

**COMPOSITING, HOMOGENIZATION, AND
CHARACTERIZATION OF SAMPLES FROM
HANFORD TANK 241-AN-107**

OCTOBER 2003

SAVANNAH RIVER TECHNOLOGY CENTER

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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.

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Key Words:

Supernate Composition
Entrained Solids
Sample Analysis

Retention:

Permanent

Key WTP R&T References:

Test Specification
24590-WTP-TSP-RT-02-010
Test Plan - WSRC-TR-2002-00185, Rev. 1
Test Exceptions
24590-WTP-TEF-RT-02-051
24590-WTP-TEF-RT-02-055
24590-WTP-TEF-RT-03-049
R&T Focus Area - Characterization
Test Scoping Statement – S-4

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LIST OF ACRONYMS

Laboratory Control Standard (LCS)
Low-Activity Waste (LAW)
Matrix Spike (MS)
Quality Control (QC)
Quality Control Acceptance Criteria (QCAC)
Relative Standard Deviation (RSD)
River Protection Project-Waste Treatment Plant (RPP-WTP)
Savannah River Technology Center (SRTC)
Total Inorganic Carbon (TIC)
Total Organic Carbon (TOC)

1.0 SUMMARY OF TESTING

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 ~5 L sample of waste from Tank 241-AN-107 was received at the Savannah River Technology Center (SRTC). The waste sample was characterized and mixed with recycle streams to provide feed for pretreatment testing. The characterization data provides a basis for rational development of pretreatment processes, verification of tank composition, and development of physical design parameters for the pretreatment plant.

1.1 OBJECTIVES

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-AN-107 samples.
- Thoroughly mix the composite and confirm that homogenous sub-samples can be drawn for analytical measurements.
- Measure physical properties of the composite sample using one of the homogeneous samples.
- Determine chemical and radiological composition of the liquid and solid fractions using homogenous sub-samples.
- Compare liquid fraction analytical results to the low-activity waste (LAW) feed Specifications 7 requirements.
- Report analytical results.

1.2 CONDUCT OF TESTING

A total of eight 500-mL jars and fourteen 125-mL jars of 241-AN-107 tank waste were received at SRTC and composited in an 8-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 centrifuging were analyzed and the supernate found to have a sodium concentration of ~9 M. Physical properties were measured on the as-received slurry. The slurry was then separated into solid and supernatant phases by centrifugation and filtration and physical properties were measured on each phase. A portion of the supernatant fraction was removed from the cells for anions and/or cations, inorganic and organic carbon, free hydroxide and total hydroxide determinations. Another portion of the supernatant fraction was heated with nitric acid and hydrogen peroxide in sealed Teflon containers as the sample preparation step prior to elemental and radionuclide determinations.

The wet solids that remained after centrifugation and decanting off the supernatant phase were treated with three main sample preparation techniques prior to removing the solutions for instrumental analysis. Hot aqua regia (3:1, v/v, concentrated hydrochloric acid and concentrated nitric acid) in a sealed Teflon pressure vessel was used to dissolve the solids for elemental and radionuclide determinations. Wet solids were also dissolved with a mixture of nitric acid, hydrochloric acid, hydrofluoric acid, and boric acid (Microwave Digestion) for elemental and radionuclide determinations. Anion determinations were obtained on solutions after leaching the solids with warm de-ionized water. Additional specific sample preparation techniques were used in the analysis of ^{14}C , ^{129}I , and ^{79}Se .

Microwave digestion is the preferred sample preparation method for solid analyses. Analyte concentrations were higher overall for the microwave digestion. Some matrices require the presence of hydrofluoric acid to achieve complete digestions.

1.3 RESULTS AND PERFORMANCE AGAINST OBJECTIVES

All of the main objectives of the characterization for the 241-AN-107 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, a quality control (QC) flag in the data tables identifies which criteria were not met.

The analytical results from the liquid fraction of the 241-AN-107 sample were compared to the Low-Activity Waste feed limits in Specification 7. The 241-AN-107 sample met the contract feed limit specifications for all analytes and radionuclides except transuranic elements.

The Test Specification and Test Plan stated that a comparison will be made of the solids material to Specification 8, which is the Contract Specification related to compositional requirements for HLW materials (Solids). Tank AN-107 solids are primarily listed as saltcake, a material that will likely dissolve during pretreatment. The solids content on a wet basis as measured in this task was relatively low (approximately 7 wt %). Further, the weight of solids on a dry weight basis was less than 1%. (This extremely high water content of AN-107 solids had also been noted previously at Battelle – BNFL-RPT-007, Rev. 0.) At 1% solids, there are not enough solids to allow sufficient material for an accurate measurement of grams solids/gram waste oxide measurement. Therefore, the comparison with Specification 8 was not deemed prudent and was omitted from this work. A Test Exception (24590-WTP-TEF-RT-03-049) was written to omit this scope.

1.4 QUALITY REQUIREMENTS

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.

Overall the data quality for the analysis of the 241-AN-107 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 represents the composition of the 241-AN-107 sample received at SRTC and makes no assertions as to the validity of the data to the tank contents as a whole. 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.

1.5 ISSUES

None

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2.0 SAMPLE RECEIVING AND COMPOSITING

A total of eight 500-mL jars and fourteen 125-mL jars of 241-AN-107 tank waste were received at SRTC on May 13, 2002. Sixteen of the sample jars received came from the ten grab samples that were obtained in February 2001 and six of the sample jars received came from two bottles that contained composited samples from a 1998 sampling event of the 241-AN-107 waste tank (Appendix A).

Visual inspection at SRTC showed each jar contained a single dark brown liquid phase with a $\sim 1/3$ " to 1" layer of mostly crystalline white solids on the bottom. No separate organic layers were observed in any of the jars. The twenty-two jars were composited into an 8-L polyethylene carboy. Each jar was weighed before and after addition through a $1/8$ " by $1/8$ " mesh screen into the carboy. Approximately 125 grams of solids collected on the screen after the compositing, as shown in Figure 2-1. The solids were wetted with supernate and pushed through the screen. The volume of sample in the 8-L carboy after emptying all of the sample jars was ~ 4.4 L. The total weight of sample added to the carboy was 6.7 kg based on the measured net weight of each jar. Table 2-1 shows the jar and lab ID 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 (see Appendix A).

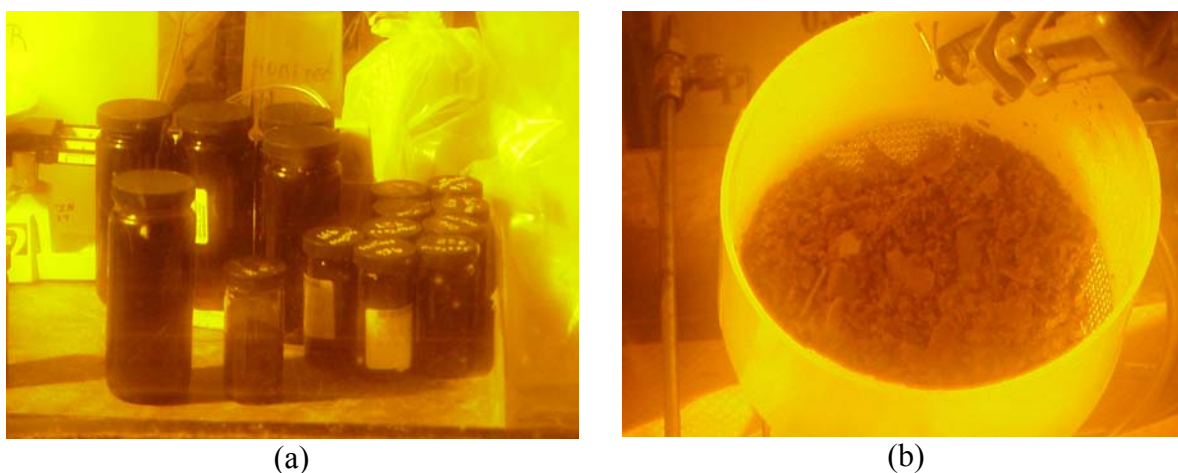


Figure 2-1. As Received AN-107 Sample (a) and Solids (b)

Table 2-1. Net Weight of Sample Jars of AN-107

Jar ID	Sample ID.	Measured Net Wt (g)	Reported Net Wt. (g)
933	7AN-01-02C	159	161.1
935	1998COMP1	153	157.6
927	7AN-01-03A	154	155.8
931	7AN-01-02A	126	137.5
940	1998COMP5	146	147.5
928	7AN-01-03B	158	159.5
929	7AN-01-03C	162	162.8
930	7AN-01-03D	164	168.7
937	1998COMP3	99	100.6
932	7AN-01-02B	164	165.7
936	1998COMP2	154	156.7
941	1998COMP6	91	93.3
934	7AN-01-02D	163	163.9
939	1998COMP4	156	157.6
PIG 17	7AN-01-09	643	648
PIG 21	7AN-01-12	119	125
PIG 24	7AN-01-10	668	670
PIG 11	7AN-01-13	623	632
PIG 6	7AN-01-05	668	669
PIG 10	7AN-01-08	668	662
PIG 5	7AN-01-07	652	648
PIG 29	7AN-01-04	649	644
	Total	6739	6786

3.0 HOMOGENEITY TESTING AND SUB-SAMPLING DISCUSSION

The 8-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 the top, bottom, or midpoint of the original sample height in the 8-L carboy. 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-AN-107 sample. The average volume percent settled solids of the twelve sub-samples was 4.99 with a percent relative standard deviation of 8.6. The data collected indicated the sampling system provided representative sub-samples independent of sampling height or sampling order. Of the twelve 225-mL sub-samples, one was kept as an archive sample, one for physical characterization, and one for chemical and radiological characterization. The remaining nine 225-mL sub-samples were returned to the carboy for subsequent process testing.

Table 3-1. Results of the Homogeneity Test for As-Received 241-AN-107

Cylinder No.	Sampling Region	Sample Volume (mL)	Volume of settled Solids (mL)	Volume Percent Solids (%)
1	Top	224	12	5.36
2	Midpoint	228	12	5.26
3	Bottom	224	12	5.36
4	Top	226	10	4.42
5	Midpoint	228	10	4.39
6	Bottom	226	10	4.42
7	Top	224	10	4.46
8	Midpoint	224	11	4.91
9	Bottom	226	12	5.31
10	Top	225	12	5.33
11	Midpoint	225	12	5.33
12	Bottom	228	12	5.26

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4.0 SAMPLE PREPARATION FOR ANALYSIS

4.1 PREPARATION OF SAMPLES FOR THE ANALYSIS OF LIQUID FRACTIONS

Samples of the supernate were obtained by centrifuging a portion of the sample, decanting the supernate, and filtering it through a 0.45 μ Nylon filter disc. Portions of the filtered supernate were diluted with de-ionized distilled water or a nitric acid and hydrogen peroxide mixture to reduce the sample activity and allow removal from the Shielded Cells for chemical characterization. The weight of the solids collected was recorded to calculate the weight percent of wet centrifuged solids. Solids collected in the centrifuge tube 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 specification¹.

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.

4.2 PREPARATION OF SAMPLES FOR THE ANALYSIS OF SOLIDS FRACTIONS

Triplicate aliquots of the solids collected after centrifugation of the as-received solids were analyzed for the analytes listed in Table 4 of the test specification¹. Samples were prepared for analysis by aqua-regia digestion, microwave digestion, and a water leach procedure.

Sample preparations for each method consisted of solids being collected by centrifuging a portion of the sample and decanting of the supernate. Solids collected in the centrifuge tube were not washed to remove interstitial supernate and were digested on a wet basis. Samples were further diluted to reduce activity and allow removal from the Shielded Cells.

Digestions of a glass standard containing many of the elements found in tank samples were prepared concurrently with the sample preparations. Table 4-1 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. The blank incorporated sample weight measurements for solid conversion data.

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.

Table 4-1. Composition of the Analytical Reference Glass-1 Standard⁴

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%

4.3 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.

5.0 PHYSICAL PROPERTIES MEASUREMENTS

5.1 WEIGHT PERCENT SOLIDS

The weight percent total solids in the sample were measured in the Shielded Cells using a conventional drying oven at 115 °C. The weight percent dissolved solids in a sample of the filtered supernate were measured in the same manner. The weight percent insoluble solids and soluble solids in the sample were calculated from the measurements of the weight percent total solids and the weight percent dissolved solids. Calculation of the weight percent insoluble solids of samples in this manner avoids difficulties associated with reproducibly measuring the insoluble solids directly. Equations 1 and 2 allow calculation of the weight percent of insoluble and soluble solids from the weight percent total solids and dissolved solids measurements. The weight percent soluble solids gives the mass of the dissolved solids in the supernate expressed as a percentage of the mass of the sample. The weight percent of insoluble solids represents the mass of insoluble solids expressed as a percentage of the mass of the sample. All measurements were made in triplicate.

