to produce a total of four samples for subsequent chemical analysis.

Table A1-4.6 Bulk Weight Percent Solids

Lab Number/Sample Identification	Weight Percent Solids
97-01112 / C106-FP-V1-1	12.35
C106-FP-V1-1-B	11.31
97-01113 / C106-FP-V1-6	11.61
C106-FP-V1-1-B (lost)	n/a
97-01114 / C106-FP-V2-3	12.58
C106-FP-V2-3-B	11.24
97-01115 / C106-FP-V2-8	12.45
C106-FP-V2-8-B	11.47
Final Vendor Product - Average	11.86
Final Vendor Product - Std Dev	0.58

4.2 CHEMICAL PREPARATION FOR ANALYSIS

Full Quality Control (QC) was applied to all batch processing techniques in accordance with the Quality Assurance Plan MCS-033. Full QC incorporates the use of methods blanks, spiked blanks, duplicate samples, matrix spiked samples and radiochemical tracers where available.

4.2.1 Acid Digestion

Each dried slurry sample was acid digested (PNL-ALO-129 "HNO₃-HCl Acid Extraction of Solids Using a Dry-Block Heater") and distributed for ICP/AES. It was noted during sample blending that the stir bars introduced into the containers by the HLRF were covered with magnetic solids. The stir bars were subjected to the same acid digestion treatment as the samples and the resulting solutions provided for ICP/AES.

4.2.2 Fusion

Caustic fusions (PNL-ALO-114 "Solubilization of Metals from Solids Using a Na₂O₂-NaOH Fusion", PNL-ALO-115 "Solubilization of Metals from Solids Using KOH-KNO₃ Fusion") were performed and aliquots distributed for ICP/AES and uranium analyses. The fusion technique is usually applied where total solubilization of the sample material is required. QC is limited to sample duplicates, a methods blank and post fusion spiking for selective analytes applied at the bench.

4.2.3 Water Leach

Each dried slurry sample was water leached (PNL-ALO-103 "Water Leach of Sludges, Soils and Other Solid Samples") filtered and aliquots distributed for anion analysis by Ion Chromatography.

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4.2.4 Carbon Analysis

Total Carbon, Total Inorganic Carbon and Total Organic Carbon analyses (PNL-ALO-381 "Direct Determination of TC, TOC, and TIC in Radioactive Sludges and Liquids by Hot Persulfate Method") is usually performed remotely using a UIC Coulemetrics Carbon Analyzer. However, due to hardware failures, the TOC analysis can not be reported at this time.

4.3 MEASURED ANALYTICAL RESULTS

The results of the analyses conducted on the Final Vendor Product are summarized in Table A1-1 (FP Measured). Table A1-4.1 shows the average ICP-AES results from each sample preparation and the "most reasonable" result selected for inclusion in Table A1-1. Tables A1-4.2 through A1-4.5 contain the chemical results obtained on the four selected Final Vendor Product samples.

4.3.1 ICP-AES Analysis

ICP-MS and ICP-AES were both used to analyze the Initial Vendor Product and the majority of the results summarized in Table A1-1 are from the ICP-MS analysis; primarily since the ICP-MS provides significant higher sensitivity. For the Final Vendor Product only ICP-AES analyses were performed since the ICP/MS was unavailable. Due to the limited sensitivity of the ICP-AES, only a subset of the analytes of interest are reported for the Final Vendor Product. However, the agreement between the calculated "adjusted" value and the ICP-AES measured value are very good. Except for silver, phosphorus and Strontium, the measured results is with $\pm 30\%$ (and most analytes are within $\pm 10\%$) of the calculated result. The Final Vendor Product silver result is only half of that calculated, which is attributed to difficulties of the specific dissolution procedure to maintain silver in solution. Strontium in the Initial Vendor Product was report by ICP-MS. Although ^{90}Sr was detected in the Initial Vendor Product decanted supernatant, the ICP-AES analysis of the supernatant detected no strontium and no correction for strontium in the decant supernatant could be made resulting in a high estimate for the

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"adjusted" value. The phosphorus result is higher for the Final Vendor Product than the Initial Vendor Product; the reason for this is unclear but appears to be related to difference in fusion dissolution (See Table A1-4.1 for phosphorus values obtained from each preparatory technique).

In most cases, results from ICP-AES (Initial and Final Vendor Product) and ICP-MS (Initial Product Only) were obtained from three different preparation techniques; specifically KOH fusion, Na_2O_2 fusion and HNO_3 -HCl acid digestion. In selecting the most reasonable result (Table A1-4.1), an effort has been made to evaluate the results based on the characteristics of the analytical technique and the limitation of the preparation method. A description of the selection basis for the Initial Vendor Product characterization is provided in the Analytical Summary Report². For the analysis of the Final Vendor Product, only the limitation of the preparation method needs to be considered, since ICP-MS was not available. Where the preparation methods are considered equivalent and the measured value for analyte is above the EQL, the highest reported result is used for comparison to the envelope specification. This applies to 16 of the 23 analytes detected by ICP-AES in the Final Vendor Product. Results from the acid digest preparations were selected for the other seven analytes, primarily because of better sample sensitivity (i.e., the samples prepared by fusion methods require 10 to 40 times higher dilutions than samples prepared by acid digestion, and most of the results from the fusion methods were below the EQL). Due to excessively high and variable calcium results for the fusion processing blanks (evident in both the Initial Vendor Product and Final Vendor Product analyses), the calcium results from the acid digestion are considered more reliable, and therefore selected for reporting.

4.3.2 IC Analysis

Following the water leaching of the Final Vendor Product, IC analyses were performed to determine the anion concentrations. Except for nitrate+nitrite, the calculated "adjusted" anion concentrations do not agree with the measure Final Vendor Product results. This is to be expected, since the water leaching does not provide 100% dissolutions, some anion such as oxalate are at solubility limits, and other anions are at the measurement quantitation limit. The Final Vendor Product results represent the anion solution concentrations for product material leached at a 1:100 ratio.

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Table A1-4.1 ICP-AES Summary of Results for Final Vendor Product

****All Results in ug/g -- dry weight****

Analysis	Acid Digestion		KOH Fusion		Na ₂ O ₂ Fusion		Result		Comments
Analyte	Blank	Average	Blank	Average	Blank	Average	Selected		
Ag		125		1,820		749	1,820	Highest selected	
Al		59,050	[324]	66,800	[85]	64,100	66,800	Highest selected	
As								not detected	
B	131.0	145		[159]				not detected - high blank/uncertainty	
Ba		487		527		544	544	Highest selected	
Be								not detected	
Bi		530		[633]		[660]	[660]	High uncertainty -- below EQL	
Ca	[54.0]	4,825	[1,570]	8,963	[2,180]	12,075	4,825	Fusions have high blanks	
Cd		84		[110]		[99]	84	Fusions are below EQL	
Ce		[328]		[577]	[428]	[1,820]		not detected -- too close to IDL to use	
Co		[31]						not detected	
Cr		570		806		765	806	Highest selected	
Cu	[2.0]	130		[206]		[201]	130	Fusions are below EQL	
Dy								not detected	
Fe	[2.0]	145,000	[91]	158,250	[84]	153,000	158,250	Highest selected	
K						[5,103]		not detected - fusion high uncertainty	
La		[112]		[162]		[175]	[112]	High uncertainty -- below EQL	
Li		[23]						not detected -- to close to IDL to use	
Mg		803		[1,478]		[1,375]	[1,478]	High uncertainty -- below EQL	
Mn		3,010	[189]	3,820		3,200	3,200	Highest selected	
Mo		[22]						not detected -- to close to IDL to use	
Na	152.0	120,000	2,710	120,250			120,000	Fusion has high blank	
Nd		332	[172]	[539]			332	Fusion has high uncertainty	
Ni	[10.0]	1,300			[98]	[550]	1,905	Highest selected	
P		1,653		3,070		4,805	4,805	Highest selected	
Pb		3,893		3,473		4,118	4,118	Highest selected	
Pd								not detected	
Rh								not detected	
Ru								not detected	
Sb								not detected	
Se		[159]						not detected	
Si	[197.0]	2,678		86,800		78,850	86,800	Highest selected	
Sn								not detected	
Sr		62		[100]	[41]	[163]	[100]	Fusion have higher uncertainty	
Te		[506]					[506]	High uncertainty	
Th		[428]					[428]	High uncertainty	
Ti		364				1,238	1,238	Highest selected	
Tl								not detected	
U		[2,530]		[5,602]		[4,802]		Use Laser Fluorimetry Results	
V								not detected	
W								not detected	
Y		[86]		[105]		[100]		not detected -- to near IDL to use	
Zn	[5.0]	[111]		[196]		[209]		not detected -- to near IDL to use	
Zr		[85]		1,252			1,252	Highest selected	

Results for each preparation (KOH/Na₂O₂/Acid) are average of 4 samples 97-1112 through 97-1115

Results in brackets are below Estimated Quantitation Limit but above Instrument Detection Limit

Acid digestion results selected for Ca due to excessively high and variable processing blank from Shielded Analytical Laboratory

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Table A1-4.2 Wet Chemistry Analysis--Vendor Product
C106 Privatization Contractor Sample

Analyte	Process Blen ¹ 97-1112-PB ug/g	C106-FP-V-1 97-1112 ug/g	C106-FP-V-1-1 97-1112 Dup ug/g	C106-FP-V-6 97-1113 ug/g	C106-FP-V-2-3 97-1114 ug/g	C106-FP-V-2-8 97-1115 ug/g	Average ug/g	Stddev ug/g	Envelope D Specification ug/g	Comparison Ave/Spec Flag
Water Leach										
Br	< 3.00E+01	< 1.10E+02	< 1.10E+02	< 1.10E+02	< 1.10E+02	< 1.00E+02	< 1.00E+02	N/A	N/A	N/A
C2O4	< 5.00E+01	8.00E+04	7.80E+04	8.30E+04	8.40E+04	8.00E+04	8.33E+04	3.19E+03	N/A	N/A
Cl	5.00E+01	< 1.10E+02	< 1.10E+02	< 1.10E+02	1.10E+02	< 1.00E+02	< 1.00E+02	N/A	2.58E+03	4.2E-02
F	< 3.00E+01	< 1.40E+03	< 1.40E+03	< 1.40E+03	< 1.30E+03	< 1.30E+03	< 1.35E+03	N/A	2.83E+04	4.8E-02
NO2	< 5.00E+01	4.00E+02	4.00E+02	4.00E+02	4.00E+02	4.00E+02	4.00E+02	0.00E+00	N/A	N/A
NO3	1.30E+02	6.00E+02	6.00E+02	5.00E+02	5.00E+02	5.00E+02	5.25E+02	5.48E+01	2.89E+05	3.2E-03
PO4	< 5.00E+01	3.00E+02	3.00E+02	3.00E+02	3.00E+02	2.00E+02	2.75E+02	4.47E+01	N/A	N/A
SO4	9.00E+01	4.00E+02	3.00E+02	3.00E+02	3.00E+02	3.00E+02	3.23E+02	4.47E+01	N/A	N/A
Direct										
OH (N)	2.00E-02	2.30E-02	2.10E-02	2.30E-02	2.10E-02	2.10E-02	2.13E-02	1.26E-03		
pH	1.16E+01	1.18E+01	1.18E+01	1.18E+01	1.18E+01	1.17E+01	1.17E+01	9.57E-02		
TC										
TiC									N/A	
TiOC									4.79E+04	
WT% Solids	1.18E+01	1.16E+01	1.16E+01	1.16E+01	1.19E+01	1.20E+01	1.18E+01	1.55E-01	8.56E+04	
* Envelope D Specification for NO3+NO2, NO3 Ave/Spec = (Average NO2+Average NO3)/Spec										
									N/A	N/A

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Table A1-4.3. ICP-AES KOH Fusion--Vendor Product
C106 Privatization Contractor Sample

Analyte	Process Blank 97-1112-PB	KOH Fusion Utilizing Ni Crucibles				Average	Stdev	Envelope D Specification	Comparison	
		C106-FP-V1-1 97-1112-1	C106-FP-V1-6 97-1113-1	C106-FP-V2-3 97-1113 Dup	C106-FP-V2-8 97-1114-1				Ave/Spec	Flag
Ag	—	2,400	1,130	877	1,150	1,800	1,820	4.38E+03	3.70E-01	
Al	[324]	65,000	70,300	68,400	71,000	62,900	66,800	1.11E+05	6.03E-01	
As	—	—	—	—	—	< 473	—	1.29E+03	3.67E-01	
B	—	[185]	[207]	[105]	[130]	[115]	[159]	1.03E+04	1.55E-02	
Ba	—	509	533	518	554	513	527	3.61E+04	1.40E-02	
Be	—	—	—	—	—	< 19	—	5.15E+02	3.87E-02	
Bi	—	[660]	[611]	[597]	[670]	[573]	[634]	2.22E+04	2.96E-02	
Ca	[1570]	8,500	9,210	8,830	8,890	8,250	8,960	5.67E+04	1.58E-01	
Cd	—	[113]	[113]	[103]	[113]	[101]	[110]	3.61E+04	3.05E-03	
Ce	—	[838]	[547]	[524]	[611]	[510]	[577]	5.44E+03	8.96E-02	
Co	—	—	[101]	[75]	—	[101]	—	3.81E+03	2.80E-02	
Cr	—	765	808	753	856	763	806	5.41E+03	1.49E-01	
Cu	—	[223]	[215]	[180]	[209]	[176]	[206]	3.86E+03	5.34E-02	
Dy	—	—	—	—	—	< 85	—	2.06E+02	4.59E-01	
Eu	—	—	—	—	—	< 189	—	1.29E-02	1.47E+00	*
Fe	[91]	152,000	160,000	157,000	167,000	154,000	158,000	2.29E+05	6.80E-01	
K	N/U	N/U	N/U	N/U	N/U	N/U	N/U	1.06E+04	N/U	N/U
La	—	[172]	[157]	[147]	[167]	[150]	[162]	2.06E+04	7.84E-03	
Mg	—	[1410]	[1480]	1,430	[1590]	1,430	1,480	1.11E+03	5.12E-02	
Mn	[189]	3,750	4,110	3,520	3,670	3,450	3,820	5.15E+04	7.41E-02	
Mo	—	—	—	—	—	< 95	—	5.15E+03	1.84E-02	
Na	2,710	124,000	116,000	112,000	114,000	127,000	120,000	1.59E+05	7.78E-01	
Nd	[172]	[847]	[519]	[487]	[567]	[491]	[539]	1.37E+04	3.94E-02	
Ni	N/U	N/U	N/U	N/U	N/U	N/U	N/U	1.86E+04	N/U	N/U
P	—	3,510	3,390	2,540	2,720	2,380	3,070	1.39E+04	2.21E-01	
Pb	—	3,600	3,320	3,590	3,680	3,290	3,470	8.75E+03	3.96E-01	
Pd	—	—	—	—	—	< 1,420	—	1.03E+03	1.38E+00	*
Rh	—	—	—	—	—	< 568	—	1.03E+03	5.51E-01	
Ru	—	—	—	—	—	< 2,080	—	2.83E+03	7.35E-01	
Sb	—	—	—	—	—	< 946	—	6.70E+03	1.41E-01	
Se	—	—	—	—	—	< 473	—	4.12E+03	1.15E-01	
Si	—	82,500	90,800	88,800	92,100	81,800	86,800	1.49E+05	5.81E-01	
Sn	—	—	—	—	—	< 2,840	—	2.83E-02	1.00E+01	*
Sr	—	[98]	[101]	[99]	[104]	[95]	[99]	4.12E+03	2.41E-02	
Te	—	—	—	—	—	< 2,840	—	1.03E+03	2.75E+00	*
Th	—	—	—	—	—	< 1,890	—	4.12E+03	4.59E-01	
Ti	—	1,200	1,160	1,190	1,300	1,160	1,210	1.03E+04	1.17E-01	
Tl	—	—	—	—	—	< 946	—	3.61E+03	2.62E-01	
U	—	[660]	[610]	[4760]	[870]	[4410]	[5,600]	920	1.08E+05	5.18E-02
V	—	—	—	—	—	< 95	—	2.88E+02	3.87E-01	
W	—	—	—	—	—	< 3,780	—	1.91E+03	1.99E+00	*
Y	—	—	[103]	[103]	[113]	[100]	[105]	1.29E+03	8.18E-02	
Zn	—	[210]	[187]	[181]	[198]	[164]	[196]	3.55E+03	5.84E-02	
Zr	—	1,850	1,160	1,050	1,280	707	1,250	1.19E+05	1.06E-02	

— = Analyte not detected above the IDL, only average IDL reported as less than (<) value.
Results in brackets are qualified but they are included in the calculation of the average.

N/A Not Applicable
N/U Not Usable

Analytical Summary Report Addendum 1, Revision 2 - March 7, 1997

Table A1-4.4. ICP-AES Na2O2 Fusion--Vendor Product
C106 Privatization Contractor Sample

Analyte	Process Blank 97-1112-PB	Na2O2 Fusion Utilizing Zr Crucibles						Average	Stdev	Envelope D Specification	Comparison
		C106-FP-V1-1 97-1112-PB	C106-FP-V1-2 97-1113-PB	C106-FP-V2-1 97-1114-PB	C106-FP-V2-2 97-1114 Dup	C106-FP-V2-3 97-1115-PB	C106-FP-V2-4 97-1115-PB				
		ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	Ava/Spe	Flag
Ag	[85]	1,160	663	636	478	536	749	280	4.38E+03	1.71E-01	
Al	—	65,500	67,700	61,800	64,100	61,000	64,100	3200	1.11E+05	5.79E-01	
As	—	—	—	—	—	—	< 469	—N/A	1.29E+03	3.64E-01	
B	—	—	—	—	—	—	< 94	—N/A	1.03E+04	9.11E-03	
Ba	—	565	548	528	551	533	544	17	3.61E+04	1.51E-02	
Be	—	—	—	—	—	—	< 19	—N/A	5.15E+02	3.84E-02	
Bi	—	[702]	[652]	[667]	[657]	[619]	[660]	34	2.22E+04	2.98E-02	
Ca	[2180]	12,500	12,200	12,300	10,500	11,200	12,100	610	5.67E+04	2.13E-01	
Cd	—	[105]	[102]	[97]	[99]	[97]	[99]	2	3.61E+04	2.74E-03	
Ce	[428]	[2560]	[1810]	[1530]	[1870]	[1390]	[1,820]	520	8.44E+03	2.83E-01	
Co	—	—	—	—	—	—	< 94	—N/A	3.61E+03	2.60E-02	
Cr	—	818	738	744	804	758	765	37	5.41E+03	1.41E-01	
Cu	—	[226]	[197]	[191]	[147]	[191]	[201]	17	3.86E+03	5.21E-02	
Dy	—	—	—	—	—	—	< 94	—N/A	2.06E+02	4.56E-01	
Eu	—	—	—	—	—	—	< 188	—N/A	1.29E+02	1.46E+00	
Fe	[84]	158,000	153,000	148,000	155,000	151,000	153,000	4300	2.29E+05	8.67E-01	
K	—	[5230]	[4930]	[5640]	[3670]	[4610]	[5100]	440	1.06E+04	4.83E-01	
La	—	[122]	[175]	—	[177]	[159]	[167]	8	2.96E+04	3.50E-03	
Li	—	[63]	—	—	[49]	[50]	[59]	8	1.11E+03	5.19E-02	
Mg	—	[1400]	[1350]	[1380]	[1320]	[1370]	[1,380]	21	1.67E+04	8.21E-02	
Mn	—	3,330	3,200	3,120	3,270	3,150	3,200	93	5.16E+04	8.21E-02	
Mo	—	—	—	—	—	—	< 94	—N/A	5.15E+03	1.82E-02	
Na	N/U	N/U	N/U	N/U	N/U	N/U	N/U	N/U	1.55E+05	N/U	N/U
Nd	—	[580]	[546]	[552]	[511]	[523]	[550]	23	1.37E+04	4.03E-02	
Ni	[98]	2,020	1,980	1,860	1,760	1,760	1,910	120	1.88E+04	1.01E-01	
P	—	4,740	4,640	4,750	4,490	5,090	4,810	200	1.39E+04	3.45E-01	
Pb	—	4,240	4,190	4,050	4,190	3,990	4,120	120	8.76E+03	4.70E-01	
Pd	—	—	—	—	—	—	< 1,410	—N/A	1.03E+03	1.37E+00	
Rh	—	—	—	—	—	—	< 563	—N/A	1.03E+03	5.47E-01	
Ru	—	—	—	—	—	—	< 2,070	—N/A	2.83E+03	7.29E-01	
Sb	—	—	—	—	—	—	< 939	—N/A	6.70E+03	1.40E-01	
Se	—	—	—	—	—	—	< 469	—N/A	4.12E+03	1.14E-01	
Si	—	77,100	82,100	77,500	79,400	78,700	78,800	2300	1.49E+05	5.28E-01	
Sn	—	—	—	—	—	—	< 2,820	—N/A	2.83E+02	9.64E+00	
Sr	[41]	[168]	[163]	[167]	[139]	[153]	[163]	7	4.12E+03	3.85E-02	
Te	—	—	—	—	—	—	< 2,820	—N/A	1.03E+03	2.73E+00	
Th	—	—	—	—	—	—	< 1,880	—N/A	4.12E+03	4.56E-01	
Ti	—	1,290	1,210	1,200	1,220	1,250	1,240	41	1.03E+04	1.20E-01	
Tl	—	—	—	—	—	—	< 47	—N/A	3.61E+03	1.30E-02	
U	—	[4530]	[4890]	[5190]	[4160]	[4600]	[4,800]	300	1.08E+05	4.44E-02	
V	—	—	—	—	—	—	< 94	—N/A	2.58E+02	3.64E-01	
W	—	—	—	—	—	—	< 3,750	—N/A	1.91E+03	1.87E+00	
Y	—	[101]	[101]	—	[87]	[99]	[100]	1	1.29E+03	7.79E-02	
Zn	—	[230]	[219]	[178]	[168]	[209]	[209]	22	3.35E+03	6.24E-02	
Zr	N/U	N/U	N/U	N/U	N/U	N/U	N/U	N/U	1.19E+05	N/U	N/U

— = Analyte not detected above the IDL; only average IDL reported as less than (<) value.
Results in brackets are qualified but they are used in the calculation of the average.

