

## **Inorganic and Radiochemical Analysis of AW-101 and AN-107 Tank Waste**

### **Inorganic**

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## Summary

Thirty jars of waste material from tank AW-101 and seventeen jars of waste material from tank AN-107 were received by Battelle. The contents of all jars of AW-101 were mixed to provide a single composite, as were the contents of all jars of AN-107. Each composite was homogenized and representative sub-samples extracted for organic, radiochemical and inorganic regulatory analyses.

The representative sub-samples were analyzed for inorganic, radiochemical, and organic analyses for analytes of interest as defined in Test Plan BNFL-29953-2. This report presents the inorganic (including TCLP metals) and radiochemical results obtained to date. Organic results are reported in report BNFL-RPT-001 (PNWD-2461).

The characterization analyses of the as received material for AW-101 and AN-107 include:

- Inductively-coupled plasma spectrometry for Ag, Al, As, B, Ba, Be, Bi, Ca, Cd, Co, Cr, Cu, Fe, K, Li, Mg, Mn, Mo, Na, Ni, P, Pb, Pd, Rh, Sb, Se, Si, Sn, Tl, U, V, W, Y, Zn, and Zr (Note: Although not specified in the test plan Ce, La, Nd, and Sr were also measured and reported for information only)
- Radiochemical analyses for total alpha and total beta activity, H-3, C-14, Co-60, Se-79, Sr-90/Y-90, Nb-94, Ru-106/Rh-106, Sb-125, Sn-126, Cs-134, Cs-137, Eu-154, Eu-155, Pu-238, Pu-239+240, Pu-241, Am-241 (by GEA and AEA), Cm-242, and Cm-243+244
- Inductively-coupled mass spectrometry for Pt, Ta, Tc-99, I-129, U-233, U-234, U-235, U-236, Np-237, U-238, Pu-239, Pu-240
- Total uranium by kinetic phosphorescence
- Ion chromatography for  $\text{Br}^-$ ,  $\text{Cl}^-$ ,  $\text{F}^-$ ,  $\text{NO}_2^-$ ,  $\text{NO}_3^-$ ,  $\text{PO}_4^{3-}$ , and  $\text{SO}_4^{2-}$  (Note: Oxalate,  $\text{C}_2\text{O}_4^{2-}$ , was also measured and reported for information only.)
- Mercury, cyanide, ammonia, and inorganic and organic carbon
- Free hydroxide and pH determination (supernatant only)
- Flashpoint determinations (supernatant only)

Except for a very few cases, the characterization results met or exceeded the quality control requirements established by the governing quality assurance plan, and met or exceeded the minimum reportable quantity requirements specified by BNFL. Whenever possible the analyses were performed to SW-846 protocols so that the results can be used to support permit application, as well as to provide feed envelope characterization data.

Results for all inorganic and radiochemical analytes of interest are included in this report except cyanide, mercury, carbon-14, and flashpoint, which are to be included in revision 1 of this report.

## Terms and Abbreviations

AEA	alpha energy analysis
ALARA	as low as reasonably achievable
BNFL	BNFL, Inc; subsidiary of British Nuclear Fuels, Ltd.
COC	chain of custody
CVAA	cold vapor atomic absorption
EQL	estimated quantitation level
GEA	gamma energy analysis
HLRF	High Level Radiation Facility
IC	ion chromatography
ICP	inductively coupled plasma/atomic emission spectrometry
ICP/MS	inductively coupled plasma/mass spectrometry
ISE	ion specific electrode
LCS	laboratory control standard
MDL	method detection limit
MRQ	minimum reportable quantity
MSA	method of standard addition
NIST	National Institute of Standards and Technology
%D	percent difference
PGM	platinum group metals
QA	quality assurance
QC	quality control
QPR	Quality Problem Report
RPD	relative percent difference
SAL	Shielded Analytical Laboratory
SRM	Standard Reference Material
TC	total carbon
TCLP	toxicity characteristic leaching procedure
TIC	total inorganic carbon
TOC	total organic carbon
TRU	transuranic
W-DOE	Washington State Department of Ecology

## Units

$^{\circ}\text{C} - ^{\circ}\text{F}$	degrees Centigrade / degrees Fahrenheit
g	gram
g/mL	gram per milliliter
$\mu\text{g/g} - \mu\text{g/mL}$	microgram per gram / microgram per milliliter
$\mu\text{Ci/g} - \mu\text{Ci/mL}$	microcurie per gram / microcurie per milliliter
mL	milliliter
mmole/mL	millimole per milliliter
nCi/g	nanocurie per gram
pCi/g	picocurie per gram
Vol%	volume percent
Wt%	weight percent

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## 1.0 Introduction

This report presents the inorganic and radiochemical analytical results for AW-101 and AN-107 as received materials. The analyses were conducted in support of the BNFL Proposal No. 30406/29274 Task 5.0. The inorganic and radiochemical analysis results obtained from the as received materials are used to provide initial characterization information for subsequent process testing and to provide data to support permit application activities. Quality Assurance (QA) Plan MCS-033 provides the operational and quality control protocols for the analytical activities, and whenever possible, analyses were performed to SW-846 equivalent methods and protocols.

The inorganic and radiochemical analytes of interest and recommended methods are defined in the BNFL Proposal No. 30406/29274 and Test Plan BNFL-29953-2 Revision 1. All inorganic and radiochemical analytes of interest defined by these documents, except cyanide, mercury, carbon-14, and flashpoint are reported, with estimated method detection limits (MDL) provided where analytes of interest are not detected. When completed, cyanide, mercury, carbon-14, and flashpoint will be included in revision 1 of this report. Certain other originally requested analyses have not been performed based on agreements between Battelle, BNFL, and/or W-DOE. These inorganic analyses are total sulfide, reactive sulfide, reactive cyanide, total sulfur, total nitrogen, total iodine, and stainless steel corrosion testing.

Per the analysis protocols established by the QA Plan, process blanks, samples, duplicates, blank spikes (or lab control standards) and matrix spikes (or post spikes) were analyzed, as appropriate. Recoveries for quality control samples (such as matrix spikes and blank spikes) are discussed in this report and evaluated for their effect on the reported results if they fail to meet the acceptance criteria of QA Plan MCS-033. Matrix spike, post spike, and lab control standard/blank spike results, as well as calibration verification standards data, are included in the Project File 29274 (Record Inventory and Disposition Schedule, Technical Support to BNFL for Phase 1B, T5.5).

Composites of both AW-101 and AN-107 as received material were prepared per Test Plan BNFL-29953-1, Revision 0. The AN-107 composite (from 17 shipping jars) and AW-101 composite (from 30 shipping jars) were prepared in separate four liter glass kettles. A bladed impeller was used to homogenize the material. While the composite was being stirred, a 1/4-inch (outside diameter) stainless steel probe was used to vacuum transfer sub-samples of the homogenized slurry to a minimum of five 125-mL glass jars. The contents of each 125-mL glass jar were extracted from different elevations and radial locations within the kettles during mixing. These sub-samples were allowed to settle for approximately 12 hours. After this settling period, the volume percent of settled solids in each of the 125-mL glass jars were similar (i.e., 19-21% for AW-101 and 18-22% for AN-107), providing indication that the sub-samples are representative of the composite.

Figures 1.1 and 1.2 provide sample flow diagrams for the preparation of the AW-101 and AN-107 as received analytical characterization sub-samples. Six 125-mL glass jars of AN-107 and five 125-mL jars of AW-101 were sub-sampled for characterization analysis. The compositing and sub-sampling operations were conducted in the High Level Radiation Facility (HLRF). The sub-samples were transferred under chain-of-custody (COC) to the Shielded Analytical Laboratory (SAL) for characterization analysis preparation and distribution.



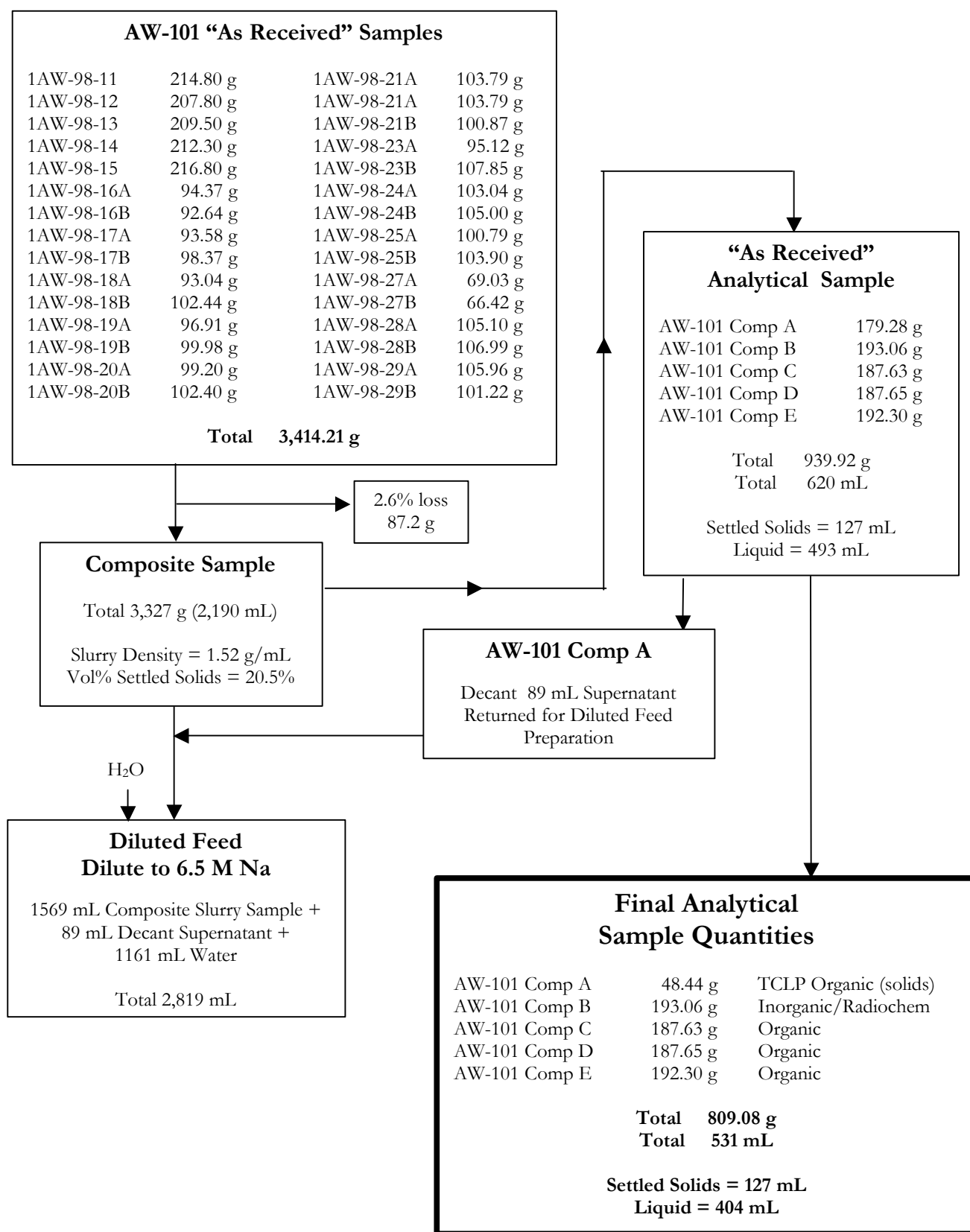


Figure 1.1 Flow Diagram for AW-101 As Received Analytical Samples

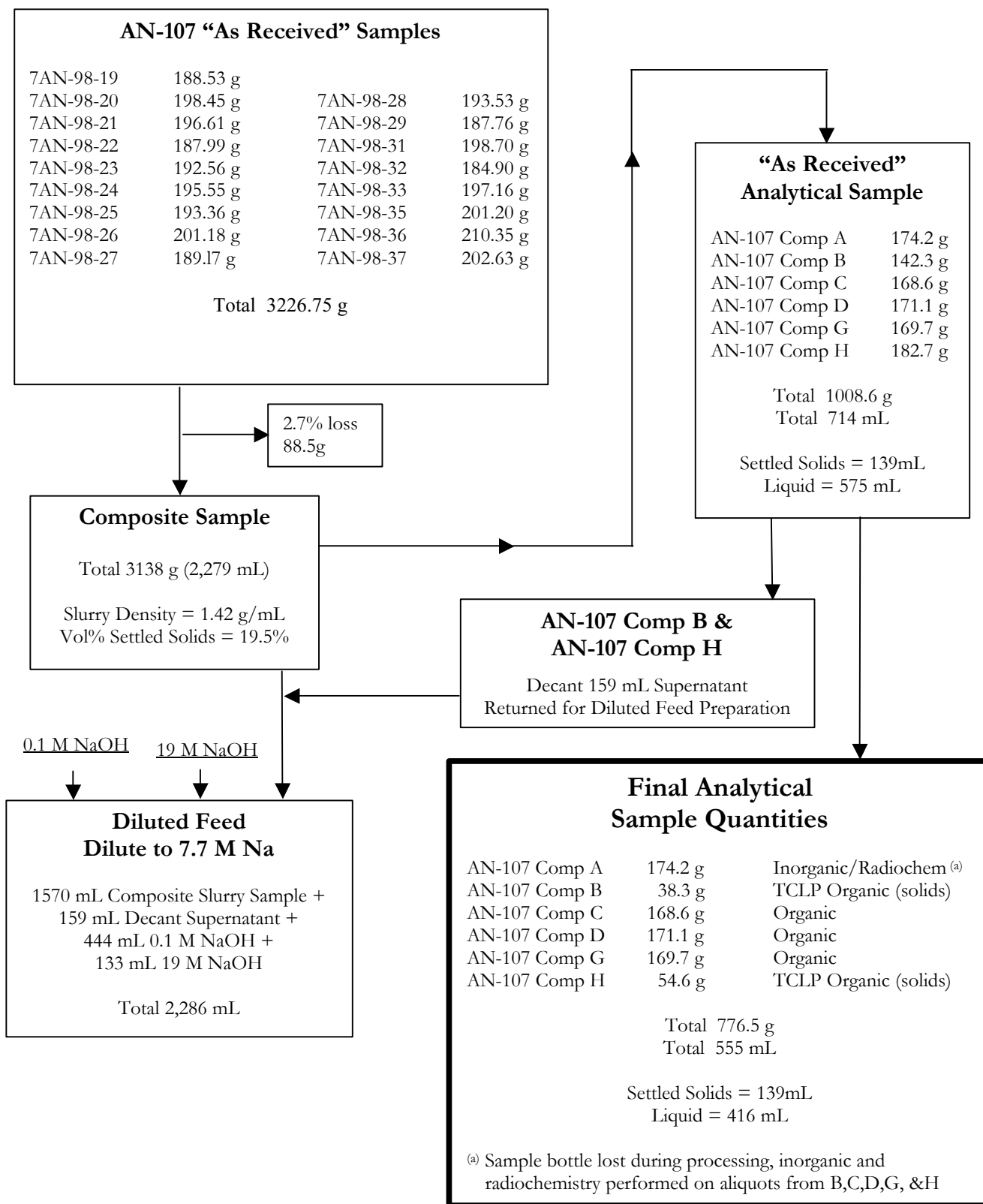


Figure 1.2 Flow Diagram for AN-107 As Received Analytical Samples

## 2.0 Sample Processing

### 2.1 Phase Separation

Prior to phase separation, the jars identified for inorganic and radiochemical analysis were processed for weight percent filtered solids and weight percent filtered dried solids. The percent filtered solids were needed to provide data for the TCLP calculations without opening the jars identified for organic analysis (particularly to minimize loss of volatiles). The jars identified for the TCLP (solids for organic TCLP constituents only) could not be used for this measurement, since most of the supernatant had been removed to provide additional supernatant for process testing in the HLRF. For the percent solids sub-sampling, it was necessary to homogenize the as received slurry by vigorous stirring (magnetic stirrer) to ensure valid sub-samples. During this stirring, the AN-107 glass jar identified for inorganic and radiochemical analysis broke and all contents were lost. This problem was reported via Quality Problem Report (QPR) 99-BNFL-001. Because of this loss, the inorganic and radiochemical analyses for AN-107 were performed on supernatants and solids from the jars identified for organic analysis. The inorganic and radiochemical analysis sub-sampling plan was provided to the SAL via special instruction included with Analytical Service Request number 5188. The inorganic and radiochemical sub-sampling from the AN-107 organic jars was performed after all organic sub-sampling had been completed, to minimize loss of volatile organic compounds. The inorganic and radiochemical sub-sampling for AW-101 was performed from the jar identified for inorganic and radiochemical analysis.