Equation 1
$$w_{is} = (w_{ts} - w_{ds}) / (1 - w_{ds})$$

Equation 2
$$w_{ss} = w_{ts} - w_{is}$$

where:

w_{ds} = weight fraction of dissolved solids	(wt dissolved solids/ wt of supernate)
w_{ts} = weight fraction of total solids	(wt total solids/ wt of sludge slurry)
w_{is} = weight fraction of insoluble solids	(wt insoluble solids/ wt of sludge slurry)
w_{ss} = weight fraction of soluble solids	(wt dissolved solids/ wt of sludge slurry)

5.2 DENSITY AND PARTICLE MORPHOLOGY

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 based on the temperature outside the building. The temperature was recorded for all density measurements and noted in the tables of analytical results.

The Contained Scanning Electron Microscope (CSEM) was used to study the physical features of the AN-107 particles and their structural relationships. It was used to produce an EDX (energy dispersive x-ray) spectrum, which gives a qualitative elemental analysis of the sample constituents. Samples were measured in triplicate. Particle size measurements could not be obtained because the dose rates were too high for the quantity of sample required for this analysis. Qualitative analysis of the AN-107 solids shows the following elements: Na, S, Cl, K, Ca, Cr, Mn, Fe, Ni, Al, and U. Micrographs are shown in Appendix B. The solids are classified as saltcake and considered precipitated salts. SEMs indicate two types of chemical composition in the particles: one dominated by salts, e.g. Na, and the other high in metals. This contributes to the heterogeneity of the solids.

5.3 HEAT CAPACITY AND RHEOLOGY

Heat capacity measurements were performed on the slurry sample in triplicate using a Differential Scanning Calorimeter. Rheological measurements were performed in triplicate using a concentric cylinder geometry head. Measurements were performed at 25 °C and 40 °C.

6.0 ANALYTICAL RESULTS AND DATA EVALUATION

6.1 GENERAL INFORMATION

Table 6-5 through Table 6-8 provide the chemical and radiochemical composition of the as-received samples of the 241-AN-107 tank waste samples 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 solid samples use mg/kg or mCi/kg units. The following list identifies the data tables included in this section with a brief description of the samples analyzed:

6-1	Abbreviations of analytical methods used for each analysis
6-2	Physical properties of the as-received 241-AN-107 sample
6-3	Rheology measurements at 25 °C
6-4	Rheology measurements at 40 °C
6-5	Chemical (inorganic and organic) and radiochemical composition of the filtered centrifuged supernate from the as-received 241-AN-107 sample
6-6	Chemical (inorganic) and radiochemical composition of the solids centrifuged from the as-received 241-AN-107 sample. The samples were digested using a microwave digestion method prior to analysis.
6-7	Chemical (inorganic) and radiochemical composition of the solids centrifuged from the as-received 241-AN-107 sample. The samples were digested using aqua-regia prior to analysis.
6-8	Chemical (inorganic and organic) composition of the solids centrifuged from the as-received 241-AN-107 sample. The samples were digested using a water leaching procedure prior to analysis.

6.2 QC FLAGS

The analytical result tables include a quality control (QC) flag in the last column to indicate 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-AN-107 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) analysis for each analyte. In addition, analyses in which the concentration of the analyte in the blank exceeded 5% of the concentration in the sample and instances in which the analyte concentration detected in the samples was less than ten times the detection level for the sample were also flagged.

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 detection level for the sample.

Most of the QC failures can be attributed to low concentrations present in the sample or 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, a large percentage of the uncertainty is due to the sampling method specified by the test specification. Sampling the solids using centrifugation does not work well with samples having concentrated supernates and low insoluble solids that can't be washed to remove interstitial supernate. The solids are classified as saltcake and considered precipitated salts. The SEMs indicate two types of chemical composition in the particles, one dominated by salts, e.g. Na, the other high in metals. This also contributes to the heterogeneity of the solids. Extensive sample preparation in the Shielded Cells may 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 led 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.

6.3 DATA EVALUATION

6.3.1 Discussion of Table 6-2, Physical Properties of the As-Received 241-AN-107 Sample

Data for the volume percent centrifuged solids and weight percent insoluble solids show high percent relative standard deviations for the replicate measurements. Both measurements have inherently low precision owing to the low solids concentration and presence of variable amounts of interstitial supernate trapped in the solids. In both measurements, the solids were not washed free of interstitial supernate. It is likely a substantial percentage of the solids would dissolve during washing. The amount of supernatant contamination can affect the weight and thus the final concentration significantly for small sample sizes.

6.3.2 Discussion of Table 6-5, Composition of the As-Received 241-AN-107 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. Ag, Li, Mg, P, U, and V were flagged as U_M for not meeting the required MRQ. These failures were attributed to the dilutions necessary to allow removal from the Shielded Cells for analysis. The unmet MRQ for P and U is not critical since the concentrations are well above the detection limit.
- Sodium results by ICP-ES and AA show good agreement. The difference between the two methods is less than 10%.
- High relative standard deviation for ^{237}Np from ICP-MS is attributed to the low concentration in the sample. W failed to meet the matrix spike recovery and relative standard deviation requirements. It is attributed to W being a contaminant in the system.
- High relative standard deviation for TIC/TOC is attributed to the limitation of the method. Samples that contain complex organic acids may not be entirely combusted by the method. This is evident when comparing the sample against the serial dilution. Serial dilution analyzed higher than the undiluted sample indicating incomplete combustion in the original measurement.
- Total base and free OH were flagged for not meeting the MRQ. The unmet MRQ for total base is of minimal impact because the total base concentration is well above the detection limit.
- Sulfur concentrations obtained from IC (as sulfate) and ICP-ES show reasonable agreement. The difference is approximately 25%.
- Phosphorus analysis by ICP-ES yielded a result approximately two times less than the concentration measured by IC (as phosphate). The ratio between the two measurements should be a factor of 3 if the phosphorus is present entirely as phosphate.
- Acetate was flagged as not meeting the required recovery for the matrix spike. Carbonate showed inconsistent data that caused it to fail the %RSD, matrix spike recovery, and the laboratory control standard recovery.
- ^{90}Sr was flagged for not meeting the matrix spike recovery. The matrix spike recovery was high, but this is not judged to impact data quality. The recovery was poor due to the relatively large amount of ^{90}Sr in the sample relative to the added ^{90}Sr spike. However, each individual analysis in the batch is yielded independently using a neutron activation analysis (NAA) step, so the data is technically sound and defensible.
- ^{154}Eu , ^{155}Eu , ^{60}Co , $^{126}\text{Sb}/^{126}\text{Sn}$, and ^{125}Sb from gamma spectrometry were flagged as not meeting the %RSD. Sn and Sb isotopes were flagged for not meeting the MRQ requirements. This is attributed to the Sn and Sb isotopes being within an order of magnitude of the detection limits for the respective samples analyzed and low sample concentration.

- Results for the ^{99}Tc pertechnetate are reported as upper limits due to interference evident in the liquid scintillation analysis. ^{99}Tc measured as pertechnetate and the total ^{99}Tc measured differ by ~51%, indicating approximately half of the technetium is in forms other than pertechnetate.
- ^{129}I was flagged for not meeting the %RSD requirement. 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.
- ^{14}C analysis was flagged for not meeting the required MRQ and %RSD. All sample results were within an order of magnitude of analytical detection limits, the beta spectrum of the 3rd replicate and the serial dilution still showed evidence of residual higher energy beta, and as a result were caveated as upper limits. The failure to meet the MRQ and %RSD requirement was attributed to low concentrations present in the sample and the dilutions necessary to allow removal from the Shielded Cells for analysis.
- ^{137}Cs concentration measured by gamma spectroscopy and ICP-MS show agreement with a difference of approximately 23%. The ICP-MS measurement is biased high due to Ba concentrations in the supernate.
- Higher energy beta was observed in the ^{79}Se spectra so the results are caveated as upper limits.
- ^{241}Am , ^{243}Am , and ^{244}Cm were flagged for not meeting the required %RSD. ^{243}Am and ^{242}Cm were flagged for not meeting the required minimum reportable quantity. The blank sample showed ^{241}Am , ^{243}Am , ^{242}Cm , and ^{244}Cm contamination. A potential positive bias for the ^{242}Cm may exist if any ^{252}Cf is present in the samples.

6.3.3 Discussion of Table 6-6, Composition of the As-Received 241-AN-107 Solids Microwave Prep

- Several of the elements were flagged for not meeting the required %RSD. Two sets of samples were analyzed and the problem of high RSDs occurred in both sets. This is attributed to the solid/liquid separation method. Many of what are generally thought of as solids associated analytes (e.g., Mn, Fe, Ni, Pb, Zn) are as high or higher in the supernate than in the solids for AN-107 because of the complexants in the tank. Several of the elements were flagged for not meeting the required MRQ and having values less than 10 times the detection limit. These failures were attributed to low concentrations present in the sample and the dilutions necessary to allow removal from the Shielded Cells for analysis.

- ICP-ES data set (IE designator in table) for the second solid digestion was used because of significant contamination of the blank and glass standard in the first data set. Silver, barium, magnesium, zinc, and titanium were flagged for not meeting the required MRQ. Copper was flagged for the blank exceeding 5% of the sample concentration. Calcium, iron, manganese, sodium, nickel, and strontium were flagged for not meeting the %RSD. Sulfur and silicon were flagged for the blank exceeding 5% of sample concentration and not meeting the required %RSD. Aluminum was flagged for the blank exceeding 5% of sample concentration, not meeting MRQ and values less than 10 times the detection limits. Lanthanum was flagged for the blank exceeding 5% of sample concentration, not meeting the required %RSD, and MRQ. Vanadium was flagged for the blank exceeding 5% of sample concentration, not meeting the required %RSD, and values less than 10 times the detection limits. Chromium was flagged for not meeting the required %RSD and MRQ. Cadmium was flagged for values less than 10 times the detection limits and not meeting the required %RSD. Phosphorus was flagged for values not meeting the required MRQ and values less than 10 times the detection limits.
- Aluminum, chromium, lead, and lanthanum unmet MRQs are of minimal impact since the concentrations present are well above the detection limits.
- Cu, S, V, ²³⁸U, ¹³³Cs, and Ce by ICP-ES and ICP-MS may be biased high due to the presence of a significant interference or contamination observed in the glass standard.
- Sodium did not meet the %RSD by AA which was attributed to variable amounts of interstitial liquid in the solids at high sodium concentrations and selenium did not meet the required MRQ. This was attributed to low concentration in the sample.
- Sodium by AA and ICP-ES are in good agreement. The difference between the two measurements is 0.2%.
- Thorium, molybdenum, ruthenium, and rhodium were flagged for not meeting the required MRQ and %RSD. This was attributed to low sample concentration. Thorium unmet MRQ is of minimal impact since the concentrations present are above the detection limit.
- U²³⁵ and Cobalt by ICP-MS were flagged for not meeting the % recovery of LCS.
- Vanadium was flagged for the blank exceeding 5% of the sample concentration and not meeting the required % recovery of LCS. This is attributed to a contaminant in the system.
- Selenium, lead, antimony, barium, platinum, thallium, plutonium, tellurium, bismuth, and neodymium were flagged for not meeting the required MRQ. This was attributed to low sample concentration and the dilutions necessary to allow removal from the Shielded Cells for analysis. Neodymium values are upper limits. Neodymium unmet MRQ is of minimal impact since the concentrations present are above the detection limit.
- Rubidium, tantalum, and yttrium were flagged for not meeting the %RSD. This was attributed to low sample concentration and the dilutions necessary to allow removal from the Shielded Cells for analysis.