N/A Not Applicable
N/U Not Used

Analytical Summary Report Addendum 1, Revision 2 - March 7, 1997

Table A1-4.5. ICP-AES Acid Digestion--Vendor Product
C106 Privatization Contractor Sample

Analyte	Acid Digestion										Envelope D Specification		Comparison	
	Process Blank 97-1112-PB-3	C106-PPA/V1-3 97-1112-PB-3	C106-PPA/V1-3 97-1113-PB-3	C106-PPA/V2-3 97-1114-PB-3	C106-PPA/V2-3 97-1115-PB-3	C106-PPA/V2-3 97-1115-DPB-3	Average	Stdev	Uppr	Lower	Ave/Spe	Flag		
	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g		
Ag	--	119	140	134	112	107	128	13	4.38E+03	2.88E-02				
Al	[11]	60,400	60,300	62,200	53,300	51,400	59,100	3900	1.11E+05	5.33E-01				
As	--	--	--	--	--	--	< 62	--N/A--	1.29E+03	4.84E-02				
B	131	138	165	[120]	156	140	145	20	1.03E+04	1.40E-02				
Ba	--	500	495	505	449	432	487	26	3.61E+04	1.35E-02				
Be	--	--	--	--	--	--	< 2	--N/A--	5.15E+02	4.84E-03				
Bi	--	540	538	548	493	481	530	25	2.22E+04	2.39E-02				
Ca	[54]	4,780	5,060	5,130	4,330	4,170	4,830	360	5.67E+04	8.51E-02				
Cd	--	86.3	84.8	83.5	78.4	77.9	84	3	3.81E+04	2.32E-03				
Ce	--	[332]	[344]	[321]	[316]	[306]	[328]	12	6.44E+03	5.10E-02				
Co	--	[31]	[31]	[31]	[29]	[28]	[31]	1	3.61E+03	8.48E-03				
Cr	--	588	569	564	529	509	570	29	5.41E+03	1.05E-01				
Cu	[2]	140	129	130	121	117	130	8	3.86E+03	3.36E-02				
Dy	--	--	--	--	--	--	< 13	--N/A--	2.05E+02	6.05E-02				
Eu	--	--	--	--	--	--	< 25	--N/A--	1.29E+02	1.94E-01				
Fe	[2]	149,000	146,000	150,000	135,000	131,000	145,000	6900	2.29E+05	6.32E-01				
K	--	--	--	--	--	--	< 499	--N/A--	1.06E+04	4.72E-02				
La	--	114	[116]	[109]	[108]	[105]	112	4	2.06E+04	5.42E-03				
Li	--	[23]	[23]	[25]	[21]	[21]	[23]	2	1.11E+03	2.06E-02				
Mg	--	827	785	839	750	720	853	40	1.67E+04	4.79E-02				
Mn	--	3,110	3,040	3,080	2,810	2,700	3,010	140	5.15E+04	5.84E-02				
Mo	--	[23]	[23]	[22]	[21]	[21]	[22]	1	5.15E+03	4.27E-03				
Na	152	128,000	119,000	123,000	110,000	108,000	120,000	7600	1.55E+05	7.76E-01				
Nd	--	340	342	333	314	305	332	13	1.37E+04	2.43E-02				
Ni	[10]	1,360	1,320	1,300	1,220	1,180	1,300	59	1.88E+04	6.81E-02				
P	--	1,670	1,670	1,760	1,510	1,490	1,650	100	1.39E+04	1.19E-01				
Pb	--	3,980	3,970	3,990	3,630	3,520	3,890	180	8.76E+03	4.44E-01				
Pd	--	[201]	--	--	--	--	[201]	--N/A--	1.03E+03	1.95E-01				
Rh	--	--	--	--	--	--	< 75	--N/A--	1.03E+03	7.26E-02				
Ru	--	[567]	--	--	--	--	[567]	--N/A--	2.83E+03	1.67E-01				
Sb	--	--	--	--	--	--	< 128	--N/A--	6.70E+02	1.86E-02				
Se	--	[161]	[157]	[169]	[150]	[147]	[159]	8	4.12E+03	3.85E-02				
Si	[197]	1,580	2,260	5,440	1,430	[1170]	2,680	1900	1.49E+05	1.79E-02				
Sn	--	[1590]	--	--	--	--	[1,140]	640	2.83E+02	4.02E+00				
Sr	--	63.7	63.4	63.2	67.3	65.6	62	3	4.12E+03	1.50E-02				
Te	--	[614]	[617]	[632]	[672]	[698]	[606]	23	1.03E+03	4.91E-01				
Th	--	[457]	[445]	[410]	[401]	[375]	[428]	27	4.12E+03	1.04E-01				
Ti	--	369	360	354	334	314	364	25	1.03E+04	3.93E-02				
Tl	--	--	--	--	--	--	< 125	--N/A--	3.61E+03	3.48E-02				
U	--	[2330]	[2620]	[2640]	[2330]	[2270]	[2,530]	140	1.08E+05	2.34E-02				
V	--	[15]	--	--	--	--	[15]	--N/A--	2.88E+02	5.71E-02				
W	--	--	--	--	--	--	< 499	--N/A--	1.91E+03	2.62E-01				
Y	--	[88]	[88]	[89]	[79]	[76]	[86]	4	1.29E+03	6.66E-02				
Zn	[5]	114	[111]	[114]	[104]	[100]	[111]	5	3.35E+03	3.31E-02				
Zr	--	[62]	[105]	[104]	[68]	[66]	[85]	23	1.19E+05	7.15E-04				

-- = Analyte not detected above the IDL; only average IDL reported as less than (<) value.
Results in brackets are qualified but they are included in the calculation of the average.

N/A Not Applicable

5.0 REFERENCES

Brooks, KP, et al. 1997. *Bench-Scale Enhancement Sludge Washing and Gravity Settling of Hanford Tank C-106 Sludge*. PNNL-11432. Pacific Northwest National Laboratory, Richland, Washington.

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Wiemers, KD. 1996a. *Addendum 1 of the Letter of Instruction (LOI) for TWRS Privatization Contractor Samples Addressing Waste Envelope D Materials, Revision 1*. Pacific Northwest National Laboratory, Richland, Washington.

Wiemers, KD. 1996b. *Addendum 1 of the Letter of Instruction (LOI) for TWRS Privatization Contractor Samples Addressing Waste Envelope D Materials, Revision 2*. Pacific Northwest National Laboratory, Richland, Washington.

Analytical Summary Report Addendum 1, Revision 2 - March 7, 1997

Appendix A:

C106 Calculations for the Final Vendor Product Adjustment - After Decant/Dilute Treatment

A =	3511.45	g	Final VP weight (Table A1-2.1)
B =	2602.85	g	Distilled Water Added (Table A1-2.1)
C =	2002.32	g	Decanted Liquid (Table A1-2.1)
D =	1.10	g/ml	Density of initial slurry (Table 4.23 Summary Report++)
E =	126.5	g oxide/L	(Table 4.23 Summary Report ++ - $[(\% \text{oxide}/100)] \times [\text{density g/ml}] \times 1000 \text{ ml/L}]$)
F =	11.5	% oxide	Initial Vendor Product (Table 4.23 Summary Report ++)
G =	14.4	% solids	Initial Vendor Product (Table 4.23 Summary Report ++)
H =	162,000	ug Na/g	dried solids (CP Na average Table 4.3 Summary Report)
I =	14,950	ug/ml	ICP Na average Sample 97-0180 (Table A1-3.1)
J =	1.036	g/ml	Density of Sample 97-0180 (Table A1-3.2)
K =	1.348	g Na2O/g	Na factor $[(1 \text{ mole Na}/23 \text{ g}) / (1 \text{ mole Na}_2\text{O}/2 \text{ mole Na})] \times [62 \text{ g Na}_2\text{O}/\text{mole Na}_2\text{O}]$

++ For Summary Report Reference - See Section 1 Footnote 2

Weight of Initial slurry (A - B + C)	R =	2910.92	g
Volume of Initial slurry $[(R / D) / 1e3]$	S =	2.646	L
Total Oxide Initial Vendor Product (E * S)	T =	334.76	g oxide
Total Mass of Dried Solids Initial Vendor Product (T * [G/F])	U =	419.17	g dried solids
Total Sodium $[(H * U) / 1e6]$	V =	67.91	g Na
Sodium in Supernate $[(I/J) * [C/1e6]]$	W =	28.89	g Na
Sodium in Final Vendor Product (V - W)	X =	39.01	g Na
Final Oxide -- Adjusted only for Sodium Removal (T - [W*K])	Y =	295.81	g oxide

Adjustment calculation for each analyte measured in supernate >> g Analyte/31 g oxide:

$$\text{g Analyte} / 31 \text{ g oxide} = (M1 * U - M2 * C) * (M3/Y) * 1e-6$$

M1	Value of analyte concentration in dried slurry material (ug/g)
U	"Total Mass of Dried Solids Initial Vendor Product" (g)
M2	Value of analyte concentration in supernate (ug/g)
C	"Decanted Liquid Mass" (g)
M3	31 (number of g oxide / 31 g oxide)
Y	"Final Oxide Adjusted for Na Removal" (g oxide)

Na Example	M1 =	162000		
	M2 =	14431		
	M3 =	31	g Na/31 g Oxide =	4.69
	U =	419.17		
	C =	2002.32		
	V =	295.8		

**TANK WASTE REMEDIATION SYSTEM (TWRS)
PRIVATIZATION CONTRACTOR**

**ENVELOPE D SAMPLES
(C-106)**

ATTACHMENT 3

**PNNL ACL SUBMITTAL OF ORGANIC
AND RADIONUCLIDE RESULTS
FROM ANALYSIS OF
C-106 ORIGINAL MATERIAL SUPERNATE**

March 1997

Pacific Northwest National Laboratory

Pacific Northwest National Laboratory

Operated by Battelle for the U.S. Department of Energy

HNF-SD-WM-DP-225, REV. 1

October 31, 1996

Mr. Cary M. Seidel
Rust Federal Services, Hanford
Mail Stop T6-04
Richland, Washington 99352

Dear Cary:

PACIFIC NORTHWEST NATIONAL LABORATORY ANALYTICAL CHEMISTRY
LABORATORY SUBMITTAL OF ORGANIC AND RADIONUCLIDE RESULTS FROM
ANALYSIS OF C-106 ORIGINAL MATERIAL SUPERNATE

Attached is a copy of the ACL results from the organic and radiochemical analysis of the supernate from the C106 Original Material. Please recall that this additional scope was added in early September.

In late August the ACL received the C-106 Original Material for testing. The three subsamples received were mixed and transferred to three beakers in preparation for drying. It was observed that a clear layer developed rapidly. This layer appeared to be organic in nature and thus raised concern for the drying process. The decision was therefore made to remove the clear layer of supernate and save it for potential analysis at a later date. In early September the decision was made to go forward with limited analysis on the clear layer. A portion of the supernate was aliquoted and transferred for total alpha and beta analysis and organic characterization. These results are included herein. You will find a table (Table X) containing the alpha and beta results, performed in duplicate, and an organic report prepared by Gary Mong and Jim Campbell.

If you have any questions or concerns please contact me on 376-9454.

Very truly yours,

Michael W. Urie, Envelope D, C106 Project Manager
Analytical Chemistry Laboratory

cc: Karyn D. Wiemers, PNNL
Neil R. Brown, DOE

MWU/ean
Attachments (2)

Battelle Boulevard ■ P.O. Box 999 ■ Richland, WA 99352

Telephone (509) 376-9454 ■ Email mw_urie@pnl.gov ■ Fax (509) 372-2624

Cary Seidel
October 31, 1996
Page 2

bcc: KJ Kuhl-Klinger
File/LB

Analysis of C-106 Sample
G.M. Mong and J.A. Campbell

Introduction

The TWRS Privatization Envelope D starting composite material (96-05892, SPDL1-C106-003), was transferred from 150 ml bottles to 150 ml beakers for drying. The bottles were shaken vigorously before the materials were transferred to the beakers in an effort for a quantitative transfer with no additional liquid added. Ten to fifteen minutes after transferring to the beaker, the slurry solids settled. This resulted in an approximately 10 ml transparent liquid layer on top of the slurry. A concern that this layer might contain concentrated insoluble organics lead to a decision to pipette the clear supernatant before drying the solids. A portion of this transparent layer was submitted for organic analyses.

This report describes the organic analyses regime which was conducted on the separated supernate.

The analysis of potential major organic components related to tank wastes cannot be accomplished via a single analytical scheme. The carboxylates and chelators may be screened using gas chromatography/mass spectrometry (GC/MS) after methylation with BF_3 -methanol; acidic phosphate esters (such as bis(2-ethylhexyl)phosphate) require diazomethane derivatization for analysis. Nonpolar organics are commonly extracted with methylene chloride and identified directly using GC/MS analysis. The supernatant sample was analyzed in this fashion for chelators and chelator fragments, phosphate-containing components, and organic-soluble carbon using GC/MS.

Methylene chloride extracts of the sludge oil resulting from C-106 waste treatments have been previously analyzed¹, and the major component identified using derivatization gas chromatography/mass spectrometry (GC/MS) was bis(2-ethylhexyl)phosphate.

Experimental Methods

A portion of the sample was stirred with water to dissolve the salts and then extracted with measured volumes of methylene chloride. The methylene chloride extracts were separated, dried with a bed of sodium sulfate to remove residual aqueous radioactive components, taken to a minimum volume (300 μL) using a gentle stream of dry nitrogen and reconstituted to a known volume with methylene chloride. Solvent extracted samples were acidified and treated with an unquantified excess of ethereal diazomethane (formed from base treatment of N-methyl-N-nitrosourea) to simultaneously determine phosphate-containing organic components and organically soluble materials (e.g. TBP) by GC/MS analysis.

Aliquots of the aqueous portion were taken to dryness for analysis of chelators and chelator fragments. These samples were treated with BF_3 /methanol complex (2 mL) for 1 h at 100° C to derivatize potential chelator components such as EDTA, NTA, and HEDTA for subsequent analysis by GC/MS.

Mass spectrometric data were obtained on low-resolution GC/MS systems (HP 5988A and 5987) that were tuned with perfluorotributylamine (PFTBA) before use. Additional GC/MS data were obtained using a HP 5970 mass selective detector system, tuned with PFTBA before use.

Results and Discussion

Methylene chloride extraction of the C-106 sample did not afford appreciable residual material upon evaporation of the solvent. This minor residual was treated with ethereal HCl, evaporated a second time, and quenched with diazomethane. Very little of the diazomethane was consumed, indicating acidic phosphates at minimal concentration. Gas chromatography/mass spectrometry analysis of this extract indicates a value of about 60 $\mu\text{g}/\text{gram}$ bis(2-ethylhexyl)phosphate (DE2HP) to be present in the liquid supernatant as delivered from the hot cell. This value is based on a single point calibration and imperfect measurement of the amount of solvents used in the work-up of the small sample. More sample is required to refine this number. The mass spectrum of the analyte (methyl-DE2HP) appears to contain one major ion which may be assigned as m/z 125 or m/z 126 depending upon instrumental parameters. We will be pursuing a high-resolution mass spectrometric investigation of this material to further understand the mass spectral properties of DE2HP.

The remainder of the sample (after a 0.1 mL aliquot for potential TOC analysis and an additional subsample were placed in reserve for HPLC analysis), was dried, weakly acidified, and dried again. Boron trifluoride/methanol was added, the derivatized-material separated, and subsequently analyzed using GC/MS. The only organic of any consequence was oxalic acid, to the extent of about 6 mg/g of sample supernatant.

Typical organic components such as tributyl phosphate or normal paraffin hydrocarbons identified in other tanks were not present in any of the samples examined.

In summary, the major organic components identified and quantitated in the supernatant sample were bis(2-ethylhexyl)phosphate and oxalic acid. The concentrations were 60 $\mu\text{g}/\text{g}$ and 6 mg/g, respectively. Other organics such as EDTA, NTA, IDA, ED3A, and HEDTA were not observed. In addition, organically soluble components such as TBP and NPH were not observed.

Reference

Campbell, JA et al. 1996. Organic Tanks Safety Program: Advanced Organic Analysis FY 1996 Progress Report. PNNL-11309, Pacific Northwest National Laboratory, Richland, Washington.

Table X: Tank C106 Total Alpha and Beta Results (Original Material, Liquid Layer)

ALO #	CORE ID	Total Alpha uCi/ml	+/-% Error	Total Beta uCi/ml	+/-% Error
96-5893-Ni	SPDL1-C106-004 Liquid Layer	2.21E-01	4%	3.34E+01	4%
96-5893-Ni-Rep	SPDL1-C106-004 Liquid Layer	2.35E-01	4%	3.37E+01	4%
	RPD	6.1%		0.9%	
Sample Spike				98%	
Standard		110%		102%	
Blank		<2.E-4		<2.E-2	

L.P. Greenwood 9-17-96
Reviewed V. L. Seely 9/17/96

**TANK WASTE REMEDIATION SYSTEM (TWRS)
PRIVATIZATION CONTRACTOR**

**ENVELOPE D SAMPLES
(C-106)**

**ATTACHMENT 4
FINAL STATISTICAL REPORT**

March 1997

Pacific Northwest National Laboratory

ATTACHMENT TO RFSH-9752732

TANK WASTE REMEDIATION SYSTEM PRIVATIZATION
PRIVATE CONTRACTOR SAMPLES
FOR HIGH-LEVEL WASTE FEED ENVELOPE D, TANK 241-C-106
FINAL STATISTICAL REPORT

Consisting of 45 pages
(including cover page)

97012-RFSH

ATTACHMENT

TANK WASTE REMEDIATION SYSTEM PRIVATIZATION
CONTRACTOR SAMPLES

WASTE ENVELOPE D MATERIAL
Tank 241-C-106

Final Statistical Analysis Report

Prepared by: Terri L Welsh
Babcock & Wilcox Protec, Inc.
Safeguards Material Control

March 27, 1997

Consisting of 44 pages
including the cover page

LIST OF TERMS

Acid	nitric acid-hydrochloric acid digestion
ANOVA	analysis of variance
CI	confidence interval
CI/L	curies per liter (at 31 gram waste oxide)
CVAA	cold vapor atomic absorption
df	degrees of freedom
DQO	data quality objective
EL	"D" Envelope Limit
EQL	estimated quantitation limit
FS.Ni	potassium hydroxide fusion utilizing Ni crucibles
FS.Zr	sodium peroxide-sodium chloride fusion utilizing zirconium crucibles
GEA	gamma energy analysis
g/L	grams per liter (at 31 gram waste oxide)
HLW	high level waste
IC	ion chromatography
ICP/AES	inductively coupled plasma/atomic emission spectrometry
ICP/MS	inductively coupled plasma/mass spectrometry
IDL	instrument detection limit
IPT	integrated process product team
LL	lower limit
LOI	letter of instruction
$\mu\text{Ci/g d}$	microcuries per gram dried slurry
$\mu\text{g/g d}$	microgram per gram dried slurry
m/z	mass-to-charge ratio (ICP/MS)
NA	not available or not applicable
NH ₃ -N	the ammonia procedure reports the results as nitrogen
PNNL	Pacific Northwest National Laboratory
PHMC	Project Hanford Management Contract
Rad	Radiochemical analysis
REML	restricted maximum likelihood estimation
RSD	relative standard deviation
SAL	Shielded Analytical Laboratory
SYS	systematic variability
TIC	total inorganic carbon
TOC	total organic carbon
TWRS	tank waste remediation system
UL	upper limit
Water	water leaching
WtChem	wet chemistry
.W	indicates that the "less than" values were used in the statistical analysis at the upper value (e.g. 3 for < 3).
.WO	indicates that the "less than" values were deleted from the statistical analysis
$\bar{x}$	estimate of the mean
σ_{μ}	standard deviation associated with the mean, random variability
σ_{total}	standard deviation associated with the mean, combined random and systematic variability

TANK WASTE REMEDIATION SYSTEM PRIVATIZATION
CONTRACTOR SAMPLESWASTE ENVELOPE D MATERIAL
Tank 241-C-106

Final Statistical Analysis Report

1.0 INTRODUCTION

This statistical analysis report presents the evaluation of a set of analytical data generated for the Tank Waste Remediation System (TWRS) Privatization Contractor Samples, High-Activity Waste Feed Envelope D, Tank 241-C-106. Samples were received at the 325 building High Level Radiochemistry Facility and composited. The composited material was then transferred to the 324 building for Envelope D batch processing (Enhanced Sludge Washing). Subsamples of the original composited materials were collected from the batch processing system for subsequent chemical, radiological, and physical property measurements. Following batch processing, the resulting material (Vendor Product) was subsampled. The subsamples were sent to the 325 building for analysis in accordance with Addendum 1 of the Letter of Instruction (LOI) (Jones, 1996). Further treatment was required to meet the envelope D sodium specification. A limited set of analyses were completed for the resultant "treated" materials (Final Vendor Product). The Final Vendor Product was then bottled for shipment to the private contractors. This report uses data collected for the Vendor Product materials.

Twenty grab samples were taken from tank 241-C-106 between June 21, 1996, and June 24, 1996. All of the samples were taken from Riser 7 at sample depths between 406 and 414 inches. Sample depth is measured from the top of the riser to the mouth of the sample bottle. Each grab sample was individually homogenized. The homogenized samples were then transferred to a 2 liter carboy. The details regarding the compositing of the grab samples are provided by Urie (1997).

Following the batch processing as described by Brooks (1997), five subsamples of the Vendor Product were obtained for subsequent physical and chemical characterization and archive. Three of the five samples were submitted to the Shielded Analytical Laboratory (SAL) for analysis of chemical and radionuclide constituents. Each of the three subsamples (~125 mL) were placed into glass beakers for mixing and drying. After drying, each of the three dried slurries were homogenized, and then divided into two halves; forming a sample and a sample duplicate. Analytical sample preparations were conducted on these Vendor Product dried slurries. Additional details regarding the subsampling and sample preparations are provided by Urie (1997).

2.0 OBJECTIVES

The objectives of the statistical analysis were

- to provide estimates of the subsample variability for the analytes,

- to provide estimates of the analytical variability for the analytes,
- to compare the upper limit of a 95% confidence interval (CI) for the mean analyte concentration with the respective envelope limit.

Both the subsample variability and the analytical variability, as determined from the analytical measurements, are used to estimate the compositional ranges of the sample concentrations. The magnitude of the subsample variability also identifies possible sample inhomogeneity where the inhomogeneity could have been caused by entrained solids. The magnitude of the analytical variability identifies, for this sample matrix, those analytes with large analytical uncertainties. The large analytical uncertainty could be due to sample inhomogeneity, matrix interferences, or analyte concentrations close to the detection limit of the analytical procedure. The magnitude of the analytical uncertainties will be used to develop the integrated process product team (IPT) feed validation procedures and the data quality objectives (DQO) for high level waste (HLW) feed staging.

The upper limit of a one-sided 95% CI on the mean concentration takes into account the sampling and analytical uncertainties. Comparing the upper limit of this 95% CI for the mean concentration with the respective envelope limit is one indicator of the Project Hanford Management Contract's (PHMC) risk of delivering feed within the envelope specification.