The contents of each of the jars (AW-101 Comp B, AN-107 Comp C, D, and G) were separated into solids and supernatant phases so that inorganic and radiochemical analyses could be performed on each phase. The phase separation was performed by centrifuging and decanting the supernatant, since previous experience indicated that filtering of the samples would be extremely difficult. Each sample was centrifuged in its original jar, and the supernatant decanted to pre-labeled teflon bottles (i.e., the wet solids remained in the original jar). Following processing for organic analysis, sub-samples of the supernatant and solids from AW-101 and AN-107 were processed for inorganic and radiochemical analysis. Table 2.1 details the quantity of supernatant and wet solids collected from each sub-sample.

The weight percent supernatant and weight percent centrifuged solids for the AW-101 Comp B (used for the inorganic analysis) is considerably different than the results obtained for AW-101 Comp C, D, and E (used for organic analysis). The weight percent centrifuged solids fraction for AW-101 Comp C, D, and E ranged from 16.1% to 16.3%, whereas the weight percent solids for the AW-101 Comp B was 20.6%. This difference does not affect the supernatant and solids result reported for AW-101; however, it does affect the slurry concentration calculated from the supernatant and solids concentrations adjusted for the supernatant and solids weight fractions. Therefore, the maximum slurry concentrations reported for the inorganic and radiochemical analytes are calculated based on a supernatant to solids weight fractions of 83.8 to 16.2, the same as used for calculating the maximum slurry concentration of organic compounds (Report BNFL-29953-001, PNWD-2461).

**Table 2.1 Supernatant and Solids Masses after Phase Separation of As Received Material**

Sample <sup>(1)</sup>	Lab ID	Total Mass (g) <sup>(2)</sup>	Supernatant		Centrifuged Wet Solids	
			Mass (g)	Wt%	Mass (g)	Wt%
AW-101 Comp (B)	99-0648 Supernatant 99-0650 Solids	195.5	155.2	79.4	40.3	20.6
AW-101 Comp (C) <sup>(3)</sup>	99-0648 Supernatant 99-0650 Solids	188.2	157.6	83.7	30.6	16.3
AW-101 Comp (D) <sup>(3)</sup>		190.7	160.0	83.9	30.7	16.1
AW-101 Comp (E) <sup>(3)</sup>		190.1	159.3	83.8	30.8	16.2
AN-107 Comp (C)	99-0649 Supernatant 99-0651 Solids	170.4	151.2	88.8	19.2	11.2
AN-107 Comp (D)		170.8	151.4	88.6	19.4	11.4
AN-107 Comp (G)		162.5	143.4	88.2	19.1	11.8

<sup>(1)</sup> Suffix ( ) represents individual jar/bottle identification number.

<sup>(2)</sup> Total mass remaining following phase separation activities in the SAL. In some cases, small differences in total mass from original sub-sample due to transferring a few mL of supernatant to balance jars for centrifuging.

<sup>(3)</sup> AW-101 C, D, and E used only for organic analysis, included in table only for comparison with AW-101 B.

## 2.2 Supernatant Density Measurements

Due to the viscous nature of the as received supernatants, all supernatant samples were processed by weight (i.e., analytical sub-samples for processing were taken by weight instead of by volume). The density of each as received supernatant was determined using method PNL-ALO-501. The resulting densities are used to convert reported supernatant results from mass/mass to mass/volume.

Table 2.2 provides the density results obtained on AW-101 and AN-107 as received supernatants following phase separation.

**Table 2.2 Density Results for AW-101 and AN-107 As Received Supernatants**

Bottle ID <sup>(1)</sup>	Lab ID	Sample Density (g/mL)	Duplicate Density (g/mL)	Average Density (g/mL)
AW-101 Comp (C)	99-0648	1.487	1.480	1.484
AN-107 Comp (C)	99-0649	1.421	1.418	1.420

<sup>(1)</sup> Suffix ( ) represents individual jar/bottle identification number.

## 2.3 Sample Preparation of Supernatants and Solids

The processing of AW-101 and AN-107 as received supernatants and solids and distribution of the processed samples is detailed in Figure 2.1. Except for the source of AW-101 and AN-107 analytical sub-samples (i.e., AW-101 sub-samples extracted from a single jar and AN-107 sub-samples extracted from five different jars), the processing of the supernatants and solids is the same for both materials.

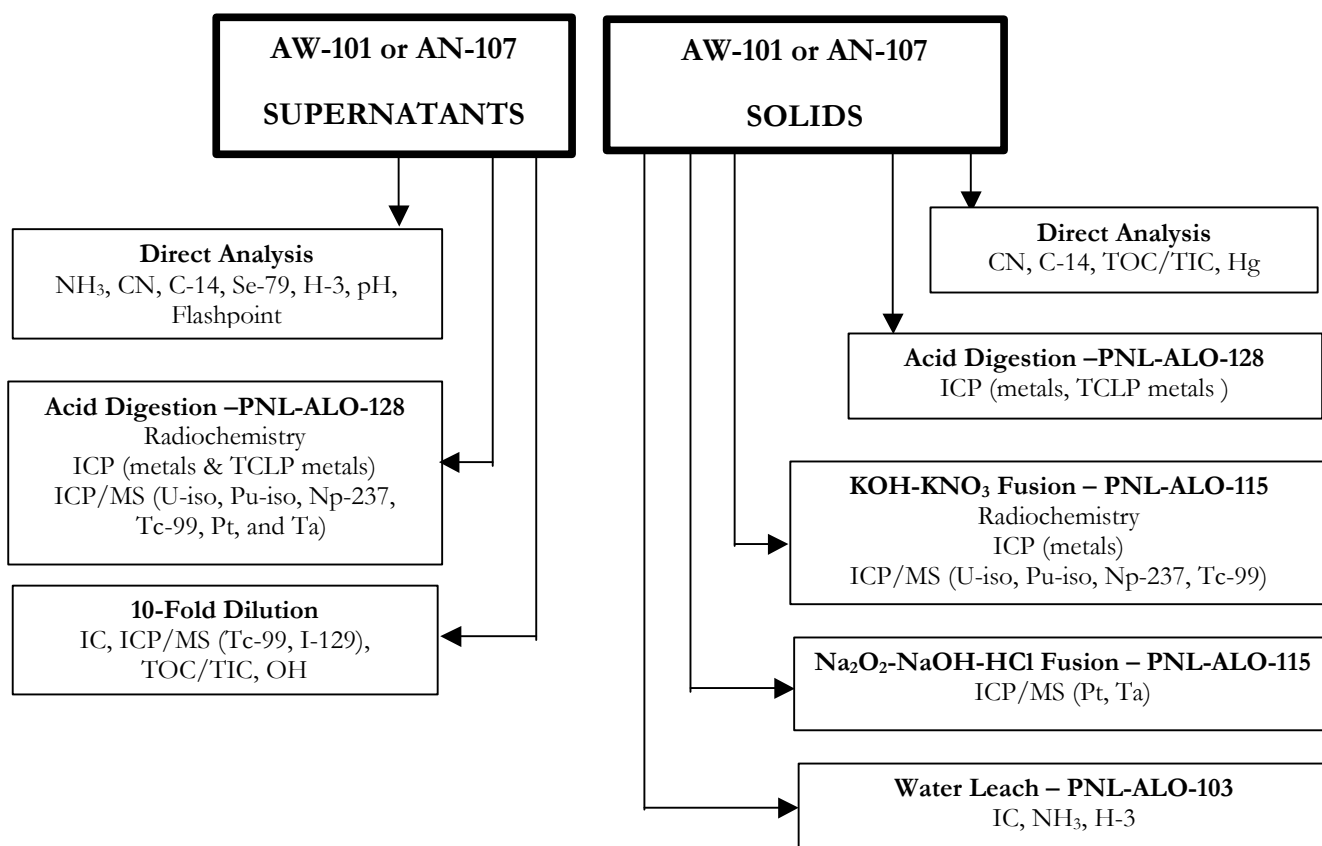
### Supernatants

As received supernatant samples were prepared by acid digestion per procedure PNL-ALO-128 (HNO<sub>3</sub>-HCl) for metals analysis (by inductively coupled plasma spectrometry [ICP] and inductively coupled plasma – mass spectrometry [ICP/MS]), radiochemical analysis, and metal and

radionuclides analysis by ICP/MS. The resulting digestion solutions from AW-101 were clear and those from AN-107 were slightly brown in color. The acid digestion solutions from both supernatants had no visible residue or precipitate. All other analyses were performed directly on the supernatant or on a prepared 10-fold water dilution of the supernatant.

### Solids

Four preparative methods, PNL-ALO-129 (HNO<sub>3</sub>-HCl acid digestion), PNL-ALO-115 (KOH-KNO<sub>3</sub> fusion), PNL-ALO-116 (Na<sub>2</sub>O<sub>2</sub>-NaOH-NaCl fusion), and PNL-ALO-103 (water leach), were performed on the wet centrifuged solids to provide analysis solutions for analytes of interest. The acid digestion and KOH-KNO<sub>3</sub> fusion preparations were analyzed for metals by ICP, and the KOH-KNO<sub>3</sub> fusion preparations were used for radiochemical analyses and for metals and radionuclides by ICP/MS. The Na<sub>2</sub>O<sub>2</sub>-NaOH-NaCl fusion was prepared specifically to obtain solutions for analysis of platinum group metals (PGM) by ICP/MS. All acid digestion and fusion preparations produced clear solutions with no visible residue, except for a few of the sample prepared by the Na<sub>2</sub>O<sub>2</sub>-NaOH-NaCl fusion, which demonstrated a slight cloudiness. Aliquots from the water leaching of the solids were analyzed for soluble anions by ion chromatography (following filtering), ammonia by ion specific electrode, and tritium by liquid scintillation counting. Some analyses, e.g., total organic carbon (TOC), were performed directly on the solids material.



**Figure 2.1 Flow Diagram for Analytical Preparation of Supernatants and Solids**

### 3.0 Analysis Results for Analytes of Interest

Tables 3.1 through 3.6 provide the results for all inorganic and radiochemical analyses performed on the AW-101 and AN-107 as received materials. Results for samples and duplicates, as well as processing blanks, are reported. Although the supernatants were processed by weight, the density of the supernatants has been used to provide the results in  $\mu\text{g/mL}$  or  $\mu\text{Ci/mL}$ , as appropriate. Solids are reported in  $\mu\text{g/g}$  or  $\mu\text{Ci/g}$ , as appropriate, where g represents mass of centrifuged wet solids.

The ICP/MS results are reported in both  $\mu\text{g/mL}$  and  $\mu\text{Ci/mL}$  for supernatants and  $\mu\text{g/g}$  and  $\mu\text{Ci/g}$  for solids. The results are provided based on both curies and mass so that a direct comparison can be made against the minimum reportable quantity (MRQ) specifications. The radionuclides measured by ICP/MS have supernatant MRQs specified in  $\mu\text{Ci/mL}$  and solids MRQs specified in  $\mu\text{g/g}$ .

To evaluate the concentrations of analytes of interest in the as received slurry material, slurry results have been calculated from the concentrations measured in the supernatant and in the wet centrifuges solids and the weight fractions of each phase. To provide a conservative total slurry concentration, the highest measured concentration from either the sample or the duplicate for each phase is used in the calculation. Where no analyte concentration is measured (i.e., results less than MDL), the lowest MDL is used in the calculation. The “maximum” slurry concentration is calculated by Equation (1).

$$C_m = ((C_l / D_l) * W_l) + (C_s * W_s) \quad (1)$$

Where:  $C_m$  = Maximum slurry concentration in  $\mu\text{g/g}$  or  $\mu\text{Ci/g}$   
 $C_l$  = Concentration of supernatant in  $\mu\text{g/mL}$  or  $\mu\text{Ci/mL}$   
 $D_l$  = Density of supernatant in  $\text{g/mL}$   
 $W_l$  = Weight fraction of supernatant  
(i.e., 0.838 for AW-101, 0.886 for AN-107)  
 $C_s$  = Concentration of solids in  $\mu\text{g/g}$  or  $\mu\text{Ci/g}$   
 $W_s$  = Weight fraction of solids  
(i.e., 0.162 for AW-101, 0.114 for AN-107)

An evaluation of the TCLP metals is provided in Section 4.0.

