

COMPOSITING, CHARACTERIZATION, AND DILUTION OF SAMPLES FROM HANFORD TANK 241-AW-101

4/15/03

M. S. Hay, SRTC/WPT
C. J. Coleman SRTC/ADS
K. B. Martin SRTC/WPT

UNCLASSIFIED
DOES NOT CONTAIN
UNCLASSIFIED CONTROLLED
NUCLEAR INFORMATION

ADC &
Reviewing
Official:

M. S. Hay
(Name and Title)

Date:

4/28/03

ADDITIONAL APPROVAL:

Official:

C. J. Coleman for *W. L. Tompkins*
R&T Manager, RPP Hanford

Date:

5/2/2003

Westinghouse Savannah River Company
Savannah River Site
Aiken, SC 29808

Prepared for the U.S. Department of Energy Under Contract Number DEAC09-96SR18500



This document was prepared in conjunction with work accomplished under Contract No. DE-AC09-96SR18500 with the U. S. Department of Energy.

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

This report has been reproduced directly from the best available copy.

**Available for sale to the public, in paper, from: U.S. Department of Commerce, National Technical Information Service, 5285 Port Royal Road, Springfield, VA 22161,
phone: (800) 553-6847,
fax: (703) 605-6900
email: orders@ntis.fedworld.gov
online ordering: <http://www.ntis.gov/help/index.asp>**

**Available electronically at <http://www.osti.gov/bridge>
Available for a processing fee to U.S. Department of Energy and its contractors, in paper, from: U.S. Department of Energy, Office of Scientific and Technical Information, P.O. Box 62, Oak Ridge, TN 37831-0062,
phone: (865)576-8401,
fax: (865)576-5728
email: reports@adonis.osti.gov**

Key Words:

Supernate Composition
Entrained Solids
Sample Analysis

Retention: Permanent

Key WTP R&T References:

Test Specification: 24590-WTP-TSP-RT-01-001
Task Plan: WSRC-TR-2001-00244

**COMPOSITING, CHARACTERIZATION, AND
DILUTION OF SAMPLES FROM HANFORD TANK
241-AW-101**

**M. S. Hay, SRTC/WPT
C. J. Coleman, SRTC/ADS
K. B. Martin, SRTC/WPT**

Issue Date: 4/15/03

Westinghouse Savannah River Company
Savannah River Site
Aiken, SC 29808

Prepared for the U.S. Department of Energy Under Contract Number DE-AC09-96SR18500

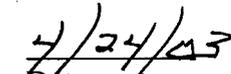


Completeness of Testing

This report describes the results of work and testing specified by test specification 24590-WPT-RT-01-001, Rev 0.. The work and any associated testing followed the quality assurance requirements outlined in the test specification. The descriptions provided in this test report are an accurate account of both the conduct of the work and the data collected. Test results are reported as well as any unusual or anomalous occurrences that are different from expected results. The test results and this report have been reviewed and verified.

Approved:


John Marra, Manager
WTP R&T Support Project


Date

This page intentionally left blank

Summary

As part of the program to provide pretreatment development and testing services to support the River Protection Project-Waste Treatment Plant (RPP-WTP) mission to treat Hanford tank waste, a ~15 L sample of waste from tank 241-AW-101 was received at the Savannah River Technology Center (SRTC). The waste sample was characterized and diluted to provide feed for pretreatment testing. The characterization data provides a basis for rational development of pretreatment processes, determination of reagent requirements, and development of physical design parameters for the pretreatment plant.

The main objectives of this work as defined by the test specification and approved task plan were to:

- Prepare a composite sample from the multiple 241-AW-101 samples
- Verify the homogeneity of the composite sample
- Analyze the composite sample
- Dilute the composite sample to 5 M sodium to provide feed for pretreatment testing
- Analyze the diluted 5 M composite sample
- Compare the as-received composite sample analytical results to the low-activity waste (LAW) feed specifications
- Analyze the undissolved solids
- Report analytical results

A total of thirty 500 mL jars of 241-AW-101 tank waste were received at SRTC and composited in a 40 L carboy. A homogeneity test indicated the mixing and sampling system were capable of providing representative samples of the composite. The as-received filtered supernate and the solids collected from the filtration were analyzed and the supernate found to have a sodium concentration of ~10.2 M.

The as-received sample was diluted with de-ionized water to a sodium concentration of 5.0 M. During the bulk dilution of the sample the 5.0 M sodium endpoint was overshoot with a resulting sodium concentration of 4.5 M. A portion of the sample was concentrated using vacuum distillation and added back to the bulk solution to bring the concentration back to 5.0 (+/-0.1) M sodium. The diluted bulk solution was sampled and sent to the CUF unit for filtration. The filtered supernate from the diluted 5 M sodium was analyzed. Due to the low entrained solids content (~0.1 wt%) of the diluted 5 M sodium 241-AW-101, the decision was made to allow the Cell Unit Filter (CUF) testing to proceed and obtain a portion of the concentrated solids for analysis at completion of the testing. Due to a mishap in the CUF testing, less than 0.5 g of solids were available for analysis. There were only sufficient solids to allow a single sample digestion method instead of the normal two methods.

All of the main objectives of the characterization and dilution program for the 241-AW-101 sample were met. However, as part of the reporting objective, data quality requirements were specified for each analyte. Due to the radioactive nature of the sample, dilutions of the raw

samples were required to allow removal from the Shielded Cells facility for analysis. These dilutions combined with the presence of very low concentrations of some analytes in the sample make it difficult to meet all of the data quality requirements for all analytes. In those cases where the data quality requirements were not met quality control (QC) flags in the data tables identify which criteria were not met.

The analytical results from the 241-AW-101 sample were compared to the Low-Activity Waste feed limits in Specification 7. The 241-AW-101 sample met all of the contract feed limit specifications.

Overall the data quality for the analysis of the 241-AW-101 sample was reasonably good. The quality control of the sample analysis consisted of the use of triplicate sample analysis, blanks, laboratory control standards (LCS), and a matrix spike.

The characterization data presented represent the composition of the 241-AW-101 sample received at SRTC and makes no assertions as to the validity of the data with respect to the tank contents as a whole or to any feed delivered from this tank. Recent experience at SRS indicates a combined sampling and analytical error on the order of 15 - 20% associated with obtaining small samples from a well mixed waste tank.

The test specification also included dilution of a portion of the composite sample to 7 M sodium concentration, analysis of the 7 M diluted sample, and a 6 month stability study of the 7 M diluted sample. The results of the 7 M dilution study will be reported separately at the completion of the 6 month stability study.

Contents

Summary	iv
Contents.....	vi
List of Tables.....	vii
1.0 Introduction	1
2.0 Sample Receiving and Compositing	3
3.0 Homogeneity Testing and Sub-Sampling	4
4.0 Bulk Dilution to 5 M Sodium and Sampling of the Diluted 241-AW-101 Sample	5
5.0 Sample Preparation for Analysis.....	6
5.1 Preparation of Samples for the Analysis of Liquid Fractions.....	6
5.2 Preparation of Samples for the Analysis of Solids Fractions	6
5.3 Dose Rate Limits for Analytical Laboratories	7
6.0 Density and Weight Percent Solids Measurements.....	9
7.0 Analytical Results and Data Evaluation.....	10
7.1 General Information.....	10
7.2 QC Flags	10
7.3 Data Evaluation.....	11
7.4 Data Tables	21
8.0 Comparison of the 241-AW-101 Sample to Specification 7.....	46
9.0 General Description of Analytical Procedures.....	51
9.1 Inductively Coupled Plasma-Atomic Emission Spectroscopy	51
9.2 Ion Chromatography for Anions and Organics Acids	51
9.3 Free Hydroxide and Total Base Titrations.....	51
9.4 Atomic Absorption Spectroscopy	52
9.5 Ammonia.....	52
9.6 Organics	52
9.7 Total Inorganic Carbon/Total Organic Carbon.....	52
9.8 Inductively Coupled Plasma-Mass Spectrometry	53
9.9 Uranium by Chemchek (Phosphorescence Method).....	53
9.10 Alpha Counting.....	53
9.11 Gamma Spectrometry	54
9.12 H ³ Analysis	54
9.13 Tc ⁹⁹ Analysis (for Pertechnetate Form).....	54
9.14 Sr ⁹⁰ Analysis	54
9.15 Se ⁷⁹ Analysis.....	55
9.16 Alpha Spectroscopy for Plutonium Isotopics	55
9.17 I ¹²⁹ Analysis	55
9.18 Am/Cm Analysis.....	56
9.19 C ¹⁴ Analysis	56
10.0 References	57
Appendix 1	A.1
Appendix 2	A.2
Appendix 3	A.3

List of Tables

Table 2.1. Net Weight of Sample Jars of AW-101.	3
Table 3.1. Results of the Homogeneity Test for As-Received 241-AW-101.	4
Table 5.1. Composition of the Analytical Reference Glass-1 Standard.....	8
Table 7.1. Abbreviations for Analytical Methods in Tables 7.2 through 7.11.....	21
Table 7.2. Properties of the As-Received 241-AW-101 Sample	22
Table 7.3. Composition of the As-Received 241-AW-101 Filtered Supernate	23
Table 7.4. Properties of the Diluted 5 M Sodium 241-AW-101 Sample	27
Table 7.5. Composition of the Diluted 5 M Sodium 241-AW-101 Filtered Supernate	28
Table 7.6. Composition of the Aqua-Regia Digested As-Received 241-AW-101 Filterable Solids.....	32
Table 7.7. Composition of the Sodium Peroxide Fusion Digested As-Received 241-AW-101 Filterable Solids.....	36
Table 7.8. Composition of the Aqua-Regia Digested Diluted 5 M Sodium 241-AW-101 Filterable Solids.....	40
Table 7.9. Composition of the Water Contact of the As-Received 241-AW-101 Filterable Solids.....	42
Table 7.10. Composition of the 1st Condensate from Concentration of the Diluted 5 M Sodium 241-AW-101 Sample	44
Table 7.11. Composition of the 4th Condensate from Concentration of the Diluted 5 M Sodium 241-AW-101 Sample	45
Table 8.1. Comparison of the As-Received 241-AW-101 Filtered Supernate to Specification 7 Chemical Composition Limits.	47
Table 8.2. Comparison of the As-Received 241-AW-101 Filtered Supernate to Specification 7 Radionuclide Limits.	48
Table 8.3. Comparison of the 5 M Sodium 241-AW-101 Filtered Supernate to Specification 7 Chemical Composition Limits.	49
Table 8.4. Comparison of the 5 M Sodium 241-AW-101 Filtered Supernate to Specification 7 Radionuclide Limits.	50

1.0 Introduction

The Savannah River Technology Center (SRTC) has been contracted to provide pretreatment development and testing services to support the River Protection Project-Waste Treatment Plant (RPP-WTP) mission to treat Hanford tank waste. As part of the program, SRTC received radioactive Hanford tank waste samples to allow testing of the pretreatment processes with actual waste samples. The first step in this program entails detailed characterization of the radioactive waste samples. The characterization data provide a basis for rational development of pretreatment processes, determination of reagent requirements, and development of physical design parameters for the pretreatment plant.

The characterization portion of the SRTC program was conducted under an approved task and quality assurance plan based on a test specification issued by Bechtel National, Inc./Washington Group International (BNI/WGI).^{1,2} The results and the associated uncertainties presented provide a description of the sample received at SRTC. The highly radioactive nature of the samples adds complexity to the analysis. Sub-sampling, large dilutions, and remote handling potentially add error to the analytical accuracy. Replicate sample analysis, matrix spike and laboratory control standards, and submission of blanks allow some definition of the magnitude of this error. However, the error associated with obtaining small samples from large non-homogenized waste tanks will be significant. Recent experience at the Savannah River Site (SRS) indicates a combined sampling and analytical error on the order of 15 - 20% associated with obtaining small samples from a well mixed waste tank.³

The data presented in this report documents the chemical characterization of a ~15 L sample of Hanford waste tank 241-AW-101. The objectives of this work were to:

- Prepare a composite sample from the multiple 241-AW-101 samples
- Verify the homogeneity of the composite sample
- Analyze the composite sample
- Dilute the composite sample to 5 M sodium to provide feed for pretreatment testing
- Analyze the diluted 5 M composite sample
- Compare as-received composite sample analytical results to the low-activity waste (LAW) feed specifications
- Analyze the undissolved solids
- Report analytical results

The test specification also includes dilution of a portion of the composite sample to 7 M sodium concentration, analysis of the 7 M diluted sample, and a 6 month stability study of the 7 M diluted sample. The results of the 7 M dilution study will be reported separately at the completion of the 6 month stability study.

The test specification provides detailed requirements for the task with respect to quality assurance. The WSRC Quality Assurance Program and the WSRC Quality Assurance

Management Plan (WSRC-RP-92-225) were followed during the task. The WSRC Quality Assurance Program was approved by WTP. The appropriate quality assurance requirements for this task from NQA-1-1989 (Part II, Basic and Part III Supplementary Requirements) and NQA-2a-1990, Part 2.7, as indicated by the QA Plan Checklist in Section VIII of the task plan, were applied to this task. The test specification also states that the Department Of Energy (DOE) Quality Assurance Requirements and Description (QARD), DOE/RW-00333P, Rev. 10, does not apply to this task.

2.0 Sample Receiving and Compositing

A total of thirty 500 mL glass jars of 241-AW-101 tank waste were received at SRTC in January and March 2001. The 500 mL grab samples were obtained from riser 22 of the 241-AW-101 waste tank in July, 2000 (Appendix 1).

Visual inspection at SRTC showed each jar contained a clear liquid phase with an ~1/4" layer of mostly crystalline white solids on the bottom. No organic layers were observed in any of the jars. The thirty jars were composited into a 40 L polyethylene carboy. Each jar was weighed before and after addition through a 1/4" by 1/4" mesh screen into the carboy. There were no solids collected on the screen after the compositing. The volume of sample in the 40 L carboy after emptying all of the sample jars was ~13.5 L. The total weight of sample added to the carboy was 19.8 kg based on the measured net weight of each jar. Table 1 shows the jar and lab ID's taken from each jar, the measured net weight based on the full and empty weight of each jar, and the reported net weight of each jar from the chain of custody documentation (Appendix 1).

Table 2.1. Net Weight of Sample Jars of AW-101.

Jar ID	Lab ID	Measured Net Wt.	Reported Net Wt.
1AW-00-1	S00T001466	664 g	671 g
1AW-00-2	S00T001467	654 g	665 g
1AW-00-3	S00T001468	663 g	672 g
1AW-00-4	S00T001469	675 g	678 g
1AW-00-5	S00T001470	663 g	679 g
1AW-00-6	S00T001471	665 g	678 g
1AW-00-7	S00T001472	599 g	686 g
1AW-00-8	S00T001473	655 g	673 g
1AW-00-9	S00T001474	670 g	684 g
1AW-00-10	S00T001475	655 g	667 g
1AW-00-11	S00T001476	668 g	672 g
1AW-00-12	S00T001477	670 g	674 g
1AW-00-13	S00T001493	655 g	664 g
1AW-00-14	S00T001492	661 g	664 g
1AW-00-15	S00T001494	657 g	663 g
1AW-00-16	S00T001495	665 g	676 g

Jar ID	Lab ID	Measured Net Wt.	Reported Net Wt.
1AW-00-17	S00T001496	650 g	667 g
1AW-00-18	S00T001497	669 g	678 g
1AW-00-19	S00T001498	661 g	666 g
1AW-00-20	S00T001499	670 g	667 g
1AW-00-21	S00T001478	654 g	659 g
1AW-00-22	S00T001479	664 g	670 g
1AW-00-23	S00T001480	666 g	671 g
1AW-00-24	S00T001481	668 g	676 g
1AW-00-25	S00T001482	662 g	671 g
1AW-00-26	S00T001483	654 g	663 g
1AW-00-27	S00T001484	646 g	653 g
1AW-00-28	S00T001485	664 g	675 g
18316	S00T001660	664 g	677 g
18318	S00T001661	649 g	666 g
	Total	19780 g	20125 g

3.0 Homogeneity Testing and Sub-Sampling

The 40 L carboy was equipped with a mechanical stirrer and a steel dip leg (3/8" ID) connected with tubing (3/8" ID) to a peristaltic pump for sampling purposes. The steel dip leg could be raised or lowered to collect sample from any height in the carboy. A homogeneity test was conducted to ensure that the agitation and sampling system could provide representative samples of the slurry. Twelve 225 mL sub-samples were obtained and collected in graduated cylinders. The sub-samples were obtained from either the top, bottom, or midpoint of the original sample height in the 40 L carboy. Concurrently a 200 mL sub-sample for chemical and physical characterization and a 500 mL sub-sample for use in the dilution study to 7 M sodium were also obtained. After settling for 24 hours the volume of settled solids in each of the 12 graduated cylinders was recorded. Table 3.1 shows the results of the homogeneity test on the as-received 241-AW-101 sample. The average volume percent settled solids of the twelve sub-samples was 5.7 with a percent relative standard deviation of 7 %. The data collected indicated the sampling system provided representative sub-samples independent of sampling height or sampling order. One of the twelve 225 mL sub-samples, one was kept as an archive sample. The remaining eleven 225 mL sub-samples were returned to the carboy for bulk dilution of the sample to 5 M sodium.

Table 3.1. Results of the Homogeneity Test for As-Received 241-AW-101.

Cylinder No.	Sampling Height	Volume of Sample (mL)	Volume of Settled Solids* (mL)	Volume Percent Solids
1	Midpoint	220	13	5.91%
2	Top	217	14	6.45%
3	Bottom	218	13	5.96%
4	Midpoint	220	12	5.45%
5	Top	220	12	5.45%
6	Bottom	217	12	5.53%
7	Midpoint	231	14	6.06%
8	Top	234	12	5.13%
9	Bottom	218	12	5.50%
10	Midpoint	228	14	6.14%
11	Top	226	12	5.31%
12	Bottom	232	13	5.60%

* The solids in each cylinder were composed of a layer of white crystalline solids with a thin layer (~4 mL) of darker colored solids on top after settling for 24 hours.

4.0 Bulk Dilution to 5 M Sodium and Sampling of the Diluted 241-AW-101 Sample

The as-received 241-AW-101 sample was diluted with deionized distilled water to provide a sodium concentration in the supernate of 5 M. The volume of water required to dilute 12.6 L of the as-received AW-101 sample to a 5 M sodium concentration was calculated based on the assumption of no dissolution of the solids in the sample. Since a large percentage of the solids were expected to dissolve during the dissolution, the calculation provided a measure of conservatism to ensure the target sodium concentration would not be overshoot. A volume of 11.7 L of de-ionized distilled water was added to the sample while mixing and allowed to equilibrate for 24 hours. The slurry was sampled and the filtered supernate analyzed indicating the sodium concentration had dropped to ~7.3 M. A second calculation indicated another 8.7 L of water would be required to reach the 5 M sodium endpoint. After adding the additional 8.7 L water a filtered supernate sample was analyzed and indicated a sodium concentration of 4.5 M. The 5 M sodium target concentration was overshoot due to erroneous analytical data after the first dilution. After consultation with the BNI R&T representative, the sodium concentration was brought back up to 5 M by vacuum distilling (at 55°C) 10.8 L of the solution to a volume of 5.04 L. The concentrated solution was added back to the bulk diluted sample. Analysis of the filtered supernate indicated a final sodium concentration of 4.95 M. The final sample volume after dilution to a 5 M sodium concentration was approximately 27.2 L.

The volume percent solids of the 5 M sodium diluted sample were determined to be much less than 2 volume percent. Therefore, after consultation with the BNI R&T representative, a homogeneity test was not required prior to obtaining sub-samples of the 5 M diluted 241-AW-101. The bulk diluted 241-AW-101 was sampled after mixing for 1 hour. A 220 mL sample was obtained for characterization and a second 220 mL sample archived.

Due to the low entrained solids content (~0.1 wt%) of the diluted 5 M sodium 241-AW-101, the decision was made, after consultation with the BNI R&T representative, to allow the CUF unit testing to proceed and obtain a portion of the concentrated solids for analysis at completion of the testing. Due to a mishap in the CUF testing less than 0.5 g of solids were available for analysis. There were only sufficient solids to allow a single sample digestion method instead of the normal two methods.

5.0 Sample Preparation for Analysis

5.1 Preparation of Samples for the Analysis of Liquid Fractions

Samples of the supernate were obtained by vacuum filtering a portion of the sample through a 0.45μ Nylon filter disc. Portions of the filtered supernate were diluted with de-ionized distilled water or nitric acid to reduce the sample activity and allow removal from the Shielded Cells for chemical characterization. Generally a 15 - 20 fold dilution was required on supernate samples to meet the dose limits for the analytical laboratories. The weight of the solids collected was recorded to calculate the weight percent of damp vacuum filtered solids. Solids collected on the filter were not washed to remove interstitial supernate. All sample preparations of the filtered supernate samples were conducted in triplicate. Supernate samples were analyzed for species listed in Table 2 of the test specification. Section 7.0 presents the results of the analysis of the 241-AW-101 sample.

A blank was prepared concurrently with the sample preparations substituting de-ionized distilled water in place of the sample aliquot.

Additional sample preparations specific to individual analytical methods were conducted by the Analytical Development Section on the samples removed from the Shielded Cells as necessary.

5.2 Preparation of Samples for the Analysis of Solids Fractions

Samples of the solids fraction were prepared for analysis by aqua-regia digestion and fusion with sodium peroxide followed by uptake in hydrochloric acid. A water contact of the unwashed, damp, as-received solids collected by vacuum filtering a portion of the sample through a 0.45μ Nylon filter disc was also conducted to allow analysis of water soluble species present in the solids. The water contact analysis was provided as additional information.

For the aqua-regia digestion and fusion with sodium peroxide methods, solids were collected by vacuum filtering a portion of the sample through a 0.45μ Nylon filter disc. Solids collected on the filter were not washed to remove interstitial supernate. The solids collected were dried at 100° C until the dry weight of the solids remained stable (i.e., less than 5 mg change in weight between measurements). Weighed portions of the dried solids were digested and diluted to reduce activity and allow removal from the Shielded Cells. Sample preparation for some radiochemical analyses, such as C^{14} and I^{129} , were conducted in the Shielded Cells with the undigested dry solids prior to removal for analysis. All sample preparations were conducted in triplicate. Samples with a volume percent solids content of >2 vol % were analyzed for species listed in Table 2 of the test specification. Samples with a volume percent solids content of <2 vol % were analyzed for species listed in Table 3 of the Test Specification. Section 7.0 presents the results of the analysis of the 241-AW-101 sample.

Duplicate digestions of a glass standard containing many of the elements found in tank samples were prepared concurrently with the sample preparations. Table 5.1 lists the composition of the Analytical Reference Glass-1 (ARG-1) glass standard.⁴ A blank was prepared concurrently with the sample preparations consisting of the digestion reagents and incorporated any manipulations and dilutions conducted on the sample.

Additional sample preparations specific to individual analytical methods were conducted by the Analytical Development Section on the samples removed from the Shielded Cells as necessary.

5.3 Dose Rate Limits for Analytical Laboratories

Samples sent to the analytical laboratories require dose rates of <10 mrem/hr whole body dose and <1000 mrem/hr extremity dose. In order to avoid exceeding the dose rate limits and the need for re-preparing the samples, dilutions were made targeting a whole body dose rate of between 2 - 5 mrem/hr. This working dose rate range provides some cushion against the unavoidable presence of contamination on the outside of the sample bottles. Additionally, sample handling in the cells, such as pipetting, weighing, or transferring from one container to another invariably leads to some contamination of the sample. Efforts to minimize the level of contamination include regular cleaning of the manipulator fingers, cleaning of cell surfaces, and the use of clean supplies. Blanks prepared in the Shielded Cells in the same manner as the sample provide some indication of the level of contamination introduced.

Table 5.1. Composition of the Analytical Reference Glass-1 Standard.

Element	Wt% in Glass
Al	2.50%
B	2.69%
Ba	0.079%
Ca	1.02%
Cr	0.064%
Cu	0.003%
Fe	9.79%
K	2.26%
Li	1.49%
Mg	0.52%
Mn	1.46%
Na	8.52%
Ni	0.83%
P	0.11%
Si	22.4%
Sr	0.003%
Ti	0.69%
Zn	0.016%
Zr	0.096%

6.0 Density and Weight Percent Solids Measurements

The weight percent total solids in the samples were measured in the Shielded Cells using a conventional drying oven at 100 °C. The weight percent dissolved solids in a sample of the filtered supernate were measured in the same manner. The weight percent insoluble solids were measured by filtering a known weight of the sample and drying the solids using a conventional drying oven at 100 °C. The weight percent vacuum filtered solids were measured by filtering a known weight of the sample and determining the weight of damp solids recovered. All filtrations were made through a 0.45 μ Nylon membrane. All measurements were made in triplicate.

Wt% dissolved solids	$(\text{wt dissolved solids}/\text{wt of supernate}) \times 100$
Wt% total solids	$(\text{wt total solids}/\text{wt of total sample}) \times 100$
Wt% insoluble solids	$(\text{wt insoluble solids}/\text{wt of total sample}) \times 100$
Wt% vacuum filterable solids	$(\text{wt of damp solids}/\text{wt of total sample}) \times 100$

Density measurements were made in the Shielded Cells on both the total sample and the filtered supernate. The temperature in the Shielded Cells varies between ~18 - 25 °C depending on the temperature outside the building. The temperature was recorded for all density measurements and noted in the tables of analytical results.

7.0 Analytical Results and Data Evaluation

7.1 General Information

Tables 7.1 through 7.11 provide the chemical and radiochemical composition of the , as-received and 5 M diluted samples of the 241-AW-101 tank waste sample received at SRTC. The tables include the results of all replicates, blanks, laboratory control and matrix spike recoveries, and quality control flags within each table to allow easy identification of data quality. Analytical results for liquid samples use units of mg/L or mCi/L while results for solids from the sample use mg/Kg or mCi/Kg units. The following list identifies the data tables included in this section with a brief description of the sample analyzed:

- Table 7.1 Abbreviations of analytical methods used for each analysis.
- Table 7.2 Physical properties (density and wt% solids measurements) of the as-received 241-AW-101 sample.
- Table 7.3 Chemical (inorganic and organic) and radiochemical composition of the filtered supernate from the as-received 241-AW-101 sample.
- Table 7.4 Physical properties (density and wt% solids measurements) of the diluted (5 M sodium) 241-AW-101 sample.
- Table 7.5 Chemical (inorganic and organic) and radiochemical composition of the filtered supernate from the diluted (5 M sodium) 241-AW-101 sample.
- Table 7.6 Chemical (inorganic) and radiochemical composition of the solids filtered from the as-received 241-AW-101 sample. The samples were digested using aqua-regia prior to analysis.
- Table 7.7 Chemical (inorganic) and radiochemical composition of the solids filtered from the as-received 241-AW-101 sample. The samples were digested using a sodium peroxide fusion method prior to analysis.
- Table 7.8 Chemical (inorganic) and radiochemical composition of the solids filtered from the diluted (5 M sodium) 241-AW-101 sample. The samples were digested using aqua-regia prior to analysis.
- Table 7.9 Chemical (inorganic and organic) composition of the solids filtered from the as-received 241-AW-101 sample. The samples were contacted with water prior to analysis. Provided for information only.
- Table 7.10 Chemical (inorganic) composition of the first condensate collected during the concentration of the over-diluted 5 M sodium 241-AW-101 sample. Provided for information only.
- Table 7.11 Chemical (inorganic) composition of the last condensate collected during the concentration of the over-diluted 5 M sodium 241-AW-101 sample. Provided for information only.

7.2 QC Flags

The analytical results in tables include a quality control (QC) flag in the last column to indicate potential problems with the data and failure to meet the quality control requirements

stated in the test specification. The test specification and approved task plan for the characterization of the 241-AW-101 samples set requirements for the minimum reportable quantity (MRQ), the percent relative standard deviation (%RSD) for the three replicate samples, the percent recovery of the laboratory control standard (LCS), and the percent recovery of the matrix spike (MS) in the analysis of each analyte. In addition analyses in which the concentration of the analyte in the blank exceeded 5% of the concentration in the sample were flagged. Results where the analyte concentration was less than ten times the estimated detection limit (DL) for the analyte were also flagged. The DL for the analyte was determined based on the analysis of high purity standards. Values less than ten times the DL typically have greater uncertainty. The following defines the QC flags used in the tables:

- None Analyte meets all QC requirements.
- U_M Analyte does not meet the MRQ requirement.
- U_R Analyte does not meet the %RSD requirement.
- U_L Analyte does not meet the percent recovery of the LCS requirement.
- U_S Analyte does not meet the percent recovery of the MS requirement.
- U_B Analyte concentration in the blank exceeds 5% of the concentration measured in the sample.
- U_E Analyte concentration measured at less than ten times the DL for the sample.

In general, most of the QC failures can be attributed to low concentrations present in the sample or to low concentrations in the analytical samples due to the dilution necessary to allow removal of the samples from the Shielded Cells for analysis in the ADS laboratory hoods. In particular, meeting the MRQ requirement depends heavily on the dilution required for handling the sample. In other cases, for example digestion of solid samples, extensive sample preparation in the Shielded Cells lead to contamination of the sample as evidenced by significant concentrations of radionuclides in the blanks and standards. The presence of contamination in the sample generally lead to large %RSD for the replicate analyses. With the low concentration of many of the radionuclides in the sample, a small amount of contamination leads to large errors in the measurement.

7.3 Data Evaluation

The following discussion provides further explanation and evaluation of the data quality in Tables 7.2 through 7.11.

Table 7.2. (Properties of the As-Received 241-AW-101 Sample)

- The data for the weight percent vacuum filterable solids and the weight percent insoluble solids show high percent relative standard deviations for the replicate measurements. Both measurements have inherently low precision owing to the presence of variable amounts of interstitial supernate trapped in the solids. In both measurements, the solids can not be washed free of interstitial supernate because a high percentage of the solids would dissolve.