3.0 STATISTICAL METHODOLOGY

The data used in the statistical analysis were obtained from Urie (1997). The differences between the three subsample results are used in computing the random "sampling" variability. The differences between the two measurements per subsample are used in computing the random "analytical" variability.

The data were statistically evaluated using one-way analysis of variance (ANOVA): the data are identified by one variable (the subsample). The one-way analysis of variance statistical model used to describe the structure of the data is

$$Y_{ij} = \mu + S_i + A_{ij},$$

$$i=1,2,\dots,a, \quad j=1,2,\dots,n_i,$$

where

Y_{ij}	=	concentration from the j^{th} analytical result from the i^{th} subsample
μ	=	the grand mean of the population
S_i	=	the effect of the i^{th} subsample
A_{ij}	=	the effect of the j^{th} analytical result from the i^{th} subsample

- a = the number of subsamples
- n_i = the number of analytical results from the i^{th} subsample.

The variable S_i , which represents the difference between the concentration of the i^{th} sample and the average content over the population, is assumed to be a random effect. This variable, as well as A_{ij} (which represents the analytical variability), are assumed to be uncorrelated and normally distributed with means zero and variances $\sigma^2(S)$ and $\sigma^2(A)$, respectively. Since every sample has its value of S_i , S_i is considered to be a random variable with a distribution over the population. This distribution has mean 0, since the S_i are defined as deviations from the population mean.

Estimates of $\sigma^2(S)$ and $\sigma^2(A)$ were obtained using Restricted Maximum Likelihood Estimation (REML) techniques. This method, applied to variance component estimation, is described in Harville, 1977. The results using the REML techniques were obtained using the statistical analysis package S-PLUS (Statistical Sciences, 1993).

The following paragraphs describe how the mean analyte concentration ($\bar{\mu}$), the standard deviation of the mean ($\hat{\sigma}_{\bar{\mu}}$), and the degrees of freedom (df) were determined using the results from the statistical model fit to the data.

Mean Analyte Concentration

The estimator, $\hat{\mu}$, is the maximum likelihood estimate of the mean. This estimator was determined by the structure of the data reflected by the statistical model. The estimate of $\bar{\mu}$ was obtained using REML techniques in S-PLUS.

Standard Deviation of the Mean

The estimated standard deviation of the mean (where the mean is the maximum likelihood estimate), $\hat{\sigma}_{\bar{\mu}}$, is the square root of a function of the variance estimates:

$$\hat{\sigma}^2(S) \text{ and } \hat{\sigma}^2(A).$$

These estimates were obtained using REML techniques. For unbalanced data, $\hat{\sigma}_{\bar{\mu}}$ is a complicated function of these variances.

Degrees of Freedom

The degrees of freedom (df) are dependent on the data structure or the statistical model used. The df associated with $\bar{\mu}$, the mean analyte concentration, for balanced data (every subsample has the same number of analytical measurements) is the number of subsamples minus one. For unbalanced data the df associated with $\bar{\mu}$ is approximated by the number of subsamples minus one.

Reference standards were analyzed as specified in the analytical procedures. The majority of the reference standards were within quality control limits. The reference standards and spike recoveries provide assurances that the analytical procedures are producing valid results. The majority of the spike

recoveries were within the customer specified limits. The analytes which had reference standard recoveries or spike recoveries outside the customer specified limits will be identified in the Final Analytical Report.

4.0 STATISTICAL RESULTS - Random Variability

The results of the statistical analyses for the Vendor Product data provided in Urie (1997) are listed in Table 4.1. The mean analyte concentration ($\bar{\mu}$), the relative standard deviation of the mean (RSD), the degrees of freedom (df), and the upper limit to the one-sided 95% confidence interval on the mean are included in Table 4.1. The RSD of the mean ($\hat{\sigma}_\mu/\bar{\mu}$) is defined as the standard deviation associated with the mean ($\hat{\sigma}_\mu$) divided by the mean.

The standard deviation for each analyte listed in Urie (1997) was computed by pooling all the analytical results (up to 6 measurements, 3 samples in duplicate). The standard deviation of the mean is then computed by dividing the standard deviation by the square root of n, where n is the number of measurements. The standard deviation of the mean in Table 4.1 was calculated using the sampling variability and the analytical variability (square root of $[\hat{\sigma}^2(S)/3 + \hat{\sigma}^2(A)/6]$ in the balanced case). The two values for the standard deviation of the mean will be different due to the assumptions used for the calculations.

The mean results provided in Urie (1997) may also be different from those listed in Table 4.1 due to the treatment of the data (especially the "less than" values).

The upper limit (UL) to a one-sided 95% confidence interval for the mean is

$$UL = \bar{\mu} + t_{(df, 0.05)} \times \hat{\sigma}_\mu. \quad (1)$$

In this equation, $t_{(df, 0.05)}$ is the quantile from Student's t distribution with df degrees of freedom for a one-sided 95% confidence interval. The upper limit of the one-sided confidence interval on the mean in Table 4.1 takes into account both the random sampling variability and the random analytical variability.

The analytical data for several analytes consisted of both quantitative values and "less than" values. A statistical analysis of the data was performed if at least 50% of the data were quantitative values. The ANOVA was computed using two different methodologies for these data.

The upper value of the "less than" (e.g., 3.5 for < 3.5) was used to represent all "less than" analytical values in the first computation. This produces a bias of unknown magnitude in both the mean analyte concentration and the variance associated with the mean; the mean analyte concentration is biased high. The extension ".w" was added to the analyte name in the tables to distinguish which analyte was statistically analyzed using "less than" values.

The "less than" values were deleted in the second computation. Deleting data produces unbalanced data sets which complicates the statistical analysis. Deleting data decreases the number of degrees of freedom. Deleting data also produces a bias of unknown magnitude in both the mean analyte concentration and the variance associated with the mean. The extension ".wo" was added to the analyte name in the tables to distinguish which analyte was statistically analyzed with the "less than" values deleted.

The statistical analysis was not performed for those analytes which had more than 50% of the analytical results reported as "less than" values. These analytes are listed in Table 4.2. Table 4.2 cites the largest value listed from the six analytical results.

The random variance component estimates were determined from ANOVA using REML techniques. These variance component estimates (from which the standard deviation associated with the mean concentration were calculated) are provided in Table 4.3. The RSD for each variance component has also been provided in Table 4.3. The RSDs give an indication of the magnitude of the random sampling variability and the magnitude of the random analytical variability.

The analytes which have a large (> 20%) sampling variability and a large (> 20%) analytical variability are listed in Table 4.4. The concentration of many of these analytes are near the measurement detection limits causing a large variability in their results. These analytes are marked with a "*" in the table. The analytical measurements are discussed in Urie (1997). Since the analyses were performed on "slurry" samples, the large sampling variability may be attributed to inhomogeneity in the suspended solids composition and reproducibility of the solids fraction sampled.

5.0 STATISTICAL RESULTS - Random and Systematic Variability

There are two sources of variability; random variability and systematic variability. Random variability (the chance variability encountered in all measurement work) is the variability that affects each member of a set of data (measurements) in a different manner. For this data set, the random analytical variability is estimated from the duplicate results obtained from each subsample (within-subsample differences) and the random sampling variability is estimated from the between-subsample differences.

Systematic variability (sometimes referred to as accuracy) is a variability that affects all members of a data set (the variability remains constant over a series of replicated measurements). Spike recoveries or reference standard recoveries are used to estimate the systematic analytical variability. Associated with analytical procedures for a particular sample matrix. Assigned values of 10%, 25%, 50%, 75%, and 100% were used as estimates of the systematic analytical variability. It is not possible to estimate the systematic sampling variability.

The total measurement uncertainty (random and systematic variability combined) was estimated using the following equation

$$\hat{\sigma}_{total}^2 = \hat{\sigma}_{random}^2 + \hat{\sigma}_{systematic}^2 \quad (2)$$

where $\hat{\sigma}_{random}$ is the relative standard deviation of the mean provided in Table 4.1 and $\hat{\sigma}_{systematic}$ is the systematic variability (in terms of RSD).

For the analytes listed in Table 4.1, the RSD of the mean, $\hat{\sigma}_{total}/\bar{\mu}$, was determined using each assigned systematic variability estimate (in terms of RSD, $\hat{\sigma}_{systematic} = 10\%, 25\%, 50\%, 75\%$, and 100%). The upper limit of the one-sided 95% confidence interval on the mean was then calculated for each analyte using the df associated with the random variability. The upper limit (UL) to a one-sided 95% confidence interval for the mean is

$$UL = \bar{\mu} + t_{(df, 0.05)} \times \hat{\sigma}_{total} \quad (3)$$

In this equation, $t_{(df, 0.05)}$ is the quantile from Student's t distribution with df degrees of freedom for a one-sided 95% confidence interval. The upper limit to a one-sided confidence interval on the mean using $\hat{\sigma}_{total}$ takes into account the random sampling variability, the random analytical variability, and the systematic analytical variability.

The use of "assigned" systematic variability estimates was to determine the effect of the total measurement uncertainty on the ratio UL/EL (UL is the upper limit of the one-sided 95% CI on the mean concentration and EL is the envelope D specification limit). A ratio of less than one indicates that the upper limit to the one-sided 95% CI on the mean analyte concentration is within the envelope D specification.

The UL/EL ratios, determined from varying the systematic variability, indicate when the upper limit to the one-sided 95% CI on the mean analyte concentration might exceed the envelope criterion. For those analyte concentrations which exceed the envelope D specifications ($UL/EL > 1$), the observed systematic variability is compared to the "assigned" systematic variability. If the observed systematic variability is less than this "assigned" systematic variability, the upper limit to the one-sided 95% CI on the mean concentration is below the envelope D specifications. The analytes flagged in this assessment have a higher risk with respect to exceeding the envelope criterion. Further discussion of analytes "at risk" is provided in Section 6.0.

6.0 COMPARISON OF ANALYTICAL RESULTS TO THE ENVELOPE D SPECIFICATION

The envelope "D" specification limits are given in terms of g/L (at 31 gram waste oxide). The analytes reported with units of $\mu\text{g/g}$ dried slurry were converted to g/L (at 31 gram waste oxide) using equation 4. The analytes reported with units of $\mu\text{Ci/g}$ dried slurry were converted to Ci/L (at 31 gram waste oxide) using equation 5. The weight percent dried slurry and the weight percent oxide were obtained from laboratory measurements and are listed in Kuhl-Klinger (1997).

$$\frac{g}{L} = \frac{\mu g_{\text{analyte}}}{g_{\text{dried slurry}}} \times \frac{g}{1000000 \mu g} \times \frac{14.4 \frac{g_{\text{dried slurry}}}{\text{total } g}}{11.5 \frac{g_{\text{oxide}}}{\text{total } g}} \times \frac{31 \frac{g_{\text{oxide}}}{L}}{L} \quad (4)$$

$$\frac{Ci}{L} = \frac{\mu Ci_{\text{analyte}}}{g_{\text{dried slurry}}} \times \frac{Ci}{1000000 \mu Ci} \times \frac{14.4 \frac{g_{\text{dried slurry}}}{\text{total } g}}{11.5 \frac{g_{\text{oxide}}}{\text{total } g}} \times \frac{31 \frac{g_{\text{oxide}}}{L}}{L} \quad (5)$$

Many of the analyte concentrations were determined using two analytical procedures (ICP/AES and ICP/MS) with multiple sample preparations (Acid, FS.Ni, and FS.Zr). The summary statistics were reported for each measurement method and sample preparation in Table 4.1.

The Analytical Summary Report (Urie, 1997) lists the "most reasonable" average per analyte for comparison to the Envelope D specifications. In most cases, the "most reasonable" estimate reflects a specific sample preparation and analysis method. The results are presented in units of g/L and Ci/L (at 31 gram waste oxide) for comparison with the envelope specifications. The statistical evaluation of the analytical data selected as "most reasonable" is presented in Tables 6.1 and 6.2. These tables contain (if applicable) the mean analyte concentration ($\bar{\mu}$), the RSD of the mean ($\sigma_p/\bar{\mu}$), the upper limit to the one-sided 95% confidence interval on the mean (random variability only), and the ratio of the upper limit to the one-sided 95% CI on the mean to the envelope "D" specification limit.

The ratio of the upper limit to the one-sided 95% CI on the mean concentration to the envelope D specification limits provides a method of comparing the analyte concentration to the envelope criterion. A ratio less than 1.0 indicates that the mean analyte concentration is below the envelope criterion. For the analytes which had concentrations reported as "less than" values, the ratio of the analyte to the envelope specification limit was calculated using the upper limit of the less than value listed; a conservative approach.

The upper limits of the 95% CI for the mean concentration were below the respective envelope "D" maximum specification limits for the majority of the analytes. The Vendor Product analytes for which the UL of the 95% CI for the mean concentration exceeded the Envelope D specifications are Na, Pt (a trace element), ^{121m}Sn , and ^{235}U (a trace radionuclide). Three radionuclides (^{110m}Ag , ^{242}Pu , and ^{119m}Sn) exceeded the Envelope D specifications if the maximum detection limits are used as the results.

The result for $^{243/244}\text{Cm}$ exceeds the Envelope D specification if compared to the ^{243}Cm specification. The result for $^{144}\text{Ce}/^{144}\text{Pr}/^{144m}\text{Pr}$ exceeds the Envelope D specification if compared to the ^{144m}Pr specification.

These results do not differ from those presented in Urie (1997).

Five analytes (Al, Fe, Na, Ni, and CO_3) have Envelope D minimum specification limits. Compliance with the minimum specification limits is not addressed in this report.

The comparison of the 95% UL with the envelope limit provides an indicator of the risk to be assumed by the PHMC in delivering feed that meets the envelope specification. It is not intended by this comparison to imply that the 95% UL will be the basis for feed composition verification. Current U.S. Department of Energy, Richland Operations Office strategy is to use the mean concentration rather than the 95% UL for the feed composition verification.

Table 6.3 contains the ratio of the upper limit to the one-sided 95% CI on the mean to the envelope specification limit where the total measurement uncertainty (σ_{total}) was used in calculating UL (the upper limit to the one-sided 95% CI on the mean). Five ratios (as explained in Section 5.0) are presented in Table 6.3 since σ_{total} was calculated using the five assigned values for the systematic variability. The five ratios indicate the effect of the total measurement uncertainty on the UL.

Using a total measurement uncertainty of 100% (calculated from equation 2 using the random variability listed in Table 4.1 and a systematic variability of 100%), the UL is below the envelope D specification for 52 of the 71 comparisons in Table 6.3. The systematic variability at which the remaining 19 comparisons have a ratio greater than one is presented in Table 6.4. This table also includes the observed systematic variability.

The UL based on σ_a (the random sampling and analytical variability) identified Na, Pt (a trace element), ^{121}mSn , and ^{235}U (a trace radionuclide) as analytes exceeding the envelope D specifications. The UL based on σ_{total} (the total measurement uncertainty) adds Ag and Yb (a trace element) to the list of analytes whose UL exceeds the envelope D specifications.

7.0 REFERENCES

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Table 4.1. Tank 241-C-106 Vendor Product
Summary Statistics - Random Variability. (page 1 of 9)

Analyte @	Method	Digest	Units \$	$\bar{x}$		RSD ($\bar{x}$) (%)	df	95% UL
Ag	ICP/AES	Acid	$\mu\text{g/g d}$	7.56E+01		2.69	2	8.15E+01
Ag	ICP/AES	FS.Ni	$\mu\text{g/g d}$	1.03E+03		9.34	2	1.31E+03
Ag	ICP/AES	FS.Zr	$\mu\text{g/g d}$	3.18E+03		8.76	2	3.99E+03
Al	ICP/AES	Acid	$\mu\text{g/g d}$	4.79E+04		1.40	2	4.99E+04
Al	ICP/AES	FS.Ni	$\mu\text{g/g d}$	5.12E+04		2.63	2	5.51E+04
Al	ICP/AES	FS.Zr	$\mu\text{g/g d}$	4.95E+04		2.88	2	5.37E+04
Al	ICP/MS	Acid	$\mu\text{g/g d}$	3.34E+04		2.96	2	3.63E+04
²⁴¹ Am.aaa	Rad	FS.Ni	$\mu\text{Ci/g d}$	2.77E+00		2.55	2	2.97E+00
²⁴¹ Am.gea	Rad	FS.Ni	$\mu\text{Ci/g d}$	3.15E+00		9.40	2	4.01E+00
As	ICP/MS	Acid	$\mu\text{g/g d}$	1.55E+02		4.04	2	1.73E+02
As	ICP/MS	FS.Ni	$\mu\text{g/g d}$	2.86E+02		7.38	2	3.48E+02
As	ICP/MS	FS.Zr	$\mu\text{g/g d}$	2.56E+02		13.55	2	3.57E+02
B	ICP/AES	Acid	$\mu\text{g/g d}$	2.25E+02		17.58	2	3.40E+02
B	ICP/AES	FS.Ni	$\mu\text{g/g d}$	3.03E+02	*	18.37	2	4.66E+02
B	ICP/MS	Acid	$\mu\text{g/g d}$	1.86E+02		21.29	2	3.02E+02
B	ICP/MS	FS.Ni	$\mu\text{g/g d}$	2.29E+02		21.41	2	3.73E+02
B	ICP/MS	FS.Zr	$\mu\text{g/g d}$	1.01E+02		38.58	2	2.15E+02
Ba	ICP/AES	Acid	$\mu\text{g/g d}$	4.15E+02		1.17	2	4.29E+02
Ba	ICP/AES	FS.Ni	$\mu\text{g/g d}$	4.07E+02		2.85	2	4.40E+02
Ba	ICP/AES	FS.Zr	$\mu\text{g/g d}$	4.37E+02		3.08	2	4.76E+02
Ba	ICP/MS	Acid	$\mu\text{g/g d}$	3.62E+02		1.11	2	3.73E+02
Ba	ICP/MS	FS.Ni	$\mu\text{g/g d}$	4.12E+02		1.94	2	4.35E+02
Ba	ICP/MS	FS.Zr	$\mu\text{g/g d}$	4.27E+02		1.29	2	4.43E+02
Bi	ICP/AES	Acid	$\mu\text{g/g d}$	4.30E+02		1.18	2	4.45E+02
Bi	ICP/AES	FS.Ni	$\mu\text{g/g d}$	5.38E+02	*	1.42	2	5.60E+02
Bi	ICP/AES	FS.Zr	$\mu\text{g/g d}$	5.82E+02	*	4.06	2	6.50E+02
C ₂ O ₄	IC	Water	$\mu\text{g/g d}$	1.70E+05		4.24	2	1.91E+05
¹⁴ C	Rad	FS.Ni	$\mu\text{Ci/g d}$	2.87E-03		78.53	2	9.45E-03
CN ⁻	WtChem	Direct	$\mu\text{g/g d}$	9.50E+00		5.59	2	1.11E+01
Ca	ICP/AES	Acid	$\mu\text{g/g d}$	3.84E+03		1.22	2	3.98E+03
Ca	ICP/AES	FS.Ni	$\mu\text{g/g d}$	6.69E+03		4.37	2	7.54E+03
Ca	ICP/AES	FS.Zr	$\mu\text{g/g d}$	9.85E+03		4.26	2	1.11E+04

@ = Table does not include analytes where more than 50% of the data are "less than" values.

\$ = $\mu\text{g/g d}$ is $\mu\text{g/g}$ dried slurry; $\mu\text{Ci/g d}$ is $\mu\text{Ci/g}$ dried slurry.

* = At least one analytical result was above the IDL but below the EQL.

Table 4.1. Tank 241-C-106 Vendor Product
Summary Statistics - Random Variability. (page 2 of 9)

Analyte a	Method	Digest	Units \$	$\bar{A}$		RSD($\bar{A}$) (%)	df	95% UL
Ca	ICP/MS	Acid	$\mu\text{g/g d}$	9.57E+03		4.79	2	1.09E+04
Ca	ICP/MS	FS.Ni	$\mu\text{g/g d}$	1.72E+03		7.78	2	2.11E+03
Ca	ICP/MS	FS.Zr	$\mu\text{g/g d}$	3.77E+03		5.02	2	4.32E+03
Cd	ICP/AES	Acid	$\mu\text{g/g d}$	7.30E+01		7.35	2	8.86E+01
Cd	ICP/AES	FS.Ni	$\mu\text{g/g d}$	8.10E+01	*	1.71	2	8.51E+01
Cd	ICP/AES	FS.Zr	$\mu\text{g/g d}$	8.08E+01	*	2.32	2	8.62E+01
Cd	ICP/MS	Acid	$\mu\text{g/g d}$	5.81E+01		1.22	2	6.01E+01
Cd	ICP/MS	FS.Ni	$\mu\text{g/g d}$	9.05E+01		5.13	2	1.04E+02
Cd	ICP/MS	FS.Zr	$\mu\text{g/g d}$	7.41E+01		6.60	2	8.83E+01
Ce	ICP/AES	Acid	$\mu\text{g/g d}$	2.99E+02	*	1.75	2	3.14E+02
Ce	ICP/MS	Acid	$\mu\text{g/g d}$	1.59E+02		1.68	2	1.67E+02
Ce	ICP/MS	FS.Ni	$\mu\text{g/g d}$	1.60E+02		8.96	2	2.01E+02
Ce	ICP/MS	FS.Zr	$\mu\text{g/g d}$	2.02E+02		6.52	2	2.41E+02
Cl ⁻	IC	Water	$\mu\text{g/g d}$	1.26E+02		8.49	2	1.57E+02
$^{242}\text{Cm}/^{242}\text{Am}/^{242m}\text{Am}$	Rad	FS.Ni	$\mu\text{Ci/g d}$	8.07E-03		10.14	2	1.05E-02
$^{243}/^{244}\text{Cm}$	Rad	FS.Ni	$\mu\text{Ci/g d}$	9.86E-02		12.97	2	1.36E-01
Co	ICP/AES	Acid	$\mu\text{g/g d}$	2.81E+01	*	1.06	2	2.90E+01
Co	ICP/MS	Acid	$\mu\text{g/g d}$	1.57E+01		1.23	2	1.62E+01
Co	ICP/MS	FS.Ni	$\mu\text{g/g d}$	2.62E+01		12.15	2	3.55E+01
Co	ICP/MS	FS.Zr	$\mu\text{g/g d}$	2.18E+01		5.95	2	2.56E+01
^{60}Co	Rad	FS.Ni	$\mu\text{Ci/g d}$	4.91E-01		3.11	2	5.36E-01
Cr	ICP/AES	Acid	$\mu\text{g/g d}$	5.33E+02		0.98	2	5.48E+02
Cr	ICP/AES	FS.Ni	$\mu\text{g/g d}$	8.02E+02		2.70	2	8.66E+02
Cr	ICP/AES	FS.Zr	$\mu\text{g/g d}$	7.00E+02		4.02	2	7.82E+02
Cr	ICP/MS	Acid	$\mu\text{g/g d}$	4.26E+02		1.05	2	4.39E+02
Cr	ICP/MS	FS.Ni	$\mu\text{g/g d}$	7.74E+02		6.05	2	9.11E+02
Cr	ICP/MS	FS.Zr	$\mu\text{g/g d}$	7.39E+02		3.93	2	8.24E+02
^{133}Cs	ICP/MS	Acid	$\mu\text{g/g d}$	1.04E+01		3.85	2	1.16E+01
^{133}Cs	ICP/MS	FS.Ni	$\mu\text{g/g d}$	1.80E+01		9.06	2	2.27E+01
^{133}Cs	ICP/MS	FS.Zr	$\mu\text{g/g d}$	7.18E+00		12.39	2	9.78E+00
^{133}Cs	Rad(MS)	FS.Ni	$\mu\text{g/g d}$	1.57E+01		4.71	2	1.78E+01
^{135}Cs	Rad	FS.Ni	$\mu\text{Ci/g d}$	2.86E-03		3.15	2	3.13E-03
$^{137}\text{Cs/Ba}$	Rad	FS.Ni	$\mu\text{Ci/g d}$	4.96E+02		3.29	2	5.43E+02

a = Table does not include analytes where more than 50% of the data are "less than" values.