**Table 3.1 AW-101 As Received --- ICP Metals Results**

<i>Tank Material</i>	AW 101 As Received <sup>(1)</sup>												
<i>Matrix Dissolution Lab ID Bottle/Jar ID Units</i>	Supernatant				Centrifuged Wet Solids								
	Acid Digest				KOH-KNO <sub>3</sub> fusion				Acid Digest				Max Slurry Conc. µg/g
	99-0644-pb	99-0648	99-0648-d	<sup>(2)</sup>	99-0650-pb	99-0650	99-0650-d	<sup>(2)</sup>	99-0650-pb	99-0650	99-0650-d	<sup>(2)</sup>	
	ProcBlnk	Comp B	Comp B	RPD	ProcBlnk	Comp B	Comp B	RPD	ProcBlnk	Comp B	Comp B	RPD	
	µg/mL	µg/mL	µg/mL	(%)	µg/g	µg/g	µg/g	(%)	µg/g	µg/g	µg/g	(%)	
<b>RNFI List <sup>(4)</sup></b>													
Ag	< 2	< 2	< 2		< 170	< 160	< 160		< 3	[16]	[16]		3.7
Al	< 5	31,100	31,200	0	< 400	17,100	17,800	4	< 7	14,500	14,500	0	20,500
As	< 21	[140]	[140]		< 1,700	< 1,700	< 1,600		< 29	[100]	[110]		96
B	[15]	[62]	70		< 330	< 320	< 330		82.6	75.6	59.6	24	52
Ba	< 1	< 1	< 1		< 66	< 64	< 65		< 1	[5.0]	[4.9]		1.3
Be	< 1	[2.7]	[2.7]		< 66	< 64	< 65		< 1	< 1	[1.2]		1.7
Bi	< 9	< 8	< 8		< 660	< 650	< 650		< 12	< 13	< 10		< 6
Ca	[38]	< 21	< 20		< 1,700	< 1,600	< 1,600		< 29	[280]	271		55
Cd	< 1	[2.1]	[2.1]		< 99	< 97	< 98		< 2	[6.7]	[6.5]		2.2
Co	< 4	< 4	< 4		< 330	< 320	< 330		< 6	< 6	< 5		< 3
Cr	< 2	94	94	0	< 130	[830]	[620]		< 2	400	388	3	118
Cu	< 2	< 2	[3.4]		< 170	< 160	< 160		< 3	[4.1]	[4.4]		2.6
Fe	[4.1]	[7.7]	[7.3]		< 170	[1,500]	[740]		< 3	281	271	4	50
K	< 170	34,800	34,900	0	n/a	n/a	n/a		< 240	44,300	45,900	4	27,200
Li	< 3	< 3	< 2		< 200	< 190	< 200		< 4	< 4	< 3		< 2
Mg	< 9	< 8	< 8		< 660	< 650	< 650		< 12	[65]	[65]		15
Mn	< 4	< 4	< 4		< 330	[460]	[480]		< 6	267	256	4	46
Mo	< 4	< 4	< 4		< 330	< 320	< 330		< 6	< 6	< 5		< 3
Na	[25]	246,000	249,000	1	< 990	192,000	199,000	4	[94]	183,000	183,000	0	173,000
Ni	< 3	[6.1]	[6.1]		n/a	n/a	n/a		< 4	50.2	48.6	3	12
P	< 9	210	220	1	< 660	[1,300]	[1,500]		< 12	1,310	1,280	2	335
Pb	< 9	[73]	[74]		< 660	< 650	< 540		< 12	[38]	[40]		47
Pd	< 64	< 63	< 61		< 5,000	< 4,800	< 4,900		< 88	< 94	< 77		< 47
Pt (ICP/MS)	< 0.05	< 0.07	< 0.07		< 0.2	< 0.2	< 0.2		n/a	n/a	n/a		< 0.07
Rh	< 26	< 25	< 24		< 2,000	< 1,900	< 2,000		< 35	< 38	< 31		< 19
Sb	< 43	< 42	< 41		< 3,300	< 3,200	< 3,300		< 59	< 63	< 51		< 31
Se	< 21	< 21	< 20		< 1,700	< 1,600	< 1,600		< 29	< 31	< 26		< 16
Si	< 43	[83]	[93]		< 3,300	< 3,200	< 3,300		[140]	[380]	[320]		99
Sn	< 130	< 130	< 120		< 9,900	< 9,700	< 9,800		< 180	< 190	< 150		< 94
Ta (ICP/MS)	< 0.05	< 0.07	< 0.07		< 0.2	< 0.2	< 0.2		n/a	n/	n/a		< 0.07
Tl	< 43	< 42	< 41		< 3,300	< 3,200	< 3,300		< 59	< 63	< 51		< 31
U	< 170	< 170	< 160		< 13,000	< 13,000	< 13,000		< 240	[1,000]	[1,000]		254
U (KinPhos)	0.007	3.47	3.59	3	0.45	1,180	1,050	12	n/a	n/a	n/a		193
V	< 4	< 4	< 4		< 330	< 320	< 330		< 6	< 6	< 5		< 3
W	< 170	< 170	< 163		< 13,000	< 13,000	< 13,000		< 240	< 250	< 200		125
Y	< 4	< 4	< 4		< 330	< 320	< 330		< 6	< 6	< 5		< 3
Zn	[8.0]	[13]	[12]		< 330	< 320	< 330		[9.2]	[16]	[11]		8.8
Zr	< 4	[6.8]	[8.1]		< 330	< 320	< 330		< 6	73.5	73.2	0	16
<b>Other Analytes Detected</b>													
Ce	< 17	< 17	< 16		< 1,300	< 1,300	< 1,300		< 24	< 25	< 21		< 13
La	< 4	< 4	< 4		< 330	< 320	< 330		< 6	< 6	< 5		< 3
Nd	< 9	< 8	< 8		< 660	< 650	< 650		< 12	< 13	< 10		< 6
Sr	< 1	< 1	< 1		< 99	< 97	< 98		< 2	[3.6]	[3.5]		1.3

- (1) Overall error for reported results is estimated to be within +15%; however results in brackets "[ ]" are less than the estimated quantitation level (i.e., 10-times MDL listed in Table 6.1) and error is anticipated to be greater than ±15%.
- (2) RPD only calculated when both sample and duplicate exceed estimated quantitation level.
- (3) Maximum slurry µg/g calculated using results from Tables 2.1 (weight fractions) and 2.2 (supernatant density).
- (4) Pt obtained by ICP/MS (solids results from Na<sub>2</sub>O<sub>2</sub>-NaOH-HCl fusion); Ta obtained by ICP/MS; U (KinPhos) obtained by kinetic phosphorescence.

**Table 3.2 AN-107 As Received --- ICP Metals Results**

<i>Tank Material</i>	AN-107 As Received <sup>(1)</sup>												
<i>Matrix</i>	Supernatant				Centrifuged Wet Solids								<sup>(3)</sup>
<i>Dissolution</i>	Acid Digest				KOH-KNO <sub>3</sub> Fusion				Acid Digest				<i>Max</i>
<i>Lab ID</i>	99-0648-pb	99-0649	99-0649-d	<sup>(2)</sup>	99-0650-pb	99-0651	99-0651-d	<sup>(2)</sup>	99-0650-pb	99-0651	99-0651-d	<sup>(2)</sup>	<i>Slurry</i>
<i>Bottle/Jar ID</i>	ProcBlnk	Comp C	Comp D	RPD	ProcBlnk	Comp C	Comp G	RPD	ProcBlnk	Comp C	Comp D	RPD	<i>Conc.</i>
<i>Units</i>	µg/mL	µg/mL	µg/mL	(%)	µg/g	µg/g	µg/g	(%)	µg/g	µg/g	µg/g	(%)	µg/g
<b>RNFL List <sup>(4)</sup></b>													
<b>Ag</b>	< 2	< 2	< 2		< 170	< 150	< 190		< 3	< 3	< 3		1.7
<b>Al</b>	< 6	273	260	4	< 400	37,300	24,700	41	< 7	37,500	36,200	4	4,480
<b>As</b>	< 23	[112]	[111]		< 1,700	< 1,500	< 1,900		< 29	[110]	[100]		81
<b>B</b>	[25]	[47]	[48]		< 330	< 310	< 370		83	115	95	20	42
<b>Ba</b>	< 1	[6.4]	[6.2]		< 66	< 62	< 74		< 1	29	28	2	7.2
<b>Be</b>	< 1	< 1	< 1		< 66	< 62	< 74		< 1	< 1	< 1		0.7
<b>Bi</b>	< 9	< 9	< 9		< 660	< 620	< 740		< 12	< 12	< 12		< 7
<b>Ca</b>	< 23	595	578	3	< 1,700	< 1,500	[3,300]		< 29	587	568	3	438
<b>Cd</b>	< 1	67	65		< 99	< 92	< 110		< 2	33	34	2	45
<b>Co</b>	< 5	< 5	< 4		< 330	< 310	< 370		< 6	< 6	< 6		< 3
<b>Cr</b>	< 2	173	169	2	< 130	[720]	[520]		< 2	743	722		193
<b>Cu</b>	< 2	[31]	[30]		< 170	< 150	< 190		< 3	[18]	[17]		21
<b>Fe</b>	< 2	1,800	1,760	2	< 170	5,530	3,540	44	< 3	4,990	4,850	3	1,760
<b>K</b>	< 190	[1700]	[1560]		n/a	n/a	n/a		< 240	[660]	[650]		1,050
<b>Li</b>	< 3	< 3	< 3		< 200	< 190	< 220		< 4	< 4	< 4		< 2
<b>Mg</b>	< 9	< 9	< 9		< 660	< 620	< 740		< 12	[23]	[21]		8.0
<b>Mn</b>	< 5	628	612	2	< 330	[690]	[650]		< 6	611	600	2	461
<b>Mo</b>	< 5	< 5	< 4		< 330	< 310	< 370		< 6	< 6	< 6		< 3
<b>Na</b>	[31]	210,000	205,000	2	< 990	159,000	164,000	3	[94]	169,000	169,000	0	150,000
<b>Ni</b>	< 3	569	557	2	n/a	n/a	n/a		< 4	283	282	0	387
<b>P</b>	< 9	473	459	3	< 660	[1,700]	[1,400]		< 12	1,830	1,760	4	505
<b>Pb</b>	< 9	409	398	2	< 660	< 620	< 740		< 12	331	322	3	293
<b>Pd</b>	< 70	< 68	< 67		< 5,000	< 4,600	< 5,500		< 88	[100]	[94]		< 53
<b>Pt (ICP/MS)</b>	< 0.05	< 0.07	< 0.07		< 0.2	< 0.2	< 0.2		n/a	n/a	n/a		< 0.07
<b>Rh</b>	< 28	< 27	< 27		< 2,000	< 1,800	< 2,200		< 35	< 36	< 37		< 21
<b>Sb</b>	< 47	< 45	< 45		< 3,300	< 3,100	< 3,700		< 59	< 59	< 62		< 35
<b>Se</b>	< 23	< 23	< 22		< 1,700	< 1,500	< 1,900		< 29	< 30	< 31		< 17
<b>Si</b>	[55]	< 45	< 45		< 3,300	< 3,100	< 3,700		[140]	[180]	[140]		44
<b>Sn</b>	< 140	< 140	< 140		< 9,900	< 9,200	< 11,000		< 180	< 180	< 190		< 104
<b>Ta (ICP/MS)</b>	< 0.05	[0.18]	[0.18]		< 0.2	< 0.2	[0.3]		n/a	n/a	n/a		0.15
<b>Tl</b>	< 47	< 45	< 45		< 3,300	< 3,100	< 3,700		< 59	< 59	< 62		< 35
<b>U</b>	< 190	< 180	< 180		< 13,000	< 12,000	< 15,000		< 240	< 240	< 250		139
<b>U (KinPhos)</b>	0.017	108	102	6	0.45	83.8	86.8	4	n/a	n/a	n/a		77.2
<b>V</b>	< 5	< 5	< 4		< 330	< 310	< 370		< 6	< 6	< 6		< 3
<b>W</b>	< 190	< 180	< 180		< 13,000	< 12,000	< 15,000		< 240	< 240	< 250		139
<b>Y</b>	< 5	[9]	[8]		< 330	< 310	< 370		< 6	64	[61]		< 13
<b>Zn</b>	[5.8]	[28]	[28]		< 330	< 310	< 370		[9.2]	[56]	[55]		24
<b>Zr</b>	< 5	[34]	[33]		< 330	< 310	< 370		< 6	136	134	1	36
<b>Other Analytes Detected</b>													
<b>Ce</b>	< 19	[57]	[54]		< 1,300	< 1,200	< 1,500		< 24	[44]	[42]		< 38
<b>La</b>	< 5	[38]	[37]		< 330	< 310	< 370		< 6	[30]	[29]		< 26
<b>Nd</b>	< 9	[110]	[110]		< 660	< 620	< 740		< 12	[120]	[110]		< 79
<b>Sr</b>	< 1	[3.0]	[3.0]		< 99	< 92	< 110		< 2	[9.0]	[8.7]		2.9

(1) Overall error for reported results is estimated to be within ±15%; however results in brackets "[]" are less than the estimated quantitation level (i.e., 10-times MDL listed in Table 6.1) and error is anticipated to be greater than ±15%.

(2) RPD only calculated when both sample and duplicate exceed estimated quantitation level.

(3) Maximum slurry µg/g calculated using results from Tables 2.1 (weight fractions) and 2.2 (supernatant density)

(4) Pt obtained by ICP/MS (solids results from Na<sub>2</sub>O<sub>2</sub>-NaOH-HCl fusion); Ta obtained by ICP/MS; U (KinPhos) obtained by kinetic phosphorescence.



Table 3.3 AW-101 &amp; AN-107 As Received --- Radiochemical Results

Tank Material	AW-101 As Received														
Matrix/Dissolution	Supernatant -- Acid Digest							Centrifuged Wet Solids -- KOH-KNO3 Fusion							Max
Lab ID	99-0644-pb		99-0648		99-0648-d		(1)	99-0650-pb		99-0650		99-0650-d		(1)	Slurry
Bottle/Jar ID	ProcBlink		Comp B		Comp B		RPD	ProcBlink		Comp B		Comp B		RPD	Conc.
Units (%Error ±1σ)	μCi/mL	%Err	μCi/mL	%Err	μCi/mL	%Err	(%)	μCi/g	%Err	μCi/g	%Err	μCi/g	%Err	(%)	μCi/g
H-3	1.83E-4	9	6.75E-3	2	6.62E-3	2	2	1.07E-4	14	8.98E-3	3	8.32E-3	3	8	5.27E-3
C-14	TBD		TBD		TBD			TBD		TBD		TBD			TBD
Co-60 (GEA)	<2.E-4		<1.E-2		<1.E-2			<2.E-3		<5.E-2		<6.E-2			< 2.E-2
Se-79 (2)	n/a		1.68E-4	5	1.78E-4	5	6	< 4.E-5		< 4.E-4		< 2.E-4			1.30E-4
Sr-90	< 3.E-3		< 6.E-1		< 4.E-1			7.58E-2	5	3.47E+1	4	3.17E+1	5	9	5.87E+0
Nb-94 (GEA)	<2.E-4		<1.E-2		<1.E-2			<2.E-3		<5.E-2		<5.E-2			< 2.E-2
Ru-106/Rh-106 (GEA)	<2.E-3		<1.E+0		<1.E+0			<2.E-2		<2.E0		<2.E0			< 1.E+0
Sb-125 (GEA)	<5.E-4		<9.E-1		<9.E-1			<5.E-3		<9.E-1		<9.E-1			< 6.E-1
Sn-126 (GEA)	<2.E-4		<3.E-1		<3.E-1			<2.E-3		<9.E-2		<1.E-1			< 2.E-1
Cs-134 (GEA)	<2.E-4		8.79E-2	12	9.36E-2	12		<2.E-3		<7.E-2		<7.E-2			6.42E-2
Cs-137 (GEA)	3.44E-4	27	4.30E+2	2	4.33E+2	2	1	5.57E-2	3	2.21E+2	2	2.23E+2	2	1	2.81E+2
Eu-154 (GEA)	<4.E-4		<4.E-2		<4.E-2			<4.E-3		<2.E-1		<2.E-1			< 6.E-2
Eu-155 (GEA)	<5.E-4		<4.E-1		<4.E-1			<5.E-3		<6.E-1		<6.E-1			< 3.E-1
Pu-238	5.53E-6	21	5.37E-5	17	4.53E-5	26		2.20E-4	8	7.63E-3	6	5.66E-3	6	30	1.27E-3
Pu-239+Pu-240	2.97E-6	31	5.12E-4	6	5.16E-4	6	1	1.48E-4	10	5.59E-2	4	5.62E-2	4	1	9.40E-3
Pu-241	< 3.E-4		2.51E-3	7	1.99E-3	8	23	2.72E-3	22	2.79E-1	5	2.83E-1	4	1	4.73E-2
Am-241(GEA)	<4.E-4		<4.E-1		<4.E-1			<6.E-3		<7.E-1		<7.E-1			< 4.E-1
Am-241 (AEA)	4.83E-6	18	2.63E-4	11	2.27E-4	15		2.77E-4	7	4.66E-2	5	4.42E-2	5	5	7.70E-3
Cm-242	< 3.E-7		< 4.E-6		< 4.E-6			< 2.E-6		< 6.E-5		< 6.E-5			< 1.E-5
Cm-243+Cm-244	2.51E-6	25	< 9.E-6		< 1.E-5			9.67E-5	11	1.14E-3	17	6.35E-4	22		1.90E-4
Beta	1.02E-2	3	4.66E+2	3	4.63E+2	3	1	1.86E-1	5	2.66E+2	4	2.41E+2	4	10	3.06E+2
Alpha	<7.E-5		< 1.E-2		< 1.E-2			< 2.E-3		7.70E-2	24	4.51E-2	28		1.92E-2
Alpha Sum (3)	1.58E-5	12	8.28E-4	5	7.89E-4	6	5	7.42E-4	4	1.11E-1	3	1.07E-1	4	4	1.85E-2