- ^{133}Cs was flagged for the blank exceeding 5% of the sample concentration and not meeting the required %RSD. This is attributed to contamination in the cells.
- Cerium was flagged for the blank exceeding 5% of the sample concentration.
- $^{126}\text{Sb}/^{126}\text{Sn}$, ^{152}Eu , and ^{125}Sb were flagged for not meeting the required MRQ. This was attributed to low sample concentration and the dilutions necessary to allow removal from the Shielded Cells for analysis.
- ^{79}Se was flagged for not meeting the required MRQ. This was attributed to low sample concentration and the dilutions necessary to allow removal from the Shielded Cells for analysis. Higher energy beta was observed in the samples' ^{79}Se spectra, therefore those results were caveated as upper limits.
- ^{241}Am was flagged for not meeting the required %RSD. ^{244}Cm was flagged for the blank exceeding 5% of the sample concentration and not meeting the required %RSD. ^{242}Cm was flagged for not meeting the required MRQ and %RSD. A potential positive bias for the ^{242}Cm may exist if any ^{252}Cf is present in the samples. The blank sample generated in the cells showed significant ^{244}Cm contamination.
- ^{59}Ni was flagged for not meeting the required MRQ and %RSD. This was attributed to low sample concentration and the dilutions necessary to allow removal from the Shielded Cells for analysis.
- Gross alpha was flagged for not meeting the required %RSD. Prior to the analyses, ^{137}Cs was removed from aliquots of the samples in order to reduce bias effects caused by large beta/alpha ratios. This was accomplished using Bio-Rad AMP1 resin. Following the Cs removal process, aliquots of the Cs-stripped samples were mounted on stainless steel counting planchets and analyzed for alpha activity using a gas-flow proportional counter. Results are background subtracted. The analysis was carried out in quadrature to illustrate the consistency within measurements for each given sample. Average results were provided. The %RSD was outside of the requested range. However, results of the four individual analyses used for each single sample all had %RSD values of <11.4%. This indicates inconsistencies between samples and that the solids are somewhat heterogeneous..
- ^3H was flagged for not meeting the required MRQ and percent recovery of the LCS. This was attributed to low sample concentration and the dilutions necessary to allow removal from the Shielded Cells for analysis.
- ^{137}Cs concentration measured by gamma spectroscopy and ICP-MS show a difference of approximately 78%. The values for ^{135}Cs and ^{137}Cs from the ICP-MS are upper limits and 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 based upon the presence of a significant interference or contamination in the blank and glass standard.
- ^{90}Sr was flagged for not meeting the required %RSD.
- ^{238}Pu and $^{239/240}\text{Pu}$ were flagged for the blank exceeding 5% of the sample concentration and not meeting the required MRQ and %RSD. ^{238}Pu was flagged for not meeting the required %RSD. This was attributed to the dilutions necessary to allow removal from the Shielded Cells for analysis and contamination in the cells.

- ^{14}C was flagged for not meeting the required MRQ and percent recovery of the LCS. These solids were so high in activity that they couldn't be run through the routine ^{14}C method, which was adjusted to enhance the separation of ^{14}C beta from the other activity. The provided sub-samples were washed from their respective centrifuge tubes with dilute caustic; the wash solution was wet-ashed with a sodium persulfate/silver nitrate oxidation in conjunction with concentrated sulfuric acid. The carbon dioxide emitted was absorbed with 3M NaOH over a period of several days. The 3M NaOH was then acidified, liberating the carbon dioxide, which was re-absorbed with Packard Instruments Carbosorb E over a period of several days. The Carbosorb E was then slurried into Ultima Gold AB, and analyzed by liquid scintillation analysis for ^{14}C . A laboratory control blank solution, spiked with a ^{14}C standard, was run in duplicate, in parallel with the samples to determine ^{14}C recoveries, the average of which were applied to the sample ^{14}C LSC results to quantify the ^{14}C concentrations in the samples. One set of AN-107 samples was spiked with ^{14}C (again in duplicate) and run through the process to serve as the matrix spike. A second laboratory control blank solution, spiked with a ^{14}C standard, was run through the process in duplicate to serve as the LCS sample.

6.3.4 Discussion of Table 6-7, Composition of the As-Received 241-AN-107 Solids Aqua Regia Prep

- All analytes analyzed by ICP-AES were flagged for quality control. This is attributed to low sample concentration and dilutions necessary to allow removal from the Shielded Cells for analysis and to the solid/liquid separation method required by the test specification. Many of what are generally thought of as solids associated analytes (e.g., Mn, Fe, Ni, Pb, Zn) are as high or higher in the supernate than in the solids for AN-107 because of the complexants in the tank.
- Magnesium, Silver, Barium, Lithium, Titanium, and Vanadium were flagged for not meeting the required MRQ. Cd and S were flagged for not meeting the %RSD. Ca and Sr were flagged for the blank exceeding 5% of the sample concentration. Copper was flagged for the blank exceeding 5% of the sample concentration, being less than 10x the detection limit, and not meeting the required MRQ. The following were flagged for being less than 10x the detection limit and not meeting the required MRQ: Be, Si, U, and Zr. Manganese, Nickel, and Lead were flagged for not meeting the required MRQ and %RSD. Aluminum and Phosphorus were flagged for the blank exceeding 5% of the sample concentration and not meeting the required MRQ and %RSD. Chromium, Iron, and Zinc were flagged for the blank exceeding 5% of the sample concentration and not meeting the %RSD.
- Aluminum, phosphorus, lead, manganese, and nickel unmet MRQs are of minimal impact since the concentrations present are well above the detection limit.
- Potassium was flagged for the blank exceeding 5% of the sample concentration and not meeting the %RSD. Sodium was flagged for not meeting the required %RSD and Selenium was flagged for not meeting the required MRQ.

- ^{233}U , ^{234}U , Se, Te, Pr, Ta, Pt, and Tl were flagged for not meeting the required MRQ. This is attributed to low sample concentration and dilutions necessary to allow removal from the Shielded Cells for analysis.
- ^{238}U , total uranium, ^{133}Cs , and Ce were flagged for not meeting the required %RSD. This is attributed to dilutions necessary to allow removal from the Shielded Cells for analysis.
- Thorium, Cobalt, Ruthenium, Molybdenum, Lead, Rhodium, Plutonium, Yttrium, and Neodymium were flagged for not meeting the required MRQ and %RSD. This is attributed to dilutions necessary to allow removal from the Shielded Cells for analysis and low concentrations in the sample.
- Thorium, ruthenium, molybdenum, rubidium, rhodium, yttrium, neodymium, and praseodymium unmet MRQs are of minimal impact since the concentrations are well above the detection limit.
- ^{236}U and ^{237}Np were flagged for values being less than 10x the detection limit and not meeting the required %RSD. This is attributed to low concentrations in the sample.
- ^{235}U , Vanadium, ^{135}Cs , and ^{137}Cs were flagged for the blank exceeding 5% of the sample concentration and not meeting the required %RSD. This is attributed sample contamination.
- Arsenic, Rubidium, Antimony, and Barium were flagged for not meeting the required MRQ and %RSD and the blank exceeding 5% of the sample concentration.
- Tungsten was flagged for not meeting the percent recovery of the MS and %RSD. This is attributed to contamination within the system.
- Bismuth was flagged for not meeting the required MRQ and the blank exceeding 5% of the sample concentration.
- ^{137}Cs was flagged for not meeting the required %RSD. The third replicate appeared higher than the other two replicates. The third replicate was reanalyzed and the second analysis confirmed the first analysis. This trend of the third replicate appearing higher than the other two replicates was also noted in the Sr-90 results.
- ^{137}Cs concentration measured by gamma spectroscopy and ICP-MS show a difference of ~50%. The values for ^{135}Cs and ^{137}Cs from the ICP-MS have a high bias due to the stable 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 an interference or contamination in the glass standard.
- Tritium was flagged for not meeting the required MRQ. The third replicate did not undergo a successful separation. This was evident in the spectrum of the distillate, therefore the data is not reported.

- The gross alpha was flagged for the blank exceeding 5% of the sample concentration and not meeting the %RSD. ^{137}Cs was removed from aliquots of the samples in order to reduce bias effects caused by large beta/alpha ratios. This was accomplished using Bio-Rad AMP1 resin. Following the Cs removal process, aliquots of the Cs-stripped samples were mounted on stainless steel counting planchets and analyzed for alpha activity using a gas-flow proportional counter. Results are background subtracted. The analysis was carried out in quadrature to illustrate the consistency between the measurement for each given sample. Average results are provided. The %RSD was 20%, slightly outside of the requested range. However, results of the 4 individual analyses used for each single sample all had %RSD values of <10%. This indicates inconsistencies with in the samples. The blank sample contained more than 10% of the activity observed in the samples indicating contamination.
- ^{90}Sr was flagged for not meeting the required MRQ and %RSD. The MRQ is not critical in this case because the concentration is well above the method detection limit.
- ^{241}Am , ^{243}Am , ^{242}Cm , and ^{244}Cm all were flagged for the blank exceeding 5% of the sample concentration and not meeting the %RSD. ^{242}Cm was flagged for not meeting the required MRQ as well. A potential positive bias for the ^{242}Cm may exist if any ^{252}Cf is present in the samples. The blank sample generated in the cells showed significant contamination in the blank and glass standard.
- ^{238}Pu and $^{239/240}\text{Pu}$ were flagged for the blank exceeding 5% of the sample concentration and not meeting the required MRQ and %RSD. ^{238}Pu was flagged for not meeting the required %RSD. Comparable amounts of Pu were present in the blank and the samples. Each sample was subjected to two different spike steps. Results from both experiments were averaged and each lab replicate agreed within 1 sigma. 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.
- ^{129}I was flagged for not meeting the required MRQ. This was attributed to low concentrations present in the sample and the dilutions necessary to allow removal from the Shielded Cells for analysis.

6.3.5 Discussion of Table 6-8, Composition of the As-Received 241-AN-107 Solids Water Leach Prep

- Fluoride was flagged for not meeting the MRQ. This failure was attributed to low concentrations present in the sample.
- The blank for the analytes appear high because the sample dilution factor is applied to the blank.

6.4 DATA TABLES

Data are presented in this section in Table 6-1 through Table 6-8.

Table 6-1. Abbreviations for Analytical Methods in Tables 6-5 through 6-8

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
Gross Alpha and Beta		ADS-2424
Tc-99		ADS-2445 ADS-2446
GC-MS	GM	ADS-2661

Table 6-2. Physical Properties of the As-Received 241-AN-107 Sample

Property	Units	1st Replicate	2 nd Replicate	3rd Replicate	Average	%RSD	QC Flag
Slurry Density	g/mL	1.42	1.41	1.42	1.42	0.41	-
Filtered Supernate Density	g/mL	1.414	1.415	1.415	1.415	0.04	-
Vol % Centrifuged Solids	Vol%	8	10	6	8	25	U _R
Wt. % Centrifuged Solids	Wt%	7.18	6.51	6.95	6.88	5	-
Wt% Total Dried Solids	Wt%	49.65	49.45	49.4	49.50	0.27	-
Wt% Dissolved Solids	Wt%	49.49	48.99	49.29	49.25	0.51	-
Wt% Insoluble solids	Wt%	0.323	0.911	0.215	0.483	78	U _R
Heat Capacity	J/g-°C	3.39	3.39	3.39	3.39	0.08	-

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 6-3. Rheology Measurements at 25 °C

Replicates	Sweep Direction	Viscosity cP	Offset Pa	R²	Range sec⁻¹
Replicate1	Up	9.76	1.11	0.9972	0 - 1000
Replicate2		9.24	1.67	0.9966	0 - 1000
Replicate3		9.27	1.71	0.9973	0 - 1000
Replicate1	Down	9.18	1.48	0.9977	0 - 1000
Replicate2		9.37	1.43	0.9976	0 - 1000
Replicate3		9.34	1.48	0.9986	0 - 1000
	Average	9.36	1.48		
	%RSD	2.23	14.52		

Table 6-4. Rheology Measurements at 40 °C

Replicates	Sweep Direction	Viscosity cP	Offset Pa	R²	Range sec⁻¹
Replicate1	Up	5.87	1.07	0.9934	0 - 850
Replicate2		5.89	1.17	0.9925	0 - 850
Replicate3		5.89	1.20	0.9951	0 - 850
Replicate1	Down	5.85	0.96	0.9934	0 - 850
Replicate2		5.85	1.01	0.9925	0 - 850
Replicate3		5.86	1.00	0.9951	0 - 850
	Average	5.87	1.07		
	%RSD	0.28	9.27		