\$ = $\mu\text{g/g d}$ is $\mu\text{g/g}$ dried slurry; $\mu\text{Ci/g d}$ is $\mu\text{Ci/g}$ dried slurry.

* = At least one analytical result was above the IDL but below the EOL.

HNF-SD-WM-DP-225, REV. 1

Table 4.1. Tank 241-C-106 Vendor Product
Summary Statistics - Random Variability. (page 3 of 9)

Analyte a	Method	Digest	Units b	$\bar{x}$		RSD ($\bar{x}$) (%)	df	95% UL
Cu	ICP/AES	Acid	$\mu\text{g/g d}$	7.75E+01		0.99	2	7.97E+01
Cu	ICP/AES	FS.Ni	$\mu\text{g/g d}$	1.09E+02	*	1.78	2	1.15E+02
Cu	ICP/AES	FS.Zr	$\mu\text{g/g d}$	1.09E+02	*	6.83	2	1.30E+02
Cu	ICP/MS	Acid	$\mu\text{g/g d}$	1.10E+02		0.75	2	1.12E+02
Cu	ICP/MS	FS.Zr	$\mu\text{g/g d}$	1.56E+02		4.22	2	1.76E+02
Cu.w	ICP/MS	FS.Ni	$\mu\text{g/g d}$	1.13E+01		71.06	2	3.48E+01
Cu.wo	ICP/MS	FS.Ni	$\mu\text{g/g d}$	1.89E+01		43.91	1	7.13E+01
Dy	ICP/MS	Acid	$\mu\text{g/g d}$	1.47E+00		2.56	2	1.58E+00
Dy	ICP/MS	FS.Ni	$\mu\text{g/g d}$	2.02E+01		17.37	2	3.04E+01
Dy	ICP/MS	FS.Zr	$\mu\text{g/g d}$	3.60E+00		32.62	2	7.04E+00
Er	ICP/MS	Acid	$\mu\text{g/g d}$	7.67E-01		2.75	2	8.28E-01
Er	ICP/MS	FS.Ni	$\mu\text{g/g d}$	1.31E+01		14.64	2	1.87E+01
Eu	ICP/MS	Acid	$\mu\text{g/g d}$	6.46E+00		1.49	2	6.74E+00
Eu	ICP/MS	FS.Ni	$\mu\text{g/g d}$	7.87E+00		30.72	2	1.49E+01
Eu	ICP/MS	FS.Zr	$\mu\text{g/g d}$	7.84E+00		6.54	2	9.34E+00
¹⁵⁴ Eu	Rad	FS.Ni	$\mu\text{Ci/g d}$	3.79E+00		1.76	2	3.99E+00
¹⁵⁵ Eu	Rad	FS.Ni	$\mu\text{Ci/g d}$	3.13E+00		4.02	2	3.50E+00
Fe	ICP/AES	Acid	$\mu\text{g/g d}$	1.37E+05		1.06	2	1.41E+05
Fe	ICP/AES	FS.Ni	$\mu\text{g/g d}$	1.41E+05		3.29	2	1.54E+05
Fe	ICP/AES	FS.Zr	$\mu\text{g/g d}$	1.31E+05		3.65	2	1.44E+05
Fe	ICP/MS	Acid	$\mu\text{g/g d}$	1.10E+05		3.24	2	1.20E+05
⁵⁵ Fe	Rad	FS.Ni	$\mu\text{Ci/g d}$	1.53E-01		2.62	2	1.65E-01
Gd	ICP/MS	Acid	$\mu\text{g/g d}$	1.36E+01		3.49	2	1.50E+01
Gd	ICP/MS	FS.Ni	$\mu\text{g/g d}$	2.50E+01		11.29	2	3.32E+01
Gd	ICP/MS	FS.Zr	$\mu\text{g/g d}$	1.21E+01		17.49	2	1.83E+01
Hg	CVAA	Direct	$\mu\text{g/g d}$	3.43E+02		1.94	2	3.63E+02
Ho	ICP/MS	Acid	$\mu\text{g/g d}$	1.42E-01		11.00	2	1.87E-01
Ho	ICP/MS	FS.Ni	$\mu\text{g/g d}$	6.07E+00		14.78	2	8.69E+00
¹²⁹ I.w	ICP/MS	Acid	$\mu\text{g/g d}$	1.18E+00		11.16	2	1.56E+00
¹²⁹ I.w	ICP/MS	Acid	$\mu\text{Ci/g d}$	2.18E-04		7.28	2	2.65E-04
¹²⁹ I.wo	ICP/MS	Acid	$\mu\text{g/g d}$	1.25E+00		10.80	2	1.64E+00
¹²⁹ I.wo	ICP/MS	Acid	$\mu\text{Ci/g d}$	2.37E-04		9.24	2	3.00E-04
K	ICP/AES	FS.Zr	$\mu\text{g/g d}$	7.59E+03	*	8.91	2	9.56E+03

a = Table does not include analytes where more than 50% of the data are "less than" values.

b = $\mu\text{g/g d}$ is $\mu\text{g/g}$ dried slurry; $\mu\text{Ci/g d}$ is $\mu\text{Ci/g}$ dried slurry.

* = At least one analytical result was above the IDL but below the ECL.

Table 4.1. Tank 241-C-106 Vendor Product
Summary Statistics - Random Variability. (page 4 of 9)

Analyte a	Method	Digest	Units b	$\bar{x}$		RSD($\bar{x}$) (%)	df	95% UL
La	ICP/AES	Acid	$\mu\text{g/g d}$	9.61E+01	*	1.37	2	8.91E+01
La	ICP/AES	FS.Ni	$\mu\text{g/g d}$	1.19E+02	*	3.45	2	1.30E+02
La	ICP/AES	FS.Zr	$\mu\text{g/g d}$	1.64E+02	*	6.48	2	1.95E+02
La	ICP/MS	Acid	$\mu\text{g/g d}$	8.50E+01		1.68	2	8.91E+01
La	ICP/MS	FS.Ni	$\mu\text{g/g d}$	8.06E+01		7.53	2	9.83E+01
La	ICP/MS	FS.Zr	$\mu\text{g/g d}$	9.22E+01		9.41	2	1.18E+02
Li	ICP/AES	Acid	$\mu\text{g/g d}$	4.59E+01		42.50	2	1.03E+02
Li	ICP/AES	FS.Zr	$\mu\text{g/g d}$	9.39E+01	*	14.81	2	1.34E+02
Mg	ICP/AES	Acid	$\mu\text{g/g d}$	7.54E+02		1.53	2	7.88E+02
Mg	ICP/AES	FS.Ni	$\mu\text{g/g d}$	1.36E+03	*	6.19	2	1.60E+03
Mg	ICP/AES	FS.Zr	$\mu\text{g/g d}$	1.31E+03	*	3.88	2	1.45E+03
Mg	ICP/MS	Acid	$\mu\text{g/g d}$	4.45E+02		1.96	2	4.71E+02
Mg	ICP/MS	FS.Ni	$\mu\text{g/g d}$	1.22E+03		7.52	2	1.49E+03
Mg	ICP/MS	FS.Zr	$\mu\text{g/g d}$	1.26E+03		3.35	2	1.52E+03
Mn	ICP/AES	Acid	$\mu\text{g/g d}$	2.60E+03		0.95	2	2.67E+03
Mn	ICP/AES	FS.Ni	$\mu\text{g/g d}$	3.14E+03		2.34	2	3.35E+03
Mn	ICP/AES	FS.Zr	$\mu\text{g/g d}$	2.59E+03		3.58	2	2.86E+03
Mn	ICP/MS	Acid	$\mu\text{g/g d}$	2.04E+03		2.29	2	2.18E+03
Mn	ICP/MS	FS.Ni	$\mu\text{g/g d}$	2.81E+03		4.03	2	3.14E+03
Mn	ICP/MS	FS.Zr	$\mu\text{g/g d}$	2.56E+03		2.77	2	2.77E+03
Mo	ICP/AES	Acid	$\mu\text{g/g d}$	2.67E+01	*	0.90	2	2.74E+01
Mo	ICP/MS	Acid	$\mu\text{g/g d}$	9.60E+00		2.59	2	1.03E+01
Mo	ICP/MS	FS.Ni	$\mu\text{g/g d}$	5.70E+01		13.98	2	8.02E+01
Mo	ICP/MS	FS.Zr	$\mu\text{g/g d}$	2.79E+01		9.95	2	3.60E+01
NH ₃ -N	WChem	Water	$\mu\text{g/g d}$	1.75E+01		20.07	2	2.78E+01
NO ₂ ⁻	IC	Water	$\mu\text{g/g d}$	2.07E+03		10.20	2	2.68E+03
NO ₃ ⁻	IC	Water	$\mu\text{g/g d}$	8.86E+02		30.56	2	1.68E+03
Na	ICP/AES	Acid	$\mu\text{g/g d}$	1.62E+05		2.93	2	1.76E+05
Na	ICP/AES	FS.Ni	$\mu\text{g/g d}$	1.42E+05		3.88	2	1.58E+05
Na	ICP/MS	Acid	$\mu\text{g/g d}$	1.82E+05		6.11	2	2.14E+05
^{93m} Nb	Rad	FS.Ni	$\mu\text{Ci/g d}$	2.37E-01		30.19	2	4.47E-01
Nd	ICP/AES	Acid	$\mu\text{g/g d}$	2.82E+02		1.19	2	2.91E+02
Nd	ICP/AES	FS.Ni	$\mu\text{g/g d}$	3.79E+02	*	4.73	2	4.31E+02

a = Table does not include analytes where more than 50% of the data are "less than" values.

b = $\mu\text{g/g d}$ is $\mu\text{g/g}$ dried slurry; $\mu\text{Ci/g d}$ is $\mu\text{Ci/g}$ dried slurry.

* = At least one analytical result was above the IDL but below the EQL.

Table 4.1. Tank 241-C-106 Vendor Product
Summary Statistics - Random Variability. (page 5 of 9)

Analyte a	Method	Digest	Units \$	$\bar{x}$		RSD($\bar{x}$) (%)	df	95% UL
Nd	ICP/AES	FS.Zr	$\mu\text{g/g d}$	5.17E+02	*	4.69	2	5.87E+02
Nd	ICP/MS	Acid	$\mu\text{g/g d}$	1.93E+02		1.06	2	1.99E+02
Nd	ICP/MS	FS.Ni	$\mu\text{g/g d}$	2.33E+02		8.00	2	2.87E+02
Nd	ICP/MS	FS.Zr	$\mu\text{g/g d}$	2.24E+02		3.99	2	2.50E+02
Ni	ICP/AES	Acid	$\mu\text{g/g d}$	1.13E+03		1.10	2	1.17E+03
Ni	ICP/AES	FS.Zr	$\mu\text{g/g d}$	1.66E+03		4.53	2	1.88E+03
Ni	ICP/MS	Acid	$\mu\text{g/g d}$	9.49E+02		2.42	2	1.02E+03
Ni	ICP/MS	FS.Zr	$\mu\text{g/g d}$	1.61E+03		2.35	2	1.72E+03
⁵⁹ Ni.w	Rad	FS.Zr	$\mu\text{Ci/g d}$	2.20E-02		10.23	2	2.86E-02
⁵⁹ Ni.wo	Rad	FS.Zr	$\mu\text{Ci/g d}$	1.97E-02		14.16	2	2.79E-02
⁶³ Ni	Rad	FS.Zr	$\mu\text{Ci/g d}$	2.40E+00		2.39	2	2.57E+00
²³⁷ Np	ICP/MS	Acid	$\mu\text{g/g d}$	4.48E+00		1.70	2	4.70E+00
²³⁷ Np	ICP/MS	Acid	$\mu\text{Ci/g d}$	3.16E-03		1.70	2	3.32E-03
²³⁷ Np	ICP/MS	FS.Zr	$\mu\text{g/g d}$	5.57E+00		9.49	2	7.11E+00
²³⁷ Np	ICP/MS	FS.Zr	$\mu\text{Ci/g d}$	3.92E-03		9.49	2	5.01E-03
P	ICP/AES	Acid	$\mu\text{g/g d}$	1.34E+03		2.94	2	1.46E+03
P	ICP/AES	FS.Ni	$\mu\text{g/g d}$	3.33E+03		4.58	2	3.77E+03
P	ICP/AES	FS.Zr	$\mu\text{g/g d}$	3.84E+03		10.59	2	5.02E+03
PO ₄ ³⁻	IC	Water	$\mu\text{g/g d}$	1.60E+02		8.94	2	2.02E+02
Pb	ICP/AES	Acid	$\mu\text{g/g d}$	3.43E+03		1.11	2	3.54E+03
Pb	ICP/AES	FS.Ni	$\mu\text{g/g d}$	2.95E+03		8.53	2	3.69E+03
Pb	ICP/AES	FS.Zr	$\mu\text{g/g d}$	3.53E+03		3.49	2	3.89E+03
Pb	ICP/MS	Acid	$\mu\text{g/g d}$	2.85E+03		3.52	2	3.15E+03
Pb	ICP/MS	FS.Zr	$\mu\text{g/g d}$	2.55E+03		1.89	2	2.69E+03
Pd	ICP/MS	Acid	$\mu\text{g/g d}$	9.99E+00		2.69	2	1.08E+01
Pd	ICP/MS	FS.Ni	$\mu\text{g/g d}$	2.35E+01		22.75	2	3.91E+01
Pd	ICP/MS	FS.Zr	$\mu\text{g/g d}$	2.74E+01		7.63	2	3.35E+01
¹⁰⁷ Pd.w	ICP/MS	Acid	$\mu\text{g/g d}$	1.35E+00		61.12	2	3.76E+00
¹⁰⁷ Pd.w	ICP/MS	Acid	$\mu\text{Ci/g d}$	7.18E-04		58.24	2	1.94E-03
¹⁰⁷ Pd.wo	ICP/MS	Acid	$\mu\text{g/g d}$	2.20E+00		63.85	1	1.11E+01
¹⁰⁷ Pd.wo	ICP/MS	Acid	$\mu\text{Ci/g d}$	1.14E-03		64.59	1	5.77E-03

a = Table does not include analytes where more than 50% of the data are "less than" values.

\$ = $\mu\text{g/g d}$ is $\mu\text{g/g}$ dried slurry; $\mu\text{Ci/g d}$ is $\mu\text{Ci/g}$ dried slurry.

* = At least one analytical result was above the IDL but below the EGL.

Table 4.1. Tank 241-C-106 Vendor Product
Summary Statistics - Random Variability. (page 6 of 9)

Analyte a	Method	Digest	Units \$	$\bar{x}$	RSD($\bar{x}$) (%)	df	95% UL
Pr	ICP/MS	Acid	$\mu\text{g/g d}$	7.05E+01	1.33	2	7.32E+01
Pr	ICP/MS	FS.Ni	$\mu\text{g/g d}$	7.71E+01	5.62	2	8.97E+01
Pr	ICP/MS	FS.Zr	$\mu\text{g/g d}$	8.41E+01	3.73	2	9.32E+01
Pt	ICP/MS	Acid	$\mu\text{g/g d}$	2.33E-01	18.07	2	3.56E-01
Pt	ICP/MS	FS.Ni	$\mu\text{g/g d}$	3.07E+01	16.03	2	4.51E+01
Pt	ICP/MS	FS.Zr	$\mu\text{g/g d}$	5.19E+00	13.45	2	7.23E+00
²³⁸ Pu	Rad	FS.Ni	$\mu\text{Ci/g d}$	6.31E-01	2.43	2	6.76E-01
²³⁹ Pu	ICP/MS	Acid	$\mu\text{g/g d}$	7.34E+00	3.21	2	8.02E+00
²³⁹ Pu	ICP/MS	Acid	$\mu\text{Ci/g d}$	4.55E-01	3.21	2	4.97E-01
²³⁹ Pu	ICP/MS	FS.Zr	$\mu\text{g/g d}$	2.46E+01	3.43	2	2.70E+01
²³⁹ Pu	ICP/MS	FS.Zr	$\mu\text{Ci/g d}$	1.52E+00	3.38	2	1.67E+00
^{239/240} Pu	Rad	FS.Ni	$\mu\text{Ci/g d}$	2.99E+00	2.60	2	3.21E+00
²⁴⁰ Pu	ICP/MS	Acid	$\mu\text{g/g d}$	5.58E-01	4.18	2	6.26E-01
²⁴⁰ Pu	ICP/MS	Acid	$\mu\text{Ci/g d}$	1.25E-01	4.62	2	1.42E-01
²⁴¹ Pu	Rad	FS.Ni	$\mu\text{Ci/g d}$	6.96E+00	2.43	2	7.45E+00
²⁴² Pu.w	ICP/MS	Acid	$\mu\text{Ci/g d}$	5.33E-05	25.00	2	9.23E-05
Rb	ICP/MS	Acid	$\mu\text{g/g d}$	1.11E+00	4.84	2	1.26E+00
Rb	ICP/MS	FS.Zr	$\mu\text{g/g d}$	7.68E+00	18.38	2	1.18E+01
Re	ICP/MS	Acid	$\mu\text{g/g d}$	7.83E-02	17.26	2	1.18E-01
Re	ICP/MS	FS.Ni	$\mu\text{g/g d}$	6.61E+00	15.76	2	9.66E+00
Rh	ICP/MS	Acid	$\mu\text{g/g d}$	1.05E+02	1.93	2	1.11E+02
Rh	ICP/MS	FS.Zr	$\mu\text{g/g d}$	1.44E+02	5.44	2	1.67E+02
Rh.w	ICP/MS	FS.Ni	$\mu\text{g/g d}$	1.90E+01	81.67	2	6.43E+01
Rh.wo	ICP/MS	FS.Ni	$\mu\text{g/g d}$	3.70E+01	64.11	1	1.87E+02
Ru	ICP/MS	FS.Ni	$\mu\text{g/g d}$	9.57E+02	3.88	2	1.07E+02
SO ₄ ²⁻	IC	Water	$\mu\text{g/g d}$	4.70E+02	8.85	2	5.92E+02
Sb.w	ICP/MS	Acid	$\mu\text{g/g d}$	3.77E-01	47.84	2	9.03E-01
Sb.wo	ICP/MS	Acid	$\mu\text{g/g d}$	7.88E-01	25.35	1	2.05E+00
Si	ICP/AES	Acid	$\mu\text{g/g d}$	2.80E+03	6.19	2	3.31E+03
Si	ICP/AES	FS.Ni	$\mu\text{g/g d}$	6.89E+04	3.91	2	7.67E+04
Si	ICP/AES	FS.Zr	$\mu\text{g/g d}$	6.46E+04	3.69	2	7.16E+04
¹⁵¹ Sm/ ¹⁴⁷ Pm	Rad	FS.Ni	$\mu\text{Ci/g d}$	9.23E+01	2.84	2	9.99E+01

a = Table does not include analytes where more than 50% of the data are "less than" values.
\$ = $\mu\text{g/g d}$ is $\mu\text{g/g}$ dried slurry; $\mu\text{Ci/g d}$ is $\mu\text{Ci/g}$ dried slurry.

* = At least one analytical result was above the IDL but below the EQL.

Table 4.1. Tank 241-C-106 Vendor Product
Summary Statistics - Random Variability. (page 7 of 9)

Analyte a	Method	Digest	Units \$	$\bar{x}$		RSD($\bar{x}$) (%)	df	95% UL
Sn	ICP/MS	Acid	$\mu\text{g/g d}$	3.48E+01		2.08	2	3.69E+01
Sn	ICP/MS	FS.Ni	$\mu\text{g/g d}$	2.93E+01		42.26	2	6.54E+01
Sn	ICP/MS	FS.Zr	$\mu\text{g/g d}$	1.02E+02		15.64	2	1.49E+02
$^{121\text{m}}\text{Sn}$	Rad	FS.Ni	$\mu\text{Ci/g d}$	1.23E+00		24.84	2	2.12E+00
$^{126}\text{Sn}/^{126\text{m}}\text{Sb}/^{126}\text{Sb}$	Rad	FS.Ni	$\mu\text{Ci/g d}$	1.15E-02		15.50	2	1.68E-02
Sr	ICP/AES	Acid	$\mu\text{g/g d}$	5.38E+01		0.99	2	5.54E+01
Sr	ICP/AES	FS.Ni	$\mu\text{g/g d}$	7.91E+01	*	4.23	2	8.89E+01
Sr	ICP/AES	FS.Zr	$\mu\text{g/g d}$	1.45E+02	*	3.49	2	1.60E+02
Sr	ICP/MS	Acid	$\mu\text{g/g d}$	4.51E+01		0.81	2	4.61E+01
Sr	ICP/MS	FS.Ni	$\mu\text{g/g d}$	5.88E+01		15.86	2	8.61E+01
Sr	ICP/MS	FS.Zr	$\mu\text{g/g d}$	1.50E+02		3.55	2	1.66E+02
$^{90}\text{Sr}/\text{Y}$	Rad	FS.Ni	$\mu\text{Ci/g d}$	7.30E+02		2.45	2	7.83E+02
TIC	WtChem	Direct	$\mu\text{gC/g d}$	5.30E+03		4.36	2	5.97E+03
TOC	WtChem	Direct	$\mu\text{gC/g d}$	4.79E+04		3.30	2	5.26E+04
Ta	ICP/MS	Acid	$\mu\text{g/g d}$	1.04E+01		11.64	2	1.40E+01
Ta	ICP/MS	FS.Ni	$\mu\text{g/g d}$	2.13E+01		26.27	2	3.77E+01
Ta	ICP/MS	FS.Zr	$\mu\text{g/g d}$	2.86E+01		22.67	2	4.75E+01
Tb	ICP/MS	Acid	$\mu\text{g/g d}$	1.46E+00		2.52	2	1.57E+00
Tb	ICP/MS	FS.Ni	$\mu\text{g/g d}$	8.20E+00		10.53	2	1.07E+01
Tb	ICP/MS	FS.Zr	$\mu\text{g/g d}$	2.34E+00		18.00	2	3.57E+00
^{99}Tc	ICP/MS	Acid	$\mu\text{g/g d}$	3.30E-01		10.83	2	4.34E-01
^{99}Tc	ICP/MS	Acid	$\mu\text{Ci/g d}$	5.60E-03		10.83	2	7.37E-03
Te	ICP/AES	Acid	$\mu\text{g/g d}$	5.23E+02		1.03	2	5.39E+02
Te	ICP/MS	Acid	$\mu\text{g/g d}$	2.03E+02		1.57	2	2.13E+02
Te	ICP/MS	FS.Ni	$\mu\text{g/g d}$	4.24E+02		8.96	2	5.35E+02
Te	ICP/MS	FS.Zr	$\mu\text{g/g d}$	2.88E+02		16.97	2	4.31E+02
Th	ICP/AES	Acid	$\mu\text{g/g d}$	3.49E+02	*	0.55	2	3.54E+02
^{232}Th	ICP/MS	Acid	$\mu\text{g/g d}$	1.85E+02		1.23	2	1.91E+02
^{232}Th	ICP/MS	Acid	$\mu\text{Ci/g d}$	2.03E-05		1.20	2	2.10E-05
^{232}Th	ICP/MS	FS.Zr	$\mu\text{g/g d}$	4.99E+02		20.45	2	7.96E+02
^{232}Th	ICP/MS	FS.Zr	$\mu\text{Ci/g d}$	5.48E-05		20.45	2	8.76E-05

a = Table does not include analytes where more than 50% of the data are "less than" values.