Table 7.3. (Composition of the As-Received 241-AW-101 Filtered Supernate)

- Many of the elements from the ICP-ES analysis (IE designator in table) were flagged as U_E since the values were less than 10 times the detection limits. Li and Ce were flagged as U_M for not meeting the required MRQ. These failures were attributed to low concentrations present in the sample and the dilutions necessary to allow removal from the Shielded Cells for analysis.
- Missing the matrix spike recovery for nitrite. As the first sample analyzed for the task the analyst assumed a nitrite spike would be sufficient to cover both nitrite and nitrate.
- The organic acids and complexants were flagged as not meeting the required MRQ. As stated in the task plan, these analytes were provided as information only since there was insufficient method data to set QC parameters. The QC parameters were provided as target values.
- The Eu^{154} , Eu^{155} , Co^{60} , Sn^{126} , and Pa^{231} from gamma spectrometry were flagged as not meeting the required MRQ.
- For details on missing LCS and matrix spike recovery's for ICP-MS analysis (IM in tables) for W, I^{127} , Np^{237} , Pu^{239} , Pu^{240} , U^{233} , U^{234} and U^{236} see the description in section 9.8. Although the LCS contains Cs (mass 133) no mass response was obtained from the analysis.
- The Pu^{238} , and $Pu^{239/240}$ results from the separation/alpha spectroscopy analysis were flagged as U_R . The percent relative standard deviations were slightly higher than the required 15% principally due to the low concentrations present in the sample. A re-analysis obtained poorer percent relative standard deviations for these analytes.
- The $Cm^{243/244}$ was flagged as U_B due to the presence of contamination present in the blank at levels greater than 5% of the sample concentration.
- High percent relative standard deviations were found in the actinide mass region (mass 232 to 244) of the ICP-MS analysis. The samples were re-analyzed but the results showed no significant improvement.
- The alpha counting results show high scatter attributed to the concentrations in the sample being near the detection limits of the method.
- The sodium results by ICP-ES and AA show good agreement. With the sodium concentration measured by the ICP-ES corrected for the high bias shown by the percent recoveries of the LCS and MS the difference between the two methods drops to less than 2%.
- Good agreement was also obtained for the cation/anion balance. Using the sodium and potassium values from AA for the cation concentration yields 10.5 M. Summing the

concentrations of free hydroxide, Al from ICP-ES, TIC calculated as a carbonate concentration, and the anions obtained from IC produces an anion concentration of 11.1 M.

- The sulfur concentrations obtained from IC (as sulfate) and ICP-ES show reasonable agreement with a difference of ~20% based on molar concentrations of sulfur.
- The phosphorus analysis by ICP-ES yielded a result approximately two times the concentration measured by IC (as phosphate).
- The potassium values obtained by ICP-ES and AA show a difference of approximately 12%.
- The sum of the formate and oxalate concentrations by IC only equate to ~35% of the TOC result indicating the presence of other sources of organic carbon in the sample. However, the analysis of organic acid, complexants, and degradation products by IC, HPLC, and GC-MS did not find measurable quantities although the detection limits for the analyses were not very low.
- The uranium concentration by Chemchek and ICP-MS show an order of magnitude (10X) difference. Although the recoveries for the LCS and MS were within the test specification requirements, the ICP-MS data for uranium masses all have very high percent relative standard deviations for the replicate samples. The samples were re-analyzed but the results showed no significant improvement.
- The Cs¹³⁷ concentration measured by gamma spectroscopy and ICP-MS show good agreement with a difference of approximately 7%. The Tc⁹⁹ measured as pertechnetate and the total Tc⁹⁹ measured by ICP-MS also differ by ~7% indicating all or nearly all the technetium is in the pertechnetate form.
- The actinide region of the ICP-MS data shows high scatter for the replicate samples
- The I¹²⁷ value from ICP-MS is unreliable due to memory effects.

Table 7.4. (Properties of the Diluted 5 M Sodium 241-AW-101 Sample)

- The low volume of solids in the sample precluded replicate analyses of the weight percent vacuum filtered solids and weight percent insoluble solids.

Table 7.5. (Composition of the Diluted 5 M Sodium 241-AW-101 Filtered Supernate)

- The diluted 5 M sodium values, in general, are approximately one-half the concentrations measured in the as-received supernate as expected. However, the large amount of undissolved salt present in the as-received sample makes comparison of the analytical results of the 5 M sodium supernate to the original as-received supernate problematic. During dilution, the majority of the salts in the as-received sample dissolve negating the use of simple dilution models.

- Many of the elements from the ICP-ES analysis (IE designator in table) were flagged as U_E since the values were less than 10 times the detection limits. Li and Ce were flagged as U_M for not meeting the required MRQ. These failures were attributed to low concentrations present in the sample and the dilutions necessary to allow removal from the Shielded Cells for analysis.
- Some of the organic acids and complexants were flagged as not meeting the required QC. As stated in the task plan, these analytes were provided as information only since there was insufficient method data to set QC parameters. The QC parameters were provided as target values.
- The results of the Hg analysis were below detection limits and flagged for not meeting the required MRQ. The failure to meet the MRQ was attributed to the low concentration in the sample and the dilutions necessary to allow removal from the Shielded Cells for analysis.
- The Pa^{231} from gamma spectrometry were flagged as not meeting the required MRQ.
- For details on missing LCS and matrix spike recovery's for ICP-MS analysis (IM in tables) for W, I^{127} , Np^{237} , Pu^{239} , Pu^{240} , U^{233} , U^{234} and U^{236} see the description in section 9.8.
- The C^{14} result was flagged due to the high percent relative standard deviation. The failure to meet the %RSD requirement was attributed to low concentrations present in the sample and the dilutions necessary to allow removal from the Shielded Cells for analysis.
- The $Pu^{239/240}$ result from the separation/alpha spectroscopy analysis was flagged as U_R . The percent relative standard deviations were higher than the required 15% principally due to the low concentrations present in the sample.
- The sodium results by ICP-ES and AA show good agreement with a difference between the two methods of less than 5%.
- Good agreement was obtained for the cation/anion balance. Using the sodium and potassium values from AA for the cation concentration yields 5.2 M. Summing the concentrations of free hydroxide, Al from ICP-ES, TIC calculated as a carbonate concentration, and the anions obtained from IC produces an anion concentration of 5.57 M.
- The phosphorus and sulfur concentrations obtained from IC (as sulfate and phosphate) were approximately one half the concentrations measured by ICP-ES.
- The potassium values obtained by ICP-ES and AA show a difference of approximately 20%.

- The alpha counting results were flagged due to the failure of the matrix spike recovery.
- The sum of the formate and oxalate concentrations by IC only equate to ~60% of the TOC result indicating the presence of other sources of organic carbon in the sample. The addition of the organic acids, complexants, and degradation products measured by IC, HPLC, and GC-MS accounts for 85% of the TOC.
- The uranium concentration by Chemchek and ICP-MS show more than an order of magnitude (10X) difference. The uranium values from the Chemchek method show a percent relative standard deviation slightly higher than the 15% requirement. The scatter in the data was attributed to low concentrations present in the sample and the dilutions necessary to allow removal from the Shielded Cells for analysis.
- The Cs¹³⁷ concentration measured by gamma spectroscopy and ICP-MS show good agreement with a difference of approximately 2.5%. The isotopic ratios of Cs¹³³:Cs¹³⁵:Cs¹³⁷ agree well with the ratios from the as-received supernate results.
- The Tc⁹⁹ measured as pertechnetate and the total Tc⁹⁹ measured by ICP-MS differ by ~18% indicating some of the Tc⁹⁹ may be in a non-pertechnetate form.
- The I¹²⁷ value from ICP-MS is unreliable due to memory effects.

Table 7.6. (Composition of the Aqua-Regia Digested As-Received 241-AW-101 Filterable Solids)

- Many of the elements from the ICP-ES analysis (IE designator in table) were flagged as U_E since the values were less than 10 times the detection limits. B and Ce were flagged as U_M for not meeting the required MRQ. Several of the elements were flagged for not meeting the required %RSD, however, in most cases the values were also less than 10 times the detection limits. These failures were attributed to low concentrations present in the sample and the dilutions necessary to allow removal from the Shielded Cells for analysis. The Fe results were flagged for not meeting the %RSD requirement.
- The sodium result by AA was flagged for a LCS recovery slightly below the required 90%.
- The results of the Hg analysis were below detection limits and flagged for not meeting the required MRQ. The failure to meet the MRQ was attributed to the low concentration in the sample and the dilutions necessary to allow removal from the Shielded Cells for analysis.
- For details on missing LCS and matrix spike recovery's for ICP-MS analysis (IM in tables) for W, I¹²⁷, Np²³⁷, Pu²³⁹, Pu²⁴⁰, U²³³, U²³⁴ and U²³⁶ see the description in section 9.8. Although the LCS contains Cs (mass 133) no mass response was obtained from the

analysis. The matrix spike only contained U so no other matrix spike recovery's were available.

- The Pa²³¹ from gamma spectrometry was flagged as not meeting the required MRQ.
- The Se⁷⁹ result was flagged for not meeting the required MRQ. The failure was attributed to low concentration present in the sample and the dilutions necessary to allow removal from the Shielded Cells for analysis.
- The sodium results by ICP-ES and AA show a difference between the two methods of ~50%. A poor recovery was obtained for sodium with AA method. Also, the glass standards show a low bias for sodium in the AA.
- The potassium values obtained by ICP-ES and AA show a difference of approximately 17%.
- The C¹⁴ result was flagged due to the high percent relative standard deviation. The failure to meet the %RSD requirement was attributed to low concentrations present in the sample and the dilutions necessary to allow removal from the Shielded Cells for analysis. For one replicate the sample preparation failed and the analysis was not completed.
- The I¹²⁹ result was flagged for not meeting the MRQ. The failure to meet the MRQ requirement was attributed to low concentrations present in the sample and the dilutions necessary to allow removal from the Shielded Cells for analysis.
- The Cs¹³⁷ concentration measured by gamma spectroscopy and ICP-MS show a difference of ~5x. The values for Cs¹³⁵ and Cs¹³⁷ from the ICP-MS have a high bias due to the significant Ba concentration in the solids and should not be considered reliable. The results for mass 133 may be biased high also due to the presence of a significant interference or contamination in the glass standard. The glass standards containing Ba show the expected values at mass 135 and 137 based on natural abundance.

Using the Cs isotopic ratios from the as-received and diluted 5 M supernate data of 3.2:1:1.4 (Cs¹³³:Cs¹³⁵:Cs¹³⁷) and the Cs¹³³ result from ICP-MS, the estimated value for Cs¹³⁷ (97 mCi/Kg) agrees reasonably well with the value for Cs¹³⁷ measured by gamma spectroscopy (77 mCi/Kg). A better estimate for the Cs¹³³ and Cs¹³⁵ concentrations can be obtained by using the Cs isotopic ratios from the as-received and diluted 5 M supernate data of 3.2:1:1.4 (Cs¹³³:Cs¹³⁵:Cs¹³⁷) and the Cs¹³⁷ value measured by gamma spectroscopy (0.89 mg/Kg). This calculation yields Cs¹³³ and Cs¹³⁵ concentrations of 2.0 mg/Kg and 0.64 mg/Kg respectively.

- Based on the ICP-MS data the Ba concentration measured by ICP-ES would seem to be significantly too high. The Ba concentration by ICP-ES has been flagged as being less than 10 times the minimum detection level.

- The total Tc⁹⁹ measured by ICP-MS was found to be ~30% higher than the Tc⁹⁹ measured as pertechnetate. As a result of the aqua-regia digestion both methods should provide a measure of the total Tc⁹⁹. However, some of the non-pertechnetate form present in the solids may not be converted to pertechnetate by the digestion which could lead to a low bias in the counting method.
- The uranium concentration measured by three methods show a high degree of scatter among the values (4.64E+01 mg/Kg by Chemchek, 2.05E+03 mg/Kg by ICP-ES, and 3.19E+02 mg/Kg by ICP-MS). The ICP-ES results shows a large concentration of uranium in the glass standard which does not contain uranium. The ICP-MS and Chemchek methods show insignificant quantities of uranium in the glass standard. The Chemchek result was also flagged for a %RSD slightly higher than the required 15%.
- The low concentrations of plutonium isotopes measured by alpha spectroscopy show high scatter, significant levels of contamination in the blanks and glass standards, and magnitudes less than ten times the minimum detection level for the method. The ICP-MS results for mass 239 and 240 also obtained values less than ten times the minimum detection level for the method. These QC failures were attributed to low concentrations present in the sample and the dilutions necessary to allow removal from the Shielded Cells for analysis.
- The Cm^{243/244} show significant levels of contamination in the blanks and glass standards. The results were flagged as U_R, U_B, and U_G. The Cm²⁴² results were less than 10 times the minimum detection level. These failures were attributed to low concentrations present in the sample and the dilutions necessary to allow removal from the Shielded Cells for analysis.

Table 7.7. (Composition of the Sodium Peroxide Fusion Digested As-Received 241-AW-101 Filterable Solids)

- Many of the elements from the ICP-ES analysis (IE designator in table) were flagged as U_E since the values were less than 10 times the detection limits. B, Ce, and Li were flagged as U_M for not meeting the required MRQ. Several of the elements were flagged for not meeting the required %RSD, however, in most cases the values were also less than 10 times the detection limits. These failures were attributed to low concentrations present in the sample and the dilutions necessary to allow removal from the Shielded Cells for analysis. The Al and Fe results were flagged for not meeting the %RSD requirement.
- For details on missing LCS and matrix spike recovery's for ICP-MS analysis (IM in tables) for W, I¹²⁷, Np²³⁷, Pu²³⁹, Pu²⁴⁰, U²³³, U²³⁴ and U²³⁶ see the description in section 9.8. Although the LCS contains Cs (mass 133) no mass response was obtained from the analysis. The matrix spike only contained Th and U so no other matrix spike recovery's were available.

- The Eu^{154} , Eu^{155} , Co^{60} from gamma spectrometry were flagged for contamination present in the blank and glass standard samples.
- The Pa^{231} from gamma spectrometry was flagged for not meeting the required MRQ.
- The Se^{79} result was flagged for not meeting the required MRQ. The failure was attributed to low concentration present in the sample and the dilutions necessary to allow removal from the Shielded Cells for analysis.
- The potassium values obtained by ICP-ES and AA show a difference of approximately 9%. The values reasonably good agreement with those obtained from the aqua-regia digestion.
- As observed in the aqua-regia digestions of the as-received solids, the Cs^{137} concentration measured by gamma spectroscopy and ICP-MS show a difference of $\sim 5\times$. The values for Cs^{135} and Cs^{137} from the ICP-MS have a high bias due to the significant Ba concentration in the solids and should not be considered reliable. The value for the 2nd replicate at mass 133 shows a significantly higher concentration than the other two replicates leading to a high percent relative standard deviation. The results for mass 133 may be biased high also due the presence of a significant interfering ion or contamination in the glass standard. The glass standards containing Ba show the expected values at mass 135 and 137 based on natural abundance.

An estimate for the Cs^{133} and Cs^{135} concentrations can be obtained by using the Cs isotopic ratios from the as-received and diluted 5 M supernate data of 3.2:1:1.4 ($\text{Cs}^{133}:\text{Cs}^{135}:\text{Cs}^{137}$) and the Cs^{137} value measured by gamma spectroscopy (0.93 mg/Kg). This calculation yields Cs^{133} and Cs^{135} concentrations of 2.1 mg/Kg and 0.66 mg/Kg respectively.

- Based on the ICP-MS data the Ba concentration measured by ICP-ES would seem to be significantly too high. The Ba concentration by ICP-ES has been flagged as being less than 10 times the minimum detection level.
- The total Tc^{99} measured by ICP-MS was found to be $\sim 22\%$ higher than the Tc^{99} measured as pertechnetate. As a result of the digestion both methods should provide a measure of the total Tc^{99} . However, some of the non-pertechnetate form present in the solids may not be converted to pertechnetate by the digestion which could lead to a low bias in the counting method. The measurements from the sodium peroxide fusion digestion agree well with those from the aqua-regia digestion.
- The uranium concentration measured by three methods shows a high degree of scatter among the values (5.05E+02 mg/Kg by Chemchek, 3.47E+04 mg/Kg by ICP-ES, and 1.58E+02 mg/Kg by ICP-MS). The ICP-ES and ICP-MS results shows significant concentrations of uranium in the blank and in the glass standard which does not contain uranium. The Chemchek methods shows a high percent relative standard deviation. The

measurements from the sodium peroxide fusion digestion show poor agreement with those from the aqua-regia digestion.

- The low concentrations of plutonium isotopes measured by alpha spectroscopy show high scatter, significant levels of contamination in the blanks and glass standards. The ICP-MS results for mass 239 obtained values less than ten times the minimum detection level for the method. ICP-MS results for mass 240 was below detection limits and was flagged for not meeting the required MRQ. These QC failures were attributed to low concentrations present in the sample and the dilutions necessary to allow removal from the Shielded Cells for analysis. The measurements from the sodium peroxide fusion digestion show poor agreement with those from the aqua-regia digestion.
- The Cm^{243/244} show significant levels of contamination in the blanks and glass standards. The results were flagged as U_B and U_G. The Cm²⁴² results were less than 10 times the minimum detection level. These QC failures were attributed to low concentrations present in the sample and the dilutions necessary to allow removal from the Shielded Cells for analysis.

Table 7.8. (Composition of the Aqua-Regia Digested Diluted 5 M Sodium 241-AW-101 Filterable Solids)

- S and U from the ICP-ES analysis (IE designator in table) were flagged as U_E since the values were less than 10 times the detection limits. These results were attributed to low concentrations present in the sample and the dilutions necessary to allow removal from the Shielded Cells for analysis.
- The TIC results were flagged for not meeting the required MRQ. Due to the acid digestion method, any carbonate present will be released as CO₂ making measurement of TIC difficult.
- The sodium results by ICP-ES shows a slightly high percent relative standard deviation.
- The glass standards show values sodium and phosphorus approximately 50% low based on the expected composition.
- The Eu¹⁵⁴, Eu¹⁵⁵, Co⁶⁰ from gamma spectrometry were flagged for contamination present in the blank. The average values were calculated from replicates 2 and 3 only. The first replicate contained high levels of europium isotopes producing a large dead time. The values for the first replicate were marked as qualitative do to the high dead time.
- The uranium concentration measured by ICP-ES (4.22E+03 mg/Kg) and ICP-MS (4.10E+03 mg/Kg) agree within 3% although the ICP-ES and mass 235 results show values less than ten times the minimum detection level for the sample. The Chemchek method shows an order of magnitude lower result (5.11E+02 mg/Kg). The ICP-ES results shows a large concentration of uranium in the glass standard which does not contain uranium. The Chemchek method was flagged for not meeting the required %RSD.

- For details on missing LCS and matrix spike recovery's for ICP-MS analysis (IM in tables) for Pu²³⁹, Pu²⁴⁰, U²³³, U²³⁴ and U²³⁶ see the description in section 9.8. The matrix spike used the LCS for these samples.
- The low concentrations of plutonium isotopes measured by alpha spectroscopy show some scatter and significant levels of contamination in the glass standards. However, the Pu^{239/240} from alpha spectroscopy and the ICP-MS results for mass 239 and 240 agree reasonably well (~17% difference). The ICP-MS results do not show contamination in the glass standards at mass 239 and 240.

Table 7.9. (Composition of the Water Contact of the As-Received 241-AW-101 Filterable Solids)

- The data is for information only. The replicate samples show high percent relative standard deviations as a result of the incomplete digestion of the sample, difficulties with sampling damp solids reproducibly, and the potential for variable amounts of interstitial supernate incorporated into the solids.

Table 7.10. (Composition of the 1st Condensate from the Concentration of the Diluted 5 M Sodium 241-AW-101 Sample)

- The data is for information only.

Table 7.11. (Composition of the 4th Condensate from the Concentration of the Diluted 5 M Sodium 241-AW-101 Sample)

- The data is for information only.

7.4 Data Tables

All blank cells were filled with dash (-) to indicate the cell was intentionally left empty.

Table 7.1. Abbreviations for Analytical Methods in Tables 7.2 through 7.11

Analytical Method	Abbreviation in Tables	ADS Procedure No.
Ion Chromatography	IC	ADS-2306
Ammonia Purge and Trap	PT	ADS-2306
Titration	T	ADS-1206 Rev. 1
ICP-AES	IE	ADS-1564
ICP-MS	IM	ADS-1543
AA	AA	ADS-1554 Rev. 3
Calc. By Difference	Diff	NA
Acidification	A	ADS-1206 Rev. 1
Chemchek (Uranium)	CC	ADS-2236 Rev. 4
Gamma Spec.	GS	ADS-2420
Separation/Gamma Spec.	SG	ADS-2420
Separation/Alpha Spec.	SA	ADS-2453 ADS-2449
Separation/Liquid Scintillation	SL	ADS-2447 ADS-2444 ADS-2407
Alpha Counting	AC	ADS-2402
HPLC	HL	ADS-2660
GC-MS	GM	ADS-2661

Table 7.2. Properties of the As-Received 241-AW-101 Sample

Property	Units	1st Replicate	2nd Replicate	3rd Replicate	Average	%RSD	QC Flag
Density of Filtered Supernate	g/mL	1.41	1.41	1.41	1.41	0.0%	-
Density of Slurry	g/mL	1.51	1.50	1.49	1.50	0.7%	-
Wt% Vacuum Filtered Solids	Wt%	1.04%	2.67%	-	1.86%	62%	U _R
Wt% Dissolved Solids	Wt%	57.3%	58.2%	59.1%	58.2%	1.6%	-
Wt% Total Solids	Wt%	54.7%	56.4%	56.9%	56.0%	2.0%	-
Wt% Insoluble Solids (measured)	Wt%	0.65%	1.87%	-	1.26%	68%	U _R

QC Flags: none - meets all QC

U_B - blank exceeds 5% of sample concentration

U_E - value less than 10x the DL

U_R - fails %RSD criteria

U_L - fails % Recovery of LCS

ND - not detected

U_M - fails minimum MRQ criteria

U_S - fails % Recovery of MS

N/A - not applicable

Table 7.3. Composition of the As-Received 241-AW-101 Filtered Supernate

Analyte	1st Replicate (mg/L)	2nd Replicate (mg/L)	3rd Replicate (mg/L)	Average (mg/L)	%RSD	Blank (mg/L)	LCS % Recovery	MS % Recovery	QC Flag
NO ₃ ⁻ (IC)	1.14E+05	1.16E+05	1.23E+05	1.18E+05	4.1%	2.70E+01	100%	97%	-
NO ₂ ⁻ (IC)	9.12E+04	9.18E+04	9.20E+04	9.17E+04	0.5%	<1.0E+01	98%	-	-
PO ₄ ³⁻ (IC)	2.89E+02	2.21E+02	2.44E+02	2.51E+02	13.8%	<1.0E+01	100%	100%	-
SO ₄ ²⁻ (IC)	3.37E+02	3.19E+02	3.17E+02	3.24E+02	3.4%	<5.0E+00	100%	96%	-
C ₂ O ₄ ²⁻ (IC)	7.22E+01	7.36E+01	7.31E+01	7.29E+01	1.0%	<1.0E+01	101%	98%	-
Cl ⁻ (IC)	6.88E+03	6.35E+03	6.36E+03	6.53E+03	4.6%	3.00E+00	101%	97%	-
F ⁻ (IC)	3.37E+02	3.19E+02	2.93E+02	3.16E+02	7.0%	<2.0E+00	102%	99%	-
CHO ₂ ⁻ (IC)	3.44E+03	3.26E+03	3.29E+03	3.33E+03	2.9%	<1.0E+01	100%	97%	-
NH ₄ ⁺ (PT)	3.13E+02	3.19E+02	3.17E+02	3.16E+02	1.0%	<1.0E+01	102%	75%	-
OH _{free} ⁻ (T)	7.48E+04	7.15E+04	7.10E+04	7.24E+04	2.8%	<3.4E+02	N/A	N/A	-
OH _{total} ⁻ (T)	1.04E+05	1.03E+05	1.00E+05	1.03E+05	2.0%	<3.4E+02	101%	N/A	-
TIC (A)	1.10E+03	1.14E+03	1.13E+03	1.13E+03	1.8%	<1.0E+00	98%	110%	-
TOC (Diff)	2.52E+03	2.46E+03	2.86E+03	2.61E+03	8.4%	1.67E+01	N/A	N/A	-
Al (IE)	5.07E+04	5.11E+04	5.09E+04	5.09E+04	0.4%	<1.3E-01	97%	N/A	-
B (IE)	3.44E+01	3.38E+01	3.31E+01	3.38E+01	1.9%	<1.1E-01	102%	N/A	U _E
Ba (IE)	2.08E+00	2.18E+00	1.99E+00	2.08E+00	4.7%	<7.6E-02	102%	102%	U _E
Ca (IE)	9.74E+00	1.01E+01	9.26E+00	9.71E+00	4.5%	<1.0E-02	99%	116%	U _E
Cd (IE)	2.81E+00	2.45E+00	2.51E+00	2.59E+00	7.5%	<1.7E-02	103%	N/A	U _E
Cr (IE)	9.53E+01	9.30E+01	9.16E+01	9.33E+01	2.0%	<2.4E-02	102%	91%	U _E
Fe (IE)	4.98E+00	4.18E+00	6.57E+00	5.25E+00	23%	<1.4E-02	102%	92%	U _E
La (IE)	2.52E+00	2.55E+00	2.25E+00	2.44E+00	6.9%	4.00E-02	99%	N/A	U _E
Li (IE)	<4.1E+00	<4.2E+00	<4.2E+00	<4.2E+00	-	<1.7E-01	106%	N/A	U _M
Mg (IE)	<1.9E+00	<2.0E+00	<2.0E+00	<2.0E+00	-	<4.0E-03	97%	N/A	-
Na (IE)	2.65E+05	2.55E+05	2.58E+05	2.59E+05	1.9%	<2.2E-01	107%	108%	-
Ni (IE)	6.23E+00	5.27E+00	6.34E+00	5.95E+00	9.9%	<5.4E-02	101%	92%	U _E
P (IE)	2.00E+02	2.02E+02	1.95E+02	1.99E+02	1.9%	<1.0E-01	96%	N/A	U _E

QC Flags: none - meets all QC

U_B - blank exceeds 5% of sample concentration

U_E - value less than 10x the DL

U_R - fails %RSD criteria

U_L - fails % Recovery of LCS

ND - not detected

U_M - fails minimum MRQ criteria

U_S - fails % Recovery of MS

N/A - not applicable

**Table 7.3. Composition of the As-Received 241-AW-101 Filtered Supernate
(Continued)**

Analyte	1st Replicate (mg/L)	2nd Replicate (mg/L)	3rd Replicate (mg/L)	Average (mg/L)	%RSD	Blank (mg/L)	LCS % Recovery	MS % Recovery	QC Flag
Pb (IE)	4.35E+01	4.23E+01	4.27E+01	4.28E+01	1.4%	<1.3E-01	102%	N/A	-
Sr (IE)	2.92E+00	2.18E+00	3.39E+00	2.83E+00	21%	<3.0E-02	101%	N/A	U _R
K (IE)	3.81E+04	3.73E+04	3.70E+04	3.75E+04	1.5%	<3.8E+00	105%	N/A	-
S (IE)	4.13E+02	4.09E+02	4.06E+02	4.09E+02	0.8%	<1.1E-01	101%	N/A	-
Ce (IE)	<2.6E+00	<2.6E+00	<2.6E+00	<2.6E+00	-	<1.1E-01	99%	N/A	U _M
V (IE)	<1.9E-01	<2.0E-01	<2.0E-01	<1.9E-01	-	<8.0E-03	103%	N/A	-
Na (AA)	2.36E+05	2.39E+05	2.28E+05	2.34E+05	2.4%	3.6E-02	99%	100%	-
K (AA)	3.41E+04	3.34E+04	3.27E+04	3.34E+04	2.0%	3.3E-02	101%	99%	-
Hg (AA)	<2.5E-01	<2.6E-01	<2.5E-01	<2.5E-01	-	<1.0E-02	97%	97%	-
U (CC)	<2.4E-01	<2.5E-01	<2.4E-01	<2.4E-01	-	<1.0E-02	104%	92%	-
Citrate (IC)	<2.4E+03	<2.5E+03	<2.4E+03	<2.4E+03	-	<1.0E+02	102%	96%	U _M
Glycolate (IC)	<2.4E+03	<2.5E+03	<2.4E+03	<2.4E+03	-	<1.0E+02	99%	115%	U _M
Acetate (IC)	<2.4E+03	<2.5E+03	<2.4E+03	<2.4E+03	-	<1.0E+02	95%	86%	U _M
EDTA (HL)	<4.8E+02	<4.9E+02	<4.9E+02	<4.9E+02	-	<2.0E+01	94%	90%	U _M
HEDTA (HL)	<4.8E+02	<4.9E+02	<4.9E+02	<4.9E+02	-	<2.0E+01	96%	90%	U _M
IDA (GM)	<6.0E+03	<6.1E+03	<6.1E+03	<6.1E+03	-	<2.5E+02	86%	104%	U _M
NTA (GM)	<6.0E+03	<6.1E+03	<6.1E+03	<6.1E+03	-	<2.5E+02	86%	104%	U _M
ED3A (GM)	ND	ND	ND	-	-	ND	86%	104%	-
Rb (IM)	1.06E+01	1.05E+01	1.03E+01	1.05E+01	1.3%	<3.2E-04	102%	98%	-
W (IM)	1.67E+02	1.60E+02	1.58E+02	1.62E+02	3.1%	<8.8E-04	-	-	-
Th (IM)	4.37E-02	3.92E-01	8.02E-02	1.72E-01	111%	<1.2E-05	100%	98%	U _R
I ¹²⁷ (IM)	2.21E+00	2.52E+00	2.48E+00	2.40E+00	7.0%	8.66E-03	-	-	-
Cs ¹³³ (IM)	1.08E+01	1.08E+01	1.08E+01	1.08E+01	0.2%	<6.6E-05	-	99%	-
Cs ¹³⁵ (IM)	3.36E+00	3.37E+00	3.35E+00	3.36E+00	0.3%	5.27E-05	104%	99%	-
Cs ¹³⁷ (IM)	4.86E+00	4.85E+00	4.87E+00	4.86E+00	0.3%	7.36E-05	104%	100%	-