Table 6-5. Composition of the As-Received 241-AN-107 Filtered Supernate

Analyte	1 st Replicate (mg/L)	2 nd Replicate (mg/L)	3 rd Replicate (mg/L)	Average (mg/L)	%RSD	Blank (mg/L)	LCS % Recovery	MS % Recovery	QC Flag
Ag* (IE)	2	<2.27	2	2	4	<0.880	101	NA	U _E U _M
Al (IE)	314	295	308	306	3	<6.27	100	NA	-
B* (IE)	13	<7.95	12	13	4	<3.08	94	NA	U _E
Ba (IE)	10	9	10	10	3	<2.09	103	118	U _E
Be (IE)	<0.156	<0.284	<0.156	<0.199	-	<0.110	100	NA	U _E
Ca (IE)	565	577	557	566	2	<2.53	102	NA	-
Cd (IE)	73	72	71	72	1	<0.462	102	110	-
Cr (IE)	195	189	190	191	2	<0.660	102	109	-
Cu (IE)	38	35	41	38	8	1.02	99	NA	-
Fe (IE)	1889	1860	1846	1865	1	2.13	102	105	-
Li (IE)	<6.72	<12.2	<6.72	<8.55	-	<4.73	96	NA	U _E U _M
Mg ~ (IE)	0.879	<1.51	<0.828	0.879	-	<0.583	103	NA	U _E U _M
Mn (IE)	673	659	659	664	1	<0.088	102	NA	-
Na (IE)	210,160	207,320	205,900	207,793	1	36.3	98	95	-
Ni (IE)	589	582	572	581	1	<1.49	103	99	-
P (IE)	558	558	565	560	1	<7.59	103	NA	U _M
Pb (IE)	471	447	460	460	3	<3.51	102	NA	-
S (IE)	3877	3664	3749	3763	3	<15.1	100	NA	-
Si (IE)	16	15	14	15	6	<1.84	102	NA	U _E
Sr (IE)	120	124	118	121	3	<0.836	101	NA	-
Ti (IE)	<1.20	<2.19	<1.20	<1.53	-	<0.847	N/A	NA	U _E
U (IE)	172	168	169	169	1	<25	98	NA	U _E U _M
V (IE)	0.581	0.859	0.914	0.785	23	<0.220	101	NA	U _R U _E U _M
Zn (IE)	30	27	31	29	7	<0.352	102	NA	-
As (AA)	<0.9372	<0.937	<0.937	<0.937	-	<0.05	91	116	-
K (AA)	1541	1555	1571	1556	1	<5.22	100	97	-
Na (AA)	192,166	185,149	187,647	188,321	2	<280	96	98	-
Se (AA)	<0.937	<0.937	<0.937	<0.937	-	<0.05	110	98	-
Hg (AA)	<2.29	<2.30	<2.27	<2.29	-	<0.11	104	107	-
Th (IM)	16	18	19	18	7	0.02	99	110	-
²³⁴ U (IM)	<0.033	<0.033	<0.033	<0.0327	-	<0.023	NA	NA	-
²³⁵ U (IM)	0.83	0.71	0.81	0.78	8	<0.023	97	118	-
²³⁶ U ~ (IM)	0.04	<0.033	<0.033	0.04	-	<0.023	NA	NA	-
²³⁷ Np (IM)	0.11	0.08	0.08	0.09	21	<0.023	NA	NA	U _R
²³⁸ U (IM)	127	126	123	125	2	0.49	97	98	-
²³⁹ Pu (IM)	0.74	0.70	0.69	0.71	4	<0.023	NA	NA	-
²⁴⁰ Pu (IM)	0.06	0.05	0.05	0.05	8	<0.023	NA	NA	-
Total U (IM)	128	127	124	126	2	0.49	NA	NA	-
Rb (IM)	7	8	8	8	2	0.004	105	113	-

Table 6-5. Composition of the As-Received 241-AN-107 Filtered Supernate (page 2 of 3)

Analyte	1 st Replicate (mg/L)	2 nd Replicate (mg/L)	3 rd Replicate (mg/L)	Average (mg/L)	%RSD	Blank (mg/L)	LCS % Recovery	MS % Recovery	QC Flag
¹³³ Cs (IM)	11	11	11	11	1	0.01	104	111	-
¹³⁵ Cs (IM)	2	2	2	2	1	0.13	104	NA	-
¹³⁷ Cs (IM)	5	5	5	5	1	0.25	98	NA	-
La (IM)	35	35	36	35	2	0.01	101	NA	-
W (IM)	114	214	166	165	30	0.024	113	167	U _S U _R
HEDTA (HL)	1111	935	1321	1123	17	<10	95	92	-
EDTA (HL)	3607	3403	3870	3627	6	<10	105	93	-
IDA (GM)	3783	4344	4034	4054	7	<25	109	107	-
TIC (A)	27500	20800	31500	26600	20	0	99.6	102	U _R
TOC (Diff)	27200	34500	38200	33300	17	9.44	101	99	U _R
¹³³ Cs (IM)	11	11	11	11	1	0.01	104	111	-
Total Base (T)	1.32 M	1.32 M	1.32 M	1.32 M	0	<0.02 M	96	99	U _M
Free OH (T)	<340	<340	-	<340	-	-	103	98.6	U _M
F (IC)	3440	3047	3471	3319	7	<20	105	115	-
CHO ₂ (IC)	11503	10332	11503	11112	6	<100	102	113	-
Cl (IC)	1440	1275	1461	1392	7	<20	99	103	-
NO ₂ (IC)	62694	56373	63212	60760	6	<100	105	105	-
NO ₃ (IC)	232123	207253	235232	224869	7	<100	100	115	-
PO ₄ (IC)	984	881	974	946	6	<100	98	97	-
SO ₄ (IC)	8715	7668	9057	8480	9	<50	99	108	-
C ₂ O ₄ (IC)	694	591	705	663	10	<100	100	105	-
NH ₃ (PT)	404	425	415	415	3	<10	109	116	-
Citrate (IC)	12124	12435	12021	12193	2	<10	94	95	-
Glycolate (IC)	24767	24870	24767	24801	0.2	<10	86	125	-
Acetate (IC)	1534	1420	1368	1440	6	<10	87	67	U _S
Carbonate (IC)	20933	19585	12953	17824	24	<50	155	174	U _R U _S U _L

~ One Value

QC Flags: none - meets all QC

U_B - blank exceeds 5% of sample concentration

U_E - value less than 10x the MDL

N/M- not measured

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

NA - not applicable

Table 6-5. Composition of the As-Received 241-AN-107 Filtered Supernate (page 3 of 3)

Analyte	1 st Replicate (mCi/L)	2 nd Replicate (mCi/L)	3 rd Replicate (mCi/L)	Average (mCi/L)	%RSD	Blank (mCi/L)	LCS % Recovery	MS % Recovery	QC Flag
Gross Beta (SL)	6.03E+02	5.88E+02	5.85E+02	5.92E+02	1.61	<2.20E-02	100.77	94.01	-
²³⁸ Pu (SA)	1.21E-02	1.34E-02	1.25E-02	1.26E-02	5.19	2.98E-04	NA	NA	-
^{239/240} Pu (SA)	4.54E-02	5.67E-02	4.75E-02	4.99E-02	12.12	2.82E-05	NA	NA	-
²⁴¹ Pu (SA)	8.38E-02	9.34E-02	8.25E-02	8.66E-02	6.87	1.05E-03	NA	NA	-
¹³⁷ Cs (GS)	3.36E+02	3.31E+02	3.37E+02	3.35E+02	0.90	<1.18E-02	NA	NA	-
⁹⁰ Sr (SL)	8.25E+01	8.70E+01	8.32E+01	8.42E+01	2.88	<1.93E-03	90.61	164.32	U _S
⁹⁹ Tc Pertech. (SL)	<5.23E-02	<4.73E-02	<5.36E-02	<5.11E-02	-	<6.26E-06	NA	NA	-
⁹⁹ Tc (total) (SL)	1.02E-01	1.02E-01	1.07E-01	1.04E-01	2.69	<6.94E-06	NA	NA	-
⁵⁹ Ni (SL)	2.11E-02	2.82E-02	2.48E-02	2.47E-02	14.29	<9.96E-06	NA	NA	-
⁶³ Ni (SL)	1.66E+00	1.73E+00	1.61E+00	1.66E+00	3.44	<1.59E-05	NA	NA	-
⁶⁰ Co (GS)	9.63E-02	6.87E-02	7.98E-02	8.16E-02	17.01	<1.60E-06	NA	NA	U _R
¹²⁶ Sb/ ¹²⁶ Sn (GS)	9.01E-04	5.73E-04	7.10E-04	7.28E-04	22.63	<1.22E-06	NA	NA	U _E U _M U _R
¹²⁵ Sb (GS)	3.06E-03	1.74E-03	2.48E-03	2.42E-03	27.36	<3.67E-06	NA	NA	U _E U _R
¹⁵² Eu (GS)	8.29E-03	6.63E-03	7.20E-03	7.37E-03	11.40	<1.08E-05	NA	NA	-
¹⁵⁴ Eu (GS)	5.11E-01	3.64E-01	4.25E-01	4.33E-01	17.00	4.13E-06	NA	NA	U _R
¹⁵⁵ Eu (GS)	3.02E-01	2.07E-01	2.47E-01	2.52E-01	19.04	<2.75E-06	NA	NA	U _R
²³¹ Pa (GS)	<1.37E-02	<1.14E-02	<1.23E-02	<1.25E-02	-	<4.21E-05	NA	NA	-
¹²⁹ I (SG)	2.31E-04	9.15E-05	1.25E-04	1.49E-04	48.81	1.87E-05	NA	NA	U _R
¹⁴ C* (SL)	6.22E-04	3.96E-04	<1.07E-03	6.97E-04	49.42	<3.33E-05	102.7	104.8	U _E U _M U _R
⁷⁹ Se (SL)	<4.55E-03	<6.49E-03	<3.24E-03	<4.76E-03	-	<1.37E-04	NA	NA	-
²⁴¹ Am (SG)	6.99E-01	1.01E+00	7.10E-01	8.06E-01	22	5.05E-05	NA	NA	U _R
²⁴³ Am (SA)	1.02E-03	1.86E-03	1.03E-03	1.30E-03	37	3.03E-06	NA	NA	U _M U _R
²⁴⁴ Cm (SA)	4.92E-02	3.48E-02	2.70E-02	3.70E-02	30	2.32E-04	NA	NA	U _R
²⁴² Cm (SA)	2.63E-03	3.37E-03	2.95E-03	2.98E-03	12	5.00E-07	NA	NA	U _M

* 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

N/M- not measured

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 6-6. Composition of the Microwave Digested As-Received 241-AN-107 Centrifuged Solids

Analyte	1 st Replicate (mg/kg)	2 nd Replicate (mg/kg)	3 rd Replicate (mg/kg)	Average (mg/kg)	%RSD	Blank (mg/kg)	Glass Std. (mg/kg)	LCS % Recovery	MS % Recovery	QC Flag
Ag (IE)	<10.6	<13.7	<10.6	<11.63	-	<11.5	<32.9	100	NA	U _M
Al (IE)	260	300	231	264	13	47.2	22100	104	NA	U _B U _M U _E
Ba (IE)	<25.2	<32.6	<25.1	<27.63	-	<27.2	867	99	104	U _M
Be (IE)	<5.11	<6.62	<5.11	<5.61	-	<5.53	19.8	99	NA	-
Ca (IE)	737	716	494	649	21	<33	10100	94	NA	U _R
Cd (IE)	41.1	47	32	40	19	<6.02	<17.3	97	97	U _E U _R
Cr (IE)	122	154	73.7	117	35	<8.6	669	96	102	U _R U _M
Cu (IE)	26.5	28.6	21.3	25	14.76	8.54	53.6	99	NA	U _B
Fe (IE)	1350	1530	908	1263	25	49.5	101000	98	102	U _R
La (IE)	30.3	42.8	28.1	34	23	10.8	66.5	100	NA	U _B U _R U _M
Li (IE)	<57	<73.8	<56.9	<62.57	-	<61.6	15300	95	NA	-
Mg (IE)	<7.02	<9.09	<7.01	<7.71	-	<7.59	5320	98	NA	U _M
Mn (IE)	419	503	300	407	25	<1.15	15100	98	NA	U _R
Na (IE)	159000	188000	118000	155000	23	96.6	65500	95	103	U _R
Ni (IE)	383	489	284	385	27	<19.3	8920	102	106	U _R
P~ (IE)	182	<118	<91.3	182	-	<98.9	509	99	NA	U _E U _M
Pb (IE)	236	371	120	242	52	<45.7	<131	100	NA	U _R U _E U _M
S (IE)	4120	4680	3190	3997	19	2060	7410	96	NA	U _B U _R
Si (IE)	552	434	340	442	24	326	206000	98	NA	U _B U _R
Sr (IE)	164	158	101	141	25	<10.9	2290	100	NA	U _R
Ti (IE)	<10.2	<13.2	<10.2	<11	-	<11	7080	99	NA	U _M
U (IE)	<301	<389	<300	<330	-	<325	2800	100	NA	-
V (IE)	15.2	16.7	11.3	14	19	9.36	155	99	NA	U _B U _E U _R
Zn (IE)	<4.24	<5.49	<4.23	<5	-	<4.58	139	97	NA	U _M
Zr (IE)	50.1	58.4	32.4	47	28	<32.7	1020		NA	U _R U _E U _M

Table 6-6. Composition of the Microwave Digested As-Received 241-AN-107 Centrifuged Solids (page 2 of 4)