Tank Material	AN-107 As Received														
Matrix/Dissolution	Supernatant -- Acid Digest							Centrifuged Wet Solids -- KOH-KNO3 Fusion							Max
Lab ID	99-0648-pb		99-0649		99-0649-d		(1)	99-0650-pb		99-0651		99-0651-d		(1)	Slurry
Bottle/Jar ID	ProcBlink		Comp C		Comp D		RPD	ProcBlink		Comp C		Comp G		RPD	Conc.
Units (%Error ±1σ)	μCi/mL	%Err	μCi/mL	%Err	μCi/mL	%Err	(%)	μCi/g	%Err	μCi/g	%Err	μCi/g	%Err	(%)	μCi/g
H-3	1.65E-4	10	2.03E-3	4	1.78E-3	4	13	1.07E-4	14	1.17E-2	3	9.28E-3	3	23	2.61E-3
C-14	TBD		TBD		TBD			TBD		TBD		TBD			TBD
Co-60 (GEA)	<2.E-4		1.37E-1	7	1.45E-1	7	6	<2.E-3		1.02E-1	14	8.87E-2	20		1.02E-1
Se-79 (2)	n/a		6.60E-4	5	6.72E-4	5	2	< 4.E-5		< 3.E-4		< 4.E-4			4.49E-4
Sr-90	1.28E-2	21	9.61E+1	5	8.62E+1	5	11	7.58E-2	5	2.25E+2	4	1.98E+2	4	13	8.58E+1
Nb-94 (GEA)	<2.E-4		<4.E-2		<4.E-2			<2.E-3		<6.E-2		<7.E-2			< 3.E-2
Ru-106/Rh-106 (GEA)	<2.E-3		<1.E+0		<1.E+0			<2.E-2		<1.E0		<2.E0			< 9.E-1
Sb-125 (GEA)	<6.E-4		<7.E-1		<7.E-1			<5.E-3		<7.E-1		<8.E-1			< 5.E-1
Sn-126 (GEA)	<2.E-4		<3.E-1		<3.E-1			<2.E-3		<9.E-2		<1.E-1			< 2.E-1
Cs-134 (GEA)	<2.E-4		<3.E-2		<3.E-2			<2.E-3		<5.E-2		<6.E-2			< 2.E-2
Cs-137 (GEA)	6.58E-4	13	3.65E+2	2	3.55E+2	2	3	5.57E-2		1.82E+2	2	2.16E+2	2	17	2.52E+2
Eu-154 (GEA)	<5.E-4		7.71E-1	4	7.40E-1	4	4	<4.E-3		1.33E+0	4	1.07E+0	6	22	6.34E-1
Eu-155 (GEA)	<5.E-4		5.85E-1	15	4.97E-1	16		<5.E-3		8.70E-1	13	7.19E-1	19		4.65E-1
Pu-238	5.37E-6	15	1.31E-2	8	1.40E-2	8	7	2.20E-4	8	1.75E-2	7	1.79E-2	8	2	1.98E-2
Pu-239+Pu-240	1.89E-6	29	5.08E-2	5	4.46E-2	5	13	1.48E-4	10	7.51E-2	5	5.98E-2	5	23	4.03E-2
Pu-241	< 7.E-5		1.07E-1	6	1.07E-1	6	0	2.72E-3	22	1.80E-1	6	1.40E-1	6	25	8.73E-2
Am-241(GEA)	<5.E-4		9.12E-1	17	6.87E-1	22		<6.E-3		7.22E-1	30	4.33E-1	50		6.51E-1
Am-241 (AEA)	6.92E-6	19	6.39E-1	5	5.98E-1	5	7	2.77E-4	7	5.40E-1	5	5.36E-1	5	1	4.60E-1
Cm-242	< 9.E-7		1.17E-3	34	2.97E-3	25		< 2.E-6		9.38E-4	40	1.45E-3	36		2.02E-3
Cm-243+Cm-244	7.56E-6	18	1.52E-2	11	1.43E-2	12		9.67E-5	11	1.22E-2	11	9.75E-3	14		1.09E-2
Beta	3.40E-2	3	5.86E+2	3	5.44E+2	3	8	1.86E-1	5	5.81E+2	4	5.52E+2	4	5	4.32E+2
Alpha	< 2.E-4		7.60E-1	3	7.50E-1	3	1	< 2.E-3		5.72E-1	6	3.98E-1	8	36	5.39E-1
Alpha Sum (3)	2.17E-5	10	7.19E-1	4	6.74E-1	4	7	7.42E-4	4	6.46E-1	4	6.25E-1	4	3	5.23E-1

(1) RPD only calculated when both sample and duplicate have error uncertainties &lt;10%.

(2) Se-79 performed directly on supernatant material, not on acid digestion preparation.

(3) Alpha Sum equals the μCi/mL or μCi/g summation of Pu-238, Pu-239+240, Am-241, Cm-242, and Cm-243+244.

**Table 3.4 AW-101 & AN-107 As Received --- ICP/MS Radionuclides in Microcuries**

Tank Material	AW-101 As Received <sup>(1)</sup>																			
	Supernatant										Centrifuged Wet Solids									
	Acid Digest					Water Dilution					KOH fusion					PGM Fusion				
	99-0644-pb	99-0648	99-0648-d	RPD	99-0644-pb	99-0648	99-0648-d	RPD	99-0644-pb	99-0648	99-0648-d	RPD	99-0650-pb	99-0650-d	99-0650-pb	99-0650-d	99-0650-pb	99-0650-d	99-0650-pb	99-0650-d
<b>Matrix</b>	ProcBlnk	Comp B	Comp B	RPD	ProcBlnk	Comp B	Comp B	RPD	ProcBlnk	Comp B	Comp B	RPD	ProcBlnk	Comp B	Comp B	RPD	ProcBlnk	Comp B	Comp B	RPD
<b>Lab ID</b>	μCi/mL	μCi/mL	μCi/mL	(%)	μCi/mL	μCi/mL	μCi/mL	(%)	μCi/mL	μCi/mL	μCi/mL	(%)	μCi/g	μCi/g	μCi/g	μCi/g	μCi/g	μCi/g	μCi/g	μCi/g
<b>Bottle/Jar ID</b>																				
<b>Units</b>																				
<b>Tc-99</b>	< 3.E-4	1.91E-1	1.92E-1	1	< 3.E-4	1.49E-1	1.59E-1	6	< 2.E-3	1.56E-1	1.71E-1	10	[1.7E-3]	1.12E-1	1.26E-1	11	< 7.E-5	< 7.E-5	< 7.E-5	[6.8E-4]
<b>I-129</b>	< 2.E-5	[6.8E-4]	[1.2E-3]		3.18E-5	3.70E-4	4.41E-4	17	< 7.E-5	< 7.E-5	< 7.E-5		< 7.E-5	< 7.E-5	< 7.E-5		[9.6E-4]	< 1.E-3	< 1.E-3	< 5.E-4
<b>U-233</b>	< 5.E-4	< 7.E-4	< 7.E-4		< 5.E-4	< 7.E-4	< 7.E-4		< 1.E-3	< 1.E-3	< 1.E-3		< 1.E-3	< 1.E-3	< 1.E-3		< 1.E-3	< 1.E-3	< 1.E-3	< 3.E-4
<b>U-234<sup>(2)</sup></b>	< 1.E-4	< 2.E-4	< 2.E-4		< 1.E-4	< 2.E-4	< 2.E-4		< 1.E-3	< 1.E-3	< 1.E-3		< 1.E-3	< 1.E-3	< 1.E-3		< 1.E-3	< 1.E-3	< 1.E-3	< 3.E-4
<b>U-235<sup>(2)</sup></b>	< 4.E-8	< 6.E-8	[1.9E-7]		< 4.E-8	< 6.E-8	< 6.E-8		< 4.E-7	2.72E-5	2.76E-5	2	< 4.E-7	1.37E-5	1.91E-5	33	< 4.E-7	1.37E-5	1.91E-5	4.59E-6
<b>U-236<sup>(2)</sup></b>	< 1.E-6	< 2.E-6	< 1.E-6		< 1.E-6	< 2.E-6	< 2.E-6		< 1.E-5	[5.6E-5]	6.92E-5		< 1.E-5	[2.6E-5]	[2.6E-5]		< 1.E-5	[2.6E-5]	[2.6E-5]	1.19E-5
<b>U-238</b>	< 7.E-8	1.46E-6	1.44E-6	1	< 2.E-8	1.05E-6	9.87E-7	6	[2.7E-7]	4.07E-4	3.96E-4	3	2.44E-6	1.80E-4	2.81E-4	44	< 7.E-5	[7.0E-5]	[8.5E-5]	[2.6E-5]
<b>Np-237</b>	< 1.E-5	< 2.E-5	< 2.E-5		< 1.E-5	< 2.E-5	< 2.E-5		< 6.E-3	5.75E-2	4.84E-2	17	< 6.E-3	2.95E-2	3.50E-2	17	< 6.E-3	2.95E-2	3.50E-2	1.04E-2
<b>Pu-239</b>	< 1.E-3	< 2.E-3	< 2.E-3		< 1.E-3	< 2.E-3	< 2.E-3		< 2.E-2	< 2.E-2	< 2.E-2		< 2.E-2	< 2.E-2	< 2.E-2		< 2.E-2	< 2.E-2	< 2.E-2	< 8.E-3
<b>Pu-240<sup>(2)</sup></b>	< 5.E-3	< 7.E-3	< 7.E-3		< 5.E-3	< 7.E-3	< 7.E-3													
Tank Material	AN-107 As Received <sup>(1)</sup>																			
	Supernatant										Centrifuged Wet Solids									
	Acid Digest					Water Dilution					KOH Fusion					PGM Fusion				
	99-0648-pb	99-0649	99-0649-d	RPD	99-0648-pb	99-0649	99-0649-d	RPD	99-0648-pb	99-0649	99-0649-d	RPD	99-0650-pb	99-0651-d	99-0651-d	99-0650-pb	99-0651-d	99-0651-d	99-0650-pb	99-0651-d
<b>Matrix</b>	ProcBlnk	Comp C	Comp D	RPD	ProcBlnk	Comp C	Comp D	RPD	ProcBlnk	Comp C	Comp C	RPD	ProcBlnk	Comp G	Comp G	ProcBlnk	Comp C	Comp G	Comp G	RPD
<b>Lab ID</b>	μCi/mL	μCi/mL	μCi/mL	(%)	μCi/mL	μCi/mL	μCi/mL	(%)	μCi/mL	μCi/mL	μCi/mL	(%)	μCi/g	μCi/g	μCi/g	μCi/g	μCi/g	μCi/g	μCi/g	μCi/g
<b>Bottle/Jar ID</b>																				
<b>Units</b>																				
<b>Tc-99</b>	< 3.E-4	1.28E-1	1.29E-1	0	< 3.E-4	1.09E-1	1.05E-1	3	< 2.E-3	7.70E-2	1.02E-1	28	[1.7E-3]	6.11E-2	5.67E-2	7	< 7.E-5	< 7.E-5	< 7.E-5	[5.2E-4]
<b>I-129</b>	< 2.E-5	[8.3E-4]	[7.0E-4]		< 2.E-5	2.00E-4	1.94E-4	3	< 7.E-5	< 7.E-5	< 7.E-5		< 7.E-5	< 7.E-5	< 7.E-5		[9.6E-4]	< 1.E-3	< 1.E-3	< 5.E-4
<b>U-233</b>	< 5.E-4	< 7.E-4	< 7.E-4		< 5.E-4	< 7.E-4	< 7.E-4		< 1.E-3	< 1.E-3	< 1.E-3		< 1.E-3	< 1.E-3	< 1.E-3		< 1.E-3	< 1.E-3	< 1.E-3	< 3.E-4
<b>U-234<sup>(2)</sup></b>	< 1.E-4	< 2.E-4	< 2.E-4		< 1.E-4	< 2.E-4	< 2.E-4		< 1.E-3	< 1.E-3	< 1.E-3		< 1.E-3	< 1.E-3	< 1.E-3		< 1.E-3	< 1.E-3	< 1.E-3	< 3.E-4
<b>U-235<sup>(2)</sup></b>	< 4.E-8	[3.1E-6]	[3.1E-6]		< 4.E-8	2.65E-6	2.47E-6	7	< 4.E-7	1.67E-6	[1.6E-6]		< 4.E-7	[1.3E-6]	[1.5E-6]		< 4.E-7	[1.3E-6]	[1.5E-6]	2.10E-6
<b>U-236<sup>(2)</sup></b>	< 1.E-6	< 2.E-6	< 2.E-6		< 1.E-6	< 2.E-6	< 2.E-6		< 1.E-5	< 1.E-5	< 1.E-5		< 1.E-5	< 1.E-5	< 1.E-5		< 1.E-5	< 1.E-5	< 1.E-5	< 3.E-6
<b>U-238</b>	< 7.E-8	3.97E-5	3.83E-5	4	[4.3E-8]	5.06E-5	4.87E-5	4	[2.7E-7]	3.25E-5	3.28E-5	1	2.44E-6	3.27E-5	3.24E-5	1	< 7.E-5	[2.1E-4]	[2.1E-4]	[6.2E-5]
<b>Np-237</b>	< 1.E-5	[6.0E-5]	[6.0E-5]		< 1.E-5	[3.7E-5]	4.20E-5		< 7.E-5	[1.4E-4]	[1.4E-4]		< 7.E-5	[2.1E-4]	[2.1E-4]		< 7.E-5	[2.1E-4]	[2.1E-4]	5.07E-2
<b>Pu-239</b>	< 1.E-3	5.98E-2	6.49E-2	8	< 1.E-3	4.14E-2	4.08E-2	2	< 6.E-3	[6.2E-2]	[6.8E-2]		< 6.E-3	8.94E-2	7.88E-2	13	< 6.E-3	8.94E-2	7.88E-2	[2.1E-2]
<b>Pu-240<sup>(2)</sup></b>	< 5.E-3	[2.9E-2]	[2.3E-2]		< 5.E-3	[9.7E-3]	7.41E-3		< 2.E-2	< 2.E-2	< 2.E-2		< 2.E-2	< 2.E-2	< 2.E-2		< 2.E-2	< 2.E-2	< 2.E-2	< 2.E-2

(1) The overall uncertainty for reported results is estimated at ±10% (±15% for I-129); results in brackets "[]" are less than the estimated quantitation limit and have higher uncertainties.