QC Flags: none - meets all QC

U_B - blank exceeds 5% of sample concentration

U_E - value less than 10x the DL

U_R - fails %RSD criteria

U_L - fails % Recovery of LCS

ND - not detected

U_M - fails minimum MRQ criteria

U_S - fails % Recovery of MS

N/A - not applicable

**Table 7.3. Composition of the As-Received 241-AW-101 Filtered Supernate
(Continued)**

Analyte	1st Replicate (mCi/L)	2nd Replicate (mCi/L)	3rd Replicate (mCi/L)	Average (mCi/L)	%RSD	Blank (mCi/L)	LCS % Recovery	MS % Recovery	QC Flag
Cs ¹³⁷ (GS)	4.10E+02	3.82E+02	3.83E+02	3.92E+02	4.0%	<9.8E-04	N/A	N/A	-
Eu ¹⁵⁴ (GS)	<7.5E-01	<2.2E-01	<6.0E-01	<5.3E-01	-	<8.6E-04	N/A	N/A	U _M
Eu ¹⁵⁵ (GS)	<2.0E+00	<1.8E+00	<1.9E+00	<1.9E+00	-	<1.5E-03	N/A	N/A	U _M
Co ⁶⁰ (GS)	<2.12E-01	<3.45E-01	<2.15E-01	<2.57E-01	-	<1.3E-03	N/A	N/A	U _M
Sn ¹²⁶ (GS)	<1.7E+00	<1.6E+00	<1.6E+00	<1.6E+00	-	<1.2E-03	N/A	N/A	U _M
Pa ²³¹ (GS)	<4.3E+01	<4.2E+01	<4.3E+01	<4.3E+01	-	<2.3E-02	N/A	N/A	U _M
Sr ⁹⁰ (SL)	<8.3E-02	<8.4E-02	<8.4E-02	<8.3E-02	-	<3.4E-03	104%	88%	-
Se ⁷⁹ (SL)	<4.7E-05	<4.8E-05	<5.8E-05	<5.1E-05	-	<2.5E-06	N/A	N/A	-
H ³ (SL)	<5.1E-03	<1.1E-02	<1.1E-02	<8.9E-03	-	<5.9E-04	88%	82%	-
C ¹⁴ (SL)	<5.6E-04	<1.4E-03	<3.7E-04	<7.9E-04	-	<6.4E-05	100%	100%	-
Tc ⁹⁹ _{pertech} (SL)	1.67E-01	1.50E-01	1.55E-01	1.57E-01	5.5%	<7.0E-05	tracer	tracer	-
I ¹²⁹ (SG)	1.58E-04	1.83E-04	1.75E-04	1.72E-04	7.4%	<2.2E-07	N/A	N/A	-
Pu ²³⁸ (SA)	2.55E-03	1.77E-03	2.04E-03	2.12E-03	19%	9.86E-07	N/A	N/A	U _R
Pu ^{239/240} (SA)	6.07E-04	5.09E-04	4.36E-04	5.17E-04	17%	9.50E-07	N/A	N/A	U _R
Pu ²⁴¹ (SA)	1.58E-03	1.41E-03	1.83E-03	1.61E-03	13%	<2.7E-07	N/A	N/A	-
Am ²⁴¹ (SG)	<5.0E-04	<5.0E-04	<5.9E-04	<5.3E-04	-	<3.4E-04	N/A	N/A	-
Cm ²⁴² (SA)	<3.0E-06	<2.5E-06	<7.1E-06	<4.2E-06	-	<2.4E-06	N/A	N/A	-
Cm ^{243/244} (SA)	2.11E-04	2.71E-04	2.46E-04	2.43E-04	12%	2.80E-05	N/A	N/A	U _B
Tc ⁹⁹ _{total} (IM)	1.74E-01	1.68E-01	1.65E-01	1.69E-01	2.6%	<4.1E-06	-	95%	-
Np ²³⁷ (IM)	2.15E-06	<2.1E-07	<2.0E-07	-	-	<8.4E-09	-	-	-
Pu ²³⁹ (IM)	2.31E-03	2.91E-02	2.75E-03	1.14E-02	135%	<7.3E-07	-	-	U _R
Pu ²⁴⁰ (IM)	2.65E-03	2.27E-02	<6.5E-05	1.27E-02*	98%	<2.7E-06	-	-	U _R
U ²³³ (IM)	2.96E-05	<2.8E-06	<2.8E-06	-	-	<1.1E-07	-	-	-
U ²³⁴ (IM)	2.68E-05	<1.8E-06	<1.8E-06	-	-	<7.4E-08	-	-	-

* Average of two replicates

QC Flags: none - meets all QC

U_B - blank exceeds 5% of sample concentration

U_E - value less than 10x the DL

U_R - fails %RSD criteria

U_L - fails % Recovery of LCS

ND - not detected

U_M - fails minimum MRQ criteria

U_S - fails % Recovery of MS

N/A - not applicable

**Table 7.3. Composition of the As-Received 241-AW-101 Filtered Supernate
(Continued)**

Analyte	1st Replicate (mCi/L)	2nd Replicate (mCi/L)	3rd Replicate (mCi/L)	Average (mCi/L)	%RSD	Blank (mCi/L)	LCS % Recovery	MS % Recovery	QC Flag
U ²³⁵ (IM)	3.48E-07	7.73E-07	2.59E-07	4.60E-07	60%	9.20E-09	96%	87%	U _R
U ²³⁶ (IM)	2.11E-06	5.11E-06	1.56E-06	2.93E-06	65%	6.43E-08	-	-	U _R
U ²³⁸ (IM)	5.55E-07	1.35E-06	7.35E-07	8.79E-07	47%	1.05E-08	98%	89%	U _R
Alpha (AC)	1.45E-02	7.27E-03	8.21E-03	1.00E-02	39%	<2.8E-04	83%	69%	U _E U _R U _S
Alpha _{sum}	-	-	-	<3.2E-03	-	-	N/A	N/A	-
Density (g/mL)	1.41	1.41	1.41	1.41	0.0%	-	N/A	N/A	-
Wt% Dissolved Solids	54.7%	56.4%	56.9%	56.0%	2.0%	-	N/A	N/A	-

QC Flags: none - meets all QC

U_B - blank exceeds 5% of sample concentration

U_E - value less than 10x the DL

U_R - fails %RSD criteria

U_L - fails % Recovery of LCS

ND - not detected

U_M - fails minimum MRQ criteria

U_S - fails % Recovery of MS

N/A - not applicable

Table 7.4. Properties of the Diluted 5 M Sodium 241-AW-101 Sample

Property	Units	1st Replicate	2nd Replicate	3rd Replicate	Average	%RSD	QC Flag
Density of Filtered Supernate	g/mL	1.24	1.25	1.25	1.25	0.5%	-
Wt% Vacuum Filtered Solids	Wt%	0.39%	-	-	-	-	-
Wt% Dissolved Solids	Wt%	34.1%	34.4%	33.9%	34.1%	0.7%	-
Wt% Insoluble Solids (measured.)	Wt%	0.11%	-	-	-	-	-

QC Flags: none - meets all QC

U_B - blank exceeds 5% of sample concentration

U_E - value less than 10x the DL

U_R - fails %RSD criteria

U_L - fails % Recovery of LCS

ND - not detected

U_M - fails minimum MRQ criteria

U_S - fails % Recovery of MS

N/A - not applicable

Table 7.5. Composition of the Diluted 5 M Sodium 241-AW-101 Filtered Supernate

Analyte	1st Replicate (mg/L)	2nd Replicate (mg/L)	3rd Replicate (mg/L)	Average (mg/L)	%RSD	Blank (mg/L)	LCS % Recovery	MS % Recovery	QC Flag
NO ₃ ⁻ (IC)	9.75E+04	9.38E+04	1.08E+05	9.99E+04	7.6%	<1.0E+01	106%	90%	-
NO ₂ ⁻ (IC)	5.01E+04	4.92E+04	5.64E+04	5.19E+04	7.5%	<1.0E+01	101%	95%	-
PO ₄ ³⁻ (IC)	<1.9E+02	3.80E+02	<1.9E+02	-	-	<1.0E+01	106%	109%	-
SO ₄ ²⁻ (IC)	1.56E+02	1.27E+02	1.52E+02	1.45E+02	11%	<5.0E+00	106%	99%	-
C ₂ O ₄ ²⁻ (IC)	2.34E+02	1.99E+02	2.66E+02	2.33E+02	14%	<1.0E+01	103%	99%	-
Cl ⁻ (IC)	2.65E+03	2.50E+03	3.02E+03	2.72E+03	10%	<2.0E+00	103%	102%	-
F ⁻ (IC)	2.73E+02	2.53E+02	2.85E+02	2.70E+02	5.9%	<2.0E+00	97%	101%	-
CHO ₂ ⁻ (IC)	1.01E+03	1.12E+03	1.14E+03	1.09E+03	6.3%	<1.0E+01	94%	103%	-
NH ₄ ⁺ (PT)	<9.7E+01	<9.1E+01	<9.5E+01	<9.4E+01	-	<5.0E+00	103%	111%	-
OH _{free} ⁻ (T)	2.77E+04	3.42E+04	3.71E+04	3.30E+04	15%	<3.4E+02	N/A	N/A	-
OH _{total} ⁻ (T)	4.08E+04	4.83E+04	4.78E+04	4.56E+04	9.3%	<3.4E+02	97%	N/A	-
TIC (A)	1.16E+03	7.64E+02	8.30E+02	9.18E+02	23%	1.59E+00	100%	108%	U _R
TOC (Diff)	6.63E+02	1.28E+03	1.04E+03	9.92E+02	31%	1.56E+01	N/A	N/A	U _R
Al (IE)	1.61E+04	1.67E+04	1.62E+04	1.63E+04	2.1%	<5.7E-01	99%	N/A	-
B (IE)	1.82E+01	1.88E+01	1.78E+01	1.82E+01	2.6%	<2.8E-01	106%	N/A	U _E
Ba (IE)	4.08E+00	4.11E+00	4.04E+00	4.08E+00	0.8%	<1.9E-01	104%	111%	U _E
Ca (IE)	6.63E+00	6.07E+00	6.01E+00	6.24E+00	5.5%	<2.3E-01	105%	111%	-
Cd (IE)	1.69E+00	1.63E+00	1.76E+00	1.70E+00	3.7%	<4.2E-02	104%	N/A	U _E
Cr (IE)	4.92E+01	5.16E+01	5.00E+01	5.03E+01	2.4%	<6.0E-02	106%	97%	-
Fe (IE)	1.96E+00	2.26E+00	2.19E+00	2.14E+00	7.3%	<3.6E-02	104%	107%	U _E
La (IE)	1.38E+00	1.63E+00	1.42E+00	1.48E+00	9.0%	<7.2E-02	99%	N/A	U _E
Li (IE)	<1.1E+01	<1.1E+01	<1.1E+01	<1.1E+01	-	<5.7E-01	99%	N/A	U _M
Mg (IE)	<1.0E+00	<1.1E+00	<9.9E-01	<1.0E+00	-	<5.3E-02	106%	N/A	-
Na (IE)	1.12E+05	1.18E+05	1.12E+05	1.14E+05	2.9%	<2.0E+01	100%	100%	-
Ni (IE)	4.38E+00	4.07E+00	4.25E+00	4.24E+00	3.7%	<1.4E-01	104%	105%	U _E
P (IE)	1.64E+02	1.71E+02	1.63E+02	1.66E+02	2.6%	<6.9E-01	106%	N/A	U _E

QC Flags: none - meets all QC

U_B - blank exceeds 5% of sample concentration

U_E - value less than 10x the DL

U_R - fails %RSD criteria

U_L - fails % Recovery of LCS

ND - not detected

U_M - fails minimum MRQ criteria

U_S - fails % Recovery of MS

N/A - not applicable

Table 7.5. Composition of the Diluted 5 M Sodium 241-AW-101 Filtered Supernate (Continued)

Analyte	1st Replicate (mg/L)	2nd Replicate (mg/L)	3rd Replicate (mg/L)	Average (mg/L)	%RSD	Blank (mg/L)	LCS % Recovery	MS % Recovery	QC Flag
Pb (IE)	2.31E+01	2.32E+01	2.10E+01	2.24E+01	5.6%	<3.2E-01	104%	N/A	U _E
Sr (IE)	<9.6E+01	<1.0E+02	<9.4E+01	<9.7E+01	-	<5.0E+00	97%	N/A	-
K (IE)	2.31E+04	2.38E+04	2.32E+04	2.34E+04	1.6%	<9.4E+00	105%	N/A	-
S (IE)	2.50E+02	2.66E+02	2.53E+02	2.56E+02	3.4%	1.43E+00	92%	N/A	-
Ce (IE)	<5.2E+00	<5.4E+00	<5.1E+00	<5.2E+00	-	<2.7E-01	96%	N/A	U _M
V (IE)	2.29E+00	2.28E+00	2.27E+00	2.28E+00	0.5%	1.04E-01	97%	N/A	U _E
Na (AA)	1.08E+05	1.12E+05	1.07E+05	1.09E+05	2.6%	<9.3E-01	101%	96%	-
K (AA)	1.80E+04	1.85E+04	1.78E+04	1.81E+04	2.2%	2.80E-02	98%	96%	-
Hg (AA)	<2.1E+00	<2.2E+00	<2.1E+00	<2.1E+00	-	<1.1E-01	94%	95%	U _M
U (CC)	2.19E-01	1.50E-01	1.87E-01	1.85E-01	19%	1.00E-02	97%	99%	U _R
Citrate (IC)	<1.9E+02	<1.8E+02	<1.9E+02	<1.9E+02	-	<1.0E+01	93%	70%	U _M U _S
Glycolate (IC)	<1.9E+02	2.90E+02	3.23E+02	3.06E+02*	7.7%	<1.0E+01	98%	99%	-
Acetate (IC)	<1.9E+02	2.28E+03	2.37E+03	2.33E+03*	2.8%	<1.0E+01	94%	100%	-
EDTA (HL)	9.75E+01	9.05E+01	9.50E+01	9.43E+01	3.7%	<5.0E+00	104%	104%	U _E
HEDTA (HL)	<9.7E+01	<9.1E+01	<9.5E+01	<9.4E+01	-	<5.0E+00	100%	100%	-
IDA (GM)	<9.7E+02	<9.1E+02	<9.5E+02	<9.4E+02	-	<5.0E+01	90%	83%	-
NTA (GM)	<9.7E+02	<9.1E+02	<9.5E+02	<9.4E+02	-	<5.0E+01	90%	83%	-
ED3A (GM)	ND	ND	ND	-	-	ND	90%	83%	-
Rb (IM)	5.33E+00	5.42E+00	5.00E+00	5.25E+00	4.2%	<2.5E-05	98%	98%	-
W (IM)	4.61E+01	4.71E+01	4.74E+01	4.69E+01	1.4%	<6.1E-05	-	-	-
Th (IM)	2.02E-03	1.55E-03	<1.4E-04	1.78E-03*	19%	<7.6E-06	97%	-	U _R
I ¹²⁷ (IM)	<9.6E-04	<1.0E-03	<9.4E-04	<9.7E-04	-	<5.0E-05	-	-	-
Cs ¹³³ (IM)	4.69E+00	4.95E+00	4.59E+00	4.74E+00	4.0%	5.00E-05	97%	108%	-
Cs ¹³⁵ (IM)	1.45E+00	1.53E+00	1.40E+00	1.46E+00	4.3%	<1.5E-06	97%	104%	-
Cs ¹³⁷ (IM)	2.06E+00	2.17E+00	1.99E+00	2.08E+00	4.5%	<2.8E-06	98%	104%	-

* Average of two replicates

QC Flags: none - meets all QC

U_B - blank exceeds 5% of sample concentration

U_E - value less than 10x the DL

U_R - fails %RSD criteria

U_L - fails % Recovery of LCS

ND - not detected

U_M - fails minimum MRQ criteria

U_S - fails % Recovery of MS

N/A - not applicable

Table 7.5. Composition of the Diluted 5 M Sodium 241-AW-101 Filtered Supernate (Continued)

Analyte	1st Replicate (mCi/L)	2nd Replicate (mCi/L)	3rd Replicate (mCi/L)	Average (mCi/L)	%RSD	Blank (mCi/L)	LCS % Recovery	MS % Recovery	QC Flag
Cs ¹³⁷ (GS)	1.72E+02	1.80E+02	1.75E+02	1.76E+02	2.2%	<2.1E-04	N/A	N/A	-
Eu ¹⁵⁴ (GS)	<2.2E-05	<2.0E-05	<2.1E-05	<2.1E-05	-	<9.0E-07	N/A	N/A	-
Eu ¹⁵⁵ (GS)	<4.0E-05	<3.6E-05	<3.9E-05	<3.8E-05	-	<1.6E-06	N/A	N/A	-
Co ⁶⁰ (GS)	3.43E-04	3.32E-04	3.53E-04	3.43E-04	2.9%	7.16E-07	N/A	N/A	-
Sn ¹²⁶ (GS)	1.82E-04	1.29E-05	2.41E-04	1.45E-04	82%	<2.2E-06	N/A	N/A	-
Pa ²³¹ (GS)	<5.2E-04	<4.9E-04	<5.0E-04	<5.0E-04	-	<2.0E-05	N/A	N/A	U _M
Sr ⁹⁰ (SL)	2.42E-02	2.86E-02	2.86E-02	2.72E-02	9.4%	<3.4E-04	99%	100%	-
Se ⁷⁹ (SL)	<5.2E-05	<4.6E-05	<4.5E-05	<4.8E-05	-	<3.3E-06	N/A	N/A	-
H ³ (SL)	<1.0E-02	<1.1E-02	<9.9E-03	<1.0E-02	-	<5.3E-04	89%	78%	-
C ¹⁴ (SL)	6.37E-04	3.45E-04	7.24E-04	5.68E-04	35%	<1.4E-05	100%	81%	U _R
Tc ⁹⁹ _{pertech} (SL)	7.33E-02	7.47E-02	7.14E-02	7.31E-02	2.3%	<6.8E-06	tracer	tracer	-
I ¹²⁹ (SG)	1.17E-04	1.14E-04	1.04E-04	1.12E-04	5.8%	<1.3E-07	N/A	N/A	-
Pu ²³⁸ (SA)	4.25E-04	5.16E-04	4.24E-04	4.55E-04	12%	7.70E-07	N/A	N/A	-
Pu ^{239/240} (SA)	5.84E-05	9.36E-05	6.39E-05	7.20E-05	26%	1.11E-06	N/A	N/A	U _R
Pu ²⁴¹ (SA)	<1.6E-03	<2.2E-03	<1.3E-03	<1.7E-03	-	<1.0E-04	N/A	N/A	-
Am ²⁴¹ (SG)	<2.2E-04	<2.1E-04	<2.2E-04	<2.2E-04	-	<1.1E-05	N/A	N/A	-
Cm ²⁴² (SA)	<2.3E-04	<4.3E-04	<3.4E-04	<3.3E-04	-	<3.6E-06	N/A	N/A	-
Cm ^{243/244} (SA)	<2.3E-04	<4.3E-04	<3.4E-04	<3.3E-04	-	<3.6E-06	N/A	N/A	-
Tc ⁹⁹ _{total} (IM)	8.65E-02	8.83E-02	8.29E-02	8.59E-02	3.2%	<5.4E-08	-	-	-
Np ²³⁷ (IM)	<1.0E-07	<1.1E-07	<1.0E-07	<1.0E-07	-	<5.4E-09	-	-	-
Pu ²³⁹ (IM)	9.99E-05	1.01E-04	<8.7E-06	1.00E-04*	0.5%	<4.7E-07	-	-	-
Pu ²⁴⁰ (IM)	<3.3E-05	<3.5E-05	<3.2E-05	<3.3E-05	-	<1.7E-06	-	-	-
U ²³³ (IM)	<1.4E-06	<1.5E-06	<1.4E-06	<1.4E-06	-	<7.3E-08	-	-	-
U ²³⁴ (IM)	<9.1E-07	<9.6E-07	<8.9E-07	<9.2E-07	-	<4.8E-08	-	-	-

* Average of two replicates

QC Flags: none - meets all QC

U_B - blank exceeds 5% of sample concentration

U_E - value less than 10x the DL

U_R - fails %RSD criteria

U_L - fails % Recovery of LCS

ND - not detected

U_M - fails minimum MRQ criteria

U_S - fails % Recovery of MS

N/A - not applicable

Table 7.5. Composition of the Diluted 5 M Sodium 241-AW-101 Filtered Supernate (Continued)

Analyte	1st Replicate (mCi/L)	2nd Replicate (mCi/L)	3rd Replicate (mCi/L)	Average (mCi/L)	%RSD	Blank (mCi/L)	LCS % Recovery	MS % Recovery	QC Flag
U ²³⁵ (IM)	2.18E-08	2.47E-08	2.28E-08	2.31E-08	6.4%	<1.6E-11	94%	-	-
U ²³⁶ (IM)	<9.4E-09	<9.9E-09	<9.2E-09	<9.5E-09	-	<4.9E-10	-	-	-
U ²³⁸ (IM)	4.38E-07	4.50E-07	4.37E-07	4.41E-07	1.6%	<2.6E-12	96%	108%	-
Alpha (AC)	<8.7E-02	<7.3E-02	<8.3E-02	<8.1E-02	-	<2.3E-04	83%	37%	U _s
Alpha _{sum}	-	-	-	<1.5E-03	-	-	-	-	-
Density (g/mL)	1.24	1.25	1.25	1.25	0.5%	-	-	-	-
Wt% Dissolved Solids	34.1%	34.4%	33.9%	34.1%	0.7%	-	-	-	-

QC Flags: none - meets all QC

U_B - blank exceeds 5% of sample concentration

U_E - value less than 10x the DL

U_R - fails %RSD criteria

U_L - fails % Recovery of LCS

ND - not detected

U_M - fails minimum MRQ criteria

U_S - fails % Recovery of MS

N/A - not applicable

Table 7.6. Composition of the Aqua-Regia Digested As-Received 241-AW-101 Filterable Solids

Analyte	1st Replicate (mg/Kg)	2nd Replicate (mg/Kg)	3rd Replicate (mg/Kg)	Average (mg/Kg)	%RSD	Blank (mg/L)	1st Glass Std (mg/Kg)	2nd Glass Std (mg/Kg)	Average (mg/Kg)	%RSD	Glass Std Comp (mg/Kg)	LCS % Recovery	MS % Recovery	QC Flag
Na (AA)	8.82E+04	1.01E+05	9.57E+04	9.48E+04	6.6%	3.72E+03	5.64E+03	6.40E+03	6.02E+03	8.9%	8.52E+04	85%	101%	U _L
K (AA)	1.38E+05	1.28E+05	1.35E+05	1.33E+05	3.9%	1.41E+03	2.45E+04	2.57E+04	2.51E+04	3.6%	2.26E+04	99%	98%	-
Hg (AA)	<2.4E+01	<1.4E+01	<9.9E+00	<1.6E+01	-	<4.7E+00	<1.1E+01	<8.7E+00	<9.8E+00	-	-	94%	90%	U _M
Al (IE)	6.91E+03	7.38E+03	7.01E+03	7.10E+03	3.5%	<1.4E+02	2.53E+04	2.59E+04	2.56E+04	1.7%	2.50E+04	100%	N/A	U _E
B (IE)	<7.0E+01	<7.0E+01	<7.0E+01	<7.00E+01	-	<7.0E+01	3.03E+04	3.10E+04	3.07E+04	1.6%	2.69E+04	104%	N/A	U _M
Ba (IE)	1.57E+02	1.61E+02	1.65E+02	1.61E+02	2.5%	<4.8E+01	1.04E+03	1.07E+03	1.06E+03	2.0%	7.90E+02	102%	112%	U _E
Ca (IE)	5.10E+02	6.00E+02	4.57E+02	5.22E+02	14%	<5.8E+01	1.18E+04	1.21E+04	1.20E+04	1.8%	1.02E+04	105%	118%	-
Cd (IE)	2.36E+01	<1.1E+01	2.13E+01	2.25E+01*	7.2%	<1.1E+01	3.71E+01	3.46E+01	3.59E+01	4.9%	-	104%	N/A	U _E
Ce (IE)	<6.8E+01	<6.8E+01	<6.8E+01	<6.8E+01	-	<6.8E+01	<6.8E+01	<6.8E+01	<6.8E+01	-	-	97%	N/A	U _M
Cr (IE)	3.48E+02	3.75E+02	3.37E+02	3.53E+02	5.5%	<1.5E+01	7.35E+02	7.24E+02	7.30E+02	1.1%	6.40E+02	104%	113%	U _E
Fe (IE)	3.85E+02	3.27E+02	2.23E+02	3.12E+02	26%	<9.0E+00	1.12E+05	1.14E+05	1.13E+05	1.3%	9.79E+04	103%	112%	U _R
K (IE)	1.72E+05	1.54E+05	1.59E+05	1.62E+05	5.7%	<2.4E+03	2.51E+04	2.58E+04	2.55E+04	1.9%	2.26E+04	106%	N/A	-
La (IE)	5.38E+01	7.13E+01	5.02E+01	5.84E+01	19%	<1.8E+01	8.49E+01	6.34E+01	7.42E+01	21%	-	99%	N/A	U _E U _R
Li (IE)	1.60E+02	1.45E+02	2.25E+02	1.77E+02	24%	<1.4E+02	1.64E+04	1.36E+04	1.50E+04	13%	1.49E+04	100%	N/A	U _E U _R
Mg (IE)	<1.3E+01	<1.3E+01	<1.3E+01	<1.3E+01	-	<1.3E+01	5.89E+03	6.00E+03	5.95E+03	1.3%	5.20E+03	104%	N/A	-
Na (IE)	2.10E+05	2.04E+05	2.04E+05	2.06E+05	1.7%	2.90E+02	9.53E+04	9.82E+04	9.68E+04	2.1%	8.52E+04	101%	103%	-
Ni (IE)	1.36E+02	9.74E+01	9.30E+01	1.09E+02	22%	<3.4E+01	9.36E+03	9.46E+03	9.41E+03	0.8%	8.27E+03	103%	112%	U _E U _R

* Average of two replicates

QC Flags: none - meets all QC

U_L - fails % Recovery of LCS

ND - not detected

U_R - fails %RSD criteria

U_S - fails % Recovery of MS

N/A - not applicable

U_M - fails minimum MRQ criteria

U_E - value less than 10x the DL

U_B - blank >5% of sample concentration

U_G - glass std >5% of sample concentration

Table 7.6. Composition of the Aqua-Regia Digested As-Received 241-AW-101 Filterable Solids (Continued)

Analyte	1st Replicate (mg/Kg)	2nd Replicate (mg/Kg)	3rd Replicate (mg/Kg)	Average (mg/Kg)	%RSD	Blank (mg/L)	1st Glass Std (mg/Kg)	2nd Glass Std (mg/Kg)	Average (mg/Kg)	%RSD	Glass Std Comp (mg/Kg)	LCS % Recovery	MS % Recovery	QC Flag
P (IE)	3.44E+03	2.99E+03	3.17E+03	3.20E+03	7.1%	<1.7E+02	1.10E+03	1.75E+03	1.43E+03	32%	1.10E+03	104%	N/A	U _E
Pb (IE)	<8.0E+01	<8.0E+01	<8.0E+01	<8.0E+01	-	<8.0E+01	<8.0E+01	<8.0E+01	<8.0E+01	-	-	103%	N/A	-
S (IE)	1.32E+03	1.48E+03	1.36E+03	1.39E+03	6.0%	<3.4E+02	2.12E+03	1.91E+03	2.02E+03	7.4%	-	92%	N/A	U _E
U (IE)	1.87E+03	2.18E+03	2.09E+03	2.05E+03	7.8%	<1.3E+03	6.88E+03	6.98E+03	6.93E+03	1.0%	-	98%	N/A	U _G
V (IE)	1.23E+02	1.34E+02	1.30E+02	1.29E+02	4.3%	3.16E+01	2.41E+02	2.42E+02	2.42E+02	0.3%	-	94%	N/A	U _B U _E
U (CC)	4.98E+01	5.24E+01	3.69E+01	4.64E+01	18%	<1.0E-02	<1.0E-02	<1.0E-02	<1.0E-02	-	-	97%	98%	U _R
W (IM)	4.92E+01	6.34E+01	5.65E+01	5.63E+01	13%	<5.9E-01	2.87E+00	3.17E+00	3.02E+00	6.9%	-	-	-	-
Th (IM)	1.63E+00	1.77E+00	1.37E+00	1.59E+00	13%	<6.3E-02	1.68E+00	1.80E+00	1.74E+00	5.1%	-	103%	-	U _G
Rb (IM)	1.25E+01	1.11E+01	1.10E+01	1.15E+01	7.3%	<2.5E-01	5.79E+01	5.61E+01	5.70E+01	2.2%	-	105%	-	U _G
I ¹²⁷ (IM)	<5.3E-01	<5.3E-01	<5.3E-01	<5.3E-01	-	<5.3E-01	<5.3E-01	<5.3E-01	<5.3E-01	-	-	-	-	U _M
Cs ¹³³ (IM)	2.57E+00	2.78E+00	2.43E+00	2.59E+00	6.8%	<2.7E-01	5.32E+00	7.20E+00	6.26E+00	21%	-	-	-	U _G
Cs ¹³⁵ (IM)	9.58E-01	8.31E-01	7.33E-01	8.41E-01	13%	<1.2E-01	5.29E+01	5.41E+01	5.35E+01	1.6%	5.13E+01	107%	-	U _E
Cs ¹³⁷ (IM)	5.19E+00	5.67E+00	4.80E+00	5.22E+00	8.4%	<9.9E-02	8.94E+01	9.36E+01	9.15E+01	3.2%	8.85E+01	110%	-	-

* Average of two replicates

QC Flags: none - meets all QC

U_L - fails % Recovery of LCS

ND - not detected

U_R - fails %RSD criteria

U_S - fails % Recovery of MS

N/A - not applicable

U_M - fails minimum MRQ criteria

U_B - value less than 10x the DL

U_B - blank >5% of sample concentration

U_G - glass std >5% of sample concentration

Table 7.6. Composition of the Aqua-Regia Digested As-Received 241-AW-101 Filterable Solids (Continued)