\$ = $\mu\text{g/g d}$ is $\mu\text{g/g}$ dried slurry; $\mu\text{Ci/g d}$ is $\mu\text{Ci/g}$ dried slurry.

* = At least one analytical result was above the IDL but below the EQL.

Table 4.1. Tank 241-C-106 Vendor Product
Summary Statistics - Random Variability. (page 8 of 9)

Analyte a	Method	Digest	Units b	$\bar{x}$	RSD ($\bar{x}$) (%)	df	95% UL
Ti	ICP/AES	Acid	$\mu\text{g/g d}$	3.50E+02	1.28	2	3.63E+02
Ti	ICP/AES	FS.Ni	$\mu\text{g/g d}$	1.03E+03	4.74	2	1.17E+03
Ti	ICP/AES	FS.Zr	$\mu\text{g/g d}$	1.02E+03	6.13	2	1.21E+03
Ti	ICP/MS	Acid	$\mu\text{g/g d}$	3.58E+02	1.33	2	3.72E+02
Ti	ICP/MS	FS.Ni	$\mu\text{g/g d}$	1.12E+03	6.86	2	1.35E+03
Ti	ICP/MS	FS.Zr	$\mu\text{g/g d}$	1.31E+03	7.06	2	1.58E+03
Tm	ICP/MS	Acid	$\mu\text{g/g d}$	6.17E-02	12.85	2	8.48E-02
Tm.w	ICP/MS	FS.Ni	$\mu\text{g/g d}$	1.82E+00	39.63	2	3.92E+00
Tm.wo	ICP/MS	FS.Ni	$\mu\text{g/g d}$	2.52E+00	36.01	2	5.17E+00
Total Alpha	Rad	FS.Ni	$\mu\text{Ci/g d}$	6.78E+00	1.66	2	7.11E+00
Total Beta	Rad	FS.Ni	$\mu\text{Ci/g d}$	2.05E+03	2.93	2	2.22E+03
Total Carbon	WtChem	Water	$\mu\text{gC/g d}$	5.22E+04	4.01	2	5.83E+04
Total U	ICP/MS	Acid	$\mu\text{g/g d}$	1.91E+03	1.90	2	2.01E+03
Total U	ICP/MS	FS.Ni	$\mu\text{g/g d}$	1.90E+03	2.84	2	2.05E+03
Total U	ICP/MS	FS.Zr	$\mu\text{g/g d}$	2.01E+03	1.34	2	2.09E+03
U	ICP/AES	Acid	$\mu\text{g/g d}$	1.95E+03	0.78	2	1.99E+03
²³³ U	ICP/MS	Acid	$\mu\text{g/g d}$	1.73E-01	11.34	2	2.31E-01
²³³ U	ICP/MS	Acid	$\mu\text{Ci/g d}$	1.68E-03	11.56	2	2.25E-03
²³⁴ U	ICP/MS	Acid	$\mu\text{g/g d}$	1.23E-01	8.02	2	1.52E-01
²³⁴ U	ICP/MS	Acid	$\mu\text{Ci/g d}$	7.73E-04	7.41	2	9.40E-04
²³⁵ U	ICP/MS	Acid	$\mu\text{g/g d}$	1.30E+01	2.86	2	1.41E+01
²³⁵ U	ICP/MS	Acid	$\mu\text{Ci/g d}$	2.80E-05	2.86	2	3.04E-05
²³⁶ U	ICP/MS	Acid	$\mu\text{g/g d}$	2.87E-01	7.28	2	3.48E-01
²³⁶ U	ICP/MS	Acid	$\mu\text{Ci/g d}$	1.85E-05	7.11	2	2.23E-05
²³⁸ U	ICP/MS	Acid	$\mu\text{g/g d}$	1.90E+03	1.91	2	2.00E+03
²³⁸ U	ICP/MS	Acid	$\mu\text{Ci/g d}$	6.37E-04	1.91	2	6.72E-04
²³⁸ U	ICP/MS	FS.Ni	$\mu\text{g/g d}$	1.89E+03	2.86	2	2.04E+03
²³⁸ U	ICP/MS	FS.Ni	$\mu\text{Ci/g d}$	6.34E-04	2.86	2	6.87E-04
²³⁸ U	ICP/MS	FS.Zr	$\mu\text{g/g d}$	2.00E+03	1.28	2	2.07E+03
²³⁸ U	ICP/MS	FS.Zr	$\mu\text{Ci/g d}$	6.71E-04	1.28	2	6.97E-04
V	ICP/MS	Acid	$\mu\text{g/g d}$	3.73E+01	8.58	2	4.66E+01

a = Table does not include analytes where more than 50% of the data are "less than" values.

b = $\mu\text{g/g d}$ is $\mu\text{g/g dried slurry}$; $\mu\text{Ci/g d}$ is $\mu\text{Ci/g dried slurry}$.

* = At least one analytical result was above the IDL but below the EQL.

Table 4.1. Tank 241-C-106 Vendor Product
Summary Statistics - Random Variability. (page 9 of 9)

Analyte a	Method	Digest	Units \$	$\bar{\mu}$		RSD($\bar{\mu}$) (%)	df	95% UL
W	ICP/MS	Acid	$\mu\text{g/g d}$	2.22E+02		4.08	2	2.48E+02
W	ICP/MS	FS.Ni	$\mu\text{g/g d}$	4.99E+02		6.05	2	5.87E+02
W	ICP/MS	FS.Zr	$\mu\text{g/g d}$	3.57E+02		19.62	2	5.61E+02
Yb	ICP/MS	Acid	$\mu\text{g/g d}$	2.50E+01		23.09	2	4.19E+01
Yb	ICP/MS	FS.Ni	$\mu\text{g/g d}$	1.26E+01		29.08	2	2.33E+01
Yb	ICP/MS	FS.Zr	$\mu\text{g/g d}$	3.24E+00		18.62	2	4.99E+00
Zn	ICP/AES	Acid	$\mu\text{g/g d}$	1.16E+02	*	1.28	2	1.20E+02
Zn	ICP/AES	FS.Ni	$\mu\text{g/g d}$	1.83E+02	*	5.47	2	2.12E+02
Zn	ICP/AES	FS.Zr	$\mu\text{g/g d}$	1.61E+02	*	3.48	2	1.77E+02
Zn	ICP/MS	Acid	$\mu\text{g/g d}$	9.92E+01		3.13	2	1.08E+02
Zn	ICP/MS	FS.Ni	$\mu\text{g/g d}$	3.07E+02		9.43	2	3.91E+02
Zn	ICP/MS	FS.Zr	$\mu\text{g/g d}$	2.34E+02		11.13	2	3.10E+02
Zr	ICP/AES	Acid	$\mu\text{g/g d}$	6.61E+02		18.92	2	1.03E+03
Zr	ICP/AES	FS.Ni	$\mu\text{g/g d}$	1.11E+03		19.48	2	1.73E+03
Zr	ICP/MS	Acid	$\mu\text{g/g d}$	2.56E+02		16.67	2	3.80E+02
Zr	ICP/MS	FS.Ni	$\mu\text{g/g d}$	4.33E+02		21.68	2	7.07E+02
m/z241	ICP/MS	Acid	$\mu\text{g/g d}$	7.73E-01		4.03	2	8.64E-01
m/z241	ICP/MS	FS.Zr	$\mu\text{g/g d}$	1.65E+00		8.55	2	2.06E+00
m/z93	ICP/MS	Acid	$\mu\text{g/g d}$	5.00E+01		15.88	2	7.31E+01
m/z93	ICP/MS	FS.Ni	$\mu\text{g/g d}$	3.38E+01		31.48	2	6.48E+01
m/z93	ICP/MS	FS.Zr	$\mu\text{g/g d}$	2.35E+02		21.81	2	3.84E+02

a = Table does not include analytes where more than 50% of the data are "less than" values.

\$ = $\mu\text{g/g d}$ is $\mu\text{g/g dried slurry}$; $\mu\text{Ci/g d}$ is $\mu\text{Ci/g dried slurry}$.

* = At least one analytical result was above the IDL but below the EDL.

Table 4.2. Tank 241-C-106 Vendor Product
Analytes with More Than 50% "Less Than" Results. (page 1 of 3)

Analyte	Method	Digest	Units \$	Largest Result
^{110m} Ag	Rad	FS.Ni	µCi/g d	< 0.09
²⁴³ Am	Rad	FS.Ni	µCi/g d	< 0.02
As	ICP/AES	Acid	µg/g d	< 123
As	ICP/AES	FS.Ni	µg/g d	< 479
As	ICP/AES	FS.Zr	µg/g d	< 510
B	ICP/AES	FS.Zr	µg/g d	186
Be	ICP/AES	Acid	µg/g d	< 1
Be	ICP/AES	FS.Ni	µg/g d	< 9
Be	ICP/AES	FS.Zr	µg/g d	< 9
Br ⁻	IC	Water	µg/g d	< 116
Ce	ICP/AES	FS.Ni	µg/g d	< 523
Ce	ICP/AES	FS.Zr	µg/g d	< 1580
¹⁴⁴ Ce/ ¹⁴⁴ Pr/ ¹⁴⁴ Pr	Rad	FS.Ni	µCi/g d	< 2
Co	ICP/AES	FS.Ni	µg/g d	< 87
Co	ICP/AES	FS.Zr	µg/g d	< 93
¹³⁴ Cs	Rad	FS.Ni	µCi/g d	< 0.3
Dy	ICP/AES	Acid	µg/g d	< 12
Dy	ICP/AES	FS.Ni	µg/g d	< 87
Dy	ICP/AES	FS.Zr	µg/g d	< 93
Er	ICP/MS	FS.Zr	µg/g d	< 1
Eu	ICP/AES	Acid	µg/g d	< 25
Eu	ICP/AES	FS.Ni	µg/g d	< 174
Eu	ICP/AES	FS.Zr	µg/g d	< 185
¹⁵² Eu	Rad	FS.Ni	µCi/g d	< 2
F ⁻	IC	Water	µg/g d	< 2910
Ho	ICP/MS	FS.Zr	µg/g d	1.11
¹²⁹ I	ICP/MS	FS.Zr	µg/g d	< 20
¹²⁹ I	ICP/MS	FS.Zr	µCi/g d	< 0.004
K	ICP/AES	Acid	µg/g d	< 492
Li	ICP/AES	FS.Ni	µg/g d	94
Mo	ICP/AES	FS.Ni	µg/g d	< 87
Mo	ICP/AES	FS.Zr	µg/g d	< 93
Pd	ICP/AES	Acid	µg/g d	< 369
Pd	ICP/AES	FS.Ni	µg/g d	< 1310
Pd	ICP/AES	FS.Zr	µg/g d	< 1390

\$ = µg/g d is µg/g dried slurry; µCi/g d is µCi/g dried slurry.

Table 4.2. Tank 241-C-106 Vendor Product
Analytes with More Than 50% "Less Than" Results. (page 2 of 3)

Analyte	Method	Digest	Units \$	Largest Result
^{240}Pu	ICP/MS	FS.Zr	$\mu\text{g/g d}$	< 1
^{240}Pu	ICP/MS	FS.Zr	$\mu\text{Ci/g d}$	< 0.2
^{242}Pu	ICP/MS	Acid	$\mu\text{g/g d}$	< 0.02
^{242}Pu	ICP/MS	Acid	$\mu\text{Ci/g d}$	< 0.00008
^{242}Pu	ICP/MS	FS.Zr	$\mu\text{g/g d}$	< 1
^{242}Pu	ICP/MS	FS.Zr	$\mu\text{Ci/g d}$	< 0.004
^{242}Pu	Rad	FS.Ni	$\mu\text{Ci/g d}$	< 0.02
Re	ICP/MS	FS.Zr	$\mu\text{g/g d}$	< 1
Rh	ICP/AES	Acid	$\mu\text{g/g d}$	< 74
Rh	ICP/AES	FS.Ni	$\mu\text{g/g d}$	< 523
Rh	ICP/AES	FS.Zr	$\mu\text{g/g d}$	< 556
Ru	ICP/AES	Acid	$\mu\text{g/g d}$	< 738
Ru	ICP/AES	FS.Ni	$\mu\text{g/g d}$	< 1740
Ru	ICP/AES	FS.Zr	$\mu\text{g/g d}$	< 1850
$^{106}\text{Ru/Rh}$	Rad	FS.Ni	$\mu\text{Ci/g d}$	< 3
Sb	ICP/AES	Acid	$\mu\text{g/g d}$	< 74
Sb	ICP/AES	FS.Ni	$\mu\text{g/g d}$	< 871
Sb	ICP/AES	FS.Zr	$\mu\text{g/g d}$	< 927
Sb	ICP/MS	FS.Ni	$\mu\text{g/g d}$	< 1
Sb	ICP/MS	FS.Zr	$\mu\text{g/g d}$	< 1
$^{125}\text{Sb}/^{125\text{m}}\text{Te}$	Rad	FS.Ni	$\mu\text{Ci/g d}$	< 2
Se	ICP/AES	Acid	$\mu\text{g/g d}$	< 369
Se	ICP/AES	FS.Ni	$\mu\text{g/g d}$	< 610
Se	ICP/AES	FS.Zr	$\mu\text{g/g d}$	< 649
^{79}Se	Rad	FS.Ni	$\mu\text{Ci/g d}$	< 0.0024
^{79}Se	Rad	Acid	$\mu\text{Ci/g d}$	< 0.00021
Sn	ICP/AES	Acid	$\mu\text{g/g d}$	< 4060
Sn	ICP/AES	FS.Ni	$\mu\text{g/g d}$	< 3920
Sn	ICP/AES	FS.Zr	$\mu\text{g/g d}$	< 4170
$^{113}\text{Sn}/^{113\text{m}}\text{In}$	Rad	FS.Ni	$\mu\text{Ci/g d}$	< 0.01
$^{119\text{m}}\text{Sn}$	Rad	FS.Ni	$\mu\text{Ci/g d}$	< 0.04
^{99}Tc	ICP/MS	FS.Ni	$\mu\text{g/g d}$	< 1
^{99}Tc	ICP/MS	FS.Ni	$\mu\text{Ci/g d}$	< 0.017
Te	ICP/AES	FS.Ni	$\mu\text{g/g d}$	< 2610
Te	ICP/AES	FS.Zr	$\mu\text{g/g d}$	< 2780

\$ = $\mu\text{g/g d}$ is $\mu\text{g/g}$ dried slurry; $\mu\text{Ci/g d}$ is $\mu\text{Ci/g}$ dried slurry.

Table 4.2. Tank 241-C-106 Vendor Product
 Analytes with More Than 50% "Less Than" Results. (page 3 of 3)

Analyte	Method	Digest	Units \$	Largest Result
Th	ICP/AES	FS.Ni	$\mu\text{g/g d}$	< 1740
Th	ICP/AES	FS.Zr	$\mu\text{g/g d}$	< 1850
Tl	ICP/AES	Acid	$\mu\text{g/g d}$	< 123
Tl	ICP/AES	FS.Ni	$\mu\text{g/g d}$	< 871
Tl	ICP/AES	FS.Zr	$\mu\text{g/g d}$	< 927
Tm	ICP/MS	FS.Zr	$\mu\text{g/g d}$	1.24
U	ICP/AES	FS.Ni	$\mu\text{g/g d}$	< 4360
U	ICP/AES	FS.Zr	$\mu\text{g/g d}$	< 6950
V	ICP/AES	Acid	$\mu\text{g/g d}$	< 37
V	ICP/AES	FS.Ni	$\mu\text{g/g d}$	< 87
V	ICP/AES	FS.Zr	$\mu\text{g/g d}$	< 93
W	ICP/AES	Acid	$\mu\text{g/g d}$	< 492
W	ICP/AES	FS.Ni	$\mu\text{g/g d}$	< 3480
W	ICP/AES	FS.Zr	$\mu\text{g/g d}$	< 3710
Y	ICP/AES	Acid	$\mu\text{g/g d}$	< 86
⁹³ Zr	Rad	FS.Ni	$\mu\text{Ci/g d}$	< 0.6

\$ = $\mu\text{g/g d}$ is $\mu\text{g/g}$ dried slurry; $\mu\text{Ci/g d}$ is $\mu\text{Ci/g}$ dried slurry.

Table 4.3. Tank 241-C-106 Vendor Product - Variance Components. (page 1 of 9)

Analyte @	Method	Digest	Mean	Units \$	Random Sampling RSD %	Random Analytical RSD %
Ag	ICP/AES	Acid	7.56E+01	µg/g d	0.00 *	6.58
Ag	ICP/AES	FS.Ni	1.03E+03	µg/g d	0.00 *	22.89
Ag	ICP/AES	FS.Zr	3.18E+03	µg/g d	0.00 *	21.45
Al	ICP/AES	Acid	4.79E+04	µg/g d	2.01	1.90
Al	ICP/AES	FS.Ni	5.12E+04	µg/g d	4.20	2.50
Al	ICP/AES	FS.Zr	4.95E+04	µg/g d	4.70	2.35
Al	ICP/MS	Acid	3.34E+04	µg/g d	0.00 *	7.25
²⁴¹ Am.aea	Rad	FS.Ni	2.77E+00	µCi/g d	4.18	2.01
²⁴¹ Am.gea	Rad	FS.Ni	3.15E+00	µCi/g d	0.00 *	23.03
As	ICP/MS	Acid	1.55E+02	µg/g d	5.43	6.24
As	ICP/MS	FS.Ni	2.86E+02	µg/g d	8.90	12.98
As	ICP/MS	FS.Zr	2.56E+02	µg/g d	22.98	6.76
B	ICP/AES	Acid	2.25E+02	µg/g d	30.09	6.60
B	ICP/AES	FS.Ni	3.03E+02	µg/g d	28.49	20.04
B	ICP/MS	Acid	1.86E+02	µg/g d	36.66	5.49
B	ICP/MS	FS.Ni	2.29E+02	µg/g d	34.78	18.18
B	ICP/MS	FS.Zr	1.01E+02	µg/g d	64.91	22.50
Ba	ICP/AES	Acid	4.15E+02	µg/g d	1.28	2.22
Ba	ICP/AES	FS.Ni	4.07E+02	µg/g d	4.69	2.17
Ba	ICP/AES	FS.Zr	4.37E+02	µg/g d	5.08	2.28
Ba	ICP/MS	Acid	3.62E+02	µg/g d	0.00 *	2.72
Ba	ICP/MS	FS.Ni	4.12E+02	µg/g d	0.00 *	4.75
Ba	ICP/MS	FS.Zr	4.27E+02	µg/g d	0.00 *	3.15
Bi	ICP/AES	Acid	4.30E+02	µg/g d	1.08	2.45
Bi	ICP/AES	FS.Ni	5.38E+02	µg/g d	2.19	1.60
Bi	ICP/AES	FS.Zr	5.82E+02	µg/g d	0.00 *	9.94
C ₂ O ₄	IC	Water	1.70E+05	µg/g d	6.30	5.33
¹⁴ C	Rad	FS.Ni	2.87E-03	µCi/g d	23.15	189.54
CN ⁻	WtChem	Direct	9.50E+00	µg/g d	9.43	3.08
Ca	ICP/AES	Acid	3.84E+03	µg/g d	1.62	1.93
Ca	ICP/AES	FS.Ni	6.69E+03	µg/g d	0.00 *	10.70
Ca	ICP/AES	FS.Zr	9.85E+03	µg/g d	0.00 *	10.42

@ = Table does not include analytes where more than 50% of the data are "less than" values.

\$ = µg/g d is µg/g dried slurry; µCi/g d is µCi/g dried slurry.

* = A RSD(S) of 0.00% indicates that the variability between duplicate analytical measurements was greater than the variability between subsamples.