Analyte	1 st Replicate (mg/kg)	2 nd Replicate (mg/kg)	3 rd Replicate (mg/kg)	Average (mg/kg)	%RSD	Blank (mg/kg)	Glass Std. (mg/kg)	LCS % Recovery	MS % Recovery	QC Flag
As (AA)	<18.3	<18.5	<22.1	<19.6	-	<18.27	<21.5	119.1	88.2	-
K (AA)	969	1060	955	995	6	<60.9	22200	99.2	105	-
Na (AA)	151000	163000	150000	154667	5	<4153	73800	95.4	102	U _R
Se (AA)	<18.3	<18.5	<22.1	<19.6	-	<18.27	<21.5	104.5	106	U _M
Hg (AA)	<44.7	<45.2	<54.0	<48.0	-	<44.66	<52.5	95.6	98.3	-
Th (IM)	37	27	24	29	24	12	9	NA	113	U _R U _M
²³³ U (IM)	<1.39	<1.39	<1.39	<1.39	-	<1.39	<1.39	NA	NA	-
²³⁴ U (IM)	<1.39	<1.39	<1.39	<1.39	-	<1.39	<1.39	NA	NA	-
²³⁵ U (IM)	<1.39	<1.39	<1.39	<1.39	-	<1.39	<1.39	125	NA	U _L
²³⁶ U (IM)	<1.39	<1.39	<1.39	<1.39	-	<1.39	<1.39	NA	NA	-
²³⁷ Np (IM)	<1.39	<1.39	<1.39	<1.39	-	<1.39	<1.39	NA	NA	-
²³⁸ U (IM)	81	89	94	88	8	<1.39	10	110	114	-
²³⁹ Pu (IM)	<1.39	<1.39	<1.39	<1.39	-	<1.39	<1.39	NA	NA	-
²⁴⁰ Pu (IM)	<1.39	<1.39	<1.39	<1.39	-	<1.39	<1.39	NA	NA	-
Total U (IM)	81	89	94	88	8	<1.39	10	NA	NA	-
V (IM)	166	159	184	170	8	191	330	124	NA	U _B U _L
Co (IM)	120	124	110	118	6	<113	229	131	NA	U _L
As (IM)	<20	<20	<20	<20	-	<20	<20	111	NA	-
Se (IM)	<300	<300	<300	<300	-	<300	<300	NA	NA	U _M
Rb (IM)	39	52	53	48	16	<10	<10	NA	93	U _R
Ru (IM)	48	59	35	48	25	<0.7	<0.7	NA	NA	U _R U _M
Mo (IM)	27	30	16	24	29	<30	47	117	NA	U _R U _M
Pd (IM)	12	15	11	13	15	<4.8	12	NA	NA	U _M
Sb (IM)	<12	<12	<12	<12	-	<12	<12	112	NA	U _M
Te (IM)	<6	<6	<6	<6	-	<6	<6	NA	NA	U _M

Table 6-6. Composition of the Microwave Digested As-Received 241-AN-107 Centrifuged Solids (page 3 of 4)

Analyte	1 st Replicate (mg/kg)	2 nd Replicate (mg/kg)	3 rd Replicate (mg/kg)	Average (mg/kg)	%RSD	Blank (mg/kg)	Glass Std. (mg/kg)	LCS % Recovery	MS % Recovery	QC Flag
¹³³ Cs (IM)	90	96	176	121	40	116	106	NA	105	U _B U _R
¹³⁵ Cs (IM)	5	5	5	5	0	<1.5	<1.5	NA	NA	-
¹³⁷ Cs (IM)	10	10	10	10	0	<10	<10	NA	NA	-
Ba (IM)	13	14	14	13	6	<1	804	110	NA	U _M
Ce (IM)	189	218	185	197	9	11	98	NA	NA	U _B
Pr (IM)	<300	<300	<300	<300	-	<300	<300	NA	NA	-
Ta (IM)	20	8	8	12	56	<6	21	NA	NA	U _R
W (IM)	120	128	122	124	3	<6	7	NA	NA	-
Pt (IM)	<3	<3	<3	<3	-	<3	<3	NA	NA	U _M
Tl (IM)	<5	<5	<5	<5	-	<5	<5	NA	107	U _M
Rh (IM)	35	44	29	36	21	<0.4	<0.4	39	NA	U _R U _M
Pu (IM)	<0.14	<0.14	<0.14	<0.14	-	<0.14	<0.14	NA	NA	U _M
Y (IM)	35	62	69	55	33	<5	<5	NA	NA	U _R
Bi (IM)	<0.89	<0.89	<0.89	<0.89	-	<0.89	<0.89	NA	NA	U _M
Nd (IM)	392	443	380	405	8	<12	27	NA	NA	U _M

~ One Value

QC Flags: none - meets all QC

U_B - blank exceeds 5% of sample concentration

U_E - value less than 10x the DL

N/M- not measured

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

NA - not applicable

Table 6-6. Composition of the Microwave Digested As-Received 241-AN-107 Centrifuged Solids (page 4 of 4)

Radionuclides	1 st Replicate (mCi/kg)	2 nd Replicate (mCi/kg)	3 rd Replicate (mCi/kg)	Average (mCi/kg)	%RSD	Blank (mCi/kg)	Glass Std. (mg/kg)	LCS % Recovery	MS % Recovery	QC Flag
⁶⁰ Co (GS)	4.54E-02	5.23E-02	4.44E-02	4.74E-02	9.0	<5.75E-04	<5.53E-04	NA	NA	-
¹²⁶ Sb/ ¹²⁶ Sn (GS)	<1.29E-03	<1.39E-03	<1.46E-03	<1.38E-03	-	<4.71E-04	<4.38E-04	NA	NA	U _M
¹²⁵ Sb (GS)	<3.76E-03	<4.08E-03	<4.20E-03	<4.01E-03	-	<1.30E-03	<1.33E-03	NA	NA	U _M
¹⁵² Eu (GS)	<1.07E-02	<1.22E-02	<1.23E-02	<1.17E-02	-	<4.07E-03	<4.14E-03	NA	NA	U _M
¹⁵⁴ Eu (GS)	2.02E-01	2.57E-01	2.02E-01	2.20E-01	14	<7.19E-04	<6.82E-04	NA	NA	-
¹⁵⁵ Eu (GS)	1.15E-01	1.41E-01	1.10E-01	1.22E-01	14	<9.10E-04	<6.35E-04	NA	NA	-
²³¹ Pa (GS)	<2.97E-03	<4.89E-02	<5.21E-02	<3.47E-02	-	<1.61E-02	<1.53E-02	NA	NA	-
⁷⁹ Se (SL)	<7.61E-04	<8.14E-04	<3.42E-03	<1.67E-03	-	<1.57E-03	<7.25E-03	NA	NA	U _M
²⁴¹ Am (SG)	6.82E-01	5.28E-01	4.66E-01	5.59E-01	20	2.23E-03	2.45E-03	NA	NA	U _R
²⁴³ Am (SA)	<3.72E-03	<3.32E-03	<2.94E-03	<3.33E-03	-	<1.36E-03	<2.72E-02	NA	NA	-
²⁴⁴ Cm (SA)	4.85E-02	3.54E-02	3.14E-02	3.84E-02	23	1.49E-02	2.07E-02	NA	NA	U _B U _R
²⁴² Cm (SA)	3.52E-03	2.57E-03	3.00E-03	3.03E-03	16	<1.31E-04	<5.81E-05	NA	NA	U _M U _R
⁵⁹ Ni (SL)	2.26E-03	3.47E-03	2.98E-03	2.90E-03	21	<3.67E-03	<1.21E-03	NA	NA	U _M U _R
⁶³ Ni (SL)	7.84E-01	9.75E-01	7.64E-01	8.41E-01	14	<1.54E-03	<1.81E-03	NA	NA	-
Gross Alpha (AC)	7.07E-01	1.26E+00	1.34E+00	1.10E+00	31	<1.18E-01	<2.03E-01	96.8	92.4	U _R
³ H (SL)	<1.22E-02	<1.23E-02	<1.47E-02	<1.30E-02	-	<1.21E-02	<2.52E-02	75.8	94.6	U _L U _M
¹³⁷ Cs (GS)	1.99E+02	2.07E+02	1.76E+02	1.94E+02	8	<7.59E+00	<8.56E+00	NA	NA	-
⁹⁰ Sr (SL)	6.44E+01	9.10E+01	8.33E+01	7.96E+01	17	<1.58E-01	<1.67E-01	94.95	91.23	U _R
^{239/240} Pu (SA)	1.14E-02	8.15E-03	5.95E-03	8.48E-03	32	7.04E-03	1.03E-03	NA	NA	U _R U _M U _B
²³⁸ Pu (SA)	1.02E-02	1.23E-02	4.41E-03	8.95E-03	46	3.59E-03	2.14E-02	NA	NA	U _R U _M U _B
²⁴¹ Pu (SA)	3.46E-02	4.30E-02	2.69E-02	3.48E-02	23	<2.61E-02	<7.21E-03	NA	NA	U _R
¹⁴ C (SL)	3.87E-04	3.64E-04	2.25E-04	3.25E-04	27	<9.01E-06	-	75.2	96.2	U _L U _M

QC Flags: none - meets all QC
U_B - blank exceeds 5% of sample concentration
U_E - value less than 10x the DL
N/M- not measured

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
NA - not applicable

Table 6-7. Composition of the Aqua-Regia Digested As-Received 241-AN-107 Centrifuged Solids

Analyte	1 st Replicate (mg/kg)	2 nd Replicate (mg/kg)	3 rd Replicate (mg/kg)	Average (mg/kg)	%RSD	Blank (mg/kg)	Glass Std. (mg/kg)	LCS % Recovery	MS % Recovery	QC Flag
Ag (IE)	<1.09	<2.05	<1.07	<1.40	-	<1.09	6.13	102	NA	U _M
Al (IE)	147	122	93	121	22	23	21300	100	NA	U _B U _M U _R
B (IE)	<3.8	<7.16	<3.73	<4.90	-	<3.8	23700	91	NA	-
Ba (IE)	<2.58	<4.86	<2.53	<3.32	-	<2.58	758	100	105	U _M
Be* (IE)	0.155	<0.256	0.221	0.19	25	0.197	20.6	101	NA	U _E U _M
Ca (IE)	209	186	228	208	10	25	8940	100	NA	U _B
Cd (IE)	9	8	15	11	36	<0.57	18.9	100	105	U _R
Cr (IE)	26	28	45	33	32	3	573	99	101	U _B U _R
Cu (IE)	8	9	10	9	11	11	41.2	99	NA	U _E U _M U _B
Fe (IE)	575	310	464	450	30	30	88200	100	100	U _B U _R
Li (IE)	<5.83	<11	<5.73	<7.52	-	<5.83	13500	95	NA	U _M
Mg ~ (IE)	14	<1.36	<0.706	14	-	<0.719	4710	101	NA	U _M
Mn (IE)	112	97	149	119	22	0.134	13400	100	NA	U _M U _R
Ni (IE)	75	97	136	102	30	3	7380	103	100	U _M U _R
P (IE)	77	86	129	97	29	23	746	102	NA	U _B U _M U _R
Pb (IE)	51	59	97	69	36	<4.33	14.5	99	NA	U _M U _R
S (IE)	393	458	756	536	36	<18.6	943	99	NA	U _R
Si ~ (IE)	4	<4.27	<2.22	4	-	4	5920	100	NA	U _E U _M
Sr (IE)	44	40	48	44	9	4	1900	101	NA	U _B
Ti (IE)	<1.04	<1.97	<1.03	<1.35	-	<1.04	5010	N/A	NA	U _M
U~ (IE)	80	<58.1	<30.2	80	-	<30.8	2290	99	NA	U _E U _M
V (IE)	<0.271	<0.512	<0.266	<0.350	-	<0.271	102	100	NA	U _M
Zn (IE)	3	6	8	6	44	5	184	99	NA	U _B U _R
Zr* (IE)	9	<5.83	15	12	34	<3.09	<9.99	100	NA	U _E U _M

Table 6-7. Composition of the Aqua-Regia Digested As-Received 241-AN-107 Centrifuged Solids (page 2 of 4)