Analyte	1st Replicate (mCi/Kg)	2nd Replicate (mCi/Kg)	3rd Replicate (mCi/Kg)	Average (mCi/Kg)	%RSD	Blank (mCi/L)	1st Glass Std (mCi/Kg)	2nd Glass Std (mCi/Kg)	Average (mCi/Kg)	%RSD	Glass Std Comp (mCi/Kg)	LCS % Recovery	MS % Recovery	QC Flag
Cs ¹³⁷ (GS)	6.62E+01	8.65E+01	7.93E+01	7.73E+01	13%	4.19E-04	1.45E-01	1.32E-01	1.39E-01	6.9%	-	N/A	N/A	-
Eu ¹⁵⁴ (GS)	7.48E-03	7.49E-03	6.58E-03	7.18E-03	7.3%	<7.7E-04	<8.7E-04	<9.1E-04	<8.9E-04	-	-	N/A	N/A	-
Eu ¹⁵⁵ (GS)	7.79E-03	7.04E-03	7.07E-03	7.30E-03	5.8%	<9.8E-04	<1.1E-03	<1.6E-03	<1.4E-04	-	-	N/A	N/A	-
Co ⁶⁰ (GS)	6.59E-03	6.73E-03	6.04E-03	6.45E-03	5.7%	<6.5E-04	<7.4E-04	<7.5E-04	<7.4E-04	-	-	N/A	N/A	-
Sn ¹²⁶ (GS)	<5.9E-04	<6.0E-04	<6.2E-04	<6.0E-04	-	<5.1E-04	<6.0E-04	<5.9E-04	<5.9E-04	-	-	N/A	N/A	-
Sr ¹²⁵ (GS)	<1.8E-03	<2.5E-03	<1.8E-03	<2.0E-03	-	<1.5E-03	<1.7E-03	<1.7E-03	<1.7E-03	-	-	-	-	-
Pa ²³¹ (GS)	<2.0E-02	<2.1E-02	<2.0E-02	<2.0E-02	-	<1.7E-02	<2.0E-02	<2.0E-02	<2.0E-02	-	-	N/A	N/A	U _M
Sr ⁹⁰ (SL)	8.06E+00	1.00E+01	8.60E+00	8.89E+00	11%	<2.2E-01	-	-	-	-	-	96%	73%	-
Tc ⁹⁹ (SL)	7.74E-02	8.12E-02	7.30E-02	7.72E-02	5.3%	1.17E-03	-	-	-	-	-	tracer	tracer	-
Se ⁷⁹ (SL)	<2.6E-03	<3.1E-03	<4.2E-03	<3.3E-03	-	<6.3E-04	-	-	-	-	-	N/A	N/A	U _M
H ³ (SL)	<1.3E-02	<1.3E-02	<1.3E-02	<1.3E-02	-	<3.3E-03	-	-	-	-	-	90%	87%	-
C ¹⁴ (SL)	**	7.72E-04	1.06E-03	9.18E-04*	22%	<2.8E-04	-	-	-	-	-	N/A	N/A	U _R
I ¹²⁹ (SG)	<2.4E-04	<1.8E-02	<9.3E-04	<6.3E-03	-	<1.8E-03	<2.0E-04	<7.6E-05	<1.4E-04	-	-	N/A	N/A	U _M
Pu ²³⁸ (SA)	3.28E-02	3.17E-02	2.25E-02	2.90E-02	20%	8.42E-03	2.36E-03	4.19E-03	3.28E-03	40%	-	N/A	N/A	U _R U _B U _G
Pu ^{239/240} (SA)	3.37E-02	1.58E-02	1.40E-02	2.12E-02	52%	1.42E-03	3.13E-03	7.03E-03	5.08E-03	54%	-	N/A	N/A	U _R U _B U _G
Pu ²⁴¹ (SA)	1.25E-01	6.71E-02	5.50E-02	8.23E-02	45%	<5.0E-03	<2.8E-02	<7.2E-02	<5.0E-02	-	-	N/A	N/A	U _R U _B U _G
Am ²⁴¹ (SG)	5.68E-02	6.67E-02	5.00E-02	5.78E-02	14.5%	9.10E-03	4.21E-03	2.37E-03	3.29E-03	39%	-	N/A	N/A	U _B U _G

* Average of two replicates

** Sample preparation failed, insufficient sample to repeat analysis

QC Flags: none - meets all QC

U_L - fails % Recovery of LCS

ND - not detected

U_R - fails minimum MRQ criteria

U_S - fails % Recovery of MS

N/A - not applicable

U_M - fails minimum MRQ criteria

U_B - value less than 10x the DL

U_G - blank >5% of sample concentration

U_B - blank >5% of sample concentration

U_G - glass std >5% of sample concentration

Table 7.6. Composition of the Aqua-Regia Digested As-Received 241-A-W-101 Filterable Solids (Continued)

Analyte	1st Replicate (mCi/Kg)	2nd Replicate (mCi/Kg)	3rd Replicate (mCi/Kg)	Average (mCi/Kg)	%RSD	Blank (mCi/L)	1st Std Glass Std (mCi/Kg)	2nd Glass Std (mCi/Kg)	Average (mCi/Kg)	%RSD	Glass Std Comp (mCi/Kg)	LCS % Recovery	MS % Recovery	QC Flag
Cm ^{244/243} (SA)	2.29E-01	3.16E-01	2.20E-01	2.55E-01	21%	6.13E-02	1.18E-01	1.79E-02	6.82E-02	104%	-	N/A	N/A	U _R U _B U _G
Cm ²⁴² (SA)	9.50E-05	1.30E-04	1.02E-04	1.09E-04	17%	<6.8E-05	<4.09E-05	<1.51E-05	<2.80E-05	-	-	N/A	N/A	U _E U _R
Tc ⁹⁹ (IM)	1.15E-01	1.22E-01	1.01E-01	1.13E-01	9.4%	1.96E-06	1.30E-03	7.82E-04	1.04E-03	35%	-	105%	-	-
Np ²³⁷ (IM)	<4.4E-05	5.07E-05	<4.4E-05	-	-	<4.4E-05	<4.3E-05	<4.4E-05	<4.4E-05	-	-	-	-	U _E
Pu ²³⁹ (IM)	1.36E-02	1.82E-02	1.38E-02	1.52E-02	17%	<3.9E-03	<3.8E-03	<3.8E-03	<3.8E-03	-	-	-	-	U _E U _R
Pu ²⁴⁰ (IM)	<1.4E-02	<1.4E-02	<1.4E-02	<1.4E-02	-	<1.4E-02	<1.4E-02	<1.4E-02	<1.4E-02	-	-	-	-	U _M
U ²³³ (IM)	<6.0E-04	<6.1E-04	<6.1E-04	<6.0E-04	-	<6.1E-04	<5.9E-04	<6.0E-04	<6.0E-04	-	-	-	-	U _M
U ²³⁴ (IM)	<4.1E-04	<4.2E-04	<4.2E-04	<4.1E-04	-	<4.2E-04	<4.1E-04	<4.1E-04	<4.1E-04	-	-	-	-	U _M
U ²³⁵ (IM)	5.70E-06	6.21E-06	4.97E-06	5.63E-06	11%	<1.4E-07	<1.3E-07	<1.4E-07	<1.3E-07	-	-	105%	97%	-
U ²³⁶ (IM)	1.16E-05	1.25E-05	1.15E-05	1.18E-05	4.7%	<4.1E-06	<4.0E-06	<4.0E-06	<4.0E-06	-	-	-	-	U _E
U ²³⁸ (IM)	1.01E-04	1.20E-04	9.62E-05	1.06E-04	12%	1.34E-7	1.93E-06	1.91E-06	1.92E-06	0.6%	-	104%	102%	-
Alpha (AC)	5.05E-01	5.27E-01	4.91E-01	5.08E-01	3.6%	<1.7E-05	-	-	-	-	-	91%	71%	-
Alpha ^{Sum}	-	-	-	1.08E-01	-	1.89E-02	-	-	1.16E-02	-	-	-	-	U _B U _G

* Average of two replicates
 QC Flags: none - meets all QC
 U_L - fails % Recovery of LCS
 ND - not detected
 U_R - fails %RSD criteria
 U_S - fails % Recovery of MS
 N/A - not applicable
 U_M - fails minimum MRQ criteria
 U_E - value less than 10x the DL
 U_B - blank >5% of sample concentration
 U_G - glass std >5% of sample concentration

Table 7.7. Composition of the Sodium Peroxide Fusion Digested As-Received 241-A-W-101 Filterable Solids

Analyte	1st Replicate (mg/Kg)	2nd Replicate (mg/Kg)	3rd Replicate (mg/Kg)	Average (mg/Kg)	%RSD	Blank (mg/L)	1st Glass Std (mg/Kg)	2nd Glass Std (mg/Kg)	Average (mg/Kg)	%RSD	Glass Std Comp (mg/Kg)	LCS % Recovery	MS % Recovery	QC Flag
K (AA)	1.29E+05	1.45E+05	1.36E+05	1.37E+05	6.1%	1.52E+00	1.86E+04	2.09E+04	1.98E+04	8.2%	2.26E+04	100%	89%	-
Al (IE)	7.66E+03	5.57E+03	5.78E+03	6.34E+03	18%	<1.4E+02	2.02E+04	2.30E+04	2.16E+04	9.2%	2.50E+04	99%	N/A	U _R
B (IE)	<7.0E+01	<7.0E+01	<7.0E+01	<7.0E+01	-	<7.0E+01	2.22E+04	2.51E+04	2.37E+04	8.7%	2.69E+04	100%	N/A	U _M
Ba (IE)	6.89E+01	7.36E+01	6.94E+01	7.06E+01	3.7%	<4.8E+01	7.68E+02	8.87E+02	8.28E+02	10%	7.90E+02	100%	101%	U _E
Ca (IE)	1.67E+03	1.52E+03	1.51E+03	1.57E+03	5.7%	2.77E+02	9.25E+03	1.05E+04	9.88E+03	9.0%	1.02E+04	103%	100%	U _B
Cd (IE)	1.86E+01	<1.1E+01	<1.1E+01	-	-	<1.1E+01	<1.1E+01	1.22E+01	-	-	-	99%	N/A	U _F
Ce (IE)	<6.8E+01	<6.8E+01	<6.8E+01	<6.8E+01	-	<6.8E+01	<6.8E+01	<6.8E+01	<6.75E+01	-	-	99%	N/A	U _M
Cr (IE)	3.69E+02	2.48E+02	2.70E+02	2.96E+02	22%	<1.5E+01	5.67E+02	6.13E+02	5.90E+02	5.5%	6.40E+02	100%	100%	U _E U _R
Fe (IE)	5.61E+02	3.31E+02	5.56E+02	4.83E+02	27%	1.34E+02	8.46E+04	9.75E+04	9.11E+04	10%	9.79E+04	100%	98%	U _R U _B
K (IE)	1.38E+05	1.59E+05	1.52E+05	1.50E+05	7.1%	<2.4E+03	1.75E+04	1.96E+04	1.86E+04	8.0%	2.26E+04	97%	N/A	-
La (IE)	2.00E+01	2.51E+01	3.55E+01	2.69E+01	29%	<1.8E+01	3.27E+01	3.17E+01	3.22E+01	2.2%	-	100%	N/A	U _E U _R
Li (IE)	<1.1E+02	<1.1E+02	<1.1E+02	<1.1E+02	-	<1.1E+02	1.33E+04	1.50E+04	1.42E+04	8.5%	1.49E+04	101%	N/A	U _M
Mg (IE)	<1.3E+01	<1.3E+01	<1.3E+01	<1.33E+01	-	<1.3E+01	4.30E+03	4.99E+03	4.65E+03	10%	5.20E+03	102%	N/A	-
Ni (IE)	1.20E+02	9.01E+01	1.50E+02	1.20E+02	25%	<3.4E+01	7.06E+03	8.11E+03	7.59E+03	9.8%	8.27E+03	101%	97%	U _E U _R
P (IE)	2.86E+03	2.20E+03	2.53E+03	2.53E+03	13%	<1.7E+02	5.51E+02	<1.7E+02	-	-	1.10E+03	100%	N/A	U _E
Pb (IE)	<8.0E+01	<8.0E+01	<8.0E+01	<8.0E+01	-	<8.0E+01	<8.0E+01	<8.0E+01	<8.0E+01	-	-	100%	N/A	-

* Average of two replicates

QC Flags: none - meets all QC

U_L - fails % Recovery of LCS

ND - not detected

U_R - fails %RSD criteria

U_S - fails % Recovery of MS

N/A - not applicable

U_M - fails minimum MRQ criteria

U_F - value less than 10x the DL

U_B - blank >5% of sample concentration

U_G - glass std >5% of sample concentration

Table 7.7. Composition of the Sodium Peroxide Fusion Digested As-Received 241-AW-101 Filterable Solids (Continued)

Analyte	1st Replicate (mg/Kg)	2nd Replicate (mg/Kg)	3rd Replicate (mg/Kg)	Average (mg/Kg)	%RSD	Blank (mg/L)	1st Glass Std (mg/Kg)	2nd Glass Std (mg/Kg)	Average (mg/Kg)	%RSD	Glass Std Comp (mg/Kg)	LCS % Recovery	MS % Recovery	QC Flag
U (IE)	3.45E+04	3.50E+04	3.45E+04	3.47E+04	0.8%	8.62E+03	3.77E+04	3.82E+04	3.80E+04	0.9%	-	100%	N/A	U _G
V (IE)	1.03E+02	1.10E+02	1.01E+02	1.05E+02	4.5%	2.64E+01	1.79E+02	1.97E+02	1.88E+02	6.8%	-	99%	N/A	U _E
U (CC)	6.34E+02	<1.0E-02	4.65E+02	5.50E+02*	22%	<1.0E-02	<1.0E-02	<1.0E-02	<1.0E-02	-	-	99%	98%	U _R
W (IM)	6.62E+01	5.70E+01	5.67E+01	6.00E+01	9.0%	8.26E-01	4.50E+00	4.52E+00	4.51E+00	0.2%	-	-	-	U _G
Th (IM)	1.26E+00	1.02E+00	9.01E-01	1.06E+00	17%	1.17E-01	1.31E+00	1.36E+00	1.33E+00	2.8%	-	101%	116%	U _G
Rb (IM)	1.29E+01	1.43E+01	1.41E+01	1.38E+01	5.5%	2.17E+00	5.30E+01	5.89E+01	5.60E+01	7.4%	-	100%	-	U _G
¹²⁷ I (IM)	1.10E+00	<7.1E-01	<7.1E-01	-	-	<7.1E-01	<7.1E-01	<7.1E-01	<7.1E-01	-	-	-	-	-
Cs ¹³³ (IM)	3.35E+00	4.26E+01	3.25E+00	1.64E+01	138%	1.78E+00	9.81E+00	1.43E+01	1.20E+01	26%	-	100%	108%	U _G
Cs ¹³⁵ (IM)	1.37E+00	1.09E+00	1.31E+00	1.26E+00	12%	3.53E-01	4.86E+01	5.36E+01	5.11E+01	7.0%	5.13E+01	104%	-	-
Cs ¹³⁷ (IM)	6.27E+00	4.74E+00	5.46E+00	5.49E+00	14%	7.30E-01	7.99E+01	8.94E+01	8.47E+01	7.9%	8.85E+01	97%	-	-
TIC (A)	9.92E-01	1.00E+00	9.96E-01	9.96E-01	0.4%	1.00E-03	9.88E-01	9.88E-01	9.88E-01	0.0%	-	104%	99%	U _G
TOC (Diff)	2.55E+04	1.97E+04	1.11E+04	1.88E+04	39%	-	1.51E+04	1.42E+04	1.47E+04	4.3%	-	N/A	N/A	U _G

* Average of two replicates

QC Flags: none - meets all QC

U_L - fails % Recovery of LCS

ND - not detected

U_R - fails %RSD criteria

U_S - fails % Recovery of MS

N/A - not applicable

U_M - fails minimum MRQ criteria

U_B - value less than 10x the DL

U_B - blank >5% of sample concentration

U_G - glass std >5% of sample concentration

Table 7.7. Composition of the Sodium Peroxide Fusion Digested As-Received 241-AW-101 Filterable Solids (Continued)

Analyte	1st Replicate (mCi/Kg)	2nd Replicate (mCi/Kg)	3rd Replicate (mCi/Kg)	Average (mCi/Kg)	%RSD	Blank (mCi/L)	1st Glass Std (mCi/Kg)	2nd Glass Std (mCi/Kg)	Average (mCi/Kg)	%RSD	Glass Std Comp (mCi/Kg)	LCS % Recovery	MS % Recovery	QC Flag
Cs ¹³⁷ (GS)	8.92E+01	7.57E+01	7.66E+01	8.05E+01	9.4%	3.07E-03	3.27E+00	5.77E-01	1.93E+00	99%	-	N/A	N/A	-
Eu ¹⁵⁴ (GS)	7.37E-03	<1.0E-03	8.93E-03	8.15E-03*	13%	1.51E-03	6.68E-03	4.99E-03	5.83E-03	21%	-	N/A	N/A	U _B U _G
Eu ¹⁵⁵ (GS)	6.17E-03	<1.2E-03	8.30E-03	7.23E-03*	21%	1.23E-03	6.45E-03	4.17E-03	5.31E-03	30%	-	N/A	N/A	U _R U _B U _G
Co ⁶⁰ (GS)	6.75E-03	<7.6E-04	7.04E-03	6.90E-03*	3%	1.53E-03	5.74E-03	4.81E-03	5.27E-03	13%	-	N/A	N/A	U _B U _G
Sn ¹²⁶ (GS)	<6.8E-04	<6.2E-04	<7.0E-04	<6.7E-04	-	<6.0E-04	<6.8E-04	<6.6E-04	<6.7E-04	-	-	N/A	N/A	-
Pa ²³¹ (GS)	<2.3E-02	<2.0E-02	<2.4E-02	<2.2E-02	-	<2.0E-02	<2.3E-02	<2.2E-02	<2.3E-02	-	-	N/A	N/A	U _M
Sr ⁹⁰ (SL)	7.06E+00	7.03E+00	7.09E+00	7.06E+00	0.4%	2.84E-01	-	-	-	-	-	101%	98%	-
Tc ⁹⁹ (SL)	1.02E-01	8.56E-02	8.75E-02	9.18E-02	10%	3.29E-03	-	-	-	-	-	tracer	tracer	-
Se ⁷⁹ (SL)	<3.3E-03	<2.8E-03	<3.1E-03	<3.1E-03	-	<6.7E-04	-	-	-	-	-	N/A	N/A	U _M
H ³ (SL)	<1.2E-02	<1.2E-02	<1.4E-02	<1.3E-02	-	<3.4E-03	-	-	-	-	-	99%	87%	-
Pu ²³⁸ (SA)	4.05E-02	3.93E-02	5.32E-02	4.43E-02	17%	1.45E-01	1.16E-01	3.37E-02	7.48E-02	78%	-	N/A	N/A	U _R U _B U _G
Pu ^{239/240} (SA)	7.57E-02	1.03E-01	8.92E-02	8.93E-02	15%	3.27E-02	3.54E-02	2.83E-02	3.18E-02	16%	-	N/A	N/A	U _R U _B U _G
Pu ²⁴¹ (SA)	2.11E-01	2.52E-01	1.81E-01	2.15E-01	17%	9.28E-02	9.95E-02	7.25E-02	8.60E-02	22%	-	N/A	N/A	U _R U _B U _G
Am ²⁴¹ (SG)	5.54E-02	4.48E-02	5.56E-02	5.19E-02	12%	1.30E-01	1.08E-01	2.79E-02	6.80E-02	83%	-	N/A	N/A	U _B U _G

* Average of two replicates

QC Flags: none - meets all QC

U_L - fails % Recovery of LCS

ND - not detected

U_R - fails %RSD criteria

U_S - fails % Recovery of MS

N/A - not applicable

U_M - fails minimum MRQ criteria

U_B - value less than 10x the DL

U_B - blank >5% of sample concentration

U_G - glass std >5% of sample concentration

Table 7.7. Composition of the Sodium Peroxide Fusion Digested As-Received 241-AW-101 Filterable Solids (Continued)

Analyte	1st Replicate (mCi/Kg)	2nd Replicate (mCi/Kg)	3rd Replicate (mCi/Kg)	Average (mCi/Kg)	%RSD	Blank (mCi/L)	1st Glass Std (mCi/Kg)	2nd Glass Std (mCi/Kg)	Average (mCi/Kg)	%RSD	Glass Std Comp (mCi/Kg)	LCS % Recovery	MS % Recovery	QC Flag
Cm ^{244/243} (SA)	1.96E-01	1.75E-01	2.34E-01	2.02E-01	15%	6.71E-01	6.37E-01	9.39E-02	3.65E-01	105%	-	N/A	N/A	U _B U _G
Cm ²⁴² (SA)	8.85E-05	5.36E-05	<1.0E-04	7.10E-05*	35%	<2.2E-04	<1.2E-04	3.50E-05	-	-	-	N/A	N/A	U _E
Tc ⁹⁹ (IM)	1.31E-01	1.10E-01	1.11E-01	1.17E-01	10%	<2.3E-03	2.51E-03	2.37E-03	2.44E-03	3.9%	-	95%	97%	-
Np ²³⁷ (IM)	<5.8E-05	<5.8E-05	<5.8E-05	<5.8E-05	-	<5.8E-05	<5.8E-05	<5.8E-05	<5.8E-05	-	-	-	-	-
Pu ²³⁹ (IM)	1.29E-02	1.04E-02	1.08E-02	1.14E-02	11%	<5.1E-03	<5.0E-03	<5.0E-03	<5.0E-03	-	-	-	-	U _E
Pu ²⁴⁰ (IM)	<1.8E-02	<1.8E-02	<1.8E-02	<1.8E-02	-	<1.9E-02	<1.8E-02	<1.8E-02	<1.8E-02	-	-	-	-	U _M
U ²³³ (IM)	<7.9E-04	<7.9E-04	<7.9E-04	<7.9E-04	-	<8.0E-04	<7.9E-04	<7.9E-04	<7.9E-04	-	-	-	-	U _M
U ²³⁴ (IM)	<5.4E-04	<5.4E-04	<5.4E-04	<5.4E-04	-	<5.5E-04	<5.4E-04	<5.4E-04	<5.4E-04	-	-	-	-	U _M
U ²³⁵ (IM)	4.13E-06	3.36E-06	3.60E-06	3.70E-06	11%	<1.8E-07	<1.8E-07	<1.8E-07	<1.8E-07	-	-	103%	98%	-
U ²³⁶ (IM)	9.27E-06	5.52E-06	7.26E-06	7.35E-06	26%	<5.4E-06	<5.3E-06	<5.3E-06	<5.3E-06	-	-	-	-	U _R
U ²³⁸ (IM)	5.85E-05	4.93E-05	4.91E-05	5.23E-05	10%	3.33E-06	3.01E-06	1.88E-06	2.45E-06	33%	-	101%	110%	U _B
Alpha (AC)	6.08E-01	3.18E-01	4.68E-01	4.65E-01	31%	1.28E+00	-	-	-	-	-	103%	95%	U _R U _B U _E
Alpha _{Sum}	-	-	-	1.86E-01	-	3.07E-01	-	-	1.75E-01	-	-	-	-	U _B U _G

* Average of two replicates

QC Flags: none - meets all QC

U_L - fails % Recovery of LCS

ND - not detected

U_R - fails %RSD criteria

U_S - fails % Recovery of MS

N/A - not applicable

U_M - fails minimum MRQ criteria

U_E - value less than 10x the DL

U_B - blank >5% of sample concentration

U_G - glass std >5% of sample concentration

Table 7.8. Composition of the Aqua-Regia Digested Diluted 5 M Sodium 241-AW-101 Filterable Solids

Analyte	1st Replicate (mg/Kg)	2nd Replicate (mg/Kg)	3rd Replicate (mg/Kg)	Average (mg/Kg)	%RSD	Blank (mg/L)	1st Glass Std (mg/Kg)	2nd Glass Std (mg/Kg)	Average (mg/Kg)	%RSD	Glass Std Comp (mg/Kg)	LCS % Recovery	MS % Recovery	QC Flag
Al (IE)	1.19E+05	1.09E+05	1.23E+05	1.17E+05	6.2%	<6.3E+01	2.05E+04	2.13E+04	2.09E+04	2.7%	2.50E+04	101%	N/A	-
Cr (IE)	3.49E+03	3.55E+03	3.60E+03	3.55E+03	1.6%	1.16E+01	5.84E+02	6.11E+02	5.98E+02	3.2%	6.40E+02	98%	99%	-
Fe (IE)	7.52E+03	7.59E+03	7.86E+03	7.66E+03	2.3%	9.71E+01	8.97E+04	9.55E+04	9.26E+04	4.4%	9.79E+04	101%	100%	-
Mn (IE)	3.12E+03	3.15E+03	3.17E+03	3.15E+03	0.8%	2.95E+00	1.43E+04	1.53E+04	1.48E+04	4.8%	1.46E+04	109%	108%	-
Na (IE)	1.71E+05	1.58E+05	1.73E+05	1.67E+05	4.9%	1.49E+03	3.47E+04	3.79E+04	3.63E+04	6.2%	8.52E+04	97%	100%	U _R
P (IE)	<1.7E+02	<1.7E+02	<1.7E+02	<1.7E+02	-	<7.6E+01	5.17E+02	5.64E+02	5.41E+02	6.1%	1.10E+03	106%	N/A	-
S (IE)	1.37E+03	1.47E+03	1.41E+03	1.42E+03	3.6%	<1.5E+02	1.26E+03	1.31E+03	1.29E+03	2.8%	-	98%	N/A	U _E
Si (IE)	4.51E+05	4.45E+05	4.56E+05	4.51E+05	1.2%	4.75E+02	1.66E+05	1.82E+05	1.74E+05	6.5%	2.24E+05	104%	N/A	-
U (IE)	4.25E+03	4.20E+03	4.21E+03	4.22E+03	0.6%	<2.5E+02	2.36E+03	2.44E+03	2.40E+03	2.4%	-	100%	N/A	U _E
U (CC)	7.09E+02	4.45E+02	4.69E+02	5.41E+02	27%	<1.0E-02	<1.0E-02	7.38E+03	-	-	-	97%	100%	U _R
TIC (A)	<2.2E+03	<2.2E+03	<2.2E+03	<2.2E+03	-	<2.2E+03	<4.0E+03	<4.0E+03	<4.0E+03	-	-	101%	88%	U _M
TOC (Diff)	1.10E+05	1.05E+05	9.91E+04	1.05E+05	5.1%	1.18E+05	4.51E+04	4.39E+04	4.45E+04	1.9%	-	N/A	N/A	U _B

* Average of two replicates

QC Flags: none - meets all QC

U_L - fails % Recovery of LCS

ND - not detected

U_R - fails %RSD criteria

U_S - fails % Recovery of MS

N/A - not applicable

U_M - fails minimum MRQ criteria

U_E - value less than 10x the DL

U_B - blank >5% of sample concentration

U_G - glass std >5% of sample concentration

Table 7.8. Composition of the Aqua-Regia Digested Diluted 5 M Sodium 241-AW-101 Filterable Solids (Continued)

Analyte	1st Replicate (mCi/Kg)	2nd Replicate (mCi/Kg)	3rd Replicate (mCi/Kg)	Average (mCi/Kg)	%RSD	Blank (mCi/L)	1st Glass Std (mCi/Kg)	2nd Glass Std (mCi/Kg)	Average (mCi/Kg)	%RSD	Glass Std Comp (mCi/Kg)	LCS % Recovery	MS % Recovery	QC Flag
Cs ¹³⁷ (GS)	6.00E+02	5.56E+02	6.23E+02	5.93E+02	5.7%	5.56E+00	6.72E+00	4.08E+00	5.40E+00	34%	-	N/A	N/A	-
Eu ¹⁵⁴ (GS)	3.13E-01**	3.70E-04	4.42E-04	4.06E-04*	13%	1.00E-03	-	-	-	-	-	N/A	N/A	U _B
Eu ¹⁵⁵ (GS)	1.98E-02**	3.33E-04	3.89E-04	3.61E-04*	11%	4.49E-03	-	-	-	-	-	N/A	N/A	U _B
Co ⁶⁰ (GS)	6.76E-04**	6.58E-04	7.16E-04	6.87E-04*	6.0%	3.77E-04	-	-	-	-	-	N/A	N/A	U _B
Am ²⁴¹ (GS)	<6.9E+00	<6.2E+00	<6.6E+00	<6.6E+00	-	<6.4E-01	<2.8E+00	<2.1E+00	<2.4E+00	-	-	N/A	N/A	-
Sr ⁹⁰ (SL)	1.01E+02	1.04E+02	1.08E+02	1.04E+02	3.1%	6.44E-04	-	-	-	-	-	116%	115%	-
Pu ^{239/240} (SA)	4.00E-01	6.85E-01	6.89E-01	5.91E-01	28%	2.40E-06	5.68E-01	3.56E-01	4.62E-01	32%	-	N/A	N/A	U _R
Am ²⁴¹ (SL)	9.32E-01	1.00E+00	1.09E+00	1.01E+00	8.0%	2.86E-02	1.79E-01	9.28E-02	1.36E-01	45%	-	N/A	N/A	U _G
Tc ⁹⁹ (IM)	5.57E-01	5.66E-01	5.49E-01	5.57E-01	1.5%	<1.2E-06	<2.1E-06	<2.1E-06	<2.1E-06	-	-	109%	93%	-
U ²³³ (IM)	<2.7E-07	<2.7E-07	<2.7E-07	<2.7E-07	-	<2.7E-07	<4.9E-07	<4.9E-07	<4.9E-07	-	-	-	-	-
U ²³⁴ (IM)	<1.8E-07	<1.8E-07	<1.8E-07	<1.8E-07	-	<1.3E-05	<3.2E-07	<3.2E-07	<3.2E-07	-	-	-	-	-
U ²³⁵ (IM)	7.24E-05	7.07E-05	6.96E-05	7.09E-05	1.9%	<6.1E-11	<1.1E-10	<1.1E-10	<1.1E-10	-	-	106%	104%	U _E
U ²³⁶ (IM)	1.75E-04	1.91E-04	1.89E-04	1.85E-04	4.8%	<1.8E-09	<3.3E-09	<3.3E-09	<3.3E-09	-	-	-	-	-
U ²³⁸ (IM)	1.37E-03	1.38E-03	1.34E-03	1.36E-03	1.6%	<9.4E-12	<1.7E-11	<1.7E-11	<1.7E-11	-	-	108%	115%	-
Pu ²³⁹ (IM)	3.78E-01	3.25E-01	3.47E-01	3.50E-01	7.6%	<1.7E-06	<3.1E-06	<3.1E-06	<3.1E-06	-	-	-	-	-
Pu ²⁴⁰ (IM)	1.54E-01	1.29E-01	1.47E-01	1.43E-01	9.0%	<6.3E-06	<1.1E-05	<1.1E-05	<1.1E-05	-	-	-	-	-
Alpha (AC)	8.87E+00	6.98E+00	8.69E+00	8.18E+00	13%	1.33E-04	-	-	-	-	-	78%	80%	-
Alpha _{Sum}	-	-	-	1.6E+00	-	2.92E-02	-	-	5.98E-01	-	-	-	-	U _G