Table 4.3. Tank 241-C-106 Vendor Product - Variance Components. (page 2 of 9)

Analyte a	Method	Digest	Mean	Units \$	Random Sampling RSD %	Random Analytical RSD %
Ca	ICP/MS	Acid	9.57E+03	µg/g d	0.00 *	11.74
Ca	ICP/MS	FS.Ni	1.72E+03	µg/g d	13.03	4.89
Ca	ICP/MS	FS.Zr	3.77E+03	µg/g d	7.73	5.63
Cd	ICP/AES	Acid	7.30E+01	µg/g d	0.00 *	18.00
Cd	ICP/AES	FS.Ni	8.10E+01	µg/g d	2.79	1.36
Cd	ICP/AES	FS.Zr	8.08E+01	µg/g d	1.23	5.40
Cd	ICP/MS	Acid	5.81E+01	µg/g d	0.00 *	2.99
Cd	ICP/MS	FS.Ni	9.05E+01	µg/g d	0.00 *	12.56
Cd	ICP/MS	FS.Zr	7.41E+01	µg/g d	10.27	7.09
Ce	ICP/AES	Acid	2.99E+02	µg/g d	1.83	3.43
Ce	ICP/MS	Acid	1.59E+02	µg/g d	1.75	3.30
Ce	ICP/MS	FS.Ni	1.60E+02	µg/g d	12.01	13.90
Ce	ICP/MS	FS.Zr	2.02E+02	µg/g d	10.36	6.37
Cl ⁻	IC	Water	1.26E+02	µg/g d	0.00 *	20.80
²⁴² Cm/ ²⁴² Am/ ^{242m} Am	Rad	FS.Ni	8.07E-03	µCi/g d	12.55	17.36
^{243/244} Cm	Rad	FS.Ni	9.86E-02	µCi/g d	20.80	11.99
Co	ICP/AES	Acid	2.81E+01	µg/g d	0.00 *	2.60
Co	ICP/MS	Acid	1.57E+01	µg/g d	0.00 *	3.01
Co	ICP/MS	FS.Ni	2.62E+01	µg/g d	16.55	18.39
Co	ICP/MS	FS.Zr	2.18E+01	µg/g d	9.38	6.03
⁶⁰ Co	Rad	FS.Ni	4.91E-01	µCi/g d	0.00 *	7.61
Cr	ICP/AES	Acid	5.33E+02	µg/g d	0.51	2.29
Cr	ICP/AES	FS.Ni	8.02E+02	µg/g d	4.23	2.85
Cr	ICP/AES	FS.Zr	7.00E+02	µg/g d	6.68	2.76
Cr	ICP/MS	Acid	4.26E+02	µg/g d	0.00 *	2.58
Cr	ICP/MS	FS.Ni	7.74E+02	µg/g d	9.28	6.90
Cr	ICP/MS	FS.Zr	7.39E+02	µg/g d	6.63	2.21
¹³³ Cs	ICP/MS	Acid	1.04E+01	µg/g d	6.30	3.03
¹³³ Cs	ICP/MS	FS.Ni	1.80E+01	µg/g d	0.00 *	22.20
¹³³ Cs	ICP/MS	FS.Zr	7.18E+00	µg/g d	2.11	30.20
¹³³ Cs	Rad	FS.Ni	1.57E+01	µCi/g d	6.47	7.04
¹³⁵ Cs	Rad	FS.Ni	2.86E-03	µCi/g d	5.26	2.05
¹³⁷ Cs/Ba	Rad	FS.Ni	4.96E+02	µCi/g d	5.56	1.79

a = Table does not include analytes where more than 50% of the data are "less than" values.

\$ = µg/g d is µg/g dried slurry; µCi/g d is µCi/g dried slurry.

* = A RSD(s) of 0.00% indicates that the variability between duplicate analytical measurements was greater than the variability between subsamples.

Table 4.3. Tank 241-C-106 Vendor Product - Variance Components. (page 3 of 9)

Analyte @	Method	Digest	Mean	Units \$	Random Sampling RSD %	Random Analytical RSD %
Cu	ICP/AES	Acid	7.75E+01	µg/g d	0.00 *	2.43
Cu	ICP/AES	FS.Ni	1.09E+02	µg/g d	0.00 *	4.35
Cu	ICP/AES	FS.Zr	1.09E+02	µg/g d	9.12	10.66
Cu	ICP/MS	Acid	1.10E+02	µg/g d	0.00 *	1.83
Cu	ICP/MS	FS.Zr	1.56E+02	µg/g d	5.83	6.24
Cu.W	ICP/MS	FS.Ni	1.13E+01	µg/g d	120.62	34.61
Cu.Wo	ICP/MS	FS.Ni	1.89E+01	µg/g d	62.09	1.50
Dy	ICP/MS	Acid	1.47E+00	µg/g d	0.00 *	6.28
Dy	ICP/MS	FS.Ni	2.02E+01	µg/g d	18.61	33.42
Dy	ICP/MS	FS.Zr	3.60E+00	µg/g d	56.09	9.64
Er	ICP/MS	Acid	7.67E-01	µg/g d	0.00 *	6.74
Er	ICP/MS	FS.Ni	1.31E+01	µg/g d	0.00 *	35.87
Eu	ICP/MS	Acid	6.46E+00	µg/g d	0.00 *	3.65
Eu	ICP/MS	FS.Ni	7.87E+00	µg/g d	0.00 *	75.25
Eu	ICP/MS	FS.Zr	7.84E+00	µg/g d	0.00 *	16.02
¹⁵⁴ Eu	Rad	FS.Ni	3.79E+00	µCi/g d	2.30	2.83
¹⁵⁵ Eu	Rad	FS.Ni	3.13E+00	µCi/g d	0.00 *	9.85
Fe	ICP/AES	Acid	1.37E+05	µg/g d	1.05	2.13
Fe	ICP/AES	FS.Ni	1.41E+05	µg/g d	5.49	2.17
Fe	ICP/AES	FS.Zr	1.31E+05	µg/g d	6.06	2.60
Fe	ICP/MS	Acid	1.10E+05	µg/g d	0.00 *	7.95
⁵⁵ Fe	Rad	FS.Ni	1.53E-01	µCi/g d	3.50	4.08
Gd	ICP/MS	Acid	1.36E+01	µg/g d	3.36	7.12
Gd	ICP/MS	FS.Ni	2.50E+01	µg/g d	0.00 *	27.66
Gd	ICP/MS	FS.Zr	1.21E+01	µg/g d	29.81	7.52
Hg	CVAA	Direct	3.43E+02	µg/g d	0.00 *	4.76
Ho	ICP/MS	Acid	1.42E-01	µg/g d	0.00 *	26.94
Ho	ICP/MS	FS.Ni	6.07E+00	µg/g d	23.64	13.89
¹²⁹ I.W	ICP/MS	Acid	1.18E+00	µg/g d	16.87	13.37
¹²⁹ I.W	ICP/MS	Acid	2.18E-04	µCi/g d	9.72	11.37
¹²⁹ I.Wo	ICP/MS	Acid	1.25E+00	µg/g d	10.75	17.33
¹²⁹ I.Wo	ICP/MS	Acid	2.37E-04	µCi/g d	0.00 *	16.00
K	ICP/AES	FS.Zr	7.59E+03	µg/g d	0.00 *	21.84

@ = Table does not include analytes where more than 50% of the data are "less than" values.

\$ = µg/g d is µg/g dried slurry; µCi/g d is µCi/g dried slurry.

* = A RSD(s) of 0.00% indicates that the variability between duplicate analytical measurements was greater than the variability between subsamples.

Table 4.3. Tank 241-C-106 Vendor Product - Variance Components. (page 4 of 9)

Analyte @	Method	Digest	Mean	Units \$	Random Sampling RSD %	Random Analytical RSD %
La	ICP/AES	Acid	9.61E+01	µg/g d	0.00 *	3.36
La	ICP/AES	FS.Ni	1.19E+02	µg/g d	4.09	6.17
La	ICP/AES	FS.Zr	1.64E+02	µg/g d	0.00 *	15.87
La	ICP/MS	Acid	8.50E+01	µg/g d	1.04	3.84
La	ICP/MS	FS.Ni	8.06E+01	µg/g d	9.80	12.17
La	ICP/MS	FS.Zr	9.22E+01	µg/g d	15.30	7.95
Li	ICP/AES	Acid	4.59E+01	µg/g d	73.60	1.67
Li	ICP/AES	FS.Zr	9.39E+01	µg/g d	22.88	16.42
Mg	ICP/AES	Acid	7.54E+02	µg/g d	2.03	2.39
Mg	ICP/AES	FS.Ni	1.36E+03	µg/g d	10.55	2.74
Mg	ICP/AES	FS.Zr	1.31E+03	µg/g d	5.20	6.01
Mg	ICP/MS	Acid	4.45E+02	µg/g d	2.56	3.15
Mg	ICP/MS	FS.Ni	1.22E+03	µg/g d	12.11	6.81
Mg	ICP/MS	FS.Zr	1.26E+03	µg/g d	4.63	4.93
Mn	ICP/AES	Acid	2.60E+03	µg/g d	0.00 *	2.32
Mn	ICP/AES	FS.Ni	3.14E+03	µg/g d	3.26	3.39
Mn	ICP/AES	FS.Zr	2.59E+03	µg/g d	5.75	3.29
Mn	ICP/MS	Acid	2.04E+03	µg/g d	0.00 *	5.61
Mn	ICP/MS	FS.Ni	2.81E+03	µg/g d	6.02	4.97
Mn	ICP/MS	FS.Zr	2.56E+03	µg/g d	4.26	3.13
Mo	ICP/AES	Acid	2.67E+01	µg/g d	0.31	2.16
Mo	ICP/MS	Acid	9.60E+00	µg/g d	0.00 *	6.33
Mo	ICP/MS	FS.Ni	5.70E+01	µg/g d	22.25	13.53
Mo	ICP/MS	FS.Zr	2.79E+01	µg/g d	16.78	5.53
NH ₃ -N	WtChem	Water	1.75E+01	µg/g d	22.44	37.54
NO ₂ ⁻	IC	Water	2.07E+03	µg/g d	15.28	12.55
NO ₃ ⁻	IC	Water	8.86E+02	µg/g d	52.06	13.50
Na	ICP/AES	Acid	1.62E+05	µg/g d	4.78	2.44
Na	ICP/AES	FS.Ni	1.42E+05	µg/g d	6.45	2.63
Na	ICP/MS	Acid	1.82E+05	µg/g d	0.00 *	14.96
⁹³ Nb	Rad	FS.Ni	2.37E-01	µCi/g d	37.35	51.76

@ = Table does not include analytes where more than 50% of the data are "less than" values.

\$ = µg/g d is µg/g dried slurry; µCi/g d is µCi/g dried slurry.

* = A RSD(S) of 0.00% indicates that the variability between duplicate analytical measurements was greater than the variability between subsamples.

Table 4.3. Tank 241-C-106 Vendor Product - Variance Components. (page 5 of 9)

Analyte a	Method	Digest	Mean	Units \$	Random Sampling RSD %	Random Analytical RSD %
Nd	ICP/AES	Acid	2.82E+02	µg/g d	0.00 *	2.92
Nd	ICP/AES	FS.Ni	3.79E+02	µg/g d	7.40	4.99
Nd	ICP/AES	FS.Zr	5.17E+02	µg/g d	0.00 *	11.49
Nd	ICP/MS	Acid	1.93E+02	µg/g d	1.20	1.96
Nd	ICP/MS	FS.Ni	2.33E+02	µg/g d	11.67	10.56
Nd	ICP/MS	FS.Zr	2.24E+02	µg/g d	0.00 *	9.77
Ni	ICP/AES	Acid	1.13E+03	µg/g d	0.00 *	2.70
Ni	ICP/AES	FS.Zr	1.66E+03	µg/g d	7.51	3.22
Ni	ICP/MS	Acid	9.49E+02	µg/g d	0.00 *	5.94
Ni	ICP/MS	FS.Zr	1.61E+03	µg/g d	3.89	1.71
⁵⁹ Ni.w	Rad	FS.Zr	2.20E-02	µCi/g d	0.00 *	25.07
⁵⁹ Ni.wo	Rad	FS.Zr	1.97E-02	µCi/g d	18.21	18.37
⁶³ Ni	Rad	FS.Zr	2.40E+00	µCi/g d	0.00 *	5.85
²³⁷ Np	ICP/MS	Acid	4.48E+00	µg/g d	0.00 *	4.17
²³⁷ Np	ICP/MS	Acid	3.16E-03	µCi/g d	0.00 *	4.16
²³⁷ Np	ICP/MS	FS.Zr	5.57E+00	µg/g d	10.77	17.57
²³⁷ Np	ICP/MS	FS.Zr	3.92E-03	µCi/g d	10.75	17.58
P	ICP/AES	Acid	1.34E+03	µg/g d	4.76	2.53
P	ICP/AES	FS.Ni	3.33E+03	µg/g d	0.00 *	11.23
P	ICP/AES	FS.Zr	3.84E+03	µg/g d	17.41	8.18
PO ₄ ³⁻	IC	Water	1.60E+02	µg/g d	0.00 *	21.89
Pb	ICP/AES	Acid	3.43E+03	µg/g d	1.35	1.94
Pb	ICP/AES	FS.Ni	2.95E+03	µg/g d	10.93	14.05
Pb	ICP/AES	FS.Zr	3.53E+03	µg/g d	5.56	3.36
Pb	ICP/MS	Acid	2.85E+03	µg/g d	4.91	5.10
Pb	ICP/MS	FS.Zr	2.55E+03	µg/g d	2.53	2.96
Pd	ICP/MS	Acid	9.99E+00	µg/g d	0.00 *	6.59
Pd	ICP/MS	FS.Ni	2.35E+01	µg/g d	0.00 *	55.72
Pd	ICP/MS	FS.Zr	2.74E+01	µg/g d	0.00 *	18.69
¹⁰⁷ Pd.w	ICP/MS	Acid	1.35E+00	µg/g d	62.34	121.00
¹⁰⁷ Pd.w	ICP/MS	Acid	7.18E-04	µCi/g d	55.86	118.78
¹⁰⁷ Pd.wo	ICP/MS	Acid	2.20E+00	µg/g d	0.00 *	110.60
¹⁰⁷ Pd.wo	ICP/MS	Acid	1.14E-03	µCi/g d	0.00 *	111.87

a = Table does not include analytes where more than 50% of the data are "less than" values.

\$ = µg/g d is µg/g dried slurry; µCi/g d is µCi/g dried slurry.

* = A RSD(s) of 0.00% indicates that the variability between duplicate analytical measurements was greater than the variability between subsamples.

Table 4.3. Tank 241-C-106 Vendor Product - Variance Components. (page 6 of 9)

Analyte a	Method	Digest	Mean	Units \$	Random Sampling RSD %	Random Analytical RSD %
Pr	ICP/MS	Acid	7.05E+01	µg/g d	1.49	2.47
Pr	ICP/MS	FS.Ni	7.71E+01	µg/g d	0.00 *	13.77
Pr	ICP/MS	FS.Zr	8.41E+01	µg/g d	6.04	3.22
Pt	ICP/MS	Acid	2.33E-01	µg/g d	0.00 *	44.26
Pt	ICP/MS	FS.Ni	3.07E+01	µg/g d	27.60	4.32
Pt	ICP/MS	FS.Zr	5.19E+00	µg/g d	7.14	31.35
238 _{Pu}	Rad	FS.Ni	6.31E-01	µCi/g d	3.38	3.54
239 _{Pu}	ICP/MS	Acid	7.34E+00	µg/g d	5.52	0.82
239 _{Pu}	ICP/MS	Acid	4.55E-01	µCi/g d	5.53	0.85
239 _{Pu}	ICP/MS	FS.Zr	2.46E+01	µg/g d	4.29	5.82
239 _{Pu}	ICP/MS	FS.Zr	1.52E+00	µCi/g d	4.24	5.71
239/240 _{Pu}	Rad	FS.Ni	2.99E+00	µCi/g d	3.34	4.27
240 _{Pu}	ICP/MS	Acid	5.58E-01	µg/g d	6.05	5.62
240 _{Pu}	ICP/MS	Acid	1.25E-01	µCi/g d	6.93	5.66
241 _{Pu}	Rad	FS.Ni	6.96E+00	µCi/g d	0.00 *	5.95
Rb	ICP/MS	Acid	1.11E+00	µg/g d	6.44	7.58
Rb	ICP/MS	FS.Zr	7.68E+00	µg/g d	22.02	32.51
Re	ICP/MS	Acid	7.83E-02	µg/g d	0.00 *	42.28
Re	ICP/MS	FS.Ni	6.61E+00	µg/g d	20.88	24.86
Rh	ICP/MS	Acid	1.05E+02	µg/g d	2.87	2.44
Rh	ICP/MS	FS.Zr	1.44E+02	µg/g d	6.78	9.25
Rh.w	ICP/MS	FS.Ni	1.90E+01	µg/g d	93.76	149.79
Rh.wo	ICP/MS	FS.Ni	3.70E+01	µg/g d	0.00 *	111.03
Ru	ICP/MS	FS.Ni	9.57E+02	µg/g d	6.72 #	NA
SO ₄ ²⁻	IC	Water	4.70E+02	µg/g d	12.09	13.34
Sb.w	ICP/MS	Acid	3.77E-01	µg/g d	31.49	108.41
Sb.wo	ICP/MS	Acid	7.88E-01	µg/g d	32.37	17.96
Si	ICP/AES	Acid	2.80E+03	µg/g d	0.00 *	15.17
Si	ICP/AES	FS.Ni	6.89E+04	µg/g d	6.63	1.92
Si	ICP/AES	FS.Zr	6.46E+04	µg/g d	6.11	2.67
151 _{Gm} , 147 _{Pm}	Rad	FS.Ni	9.23E+01	µCi/g d	4.07	3.89

∅ = Table does not include analytes where more than 50% of the data are "less than" values.

\$ = µg/g d is µg/g dried slurry; µCi/g d is µCi/g dried slurry.

* = A RSD(S) of 0.00% indicates that the variability between duplicate analytical measurements was greater than the variability between subsamples.

= Duplicate measurements were not available, the sampling and analytical variability cannot be separated from each other.

Table 4.3. Tank 241-C-106 Vendor Product - Variance Components. (page 7 of 9)

Analyte @	Method	Digest	Mean	Units \$	Random Sampling RSD %	Random Analytical RSD %
Sn	ICP/MS	Acid	3.48E+01	µg/g d	2.67	3.43
Sn	ICP/MS	FS.Ni	2.93E+01	µg/g d	32.13	93.02
Sn	ICP/MS	FS.Zr	1.02E+02	µg/g d	23.42	19.26
^{121m} Sn	Rad	FS.Ni	1.23E+00	µCi/g d	39.45	24.26
¹²⁶ Sn/ ^{126m} Sb/ ¹²⁶ Sb	Rad	FS.Ni	1.15E-02	µCi/g d	23.91	17.29
Sr	ICP/AES	Acid	5.38E+01	µg/g d	0.00 *	2.43
Sr	ICP/AES	FS.Ni	7.91E+01	µg/g d	7.25	1.51
Sr	ICP/AES	FS.Zr	1.45E+02	µg/g d	0.00 *	8.55
Sr	ICP/MS	Acid	4.51E+01	µg/g d	0.00 *	1.98
Sr	ICP/MS	FS.Ni	5.88E+01	µg/g d	27.11	6.32
Sr	ICP/MS	FS.Zr	1.50E+02	µg/g d	0.00 *	8.69
⁹⁰ Sr/Y	Rad	FS.Ni	7.30E+02	µCi/g d	0.00 *	6.01
TIC	WtChem	Direct	5.30E+03	ugC/g d	0.00 *	10.69
TOC	WtChem	Direct	4.79E+04	ugC/g d	3.82	6.03
Ta	ICP/MS	Acid	1.04E+01	µg/g d	6.97	26.76
Ta	ICP/MS	FS.Ni	2.13E+01	µg/g d	40.47	29.40
Ta	ICP/MS	FS.Zr	2.86E+01	µg/g d	38.93	7.19
Tb	ICP/MS	Acid	1.46E+00	µg/g d	0.00 *	6.17
Tb	ICP/MS	FS.Ni	8.20E+00	µg/g d	15.95	12.51
Tb	ICP/MS	FS.Zr	2.34E+00	µg/g d	26.28	23.74
⁹⁹ Tc	ICP/MS	Acid	3.30E-01	µg/g d	18.26	6.06
⁹⁹ Tc	ICP/MS	Acid	5.60E-03	µCi/g d	18.24	6.04
Te	ICP/AES	Acid	5.23E+02	µg/g d	0.84	2.22
Te	ICP/MS	Acid	2.03E+02	µg/g d	0.00 *	3.86
Te	ICP/MS	FS.Ni	4.24E+02	µg/g d	8.08	18.74
Te	ICP/MS	FS.Zr	2.88E+02	µg/g d	21.27	28.70
Th	ICP/AES	Acid	3.49E+02	µg/g d	0.00 *	1.34
²³² Th	ICP/MS	Acid	1.85E+02	µg/g d	0.00 *	3.02
²³² Th	ICP/MS	Acid	2.03E-05	µCi/g d	0.00 *	2.95
²³² Th	ICP/MS	FS.Zr	4.99E+02	µg/g d	34.61	10.61
²³² Th	ICP/MS	FS.Zr	5.48E-05	µCi/g d	34.61	10.61

@ = Table does not include analytes where more than 50% of the data are "less than" values.

\$ = µg/g d is µg/g dried slurry; µCi/g d is µCi/g dried slurry.

* = A RSD(S) of 0.00% indicates that the variability between duplicate analytical measurements was greater than the variability between subsamples.

Table 4.3. Tank 241-C-106 Vendor Product - Variance Components. (page 8 of 9)

Analyte @	Method	Digest	Mean	Units \$	Random Sampling RSD %	Random Analytical RSD %
Ti	ICP/AES	Acid	3.50E+02	µg/g d	0.00 *	3.14
Ti	ICP/AES	FS.Ni	1.03E+03	µg/g d	7.96	2.85
Ti	ICP/AES	FS.Zr	1.02E+03	µg/g d	10.38	3.13
Ti	ICP/MS	Acid	3.58E+02	µg/g d	0.00 *	3.25
Ti	ICP/MS	FS.Ni	1.12E+03	µg/g d	11.76	2.51
Ti	ICP/MS	FS.Zr	1.31E+03	µg/g d	12.01	3.26
Tm	ICP/MS	Acid	6.17E-02	µg/g d	0.00 *	31.47
Tm.w	ICP/MS	FS.Ni	1.82E+00	µg/g d	62.86	38.99
Tm.wo	ICP/MS	FS.Ni	2.52E+00	µg/g d	31.98	47.42
Total Alpha	Rad	FS.Ni	6.78E+00	µCi/g d	2.19	2.64
Total Beta	Rad	FS.Ni	2.05E+03	µCi/g d	3.80	4.75
Total Carbon	WtChem	Direct	5.22E+04	ugC/g d	5.23	6.44
Total U	ICP/MS	Acid	1.91E+03	µg/g d	2.47	3.07
Total U	ICP/MS	FS.Ni	1.90E+03	µg/g d	0.00 *	6.96
Total U	ICP/MS	FS.Zr	2.01E+03	µg/g d	0.00 *	3.29
U	ICP/AES	Acid	1.95E+03	µg/g d	0.00 *	1.90
²³³ U	ICP/MS	Acid	1.73E-01	µg/g d	18.32	9.99
²³³ U	ICP/MS	Acid	1.68E-03	µCi/g d	18.24	11.69
²³⁴ U	ICP/MS	Acid	1.23E-01	µg/g d	0.00 *	19.64
²³⁴ U	ICP/MS	Acid	7.73E-04	µCi/g d	0.00 *	18.15
²³⁵ U	ICP/MS	Acid	1.30E+01	µg/g d	3.48	4.98
²³⁵ U	ICP/MS	Acid	2.80E-05	µCi/g d	3.48	4.98
²³⁶ U	ICP/MS	Acid	2.87E-01	µg/g d	9.82	11.21
²³⁶ U	ICP/MS	Acid	1.85E-05	µCi/g d	9.18	11.61
²³⁸ U	ICP/MS	Acid	1.90E+03	µg/g d	2.48	3.08
²³⁸ U	ICP/MS	Acid	6.37E-04	µCi/g d	2.48	3.08
²³⁸ U	ICP/MS	FS.Ni	1.89E+03	µg/g d	0.00 *	7.00
²³⁸ U	ICP/MS	FS.Ni	6.34E-04	µCi/g d	0.00 *	7.00
²³⁸ U	ICP/MS	FS.Zr	2.00E+03	µg/g d	0.00 *	3.14
²³⁸ U	ICP/MS	FS.Zr	6.71E-04	µCi/g d	0.00 *	3.14
V	ICP/MS	Acid	3.73E+01	µg/g d	9.64	16.01

@ = Table does not include analytes where more than 50% of the data are "less than" values.