(2) No standard Available; Results calculated from instrument response from different isotope.

**Table 3.5 AW-101 & AN-107 As Received --- ICP/MS Radionuclides in Micrograms**

Tank Material	AW-101 As Received <sup>(1)</sup>																			
	Supernatant										Centrifuged Wet Solids									
	Acid Digest					Water Dilution					KOH fusion					PGM Fusion				
	99-0644-pb	99-0648	99-0648-d	99-0648-d	99-0648-d	99-0644-pb	99-0648	99-0648	99-0648-d	99-0648-d	99-0650-pb	99-0650	99-0650-d	99-0650-d	99-0650-d	99-0650-pb	99-0650	99-0650	99-0650-d	Max Slurry Conc. $\mu\text{Ci/g}$
Matrix	ProcBlink	Comp B	Comp B	RPD	RPD	ProcBlink	Comp B	Comp B	Comp B	RPD	ProcBlink	Comp B	Comp B	Comp B	RPD	ProcBlink	Comp B	Comp B	RPD	
Bottle/Jar ID	$\mu\text{g/mL}$	$\mu\text{g/mL}$	$\mu\text{g/mL}$	(%)	(%)	$\mu\text{g/mL}$	$\mu\text{g/mL}$	$\mu\text{g/mL}$	$\mu\text{g/mL}$	(%)	$\mu\text{g/g}$	$\mu\text{g/g}$	$\mu\text{g/g}$	$\mu\text{g/g}$	(%)	$\mu\text{g/g}$	$\mu\text{g/g}$	$\mu\text{g/g}$	(%)	
Units																				
Tc-99	< 0.02	11.2	11.3	1	1	< 0.02	8.79	2.09	2.49	6	< 0.10	9.17	10.1	10	10	[0.10]	6.6	7.4	11	8.02
I-129	< 0.10	[3.86]	[6.68]			0.18	2.09	2.49	2.49	17	< 0.40	< 0.40	< 0.40	< 0.40	< 0.40	< 0.40	< 0.40	< 0.40	< 0.40	[3.84]
U-233	< 0.05	< 0.07	< 0.07			< 0.05	< 0.07	< 0.07	< 0.07		< 0.10	< 0.10	< 0.10	< 0.10	< 0.10	[0.10]	< 0.10	< 0.10	< 0.10	< 0.06
U-234 <sup>(2)</sup>	< 0.02	< 0.03	< 0.03			< 0.02	< 0.03	< 0.03	< 0.03		< 0.20	< 0.20	< 0.20	< 0.20	< 0.20	< 0.20	< 0.20	< 0.20	< 0.20	< 0.05
U-235 <sup>(2)</sup>	< 0.02	< 0.03	[0.09]			< 0.02	< 0.03	< 0.03	< 0.03		< 0.20	12.6	12.8	2	2	< 0.20	6.34	8.85	33	2.12
U-236 <sup>(2)</sup>	< 0.02	< 0.03	< 0.02			< 0.02	< 0.03	< 0.03	< 0.03		< 0.20	[0.87]	1.07			< 0.20	[0.40]	[0.40]		0.18
U-238	< 0.20	4.35	4.30	1	1	< 0.05	3.13	2.94	2.94	6	[0.80]	12.10	11.80	3	3	7.25	536	836	44	198
Np-237	< 0.02	< 0.03	< 0.03			< 0.02	< 0.03	< 0.03	< 0.03		< 0.10	< 0.10	< 0.10	< 0.10	< 0.10	< 0.10	[0.12]	[0.12]		[0.04]
Pu-239	< 0.02	< 0.03	< 0.03			< 0.02	< 0.03	< 0.03	< 0.03		< 0.10	0.93	0.78	17	17	< 0.10	0.48	0.56	17	0.17
Pu-240 <sup>(2)</sup>	< 0.02	< 0.03	< 0.03			< 0.02	< 0.03	< 0.03	< 0.03		< 0.10	< 0.10	< 0.10	< 0.10	< 0.10	< 0.10	< 0.10	< 0.10	< 0.10	< 0.03

Tank Material	AN-107 As Received <sup>(1)</sup>																			
	Supernatant										Centrifuged Wet Solids									
	Acid Digest					Water Dilution					KOH Fusion					PGM Fusion				
	99-0644-pb	99-0649	99-0649-d	99-0649-d	99-0649-d	99-0644-pb	99-0649	99-0649	99-0649-d	99-0649-d	99-0650-pb	99-0651	99-0651-d	99-0651-d	99-0651-d	99-0650-pb	99-0651	99-0651	99-0651-d	Max Slurry Conc. $\mu\text{Ci/g}$
Matrix	ProcBlink	Comp C	Comp C	RPD	RPD	ProcBlink	Comp C	Comp C	Comp C	RPD	ProcBlink	Comp C	Comp C	Comp C	RPD	ProcBlink	Comp C	Comp C	RPD	
Bottle/Jar ID	$\mu\text{g/mL}$	$\mu\text{g/mL}$	$\mu\text{g/mL}$	(%)	(%)	$\mu\text{g/mL}$	$\mu\text{g/mL}$	$\mu\text{g/mL}$	$\mu\text{g/mL}$	(%)	$\mu\text{g/g}$	$\mu\text{g/g}$	$\mu\text{g/g}$	$\mu\text{g/g}$	(%)	$\mu\text{g/g}$	$\mu\text{g/g}$	$\mu\text{g/g}$	(%)	
Units																				
Tc-99	< 0.02	7.55	7.58	0	0	< 0.02	6.40	6.19	6.19	3	< 0.10	4.54	6.04	28	28	[0.10]	3.6	3.34	7	5.42
I-129	< 0.10	[4.69]	[3.98]			< 0.10	1.13	1.10	1.10	3	< 0.40	< 0.40	< 0.40	< 0.40	< 0.40	< 0.40	< 0.40	< 0.40	< 0.40	[2.97]
U-233	< 0.05	< 0.07	< 0.07			< 0.05	< 0.07	< 0.07	< 0.07		< 0.10	< 0.10	< 0.10	< 0.10	< 0.10	[0.10]	< 0.10	< 0.10	< 0.10	< 0.06
U-234 <sup>(2)</sup>	< 0.02	< 0.03	< 0.03			< 0.02	< 0.03	< 0.03	< 0.03		< 0.20	< 0.20	< 0.20	< 0.20	< 0.20	< 0.20	< 0.20	< 0.20	< 0.20	< 0.04
U-235 <sup>(2)</sup>	< 0.02	[1.42]	[1.42]			< 0.02	1.23	1.14	1.14	7	< 0.20	0.78	[0.73]			< 0.20	[0.60]	[0.70]		0.97
U-236 <sup>(2)</sup>	< 0.02	< 0.03	< 0.03			< 0.02	< 0.03	< 0.03	< 0.03		< 0.20	< 0.20	< 0.20	< 0.20	< 0.20	< 0.20	< 0.20	< 0.20	< 0.20	< 0.04
U-238	< 0.20	118	114	4	4	[0.13]	151	145	145	4	[0.80]	96.8	97.6	1	1	7.25	97.2	96.3	1	105
Np-237	< 0.02	[0.09]	[0.09]			< 0.02	[0.05]	0.06	0.06		< 0.10	[0.20]	[0.20]			< 0.10	[0.30]	[0.30]		[0.09]
Pu-239	< 0.02	0.96	1.05	8	8	< 0.02	0.67	0.66	0.66	2	< 0.10	[1.00]	[1.10]			< 0.10	1.44	1.27	13	0.82
Pu-240 <sup>(2)</sup>	< 0.02	[0.13]	[0.10]			< 0.02	[0.04]	0.03	0.03		< 0.10	< 0.10	< 0.10	< 0.10	< 0.10	< 0.10	< 0.10	< 0.10	< 0.10	[0.09]

(1) The overall uncertainty for reported results is estimated at  $\pm 10\%$  ( $\pm 15\%$  for I-129); results in brackets “[ ]” are less than the estimated quantitation limit and have higher uncertainties.  
(2) No standard Available; Results calculated from instrument response from different isotopes.

Table 3.6 AW-101 &amp; AN-107 As Received --- Other Results

Tank Material Matrix Lab ID Bottle/Jar ID Units	AW-101 As Received										Max. Slurry Conc. µg/g
	Supernatant					Centrifuged Wet Solids					
	Type of	99-0644-pb	99-0648	99-0648-d	(1)	Type of	99-0650-pb	99-0650	99-0650-d	(1)	
		ProcBlnk	Comp B	Comp B	RPD		ProcBlnk	Comp B	Comp B	RPD	
		µg/mL	µg/mL	µg/mL	(%)		µg/g	µg/g	µg/g	(%)	
TIC	Persulfate	76	2 020	1 670	19	Persulfate	n/a	14 900	13 900	7	3 600
TOC	Persulfate	< 85	3,320	2,850	15	Persulfate	n/a	4,650	4,620	1	2,600
TC (sum)		76	5,340	4,520	17		na/	19,600	18,500	6	6,200
Fluoride	Dir./Dil.	< 17	1,800	1,700	6	Water Leach	< 120	3,300	3,400	0	1,600
Chloride	Dir./Dil.	< 17	6,000	5,700	4	Water Leach	< 120	3,700	3,300	9	4,000
Nitrite	Dir./Dil.	< 35	106,000	108,000	1	Water Leach	< 250	61,000	55,700	9	70,900
Bromide	Dir./Dil.	< 17	< 480	< 480		Water Leach	< 120	< 1300	< 1300		< 480
Nitrate	Dir./Dil.	< 35	163,000	167,000	3	Water Leach	< 250	138,000	171,000	22	122,000
Phosphate	Dir./Dil.	< 35	< 990	< 990		Water Leach	< 250	6,800	7,600	11	1,800
Sulfate	Dir./Dil.	< 35	< 990	< 990		Water Leach	< 250	7,400	7,100	4	1,800
Oxalate	Dir./Dil.	< 34	< 990	< 990		Water Leach	< 250	12,600	12,700	1	2,600
Mercury	Acid Digest	TBD	TBD	TBD		Acid Digest	TBD	TBD	TBD		TBD
Ammonia	Dir./Dil.	5	190	180	5	Water Leach	11	21	21		110
Cyanide (total)	Distillation	TBD	TBD	TBD		Distillation	TBD	TBD	TBD		TBD
		°F	°F	°F							n/a
Flashpoint	Direct	n/m	TBD	TBD							n/a
		mmole/mL	mmole/mL	mmole/mL							n/a
Hydroxide	Dir./Dil.	< 0.01 (2)	5.30	5.34	1						n/a
		pH	pH	pH							n/a
pH	Direct	n/m	13.44	13.38	0						n/a

Tank Material Matrix Lab ID Bottle/Jar ID Units	AN-107 As Received										Max. Slurry Conc. µg/g
	Supernatant					Centrifuged Wet Solids					
	Prep	99-0648-pb	99-0649	99-0649-d	(1)	Prep	99-0650-pb	99-0651	99-0651-d	(1)	
		ProcBlnk	Comp C	Comp D	RPD		ProcBlnk	Comp C	Comp D	RPD	
		µg/mL	µg/mL	µg/mL	(%)		µg/g	µg/g	µg/g	(%)	
TIC	Persulfate	73	18 100	20 100	11	Persulfate	n/a	32 600	29 600	10	16 300
TOC	Persulfate	< 85	40,200	41,900	4	Persulfate	n/a	27,500	27,400	0	29,300
TC (sum)		73	58,200	62,000	6		n/a	60,100	57,000	5	45,600
Fluoride (IC)	Dir./Dil.	< 21	8,000	8,200	2	Water Leach	< 120	4,900	5,000	2	5,700
Chloride (IC)	Dir./Dil.	< 21	1,800	1,900	10	Water Leach	< 120	< 1300	< 1100		1,300
Nitrite (IC)	Dir./Dil.	< 42	68,800	68,100	1	Water Leach	< 250	36,100	37,900	5	47,200
Bromide (IC)	Dir./Dil.	< 21	< 950	< 1000		Water Leach	< 120	< 1400	< 1200		730
Nitrate (IC)	Dir./Dil.	< 42	220,000	221,000	0	Water Leach	< 250	123,000	131,000	6	153,000
Phosphate (IC)	Dir./Dil.	< 42	4,914	5,200	6	Water Leach	< 250	6,500	5,900	10	4,000
Sulfate (IC)	Dir./Dil.	< 42	10,500	10,200	3	Water Leach	< 250	7,900	7,700	3	7,400
Oxalate (IC)	Dir./Dil.	< 41	< 1900	< 2000		Water Leach	< 240	23,100	18,300	23	3,800
Mercury	Acid Digest	TBD	TBD	TBD		Acid Digest	TBD	TBD	TBD		TBD
Ammonia	Dir./Dil.	2	79	86	11	Water Leach	11	11	13		55
Cyanide (total)	Distillation	TBD	TBD	TBD		Distillation	TBD	TBD	TBD		TBD
		°F	°F	°F							n/a
Flashpoint	Direct	n/m	TBD	TBD							n/a
		mmole/mL	mmole/mL	mmole/mL							n/a
Hydroxide	Dir./Dil.	< 0.01 (2)	< 0.01 (2)	< 0.01 (2)							n/a
		pH	pH	pH							n/a
pH (3)	Direct	n/m	10.99	11.05	0						n/a

(1) RPD only calculated when sample and duplicate results above threshold for method's RPD calculation. (Calculated prior to rounding)

(2) No titration inflection point detected; free hydroxide estimated at <0.01 mmole/mL.