* Average of two replicates

**Values are qualitative due to high dead time (see data evaluation)

QC Flags: none - meets all QC

U_L - fails % Recovery of LCS

ND - not detected

U_R - fails %RSD criteria

U_S - fails % Recovery of MS

N/A - not applicable

U_M - fails minimum MRQ criteria

U_E - value less than 10x the DL

U_B - blank >5% of sample concentration

U_G - glass std >5% of sample concentration

Table 7.9. Composition of the Water Contact of the As-Received 241-AW-101 Filterable Solids

Analyte	1st Replicate (mg/Kg)	2nd Replicate (mg/Kg)	3rd Replicate (mg/Kg)	Average (mg/Kg)	%RSD	Blank (mg/L)	LCS % Recovery	MS % Recovery	QC Flag
NO ₃ ⁻ (IC)	1.91E+05	1.59E+05	2.14E+05	1.88E+05	14%	<1.0E+01	101%	113%	-
NO ₂ ⁻ (IC)	3.65E+04	3.30E+04	3.28E+04	3.41E+04	6.2%	<1.0E+01	102%	104%	-
PO ₄ ³⁻ (IC)	1.99E+03	9.91E+02	2.31E+03	1.76E+03	39%	4.00E+00	101%	91%	U _R
SO ₄ ²⁻ (IC)	1.33E+03	<1.7E+03	<2.3E+03	-	-	<5.0E+00	101%	91%	U _E
C ₂ O ₄ ²⁻ (IC)	9.96E+02	9.91E+02	9.23E+02	9.70E+02	4.2%	<1.0E+01	102%	92%	-
Cl ⁻ (IC)	3.32E+03	1.32E+03	3.23E+03	2.62E+03	43%	4.00E+00	102%	91%	U _R
F ⁻ (IC)	6.64E+02	<6.6E+02	<9.2E+02	-	-	<2.0E+00	105%	95%	U _E
CHO ₂ ⁻ (IC)	2.66E+03	<3.3E+03	2.77E+03	2.71E+03*	2.9%	<1.0E+01	102%	92%	U _E
OH _{free} (T)	<1.1E+02	<1.1E+02	<1.6E+02	<1.3E+02	-	<2.0E-02	N/A	N/A	-
OH _{total} (T)	<1.1E+02	<1.1E+02	<1.6E+02	<1.3E+02	-	<2.0E-02	102%	N/A	-
TIC (A)	6.06E+03	2.45E+03	9.81E+03	6.11E+03	60%	<1.0E+00	102%	92%	U _R
TOC (Diff)	1.04E+04	8.82E+03	1.54E+04	1.15E+04	30%	7.06E+00	N/A	N/A	U _R
Al (IE)	1.16E+04	1.04E+04	1.08E+04	1.09E+04	5.6%	<9.3E-01	99%	N/A	-
Ba (IE)	<2.7E+01	<2.6E+01	<3.7E+01	<3.0E+01	-	<8.0E-02	99%	96%	U _M
Ca (IE)	<1.4E+01	<1.4E+01	<1.9E+01	<1.6E+01	-	1.88E-01	101%	103%	-
Cr (IE)	<1.0E+02	<1.0E+02	<1.4E+02	<1.1E+02	-	<3.1E-01	99%	97%	-
Fe (IE)	<1.3E+01	<1.3E+01	<1.8E+01	<1.5E+01	-	<4.0E-02	99%	95%	-
Na (IE)	1.43E+05	1.36E+05	1.42E+05	1.41E+05	2.7%	8.75E-01	99%	103%	-
Ni (IE)	<1.1E+02	<1.1E+02	<1.5E+02	<1.2E+02	-	<3.2E-01	99%	97%	U _M
P (IE)	7.01E+02	4.66E+02	<5.1E+02	5.83E+02*	29%	<1.1E+00	99%	N/A	U _E U _R
S (IE)	1.75E+02	1.38E+02	1.11E+02	1.42E+02	23%	<2.4E-01	101%	N/A	U _E U _R
Na (AA)	1.26E+05	1.20E+05	1.24E+05	1.23E+05	2.3%	2.59E-01	99%	97%	-
K (AA)	3.38E+04	3.14E+04	3.92E+04	3.48E+04	11%	9.10E-02	98%	96%	-

Provided as information only

*Average of two replicates

QC Flags: none - meets all QC

U_B - blank exceeds 5% of sample concentration

U_E - value less than 10x the DL

U_R - fails %RSD criteria

U_L - fails % Recovery of LCS

ND - not detected

U_M - fails minimum MRQ criteria

U_S - fails % Recovery of MS

N/A - not applicable

Table 7.9. Composition of the Water Contact of the As-Received 241-AW-101 Filterable Solids (Continued)

Analyte	1st Replicate (mg/L)	2nd Replicate (mg/L)	3rd Replicate (mg/L)	Average (mg/L)	%RSD	Blank (mg/L)	LCS % Recovery	MS % Recovery	QC Flag
Citrate (IC)	<3.3E+04	<3.3E+04	<4.6E+04	<3.7E+04	-	<1.0E+02	99%	-	U _M
Glycolate(IC)	<3.3E+04	<3.3E+04	<4.6E+04	<3.7E+04	-	<1.0E+02	100%	-	U _M
EDTA (HL)	<6.6E+03	<6.6E+03	<9.2E+03	<7.5E+03	-	<2.0E+01	102%	94%	U _M
HEDTA (HL)	<6.6E+03	<6.6E+03	<9.2E+03	<7.5E+03	-	<2.0E+01	96%	90%	U _M
IDA (GM)	<1.7E+04	<1.7E+04	<2.3E+04	<1.9E+04	-	<5.0E+01	96%	112%	U _M
NTA (GM)	<1.7E+04	<1.7E+04	<2.3E+04	<1.9E+04	-	<5.0E+01	96%	112%	U _M
ED3A (GM)	ND	ND	ND	-	-	ND	96%	112%	-

Provided as information only

QC Flags: none - meets all QC

U_B - blank exceeds 5% of sample concentration

U_E - value less than 10x the DL

U_R - fails %RSD criteria

U_L - fails % Recovery of LCS

ND - not detected

U_M - fails minimum MRQ criteria

U_S - fails % Recovery of MS

N/A - not applicable

Table 7.10. Composition of the 1st Condensate from Concentration of the Diluted 5 M Sodium 241-AW-101 Sample

Analyte	1st Replicate (mg/L)	2nd Replicate (mg/L)	3rd Replicate (mg/L)	Average (mg/L)	%RSD
NO ₃ ⁻ (IC)	1.63E+03	1.31E+03	1.02E+03	1.32E+03	23%
NO ₂ ⁻ (IC)	1.06E+03	7.91E+02	5.76E+02	8.09E+02	30%
PO ₄ ³⁻ (IC)	<1.0E+02	<1.0E+02	<1.0E+02	<1.0E+02	-
SO ₄ ²⁻ (IC)	<5.0E+01	<5.0E+01	<5.0E+01	<5.0E+01	-
C ₂ O ₄ ²⁻ (IC)	<1.0E+02	<1.0E+02	<1.0E+02	<1.0E+02	-
Cl ⁻ (IC)	4.10E+01	3.50E+01	2.60E+01	3.40E+01	22%
F ⁻ (IC)	<2.0E+01	<2.0E+01	<2.0E+01	<2.0E+01	-
CHO ₂ ⁻ (IC)	<1.0E+02	<1.0E+02	<1.0E+02	<1.0E+02	-
TIC (A)	2.77E+01	2.99E+01	2.73E+01	2.83E+01	4.9%
TOC (Diff)	4.33E+01	5.89E+01	5.41E+01	5.21E+01	15%
Al (IE)	3.00E+02	3.01E+02	2.97E+02	2.99E+02	0.7%
B (IE)	3.90E-01	3.83E-01	5.24E-01	4.32E-01	18%
Ba (IE)	<2.1E-01	<2.4E-02	<2.1E-02	<8.5E-02	-
Ca (IE)	<2.5E-01	1.07E-01	1.40E-01	1.24E-01*	19%
Cd (IE)	<4.6E-02	1.60E-02	2.70E-02	2.97E-02*	26%
Cr (IE)	8.69E-01	9.06E-01	1.28E+00	1.02E+00	22%
Fe (IE)	<4.0E-02	3.70E-02	3.90E-02	3.87E-02*	3.7%
Li (IE)	<4.7E-01	<5.4E-02	<4.7E-02	<1.9E-01	-
Mg (IE)	<5.8E-02	<7.0E-03	<6.0E-03	<2.4E-02	-
Na (IE)	2.52E+03	2.55E+03	2.51E+03	2.53E+03	0.8%
Ni (IE)	2.24E-01	7.70E-02	1.04E-01	1.35E-01	58%
P (IE)	2.54E+00	2.47E+00	3.45E+00	2.82E+00	19%
Pb (IE)	<3.5E-01	3.52E-01	3.93E-01	3.73E-01*	7.9%
Sr (IE)	<8.4E-02	1.10E-02	1.80E-02	3.77E-02*	13%
S (IE)	3.92E+00	3.89E+00	5.67E+00	4.49E+00	23%
V (IE)	<1.1E+01	<1.3E+00	<1.1E+00	<4.5E+00	-
Cs ¹³⁷ mCi/L (GS)	3.69E+00	3.65E+00	3.67E+00	3.67E+00	0.6%
Tc ⁹⁹ pertech mCi/L	1.58E-03	1.52E-03	1.56E-03	1.55E-03	2.1%

Provided as information only

*Average of two replicates

ND - not detected

Table 7.11. Composition of the 4th Condensate from Concentration of the Diluted 5 M Sodium 241-AW-101 Sample

Analyte	1st Replicate (mg/L)	2nd Replicate (mg/L)	3rd Replicate (mg/L)	Average (mg/L)	%RSD
NO ₃ ⁻ (IC)	<1.0E+02	<1.0E+02	<1.0E+02	<1.0E+02	-
NO ₂ ⁻ (IC)	<1.0E+02	<1.0E+02	<1.0E+02	<1.0E+02	-
PO ₄ ³⁻ (IC)	<1.0E+02	<1.0E+02	<1.0E+02	<1.0E+02	-
SO ₄ ²⁻ (IC)	<5.0E+01	<5.0E+01	<5.0E+01	<5.0E+01	-
C ₂ O ₄ ²⁻ (IC)	<1.0E+02	<1.0E+02	<1.0E+02	<1.0E+02	-
Cl ⁻ (IC)	<2.0E+01	<2.0E+01	<2.0E+01	<2.0E+01	-
F ⁻ (IC)	<2.0E+01	<2.0E+01	<2.0E+01	<2.0E+01	-
CHO ₂ ⁻ (IC)	<1.0E+02	<1.0E+02	<1.0E+02	<1.0E+02	-
TIC (A)	1.09E+01	9.72E+00	1.22E+01	1.09E+01	11%
TOC (Diff)	3.04E+01	2.77E+01	3.06E+01	2.96E+01	5.5%
Al (IE)	2.83E+00	2.80E+00	1.06E+00	2.23E+00	45%
B (IE)	<3.1E-02	<3.1E-02	<3.1E-02	<3.1E-02	-
Ba (IE)	<2.1E-02	<2.1E-02	<2.1E-02	<2.1E-02	-
Ca (IE)	6.80E-02	2.50E-02	<2.5E-02	4.65E-02*	65%
Cd (IE)	<5.0E-03	<5.0E-03	<5.0E-03	<5.0E-03	-
Cr (IE)	<7.0E-03	<7.0E-03	<7.0E-03	<7.0E-03	-
Fe (IE)	<4.0E-03	<4.0E-03	<4.0E-03	<4.0E-03	-
Li (IE)	<4.7E-02	<4.7E-02	<4.7E-02	<4.7E-02	-
Mg (IE)	<6.0E-03	<6.0E-03	<6.0E-03	<6.0E-03	-
Na (IE)	2.80E+01	2.77E+01	2.77E+01	2.78E+01	0.6%
Ni (IE)	1.70E-02	1.90E-02	<1.5E-02	1.80E-02*	7.9%
P (IE)	1.38E-01	1.22E-01	<7.6E-02	1.30E-01*	8.7%
Pb (IE)	<3.5E-02	<3.5E-02	<3.5E-02	<3.5E-02	-
Sr (IE)	<8.0E-03	<8.0E-03	<8.0E-03	<8.0E-03	-
S (IE)	<1.5E-01	<1.5E-01	<1.5E-01	<1.5E-01	-
V (IE)	<1.1E+00	<1.1E+00	<1.1E+00	<1.1E+00	-
Cs ¹³⁷ mCi/L (GS)	3.98E-02	4.06E-02	4.10E-02	4.05E-02	1.5%
Tc ⁹⁹ pertech mCi/L	<2.3E-05	<3.0E-05	<3.1E-05	<2.8E-05	-

Provided as information only

*Average of two replicates

ND - not detected

8.0 Comparison of the 241-AW-101 Sample to Specification 7

Specification 7 defines the Low Activity Waste Envelopes A, B, and C and the compositional and radionuclide limits for each of those envelopes. The as-received 241-AW-101 sample meets all of the specification limits with the exception of the Co^{60} and Eu^{154} . The 5 M Sodium 241-AW-101 sample meets all of the specification limits. The Co^{60} and Eu^{154} values for the as-received 241-AW-101 sample exceed the specification due to the high detection limits for these isotopes as a result of the high cesium content of the sample. A special separation to remove Cs^{137} from the 5 M Sodium sample shows the waste stream contains Co^{60} and Eu^{154} concentrations of less than 5% of the maximum.

Table 8.1. Comparison of the As-Received 241-AW-101 Filtered Supernate to Specification 7 Chemical Composition Limits.

Analyte	Average Value from Table 7.3 (M)	QC Flag	Average Analyte to Na Molar Ratio	Specification 7, Envelope A Maximum Ratio	% of Maximum	Meets Specification 7
Al	1.89E+00	-	1.67E-01	2.50E-01	67%	Yes
Ba	1.52E-05	U _E	1.34E-06	1.00E-04	1.3%	Yes
Ca	2.42E-04	U _E	2.15E-05	4.00E-02	0.1%	Yes
Cd	2.31E-05	U _E	2.04E-06	4.00E-03	0.1%	Yes
Cl	1.84E-01	-	1.63E-02	3.70E-02	44%	Yes
Cr	1.79E-03	U _E	1.59E-04	6.90E-03	2.3%	Yes
F	1.66E-02	-	1.47E-03	9.10E-02	1.6%	Yes
Fe	9.39E-05	U _E	8.33E-06	1.00E-02	0.1%	Yes
Hg	<1.3E-06	-	<1.1E-07	1.40E-05	0.8%	Yes
K	9.59E-01 ^a	-	8.50E-02	1.80E-01	47%	Yes
La	1.76E-05	U _E	1.56E-06	8.30E-05	1.9%	Yes
Ni	1.01E-04	U _E	8.98E-06	3.00E-03	0.3%	Yes
NO ₂	1.99E+00	-	1.77E-01	3.80E-01	46%	Yes
NO ₃	1.90E+00	-	1.69E-01	8.00E-01	21%	Yes
Pb	2.07E-04	-	1.83E-05	6.80E-04	2.7%	Yes
PO ₄	6.43E-03 ^b	-	5.70E-04	3.80E-02	1.5%	Yes
SO ₄	4.26E-03 ^b	-	3.78E-04	1.00E-02	3.8%	Yes
TIC	9.38E-02	-	8.32E-03	3.00E-01	2.8%	Yes
TOC	2.18E-01	-	1.93E-02	5.00E-01	3.9%	Yes
U	1.21E-05 ^c	U _R	1.07E-06	1.20E-03	0.1%	Yes

- a) The K value from ICP-ES was used in the table as being more conservative than the AA value.
b) P and S values from ICP-ES were used in the table as being more conservative than the IC values.
c) The total uranium was calculated from the ICP-MS values for mass 233, 234, 235, 236, and 238.

U_E value less than 10x the DL U_R fails %RSD criteria

Table 8.2. Comparison of the As-Received 241-AW-101 Filtered Supernate to Specification 7 Radionuclide Limits.

Radionuclide	Average Value from Table 7.3 (Bq)	QC Flag	Average Analyte to Na Molar Ratio	Specification 7, Envelope A Maximum Ratio	% of Maximum	Meets Specification 7
TRU	<1.3E+05 ^a	U _R	1.12E+04	4.80E+05	2.3%	Yes
Cs ¹³⁷	1.45E+10	-	1.28E+09	4.30E+09	30%	Yes
Sr ⁹⁰	<3.1E+06	-	2.74E+05	4.40E+07	0.6%	Yes
Tc ⁹⁹	6.26E+06 ^b	-	5.55E+05	7.10E+06	7.8%	Yes
Co ⁶⁰	<9.5E+06	U _M	<8.4E+05	6.10E+04	1400%	No
Eu ¹⁵⁴	<1.9E+07	U _M	<1.7E+06	1.20E+06	140%	No

- a) The TRU was calculated by summing the Pu²³⁸, Pu^{239/240}, Am²⁴¹, and Cm^{243/244} alpha spectroscopy results.
b) The Tc⁹⁹ result from ICP-MS for mass 99.

U_R fails %RSD criteria U_M fails MRQ criteria

Table 8.3. Comparison of the 5 M Sodium 241-AW-101 Filtered Supernate to Specification 7 Chemical Composition Limits.

Analyte	Average Value from Table 7.5 (M)	QC Flag	Average Analyte to Na Molar Ratio	Specification 7, Envelope A Maximum Ratio	% of Maximum	Meets Specification 7
Al	6.05E-01	-	1.22E-01	2.50E-01	49%	Yes
Ba	2.97E-05	U _E	6.00E-06	1.00E-04	6.0%	Yes
Ca	1.56E-04	-	3.14E-05	4.00E-02	0.1%	Yes
Cd	1.51E-05	U _E	3.05E-06	4.00E-03	0.1%	Yes
Cl	7.68E-02	-	1.55E-02	3.70E-02	42%	Yes
Cr	9.67E-04	-	1.95E-04	6.90E-03	2.8%	Yes
F	1.42E-02	-	2.87E-03	9.10E-02	3.2%	Yes
Fe	3.83E-05	U _E	7.73E-06	1.00E-02	0.1%	Yes
Hg	<1.1E-05	-	<2.2E-06	1.40E-05	<15%	Yes
K	5.98E-01 ^a	-	1.21E-01	1.80E-01	67%	Yes
La	1.07E-05	U _E	2.15E-06	8.30E-05	2.6%	Yes
Ni	7.22E-05	U _E	1.46E-05	3.00E-03	0.5%	Yes
NO ₂	1.13E+00	-	2.28E-01	3.80E-01	60%	Yes
NO ₃	1.61E+00	-	3.25E-01	8.00E-01	41%	Yes
Pb	1.08E-04	U _E	2.18E-05	6.80E-04	3.2%	Yes
PO ₄	5.36E-03 ^b	U _E	1.08E-03	3.80E-02	2.9%	Yes
SO ₄	2.67E-03 ^b	-	5.39E-04	1.00E-02	5.4%	Yes
TIC	7.65E-02	U _R	1.55E-02	3.00E-01	5.2%	Yes
TOC	8.27E-02	U _R	1.67E-02	5.00E-01	3.3%	Yes
U	5.57E-06 ^c	-	1.12E-06	1.20E-03	0.1%	Yes

- a) The K value from ICP-ES was used in the table as being more conservative than the AA value.
b) P and S values from ICP-ES were used in the table as being more conservative than the IC values.
c) The total uranium was calculated from the ICP-MS values for mass 235 and 238.

U_E value less than 10x the DL U_R fails %RSD criteria

Table 8.4. Comparison of the 5 M Sodium 241-AW-101 Filtered Supernate to Specification 7 Radionuclide Limits.

Radionuclide	Average Value from Table 7.5 (Bq)	QC Flag	Average Analyte to Na Molar Ratio	Specification 7, Envelope A Maximum Ratio	% of Maximum	Meets Specification 7
TRU	3.98E+04 ^a	U _R	8.05E+03	4.80E+05	1.7%	Yes
Cs ¹³⁷	6.50E+09	-	1.31E+09	4.30E+09	31%	Yes
Sr ⁹⁰	1.01E+06	-	2.03E+05	4.40E+07	0.5%	Yes
Tc ⁹⁹	3.18E+06 ^b	-	6.42E+05	7.10E+06	9.0%	Yes
Co ⁶⁰	1.27E+04	-	2.56E+03	6.10E+04	4.2%	Yes
Eu ¹⁵⁴	<7.8E+02	-	<1.6E+02	1.20E+06	0.01%	Yes

- a) The TRU was calculated by summing the Pu²³⁸, Pu^{239/240}, Am²⁴¹, and Cm^{243/244} alpha spectroscopy results.
b) The Tc⁹⁹ result from ICP-MS for mass 99.

U_R fails %RSD criteria

9.0 General Description of Analytical Procedures

9.1 Inductively Coupled Plasma-Atomic Emission Spectroscopy

Samples were diluted as necessary to bring analytes within the instrument range. A scandium internal standard was added to all samples after dilution at a concentration of 2 mg/L. Background and internal standard correction were applied to the results. An LCS containing the analytes of interest was run with the samples. A matrix spike containing Ba., Ca, Cr, Fe, Na, and Ni was run with each set of samples. The subset of elements used for the matrix spike was specified in the task plan.

9.2 Ion Chromatography for Anions and Organics Acids

For IC Anions, samples were diluted with a carbonate/bicarbonate diluent as necessary to bring analytes to within instrument calibration. An LCS containing the analytes of interest was analyzed concurrently with samples. One sample replicate was spiked with known concentrations of the analytes of interest for each set of samples.

Organic acids were determined by ion exclusion chromatography (IEC). Samples were diluted in a high salt solution as necessary to bring analytes to within instrument calibration. An LCS containing the analytes of interest was analyzed concurrently with samples. One sample replicate was spiked with known concentrations of the analytes of interest for each set of samples.

9.3 Free Hydroxide and Total Base Titrations

Total hydroxide (total base) was determined by titration to an inflection end point closest to pH 7.00. A LCS containing 1.00 N NaOH was run in triplicate before and after each set of samples.

Free hydroxide was determined by incremental inflection point titration. The titration curves were examined to determine the free hydroxide concentration. A lab control standard containing hydroxide, aluminate, and carbonate was run in triplicate before and after each set of samples.

Carbonate was determined by precipitation using saturated barium chloride. The precipitate was dissolved in acid and the carbonate concentration determined via back titration. A LCS containing hydroxide, aluminate, and carbonate was run in triplicate before and after each set of samples.

No matrix spikes were performed on any of the base titrations.

9.4 Atomic Absorption Spectroscopy

Sodium, potassium, and mercury were analyzed by AA. The mercury was determined using the cold vapor technique. Samples were diluted as necessary to bring analytes within the instrument calibration range. An LCS containing the analyte of interest was run with the samples. A matrix spike containing the analyte of interest was made on one of the sample replicates for each set of samples.

9.5 Ammonia

Ammonia was analyzed by ion (cation) chromatography after a purge and trap procedure to isolate the ammonia from the sample.

Due to high concentrations of sodium ions which interfere with the analysis of ammonium ions (NH_4^+), dissolved ammonia in the sample was purged using helium gas and trapped in an acidic solution for cation analysis. A 1 mL sample aliquot was added to a 2 molar hydroxide solution and purged for 15 minutes. An LCS containing the sample analyte was analyzed concurrently with samples. A sample replicate was spiked with the analyte at a known concentration, purged for 15 minutes, and trapped in an acidic solution for cations analysis. Measured values were adjusted to account for purge efficiency losses.

9.6 Organics

EDTA and HEDTA were analyzed by Ion Pair Chromatography (IPC). The copper complex of EDTA and HEDTA were used as the LCS. For the matrix spike, EDTA and HEDTA were spiked into the sample followed by preparation and analysis.

IDA, NTA, and ED3A were analyzed by GC-MS. These compounds were converted from carboxylic acids to methyl esters by BF_3 /methanol reagent for GC-MS analysis. The GC-MS instrument was calibrated using naphthalene-d8 as the LCS. The matrix spike was either Adipic acid or stearic-d35 acid which was then compared to acid methyl ester. Each sample was prepared in duplicate, one containing a spike of IDA and NTA. The IDA/NTA spike served as an internal standard.

9.7 Total Inorganic Carbon/Total Organic Carbon

Total carbon was determined by combustion at 780 degrees C in a stream of pure oxygen. The CO_2 produced was then measured. The inorganic carbon was determined by injecting an aliquot into an acid medium purged by an oxygen stream. Again, the CO_2 produced was then measured. By subtracting the inorganic carbon from the total carbon, the organic carbon was calculated. Instrument calibration used NIST traceable organic and inorganic standards before and after each set of samples. These standards also served as the LCS and were used for the matrix spike for the method. The matrix spike was made on one of the sample replicates.

9.8 Inductively Coupled Plasma-Mass Spectrometry

Samples were run concurrently with an LCS containing V, Co, As, Sr, Mo, Ru, Ag, Cd, Sb, Cs, Ba, La, Eu, Ho, Yb, Tl, Pb, Th, and U. This LCS provided a mass response covering most of the mass range of interest. However, the LCS did not cover every mass of species of interest such as I^{127} , and W isotopes so no LCS recovery was provided for these analytes. In general a matrix spike using the LCS was made on one of the sample replicates. For some of the sample results without matrix spike recovery's in the data tables, a matrix spike containing only U was used. The following describes the calculation of the analytes of interest from the mass values:

Rb	sum of mass 85 and 87
Tc ⁹⁹	mass 99. Subject to interference when Ru is present in the sample.
I ¹²⁷	mass 127. Value is unreliable due to memory effects.
Cs ¹³³	mass 133
Cs ¹³⁵	mass 135. Subject to interference when Ba is present in sample.
Cs ¹³⁷	mass 137. Subject to interference when Ba is present in sample.
W	Sum of mass 182, 183, 184, 186. Mass 180 and 181 not included in summation. The isobaric chain for mass 180 stops at the long lived Hf ¹⁸⁰ . The isobaric chain for mass 181 stops at the long lived Ta ¹⁸¹ .
Th	mass 232
U ²³³	mass 233
U ²³⁴	mass 234
U ²³⁵	mass 235
U ²³⁶	mass 236
U ²³⁸	mass 238
Np ²³⁷	mass 237
Pu ²³⁹	mass 239
Pu ²⁴⁰	mass 240

9.9 Uranium by Chemchek (Phosphorescence Method)

Samples were diluted as necessary to bring analytes within the instrument calibration range. An LCS containing the analyte of interest was run with the samples. A matrix spike containing uranium was made on one of the sample replicates for each set of samples. Uranium was determined from the phosphorescence exhibited when exposed to ultraviolet light from a nitrogen-pumped dye laser at 420 nm.

9.10 Alpha Counting

Prior to the analysis, Cs¹³⁷ was removed from aliquots of the samples in order to reduce bias effects caused by large beta/alpha ratios. The Cs¹³⁷ removal was accomplished using Bio-Rad AMP1 resin. Following the Cs removal process, an aliquot of each stripped solution was added to a stainless steel planchet and analyzed for alpha activity.

9.11 Gamma Spectrometry

An aliquot of each sample was analyzed by gamma spectroscopy analysis using a high purity germanium detector. Results are background subtracted.

9.12 H³ Analysis

For the tritium analysis, an aliquot of each sample was subjected to a steam distillation to separate the tritium containing fraction from the remainder of the sample. An aliquot of each distillate was added to liquid scintillation cocktail to be analyzed for tritium. The samples were counted on a Packard Instruments liquid scintillation counter along with an instrument blank. The instrument blank was counted first and was used to establish an instrument background that was subtracted from the count results for the samples.

9.13 Tc^{99m} Analysis (for Pertech netate Form)

Tc^{99m} tracers were generated initially by neutron irradiation of natural molybdenum using SRTC's Cf²⁵² Neutron Activation Analysis facility. Mo⁹⁸ was activated to Mo⁹⁹ which subsequently beta decays to Tc^{99m}. The Tc^{99m} was then extracted from the Mo⁹⁹ to form a Tc^{99m} tracer.

Aliquots of the sample were diluted with water, Tc^{99m} tracer was added, and the Tc⁹⁹ was subsequently extracted using an Aliquat 336 based extraction. Aliquat 336 extracts Tc⁹⁹ in the pertechnetate form. The extractant was then analyzed first by gamma spectroscopy to determine Tc^{99m} tracer recoveries, and then analyzed by liquid scintillation analysis to determine Tc⁹⁹. Tc^{99m} tracer recoveries were applied to the liquid scintillation results to quantify the Tc⁹⁹. A blank solution was also run through the extraction process to ensure no cross contamination existed at the laboratory level.