\$ = µg/g d is µg/g dried slurry; µCi/g d is µCi/g dried slurry.

* = A RSD(s) of 0.00% indicates that the variability between duplicate analytical measurements was greater than the variability between subsamples.

Table 4.3. Tank 241-C-106 Vendor Product - Variance Components. (page 9 of 9)

Analyte a	Method	Digest	Mean	Units \$	Random Sampling RSD %	Random Analytical RSD %
W	ICP/MS	Acid	2.22E+02	µg/g d	6.91	2.13
W	ICP/MS	FS.Ni	4.99E+02	µg/g d	0.00 *	14.83
W	ICP/MS	FS.Zr	3.57E+02	µg/g d	33.21	10.16
Yb	ICP/MS	Acid	2.50E+01	µg/g d	34.64	28.28
Yb	ICP/MS	FS.Ni	1.26E+01	µg/g d	46.77	26.45
Yb	ICP/MS	FS.Zr	3.24E+00	µg/g d	0.00 *	45.60
Zn	ICP/AES	Acid	1.16E+02	µg/g d	1.75	1.93
Zn	ICP/AES	FS.Ni	1.83E+02	µg/g d	0.00 *	13.40
Zn	ICP/AES	FS.Zr	1.61E+02	µg/g d	0.00 *	8.52
Zn	ICP/MS	Acid	9.92E+01	µg/g d	3.25	6.12
Zn	ICP/MS	FS.Ni	3.07E+02	µg/g d	0.00 *	23.10
Zn	ICP/MS	FS.Zr	2.34E+02	µg/g d	18.01	9.71
Zr	ICP/AES	Acid	6.61E+02	µg/g d	32.55	5.44
Zr	ICP/AES	FS.Ni	1.11E+03	µg/g d	0.00 *	47.71
Zr	ICP/MS	Acid	2.56E+02	µg/g d	28.54	6.30
Zr	ICP/MS	FS.Ni	4.33E+02	µg/g d	0.00 *	53.11
m/z241	ICP/MS	Acid	7.73E-01	µg/g d	4.21	7.87
m/z241	ICP/MS	FS.Zr	1.65E+00	µg/g d	0.00 *	20.94
m/z93	ICP/MS	Acid	5.00E+01	µg/g d	27.31	4.67
m/z93	ICP/MS	FS.Ni	3.38E+01	µg/g d	0.00 *	77.10
m/z93	ICP/MS	FS.Zr	2.35E+02	µg/g d	37.31	8.31

a = Table does not include analytes where more than 50% of the data are "less than" values.

\$ = µg/g d is µg/g dried slurry; µCi/g d is µCi/g dried slurry.

* = A RSD(S) of 0.00% indicates that the variability between duplicate analytical measurements was greater than the variability between subsamples.

Table 4.4. Tank 241-C-106 Vendor Product Analytes with Sampling Variability > 20% and Analytical Variability > 20%. (page 1 of 2)

Sample Variability > 20%	Analytical Variability > 20%
As (ICP/MS.FS.Zr)	Ag (ICP/AES.FS.Ni)
B (ICP/AES.Acid)	Ag (ICP/AES.FS.Zr)
B (ICP/AES.FS.Ni)	²⁴¹ Am.gea (Rad.FS.Ni) *
B (ICP/MS.Acid)	B (ICP/AES.FS.Ni) *
B (ICP/MS.FS.Ni)	B (ICP/MS.FS.Zr) *
B (ICP/MS.FS.Zr)	Cl ⁻ (IC.Water) *
^{243/244} Cm (Rad.FS.Ni)	¹³³ Cs (ICP/MS.FS.Ni) *
¹⁴ C (Rad.FS.Ni)	¹³³ Cs (ICP/MS.FS.Zr) *
Cu.w (ICP/MS.FS.Ni)	Cu.w (ICP/MS.FS.Ni) *
Cu.wo (ICP/MS.FS.Ni)	¹⁴ C (Rad.FS.Ni) *
Dy (ICP/MS.FS.Zr)	Dy (ICP/MS.FS.Ni) *
Gd (ICP/MS.FS.Zr)	Er (ICP/MS.FS.Ni) *
Ho (ICP/MS.FS.Ni)	Eu (ICP/MS.FS.Ni) *
Li (ICP/AES.Acid)	Gd (ICP/MS.FS.Ni)
Li (ICP/AES.FS.Zr)	Ho (ICP/MS.Acid) *
Mo (ICP/MS.FS.Ni)	K (ICP/AES.FS.Zr) *
NH ₃ -N (WtChem.Water)	NH ₃ -N (WtChem.Water) *
NO ₃ ⁻ (IC.Water)	^{93m} Nb (Rad.FS.Ni) *
^{93m} Nb (Rad.FS.Ni)	⁵⁹ Ni.w (Rad.FS.Zr) *
¹⁰⁷ Pd.w (ICP/MS.Acid)	PO ₄ ³⁻ (IC.Water) *
Pt (ICP/MS.FS.Ni)	Pd (ICP/MS.FS.Ni) *
Rb (ICP/MS.FS.Zr)	¹⁰⁷ Pd.w (ICP/MS.Acid) *
Re (ICP/MS.FS.Ni)	¹⁰⁷ Pd.wo (ICP/MS.Acid) *
Rh.w (ICP/MS.FS.Ni)	Pt (ICP/MS.Acid) *
Sb.w (ICP/MS.Acid)	Pt (ICP/MS.FS.Zr) *
Sb.wo (ICP/MS.Acid)	Rb (ICP/MS.FS.Zr) *
	Re (ICP/MS.Acid) *
	Re (ICP/MS.FS.Ni) *

* = Large analytical variability is due to the results being close to the detection limits.

Table 4.4. Tank 241-C-106 Vendor Product Analytes with Sampling Variability > 20% and Analytical Variability > 20%. (page 2 of 2)

Sample Variability > 20%	Analytical Variability > 20%
Sn (ICP/MS.FS.Ni)	Rh.w (ICP/MS.FS.Ni)
Sn (ICP/MS.FS.Zr)	Rh.wo (ICP/MS.FS.Ni)
^{121m} Sn (Rad.FS.Ni)	Sb.w (ICP/MS.Acid) *
¹²⁶ Sn/ ^{126m} Sb/ ¹²⁶ Sb (Rad.FS.Ni)	Sn (ICP/MS.FS.Ni)
Sr (ICP/MS.FS.Ni)	^{121m} Sn (Rad.FS.Ni) *
Ta (ICP/MS.FS.Ni)	Ta (ICP/MS.Acid)
Ta (ICP/MS.FS.Zr)	Ta (ICP/MS.FS.Ni)
Tb (ICP/MS.FS.Zr)	Tb (ICP/MS.FS.Zr) *
Te (ICP/MS.FS.Zr)	Te (ICP/MS.FS.Zr)
²³² Th (ICP/MS.FS.Zr)	Tm (ICP/MS.Acid) *
Tm.w (ICP/MS.FS.Ni)	Tm.w (ICP/MS.FS.Ni) *
Tm.wo (ICP/MS.FS.Ni)	Tm.wo (ICP/MS.FS.Ni) *
W (ICP/MS.FS.Zr)	Yb (ICP/MS.Acid) *
Yb (ICP/MS.Acid)	Yb (ICP/MS.FS.Ni) *
Yb (ICP/MS.FS.Ni)	Yb (ICP/MS.FS.Zr) *
Zr (ICP/AES.Acid)	Zn (ICP/MS.FS.Ni)
Zr (ICP/MS.Acid)	Zr (ICP/AES.FS.Ni) *
m/z93 (ICP/MS.Acid)	Zr (ICP/MS.FS.Ni) *
m/z93 (ICP/MS.FS.Zr)	m/z241 (ICP/MS.FS.Zr) *
	m/z93 (ICP/MS.FS.Ni) *

* = Large analytical variability is due to the results being close to the detection limits.

Table 6.1. Tank 241-C-106 Vendor Product
Envelope "D" Specification Analytes - Random Variability. (page 1 of 4)

Analyte a	Method	Digest	Units &	Δ	RSD(Δ) (%)	95% UL	"D" Envelope Limit (EL)	UL/EL \$
Ag	ICP/AES	FS.Zr	g/L	1.23E-01	8.76	1.55E-01	0.17	0.911
Al	ICP/AES	FS.Ni	g/L	1.99E+00	2.63	2.14E+00	4.3	0.498
²⁴¹ Am.gea	Rad	FS.Ni	Ci/L	1.22E-04	9.40	1.56E-04	0.043	0.004
As	ICP/MS	FS.Ni	g/L	1.11E-02	7.38	1.35E-02	0.05	0.270
B	ICP/MS	FS.Ni	g/L	8.90E-03	21.41	1.45E-02	0.4	0.036
Ba	ICP/AES	FS.Zr	g/L	1.70E-02	3.08	1.85E-02	1.4	0.013
Bi	ICP/AES	Acid	g/L	1.67E-02	1.18	1.73E-02	0.86	0.020
¹⁴ C	Rad	FS.Ni	Ci/L	1.11E-07	78.53	9.45E-03	2E-06	0.183
CN ⁻	WtChem	Direct	g/L	3.69E-04	5.59	4.29E-04	0.5	0.001
Ca	ICP/AES	Acid	g/L	1.49E-01	1.22	1.54E-01	2.2	0.070
Cd	ICP/MS	FS.Ni	g/L	3.51E-03	5.13	4.04E-03	1.4	0.003
Ce	ICP/MS	FS.Zr	g/L	7.85E-03	6.52	9.35E-03	0.25	0.037
Cl ⁻	IC	Water	g/L	4.89E-03	8.49	6.10E-03	0.1	0.061
²⁴² Cm/ ²⁴² Am/ ^{242m} Am	Rad	FS.Ni	Ci/L	3.13E-07	10.14	4.06E-07	0.000037	0.011
²⁴² Cm/ ²⁴² Am/ ^{242m} Am	Rad	FS.Ni	Ci/L	3.13E-07	10.14	4.06E-07	3.1E-05	0.013
²⁴² Cm/ ²⁴² Am/ ^{242m} Am	Rad	FS.Ni	Ci/L	3.13E-07	10.14	4.06E-07	0.000032	0.013
²⁴³ Cm/ ²⁴⁴ Cm	Rad	FS.Ni	Ci/L	3.83E-06	12.97	5.28E-06	1.00E-09	5277
²⁴³ Cm/ ²⁴⁴ Cm	Rad	FS.Ni	Ci/L	3.83E-06	12.97	5.28E-06	9.3E-04	0.006
Co	ICP/MS	FS.Ni	g/L	1.02E-03	12.15	1.38E-03	0.14	0.010
⁶⁰ Co	Rad	FS.Ni	Ci/L	1.91E-05	3.11	2.08E-05	0.003	0.007
Cr	ICP/MS	FS.Ni	g/L	3.00E-02	6.05	3.53E-02	0.21	0.168
¹³⁵ Cs	Rad	FS.Ni	Ci/L	1.11E-07	3.15	1.21E-07	0.00003	0.004
¹³⁷ Cs/ ¹³⁷ Ba	Rad	FS.Ni	Ci/L	1.92E-02	3.29	2.11E-02	3	0.007
¹³⁷ Cs/ ¹³⁷ Ba	Rad	FS.Ni	Ci/L	1.92E-02	3.29	2.11E-02	3	0.007
Cu	ICP/MS	FS.Zr	g/L	6.07E-03	74.22	6.81E-03	0.15	0.045
Dy	ICP/MS	FS.Ni	g/L	7.84E-04	17.37	1.18E-03	0.008	0.148
Er	ICP/MS	FS.Ni	g/L	5.08E-04	14.64	7.25E-04	0.001	0.725
Eu	ICP/MS	FS.Ni	g/L	3.06E-04	30.72	5.80E-04	0.005	0.116
¹⁵⁴ Eu	Rad	FS.Ni	Ci/L	1.47E-04	1.76	1.55E-04	0.016	0.010
¹⁵⁵ Eu	Rad	FS.Ni	Ci/L	1.21E-04	4.02	1.36E-04	0.009	0.015
Fe	ICP/AES	FS.Ni	g/L	5.46E+00	3.29	5.99E+00	8.9	0.672
⁵⁵ Fe	Rad	FS.Ni	Ci/L	5.94E-06	2.62	6.39E-06	0.001	0.006

a = "most reasonable" analyte as described in Urie (1997).

Δ = g/L or Ci/L is g/L (at 31 gram waste oxide) or Ci/L (at 31 gram waste oxide).

* = At least one of the six analytical results is above the IDL but below the EDL.

\$ = If more than one radionuclide is listed, then the UL is compared to the specification for each radionuclide.

= NH₃-N converted to NH₃ by multiplying by (17/14).

Table 6.1. Tank 241-C-106 Vendor Product
Envelope "D" Specification Analytes - Random Variability. (page 2 of 4)

Analyte a	Method	Digest	Units &	$\bar{x}$	RSD($\bar{x}$) (%)	95% UL	"D" Envelope Limit (EL)	UL/EL %
Gd	ICP/MS	FS.Ni	g/L	9.70E-04	11.29	1.29E-03	0.003	0.430
Hg	CVA	Direct	g/L	1.33E-02	1.94	1.41E-02	0.03	0.469
Ho	ICP/MS	FS.Ni	g/L	2.35E-04	14.78	3.37E-04	0.001	0.337
¹²⁹ I.W	ICP/MS	Acid	Ci/L	8.48E-09	7.28	1.03E-08	9.00E-08	0.114
¹²⁹ I.Wo	ICP/MS	Acid	Ci/L	9.19E-09	9.24	1.17E-08	9.00E-08	0.130
La	ICP/MS	FS.Zr	g/L	3.58E-03	9.41	4.56E-03	0.8	0.006
Li	ICP/AES	Acid	g/L	1.78E-03	42.50	3.99E-03	0.043	0.093
Mg	ICP/MS	FS.Zr	g/L	4.88E-02	3.35	5.91E-02	0.65	0.091
Mn	ICP/MS	FS.Ni	g/L	1.09E-01	4.03	1.22E-01	2	0.061
Mo	ICP/MS	FS.Ni	g/L	2.21E-03	13.98	3.11E-03	0.2	0.016
NH ₃ #	WtChem	Water	g/L	8.25E-04	20.07	1.31E-03	0.5	0.003
NO ₂ ⁻ +NO ₃ ⁻	IC	Water	g/L	1.42E-01	10.69	1.87E-01	11.2	0.017
Na	ICP/AES	Acid	g/L	6.29E+00	2.93	6.83E+00	6	1.138
^{93m} Nb	Rad	FS.Ni	Ci/L	9.22E-06	30.19	1.73E-05	8.7E-05	0.199
Nd	ICP/AES	Acid	g/L	1.09E-02	1.19	1.13E-02	0.53	0.021
Ni	ICP/MS	FS.Zr	g/L	6.23E-02	2.35	6.66E-02	0.73	0.091
⁵⁹ Ni.W	Rad	FS.Zr	Ci/L	8.53E-07	10.23	1.11E-06	0.000014	0.079
⁵⁹ Ni.Wo	Rad	FS.Zr	Ci/L	7.66E-07	14.16	1.08E-06	0.000014	0.077
⁶³ Ni	Rad	FS.Zr	Ci/L	9.33E-05	2.39	9.98E-05	0.0016	0.062
²³⁷ Np	ICP/MS	FS.Zr	Ci/L	1.52E-07	9.49	1.94E-07	0.000023	0.008
P	ICP/AES	FS.Ni	g/L	1.29E-01	4.58	1.46E-01	0.54	0.271
Pb	ICP/AES	Acid	g/L	1.33E-01	1.11	1.37E-01	0.34	0.404
Pd	ICP/MS	FS.Zr	g/L	1.06E-03	7.63	1.30E-03	0.04	0.033
¹⁰⁷ Pd.W	ICP/MS	Acid	Ci/L	2.79E-08	58.24	7.53E-08	0.000004	0.019
¹⁰⁷ Pd.Wo	ICP/MS	Acid	Ci/L	4.41E-08	64.59	2.24E-07	0.000004	0.056
Pr	ICP/MS	FS.Zr	g/L	3.26E-03	3.73	3.62E-03	0.11	0.033
Pt	ICP/MS	FS.Ni	g/L	1.19E-03	16.03	1.75E-03	0.001	1.751
²³⁸ Pu	Rad	FS.Ni	Ci/L	2.45E-05	2.43	2.62E-05	0.00011	0.238
²³⁹ Pu	ICP/MS	FS.Zr	Ci/L	5.91E-05	3.38	6.49E-05	0.00095	0.068
²⁴⁰ Pu	ICP/MS	Acid	Ci/L	4.85E-06	4.62	5.51E-06	0.00026	0.021
²⁴¹ Pu	Rad	FS.Ni	Ci/L	2.70E-04	2.43	2.89E-04	0.0069	0.042

a = "most reasonable" analyte as described in Urie (1997).

g = g/L or Ci/L (at 31 gram waste oxide) or Ci/L (at 31 gram waste oxide).

* = At least one of the six analytical results is above the IDL but below the EOL.

s = If more than one radionuclide is listed, then the UL is compared to the specification for each radionuclide.

= NH₃-W converted to NH₃ by multiplying by (17/14).

Table 6.1. Tank 241-C-106 Vendor Product
Envelope "D" Specification Analytes - Random Variability. (page 3 of 4)

Analyte a	Method	Digest	Units &	β	RSD(β) (%)	95% UL	"D" Envelope Limit (EL)	UL/EL $\pm$
Rb	ICP/MS	FS.Zr	g/L	2.98E-04	18.38	4.58E-04	0.06	0.008
Re	ICP/MS	FS.Ni	g/L	2.57E-04	15.76	3.75E-04	0.03	0.012
Rh	ICP/MS	FS.Zr	g/L	5.60E-03	5.44	6.48E-03	0.04	0.162
Ru	ICP/MS	FS.Ni	g/L	3.71E-03	3.88	4.13E-03	0.11	0.038
Sb.w	ICP/MS	Acid	g/L	1.46E-05	47.84	3.50E-05	0.26	0.000
Sb.wo	ICP/MS	Acid	g/L	3.06E-05	25.35	7.95E-05	0.26	0.000
Si	ICP/AES	FS.Ni	g/L	2.67E+00	3.91	2.98E+00	5.8	0.513
$^{151}\text{Sm}/^{147}\text{Pm}$	Rad	FS.Ni	Ci/L	3.58E-03	2.84	3.88E-03	0.093	0.042
$^{151}\text{Sm}/^{147}\text{Pm}$	Rad	FS.Ni	Ci/L	3.58E-03	2.84	3.88E-03	0.16	0.024
Sn	ICP/MS	FS.Zr	g/L	3.96E-03	15.64	5.77E-03	0.011	0.525
$^{121}\text{m}_{\text{Sn}}$	Rad	FS.Ni	Ci/L	4.77E-05	24.84	8.23E-05	0.000009	9.146
$^{126}\text{Sn}/^{126}\text{m}_{\text{Sb}}/^{126}\text{Sb}$	Rad	FS.Ni	Ci/L	4.48E-07	15.50	6.51E-07	0.000048	0.014
$^{126}\text{Sn}/^{126}\text{m}_{\text{Sb}}/^{126}\text{Sb}$	Rad	FS.Ni	Ci/L	4.48E-07	15.50	6.51E-07	0.000034	0.019
$^{126}\text{Sn}/^{126}\text{m}_{\text{Sb}}/^{126}\text{Sb}$	Rad	FS.Ni	Ci/L	4.48E-07	15.50	6.51E-07	0.000005	0.135
Sr	ICP/MS	FS.Zr	g/L	5.84E-03	3.55	6.44E-03	0.16	0.040
$^{90}\text{Sr}/^{90}\text{Y}$	Rad	FS.Ni	Ci/L	2.83E-02	2.45	3.04E-02	3.1	0.010
$^{90}\text{Sr}/^{90}\text{Y}$	Rad	FS.Ni	Ci/L	2.83E-02	2.45	3.04E-02	3.1	0.010
TIC	WtChem	Direct	g/L	2.06E-01	4.36	2.32E-01	1.86	0.125
TOC	WtChem	Direct	g/L	1.86E+00	3.30	2.04E+00	3.4	0.600
Ta	ICP/MS	FS.Zr	g/L	1.11E-03	22.67	1.84E-03	0.008	0.231
Tb	ICP/MS	FS.Ni	g/L	3.18E-04	10.53	4.16E-04	0.001	0.416
^{99}Tc	ICP/MS	Acid	Ci/L	2.18E-07	10.82	2.86E-07	0.0045	0.000
Te	ICP/MS	FS.Ni	g/L	1.65E-02	8.96	2.08E-02	0.04	0.519
Th *	ICP/AES	Acid	g/L	1.35E-02	0.55	1.38E-02	0.16	0.086
Ti	ICP/MS	FS.Zr	g/L	5.10E-02	7.06	6.15E-02	0.4	0.154
Tm.w	ICP/MS	FS.Ni	g/L	7.06E-05	39.63	1.52E-04	0.001	0.152
Tm.wo	ICP/MS	FS.Ni	g/L	9.78E-05	36.01	2.01E-04	0.001	0.201
Total U	ICP/MS	FS.Ni	g/L	7.36E-02	2.84	7.97E-02	4.2	0.019
^{233}U	ICP/MS	Acid	Ci/L	6.52E-08	11.56	8.72E-08	1.00E-09	87.2
^{234}U	ICP/MS	Acid	Ci/L	3.00E-08	7.41	3.65E-08	7.70E-07	0.047
^{235}U	ICP/MS	Acid	Ci/L	1.09E-09	2.86	1.18E-09	3.20E-08	0.037

a = "most reasonable analyte as described in Urie (1997).

* = g/L or Ci/L is g/L (at 31 gram waste oxide) or Ci/L (at 31 gram waste oxide).