(3) pH Sample and Duplicate performed from two sub-samples from bottle C for AN-107.

dir./dil = direct or dilution; n/a = not applicable; n/m = not measured due to applicability of method; TBD = to be determined

## 4.0 TCLP Metals Analysis and Evaluation

The TCLP, SW-846 Method 1311, was not performed on the AW-101 and AN-107 materials for toxic metals. The estimated TCLP metals concentrations are calculated from 1) the concentrations of the supernatant TCLP metals, 2) the concentrations of the solids TCLP metal (assuming all metals would be leached 100%), 3) the density of the supernatant, and 4) the weight percent (wt%) filtered solids performed per Method 1311. The concentrations of the TCLP metals are estimated by assuming that the supernatants and solids are analyzed separately and combined mathematically per Method 1311. Equation (2) is used to estimate the concentrations of the TCLP metals in the as received tank waste materials assuming a 100 g initial sample size for processing. The TCLP metals concentrations in the solids from the acid digestion preparations are used for the calculation. The acid digestion results are considered to be conservative since the nitric-hydrochloric acid digestion is significantly more rigorous than the TCLP acetic acid leach.

$$C = [ (L * (V_1/D_1)) + ((S * (V_2/D_2)) ] / [ (V_1/D_1) + (V_2/D_2) ] \quad (2)$$

Where: C = Waste material TCLP concentration in  $\mu\text{g/mL}$   
L = average supernatant TCLP metal concentration in  $\mu\text{g/mL}$   
S = average solids TCLP metal concentration in  $\mu\text{g/g}$   
D<sub>1</sub> = density of supernatant in g/mL  
D<sub>2</sub> = 1, the approximate density in g/mL of the TCLP extraction fluid  
V<sub>1</sub> = volume in g of liquid fraction of nominal 100 grams of waste material = (100 g) \* (1 – W/100)  
V<sub>2</sub> = volume in g of TCLP fluid to add to solids fraction of waste material for TCLP extraction = (100 g) \* (W/100) \* F  
  
F = 20, the TCLP fluid to solids extraction ratio  
W = wt% filtered solids (31.1 for AW-101, 50.8 for AN-107)

Table 4.1 provides the estimated TCLP metals results for the AW-101 and AN-107 waste material. The results indicate that both waste materials have TCLP metal concentrations that exceed the regulatory threshold, specifically arsenic, chromium, and lead. Cadmium exceeds the threshold for the AN-107 waste. Due to the dilutions required for the analyses to support ALARA (as low as reasonably achievable) radiation exposure concerns and the low regulatory threshold for selenium (i.e., 1  $\mu\text{g/mL}$ ), it can not be determined if selenium exceeds the threshold. Nickel is included in the TCLP table at the request of BNFL; however, there is no TCLP threshold associated with nickel.

The wt% filtered solids were determined per Method 1311. However, due to the difficulty in filtering the AW-101 and AN-107 as received materials, the wt% filtered solids results are considerably higher (i.e., two to five times) than the wt% centrifuged solids. Although the ICP analyses for the TCLP metal were performed on preparations using the centrifuged solids, the results of Table 4.1 use the results from the wt% filtered solids for calculating the estimated TCLP metals concentration. Since in nearly all cases the concentration of TCLP metals is higher in the solids than in the supernatants, the use of the higher wt% filtered solids provides a conservative estimate of the TCLP metals concentration.

**Table 4.1 TCLP Metals Estimated Concentrations**

TCLP Analytes	TCLP Limit µg/mL	AW-101 As Received				AN-107 As Received			
		Weight % Wet Filtered Solids = 31.1%				Weight % Wet Filtered Solids = 50.8%			
		Supernatant	Wet Solids		TCLP Conc. µg/mL	Supernatant	Wet Solids		TCLP Conc. µg/mL
		Acid Digest	Acid Digest	After Leach		Acid Digest	Acid Digest	After Leach	
		99-0648 Average µg/mL	99-0650 Average µg/g	20 : 1 Ratio Average µg/mL		99-0649 Average µg/mL	99-0651 Average µg/g	20 : 1 Ratio Average µg/mL	
Ag	5.0	< 2.1	[16]	[0.8]	0.9	< 2.0	< 3.0	< 0.2	< 0.2
As	5.0	[140]	[105]	[5.3]	14.6	[111]	[105]	[5.3]	8.8
Ba	100.0	< 0.8	[5.0]	[0.2]	0.3	[6]	28	1.4	1.6
Cd	1.0	[2.1]	[6.6]	[0.3]	0.5	66	34	1.7	3.8
Cr	5.0	94	394	19.7	24.8	171	733	36.6	41.1
Hg	0.20	TBD	TBD			TBD	TBD		
Ni	--	[6.1]	49	2.5	2.7	563	283	14.1	32.2
Pb	5.0	[73]	[39]	[2.0]	6.9	403	327	16.3	29.1
Se	1.0	< 20.7	< 20.7	< 1.0	< 2.4	< 20	< 30	< 1.5	< 2.1
* Values in [ ] are above the MDL but below the EQL and have uncertainties > 15%.									
* Shaded and boxed values exceed or potentially exceed regulatory threshold.									

## 5.0 Quality Control and Data Evaluation

General Comments: Due to a mislabeling error (reported via QPR 99-BNFL-02), the AW-101 diluted feed and AN-107 as received supernatants were processed together, and AW-101 as received and AN-107 diluted feed supernatants were processed together. This mislabeling error applied to the acid digestion, dilutions, and direct sub-sampling of each of the supernatants. The error was detected and corrected prior to the analysis of as received supernatants. Due to this error there are two batch process blanks reported: 99-0644-pb for AW-101 supernatant and 99-0648-pb for AN-107 supernatant. This causes minor confusion since the as received laboratory numbers (i.e., 99-0648 through 99-0651) do not include 99-0644 which is the laboratory number for the AW-101 diluted feed supernatant. Based on the batch processing of the samples, process blanks labeled 99-0644-pb were processed with AW-101 supernatant sample 99-0648 and process blanks labeled 99-0648-pb were processed with AN-107 supernatant sample 99-0649.

### 5.1 Metals Analysis by ICP – Tables 3.1 & 3.2

Due to the high radiological dose from the acid digestion and KOH-KNO<sub>3</sub> fusion preparations, aliquots were diluted in the SAL to reduce the radiological dose prior to transferring the samples to the ICP workstation. The acid dissolution of the AW-101 and AN-107 supernatants and the KOH-KNO<sub>3</sub> fusion dissolution and acid digestion of the AW-101 and AN-107 solids were analyzed by ICP in six analytical batches following procedure PNL-ALO-211. Where analytes are not detected, the results are reported as less than the MDL. Results present in brackets “[ ]” are less than the estimated quantitation level (EQL), and have uncertainties exceeding  $\pm 15\%$ . For the ICP, the EQL is defined as ten times the MDL. Above the EQL, results are expected to have uncertainties of less than  $\pm 15\%$ , and typically less than  $\pm 10\%$ .

Quality control for the ICP analysis consists of duplicates, process blanks, serial dilution, matrix spikes, laboratory control standards or blank spikes, post spikes, verification check standards, and high calibration standards. An evaluation of each of the quality control (QC) criteria was performed and a summary is presented below.

Duplicates: Except for a very few cases, the relative percent differences (RPD) for analytes of interest were within the acceptance criteria of 20%. Exceptions are boron (20%-24%) for AW-101 and AN-107 solids acid digestions and aluminum (41%) and iron (44%) for AN-107 solids KOH-KNO<sub>3</sub> fusion. The poor boron RPDs result from the high boron processing blank (i.e., use of boro-silicate glass for processing acid digestions) relative to the sample boron concentrations. The aluminum and iron RPDs most likely result from sample heterogeneity, since other analytes in the samples have acceptable RPDs (most notably, sodium).

Matrix Spikes: As a result of the dilutions for ALARA purposes, most matrix spike samples were diluted such that the spike concentration was below the EQL. In the cases where the matrix spikes are meaningless, post spikes are used to evaluate matrix effects. However, the matrix spikes associated with the acid digestion of AW-101 solids and AN-107 solids provided usable results. In this case, all recoveries were acceptable for analyte concentrations exceeding the EQL, except silicon recovery (i.e., 49%) for the AW-101 solids. Since acid digestion is not generally a viable dissolution method for silicon in tank waste materials, inconsistent leaching of the solids is the likely cause of the apparent poor recovery.

**Blank Spikes:** The supernatant blank spikes were subjected to the same ALARA dilution as the matrix spikes, resulting in diluting the blank spike concentration to below the EQL. The blank spike prepared with acid digestion of the AW-101 and AN-107 solids was not diluted and produced recoveries within acceptance criteria of 80% to 120% recovery, except for silver (74%). Silver has been demonstrated to be difficult to maintain in solution using the acid digestion procedure used for tank waste materials. Increasing the chloride concentration in the digestion has been used to stabilize the silver (assuming low silver concentrations). However, since neither AW-101 or AN-107 supernatants or solids appear to have any significant concentration of silver (i.e., highest measured concentration was 16 µg/g), the silver recovery is considered adequate to evaluate the validity of the sample silver concentration.

**Post Spikes:** For the acid digestion of AW-101 and AN-107 supernatants, tin failed to meet the acceptance criteria of 75% to 125%. However, based on the poor tin sensitivity and the required analytical dilutions, the tin post spike concentration falls below the tin detection limit, resulting in meaningless recovery results.

**Process Blanks:** Analytes of interest detected in the processing blanks for the acid digestions of the supernatants and solids and the KOH-KNO<sub>3</sub> fusions were below the EQL, except for boron in the acid digestions of the solids. In this case, the boron measured in the solids samples appears to be entirely from contamination from the processing blank and the actual boron results should be considered less than the MDL.

**Percent Difference (%D):** The percent difference is based on the difference in the calculate concentration of a sample when compared to the sample diluted by five-fold. Where analyte concentrations exceed the EQL, all %D results meet the acceptance criteria of ±10%, except for calcium (+80% and +55%) from the acid digestion preparation for the AN-107 solids. The calcium deviation appears to be from excess calcium in the five-fold diluted sample, possibly from contaminant introduced during the dilution process. However, for the AW-101 solids analyzed at the same time the %D was <1% for calcium, suggesting that the high %D may be matrix-related.

**Laboratory Control Standard (LCS):** A NIST SRM-2710 reference standard was processed with all fusion prepared samples as a LCS. For all analytes in the LCS above the EQL, recoveries were within acceptance criteria.

**ICP QC Check Standard and Linear Range Check Standard:** All standards provided results within acceptance criteria, except for a few analytes from the QC Check Standard. Acceptance criteria for the QC Check Standard is ±10% of true value. Silicon, rhodium, tin, and palladium typically provided results at the ±11% to ±13% range, with one tin value being at 20%. This deviation is not expected to impact the reported results for these analytes.



## 5.2 Radiochemical Analysis

### Gamma Emitters by Gamma Energy Analysis (GEA) – Table 3.3

The sample preparations from the SAL were diluted further in a laboratory, then packaged for gamma counting following procedure PNL-ALO-450. Most of the gamma emission from the samples is from Cs-137. Other detected gamma emitters are Co-60, Cs-134, Eu-154, Eu-155, and Am-241. All of these gamma emitters are at much lower concentration than the Cs-137, and not all were detected in every sample. No gamma activity was detectable for Nb-94, Ru/Rh-106, Sb-125, or Sn-126.

Two of the three SAL process blanks had detectable quantities of Cs-137, but at insignificant levels when compared to the Cs-137 levels in the samples. No other gamma emitters were detected in the blanks.

Where the counting uncertainties are less than 10% for the sample and duplicate, the RPD results are within the acceptance criteria of 20%, except for AN-107 solids Eu-154. The AN-107 solids Eu-154 RPD is considerably higher than predicted from the counting uncertainties (i.e., counting uncertainties of 4% and 6%, but an RPD of 22%), as is the AN-107 solids Cs-137 RPD (i.e., counting uncertainties of 2%, but an RPD of 17%). Poor RPDs are also evident for other analytes of interest analyzed from the same fusion preparation (e.g., some ICP analytes, Pu-239+240, Pu-241, and total alpha). Since some analytes demonstrate good precision (i.e., low RPD values), the poor precision is most likely due to sample heterogeneity or inability to obtain a representative sample (i.e., fusion preparations typically use less than 0.2 g of material).

Since the Am-241 activity in both AW-101 and AN-107 is reasonably low, the results from the alpha energy analysis (AEA) should provide better Am-241 results than GEA. Americium-241 was not detected by GEA in the AW-101 samples, and the counting uncertainties ranged from 12% to 50% for the AN-107 samples. Although Am-241, along with Pu-239+240, makes up most of the alpha activity for the AW-101 and AN-107 samples, the Am-241 gamma activities are too low to measure accurately by GEA.

Since gamma energy analyses do not involve chemical separations, no sample spiking is performed.

### Total Alpha and Total Beta – Table 3.3

For total alpha and total beta activity measurements, the SAL preparations were further diluted, then small aliquots were evaporated on planchets for counting following procedures PNL-ALO-420, 421, 430, and 431.

The AW-101 supernatant sample had no detectable alpha activity by this method since the sample has high dissolved solids, which causes high mass loading on the counting planchet. Due to the high dissolved solids, sample volumes were reduced to minimize the self-absorption effect from the solids/salts. The reduced sample volume increases the minimum detectable activity level. The other samples have high dissolved solids, but also have more alpha activity, so that alpha activity was detectable by this method.

The high dissolved solids caused poor agreement between sample and duplicate alpha activity results for AW-101 solids and AN-107 solids samples. The effect of solids loading on the analytical precision is very difficult to accurately calculate, and has not been added into the uncertainty. Therefore, the actual analytical uncertainty is higher than the composite analytical and counting error presented in Table 3.3.

In some cases, a better estimate of the total alpha activity can be obtained by summing the individual alpha emitters measured by alpha energy analysis (i.e., Pu-238, Pu-239+240, Am-241, Cm-242, and Cm-243+244). The “Alpha Sum” in Table 3.3 provides the sum of the individual alpha emitters. This sum agrees fairly well with the total alpha activity results, even for those samples containing high dissolved solids. The total alpha activity from the sum of the alpha emitters is not biased from high dissolved solids and the error in the “Alpha Sum” is primarily from counting statistics. The “Alpha Sum” show good agreement between sample and duplicate samples. Although the sum of the individual alpha emitters show weak alpha activities in the process blanks, no alpha activity was detectable in the process blanks from the total alpha activity analysis.

The total beta activity results are considerably less affected by mass loading than the total alpha activity results, and the much higher beta activity in these samples allows a smaller aliquot than alpha counting requires. The duplicate beta activity results agree within expected uncertainties for all samples. The SAL process blanks have beta activity a factor of  $10^3$  to  $10^4$  less than the samples. The total beta activities show reasonable agreement with the sum of the beta emitters (i.e., mainly Cs-137 plus two times Sr-90) for all samples. All of the total alpha and total beta matrix and blank spikes were recovered within the acceptance criteria.

#### Plutonium, Americium, and Curium – Table 3.3

Plutonium, americium, and curium were separated from the SAL preparations using Eichrom TRU resin according to procedure PNL-ALO-417. The separated fractions were mounted for alpha spectroscopy by co-precipitation on a neodymium fluoride ( $\text{NdF}_3$ ) carrier (procedure PNL-ALO-496). Absolute activity of the alpha emitters was calculated using Pu-242 and Am-243 tracers added to the sample aliquots at the start of the chemistry in the laboratory.

The SAL process blanks indicated detectable Pu-239+240, but the activities were 50 times lower than the lowest sample and  $10^5$  less than the highest sample. Two SAL process blanks had slightly detectable Am-241. The largest effect is on the AW-101 supernatant, where the Am-241 result for the highest SAL process blank is about 3% the activity of the AW-101 supernatant.

Where the counting uncertainties are less than 10% for the sample and duplicate, the plutonium and americium RPD results are within the acceptance criteria of 20%, except for Pu-241 in the AW-101 supernatant, Pu-238 in the AW-101 solids, and Pu-239+240 and Pu-241 in the AN-107 solids. Plutonium-238 is occasionally a contaminant from the SAL hot cells, and either the sample or duplicate could be contaminated. Since the RPDs for other analytes measured in the AW-101 supernatant are well within the acceptance criteria, the poor precision demonstrated by the Pu-241 is most likely due to sample heterogeneity. For the AN-107 solids, the RPDs for the Pu-239+240 and Pu-241 are significantly higher than predicted from the good counting statistics. The RPDs for AN-107 solids are highly variable, ranging from 1% to 25%, indicating significant sample heterogeneity or inability to extract small ( $<0.2$  g) homogeneous sub-samples for fusion preparation.