9.14 Sr⁹⁰ Analysis

An aliquot of each sample was analyzed for Sr⁹⁰ using an Eichrom Sr-Spec based extraction procedure. A Sr⁹⁰ spiked blank was analyzed with the sample batch to establish Sr⁹⁰/Y⁹⁰ counting efficiencies and Sr chemical recoveries. Aliquots of each sample's Sr extract were analyzed by neutron activation analysis to determine Sr carrier recoveries, the results of which were normalized to the results of the Sr⁹⁰ spiked blank sample so each sample could be yielded by the recovery of its stable Sr carrier recovery. The LCS and matrix spike samples were treated exactly like the samples. Once the extractions were complete, aliquots of the resultant Sr⁹⁰/Y⁹⁰ containing extracts mixed with liquid scintillation cocktail were counted in the ADS Radiochemistry Counting Facility. The samples were counted on a Packard Instruments liquid scintillation counter along with an instrument blank. The instrument blank was counted first and was used to establish an instrument background that was subtracted from the count results for the samples.

9.15 Se⁷⁹ Analysis

Aliquots of the sample were spiked initially with stable Se which acted as both a chemical carrier and a Se yield tracer for the Se⁷⁹ measurements. The samples were then oxidized. Next the solutions were reduced to precipitate out Se metal. The Se metal was washed repeatedly, redissolved, and the dissolution was then subjected to a series of decontamination steps with several types of analytical resins added in batch mode, which were subsequently filtered off. The decontaminated solutions were then concentrated. Aliquots of the concentrate were analyzed by neutron activation analysis to determine Se carrier yields, and by liquid scintillation to measure Se⁷⁹ activities.

9.16 Alpha Spectroscopy for Plutonium Isotopics

An aliquot of each sample dissolution was subjected to a thenoyltrifluoroacetone (TTA) separation. An aliquot of the sample dissolution was initially spiked with a Pu²³⁸ tracer. A second aliquot of straight sample dissolution was analyzed along with the spiked sample. In addition, a third aliquot was used for determining the Pu²⁴¹ concentration. All of the plutonium in the samples was reduced once using hydroxylamine. An anion complexing reagent (aluminum nitrate) was then added, and the solutions were oxidized with 4M sodium nitrite. The plutonium was then extracted from the matrix using a TTA solution. The TTA layer was mounted on a counting dish, the mount was then analyzed by alpha spectroscopy. A blank sample was run with the sample set.

The analysis results for the Pu^{239/240} alpha peak were yielded using the Pu²³⁸ recoveries from the Pu²³⁸ traced sample separation. The ratio of the Pu^{239/240} to the Pu²³⁸ in the sample was obtained from the alpha spectroscopy analysis of the non-spiked sample. That ratio was applied to the determined Pu^{239/240} value to determine the Pu²³⁸ activity in the sample.

The sample aliquot dedicated to the Pu²⁴¹ analysis was added to liquid scintillation cocktail following the separation and analyzed for both Pu²⁴¹ and gross Pu-alpha constituents. The ratio of Pu²⁴¹ to total Pu alpha was determined and applied to the results from the plates in order to determine a Pu²⁴¹ concentration.

9.17 I¹²⁹ Analysis

An aliquot of each sample was spiked with stable iodide and was subjected to a silver iodide precipitation method to separate any iodide in the matrix from other radionuclides. A blank DI water sample was analyzed along with the batch. The precipitates were analyzed for I¹²⁹ activity with a low energy HPGe gamma spectroscopy detector. After the gamma analyses, the precipitates were analyzed by neutron activation analysis (NAA) to determine the levels of stable iodide carrier in the precipitates. The recoveries of the iodide carrier were used to correct the gamma spectroscopy results for the I¹²⁹ recoveries. Uncertainties provided are 1 sigma.

9.18 Am/Cm Analysis

Aliquots of sample were run through a Am/Cm separation procedure to separate the trivalent Am/Cm isotopes from the higher valence state actinides following a sample oxidation step. Samples were run through the procedure in duplicate, one sample spiked with Am²⁴³ for yielding purposes, one sample unspiked to correct for any Am²⁴³ that may be present in the samples. The Am/Cm sample mount was analyzed by alpha spectroscopy for Cm^{244/243} and Cm²⁴² and by low energy gamma spectrometry for Am²⁴¹. The results were yielded by using the Am²⁴³ tracer alpha result and the Am²⁴³ tracer gamma result. A blank sample was run through all of the analyses with every batch of samples.

9.19 C¹⁴ Analysis

Aliquots of sample were wet-ashed with a sodium persulfate/silver nitrate oxidation in conjunction with concentrated sulfuric acid. The carbon dioxide emitted was absorbed with Packard Instruments Carbosorb E. The Carbosorb E was then slurried into Ultima Gold AB, and analyzed by liquid scintillation analysis for C¹⁴. Each sample was run through the process in duplicate. A blank solution, spiked with a C¹⁴ standard, was run (in duplicate) in parallel with the samples to determine C¹⁴ recoveries. The average recoveries were applied to the sample results to quantify the C¹⁴ concentrations. A second blank solution, spiked with the C¹⁴ standard was also run in duplicate through the process to serve as the laboratory control sample. One customer sample was spiked with some C¹⁴ (again in duplicate) and run through the process to serve as the matrix spike. A blank solution was also run through the entire process to ensure no cross contamination existed at the laboratory level.

10.0 References

1. E. Lee, I. Burgeson, "Tank 241-AW-101 Sample Composite, Homogeneity, Analysis, and Dilution - Test Specification", 24590-WTP-TSP-RT-01-001, Rev. 0, August 10, 2001.
2. M. Hay, "Task Technical and Quality Assurance Plan for the Characterization and Dilution of Samples from Hanford Tank 241-AW-101", WSRC-TR-2001-00244, SRT-RPP-2001-00063, Rev. 0, September 14, 2001, Westinghouse Savannah River Company, Aiken, SC.
3. Hay, M. S., Edwards, T. B., "Statistical Analysis of ESP Verification Test Samples", WSRC-RP-94-1224, November 4, 1994, Westinghouse Savannah River Company, Aiken, SC.
4. C. J. Coleman, R. A. Dewberry, M. F. Bryant, J. J. Gemmill, "SRL's Performance in Round Robin #6 - Analysis of Simulated Defense Waste Glass (U)", WSRC-TR-91-187, Rev. 0, May 31, 1991, Westinghouse Savannah River Company, Aiken, SC.

Appendix 1

- Letter of Instruction to Provide Sample Material to Waste Treatment Plant Contractor
- Spreadsheet of Sample Description
- Chain of Custody Documentation

Tank 241-AW-101, Letter of Instruction to Provide Sample Material
To Waste Treatment Plant Contractor.

1. Title:

Letter of Instruction to Provide Tank 241-AW-101 Sample Material to Waste Treatment Plant Contractor

2. Suggested Contractor:

Fluor Hanford 222-S Laboratory

3. Revision Number:

0

4. Date:

May 23, 2000

5. Objective:

In accordance with Interface Control Document (ICD) 23, tank 241-AW-101 waste samples are to be provided to the waste treatment plant contractor to be used for Process Verification and Waste Form Qualification Testing (BNFL 2000). This Letter of Instruction (LOI) directs the Characterization Project Operations (CPO) to obtain the required grab samples and for the 222-S Laboratory to receive, examine, and package sample material from tank 241-AW-101 for shipment to the waste treatment plant contractor.

6. Background/Introduction:

The River Protection Project (RPP) will provide samples from tank 241-AW-101 to the waste treatment plant contractor consisting of 15 L of the liquid phase to be sent by September 1, 2000. Archived liquid samples at 222-S Laboratory of approximately 1.2 L from prior sampling will be used to augment the new grab sample volume of 14 L. The 222-S Laboratory will estimate the quantities of liquid phase from archive based on sample weights, densities, volumes, and visual observations using information from previous sample events. The sample will be packaged for shipment in compliance with the appropriate transportation requirements as directed by waste treatment plant contractor BNFL (2000). Analyses of previous samples from tank 241-AW-101 will satisfy transportation requirements. Consequently, no additional analyses of the tank 241-AW-101 samples are required at this time.

7. Scope

This LOI directs the CPO to (1) obtain the required grab samples, (2) ship the samples to the 222-S Laboratory, and (3) provide sampling information to the tank 241-AW-101 tank coordinator. This LOI also directs the 222-S Laboratory to receive and process sample material from tank 241-AW-101 for shipment to the waste treatment plant contractor.

8. Functional Requirements:

CPO shall obtain a total of twenty-eight 500-mL grab samples at five elevations within the tank 241-AW-101 from riser 022 in accordance with this LOI. The sample material will be obtained from grab samples from 5 equally spaced levels in the liquid phase of the tank as shown in Table 1. The surveillance measurements indicates the surface level is 409 inches from the bottom of the tank which is the equivalent of 4,258 kL (1,125 kgals) (CHG 2000). The current best basis inventory estimates the solids level is at 111 inches from the bottom with a floating crust (69 kgals) estimated at 25 inches (CHG 2000). Therefore, the first of five levels sampled will be at 370 inches from the tank bottom. The fifth level sampled will be at 130 inches from the bottom. The 14 L of supernatant will be comprised of 3 levels at 3 L each (6*0.5L) and 2 levels of 2.5 L each (5*0.5 L). as listed in Table 1. A sludge weight is recommended to be used on the sample bottle holder to penetrate the crust.

If quality-affecting changes to the sampling requirements must be made (including the riser, sampling truck, or sample elevation to be sampled), the change must be recorded and approved by the cognizant engineer and tank coordinator before sampling. This information may be recorded on a permanent data sheet or recorded directly in sampling work packages.

CPO will record at the time of sampling (1) the riser location and sample elevation for each sample, (2) the appearance of each sample at the time of sampling (note if liquid is clear or cloudy and if solids are present, and (5) a description of any unusual observations of event during the sampling.

Table 1. Tank 241-AW-101 Supernate Sampling Information

Sample Number	Sample Type	Sample Location	Elevation From Tank Bottom (inches) ¹	Cable Length From Top of Sample Riser (inches) ²
1AW-00-1	500-mL grab	Riser 022	370	297
1AW-00-2	500-mL grab	Riser 022	370	297
1AW-00-3	500-mL grab	Riser 022	370	297
1AW-00-4	500-mL grab	Riser 022	370	297
1AW-00-5	500-mL grab	Riser 022	370	297
1AW-00-6	500-mL grab	Riser 022	370	297
1AW-00-7	500-mL grab	Riser 022	310	357
1AW-00-8	500-mL grab	Riser 022	310	357
1AW-00-9	500-mL grab	Riser 022	310	357
1AW-00-10	500-mL grab	Riser 022	310	357
1AW-00-11	500-mL grab	Riser 022	310	357
1AW-00-12	500-mL grab	Riser 022	310	357
1AW-00-13	500-mL grab	Riser 022	250	417
1AW-00-14	500-mL grab	Riser 022	250	417
1AW-00-15	500-mL grab	Riser 022	250	417
1AW-00-16	500-mL grab	Riser 022	250	417
1AW-00-17	500-mL grab	Riser 022	250	417
1AW-00-18	500-mL grab	Riser 022	190	477
1AW-00-19	500-mL grab	Riser 022	190	477
1AW-00-20	500-mL grab	Riser 022	190	477
1AW-00-21	500-mL grab	Riser 022	190	477
1AW-00-22	500-mL grab	Riser 022	190	477
1AW-00-23	500-mL grab	Riser 022	130	537
1AW-00-24	500-mL grab	Riser 022	130	537
1AW-00-25	500-mL grab	Riser 022	130	537
1AW-00-26	500-mL grab	Riser 022	130	537
1AW-00-27	500-mL grab	Riser 022	130	537
1AW-00-28	500-mL grab	Riser 022	130	537

¹Sample elevation is defined as the distance from the bottom of the tank to the mouth of the sample bottle.

²Cable length is defined as the distance from the top of the tank riser to the mouth of the sample

Shipping information: Supernate Radionuclides highest conc.): Cs¹³⁷ and Sr⁹⁰

TBq Content 500 mL bottle: 0.095 TBq/bottle

This LOI instructs the Laboratory to:

- Visually inspect, weigh, and photograph samples as they are received at the Laboratory. CHG will provide the tare weights of the capped sample bottles.
- Estimate the volume % settled solids after 1, 2, and 7 working days of settling for each sample containing > 5% solids by volume by visual inspection. Photograph the bottles containing settled solids after completion of settling and report volume % settled solids for each sample containing over >5% solids to tank coordinator by e-mail.

The tank coordinator may request additional supernatant samples in order to accumulate the 15 L required by BNFL (2000). CPO shall obtain one or more additional samples from tank 241-AW-101 when requested by the tank coordinator.

9. Deliverables

- Archived samples (that have not been composited) will be used to augment the quantity requirements. The estimated volume % of liquid for the archived samples shall be reported to the tank coordinator by e-mail before the first sample receipt at the 222-S Laboratory.
- The tank 241-AW-101 grab samples should be shipped to the 222-S Laboratory within 3 days of CPO obtaining the samples. It is recognized that conditions may not be amenable in order to comply with this requested timetable. Therefore, transport of the samples to the 222-S Laboratory should be done at the first opportunity if not within the requested time period.
- The tank coordinator shall be provided with the following information within one day of completing shipment of the samples: (1) completed chain of custody forms for each sample, (2) The riser location and sample elevation for each sample, (3) the appearance of each sample at the time of sampling (note if liquid is clear or cloudy and if solids are present, and (4) a description of any unusual observations of event during the sampling.
- The estimated volume % solids for the grab samples shall be reported to the tank coordinator by e-mail within 10 working days of sample receipt at the 222-S Laboratory.
- A supporting document describing the appearance, weight, and solids content of each sample shall be issued by 222-S laboratory for public release. This document shall contain the methodology for estimating the volume % solids in each sample containing over 5 % settled solids.
- The tank 241-AW-101 supernatant samples are to be shipped to the waste treatment plant contractor by September 1, 2000.

10. Acceptance Criteria

Characterization Project Operations (CPO) should transport each grab sample collected to the 222-S Laboratory within 3 days. Transport of the sample to the 222-S Laboratory should be done at the first opportunity if not within the requested time period. A verbal notification by CPO is to be made to the 222-S Laboratory at 373-2435 at least 24 hours in advance of an expected shipment.

The 222-S Laboratory estimate of the volume of liquid shall provide confidence that the shipment to waste treatment plant contractor contains 15 L of AW-101 liquid phase.

Processes, services, activities, and conditions adverse to the quality, which do not conform to requirements specified in this LOI or references herein, shall be controlled to prevent inadvertent use. Nonconforming sampling and analysis processes shall be identified, controlled, reported, and the disposition taken as required by the *Nonconforming Item Reporting and Control* (CHG 1999).

11. Period of Performance

The work described herein shall be completed between May 15, and September 1, 2000.

12. Technical Point of Contact

The technical point of contact for this work scope is the RPP tank coordinator for tank 241-AW-101, S. G. McKinney.

13. Applicable Environmental, Safety, and Health (E, S, & H) Requirements

All applicable DOE contractual E, S, & H Requirements hold for this sampling event. Field sampling, sample handling, and transportation shall be performed per approved CPO and/or 222-S Laboratory work packages and procedures. No additional CHG E, S, & H requirements are imposed by this LOI.

14. Safety Requirements

All DOE contractual safety requirements hold for this sampling event. For the work to be performed at the waste treatment plant contractor the Contractor's safety requirements apply. No additional CHG safety requirements are imposed by this LOI.

15. Requirements Including Quality Assurance Requirements

The subcontractor is subject to Title 10, Code of Federal Regulations, Part 830.120, *Quality Assurance Requirements*, and the enforcement actions under 10 CFR 820, *General Statement of Enforcement Policy*.

16. Hold Points

n/a

17. Configuration Management Requirements

n/a

18. Work Location/Access Requirements

This work shall be performed in accordance with the appropriate CPO sampling protocols and procedures. Sample examination and packaging shall be performed at the 222-S Laboratory. The tank coordinator or his designee shall have access to inspect the sample.

19. Provided Items

n/a

20. Training

n/a

21. Qualifications

n/a

22. Special Requirements

n/a

23. Reporting/Administration

n/a

Table 4. Tank 241-AW-101 Project Points of Contact and Key Personnel List

Responsibility	Organization	Individual
RPP 241-AW-101 Tank Coordinator	RPP Process Engineering (CHG)	S. G. McKinney
222-S Laboratory Point of Contact (day shift)	Analytical Services (FH)	S. N. Bakhtiar, 373-3015
222-S Laboratory Point of Contact	Analytical Services (FH)	222-S Laboratory Shift Manager, 373-2435
Client Services Point of Contact	Analytical Services (FH)	K. L. Powell, 372-0939
200 East Tank Farm Point of Contact	Tank Farm Operations (CHG)	East Tank Farm Operations Shift Manager, 373-3475
Data Management	Manager, Data Development and Interpretation (CHG)	J. G. Field, 376-3753
Field Sampling Point of Contact	RPP Characterization Project Operations (CHG)	J. F. Sickels, 373-0259
Process Engineering Point of Contact for Immediate Notifications	RPP Process Engineering (CHG)	On-Call Process Engineer, 539-2074 or 85-9654 (pager)
Process Chemistry Point of Contact	Manager, Technology, Operations and Process Science (NHC)	L. L. Lockrem, 373-4771
Waste treatment plant contractor Point of Contact	(TBD)	(TBD)
WIT Point of Contact	PNNL	I. E. Burgeson, 372-3650

References:

BNFL, 2000, *Interface Control Document ICD-23 Between DOE and BNFL Inc. for Waste Treatability Samples*, BNFL-5193-ID-23, Rev. 3c.rl, BNFL Inc., Richland, Washington.

CHG, 1999, *Nonconforming Item Reporting and Control*, RPP-PRO-298, Rev. 0, CH2M HILL Hanford Group, Inc., Richland, Washington.

CHG 2000, *Tank Characterization Database at <http://twins.pnl.gov:8001/TCD/main.html>*, CH2M HILL Hanford Group, Inc., Richland, Washington.

Approvals

S. G. McKinney, Tank 241-AW-101 Coordinator
Data Development and Interpretation
CH2M HILL Hanford Group, Inc.

J. G. Field, Manager
Data Development and Interpretation
CH2M HILL Hanford Group, Inc.

S. N. Bakhtiar, Manager
222-S Laboratory Operations
Fluor Daniel Hanford

J. F. Sickels, Manager
Characterization Project Operations
CH2M HILL Hanford Group, Inc.

Sampling History of 241-AW-101 Sample										
Task Name	Sampling Event	Sample Type/Core Segment ID	Device Type	Riser	Sampling Begin Date	Sampling End Date	Sampling Level	Sampling Level Units	Recovery Percentage	
241-AW-101	1AW-00-1	SUPERNATE	GRAB	22	7/12/00 0:00	7/12/00 0:00				
241-AW-101	1AW-00-10	SUPERNATE	GRAB	22	7/12/00 0:00	7/12/00 0:00				
241-AW-101	1AW-00-11	SUPERNATE	GRAB	22	7/12/00 0:00	7/12/00 0:00				
241-AW-101	1AW-00-12	SUPERNATE	GRAB	22	7/12/00 0:00	7/12/00 0:00				
241-AW-101	1AW-00-13	SUPERNATE	GRAB	22	7/13/00 0:00	7/18/00 0:00				
241-AW-101	1AW-00-14	SUPERNATE	GRAB	22	7/13/00 0:00	7/18/00 0:00				
241-AW-101	1AW-00-15	SUPERNATE	GRAB	22	7/13/00 0:00	7/18/00 0:00				
241-AW-101	1AW-00-16	SUPERNATE	GRAB	22	7/13/00 0:00	7/18/00 0:00				
241-AW-101	1AW-00-17	SUPERNATE	GRAB	22	7/13/00 0:00	7/18/00 0:00				
241-AW-101	1AW-00-18	SUPERNATE	GRAB	22	7/13/00 0:00	7/18/00 0:00				
241-AW-101	1AW-00-19	SUPERNATE	GRAB	22	7/13/00 0:00	7/18/00 0:00				
241-AW-101	1AW-00-2	SUPERNATE	GRAB	22	7/12/00 0:00	7/12/00 0:00				
241-AW-101	1AW-00-20	SUPERNATE	GRAB	22	7/13/00 0:00	7/18/00 0:00				
241-AW-101	1AW-00-21	SUPERNATE	GRAB	22	7/13/00 0:00	7/17/00 0:00				
241-AW-101	1AW-00-22	SUPERNATE	GRAB	22	7/13/00 0:00	7/17/00 0:00				
241-AW-101	1AW-00-23	SUPERNATE	GRAB	22	7/13/00 0:00	7/17/00 0:00				
241-AW-101	1AW-00-24	SUPERNATE	GRAB	22	7/13/00 0:00	7/17/00 0:00				
241-AW-101	1AW-00-25	SUPERNATE	GRAB	22	7/13/00 0:00	7/17/00 0:00				
241-AW-101	1AW-00-26	SUPERNATE	GRAB	22	7/13/00 0:00	7/17/00 0:00				
241-AW-101	1AW-00-27	SUPERNATE	GRAB	22	7/13/00 0:00	7/17/00 0:00				
241-AW-101	1AW-00-28	SUPERNATE	GRAB	22	7/13/00 0:00	7/17/00 0:00				
241-AW-101	1AW-00-3	SUPERNATE	GRAB	22	7/12/00 0:00	7/12/00 0:00				
241-AW-101	1AW-00-4	SUPERNATE	GRAB	22	7/12/00 0:00	7/12/00 0:00				
241-AW-101	1AW-00-5	SUPERNATE	GRAB	22	7/12/00 0:00	7/12/00 0:00				
241-AW-101	1AW-00-6	SUPERNATE	GRAB	22	7/12/00 0:00	7/12/00 0:00				
241-AW-101	1AW-00-7	SUPERNATE	GRAB	22	7/12/00 0:00	7/12/00 0:00				
241-AW-101	1AW-00-8	SUPERNATE	GRAB	22	7/12/00 0:00	7/12/00 0:00				
241-AW-101	1AW-00-9	SUPERNATE	GRAB	22	7/12/00 0:00	7/12/00 0:00				

Physical Appearance (continued)	Segment Serial Number	Sampling Event Comment	Segment Comment	Sample Number
Sample was allowed to settle for 10-13 days before visual description was recorded.				BOHB46T
Sample was allowed to settle for 10-13 days before visual description was recorded.				BOHB55T
Sample was allowed to settle for 10-13 days before visual description was recorded.				BOHB56T
Sample was allowed to settle for 10-13 days before visual description was recorded.				BOHB57T
Sample was allowed to settle for 10-13 days before visual description was recorded.				BOHB67T
Sample was allowed to settle for 10-13 days before visual description was recorded.				BOHB66T
Sample was allowed to settle for 10-13 days before visual description was recorded.				BOHB88T
Sample was allowed to settle for 10-13 days before visual description was recorded.				BOHB69T
Sample was allowed to settle for 10-13 days before visual description was recorded.				BOHB70T
Sample was allowed to settle for 10-13 days before visual description was recorded.				BOHB71T
Sample was allowed to settle for 10-13 days before visual description was recorded.				BOHB72T
Sample was allowed to settle for 10-13 days before visual description was recorded.				BOHB47T
Sample was allowed to settle for 10-13 days before visual description was recorded.				BOHB73T
Sample was allowed to settle for 10-13 days before visual description was recorded.				BOHB58T
Sample was allowed to settle for 10-13 days before visual description was recorded.				BOHB59T
Sample was allowed to settle for 10-13 days before visual description was recorded.				BOHB60T
Sample was allowed to settle for 10-13 days before visual description was recorded.				BOHB61T
Sample was allowed to settle for 10-13 days before visual description was recorded.				BOHB62T
Sample was allowed to settle for 10-13 days before visual description was recorded.				BOHB63T
Sample was allowed to settle for 10-13 days before visual description was recorded.				BOHB64T
Sample was allowed to settle for 10-13 days before visual description was recorded.				BOHB65T
Sample was allowed to settle for 10-13 days before visual description was recorded.				BOHB48T
Sample was allowed to settle for 10-13 days before visual description was recorded.				BOHB49T
Sample was allowed to settle for 10-13 days before visual description was recorded.				BOHB50T
Sample was allowed to settle for 10-13 days before visual description was recorded.				BOHB51T
Sample was allowed to settle for 10-13 days before visual description was recorded.				BOHB52T
Sample was allowed to settle for 10-13 days before visual description was recorded.				BOHB53T
Sample was allowed to settle for 10-13 days before visual description was recorded.				BOHB54T

CHAIN OF CUSTODY/SAMPLE ANALYSIS REQUEST

C.O.C. No. _____

Page 1 of 1

Collector N/A	Contact/Requestor Ruth Esch	Telephone No. 373-4314	MSIN 56-12	FAX 372-1878
SAF No. N/A	Sample Origin Tank 241-AW-101	Purchase Order/Charge Code N/A	Temp. N/A	
Project Title AW-101 IC023 Shipment	Logbook No. N/A	Ice Chest No. N/A	Bill of Lading/Air Bill No. N/A	
Shipped To (Lab) SRTC, Bldg. 773A, East Wing Truck Dock	Method of Shipment PMS-1 CASE	Offsite Property No. N/A		
Protocol N/A	Data Turnaround N/A			

Sample No.	Lab ID	Date	Time	No./Type Container	Net. Wt.	Sample Analysis	Preservative
LAW-00-1	S00T001466	L	N/A	1/500 mL glass	Net. Wt. 671 g		None
LAW-00-2	S00T001467	L	N/A	1/500 mL glass	Net. Wt. 665 g		None
LAW-00-3	S00T001468	L	N/A	1/500 mL glass	Net. Wt. 672 g		None
LAW-00-4	S00T001469	L	N/A	1/500 mL glass	Net. Wt. 678 g		None
LAW-00-5	S00T001470	L	N/A	1/500 mL glass	Net. Wt. 679 g		None
LAW-00-6	S00T001471	L	N/A	1/500 mL glass	Net. Wt. 678 g		None
LAW-00-7	S00T001472	L	N/A	1/500 mL glass	Net. Wt. 686 g		None
LAW-00-8	S00T001473	L	N/A	1/500 mL glass	Net. Wt. 673 g		None

POSSIBLE SAMPLE HAZARDS/REMARKS (List all known wastes) MSDS Yes No

Copies of original chain of custody forms from field sampling are attached.

SPECIAL INSTRUCTIONS

Ship to: SRTC, Bldg. 773A
East Wing Truck Dock
Aiken, SC 29802
ATTN: Debra Fields (803)725-5849 or
Bill Wilmarth (803)725-1727/(803)725-PAGE 11281
Lynda Wingard (803)725-7097/(803)725-PAGE 11880

Hold Time _____

Relinquished By <i>R. Steele</i>	Print <i>R. Steele</i>	Date/Time <i>1/24/01</i>	Signature <i>[Signature]</i>
Relinquished By <i>J.R. Sexton</i>	Print <i>J.R. Sexton</i>	Date/Time <i>1-30-01</i>	Signature <i>[Signature]</i>
Relinquished By	Print	Date/Time	Signature

FINAL SAMPLE DISPOSITION	Disposal Method (e.g., Return to customer, per lab procedure, used in process)
Received By	Received By
Date/Time	Date/Time
Disposed By	Disposed By
Date/Time	Date/Time

Matrix*

S = Soil	DS = Drum Solids
SE = Sediment	DL = Drum Liquids
SO = Solid	T = Tissue
SL = Sludge	WI = Wipe
W = Water	L = Liquid
O = Oil	V = Vegetation
A = Air	X = Other

All samples containing hazardous materials shall be picked up by requestor and returned to parent container or site of origin.

DISTRIBUTION: White - Remain with Samples Color - Customer

CHAIN OF CUSTODY/SAMPLE ANALYSIS REQUEST

C.O.C. No. _____
 Page 1 of 1

Collector N/A	Contact/Requestor Ruth Esch	Telephone No. 373-4314	MSIN 16-12	FAX 372-1878
SAF No. N/A	Sample Origin Tank 241-AW-101	Purchase Order/Charge Code N/A	Temp. N/A	
Project Title AW-101 ICD23 Shipment	Logbook No. N/A	Ice Chest No. N/A	Bill of Lading/Air Bill No. N/A	
Shipped To (Lab) SRTC, Bldg. 773A, East Wing Truck Dock	Method of Shipment PAS-1 Caak	Bill of Lading/Air Bill No. N/A	Offsite Property No. N/A	
Protocol N/A	Data Turnaround N/A	Sample Analysis		

Sample No.	Lab ID	Date	Time	No./Type Container	Net. Wt.	MSDS	Yes	No	Special Instructions	Hold Time	Preservative
IAM-00-9	S00T001474	N/A	N/A	1/500 mL glass	684 g						None
IAM-00-10	S00T001475	N/A	N/A	1/500 mL glass	667 g						None
IAM-00-11	S00T001476	N/A	N/A	1/500 mL glass	672 g						None
IAM-00-12	S00T001477	N/A	N/A	1/500 mL glass	674 g						None
IAM-00-13	S00T001493	N/A	N/A	1/500 mL glass	664 g						None
IAM-00-14	S00T001492	N/A	N/A	1/500 mL glass	664 g						None
IAM-00-15	S00T001494	N/A	N/A	1/500 mL glass	663 g						None
IAM-00-16	S00T001495	N/A	N/A	1/500 mL glass	676 g						None

POSSIBLE SAMPLE HAZARDS/REMARKS (List all known wastes) Yes No
Copies of original chain of custody forms from field sampling are attached.