= At least one of the six analytical results is above the IDL but below the ECL.

S = If more than one radionuclide is listed, then the UL is compared to the specification for each radionuclide.

= $\text{NH}_3\text{-N}$ converted to NH_3 by multiplying by (17/14).

Table 6.1. Tank 241-C-106 Vendor Product
Envelope "D" Specification Analytes - Random Variability. (page 4 of 4)

Analyte ^a	Method	Digest	Units &	$\bar{A}$	RSD($\bar{A}$) (%)	95% UL	"D" Envelope Limit (EL)	UL/EL ^c
²³⁶ U	ICP/MS	Acid	Ci/L	7.17E-10	7.11	8.66E-10	8.20E-08	0.011
²³⁸ U	ICP/MS	FS.Zr	Ci/L	2.61E-08	1.28	2.70E-08	5.80E-07	0.047
V	ICP/MS	Acid	g/L	1.45E-03	8.58	1.81E-03	0.01	0.181
W	ICP/MS	FS.Ni	g/L	1.94E-02	6.05	2.28E-02	0.074	0.308
Yb	ICP/MS	FS.Ni	g/L	4.89E-04	29.08	9.04E-04	0.001	0.904
Zn	ICP/MS	FS.Ni	g/L	1.19E-02	9.43	1.52E-02	0.13	0.117
Zr	ICP/AES	FS.Ni	g/L	4.29E-02	19.48	6.73E-02	4.6	0.015

^a = "most reasonable" analyte as described in Urie (1997).

^b = g/L or Ci/L is g/L (at 31 gram waste oxide) or Ci/L (at 31 gram waste oxide).

^c = At least one of the six analytical results is above the IDL but below the EQL.

^d = If more than one radionuclide is listed, then the UL is compared to the specification for each radionuclide.

^e = NH₃-N converted to NH₃ by multiplying by (17/14).

Table 6.2. Tank 241-C-106 Vendor Product
Envelope "D" Specification Analytes - Less Than Values.

Analyte a	Method	Digest	Units &	Largest Result	"D" Envelope Limit (EL)	Largest/EL \$
^{110m}Ag	Rad	FS.Ni	Ci/L	< 3.49E-06	1.00E-08	349
^{243}Am	Rad	FS.Ni	Ci/L	< 7.76E-07	0.000005	0.155
Be	ICP/AES	Acid	g/L	< 3.88E-05	0.02	0.002
$^{144}\text{Ce}/^{144}\text{Pr}/^{144m}\text{Pr}$	Rad	FS.Ni	Ci/L	< 7.76E-05	0.0001	0.776
$^{144}\text{Ce}/^{144}\text{Pr}/^{144m}\text{Pr}$	Rad	FS.Ni	Ci/L	< 7.76E-05	0.0001	0.776
$^{144}\text{Ce}/^{144}\text{Pr}/^{144m}\text{Pr}$	Rad	FS.Ni	Ci/L	< 7.76E-05	1.0E-07	776
^{134}Cs	Rad	FS.Ni	Ci/L	< 1.16E-05	0.0068	0.002
^{152}Eu	Rad	FS.Ni	Ci/L	< 7.76E-05	0.00014	0.555
F ⁻	WetChem	Water	g/L	< 1.13E-01	1.1	0.103
K	ICP/AES	Acid	g/L	< 1.91E-02	0.41	0.047
^{242}Pu	Rad	FS.Ni	Ci/L	< 7.76E-07	7.10E-08	10.934
$^{106}\text{Ru}/^{106}\text{Rh}$	Rad	FS.Ni	Ci/L	< 1.16E-04	0.0002	0.582
$^{106}\text{Ru}/^{106}\text{Rh}$	Rad	FS.Ni	Ci/L	< 1.16E-04	0.0002	0.582
$^{125}\text{Sb}/^{125m}\text{Te}$	Rad	FS.Ni	Ci/L	< 7.76E-05	0.01	0.008
$^{125}\text{Sb}/^{125m}\text{Te}$	Rad	FS.Ni	Ci/L	< 7.76E-05	0.003	0.026
Se	ICP/AES	Acid	g/L	< 1.43E-02	0.16	0.090
^{79}Se	Rad	Acid	Ci/L	< 8.15E-09	4.20E-07	0.019
$^{113}\text{Sn}/^{113m}\text{In}$	Rad	FS.Ni	Ci/L	< 3.88E-07	1.18E-06	0.206
$^{113}\text{Sn}/^{113m}\text{In}$	Rad	FS.Ni	Ci/L	< 3.88E-07	1.18E-06	0.206
^{119m}Sn	Rad	FS.Ni	Ci/L	< 1.55E-06	1.00E-08	155.270
Tl	ICP/AES	Acid	g/L	< 4.77E-03	0.14	0.034
Y	ICP/AES	Acid	g/L	< 3.30E-03	0.05	0.066
^{93}Zr	Rad	FS.Ni	Ci/L	< 2.33E-05	1.4E-04	0.166

a = "most reasonable" analyte as described in Urie (1997).

\$ = If more than one radionuclide is listed, then the UL is compared to the specification for each radionuclide.

& = g/L or Ci/L is g/L (at 31 gram waste oxide) or Ci/L (at 31 gram waste oxide).

Table 6.3. Tank 241-C-106 Vendor Product
Envelope "D" Specification Analytes - Total Variability. (page 1 of 4)

Analyte @	Method	Digest	Units &	μ	UL/EL \$ SYS=10%	UL/EL \$ SYS=25%	UL/EL \$ SYS=50%	UL/EL \$ SYS=75%	UL/EL \$ SYS=100%
Ag	ICP/AES	FS.Zr	g/L	1.23E-01	1.007	1.287	1.801	2.326	2.853
Al	ICP/AES	FS.Ni	g/L	1.99E+00	0.602	0.801	1.138	1.475	1.812
²⁴¹ Am.gea	Rad	FS.Ni	Ci/L	1.22E-04	0.004	0.005	0.007	0.009	0.011
As	ICP/MS	FS.Ni	g/L	1.11E-02	0.303	0.391	0.550	0.711	0.873
B	ICP/MS	FS.Ni	g/L	8.90E-03	0.038	0.044	0.058	0.073	0.089
Ba	ICP/AES	FS.Zr	g/L	1.70E-02	0.016	0.021	0.030	0.039	0.047
Bi	ICP/AES	Acid	g/L	1.67E-02	0.025	0.034	0.048	0.062	0.076
¹⁴ C	Rad	FS.Ni	Ci/L	1.11E-07	0.184	0.190	0.207	0.232	0.262
CN ⁻	WtChem	Direct	g/L	3.69E-04	0.001	0.001	0.002	0.002	0.003
Ca	ICP/AES	Acid	g/L	1.49E-01	0.088	0.117	0.167	0.216	0.266
Cd	ICP/MS	FS.Ni	g/L	3.51E-03	0.003	0.004	0.006	0.008	0.010
Ce	ICP/MS	FS.Zr	g/L	7.85E-03	0.042	0.055	0.078	0.100	0.123
Cl ⁻	IC	Water	g/L	4.89E-03	0.068	0.087	0.121	0.157	0.192
²⁴² Cm/ ²⁴² Am/ ^{242m} Am	Rad	FS.Ni	Ci/L	3.13E-07	0.012	0.015	0.021	0.027	0.033
²⁴² Cm/ ²⁴² Am/ ^{242m} Am	Rad	FS.Ni	Ci/L	3.13E-07	0.014	0.018	0.025	0.032	0.040
²⁴² Cm/ ²⁴² Am/ ^{242m} Am	Rad	FS.Ni	Ci/L	3.13E-07	0.014	0.017	0.024	0.031	0.038
²⁴³ Cm/ ²⁴⁴ Cm	Rad	FS.Ni	Ci/L	3.83E-06	5658	6975	9601	12335	15099
²⁴³ Cm/ ²⁴⁴ Cm	Rad	FS.Ni	Ci/L	3.83E-06	0.006	0.008	0.010	0.013	0.016
Co	ICP/MS	FS.Ni	g/L	1.02E-03	0.011	0.013	0.018	0.023	0.029
⁶⁰ Co	Rad	FS.Ni	Ci/L	1.91E-05	0.008	0.011	0.016	0.020	0.025
Cr	ICP/MS	FS.Ni	g/L	3.00E-02	0.192	0.250	0.353	0.457	0.561
¹³⁵ Cs	Rad	FS.Ni	Ci/L	1.11E-07	0.005	0.006	0.009	0.012	0.015
¹³⁷ Cs/ ¹³⁷ Ba	Rad	FS.Ni	Ci/L	1.92E-02	0.008	0.011	0.016	0.020	0.025
¹³⁷ Cs/ ¹³⁷ Ba	Rad	FS.Ni	Ci/L	1.92E-02	0.008	0.011	0.016	0.020	0.025
Cu	ICP/MS	FS.Zr	g/L	6.07E-03	0.053	0.070	0.100	0.129	0.159
Dy	ICP/MS	FS.Ni	g/L	7.84E-04	0.155	0.185	0.249	0.318	0.388
Er	ICP/MS	FS.Ni	g/L	5.08E-04	0.771	0.938	1.281	1.642	2.007
Eu	ICP/MS	FS.Ni	g/L	3.06E-04	0.119	0.132	0.166	0.206	0.248
¹⁵⁴ Eu	Rad	FS.Ni	Ci/L	1.47E-04	0.012	0.016	0.023	0.029	0.036
¹⁵⁵ Eu	Rad	FS.Ni	Ci/L	1.21E-04	0.018	0.023	0.033	0.043	0.053
Fe	ICP/AES	FS.Ni	g/L	5.46E+00	0.802	1.065	1.511	1.958	2.406
⁵⁵ Fe	Rad	FS.Ni	Ci/L	5.94E-06	0.008	0.010	0.015	0.019	0.023

@ = "most reasonable" analyte as described in Urie (1997).

& = g/L or Ci/L is g/L (at 31 gram waste oxide) or Ci/L (at 31 gram waste oxide).

S = if more than one radionuclide is listed, then the UL is compared to the specification for each radionuclide.

Table 6.3. Tank 241-C-106 Vendor Product
Envelope "D" Specification Analytes - Total Variability. (page 2 of 4)

Analyte @	Method	Digest	Units &	$\bar{\mu}$	UL/EL \$ SYS=10%	UL/EL \$ SYS=25%	UL/EL \$ SYS=50%	UL/EL \$ SYS=75%	UL/EL \$ SYS=100%
Gd	ICP/MS	FS.Ni	g/L	9.70E-04	0.466	0.582	0.807	1.039	1.273
Hg	CVAA	Direct	g/L	1.33E-02	0.576	0.770	1.093	1.417	1.742
Ho	ICP/MS	FS.Ni	g/L	2.35E-04	0.358	0.435	0.594	0.761	0.931
¹²⁹ I _{L.W}	ICP/MS	Acid	Ci/L	8.48E-09	0.128	0.166	0.233	0.301	0.370
¹²⁹ I _{L.WO}	ICP/MS	Acid	Ci/L	9.19E-09	0.143	0.182	0.254	0.327	0.401
La	ICP/MS	FS.Zr	g/L	3.58E-03	0.006	0.008	0.011	0.014	0.018
Li	ICP/AES	Acid	g/L	1.78E-03	0.094	0.101	0.121	0.146	0.173
Mg	ICP/MS	FS.Zr	g/L	4.88E-02	0.125	0.195	0.312	0.431	0.549
Mn	ICP/MS	FS.Ni	g/L	1.09E-01	0.072	0.095	0.134	0.174	0.214
Mo	ICP/MS	FS.Ni	g/L	2.21E-03	0.017	0.020	0.028	0.036	0.044
NH ₃ #	WtChem	Water	g/L	8.25E-04	0.003	0.003	0.004	0.005	0.007
NO ₂ ⁻ +NO ₃ ⁻	IC	Water	g/L	1.42E-01	0.018	0.021	0.028	0.035	0.043
Na	ICP/AES	Acid	g/L	6.29E+00	1.367	1.818	2.581	3.345	4.110
^{93m} Nb	Rad	FS.Ni	Ci/L	9.22E-06	0.204	0.227	0.287	0.232	0.429
Nd	ICP/AES	Acid	g/L	1.09E-02	0.027	0.036	0.051	0.066	0.081
Ni	ICP/MS	FS.Zr	g/L	6.23E-02	0.111	0.148	0.210	0.272	0.335
⁵⁹ Ni _{L.W}	Rad	FS.Zr	Ci/L	8.53E-07	0.086	0.109	0.152	0.196	0.240
⁵⁹ Ni _{L.WO}	Rad	FS.Zr	Ci/L	7.66E-07	0.082	0.101	0.138	0.177	0.216
⁶³ Ni	Rad	FS.Zr	Ci/L	9.33E-05	0.076	0.101	0.144	0.186	0.229
²³⁷ Np	ICP/MS	FS.Zr	Ci/L	1.52E-07	0.009	0.012	0.016	0.021	0.026
P	ICP/AES	FS.Ni	g/L	1.29E-01	0.316	0.416	0.589	0.763	0.938
Pb	ICP/AES	Acid	g/L	1.33E-01	0.507	0.678	0.963	1.249	1.535
Pd	ICP/MS	FS.Zr	g/L	1.06E-03	0.036	0.047	0.066	0.085	0.104
¹⁰⁷ Pd _{L.W}	ICP/MS	Acid	Ci/L	2.79E-08	0.019	0.020	0.023	0.026	0.031
¹⁰⁷ Pd _{L.WO}	ICP/MS	Acid	Ci/L	4.41E-08	0.057	0.059	0.068	0.080	0.094
Pr	ICP/MS	FS.Zr	g/L	3.26E-03	0.039	0.052	0.073	0.095	0.116
Pt	ICP/MS	FS.Ni	g/L	1.19E-03	1.851	2.228	3.022	3.865	4.721
²³⁸ Pu	Rad	FS.Ni	Ci/L	2.45E-05	0.290	0.386	0.548	0.711	0.873
²³⁹ Pu	ICP/MS	FS.Zr	Ci/L	5.91E-05	0.081	0.108	0.153	0.199	0.244
²⁴⁰ Pu	ICP/MS	Acid	Ci/L	4.85E-06	0.025	0.033	0.046	0.060	0.073
²⁴¹ Pu	Rad	FS.Ni	Ci/L	2.70E-04	0.051	0.068	0.096	0.125	0.153
Rb	ICP/MS	FS.Zr	g/L	2.98E-04	0.008	0.009	0.013	0.016	0.020

@ = "most reasonable" analyte as described in Urie (1997).

& = g/L or Ci/L is g/L (at 31 gram waste oxide) or Ci/L (at 31 gram waste oxide).

\$ = If more than one radionuclide is listed, then the UL is compared to the specification for each radionuclide.

Table 6.3. Tank 241-C-106 Vendor Product
Envelope "D" Specification Analytes - Total Variability. (page 3 of 4)

Analyte Δ	Method	Digest	Units &	μ	UL/EL Σ SYS=10%	UL/EL Σ SYS=25%	UL/EL Σ SYS=50%	UL/EL Σ SYS=75%	UL/EL Σ SYS=100%
Re	ICP/MS	FS.Ni	g/L	2.57E-04	0.013	0.016	0.022	0.028	0.034
Rh	ICP/MS	FS.Zr	g/L	5.60E-03	0.186	0.244	0.345	0.447	0.549
Ru	ICP/MS	FS.Ni	g/L	3.71E-03	0.044	0.059	0.083	0.108	0.132
Sb.w	ICP/MS	Acid	g/L	1.46E-05	0.000	0.000	0.000	0.000	0.000
Sb.wo	ICP/MS	Acid	g/L	3.06E-05	0.000	0.000	0.001	0.001	0.001
Si	ICP/AES	FS.Ni	g/L	2.67E+00	0.605	0.801	1.136	1.471	1.807
$^{151}\text{Sm}/^{147}\text{Pm}$	Rad	FS.Ni	Ci/L	3.58E-03	0.050	0.067	0.095	0.123	0.151
$^{151}\text{Sm}/^{147}\text{Pm}$	Rad	FS.Ni	Ci/L	3.58E-03	0.029	0.039	0.055	0.071	0.088
Sn	ICP/MS	FS.Zr	g/L	3.96E-03	0.556	0.671	0.911	1.166	1.425
$^{121}\text{m}_{\text{Sn}}$	Rad	FS.Ni	Ci/L	4.77E-05	9.45	10.76	13.94	17.53	21.25
$^{126}\text{Sn}/^{126\text{m}}\text{Sb}/^{126}\text{Sb}$	Rad	FS.Ni	Ci/L	4.48E-07	0.014	0.017	0.024	0.030	0.037
$^{126}\text{Sn}/^{126\text{m}}\text{Sb}/^{126}\text{Sb}$	Rad	FS.Ni	Ci/L	4.48E-07	0.020	0.024	0.033	0.042	0.052
$^{126}\text{Sn}/^{126\text{m}}\text{Sb}/^{126}\text{Sb}$	Rad	FS.Ni	Ci/L	4.48E-07	0.143	0.172	0.234	0.300	0.367
Sr	ICP/MS	FS.Zr	g/L	5.84E-03	0.048	0.063	0.090	0.116	0.143
$^{90}\text{Sr}/^{90}\text{Y}$	Rad	FS.Ni	Ci/L	2.83E-02	0.012	0.016	0.023	0.029	0.036
$^{90}\text{Sr}/^{90}\text{Y}$	Rad	FS.Ni	Ci/L	2.83E-02	0.012	0.016	0.023	0.029	0.036
TIC	WtChem	Direct	g/L	2.06E-01	0.146	0.192	0.273	0.353	0.434
TOC	WtChem	Direct	g/L	1.86E+00	0.716	0.950	1.348	1.747	2.146
Ta	ICP/MS	FS.Zr	g/L	1.11E-03	0.239	0.276	0.361	0.456	0.554
Tb	ICP/MS	FS.Ni	g/L	3.18E-04	0.453	0.570	0.793	1.022	1.252
^{99}Tc	ICP/MS	Acid	Ci/L	2.18E-07	0.000	0.000	0.000	0.000	0.000
Te	ICP/MS	FS.Ni	g/L	1.65E-02	0.573	0.731	1.022	1.319	1.618
Th	ICP/AES	Acid	g/L	1.35E-02	0.109	0.146	0.208	0.270	0.332
Ti	ICP/MS	FS.Zr	g/L	5.10E-02	0.173	0.224	0.315	0.408	0.501
Tm.w	ICP/MS	FS.Ni	g/L	7.06E-05	0.155	0.167	0.202	0.245	0.292
Tm.wo	ICP/MS	FS.Ni	g/L	9.78E-05	0.205	0.223	0.274	0.335	0.401
Total U	ICP/MS	FS.Ni	g/L	7.36E-02	0.023	0.030	0.043	0.056	0.069
^{233}U	ICP/MS	Acid	Ci/L	6.52E-08	94.26	117.59	162.84	209.59	256.75
^{234}U	ICP/MS	Acid	Ci/L	3.00E-08	0.053	0.069	0.096	0.125	0.153
^{235}U	ICP/MS	Acid	Ci/L	1.09E-09	0.044	0.059	0.084	0.109	0.133
^{236}U	ICP/MS	Acid	Ci/L	7.17E-10	0.012	0.015	0.022	0.028	0.034
^{238}U	ICP/MS	FS.Zr	Ci/L	2.61E-08	0.058	0.078	0.111	0.143	0.176

Δ = "most reasonable" analyte as described in Urie (1997).

Σ = g/L or Ci/L is g/L (at 31 gram waste oxide) or Ci/L (at 31 gram waste oxide).

Σ = If more than one radionuclide is listed, then the UL is compared to the specification for each radionuclide.

Table 6.3. Tank 241-C-106 Vendor Product
Envelope "D" Specification Analytes - Total Variability. (page 4 of 4)

Analyte ^a	Method	Digest	Units ^b	$\bar{x}$	UL/EL \$ SYS=10%	UL/EL \$ SYS=25%	UL/EL \$ SYS=50%	UL/EL \$ SYS=75%	UL/EL \$ SYS=100%
V	ICP/MS	Acid	g/L	1.45E-03	0.200	0.256	0.359	0.464	0.569
W	ICP/MS	FS.Ni	g/L	1.94E-02	0.351	0.458	0.646	0.836	1.026
Yb	ICP/MS	FS.Ni	g/L	4.89E-04	0.928	1.036	1.314	1.637	1.975
Zn	ICP/MS	FS.Ni	g/L	1.19E-02	0.128	0.163	0.227	0.294	0.360
Zr	ICP/AES	FS.Ni	g/L	4.29E-02	0.015	0.018	0.024	0.030	0.037

^a = "most reasonable" analyte as described in Urie (1997).

^b = g/L or Ci/L is g/L (at 31 gram waste oxide) or Ci/L (at 31 gram waste oxide).

\$ = If more than one radionuclide is listed, then the UL is compared to the specification for each radionuclide.

Table 6.4. Tank 241-C-106 Vendor Product - Ratio (UL/EL) > 1.

Analyte	Systematic Variability Estimate at which the Ratio (UL/EL) > 1	Observed Systematic Variability
Na	0%	6% - sd
Pt	0%	42% - rs
^{121m} Sn	0%	30% - ct
²³³ U	0%	NA
^{243/244} Cm	0% (²⁴³ Cm EL)	7% - (²⁴¹ Am sp)
Ag	between 0% & 10%	11.5% - sp
Al	between 25% & 50%	< 10% - sd
Er	between 25% & 50%	1% - rs
Fe	between 10% & 25%	0.6% - sp
Gd	between 50% & 75%	8% - rs
Hg	between 25% & 50%	< 25% - sp
Pb	between 50% & 75%	< 25% - sp
Si	between 25% & 50%	< 25% - sp
Sn	between 50% & 75%	< 25% - sp
TOC	between 25% & 75%	< 25% - sp
Tb	between 50% & 75%	< 25% - sp
Te	between 25% & 50%	< 25% - sp
W	between 75% & 100%	< 25% - sp \$
Yb	between 10% & 25%	< 25% - sp \$

sp = systematic variability estimated from spike analyses.
 sd = systematic variability estimated from serial dilutions.
 rs = systematic variability estimated from reference standards.
 ct = systematic variability estimated from counting statistics.
 \$ = spike analysis is within the specified limits, but the control standard was outside the control limits.

DISTRIBUTION SHEET

To Distribution	From Production Planning & Control	Page 1 of 1		
		Date: 03/28/97		
Project Title/Work Order HNF-SD-WM-DP-225, Rev. 1 "Tank Waste Remediation System(TWRS) Privatization Contractor Samples Waste Envelope D Material 241-C-106"		EDT NO.: N/A		
		ECN NO.: ECN-633345		
Name	MSIN	Text With all Attach	EDT/ECN ONLY	

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