All Pu-239 and Am-241 matrix spikes had acceptable recoveries. No matrix spike was prepared for curium.

Plutonium-241, a beta emitter, was measured by liquid scintillation counting. The Pu-241 spike had an acceptable but low recovery of 77%. The low recovery was most likely due to a weak spike activity level relative to the activity present in the sample.

#### Uranium – Table 3.5

Uranium was measured directly in dilutions of the hot cell KOH-KNO<sub>3</sub> fusion preparations by kinetic phosphorescence following procedure PNL-ALO-4014. The hot cell blanks have detectable uranium, but considerably less than any of the samples. The duplicates agree well in all cases. No uranium was detectable in any instrument blanks. All eight instrument check standards demonstrated recoveries between 98% and 102%.

#### Strontium-90 – Table 3.3

The Sr-90 analyses were conducted according to procedure PNL-ALO-476, which utilizes Sr-Spec resin that contains a crown-ether for the selective extraction of strontium from the radioactive and inactive matrix. After thorough washing, the strontium is back-extracted from the resin with water. The water is dried onto 2-inch planchets and counted with a gas-flow proportional counter according to procedure PNL-ALO-484. Radiochemical yields were determined with a Sr-85 tracer counted by GEA according to procedure PNL-ALO-450. The beta count rate is corrected for the interference from the Sr-85 tracer and Y-90 ingrowth.

The samples were analyzed in two batches; the supernatants were analyzed in one batch and the solids material analyzed in another batch. No analytical problems or anomalies were noted during the course of analysis. The RPDs between the samples and duplicates were equal to or less than 13%, well within acceptance criteria of 20%. Contamination apparent in the process blanks was nominally four orders of magnitude less than the Sr-90 detected in the samples. Thus contamination from the SAL hot cells has no effect on the reported results. Sample matrix spikes and blank spikes were prepared in radioanalytical laboratory and were processed with the sample batches. The blank spike recoveries were excellent at 100% and 97%. Two matrix spikes resulted in recoveries of 107% and 96%.

The AW-101 supernatant samples have no detectable Sr-90 by this method. Aliquots for Sr-90 analysis were chosen by estimating Sr-90 activity from the total beta activity counts; however, the beta activity from these samples turned out to be essentially all from Cs-137. The 0.5 µCi/mL detection limit obtained is within a factor of three of the 0.15 µCi/mL MRQ requested. Due to the cost impact for reanalyzing additional aliquots and the minimal usefulness of lowering the Sr-90 reported values, no additional Sr-90 analyses were performed.

#### Tritium – Table 3.3

Tritium was measured by distilling water dilutions of the samples per procedure PNL-ALO-418, then liquid scintillation counting the distillate per procedure PNL-ALO-474. The supernatant samples were merely mixed with water and then distilled; the solids were stirred with water and the

resultant slurry distilled. The processing blanks (distilled, de-ionized water) were mixed with water and distilled like the samples.

The process blanks have tritium at a level of about 15% of the lowest sample, and roughly 2% of the highest sample. Since the SAL hot cells are significantly contaminated with tritium, it is highly probable that the tritium levels in the processing blank are typical of the contamination levels within the samples.

The RPDs for the AW-101 supernatant and solids and the AN-107 supernatant are within the acceptance criteria of 20%, whereas the RPD for the AN-107 solids exceed this criteria. Many, but not all, radiochemical and inorganic results from the fusion preparation of AN-107 solids show large differences between the sample and duplicate analysis suggesting sample heterogeneity or inability to obtain small representative (e.g., <0.2 g for fusion preparation) analytical sub-samples.

The tritium results from the radioanalytical laboratory reagent blanks, considered to be the liquid scintillation background activities, were subtracted from the sample tritium results. Blank spikes were used to determine the detector efficiency. Matrix spikes were not prepared for tritium distillation and analysis.

#### Selenium-79 – Table 3.3

The Se-79 was measured using procedure PNL-ALO-440. In this method the selenium is separated from the sample by precipitation, followed by ion exchange, and then distillation. The product selenium is counted by liquid scintillation following procedure PNL-ALO-474. Since a Se-79 calibration standard is not commercially available, C-14 (which has a nearly identical beta energy) was used as a surrogate.

The liquid scintillation energy spectra had apparent Se-79 peaks in both the AW-101 and AN-107 supernatant samples. Both the AW-101 and AN-107 solids samples indicated beta activities above background; however, the spectra did not match Se-79. All the sample spectra indicated the presence of other beta emitters. To identify the extraneous beta emitters and eliminate the high bias and false positives caused by other beta activity, the liquid scintillation vials were gamma counted. Cs-137 was detected in most of the samples. The contribution of the Cs-137 activity in the Se-79 energy region was subtracted out, resulting in no counts above background for either the AW-101 or AN-107 solids samples. The RPDs for the AW-101 and AN-107 supernatant are well within the acceptance criteria of 20%. No Se-79 activity was seen in any of the processing blanks.

Since Se-79 is not available commercially, no blank spikes or matrix spikes were analyzed.

#### Carbon-14 – Table 3.3

Analysis not complete; to be reported in revision 1.

### 5.3 Anion Analysis by IC – Table 3.5

The AW-101 and AN-107 as received supernatants were diluted 5-fold to 15-fold during the preparation of the samples in the SAL, and were diluted an additional 100-fold to 1000-fold at the ion chromatography (IC) workstation to ensure that all anions of interest were within the calibration range. The solid samples were leached using procedure PNL-ALO-103 resulting in a SAL dilution factor of approximately 50 for each sample. The leaches were diluted an additional 100-fold prior to analysis. The supernatants and leach solutions from the solids were analyzed by IC for inorganic anions per procedure PNL-ALO-212. Although oxalate is an analyte of interest for the as received material, it is measured by organic IC analysis and reported as an organic anion in the Organic Report (BNFL-RPT-001, PNWD-2461). The oxalate results reported in Table 3.5 from the inorganic IC analysis are for information only.

Matrix spikes were prepared at the IC workstation following the dilution and leaching in the SAL. The matrix spike (AW-101 solids) and blank spikes demonstrated recoveries between 88% and 100%, well within the acceptance criteria of 75% to 125%. The analytical system blanks, as well as the dilution blanks and leach processing blank from the SAL, were all within acceptance criteria except for a single nitrate value (1.2 µg/ml) for one of 10 analytical system blanks. However, based on the very large nitrate concentrations for each of the samples, the nitrate contribution from this blank is considered negligible.

Where both the sample and duplicate results are above the EQL (i.e., for IC, the lowest calibration standard), the RPD was 11% or less for all samples and for all anions, with the exception of nitrate on AW-101 solids (RPD = 22%) and oxalate on AN-107 solids (RPD=23%). Since the other anions for the AW-101 solids have acceptable RPDs (i.e., 0% to 11%), the poor precision of the nitrate is most likely due to sub-sampling inconsistencies (i.e., the sample and duplicate solids sub-samples have difference liquid and solids fractions).

The fluoride peak was difficult to quantify largely due to the inability to deconvolute the fluoride peak from interfering peaks. Interferences from suspected organic anions are prevalent in nearly all samples. Based on the inability to deconvolute the fluoride and interference peaks, the fluoride results should be considered the “upper” bounds for the fluoride concentration.

As required by the governing QA Plan, mid-range verification standards were analyzed at the beginning of the analysis, after every 10 injections, and at the end of the analysis. For all reported results except chloride, all analytes of interest were recovered within the acceptance criteria of 90% to 110% for the verification standard. All chloride verification standards recoveries were within 88% to 90%. This low recovery was not recognized until after the completion of all the analyses. The low recovery is suspected to be due to an unknown error associated with preparation of the mid-range verification standard. Following the analyses, a fresh, mid-range verification standard solution was prepared and analyzed, resulting in recoveries in excess of 95% for chloride. Since the availability of additional sample was limited and the verification failure could be traced to an inaccurate verification standard, the results have been reported as analyzed. Since the low chloride recoveries demonstrated on the mid-range verification standard are suspected to be from a preparation error, no bias is suspected in the reported chloride results for the samples.

The phosphorus concentration based on phosphate results from the IC, both on the supernatants and leaches from the solids, are consistently higher (up to 3 times) than the total phosphorus results obtained by ICP. At this time, the cause of this discrepancy is unknown. The acid digestion and fusion preparations produced clear solutions; i.e., no indication of phosphate precipitation or loss of phosphorus. The IC chromatograms appear to have no interferences at the phosphate retention time; however, resolution of anions with similar retention times is not possible and can lead to quantitation of an unknown component as phosphate.

#### **5.4 TOC/TIC Analysis by Hot Persulfate/Coulometry – Table 3.5**

The analyses of the AW-101 and AN-107 as received samples were performed by the hot persulfate wet oxidation method, PNL-ALO-381. The hot persulfate method uses acid decomposition for total inorganic carbon (TIC) and acidic potassium persulfate oxidation at 92-95°C for TOC (total organic carbon), all on the same weighed sample, with total carbon (TC) being the sum of the TIC and TOC. Per the analytical method, the TOC and TIC results are bias corrected for average percent recovery of system calibration standards and are also corrected for contribution from the system blank. The TOC and TIC results are the only reported results subjected to this bias correction calculation.

The QC for the methods involves system calibration blanks, system calibration standards, sample duplicates, and matrix spikes. The QC system calibration standards were all within acceptance criteria, with the average recovery being 97% for TIC and 100% for TOC for the supernatant analysis and 96% TIC and 98% TOC for the solids analysis.

The TIC and TOC recovery from AW-101 supernatant and AW-101 solids matrix spikes ranged 103% to 118%, well within the acceptance criteria of 75% to 125%. The matrix spike recovery for the AN-107 solids samples produced some erratic results. The recovery for the TIC spike was 38%, whereas the recovery for the TOC spike was 155%. Based on the actual quantity of carbon added from the TIC and TOC spikes, the TC recovery is about 90%. This suggests either an entry error (e.g., TIC and TOC raw data entries for the matrix spike sample were switched) or the room temperature acid addition did not reduce the pH adequately (which is unlikely). Since the quantity of sample to acid concentration is low, it is suspected that the entries were inadvertently switched. Recalculating the matrix spike recoveries based on the assumption that the entries are switched results in recoveries of 85% for TIC and 96% for TOC. Since the matrix spike recoveries for AW-101 supernatant and solids samples were within acceptance criteria, no additional matrix spike analyses were performed on AN-107 solids.

Although an alternate furnace method was available for measuring TIC and TC, only the hot persulfate method was used on the AW-101 and AN-107 samples. Based on the analytical information available (i.e., oxalate and formate by IC reported in PNWD-2461), the major organic components appear to be oxalate and formate. Although the furnace method typically provides better total carbon results on difficult to decompose organic salts, the hot persulfate TC results are considered accurate within the acceptance criteria of the governing QA plan (and the hot persulfate method typically provides more reliable TIC results than the furnace method). Salts of oxalate and formate demonstrate reasonable recoveries (i.e., >90%) when analyzed by the hot persulfate method. Providing TOC, TIC and TC by two different method would provide additional

information; however, the benefit of this additional information could not justify the additional costs.

## 5.5 Radionuclide Analysis by ICP/MS— Table 3.4

Selected radionuclide analyses and the platinum and tantalum analyses were performed by ICP/MS per procedures PNL-ALO-280, 281, and 282. For the supernatants, the radionuclides and analytes of interest were analyzed on both the acid digestions and water dilutions of the supernatants. For the solids, the samples were analyzed on both the KOH-KNO<sub>3</sub> fusion and Na<sub>2</sub>O<sub>2</sub>-NaOH-HCl fusion preparation of the solids.

The RPDs from both the acid digestions and the water dilutions of the AW-101 and AN-107 supernatant were within the 20% acceptance criteria. The RPDs for the KOH-KNO<sub>3</sub> fusion for both AW-101 and AN-107 were within acceptance criteria, except for the AN-107 solids Tc-99. The poor RPD for Tc-99 is attributed to sample heterogeneity and the small samples sizes used for fusion preparations. The RPDs for the Na<sub>2</sub>O<sub>2</sub>-NaOH-HCl fusion for the AN-107 solids are within acceptance criteria. However, the RPDs for the AW-101 solids are all extremely poor, suggesting significant sample heterogeneity or poor sub-sampling (e.g., sample and duplicate sub-sample liquid and solid fractions are not the same, possibly due to location in the jar from which solids sub-sample was taken).

No preparation matrix spikes for radionuclides were prepared. However, post spikes were prepared and analyzed at the ICP/MS workstation. The post spike recoveries ranged from 84% to 107%, well within the acceptance criteria of 75% to 125%.

In general, the results of the continuing calibration verification check standard were within acceptance criteria. However, some radionuclides of interest did not meet the acceptance criteria on every check standard. In particular, the check standards for I-129 were consistently outside the acceptance criteria, although the post spike recoveries were acceptable. The potential bias introduced by the failure of some check standard radionuclides is considered insignificant, since most of the failures were only marginally outside the acceptance window.

The reported Tc-99 results assume that the ruthenium present is exclusively fission-product ruthenium, and therefore does not have an isotope at mass 99. The calculated results assume that everything observed at the mass 99 is due to technetium. The observed ruthenium mass spectra are not typical of natural ruthenium and are consistent with spectra observed in previous tank waste analyses. Therefore, the assumption that observed mass 99 is primarily technetium should be valid.

Off-line interference corrections were required for I-129 (for xenon correction) and Pu-239 (for uranium hydride correction). The results in Table 3.4 and Table 3.5 include the corrections.

The Na<sub>2</sub>O<sub>2</sub>-NaOH-HCl fusion blank preparation appears to have a severe tantalum contamination. This contamination is not evident in any samples. However, this is not a major concern since the tantalum results reported in Table 3.1 and Table 3.2 are obtained from the KOH-KNO<sub>3</sub> fusion preparations.

The ICP/MS results for Pu-239+240 compare very favorably to the results obtained by AEA. Also, the ICP/MS results for total uranium (i.e., U-238+235) compare well with the results obtained by kinetic phosphorescence and ICP. In general, the average results between the methods vary by less than 20%, which is considered very good agreement for a method-to-method comparison. Table 5.1 presents the comparison for these results. The ICP/MS can not distinguish between U-238 and Pu-238. However, since the Pu-238 concentration (in  $\mu\text{g/g}$  or  $\mu\text{g/mL}$ ) is negligible, the ICP/MS response at mass 238 is attributed solely to U-238.