Analyte	1 st Replicate (mg/kg)	2 nd Replicate (mg/kg)	3 rd Replicate (mg/kg)	Average (mg/kg)	%RSD	Blank (mg/kg)	Glass Std. (mg/kg)	LCS % Recovery	MS % Recovery	QC Flag
As (AA)	<5.55	<5.75	<5.45	<5.58	-	<5.55	<18	90	119	-
K (AA)	244	322	445	337	30	<29.22	20300	99	104	U _R
Na (AA)	105,000	116,000	165,000	128,667	25	<3481	196000	96	106	U _R
Se (AA)	<5.55	<5.75	<5.45	<5.58	-	<5.55	<18	119	115	U _M
Hg (AA)	<13.6	<14.1	<13.3	<13.67	-	<13.56	<43.8	96	104	-
Th (IM)	3.0	2.2	4.8	3.3	40	0.14	1.8	105	104	U _R U _M
²³³ U (IM)	0.014	<0.013	<0.013	0.014	-	<0.013	<0.013	NA	NA	U _M
²³⁴ U (IM)	<0.013	<0.013	<0.013	<0.013	-	<0.013	<0.013	NA	NA	U _M
²³⁵ U (IM)	0.43	0.23	0.20	0.29	43	0.017	0.08	101	NA	U _R U _B
²³⁶ U* (IM)	0.05	0.02	<0.013	0.03	59	<0.013	<0.013	NA	NA	U _E U _R
²³⁷ Np (IM)	0.07	0.03	0.04	0.04	49	<0.013	<0.013	NA	NA	U _E U _R
²³⁸ U (IM)	82	26	32	46.7	66	1.80	10.7	100	115	U _R
Total U (IM)	82.80	26.26	32.07	47.0	66	1.82	10.82	NA	NA	U _R
V (IM)	21	4	11	11.8	73	20	189	111	NA	U _R U _B
Co (IM)	1	1	2	1.3	26	<0.25	64	115	NA	U _R U _M
As (IM)	1	<0.46	1	0.9	40	1	8	112	NA	U _R U _M U _B
Se (IM)	<0.12	<0.12	<0.12	<0.12	-	<0.12	<0.12	105	NA	U _M
Rb (IM)	1.8	2.4	3.2	2.5	29	0.14	58.8	NA	105	U _R U _M U _B
Ru (IM)	4.9	3.8	8.4	5.7	42	<0.03	<0.03	NA	NA	U _R U _M
Mo (IM)	4.1	3.2	7.4	4.9	46	0.1	2.2	105	NA	U _R U _M
Pd (IM)	2.0	0.7	2.6	1.7	56	<0.03	0.9	NA	NA	U _R U _M
Sb (IM)	0.04	0.05	0.08	0.1	37	0.06	1.25	102	NA	U _R U _M U _B
Te (IM)	<0.08	<0.08	<0.08	<0.08	-	<0.08	<0.08	NA	NA	U _M
¹³³ Cs (IM)	1.44	1.92	2.64	2.0	30	<0.03	6	NA	110	U _R
¹³⁵ Cs (IM)	0.41	0.3	0.5	0.4	16	0.06	<0.03	NA	NA	U _R U _B
¹³⁷ Cs (IM)	0.91	1.16	1.60	1.2	28	0.11	<0.004	NA	NA	U _R U _B

Table 6-7. Composition of the Aqua-Regia Digested As-Received 241-AN-107 Centrifuged Solids (page 3 of 4)

Analyte	1 st Replicate (mg/kg)	2 nd Replicate (mg/kg)	3 rd Replicate (mg/kg)	Average (mg/kg)	%RSD	Blank (mg/kg)	Glass Std. (mg/kg)	LCS % Recovery	MS % Recovery	QC Flag
Ba (IM)	1	2	2	1.6	30	0.641	770	105	NA	U _R U _M U _B
Ce (IM)	6.0	6.9	10.6	7.8	31	0.073	9.2	NA	NA	U _R
Pr (IM)	3.3	4.1	6.2	4.5	33	0.037	0.9	NA	NA	U _M
Ta (IM)	<0.03	<0.03	<0.03	<0.03	-	<0.03	<0.03	NA	NA	U _M
W (IM)	25	7	45	25.6	76	0.399	2	136	136	U _S U _R
Pt (IM)	<0.3	<0.3	<0.3	<0.3	-	<0.3	<0.3	NA	NA	U _M
Tl (IM)	<0.2	<0.2	<0.2	<0.2	-	<0.2	<0.2	NA	NA	U _M
Rh (IM)	1.4	1.0	2.6	1.7	49	<0.04	<0.04	NA	NA	U _R U _M
Pu (IM)	0.24	0.09	0.23	0.2	44	<0.02	<0.02	NA	NA	U _R U _M
Y (IM)	1.689	1.913	2.558	2.1	22	0.018	5.783	NA	NA	U _R U _M
Bi (IM)	<0.3	<0.3	<0.3	<0.3	-	0.5	3.5	NA	NA	U _M U _B
Nd (IM)	15.5	19.0	28.8	21.1	33	0.3	4.5	NA	NA	U _R U _M

~ One Value

* Average of two data points

QC Flags: none - meets all QC

U_B - blank exceeds 5% of sample concentration

U_E - value less than 10x the DL

N/M- not measured

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

NA - not applicable

Table 6-7. Composition of the Aqua-Regia Digested As-Received 241-AN-107 Centrifuged Solids (page 4 of 4)

Radionuclides	1 st Replicate (mCi/kg)	2 nd Replicate (mCi/kg)	3 rd Replicate (mCi/kg)	Average (mCi/kg)	%RSD	Blank (mCi/kg)	Glass Std. (mg/kg)	LCS % Recovery	MS % Recovery	QC Flag
¹³⁷ Cs (GS)	4.49E+01	4.96E+01	7.34E+01	5.60E+01	27.4	<2.70E+00	<8.74E+00	NA	NA	U _R
³ H * (SL)	<6.04E-03	<6.26E-03	-	<6.15E-03	-	<6.05E-03	<1.15E-02	NA	NA	U _M
Gross Alpha (AC)	4.19E-01	6.04E-01	6.13E-01	5.45E-01	20.0	8.67E-02	<1.24E-01	NA	NA	U _R U _B
⁹⁰ Sr (SL)	1.97E+01	1.95E+01	3.04E+01	2.32E+01	26.8	1.68E-01	2.90E-01	NA	NA	U _M U _R
²⁴¹ Am (SG)	9.61E-02	1.21E-01	1.67E-01	1.28E-01	28.1	8.89E-03	5.15E-02	NA	NA	U _R U _B
²⁴³ Am (SA)	8.00E-04	5.25E-04	3.56E-04	5.60E-04	40.0	1.19E-04	<9.94E-04	NA	NA	U _R U _B
²⁴⁴ Cm (SA)	1.89E-02	2.20E-01	5.33E-02	9.74E-02	110.4	1.57E-01	4.13E-01	NA	NA	U _R U _B
²⁴² Cm (SA)	4.59E-04	6.80E-04	6.60E-04	6.00E-04	20.3	1.24E-04	1.37E-04	NA	NA	U _M U _R U _B
²³⁸ Pu (SA)	4.87E-02	1.91E-02	5.95E-03	2.46E-02	89.0	1.78E-02	<3.39E-02	NA	NA	U _R U _M U _B
^{239/240} Pu (SA)	9.91E-03	6.17E-03	8.60E-03	8.23E-03	23.1	4.24E-03	<4.55E-03	NA	NA	U _R U _M U _B
²⁴¹ Pu (SA)	4.91E-02	4.24E-02	1.74E-02	3.63E-02	46.0	<8.22E-03	<2.49E-02	NA	NA	U _R
¹²⁹ I (SG)	<6.98E-04	<5.81E-04	<7.21E-04	<6.67E-04	-	<4.32E-04	NA	NA	NA	U _M

* Average of two data points

QC Flags: none - meets all QC

U_B - blank exceeds 5% of sample concentration

U_E - value less than 10x the DL

N/M- not measured

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

NA - not applicable

Table 6-8. Composition of the Water Leach Digested As-Received 241-AN-107 Centrifuged Solids

Analyte	1 st Replicate (mg/kg)	2 nd Replicate (mg/kg)	3 rd Replicate (mg/kg)	Average (mg/kg)	%RSD	Blank (mg/kg)	LCS % Recovery	MS % Recovery	QC Flag
TIC (A)	18600	18500	19700	18933	4	0	101	102	-
TOC (Diff)	29500	30600	28700	29600	3	495	101	99.3	-
F (IC)	2000	1580	1828	1803	12	<2500	95	112	U _M
CHO ₂ (IC)	5000	4310	5091	4801	9	<12500	90	109	-
Cl (IC)	<2500	<2873	<2611	<2662	-	<2500	99	105	-
NO ₂ (IC)	32875	26868	31854	30532	11	<12500	98	116	-
NO ₃ (IC)	122750	98994	119582	113775	11	<12500	104	118	-
PO ₄ (IC)	<12500	<14368	<13055	<13308	-	<12500	104	106	-
SO ₄ (IC)	4250	3592	4178	4006	9	<6250	102	106	-
C ₂ O ₄ (IC)	3625	3736	4178	3846	8	<12500	99	108	-
Br (IC)	<12500	<14368	<13055	<13308	-	<12500	103	107	-

QC Flags: none - meets all QC

U_B - blank exceeds 5% of sample concentration

U_E - value less than 10x the DL

N/M- not measured

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

NA - not applicable

7.0 COMPARISON OF THE AS-RECEIVED SUPERNATE 241-AN-107 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-AN-107 Envelope C sample meets all of the specification limits except for the TRU limit. The TRU concentration is slightly above the specification limit. The %RSDs were high on these measurements and the overall uncertainty indicates that it is somewhere close to the limit, i.e., +/- 25-50%. The sodium concentration is averaged between the values measured by ICP-ES and AA in Table 6-5.

Table 7-1. Comparison of As-Received 241-AN-107 Envelope C Filtered Supernate to Specification 7 Chemical Composition Limits

Analyte	Average Value from Table 6-5 (M)	QC Flag	Average Analyte to Na Molar Ratio	Specification 7, Envelope C Maximum Ratio	% of Maximum	Meets Specification 7
Al	1.13E-02	-	1.32E-03	2.50E-01	1	YES
Ba	6.55E-05	U _E	7.62E-06	1.00E-04	8	YES
Ca	1.41E-02	-	1.64E-03	4.00E-02	4	YES
Cd	6.41E-04	U _M	7.45E-05	4.00E-03	2	YES
Cl	3.93E-02	-	4.57E-03	3.70E-02	12	YES
Cr	3.67E-03	-	4.27E-04	6.90E-03	6	YES
F	1.75E-01	-	2.03E-02	9.10E-02	22	YES
Fe	3.34E-02	-	3.88E-03	1.00E-02	39	YES
Hg	<1.14E-05	-	<1.33E-06	1.40E-05	9	YES
K	3.98E-02	-	4.63E-03	1.80E-01	3	YES
La	2.55E-04	-	2.97E-05	8.30E-05	36	YES
Ni	9.90E-03	-	1.15E-03	3.00E-03	38	YES
NO ₂	1.32E+00	-	1.54E-01	3.80E-01	40	YES
NO ₃	3.63E+00	-	4.22E-01	8.00E-01	53	YES
Pb	2.22E-03	U _M	2.58E-04	6.80E-04	38	YES
PO ₄	9.96E-03	-	1.16E-03	3.80E-02	3	YES
SO ₄	8.83E-02	-	1.03E-02	2.00E-02	51	YES
TIC	2.21E+00	U _R	2.58E-01	3.00E-01	86	YES
TOC	2.77E+00	U _R	3.22E-01	5.00E-01	64	YES
U	5.29E-04	-	6.16E-05	1.20E-03	5	YES

1) The total uranium was calculated from the ICP-MS values for mass 235 and 238.

Table 7-2. Comparison of As-Received 241-AN-107 Filtered Supernate to Specification 7 Radionuclide Limits

Radio-nuclides	Average Value from Table 6-5 (M)	QC Flag	Average Analyte to Na Molar Ratio	Specification 7, Envelope C Maximum Ratio	% of Maximum	Meets Specification 7
TRU ¹	3.35E+07	U _R	3.90E+06	3.00E+06	130	NO
¹³⁷ Cs	1.24E+10	-	1.44E+09	4.30E+09	34	YES
⁹⁰ Sr	3.12E+09	U _S	3.62E+08	8.00E+08	45	YES
⁹⁹ Tc	3.85E+06	-	4.47E+05	7.10E+06	6	YES
⁶⁰ Co	3.02E+06	U _R	3.51E+05	3.70E+05	95	YES
¹⁵⁴ Eu	1.60E+07	U _R	1.86E+06	4.30E+06	43	YES

1) The TRU was calculated by summing the ²³⁸Pu, ^{239/240}Pu, ²⁴¹Am, and ²⁴⁴Cm alpha spectroscopy results.

2) The ⁹⁹Tc result is from ⁹⁹Tc total .

U_R Fails % RSD criteria

U_S Fails % recovery of Matrix Spike

8.0 GENERAL DESCRIPTION OF ANALYTICAL PROCEDURES

8.1 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, Cd, 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.

8.2 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. A 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.

8.3 FREE HYDROXIDE AND TOTAL BASE TITRATIONS

Total hydroxide (total base) was determined by titration to an inflection end point closest to pH 7.00. An 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. An 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.

8.4 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.

8.5 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.

8.6 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 was 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- d_8 as the LCS. The matrix spike was deuterated stearic- d_{35} acid which was compared to acid methyl ester. The IDA spike served as an internal standard.

8.7 TOTAL INORGANIC CARBON/TOTAL ORGANIC CARBON

Total carbon was determined by combustion at 780 °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.