SPECIAL INSTRUCTIONS
 Ship to: SRTC, Bldg. 773A
 East Wing Truck Dock
 Aiken, SC 29802
 ARTN. Debra Fields (803)725-5849 or
 Bill Wilmarth (803)725-1727/(803)725-PAGE 11281
 Lynda Wingard (803)725-7097/(803)725-PAGE 11880

Relinquished By <i>Et Steele</i>	Print	Sign <i>Et Steele</i>	Date/Time <i>1/30/01 1045</i>	Received By <i>JR Sexton</i>	Print	Sign <i>JR Sexton</i>	Date/Time <i>01-30-01</i>	Matrix*
Relinquished By <i>JR Sexton</i>			Date/Time <i>1-30-01</i>	Received By <i>JR Sexton</i>			Date/Time <i>1/30/01</i>	S = Soil SE = Sediment SO = Solid SL = Sludge W = Water O = Oil A = Air
Relinquished By			Date/Time	Received By			Date/Time	DS = Drum Solids DL = Drum Liquids T = Tissue WI = Wipe L = Liquid V = Vegetation X = Other
FINAL SAMPLE DISPOSITION	Disposal Method (e.g., Return to customer, per lab procedure, used in process)			Disposed By				

All samples containing hazardous materials shall be picked up by requestor and returned to parent container or site of origin.
 DISTRIBUTION: White - Remain with Samples Color - Customer
 BC-6000-828 (04/99)

Appendix 2

- Tank 241-AW-101 Sample Composite, Homogeneity, Analysis, and Dilution, Test Specification, 24590-WTP-TSP-RT-01-001, Rev.0, August 10, 2001.

River Protection Project Waste Treatment Plant

RPP-WTP

**Tank 241-AW-101 Sample
Composite, Homogeneity, Analysis, and Dilution**

Test Specification
Revision 0

August 10, 2001

Ernest Lee & Ingrid Burgeson
Characterization R&T
BNI/WGI

Approval

Date

Ernest D. Lee Characterization R&T Engr.	
Roger Roosa / Characterization R&T Manager	
Todd Wright / R&T Manager	

Revision Summary

Revision Number	Task	Purpose
0	General	General revision to TSP-W375-00-00002 Rev 0 to provide clarification of work to perform during this task

Purpose

This specification provides instructions to Savannah River Technology Center (SRTC) personnel for:

- Preparing a composite sample from multiple tank 241-AW-101 waste samples,
- Verifying the homogeneity of the composite sample,
- Analyzing the composite,
- Diluting the composite sample, and
- Analyzing the diluted 241-AW-101 waste sample
- Comparing analytical results with the low-activity waste (LAW) specifications for the waste treatment plant (WTP)
- Characterize the undissolved solids
- Reporting analytical results

Background

Hanford Site personnel have obtained ~15-liters of waste from tank 241-AW-101 for shipment to the SRTC. The prefix "241" is common to all Hanford Site tanks and will not be used further. The tank AW-101 waste sample was shipped to the SRTC in 500-ml bottles contained in the PAS-1 cask, with shipments occurring in January, February and March 2001. Waste contained in tank AW-101 is candidate low-activity waste (LAW) feed to the River Protection Project Waste Treatment Plant (RPP-WTP).

SRTC personnel will characterize the AW-101 sample (as described by this test specification) and then use this waste sample for process verification testing and waste form qualification test activities. The data will not be used to address regulatory requirements. Separate test specifications will be issued for the process verification testing and waste form qualification test activities conducted with the AW-101 sample and will include the following tests:

- Crossflow ultrafiltration using a single, 0.1- μ m filter element to provide filter flux for WTP design verification,
- Multiple load and elution tests with SuperLig 644 (Cs) and SuperLig 639 (Tc) ion exchange columns (~10-ml resin per column) to determine useful operating life of these resins and variability in resin performance
- Evaporation of pretreated sample to determine saturation end-point, partitioning of compounds of potential regulatory concern, and provide information to verify process models,
- Melter feed rheology measurements,
- Lab-scale radioactive melter process test to determine partitioning of radionuclides and inorganic compounds, determine if hazardous compounds (e.g., dioxins) form in melter system, verify ability to process glass formulation, demonstrate compliance of LAW glass with land disposal restrictions and other contract specifications

In 1998, personnel at the Pacific Northwest National Laboratory (PNNL) analyzed a sample from tank AW-101, with inorganic and radiochemical analytes reported in *Inorganic and Radiochemical Analysis of AW-101 and AN-107 Tank Waste*¹. PNNL personnel conducted analyses of the tank AW-101 sample to determine analytes of potential regulatory concern and reported their results in *Organic Analysis of AW-101 and AN-107 Tank Waste*². These analytical results confirm that waste contained in tank 241-AW-101 meets the criteria of LAW Envelope A feed, as defined in specification 7 of request for proposal (RFP) solicitation number DE-RP27-00RV14136³. SRTC personnel are not to repeat analysis of the current AW-101 to determine analytes of potential regulatory concern.

¹ Inorganic and Radiochemical Analysis of AW-101 and AN-107 Tank Waste, PNWD-2462, BNFL-RPT-008, rev. 0, May 1999, Battelle, Richland Washington.

² Organic Analysis of AW-101 and AN-107 Tank Waste, PNWD-2461, BNFL-RPT-001, rev. 0, August 2000, Battelle, Richland Washington.

³ Waste Treatment and Immobilization Plant Request for Proposal solicitation DE-RP27-00RV14136, final draft issued August 31, 2000, U. S. Department of Energy, Office of River Protection, Richland, Washington.

The Tank Farm contractor plans calls for retrieving the supernate from Tank AW-101 into a second tank and then dissolving the salt cake in AW-101.

The sodium concentration of the liquid fraction of tank AW-101 waste is ~10.7M, based on the above referenced analyses. PNNL personnel have diluted a sample of tank AW-101 waste using de-ionized water to achieve final sodium, aluminum, and hydroxide concentrations of ~6.5M, 1.2M, and 3.0M, respectively⁴. PNNL personnel did not observe any adverse effects from adding de-ionized water to the AW-101 sample.

PNNL personnel separated entrained solids from a tank AW-101 waste sample and conducted washing of the entrained solids per specification 12 of RFP solicitation DE-RP27-00RV14136⁵. PNNL personnel have also conducted solubility versus temperature experiments with the AW-101 entrained solids slurry⁶. SRTC personnel will not repeat these tests with the current AW-101 waste sample.

⁴ Inorganic and Radiochemical Analysis of AW-101 and AN-107 "Diluted Feed" Materials, PNWD-2463, BNFL-RPT-003, revision 0, September 1999, Battelle, Richland Washington.

⁵ Washing of the AW-101 Entrained Solids, PNWD-2465, BNFL-RPT-006, rev. 0, August 1999, Battelle, Richland Washington.

⁶ AW-101 Entrained Solids – Solubility Versus Temperature, PNWD-2466, BNFL-RPT-004, rev. 0, August 1999, Battelle, Richland Washington.

Task Specification Title: AW-101 Sample Composite, Homogeneity, and Analysis

BNI R&T Plan Reference

Section 1.0 of the Research and Technology Plan identifies characterization requirements for low-activity waste (LAW) samples.

BNI Statement of Work / RPP-WTP Contract

Characterization of tank waste samples is identified in the BNI Statement of Work section 5.2.2, *Characterization of LAW and HLW Feeds* and the RPP-WTP Contract standard 2, item (a)(3)(i).

1.1 Justification

This task provides information for assessing tank 241-AW-101 waste for compliance with the LAW feed specification. It also directs the characterization of process testing solutions which provides an opportunity to underpin the pretreatment facility design basis. Additionally it will provide data to the WPT to validate assumptions in the flowsheet and to support process verification testing and LAW waste form qualification. Additionally, it is critical that the individual samples of AW-101 waste shipped to SRTC are homogeneous before sub-sampling to ensure the analysis of the sub-samples provides information representative of the bulk sample.

1.2 Objectives

The objectives of this task are to:

- Receive and composite sample bottles that contain AW-101 waste,
- Thoroughly mix the composite AW-101 sample,
- Verify sample homogeneity,
- Sub-sample the AW-101 composite sample,
- Analyze the liquid fraction to determine compliance with specification 7 of RFP solicitation DE-RP27-00RV14136,
- Dilute a sub-sample to 7 Molar sodium concentration and characterize the liquid and solid fraction,
- Dilute the remaining tank contents to 5 Molar sodium,
- Report liquid and solid analyses in accordance with *Standard Electronic Format Specification for Tank Waste Characterization Data Loader: Version 3.0*, (HNF-3638 revision 1).⁷
- Provide a summary analytical report in electronic and paper format within 90-days of completing all analyses.
- Sample residuals and contaminated waste shall be returned to Hanford within 1 year of shipment to SRTC

⁷ Note that there are specific data formats and required fields for the data to be successfully incorporated into the electronic database. It is highly recommended that these requirements be reviewed prior to beginning any analyses.

1.3 Test Plan

SRTC shall prepare a general or specific test plan containing detailed information needed to implement this test specification. The test plan shall include a table that lists the expected amount of sample needed for each characterization step and the expected amount of sample remaining and available for process verification testing. The test plan shall provide direction for the sub-sample volumes and preparation methods for each series of determinations.

The test plan shall provide in tabular form; the methods chosen for sample preparation, whether an actual analyte, surrogate, or tracer shall be used for Laboratory Control Standards (LCS), matrix spikes and the instrument calibration requirements. The estimated minimum detection target for each analyte in Tables 2 and 3 shall be reviewed and confirm that they can be met. If the target minimum reportable quantity for an analyte in Tables 2 and 3 are below the estimated minimum detection limit, they shall be flagged. The QA target requirements listed in Tables 1, 4, and 5 shall be reviewed and any requirements that are not expected to be met should be identified and the expected delta from the criteria be identified. The test plan shall provide the technical basis for calibration and demonstration methods for continued performance where LCS and spike recoveries are not performed for a specific analyte.

The test plan should identify analyses requested in the test specification which can not readily be performed by the laboratory, with an explanation of what would be required to implement the analysis, i.e., new procedure, new equipment, etc.

The test plan should detail how the data will be reported when there are failures with meeting the QA requirements, eg., RSD greater than 15%.

The test plan shall specify that all non-corrupted excess samples shall be retained for use for process verification testing.

SRTC may propose and use alternate analytical methods and QC requirements than those listed in this test specification provided justification is provided in the approved test plan.

A draft of the test plan shall be submitted to BNI R&T for review and comment. Comments labeled "Required" under the "significance" column require a disposition. That is, the comment should be incorporated into the document to the degree that it is technically correct if it is not negated by other document revision. Comments that are "acknowledged" or "noted" require written justification for not incorporating the comment to some degree.

The final test plan must be approved by a BNI R&T representative. Prior to performing testing for this work, the sub contractor's approved test plan must be submitted to BNI's Project Document Control.

1.4 Success Criteria

The analytes and physical properties listed in this test specification were measured. The QC target criteria in Tables 4 and 5 along with the QC requirements for the approved QA plan for the project are met. The target criteria presented are goals for demonstrating reliable method performance. However, the analytical data may be acceptable for its intended purpose even if some of the QC criteria is not met. It is understood that the laboratory will follow its internal QA system for required actions whenever QC failures occur. In the event of QC failures, the laboratory will follow internal procedures to resolve the issues. If the failures can not be resolved, the BNI R&T representative will be notified as soon as the inability to resolve the failures at the bench level is identified. The laboratory should provide a suggested course of action at that time. Additionally, all QC failures and limitations on the associated data shall be discussed in the narrative of the data report. All data not meeting the specified QC requirements shall be brought to the attention of the BNI R&T representative. The R&T representative will notify the contractor, in writing, the acceptance of data which did not meet the QC requirements.

1.5 Quality Assurance

Work shall be performed in accordance with applicable requirements NQA-1-1989 Part I, Basic and Supplementary Requirements and NQA-2a-1990 subpart 2.7, as required in Standard 7 section 3ii(B) of the WTP Contract. SRTC shall submit a QA checklist along with the Test Plan that identifies the sections of the WSRC Quality Assurance Plan that implements the applicable sections of NQA-1-1989 and NQA-2a-1990. The matrix shall identify:

- a) Where the requirements are addressed
- b) Where requirements are not applicable based on the scope of work.

The *Quality Assurance Requirements and Description* (DOE/RW-00333P), the principal quality assurance document for the Civilian Radioactive Waste Management Program does not apply to activities conducted as part of this task.

1.6 Test Conditions

SRTC personnel are to inspect and weigh the "as received samples" to determine what degree of sample loss has occurred during sample storage and transport. SRTC personnel are to report the visual appearance of the liquid and solid / crystalline phases present in each AW-101 sample bottles.

SRTC shall maintain a Chain of Custody for the AW-101 samples and sub-samples during performance of work specified by this test specification.

SRTC personnel are to maintain a material balance for the AW-101 samples throughout the process steps defined by this test specification. Items typically to be recorded include sample bottle identification number, bottle tare weight (if provided by Hanford 222-S Laboratory), the mass (or volume) of sample received in each bottle, loss of sample due to residual sample left in each bottle, mass (or volume) of composite AW-101 sample, mass and volume of chemical additions, mass (or volume) of sample removed for analysis, and any other significant activities that add or remove mass (or volume) from the AW-101 sample.

The sample bottle identification numbers should be correlated to the sampling event (date, riser, type of sampling) and the sampling bottle identification numbers. See Attachment 2 for tank sampling information.

SRTC personnel are to monitor the condition of all samples that are archived for extended periods. Actions shall be taken to prevent samples from drying out. At a minimum actions shall include; keeping the samples in sealed jars, replacing evaporated liquid before solids are exposed, and delay separating the liquid from solids until ready to proceed with testing or analysis.

1.6.1 Compositing Tank Samples and Sub-Sampling.

1. SRTC personnel are to record the temperature of the cell during sample compositing and sub-sampling activity.
2. SRTC personnel are to maintain a material balance for the AW-101 sample throughout the process steps defined by this test specification. Items typically to be recorded include sample bottle identification number, the mass (or volume) of sample received in each bottle, loss of sample due to residual sample left in each bottle, mass (or volume) of composite AW-101 sample, mass and volume of chemical additions, mass (or volume) of sample removed for analysis, mass (or volume) of sample transferred to ultrafiltration testing, and any other significant activities that add or remove mass (or volume) from the AW-101 sample.
3. SRTC personnel are to composite all AW-101 sample bottles into a clean, missing vessel that contains an agitation and sampling (e.g., recirculation line from pump) system. The contents of the sample bottles are to pass through a screen having an opening sufficiently small so that solids passing through the screen will not be larger than the opening in the vessel sampling system. Additionally, the screen size should be selected to ensure that the particles remain suspended in solution during mixing.
 - a. The vessel used to composite the AW-101 sample should have a capacity of at least 30-liters, since this same vessel will be used for diluting the composite sample.

- b. The vessel agitation system shall be operated continuously at least an hour prior to performing sub-sampling and shall continue to operate during sub-sampling.
 - c. There shall be at least 20 minutes between collection of each sample. During this time, the agitation system shall be in operation.
4. SRTC personnel are to receive and composite all AW-101 sample before proceeding with the remainder of this task. The compositing strategy identified in the following steps is the same compositing approach utilized by PNNL in the characterization and analysis of AW101 as-received and diluted tank waste. This compositing approach resulted in homogeneous subsamples of AW101 tank waste, based upon measurement of the volume percent solids for each subsample. It should be noted that this approach did not provide acceptable homogeneous subsamples with AN107 tank waste material.

All sample bottles shall be transferred into a clean vessel through a screen which has openings which are approximately 1/8 x 1/8 to 3/32 x 3/32 inches. The vessel shall be capable of maintaining continuous agitation and sub-sampling⁸.

Once the material is transferred into the vessel, remove the screen and place a cover on the mixing vessel. If any material remains on the sample screen after flushing the screen with the liquid samples, determine weight of material and archive it. Notify BNI representative with a proposed path forward for this material. If the mass of solid material retained by the screen is greater or equal to 2% of the total solids, notify the BNI R&T representative for direction.

The vessel agitation system shall be operated continuously at least an hour prior to performing sub-sampling and shall continue to operate during sub-sampling.

SRTC personnel are to extract twelve sub-samples from the AW-101 slurry into containers capable of holding approximately 250 mL of slurry. The volume of the composite samples will be approximately 225 mL and will be collected at three separate depths. The collection order for the sub-samples and the depth of collection must be recorded for each sub-sample

5. SRTC personnel are to extract twelve sub-samples from the AW-101 slurry into containers capable of holding approximately 250 mL of slurry. The volume of the composite samples will be approximately 225 mL for ten samples and approximately 100 mL for two of the samples (or whatever volume is needed to ensure adequate material for characterization and dilution studies). The samples will be collected at three separate depths. The collection order for the sub-samples and the depth of collection must be recorded for each sub-sample.

The samples shall be collected while operating the vessel agitation system and should be collected one sample from the top, one from the middle and one from the bottom until four samples have been collected from each level. SRTC personnel are to extract twelve 225 ± 10-ml sub samples from the AW-101 composite sample to evaluate sample homogeneity. Allow approximately 15 minutes between sample collection. During this time, the mixing vessel shall be in continuous operation and the pump should be continuously recirculating AW101 material.

- a. SRTC personnel collect the 1st, 4th, 7th and 10th sub-sample from near the midpoint of the slurry height within the homogenization vessel.
- b. SRTC personnel shall collect the 2nd, 5th, 8th, and 11th sub-sample from near the top of the slurry height within the homogenization vessel.

⁸ The screen size has been selected based upon the compositing strategy identified in "Regulatory Data Quality Objective: Sample Compositing Strategy", and is based upon ensuring there are no particles which are large enough to plug the sampling port. The screen size can be modified to meet the specific sampling tube dimensions of the SRTC homogenization system.

- c. SRTC personnel shall collect the 3rd, 6th, 9th, and 12th sub-sample from near the bottom of the slurry height within the homogenization vessel.
 - d. Allow the composite sub-samples to settle for a minimum of 24-hours.
 - e. SRTC personnel are to inspect the composite sub-samples for second phase layer and record volume or thickness and appearance of the layer.
 - f. SRTC personnel are to record the volume of settle solids / crystalline material and supernate present in each of the twelve AW-101 composite sub-samples. The volume measurement shall be reported to an accuracy of $\pm 1\%$.
 - g. SRTC personnel are to calculate the relative volume percentage of settled solids / crystalline material present in each of the twelve composite sub-samples, and compare the volume percent settled solids versus the order of sample collection.
 - h. **Administrative Hold Point:** If an organic layer is observed in any of the composite batches, the client needs to be consulted before work continues.
 - i. **Administrative Hold Point:** If the composite batch samples contains an average of less than or equal to 10 vol% settled solids, then the homogenization is complete. The vol% settled solids data on the composite batches are to be presented to BNI-WGI R&T Characterization representative for approval before proceeding. Once this approval has been received, proceed to step 6.
 - j. **Administrative Hold Point:** If the composite batches contain an average of greater than 10 vol% settled solids, then the following two criteria must be met before the composite batches will be considered homogeneous. If these two criteria are met, then data on the composite batches are to be presented to the client for approval before proceeding to step 6. the data package submitted for BNI-WGI approval is to include information on any outliers eliminated from the data set reconciled against the criteria. If the composite batches fail the criteria, then the composite sub-samples will be returned to the mixing vessel and re-homogenized and sub-sampled again under step 5 at a higher agitation rate and/or other modifications deemed necessary to obtain improved mixing. If the second homogenization fails, the client will be consulted.
 - i) Calculate a standard deviation for the entire vol% settled solids data set. This standard deviation must be no greater than 5 vol%.
 - ii) Calculate a best fit line for the data set (vol% settled solids versus collection order). This best fit line must not show a trend of greater than 5 vol% over the range, for example, if the linear best fit line is at 50 vol% for the first sample, then it cannot exceed 55 vol% or be below 45 vol% for the last sample (these are the values for the best fit line, not the samples).
6. After receiving confirmation that the homogenization is acceptable, return all but two sub-samples to the homogenization vessel. One sub-sample will be used for characterization of the as- received tank material and one sub-sample will be used in the dilution to 7M Na (detailed in step 1.6.2).
- SRTC Personnel are to analyze triplicate aliquots of one homogenous sub-sample of the "composite AW-101" sample to determine the properties of the slurry listed in Table 1. In addition, SRTC Personnel are to analyze triplicate aliquots of one homogenous sub-sample of the "composite AW-101" sample to determine the concentration of analytes listed in Table 2 contained in the filtrate and vacuum filtered solids. Quality Control parameters are defined in Tables 4 and 5. The filtered solids will be digested using two methods, acid digestion and KOH or Na₂O₂ fusion. If after the sample dissolution step during sample preparation, the solids have not completely dissolved, then filter and archive the solids. After reviewing the results from both dissolution methods, contact the BNI R&T representative if there is a concern that the undissolved material were not dissolved by either dissolution method used

8. SRTC Personnel are to archive one sub-sample used for verification of homogeneity until completion of study. One sub-sample will be used for dilution study described in section 1.6.2. The remaining sub-samples are to be returned to the compositing vessel.
9. After determining the sodium concentration in the "as received" material, SRTC personnel are to dilute the AW-101 composite sample remaining in the compositing vessel using de-ionized water to a target sodium concentration of ~5.0M.

CAUTION: The Al and OH concentrations of the AW-101 waste sample are approximately 1.2M and 5.3M, respectively. To avoid Al precipitation, always added the de-ionized water to the AW-101 sample.

- a. The de-ionized water is to be added to the AW-101 composite sample while the AW-101 sample is being agitated.
- b. SRTC personnel are to record the volume ± 10 -ml of de-ionized water that is added to the AW-101 sample.

SRTC personnel are to analyze triplicate aliquots of one homogenous sub-sample of the "diluted AW-101" slurry sample to measure the physical properties listed in Table 1. In addition, SRTC personnel are to analyze triplicate aliquots of one homogenous sub-sample of the "diluted AW-101" sample to determine the concentration of analytes listed in Table 2 contained in the filtrate. For the vacuum filtered solids, if solids were greater than 2 vol%, determine the concentration analytes listed in Table 3. The filtered solids will be digested using two methods, acid digestion and KOH or Na₂O₂ fusion. Quality Control parameters are defined in Tables 4 and 5.

- a. Do not wash the damp solids / crystalline material, since this will cause solids dissolution.
- b. If after the sample dissolution step, the solids have not completely dissolved, then filter and archive the solids. After reviewing the results from both dissolution methods, contact the BNI R&T representative if there is a concern that the undissolved material were not dissolved by either dissolution method used.

SRTC personnel are to compare the liquid and solid fraction analytical results for the "as-received AW-101" composite sample and the "diluted AW-101" sample to determine compliance with Specification 7, Low-Activity Waste Envelopes Definition, of *WTP Request for Proposal Solicitation No. DE-RP27-00RV14136*.

10. SRTC personnel are to report to the RPP-WTP technical representative the sodium concentrations for the as-received and diluted AW-101 sub-samples before proceeding with ultrafiltration tests with the diluted AW-101 sample.

1.6.2 AW-101 Sample Dilution Studies

1. SRTC personnel are to use one of the reserved sub-samples from the AW-101 composite sample that were set aside in task 1.61 for the sample dilution studies.
2. SRTC personnel are to vigorously agitate the sub-sample of AW-101 waste and add de-ionized water to dilute the AW-101 sample to a final sodium concentration of ~7M.
 - a. SRTC personnel are to record the volume (± 1 -ml) of the sub-sample.
 - b. SRTC personnel are to analyze triplicate aliquots of one homogenous sub-sample of the "diluted AW-101" slurry sample to measure the physical properties listed in Table 1. In addition, SRTC personnel are to analyze triplicate aliquots of one homogenous sub-sample of the "diluted AW-101" sample to determine the concentration of analytes listed in Table 2 contained in the filtrate. For the vacuum filtered solids, if solids were greater than 2 vol%, determine the concentration analytes listed in Table 3. The filtered solids will be digested using two methods, acid digestion and KOH or Na₂O₂ fusion. Quality Control parameters are defined in Tables 4 and 5.
 - c.

- d. Do not wash the damp solids / crystalline material, since this will cause solids dissolution.
3. SRTC personnel are to place the ~7M Na AW-101 sub-sample in a tightly sealed bottle and record on a daily basis for six-months (excluding weekends and holidays) the ambient temperature and the volume (± 1 -ml) of settle solids present in the sample.

After the six-month period of observation, SRTC personnel are to analyze in triplicate, homogenous sub-samples of the "7M Na AW-101" sample to determine the concentrations of analyte listed in Table 1 and 2 contained in the slurry, filtrate, and vacuum filtered solids. Quality Control parameters are defined in Table 4.

If after the sample dissolution step during sample preparation, the solids have not completely dissolved, then filter and archive the solids. After reviewing the results from both dissolution methods, contact the BNI R&T representative if there is a concern that the undissolved material were not dissolved by either dissolution method used.

4. SRTC personnel are to retain any remaining 7M Na AW-101 sample in a tightly sealed bottle that is labeled to distinguish this sample from other samples.

1.6.3 Characterization of AW-101 CUF Washed Entrained Solids

Following dilution of the material in the composite vessel, a LAW entrained solids ultrafiltration test will be performed (see TSP-24590-01-00001, Rev. 0). A sub-sample of the washed solids collected from the filtration test shall be analyze in triplicate, to determine the concentrations of analyte listed in Table 3. Quality Control parameters are defined in Table 5.

SRTC personnel are to compare and report the analytical results from the washed entrained solids to determine compliance with nineteen identified analytes for entrained solids identified in Specification 7, Low-Activity Waste Envelopes Definition, of *WTP Contract No. DE-AC27-01RV14136*.

1.7 Reporting

SRTC personnel are to report all characterization results in metric units, in accordance with section 6.6.3, *Convention of Units of Measure*, of the DOE-ORP statement of work to BNI/WGI. SRTC personnel are to issue the draft test results to RPP-WTP within 90 calendar days after completing the analyses identified in this test specification. SRTC personnel are to issue a final test report within 30 calendar days after receiving comments on the draft report from RPP-WTP. This report shall be issued as a document approved for public release and available to all Hanford contractors. This report will be provided to the client in both electronic and paper format.

SRTC personnel shall report all liquid and solid analyses in accordance with *Standard Electronic Format Specification for Tank Waste Characterization Data Loader: Version 3.0*, (HNF-3638 revision 1), to the extent data field information is available.

Table 1 Slurry Analyses for Feed Composition ^(a)		
Analyte	Minimum Reportable Quantity	Analysis Method ^(c)
Physical Property ^(a)	Expected Range	
Slurry Density ^(b)	1 to 1.6 gm/cm;	Gravimetric
Liquid Density ^(b)	1 to 1.6 gm/cm	Gravimetric
Wt% vacuum filtered solids ^(b)	0.1 to 30 wt%	Gravimetric
Wt% Oven Dried solids	1 to 80 wt%	Gravimetric
Wt% Dissolved solids	1 to 25 wt%	Gravimetric
Wt% Soluble solids	1 to 25 wt%	Calculation

Footnotes:

^(a) Triplicate measurements are to be made for each Physical Property at specified concentration and temperature.

Acceptable precision is <15% RSD. %RSD = (standard deviation/mean) * 100

^(b) Measurements are to be made using vendor calibration of glassware and laboratory balances.

^(c) Measurements are to be made at cell ambient temperature (normally ambient between 28-35 °C).

Table 2. Filtered Solids and Filtered Supernate Analyses			
Analyte	Target Minimum Reportable Quantity		Recommended Analysis Method ^(c)
	Filtrate mg/L	solids mg/Kg	
Al	7.5E+01	7.5E+01	ICP-AES
B	2.3E+00	2.3E+00	
Ba	2.3E+00	2.3E+00	
Ca	1.5E+02	1.5E+02	
Ce	2.3E+00	2.3E+00	
Cd	7.5E+00	7.5E+00	
Cr	1.5E+01	1.5E+01	
Fe	1.5E+02	1.5E+02	
K	7.5E+01	7.5E+01	
La	3.5E+01	3.5E+01	
Li	2.3E+00	2.3E+00	
Mg	3.0E+02	3.0E+02	
Na	7.5E+01	7.5E+01	
Ni	3.0E+01	3.0E+01	
P	6.0E+02	6.0E+02	
Pb	3.0E+02	3.0E+02	
S	1.7E+02	1.7E+02	
Th	2.3E+00	2.3E+00	
V	2.3E+00	2.3E+00	
W	2.3E+00	2.3E+00	
U	7.8E+02	7.8E+02	Kin. Phosphorescence
TOC	1.5E+03 (as C)	1.5E+03 (as C)	Furnace oxidation method
TIC	1.5E+02 (as C)	1.5E+02 (as C)	
Hg	1.5E+00	1.5E+00	Cold Vapor AA
Cl	3.0E+02	3.0E+02	IC ^(d)
F	1.5E+02	1.5E+02	
NO ₂	3.0E+03	3.0E+03	
NO ₃	3.0E+03	3.0E+03	
PO ₄	2.5E+03 (as P)	2.5E+03 (as P)	
SO ₄	2.3E+03 (as S)	2.3E+03 (as S)	
⁹⁹ Tc	mCi/L (except as noted) 6.0E+00	mCi/L (except as noted) 1.50E-03	ICP-MS
Rb	1.0E+00 (mg/L)	1.0E+00 (mg/L)	
¹²⁷ I	1.5E+00 (mg/L)	1.5E+00 (mg/L)	
¹²⁹ I ^(d)	1.8E-05	1.8E-05	
¹³³ Cs	7.0E-02 (mg/L)	7.0E-02 (mg/L)	
¹³⁵ Cs	1.5E+00	1.5E+00	
¹³⁷ Cs	1.5E+00	1.5E+00	
²³⁷ Np	2.7E-02	2.7E-02	
²³⁹ Pu	3.0E-02	3.0E-02	
²⁴⁰ Pu	1.0E-02	1.0E-02	
²⁴¹ Pu / ²⁴¹ Am	8.7E-03(mg/L)	8.7E-03(mg/L)	
⁹⁹ Tc	1.5E-03	1.5E-03	
²³³ U	4.2E-04	4.2E-04	
²³⁴ U	1.2E-04	1.2E-04	
²³⁵ U	4.5E-08	4.5E-08	
²³⁶ U	1.4E-06	1.4E-06	

Table 2 Cont.