**Table 5.1 Comparison of ICP/MS to Other Methods for Pu-239+240 and Total U**

<i>Tank Material</i> <i>Matrix</i> <i>Dissolution</i>		AW-101 <sup>(1)</sup>		AN-107 <sup>(1)</sup>	
		Supernatant	Solids	Supernatant	Solids
		Acid Digest	KOH-KNO <sub>3</sub> fusion	Acid Digest	KOH-KNO <sub>3</sub> fusion
Analyte	Method	$\mu\text{Ci/mL}$	$\mu\text{Ci/g}$	$\mu\text{Ci/mL}$	$\mu\text{Ci/g}$
Plutonium 239+240	ICP/MS	<2.0e-3	5.30E-02	6.24E-02	6.50E-02
	Radiochem	5.14E-04	5.61E-02	4.77E-02	6.75E-02
	Difference from ICP/MS	n/a	6%	23%	4%
Analyte	Method	$\mu\text{g/mL}$	$\mu\text{g/g}$	$\mu\text{g/mL}$	$\mu\text{g/g}$
Total Uranium	ICP/MS <sup>(2)</sup>	4.37E+00	1.21E+03	1.17E+02	9.80E+01
	Kinetic Phosphorescence	3.53E+00	1.12E+03	1.05E+02	8.53E+01
	Difference from ICP/MS	19%	8%	11%	13%
Total Uranium	ICP/MS <sup>(2)</sup>	4.37E+00	1.21E+03	1.17E+02	9.80E+01
	ICP <sup>(3)</sup>	<1.7e+02	1.00E+03	<1.8e+02	<2.4e+02
	Difference from ICP/MS	n/a	17%	n/a	n/a
<sup>(1)</sup> All results are average of sample and duplicate results from Table 3.1 to Table 3.5 <sup>(2)</sup> ICP/MS Total Uranium = sum of U-238 and U-235 <sup>(3)</sup> ICP solids results from acid digestion, not KOH-KNO <sub>3</sub> fusion					

## 5.6 Free Hydroxide and pH Analysis – Table 3.5

Analysis of free hydroxide was performed following analytical dilution of the supernatants in the SAL. The AW-101 and AN-107 supernatant samples were analyzed according to procedure PNL-ALO-228. The samples were titrated with NIST-traceable hydrochloric acid. Quality control check standards were prepared from NIST-traceable sodium hydroxide. The accuracy of the check standards measured within 2.4% of the true hydroxide concentration.

For AW-101, a free hydroxide concentration of about 5.3 millimoles OH per milliliter is calculated from the first equivalence point on the titration curve. To verify that the hydroxide equivalence point was the first equivalence point, AW-101 supernatant was spiked with sodium hydroxide standard; the first equivalence point was identical to that of the sample (i.e., pH = 10.27, at a 10 fold dilution). Recovery of the matrix spike was 100.2%. The RPD for the free hydroxide results for the AW-101 supernatants is less than 1%.

For AN-107, the pH was measured at about 11.0 and the TIC at about 1.6%. The first equivalence point was measured at a pH of 8.1 and produced a base-equivalence of 1.3 millimoles per milliliter. Based on the initial pH of the AN-107 supernatant and the very high TIC concentration, the first



equivalence point is likely due primarily to carbonate not free hydroxide. If all carbonate, the 1.6% inorganic carbon result calculates to a 1.3 millimoles  $\text{CO}_3$  per milliliter, essentially the same as the measured base-equivalence. Since no equivalence point was detected at higher pH, free hydroxide is estimated to be less than the detection limit of 0.01 millimoles OH per milliliter.

The pH measurements were performed in duplicate on the AW-101 and AN-107 supernatants per procedure PNL-ALO-225.

### **5.7 Mercury Analysis by CVAA – Table 3.5**

Analysis not complete; to be reported in revision 1.

### **5.8 Ammonia Analysis by ISE – Table 3.5**

Analysis of ammonia was performed on the AW-101 and AN-107 supernatants and leaches of the solids by the Method of Standard Addition (MSA) using ammonia ion specific electrode (ISE). The analysis was performed per procedure PNL-ALO-226. The MSA relies on the addition of known standards to calculate the starting concentration in the sample. Also, the ISE was calibrated from  $10^{-6}$  molar to  $10^{-2}$  molar and this calibration curve provided additional validation of the reported results.

The hot cell blanks were about 10 to 20 times the ISE ammonia detection limit. Although ammonia was detected in leaches for the AW-101 and AN-107 solids, the results are nearly identical to the hot cell preparative blanks and the reported ammonia is considered residual contamination from the hot cell preparative activities. The hot cell blank concentrations were less than 3% of the ammonia concentration measured in the supernatants, and have little affect on the reported ammonia supernatant results.

Based on the organic analysis of the AW-101 and AN-107 as received materials (PNWD-2461), amines may be present in the samples. Volatile amines are an interference for ammonia analysis performed by ammonia ISE, and the apparent ammonia concentrations in the samples may be from unresolved amine (or other) interferences.

### **5.9 Total Cyanide Analysis by Distillation/Colorimetry – Table 3.5**

Analysis not complete; to be reported in revision 1.

### **5.10 Flashpoint Determination – Table 3.5**

Analysis not complete; to be reported in revision 1.

## 6.0 Method Detection Limits & Minimum Reportable Quantities

The MDLs for specific analytes of interest vary significantly based on the procedures used for preparing the samples for analysis (e.g., acid digestion, fusion), the sample size taken for the analysis, required dilutions for ALARA safety considerations, and the magnitude of interfering analytes. For the work presented in this report, effort was made to optimize the sample preparation parameters (e.g., minimal dilutions). Table 6.1 provides a direct comparison of each analyte MDL to the target MRQ provided by BNFL. The MDLs are nominal values based on average sample sizes used for analysis. The MDLs are presented for both liquids and solids. Where solids are prepared by both acid digestion and fusion, both the acid digestion MDL and fusion MDL are provided for comparison. The MDLs that are shaded and boxed with double lines exceed the BNFL requested MRQs.

As is evident from the Table 6.1, some analytes of interest have not been measured at the target MRQ. As would be expected due to high preparative dilution factors, fusion preparations have the largest quantity of MDLs not meeting the target MRQ. For AW-101 and AN-107, this is not a significant problem, since the acid digestion of the solids provides an apparent 100% dissolution. Although the acid digestion appeared suitable for the AW-101 and AN-107 materials, silicon is still of some concern, since acid digestion is an extremely poor dissolution method for most silicon-based compounds.

Many of the high MDLs from the fusion preparation are within a factor of five of the target MRQ; specifically, aluminum, chromium, iron, magnesium, manganese, and silicon. With further optimization of sample size and analytical dilutions, these fusion MDLs may meet the target MRQs. Although the Am-241 (by GEA) and total alpha fusion MDLs are higher than the MRQs, the MDLs for Am-241 (by AEA) and total alpha by "Sum Alpha" are considerably below the MRQ. The fusion MDLs for calcium, cadmium, cobalt, molybdenum, sodium, uranium (by ICP), zinc, and Pu-241 are from 10 to 1000 times higher than the target MRQs, and it is unlikely that fusion preparation can be optimized to this extent.

Of those MDLs for solids, the one of most concern is Pu-241. It is unlikely that any preparative technique will allow the quantification of Pu-241 at the 1.2 pCi/g unless very large sample volumes can be prepared for counting. Since Pu-241 decays to Am-241, there is a question as to the rationale of the 1.2 pCi/g MRQ for Pu-241 when the MRQ for Am-241 is 1.2 nCi/g.

The solids acid digestion MDL for cobalt is within a factor of two of the target MRQ, and with additional optimization of the digestion preparation the cobalt MDL for solids may be able to be reduced. Also, the MDLs for TIC and TOC for solids are about a factor of two higher than the target MRQs. Analytically the TIC and TOC MDLs can be reduced by increasing the solids sample size; however, ALARA limitations make this approach questionable.

For liquid analysis, only potassium and chloride MDLs are above the target MRQs. The liquid acid digestion MDL for potassium is about a factor of two higher than the target MRQ, and additional optimization may be able to lower the MDL. As for chloride, it is unlikely that the MRQ of 3 µg/mL can be achieved consistently on tank waste supernatants by using IC as the analysis method. The presence of other anions (particularly very high nitrate) requires significant dilution of the samples prior to analysis to prevent IC column overloading. This required dilution, and the often presence of interfering organic anions, significantly limit the chloride MDL.

Table 6.1 Comparison of Measurement MDLs to Target MRQs

Analyte	Liquids		Solids			Radionuclide	Liquids		Solids	
	MDL (1) μg/mL	BNFL MRQ μg/mL	MDL (1) μg/g	MDL (Fusion) μg/g	BNFL MRQ μg/g		MDL (1) μCi/mL	BNFL MRQ μCi/mL	MDL (Fusion) μCi/g	BNFL MRQ μCi/g
Ag	2.14E+0	1.75E+1	2.94E+0	1.70E+2	9.00E+2	H-3	1.00E-5		1.00E-4	
Al	5.14E+0	7.50E+1	7.06E+0	4.00E+2	3.30E+2	C-14	TRD		TRD	
As	2.14E+1		2.94E+1	1.70E+3		Co-60 (GEA)	2.00E-4	2.10E-3	2.00E-3	1.20E-2
B	4.00E+0		6.00E+0	3.30E+2		Se-79	2.00E-6		4.00E-5	
Ba	8.57E-1	7.80E+1	1.18E+0	6.61E+1	6.00E+2	Sr-90	3.00E-3	1.50E-1	3.00E-2	7.01E+1
Be	8.57E-1		1.18E+0	6.61E+1		Nb-94 (GEA)	2.00E-4		2.00E-3	
Bi	8.57E+0		1.18E+1	6.60E+2		Ru-106/Rh-106 (GEA)	2.00E-3		2.00E-2	
Ca	2.10E+1	1.50E+2	2.94E+1	1.70E+3	1.80E+2	Sb-125 (GEA)	5.00E-4		5.00E-3	
Cd	1.29E+0	7.50E+0	1.77E+0	9.91E+1	1.10E+1	Sn-126 (GEA)	2.00E-4		2.00E-3	
Co	4.29E+0	3.00E+1	5.89E+0	3.30E+2	3.00E+0	Cs-134 (GEA)	2.00E-4		2.00E-3	
Cr	1.71E+0	1.50E+1	2.35E+0	1.30E+2	1.20E+2	Cs-137 (GEA)	2.00E-4	9.00E+0	2.00E-3	6.00E-2
Cu	2.14E+0	1.70E+1	2.94E+0	1.70E+2	1.80E+1	Eu-154 (GEA)	4.00E-4	2.00E-3	4.00E-3	6.00E-2
Fe	2.00E+0	1.50E+2	2.94E+0	1.70E+2	1.40E+2	Eu-155 (GEA)	5.00E-4	9.00E-2	5.00E-3	6.00E-2
K	1.70E+2	7.50E+1	2.40E+2		1.50E+3	Pu-238	2.00E-6	9.60E-3	2.00E-5	6.00E-5
La		3.50E+1			6.00E+1	Pu-239+Pu-240	2.00E-6		2.00E-5	
Li	2.57E+0		3.53E+0	2.00E+2		Pu-241	3.00E-4	9.60E-3	3.00E-3	1.20E-6
Mg	8.57E+0	1.50E+2	1.18E+1	6.60E+2	5.40E+2	Am-241(GEA)	4.00E-4	7.20E-4	6.00E-3	1.20E-3
Mn	4.29E+0	1.50E+2	5.89E+0	3.30E+2	3.00E+2	Am-241 (AEA)	2.00E-6	7.20E-4	2.00E-5	1.20E-3
Mo	4.29E+0	9.00E+1	5.89E+0	3.30E+2	6.00E+0	Cm-242	3.00E-7		2.00E-6	
Na	1.30E+1	7.50E+1	1.80E+1	9.90E+2	1.50E+2	Cm-243+Cm-244	5.00E-7	1.50E-1	5.00E-6	6.00E-5
Ni	2.57E+0	3.00E+1	3.53E+0		1.60E+2	Beta	2.00E-4		2.00E-3	
P	8.57E+0		1.18E+1	6.60E+2		Alpha	7.00E-5	2.30E-1	2.00E-3	1.00E-3
Pb	8.57E+0	3.00E+2	1.18E+1	6.60E+2	6.00E+2	Sum Alpha	2.00E-6		2.00E-5	
Pd	6.43E+1		8.83E+1	5.00E+3			μCi/mL	μCi/mL	μg/g	μg/g
Pt	7.00E-2			2.00E-1		Tc-99 (ICP/MS)	3.39E-4	1.50E-3	1.00E-1	6.00E+0
Rh	2.57E+1		3.53E+1	2.00E+3		I-129 (ICP/MS)	1.77E-5	1.10E-3	4.00E-1	3.00E+1
Sb	4.29E+1		5.89E+1	3.30E+3		U-233 (ICP/MS)	4.82E-4	1.80E-3	1.00E-1	6.00E+0
Se	2.14E+1		2.94E+1	1.70E+3		U-234 (ICP/MS)	1.24E-4		2.00E-1	
Si	4.29E+1	1.70E+2	5.90E+1	3.30E+3	3.00E+3	U-235 (ICP/MS)	4.32E-8	3.30E-6	2.00E-1	6.00E+0
Sn	1.30E+2		1.80E+2	9.90E+3		U-236 (ICP/MS)	1.20E-6		2.00E-1	
Ta	7.00E-2			2.00E-1		U-238 (ICP/MS)	6.72E-8	5.00E-7	2.00E+0	6.00E+0
Ti		1.70E+1			1.50E+2	Np-237(ICP/MS)	1.41E-5	2.70E-2	1.00E-1	1.80E+0
Tl	4.29E+1		5.89E+1	3.30E+3		Pu-239 (ICP/MS)	1.24E-3	9.60E-3	1.00E-1	6.00E+0
U	1.70E+2	6.00E+2	2.40E+2	1.30E+4	6.00E+2	Pu-240 (ICP/MS)	4.54E-3		1.00E-1	
Total U (2)	1.00E-5	6.00E+2		1.00E-4	6.00E+2					
V	4.29E+0		5.89E+0	3.30E+2			μg/mL	μg/mL	μg/g	μg/g
W	1.70E+2		2.40E+2	1.30E+4		OH (total)	1.70E+2	1.70E+2		
Y	4.29E+0		5.89E+0	3.30E+2		OH (free)	1.70E+2	1.70E+2		
Zn	4.29E+0	1.65E+1	6.00E+0	3.30E+2	6.00E+0	Ammonia	1.00E-1		1.0E+0	
Zr	4.29E+0		5.89E+0	3.30E+2	6.00E+2	Hg	TRD	2.00E+0	TRD	1.50E+0
Br	1.90E+1		1.21E+2			CN	TRD		TRD	
Cl	1.90E+1	3.00E+0	1.21E+2		2.30E+2	<p>Note: Shaded and boxed MDLs exceed the target MRQ</p> <p>← PO<sub>4</sub> solids MDL and MRQ as P</p> <p>← SO<sub>4</sub> solids MDL and MRQ as S</p> <p>(1) Acid Digestion, Water Leach, or Direct Analysis, as applicable</p> <p>(2) Total uranium by kinetic phosphorescence</p>				
F	1.90E+1	1.50E+2	1.21E+2		7.50E+3					
NO <sub>2</sub>	3.85E+1		2.46E+2							
NO <sub>3</sub>	3.85E+1	3.00E+3	2.46E+2		4.50E+2					
PO <sub>4</sub>	3.85E+1	2.50E+3	8.00E+1		6.00E+2					
SO <sub>4</sub>	3.85E+1	2.30E+3	8.20E+1		1.20E+3					
C <sub>2</sub> O <sub>4</sub>	3.75E+1		2.41E+2							
TIC	1.40E+1	1.50E+2	7.35E+1		3.00E+1					
TOC	2.55E+1	1.50E+3	1.20E+2		6.00E+1					