8.8 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 ^{127}I , 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 recoveries 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
^{99}Tc	mass 99. Subject to interference when Ru is present in the sample.
^{127}I	mass 127. Value is unreliable due to memory effects.
^{133}Cs	mass 133
^{135}Cs	mass 135. Subject to interference when Ba is present in sample.
^{137}Cs	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
^{233}U	mass 233
^{234}U	mass 234
^{235}U	mass 235
^{236}U	mass 236
^{238}U	mass 238
^{237}Np	mass 237
^{239}Pu	mass 239
^{240}Pu	mass 240

ICP-MS determinations of radionuclides were reported in $\mu\text{g/g}$ of the supernatant. To convert these values to mCi/L as required by the Test Specification, the density of the supernate and the specific activity of each radionuclide were used. The specific activity for each radionuclide was obtained from "Integrated Data Base Report -1994: U.S. Spent Nuclear Fuel and Radioactive Waste Inventories, Projections, and Characteristics," Appendix B, pages 277 and 280, DOE/RW-0006, September, 1995, prepared by the Oak Ridge National Laboratory.

8.9 ALPHA COUNTING

Prior to the analysis, ^{137}Cs was removed from aliquots of the samples in order to reduce bias effects caused by large beta/alpha ratios. The ^{137}Cs 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.

8.10 GAMMA SPECTROMETRY

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

8.11 ^3H 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.

8.12 $^{99\text{m}}\text{Tc}$ ANALYSIS (FOR PERTECHNETATE FORM)

$^{99\text{m}}\text{Tc}$ tracers were generated initially by neutron irradiation of natural molybdenum using the SRTC CF^{252} Neutron Activation Analysis facility. ^{98}Mo was activated to ^{99}Mo , which subsequently beta decays to $^{99\text{m}}\text{Tc}$. The $^{99\text{m}}\text{Tc}$ was then extracted from the ^{99}Mo to form a $^{99\text{m}}\text{Tc}$ tracer.

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

Pertechnetate is the thermodynamically stable form of technetium in oxidizing environments. It is presumed to be the predominant chemical form of technetium in the aqueous phase of nuclear waste tanks, however significant amounts of technetium appear in a different chemical form. Total technetium accounts for pertechnetate and non-pertenetate forms present.

8.13 ^{90}Sr ANALYSIS

An aliquot of each sample was analyzed for ^{90}Sr using an Eichrom Sr-Spec based extraction procedure. A ^{90}Sr spiked blank was analyzed with the sample batch to establish $^{90}\text{Sr}/^{90}\text{Y}$ 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 ^{90}Sr 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 $^{90}\text{Sr}/^{90}\text{Y}$ 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.

8.14 ^{79}Se 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 ^{79}Se 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 ^{79}Se activities.

8.15 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 ^{238}Pu 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 ^{241}Pu 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 $^{239/240}\text{Pu}$ alpha peak were yielded using the ^{238}Pu recoveries from the ^{238}Pu traced sample separation. The ratio of the $^{239/240}\text{Pu}$ to the ^{238}Pu in the sample was obtained from the alpha spectroscopy analysis of the non-spiked sample. That ratio was applied to the determined $^{239/240}\text{Pu}$ value to determine the ^{238}Pu activity in the sample.

The sample aliquot dedicated to the ^{241}Pu analysis was added to liquid scintillation cocktail following the separation and analyzed for both ^{241}Pu and gross Pu-alpha constituents. The ratio of ^{241}Pu to total Pu alpha was determined and applied to the results from the plates in order to determine a ^{241}Pu concentration.

8.16 ^{129}I ANALYSIS

An aliquot of each sample was spiked with stable iodide, was Cs-stripped using AMP-1 resin, 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 ^{129}I 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 ^{129}I recoveries. Uncertainties provided are 1 sigma.

8.17 Am/Cm ANALYSIS

Aliquots of sample were run through an 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 ^{243}Am for yielding purposes, one sample unspiked to correct for any ^{243}Am that may be present in the samples. The Am/Cm sample mount was analyzed by alpha spectroscopy for $^{244/243}\text{Cm}$ and ^{242}Cm and by low energy gamma spectrometry for ^{241}Am . The ^{241}Am activity in the samples was high enough to swamp out the ^{243}Am spikes' alpha spectrum, as a result, the alpha results of $^{244}\text{Cm}/^{242}\text{Cm}$ were quantified by ratioing their alpha peaks to that of the ^{241}Am which had been previously quantified in the gamma measurement. ^{243}Am values were quantified off a low energy gamma analysis of the mount containing no ^{243}Am spike, and using that $^{243}\text{Am}/^{241}\text{Am}$ ratio applied to the quantified ^{241}Am activity. A laboratory blank sample was run through all of the analyses with the batch of samples.

8.18 ^{14}C 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 ^{14}C . Each sample was run through the process in duplicate. A blank solution, spiked with a ^{14}C standard, was run (in duplicate) in parallel with the samples to determine ^{14}C recoveries. The average recoveries were applied to the sample results to quantify the ^{14}C concentrations. A second blank solution, spiked with the ^{14}C standard was also run in duplicate through the process to serve as the laboratory control sample. One customer sample was spiked with some ^{14}C (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.

8.19 ^{59}Ni ANALYSIS

Aliquots of aqueous sample were spiked with nickel carrier and then concentrated. The treated sample aliquots were then subjected to a Ni-DMG separation. Aliquots of the resulting separated nickel solution were analyzed by liquid scintillation to determine ^{63}Ni , x-ray spectroscopy to determine ^{59}Ni , and ICP-ES to determine the concentrations of nickel carrier recovered. The concentrations of nickel carrier recovered were used to yield the ^{59}Ni and ^{63}Ni measurements. A laboratory blank was also run through the process.

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9.0 REFERENCES

1. Arakali, A., Tank 241-AN-107 Sample Compositing, Homogenization and Analyses Test Specification 24590-WTP-TSP-RT-02-010, Rev. 0, June 03, 2002
2. Martin, K., Task Technical and Quality Assurance Plan for Compositing, Homogenizing, and Characterizing Samples from Hanford Tank 241-AN-107, WSRC-TR-2002-00185, Revision 1, August 12, 2002, 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. Smith, G. L., "Characterization of Analytical Reference Glass-1 (ARG-1)," PNL-8992, December 1993
5. Gregg J. Lumetta and F. Vaugh Hoopes., Washing of the AN-107 Entrained Solids. BNFL-RPT-007, Rev. 0., August 1999. Battelle Pacific Northwest Division, Richland, Washington.

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APPENDIX A. CHAIN OF CUSTODY DOCUMENTATION

CHAIN OF CUSTODY/SAMPLE ANALYSIS REQUEST							C.O.C. No.	
							Page <u>1</u> of <u>1</u>	
Collector Tank Farms			Contact/Requestor Christina Caprio			Telephone No. 509-372-2553 MSIN T6-12 FAX 509-373-4671		
SAF No. N/A			Sample Origin Tank 241-AN-107			Purchase Order/Charge Code CACN: 116294		
Project Title AN107 ICD23 (241-AN-107 1998 & 2001 Sample Shipment)			Logbook No. N/A			Ice Chest No. Temp. N/A N/A		
Shipped To (Lab) Savannah River Technology Center (SRTC)			Method of Shipment PAS-1 shipping cask			Bill of Lading/Air Bill No. N/A		
Protocol N/A			Data Turnaround N/A			Offsite Property No. N/A		
Sample No.	Lab ID	*	Date	Time	No./Type Container	Sample Analysis		Preservative
7AN-01-09	S02T000413	L	5/6/02	1135	1/500 mL glass	Net Wt. 648 g <i>Pig # 17</i>		N/A
						NOTE: This sample is not necessarily representative		
						of the waste in the tanks that will be shipped to the		
						Vitrification Plant		
POSSIBLE SAMPLE HAZARDS/REMARKS (List all known wastes) MSDS <input type="checkbox"/> Yes <input checked="" type="checkbox"/> No						SPECIAL INSTRUCTIONS		
Copies of original chain of custodies for parent samples and sample breakdown/composite preparation information are provided as attached paperwork.						Hold Time		
						Ship to: Bldg. 773A, East Wing Truck Dock Aiken, SC 29802 ATTN: Debra Fields, 803-725-5849 (p) Bill Wilmarth, 803-725-1727 (p), 803-725-PAGE, pager extension: 11481; Lynda Wingard, 803-725-7097 (p), 803-725-PAGE, ext. 11880		
Relinquished By	Print	Sign	Date/Time	Received By	Print	Sign	Date/Time	Matrix*
	<i>RT Steels</i>	<i>RT Steels</i>	5/9/02 1145	<i>JAMES L. BARNHART</i>	<i>JAMES L. BARNHART</i>	<i>JAMES L. BARNHART</i>	5/9/02 1145	S = Soil DS = Drum Solids
	<i>JAMES L. BARNHART</i>	<i>JAMES L. BARNHART</i>	5/13/02	<i>Samuel McDuff</i>	<i>Samuel McDuff</i>	<i>Samuel McDuff</i>	5/13/02 9:45	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
FINAL SAMPLE DISPOSITION	Disposal Method (e.g., Return to customer, per lab procedure, used in process)					Disposed By		Date/Time

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 (04/98)

CHAIN OF CUSTODY/SAMPLE ANALYSIS REQUEST							C.O.C. No. 0.	
							Page 1 of 1	
Collector Tank Farms			Contact/Requestor Christina Caprio			Telephone No. MSIN FAX 509-372-2553 T6-12 509-378-1878		
SAF No. N/A			Sample Origin Tank 241-AN-107			Purchase Order/Charge Code CACN: 116294		
Project Title AN107 ICD23 (241-AN-107 1998 & 2001 Sample Shipment)			Logbook No. N/A			Ice Chest No. Temp. N/A N/A		
Shipped To (Lab) Savannah River Technology Center (SRTC)			Method of Shipment Hedge Hog/Barney Box			Bill of Lading/Air Bill No. N/A		
Protocol N/A			Data Turnaround N/A			Offsite Property No. N/A		
Sample No.	Lab ID	*	Date	Time	No./Type Container	Sample Analysis		Preservative
7AN-01-02C	S02T000933	L			1/125 mL glass	Net Wt. 161.1g		N/A
						NOTE: This sample is not necessarily representative		
						of the waste in the tanks that will be shipped to the		
						Vitrification Plant		
						Jar # (# on the bottle): 933		
POSSIBLE SAMPLE HAZARDS/REMARKS (List all known wastes) MSDS <input type="checkbox"/> Yes <input checked="" type="checkbox"/> No Copies of original chain of custodies for parent samples and sample breakdown/composite preparation information are provided as attached paperwork. The field numbers are a subset of the Sample No. above (exclude the last character off the sample number). 7AN-01-02C is 1/4 of 7AN-01-02, which was done for shipping purposes.						SPECIAL INSTRUCTIONS Ship to: Bldg. 773A, East Wing Truck Dock Aiken, SC 29802 ATTN: Debra Fields, 803-725-5849 (p) Bill Wilmarth, 803-725-1727 (p), 803-725-PAGE, pager extension: 11481; Lynda Wingard, 803-725-7097 (p), 803-725-PAGE, ext. 11880		
Relinquished By Print Sign		Date/Time		Received By Print Sign		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
R. Steele R. Steele		5/15/02 11:43		M.R. LATHAN M.R. Lathon		5/15/02 11:43		
M.R. LATHAN M.R. Lathon		5/15/02 4:15		Kim Portlock Kim Portlock		5/15/02 4:17		
Sharon Smith		5/21/02 10:00		J. McC. (J. McC.)		5/21/02 11:00		
Relinquished By		Date/Time		Received By		Date/Time		
FINAL SAMPLE DISPOSITION		Disposal Method (e.g., Return to customer, per lab procedure, used in process)				Disposed By		Date/Time

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 (04/98)