Table 2 continued Centrifuged Solids and Filtered Supernate Analyses			
	Filtrate	solids	
	mg/L	mg/Kg	
²³⁸ U	7.2E-09	7.2E-09	ICP-MS continued
⁹⁹ Tc(perchnetate)	1.5E-03	1.5E-03	Separations / Liquid Beta Scintillation without sample oxidation to determine perchnetate
³ H	2.1E-02	2.1E-02	Separations / Liquid Scintillation
¹⁴ C	7.2E-04	7.2E-04	
⁷⁹ Se	9.0E-05	9.0E-05	
⁹⁰ Sr	1.5E-01	1.5E-01	
²³⁸ Pu	1.0E-02	1.0E-02	
^{239/240} Pu	3.0E-02	3.0E-02	Separations / AEA
²⁴¹ Am	3.0E-02	3.0E-02	
²⁴² Cm	1.5E-01	1.5E-01	
^{243/244} Cm	1.5E-02	1.5E-02	
¹⁵⁴ Eu	2.0E-03	2.0E-03	Extended Counting Time GEA
¹⁵⁵ Eu	9.0E-02	9.0E-02	
⁶⁰ Co	2.1E-03	2.1E-03	
¹²⁶ Sn	6.0E-03	6.0E-03	
¹³⁷ Cs	9.0E+00	9.0E+00	
²³¹ Pa	7.89E-05	7.89E-05	
Total Alpha	2.3E-01	2.3E-01	Alpha counting
Sum of Alpha (TRU)	N/A	N/A	Summation ^(b) of: Pu-238, Pu-239+Pu-240 (or Pu-239, Pu-240 ICP/MS) and Am-241
Total and Free OH	7.5E+04 mg/L	7.5E+04 mg/L	Titration
Ammonia ^(f)	NA	1.4E+02 mg/L	ISE or IC
Organic Analytes	mg/L	mg/L	
Oxalate	1.5E+03	1.5E+03	Ion Chromatograph
Citrate	1.5E+03	1.5E+03	
Formate	1.5E+03	1.5E+03	
Gluconate	1.5E+03	1.5E+03	
Gylcolate	1.5E+03	1.5E+03	
EDTA ^(a)	1.5E+03	1.5E+03	Derivatization/ GC-MS
HEDTA ^(a)	1.5E+03	1.5E+03	
D2EHPA ^{(a),(e)}	1.5E+03	1.5E+03	
NTA ^{(a),(e)}	1.5E+03	1.5E+03	
IDA ^{(a),(e)}	1.5E+03	1.5E+03	
Succinic Acid ^{(a),(e)}	1.5E+03	1.5E+03	
ED3A ^{(a),(e)}	1.5E+03	1.5E+03	
Density	Expected Range 0.95 –1.5 (gm/mL)	Expected Range 0.95 –1.5 (gm/mL)	Gravimetric
Dissolved solids	1 to 50 (gm solids/gm supernate)	1 to 50 (gm solids/gm supernate)	Gravimetric

Footnote:

- ^(a) MRQs are target values, measurement of chelators and organic phosphates are best effort only, since there is insufficient method data available to set QC parameters.
- ^(b) Guidance for reporting summation of isotopics and reporting isotopic values derived by different methods will be provided later after an agreement is obtained with DOE.
- ^(c) If analytical method changes result in full attainment of desired QC and MRQs, then method substitution shall be documented in Test Plan or in final test report. Method changes that don't satisfy desired QC/MRQ targets shall be communicated to BNI R&T representative for approval before proceeding.
- ^(d) Perform these analyses on non-digested sample material (water leach, etc) if analyte is not compatible with standard digestion procedures.
- ^(e) These organics are requested on an opportunistic basis. If they can be obtained with the requested organics, then run appropriate standards and report.
- ^(f) Ammonia analysis is not required on the solids.

Table 3. HLW Entrained Solids Analyses		
Analyte	Target Minimum Reportable Quantity	Recommended Analysis Method^(c)
	mg/Kg^(a)	
Al	3.3E+02	ICP-AES (AA may be used for Na)
Cr	1.2E+02	
Fe	1.4E+02	
Mn	3.0E+02	
Na	1.5E+02	
P	6.0E+02	
S	1.7E+02	
Si	3.0E+03	
TOC	6.0E+01 (as C)	
TIC	3.0E+01 (as C)	
U	7.8E+02	Kin. Phosphorescence
	mCi/Kg	
⁹⁹ Tc	6.0E+00	ICP-MS
²³³ U	6.0E+01	
²³⁵ U	6.0E+00	
²³⁴ U	3.7E-03	
²³⁶ U	3.8E-04	
²³⁸ U	2.0E-06	
⁹⁰ Sr	7.0E+01	Separations / Beta Gas Flow Proportional Counter
^{239/240} Pu	6.0E+00	Separations AEA
²⁴¹ Am	1.8E-02	
⁶⁰ Co	1.2E-02	Extended Counting Time GEA
¹⁵⁴ Eu	6.0E-02	
¹⁵⁵ Eu	9.0E-02	
²⁴¹ Am	6.0E+00	
¹³⁷ Cs	9.00E+00	
Total Alpha	1.0E-03	Alpha Count
Sum of Alpha (TRU)	N/A	Summation ^(b) of: Pu-238, Pu-239, Pu-240, and Am-241
Physical Property	Expected Range	
Wt% Oven Dried Solids	0.1 to 100 wt%	Gravimetric
Density	0.9 to 2.0 gm/ml	Gravimetric
Wt% Undissolved Solids	10 to 50 wt%	Calculation
Wt% Soluble Solids	1 to 50 wt%	Calculation

Footnote:

^(a) MRQs are based on dried solids weighs.

^(b) Guidance for reporting summation of isotopics that are derived by different methods will be provided later after an agreement is obtained with DOE

^(c) If analytical method changes result in full attainment of desired QC and MRQs, then method substitution shall be documented in Test Plan or in final test report. Method changes that don't satisfy desired QC/MRQ targets shall be communicated to BNI R&T representative for approval before proceeding.

Table 4. Quality Control Parameters for Liquid Analysis

Liquid Fraction	Recommended Analytical Technique ^(f)	QC Flagging Criteria		
		LCS %Recovery ^(a)	Spike %Recovery ^(b)	Triplicate RSD ^(c)
Al, B, Ba, Ca, Cd, Ce, Cr, Fe, K, La, Li, Mg, Mn, Ni, P, Pb, S, Si, Th, V, W	ICP/AES	80 - 120%	75 - 125%	<15%
Na	ICP/AES	90 - 110%	90 - 110%	<3.5%
U	Kin. Phosphorescence	80 - 120%	75 - 125%	<15%
Rb, ¹²⁷ I, ¹²⁹ I, ¹³³ Cs, ¹³⁵ Cs, ¹³⁷ Cs, ²³³ U, ²³⁴ U, ²³⁵ U, ²³⁶ U, ²³⁸ U, ²³⁷ Np, ²³⁹ Pu, ²⁴⁰ Pu, ²⁴¹ Pu / ²⁴¹ Am, ⁹⁹ Tc	ICP/MS	80 - 120%	70 - 130%	<15%
Cl, F, NO ₂ ⁻ , NO ₃ ⁻ , PO ₄ ⁻³ , SO ₄ ⁻²	IC	80 - 120%	75 - 125%	<15%
Hg	CVAA	80 - 120%	75 - 125%	<15%
NH ₃ /NH ₄ ⁺	ISE, standard additions	80 - 120%	75 - 125%	<15%
OH ⁻ (total and free)	Potentiometric titration /precipitation	80 - 120%	N/A	<15%
TIC	Furnace oxidation	80 - 120%	75 - 125%	<15%
TOC	Furnace oxidation	80 - 120%	75 - 125%	<15%
³ H	Separation/liq. Scintillation	80 - 120%	N/A	<15%
¹⁴ C	Separation/liq. Scintillation	80 - 120%	75 - 125%	<15%
⁶⁰ Co ^(e) , ¹²⁶ Sn, ²³¹ Pa	Extended GEA	NP	N/A ^(f)	<15%
⁷⁹ Se	Liq. scintillation	NP	N/A ^(e)	<15%
⁹⁰ Sr	Isotopic specific separation/beta count	75 - 125%	N/A ^(e)	<15%
⁹⁹ Tc (pertechnetate)	Separation/beta count	80 - 120%	70 - 130%	<15%
¹³⁷ Cs	GEA	NP	N/A ^(f)	<15%
¹⁵⁴ Eu ^(e)	GEA	NP	N/A ^(f)	<15%
¹⁵⁵ Eu ^(e)	GEA	NP	N/A ^(f)	<15%
²³⁸ Pu, ^{239/240} Pu, ²⁴¹ Am, ²⁴² Cm, ^{243/244} Cm	Separation/AEA	NP	N/A	<15%
Total Alpha	Proportional counter	70 - 130%	70 - 130%	<15%
Sum of Alpha ^(g) [TRU]	Calculation	N/A	N/A	N/A
Density	Gravimetric	N/A	N/A	<20%
Wt% dissolved solids	Gravimetric	N/A	N/A	<20%
EDTA ^(h)	Derivatization/GC-MS	80 - 120%	75 - 125%	<15%
HEDTA ^(h)	Derivatization/GC-MS	80 - 120%	75 - 125%	<15%
Oxalate	IC	80 - 120%	75 - 125%	<15%
Citrate	IC	80 - 120%	75 - 125%	<15%
Formate	IC	80 - 120%	75 - 125%	<15%
Gluconate	IC	80 - 120%	75 - 125%	<15%
Glycolate	IC	80 - 120%	75 - 125%	<15%
D2EPHA ^(h)	Derivatization/GC-MS	80 - 120%	75 - 125%	<15%
NTA ^(h)	Derivatization/GC-MS	80 - 120%	75 - 125%	<15%
IDA ^(h)	Derivatization/GC-MS	80 - 120%	75 - 125%	<15%
Succinic Acid ^(h)	Derivatization/GC-MS	80 - 120%	75 - 125%	<15%
ED3A ^(h)	Derivatization/GC-MS	80 - 120%	75 - 125%	<15%

Acronyms:

- AEA - Alpha Energy Analysis
- CVAA - Cold Vapor Atomic Absorption
- GEA - Gamma Energy Analysis
- IC - Ion Chromatography
- ICP/AES - Inductively Coupled Plasma Atomic Emission Spectroscopy
- ICP/MS - Inductively Coupled Plasma Mass Spectroscopy
- LSC - Laboratory Control Standard
- N/A - Not applicable
- NP - Not performed
- RSD - Relative Standard Deviation
- Wt% - Weight percent

Footnotes:

^(a) LCS = Laboratory Control Standard. This standard is carried through the entire method. The accuracy of a method is usually expressed as the percent recovery of the LCS. The LCS is a matrix with known concentration of analytes processed with each preparation and analyses batch. It is expressed as percent recovery; i.e., the amount measured, divided by the known concentration, times 100.

^(b) For some methods, the sample accuracy is expressed as the percent recovery of a matrix spike sample. It is expressed as percent recovery; i.e., the amount measured less the amount in the sample, divided by the spike added, times 100. One matrix spike is performed per analytical batch. Samples are batched with similar matrices. For other analytes, the accuracy is determined based on use of serial dilutions.

^(c) RSD = Relative Standard Deviation between the samples. Sample precision is estimated by analyzing replicates taken separately through preparation and analysis. Acceptable sample precision is usually <15% RSD if the sample result is at least 10 times the instrument detection limit. $RSD = (\text{standard deviation} / \text{mean}) \times 100$

^(d) Matrix spike analyses are not required for this method because a tracer is used to correct for analyte loss during sample preparation and analysis. The result generated using the tracer accounts for any inaccuracy of the method on the matrix. The reported results reflect this correction.

^(e) An extended counting time in the presence of high ¹³⁷Cs activity may be required to achieve the minimum reportable quantity for ⁶⁰Co and ¹⁵²Eu, ¹⁵⁴Eu, ¹⁵⁵Eu.

^(f) The measurement is a direct reading of the energy and the sample matrix does not affect the analysis; therefore, a matrix spike is not required.

^(g) The sum of ²³⁸Pu, ²³⁹Pu, ²⁴⁰Pu, and ²⁴¹Am activities will be used as a measurement of alpha-emitting TRU. The selected isotopes account for greater than 95% of the alpha-emitting TRU activity based on previous analysis of Phase I candidate tank waste (Esch 1997a, 1997b, 1997c). Additional isotopes that are defined as alpha-emitting TRU (e.g., ²³⁷Np, ²⁴²Pu, ²⁴²Cm, ²⁴³Am, and ²⁴³⁺²⁴⁴Cm) are not used to calculate total TRU activity because the MDAs for these isotopes are large in comparison with the envelope limits and it is expected that their concentrations are well below the MDA. Note that ²⁴¹Pu is a beta-emitting TRU whose analysis, along with ²⁴²Cm, is required specifically for class C waste determination. If any of the isotopes are below the MRQ, then the method of summation will be agreed by BNI prior to reporting values.

^(h) Measurement of chelators and organic phosphates are best effort only, since there is insufficient method data available to set QC parameters, QC acceptance criteria are target values.

⁽ⁱ⁾ If analytical method changes result in full attainment of desired QC and MRQs, then method substitution shall be documented in Test Plan or in final test report. Method changes that don't satisfy desired QC/MRQ targets shall be communicated to BNI R&T representative for approval before proceeding.

Table 5. Quality Control Parameters for Solids Analysis

Solids Fraction	Recommended Analytical Technique ^(d)	QC Flagging Criteria		
		LCS % Recovery ^(a)	Spike % Recovery ^(b)	Triplicate RSD ^(c)
Al, B, Ba, Ca, Ce, Cd, Cr, Fe, K, La, Li, Mg, Mn, Ni, P, Pb, S, Si, Th, V, W	ICP/AES	80 - 120%	75 - 125%	<15%
Na	ICP/AES	90 - 110%	90 - 110%	<3.5%
Rb, ⁹⁹ Tc, ¹²⁷ I, ¹³³ Cs, ²³³ U, ²³⁵ U, ²³⁷ Np, ¹²⁹ I, ¹³⁵ Cs, ¹³⁷ Cs, ²³⁹ Pu, ²³⁴ U, ²³⁶ U, ²³⁸ U, ²⁴⁰ Pu, AMU-241.	ICP/MS	80 - 120%	70 - 130%	<15%
Cl, F, NO ₂ , NO ₃ , PO ₄ ³⁻ , SO ₄ ²⁻	IC	80 - 120%	75 - 125%	<15%
Hg	CVAA	80 - 120%	75 - 125%	<15%
TIC/CO ₃ ⁻	Furnace oxidation	80 - 120%	75 - 125%	<15%
TOC	Furnace oxidation	80 - 120%	75 - 125%	<15%
³ H	Separation/liq. Scintillation	80 - 120%	N/A ^(d)	<15%
¹⁴ C	Separation/liq. Scintillation	80 - 120%	75 - 125%	<15%
⁶⁰ Co ⁽ⁱ⁾	Extended count GEA	NP	N/A ⁽ⁱ⁾	<15%
⁹⁰ Sr ^(d)	Isotopic specific separation/beta count	75 - 125%	N/A ^(d)	<15%
⁹⁹ Tc	ICP/MS	80 - 120%	70 - 130%	<15%
¹²⁶ Sn ^(e)	Extended Count GEA	NP	N/A	<15%
¹³⁷ Cs	GEA	NP	N/A	<15%
¹⁵² Eu ^(e)	Extended Count GEA	NP	N/A ⁽ⁱ⁾	<15%
¹⁵⁴ Eu ^(e)	Extended Count GEA	NP	N/A ⁽ⁱ⁾	<15%
¹⁵⁵ Eu ^(e)	Extended Count GEA	NP	N/A ⁽ⁱ⁾	<15%
²⁴¹ Am	Extended Count GEA	NP	N/A ⁽ⁱ⁾	<15%
Total Pu	Sum of Isotopes	N/A	N/A	N/A
²³⁸ Pu, ^{239/240} Pu, ²⁴² Pu	Separation/AEA	NP	N/A ^(d)	<15%
²⁴¹ Pu/Am, ²⁴² Pu	ICP/MS	80 - 120%	70 - 130%	<15%
²⁴¹ Am	Separation/AEA	NP	N/A ^(d)	<15%
²⁴² Cm	Separation/AEA	NP	N/A ^(d)	<15%
²⁴³ + ²⁴⁴ Cm	Separation/AEA	NP	N/A ^(d)	<15%
Total Alpha	Proportional counter	70 - 130%	70 - 130%	<15%
EDTA ^(h)	Derivatization/GC-MS	80 - 120%	75 - 125%	<15%
HEDTA ^(h)	Derivatization/GC-MS	80 - 120%	75 - 125%	<15%
Oxalate	IC	80 - 120%	75 - 125%	<15%
Citrate	IC	80 - 120%	75 - 125%	<15%
Formate	IC	80 - 120%	75 - 125%	<15%
Gluconate	IC	80 - 120%	75 - 125%	<15%
Glycolate	IC	80 - 120%	75 - 125%	<15%
D2EPHA ^(h)	Derivatization/GC-MS	80 - 120%	75 - 125%	<15%
NTA ^(h)	Derivatization/GC-MS	80 - 120%	75 - 125%	<15%
IDA ^(h)	Derivatization/GC-MS	80 - 120%	75 - 125%	<15%
Succinic Acid ^(h)	Derivatization/GC-MS	80 - 120%	75 - 125%	<15%
ED3A ^(h)	Derivatization/GC-MS	80 - 120%	75 - 125%	<15%
Bulk density	Gravimetric	N/A	N/A	<20%
Wt% solids	Gravimetric	N/A	N/A	<20%

Acronyms:

- AEA – Alpha Energy Analysis
- CVAA – Cold Vapor Atomic Absorption
- GEA – Gamma Energy Analysis
- IC – Ion Chromatography
- ICP/AES – Inductively Coupled Plasma Atomic Emission Spectroscopy
- ICP/MS – Inductively Coupled Plasma Mass Spectroscopy
- LSC – Laboratory Control Standard
- N/A – Not applicable
- NP – Not performed
- RSD – Relative Standard Deviation
- Wt% – Weight percent

Footnotes:

^(a) LCS = Laboratory Control Standard. This standard is carried through the entire method. The accuracy of a method is usually expressed as the percent recovery of the LCS. The LCS is a matrix with known concentration of analytes processed with each preparation and analyses batch. It is expressed as percent recovery; i.e., the amount measured, divided by the known concentration, times 100.

^(b) For some methods, the sample accuracy is expressed as the percent recovery of a matrix spike sample. It is expressed as percent recovery; i.e., the amount measured less the amount in the sample, divided by the spike added, times 100. One matrix spike is performed per analytical batch. Samples are batched with similar matrices. For other analytes, the accuracy is determined based on use of serial dilutions.

^(c) RSD = Relative Standard Deviation between the samples. Sample precision is estimated by analyzing replicates taken separately through preparation and analysis. Acceptable sample precision is usually <15% RSD if the sample result is at least 10 times the instrument detection limit. $RSD = (\text{standard deviation} / \text{mean}) \times 100$

^(d) Matrix spike analyses are not required for this method because a tracer is used to correct for analyte loss during sample preparation and analysis. The result generated using the tracer accounts for an inaccuracy of the method on the matrix. The reported results reflect this correction.

^(e) An extended counting time in the presence of relatively high gamma-activity may be required to achieve the minimum reportable quantity for ⁶⁰Co and ¹⁵²Eu, ¹⁵⁴Eu, ¹⁵⁵Eu.

^(f) The measurement is a direct reading of the energy and the sample matrix does not affect the analysis; therefore, a matrix spike is not required.

^(g) Combined analysis of ¹²⁶Sn, ¹²⁶Sb and ^{126m}Sb

^(h) Measurement of chelators and organic phosphates are best effort only, since there is insufficient method data available to set QC parameters, QC acceptance criteria are target values.

⁽ⁱ⁾ If analytical method changes result in full attainment of desired QC and MRQs, then method substitution shall be documented in Test Plan or in final test report. Method changes that don't satisfy desired QC/MRQ targets shall be communicated to BNI R&T representative for approval before proceeding.

ATTACHMENT 1, CONTRACT SPECIFICATIONS
(Excerpt from Contract No. DE-AC27-O1RV14136)

Specification 7: Low-Activity Waste Envelopes Definition

7.1 Scope: This Specification establishes three LAW feed envelopes, Waste Envelopes A, B, and C; and defines how a unit of LAW is determined for each LAW envelope. Each waste envelope provides the compositional limits for chemical and radioactive constituents in the waste feed to be provided to the WTP. The WTP shall be designed to treat the waste envelopes.

7.2 Requirements:

7.2.1 References:

- 7.2.1.1 HNF-SD-WM-SAR-067, Rev. 1-I. March 2000. *Tank Waste Remediation System Final Safety Analysis Report*. CH2M HILL Hanford Group, Inc., Richland, Washington.
- 7.2.1.2 HNF-SD-WM-TSR-006, Rev. 1-HE. March 2000. *Tank Waste Remediation System Technical Safety Requirements*, CH2M HILL Hanford Group, Inc., Richland, Washington.
- 7.2.1.3 OSD-T-151-00007, Rev. H-22. June 14, 2000. *Operating Specification for 241-AN, AP, AW, AY, AZ, and SY Tank Farms*. CH2M HILL Hanford Group, Inc., Richland, Washington.
- 7.2.1.4 DOE/RL-88-21, Rev. 10. December 21, 1999. *Double Shell Tank Unit Permits Application*. U.S. Department of Energy, Richland Operations Office, Richland, Washington.

7.2.2 Envelope Requirements:

7.2.2.1 Composition: This specification lists the concentration limits for the LAW Envelopes A, B, and C feed to be transferred by DOE to the Contractor for LAW services in Tables TS-7.1, *Low-Activity Waste Chemical Composition, Soluble Fraction Only*, and TS-7.2, *Low-Activity Waste Radionuclide Content, Soluble Fraction Only*. The concentration limits apply to the soluble fraction only. The Na concentration limits for the LAW feeds are identified below.

.7.1.1.1 Waste Feed	Na (mole per liter)
Envelope A, B, C	4 – 10
AZ-101 and AZ-102 Supernatant	2 – 5
HLW Slurry and other HLW Liquids (Defined in Specification 8, <i>High-Level Waste Envelope Definition</i>)	0.1 – 10

The LAW feeds may contain up to two weight percent solids. Solids are defined as the product of centrifuging the LAW feed, separating and drying the solids, and removing the dissolved solids contribution. The insoluble fraction characterization will include measurements of Al, Cr, Fe, Mn, Na, P, S, Si, U, TIC, TOC, ⁶⁰Co, ⁹⁰Sr, ⁹⁹Tc, ¹³⁷Cs, ¹⁵⁴Eu, ^{239/240}Pu, ²⁴¹Am, and total alpha concentrations. Trace quantities of unspecified radionuclides, chemicals, and other impurities may be present in the waste feed.

All LAW feed (soluble and insoluble components) will meet the Tank Farm Operations specifications given in OSD-T-151-00007 (except for free hydroxide), the *Tank Waste Remediation System Final Safety Analysis Report*, and *Technical Safety Requirements*, as applicable.

The radiochemical inventory of the LAW feed at the time of delivery shall be compared to the specification limits to assess compliance. The specifications for ^{60}Co , and ^{154}Eu shall apply at the time of delivery for ILAW immobilization.

The LAW feed provided shall not contain a visible separate organic phase.

The LAW feed provided will generate gases, including hydrogen and ammonia, at a nearly constant rate and a nearly uniform composition.

The maximum ^{137}Cs concentration equivalent in the transferred Envelope A, Envelope B, and Envelope C wastes feeds shall not exceed 1.2 Ci/l. The maximum ^{137}Cs concentration equivalent in the liquid fraction of Tank AZ-101 and AZ-102 feeds shall not exceed 3.0 Ci/l.

Dangerous waste codes are identified in the Double-Shell Tank System Unit Permit Application (DOE/RL-88-21, December 21, 1999). Multi-source leachate (F039) is included as a waste derived from non-specific source wastes F001 through F005.

7.2.3 Units of Low-Activity Waste: Units of LAW shall be defined as follows:

- (a) Envelope A: The quantity of Waste Envelope A containing one metric ton of waste sodium shall equal one unit.
- (b) Envelope B: The quantity of Waste Envelope B containing one metric ton of waste sodium shall be the lesser of the following number of units:
- (1) 2.6 units; or
 - (2) $\frac{X}{Y}$ units

where X is equal to 18-weight percent sodium oxide loading in the ILAW glass and Y is equal to the achievable waste sodium oxide loading, for the particular waste feed. The waste loading limitations shall be based solely upon effects of chlorine, chromium, phosphate, and sulfate.

- (c) Envelope C: The quantity of Waste Envelope C containing one metric ton of waste sodium shall be the lesser of the following number of units:
- (1) 1.15 units; or
 - (2) $\frac{X}{Y}$ units

where X and Y are defined above. The waste loading limitations shall be based solely upon sodium additions required for cesium, technetium, strontium and TRU removal from Envelope C for the particular waste feed.

Table TS-7.1 Low-Activity Waste Chemical Composition, Soluble Fraction Only

Chemical Analyte	Maximum Ratio, analyte (mole) to sodium (mole)		
	Envelope A	Envelope B	Envelope C
Al	2.5E-01	2.5E-01	2.5E-01
Ba	1.0E-04	1.0E-04	1.0E-04
Ca	4.0E-02	4.0E-02	4.0E-02
Cd	4.0E-03	4.0E-03	4.0E-03
Cl	3.7E-02	8.9E-02	3.7E-02
Cr	6.9E-03	2.0E-02	6.9E-03
F	9.1E-02	2.0E-01	9.1E-02
Fe	1.0E-02	1.0E-02	1.0E-02
Hg	1.4E-05	1.4E-05	1.4E-05
K	1.8E-01	1.8E-01	1.8E-01
La	8.3E-05	8.3E-05	8.3E-05
Ni	3.0E-03	3.0E-03	3.0E-03
NO ₂	3.8E-01	3.8E-01	3.8E-01
NO ₃	8.0E-01	8.0E-01	8.0E-01
Pb	6.8E-04	6.8E-04	6.8E-04
PO ₄	3.8E-02	1.3E-01	3.8E-02
SO ₄	1.0E-02	7.0E-02	2.0E-02
TIC ¹	3.0E-01	3.0E-01	3.0E-01
TOC ²	5.0E-01	5.0E-01	5.0E-01
U	1.2E-03	1.2E-03	1.2E-03

Notes:

- 1 Mole of inorganic carbon atoms/mole sodium
2 Mole of organic carbon atoms/mole sodium

Table TS-7.2 Low-Activity Waste Radionuclide Content, Soluble Fraction Only

Radionuclide	Maximum Ratio, radionuclide (Bq) to sodium (mole)		
	Envelope A	Envelope B	Envelope C
TRU ²	4.8E+05	4.8E+05	3.0E+06
¹³⁷ Cs	4.3E+09	2.0E+10	4.3E+09
⁹⁰ Sr	4.4E+07	4.4E+07	8.0E+08
⁹⁹ Tc	7.1E+06	7.1E+06	7.1E+06
⁶⁰ Co	6.1E+04	6.1E+04	3.7E+05
¹⁵⁴ Eu	1.2E+06	1.2E+06	4.3E+06

Notes:

¹ The activity limit shall apply to the feed certification date.

² TRU is defined as: Alpha-emitting radionuclides with an atomic number greater than 92 with half-life greater than 10 years.

Some radionuclides, such as ⁹⁰Sr and ¹³⁷Cs, have daughters with relatively short half-lives. These daughters have not been listed in this table. However, they are present in concentrations associated with the normal decay chains of the radionuclides.

Attachment 2

GRAB SAMPLING INFORMATION

Tank 241-AW-101 was grab sampled between May 2000 and September 2000 in accordance with Interface Control Document (ICD) 23⁽¹⁾. The purpose of the sampling event was to provide 15L of sample for Process Verification and Waste Form Qualification Testing. The grab samples were collected from a single riser (022) at 5 elevations. As of May 2000, AW101 contains 4,258 kL (1,125 kgal) of waste with a liquid phase estimated by the best basis inventory assessment at 2,831 kL (748 kgal). The following table provides the sample name and elevation for this sampling event.

Table A2.1 Tank AW-101 Supernate Sampling Information

Sample Number	Sample Type	Sample Location	Elevation from Tank Bottom (inches)	Cable Length from Top of Sample Riser (inches)
1AW-00-1	500 mL grab	Riser 022	370	297
1AW-00-2	500 mL grab	Riser 022	370	297
1AW-00-3	500 mL grab	Riser 022	370	297
1AW-00-4	500 mL grab	Riser 022	370	297
1AW-00-5	500 mL grab	Riser 022	370	297
1AW-00-6	500 mL grab	Riser 022	370	297
1AW-00-7	500 mL grab	Riser 022	310	357
1AW-00-8	500 mL grab	Riser 022	310	357
1AW-00-9	500 mL grab	Riser 022	310	357
1AW-00-10	500 mL grab	Riser 022	310	357
1AW-00-11	500 mL grab	Riser 022	310	357
1AW-00-12	500 mL grab	Riser 022	310	357
1AW-00-13	500 mL grab	Riser 022	250	417
1AW-00-14	500 mL grab	Riser 022	250	417
1AW-00-15	500 mL grab	Riser 022	250	417
1AW-00-16	500 mL grab	Riser 022	250	417
1AW-00-17	500 mL grab	Riser 022	250	417
1AW-00-18	500 mL grab	Riser 022	190	477
1AW-00-19	500 mL grab	Riser 022	190	477
1AW-00-20	500 mL grab	Riser 022	190	477
1AW-00-21	500 mL grab	Riser 022	190	477
1AW-00-22	500 mL grab	Riser 022	190	477
1AW-00-23	500 mL grab	Riser 022	130	537
1AW-00-24	500 mL grab	Riser 022	130	537
1AW-00-25	500 mL grab	Riser 022	130	537
1AW-00-26	500 mL grab	Riser 022	130	537
1AW-00-27	500 mL grab	Riser 022	130	537
1AW-00-28	500 mL grab	Riser 022	130	537

Note that prior to shipment to SRTC, the 222-S laboratory was going to collect approximately 1 L of archived AW101 material to ensure 15L of material would be shipped to SRTC. Currently, there is no specific information available on the origin of the archived material.

⁽¹⁾ Letter of Instruction from Data Development and Interpretation, Interoffice Memo, 74B20-00-033, May 23, 2000, CH2Mhill, Richland, Washington.

Appendix 3

- **Raw Analytical Results**
(The large volume of raw analytical data is available upon request.)