CHAIN OF CUSTODY/SAMPLE ANALYSIS REQUEST							C.O.C. No.	
							Page <u>1</u> of <u>1</u>	
Collector Tank Farms			Contact/Requestor Christina Caprio			Telephone No. MSIN 509-372-2553 FAX T6-12 509-378-1878		
SAF No. N/A			Sample Origin Tank 241-AN-107			Purchase Order/Charge Code CACN: 116294		
Project Title AN107 ICD23 (241-AN-107 1998 & 2001 Sample Shipment)			Logbook No. N/A			Ice Chest No. Temp. N/A N/A		
Shipped To (Lab) Savannah River Technology Center (SRTC)			Method of Shipment Hedge Hog/Barney Box			Bill of Lading/Air Bill No. N/A		
Protocol N/A			Data Turnaround N/A			Offsite Property No. N/A		
Sample No.	Lab ID	*	Date	Time	No./Type Container	Sample Analysis		Preservative
1998COMP1	S02T000935	L			1/125 mL glass	Net Wt. 157.6g		N/A
						NOTE: This sample is not necessarily representative		
						of the waste in the tanks that will be shipped to the		
						Vitrification Plant		
						Jar # (# on the bottle): 935		
POSSIBLE SAMPLE HAZARDS/REMARKS (List all known wastes) MSDS <input type="checkbox"/> Yes <input checked="" type="checkbox"/> No					SPECIAL INSTRUCTIONS			
Copies of original chain of custodies for parent samples and sample breakdown/composite preparation information are provided as attached paperwork. The field numbers are not defined on the COC because there were 11 items included in the composite. See the attached sample breakdown document in the field sample# column for that information. 1998COMP1 is 1/6 of the total composite, which was done for shipping purposes.					Ship to: Bldg. 773A, East Wing Truck Dock Aiken, SC 29802 ATTN: Debra Fields, 803-725-5849 (p) Bill Wilmarth, 803-725-1727 (p), 803-725-PAGE, pager extension: 11481; Lynda Wingard, 803-725-7097 (p), 803-725-PAGE, ext. 11880			
Relinquished By		Print	Sign	Date/Time	Received By		Print	Sign
AT Steele				5/15/02 11:35	M.R. LATHAN			
Relinquished By		Print	Sign	Date/Time	Received By		Print	Sign
M.R. LATHAN				5/15/02 4:15	Kim Postlock			
Relinquished By		Print	Sign	Date/Time	Received By		Print	Sign
Sharon Smith				5/21/02 10:00	M.R. LATHAN			
Relinquished By		Print	Sign	Date/Time	Received By		Print	Sign
FINAL SAMPLE DISPOSITION		Disposal Method (e.g., Return to customer, per lab procedure, used in process)				Disposed By		
						Date/Time		

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 (04/98)

CHAIN OF CUSTODY/SAMPLE ANALYSIS REQUEST							C.O.C. No.	
							Page <u>1</u> of <u>1</u>	
Collector Tank Farms			Contact/Requestor Christina Caprio			Telephone No. 509-372-2553 MSIN T6-12 FAX 509-378-1878		
SAF No. N/A			Sample Origin Tank 241-AN-107			Purchase Order/Charge Code CACN: 116294		
Project Title AN107 ICD23 (241-AN-107 1998 & 2001 Sample Shipment)			Logbook No. N/A			Ice Chest No. Temp. N/A N/A		
Shipped To (Lab) Savannah River Technology Center (SRTC)			Method of Shipment Hedge Hog/Barney Box			Bill of Lading/Air Bill No. N/A		
Protocol N/A			Data Turnaround N/A			Offsite Property No. N/A		
Sample No.	Lab ID	*	Date	Time	No./Type Container	Sample Analysis		Preservative
7AN-01-03A	S02T000927	L			1/125 mL glass	Net Wt. 155.8g		N/A
						NOTE: This sample is not necessarily representative		
						of the waste in the tanks that will be shipped to the		
						Vitrification Plant		
						Jar # (# on the bottle): 927		
POSSIBLE SAMPLE HAZARDS/REMARKS (List all known wastes) MSDS <input type="checkbox"/> Yes <input checked="" type="checkbox"/> No					SPECIAL INSTRUCTIONS			
Copies of original chain of custodies for parent samples and sample breakdown/composite preparation information are provided as attached paperwork. The field numbers are a subset of the Sample No. above (exclude the last character off the sample number). 7AN-01-03A is 1/4 of 7AN-01-03, which was done for shipping purposes.					Ship to: Bldg. 773A, East Wing Truck Dock Aiken, SC 29802 ATTN: Debra Fields, 803-725-5849 (p) Bill Wilmarth, 803-725-1727 (p), 803-725-PAGE, pager extension: 11481; Lynda Wingard, 803-725-7097 (p), 803-725-PAGE, ext. 11880			
Relinquished By		Print	Sign	Date/Time	Received By		Print	Sign
R. STEELE		R. Steele		5/15/02 11:45	M.R. LATHAN		M.R. Lathan	5/15/02 11:45
Relinquished By				Date/Time	Received By			Date/Time
M.R. LATHAN		M.R. Lathan		5/15/02 4:15	Kim Portlock		Kim Portlock	5/15/02 4:17
Relinquished By				Date/Time	Received By			Date/Time
Shaun Smith				5/21/02 10:00	R. Steele		R. Steele	5/21/02 11:00
Relinquished By				Date/Time	Received By			Date/Time
FINAL SAMPLE DISPOSITION		Disposal Method (e.g., Return to customer, per lab procedure, used in process)				Disposed By		Date/Time

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 (04/98)

CHAIN OF CUSTODY/SAMPLE ANALYSIS REQUEST							C.O.C. No. 0.	
							Page 1 of 1	
Collector Tank Farms			Contact/Requestor Christina Caprio			Telephone No. 509-372-2553 MSIN T6-12 FAX 509-378-1878		
SAF No. N/A			Sample Origin Tank 241-AN-107			Purchase Order/Charge Code CACN: 116294		
Project Title AN107 ICD23 (241-AN-107 1998 & 2001 Sample Shipment)			Logbook No. N/A			Ice Chest No. Temp. N/A		
Shipped To (Lab) Savannah River Technology Center (SRTC)			Method of Shipment Hedge Hog/Barney Box			Bill of Lading/Air Bill No. N/A		
Protocol N/A			Data Turnaround N/A			Offsite Property No. N/A		
Sample No.	Lab ID	*	Date	Time	No./Type Container	Sample Analysis		Preservative
7AN-01-02A	S02T000931	L			1/125 mL glass	Net Wt. 137.5 g		N/A
						NOTE: This sample is not necessarily representative		
						of the waste in the tanks that will be shipped to the		
						Vitrification Plant		
						Jar # (# on the bottle): 931		
POSSIBLE SAMPLE HAZARDS/REMARKS (List all known wastes) MSDS <input type="checkbox"/> Yes <input checked="" type="checkbox"/> No					SPECIAL INSTRUCTIONS			
Copies of original chain of custodies for parent samples and sample breakdown/composite preparation information are provided as attached paperwork. The field numbers are a subset of the Sample No. above (exclude the last character off the sample number). 7AN-01-02A is 1/4 of 7AN-01-02, which was done for shipping purposes.					Hold Time Ship to: Bldg. 773A, East Wing Truck Dock Aiken, SC 29802 ATTN: Debra Fields, 803-725-5849 (p) Bill Wilmarth, 803-725-1727 (p), 803-725-PAGE, pager extension: 11481; Lynda Wingard, 803-725-7097 (p), 803-725-PAGE, ext. 11880			
Relinquished By	Print	Sign	Date/Time	Received By	Print	Sign	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
Relinquished By			Date/Time	Received By			Date/Time	
Relinquished By			Date/Time	Received By			Date/Time	
Relinquished By			Date/Time	Received By			Date/Time	
FINAL SAMPLE DISPOSITION		Disposal Method (e.g., Return to customer, per lab procedure, used in process)			Disposed By		Date/Time	

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 (04/98)

WSRC-TR-2003-00210, REVISION 0
SRT-RPP-2003-00091, REVISION 0

BC-6000-828 (04/98)

WSRC-TR-2003-00210, REVISION 0
SRT-RPP-2003-00091, REVISION 0

BC-6000-828 (04/98)

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

BC-6000-828 (04/98)

CHAIN OF CUSTODY/SAMPLE ANALYSIS REQUEST							C.O.C. No. 0.	
							Page 1 of 1	
Collector Tank Farms			Contact/Requestor Christina Caprio			Telephone No. 509-372-2553 MSIN T6-12 FAX 509-378-1878		
SAF No. N/A			Sample Origin Tank 241-AN-107			Purchase Order/Charge Code CACN: 116294		
Project Title AN107 ICD23 (241-AN-107 1998 & 2001 Sample Shipment)			Logbook No. N/A			Ice Chest No. N/A Temp. N/A		
Shipped To (Lab) Savannah River Technology Center (SRTC)			Method of Shipment Hedge Hog/Barney Box			Bill of Lading/Air Bill No. N/A		
Protocol N/A			Data Turnaround N/A			Offsite Property No. N/A		
Sample No.	Lab ID	*	Date	Time	No./Type Container	Sample Analysis		Preservative
7AN-01-03D	S02T000930	L			1/125 mL glass	Net Wt. 168.7g		N/A
						NOTE: This sample is not necessarily representative		
						of the waste in the tanks that will be shipped to the		
						Vitrification Plant		
						Jar # (# on the bottle): 930		
POSSIBLE SAMPLE HAZARDS/REMARKS (List all known wastes) MSDS <input type="checkbox"/> Yes <input checked="" type="checkbox"/> No						SPECIAL INSTRUCTIONS		
Copies of original chain of custodies for parent samples and sample breakdown/composite preparation information are provided as attached paperwork. The field numbers are a subset of the Sample No. above (exclude the last character off the sample number). 7AN-01-03D is 1/4 of 7AN-01-03, which was done for shipping purposes.						Hold Time		
						Ship to: Bldg. 773A, East Wing Truck Dock Aiken, SC 29802 ATTN. Debra Fields, 803-725-5849 (p) Bill Wilmarth, 803-725-1727 (p), 803-725-PAGE, pager extension: 11481; Lynda Wingard, 803-725-7097 (p), 803-725-PAGE, ext. 11880		
Relinquished By	Print	Sign	Date/Time	Received By	Print	Sign	Date/Time	Matrix*
	R. STEELE	[Signature]	5/15/02 11:49		M.R. LATHAN	[Signature]	5/15/02 11:49	S = Soil DS = Drum Solids
Relinquished By	M.R. LATHAN	[Signature]	5/15/02 4:15	Received By	Kim Portlock	[Signature]	5/15/02 11:17	SE = Sediment DL = Drum Liquids
Relinquished By	Sharon Smith	[Signature]	5/21/02 10:00	Received By	[Signature]	[Signature]	5/31/02 11:00	SO = Solid T = Tissue
Relinquished By				Received By				SL = Sludge WI = Wipe
								W = Water L = Liquid
								O = Oil V = Vegetation
								A = Air X = Other
FINAL SAMPLE DISPOSITION	Disposal Method (e.g., Return to customer, per lab procedure, used in process)				Disposed By		Date/Time	

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 (04/98)

WSRC-TR-2003-00210, REVISION 0
SRT-RPP-2003-00091, REVISION 0

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 (04/98)

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

WSRC-TR-2003-00210, REVISION 0
SRT-RPP-2003-00091, REVISION 0

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

WSRC-TR-2003-00210, REVISION 0
SRT-RPP-2003-00091, REVISION 0

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 (04/98)

WSRC-TR-2003-00210, REVISION 0
SRT-RPP-2003-00091, REVISION 0

DISTRIBUTION: White - Remain with Samples Color - Customer

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BC-6000-828 (04/98)

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APPENDIX B. MICROGRAPHS OF AN-107 DRIED SOLIDS

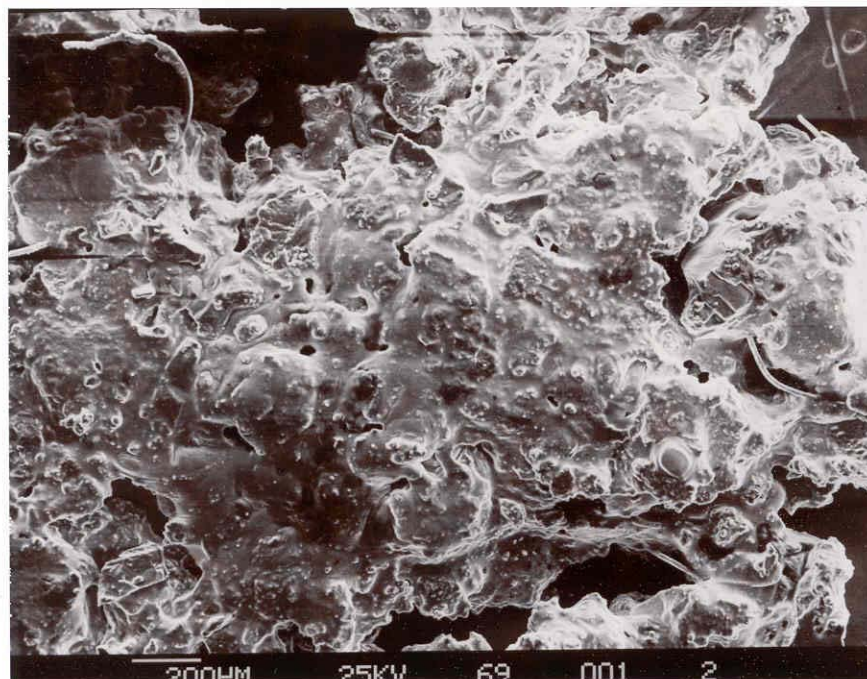


Figure B- 1. AN-107 Solid 1 50x Magnification

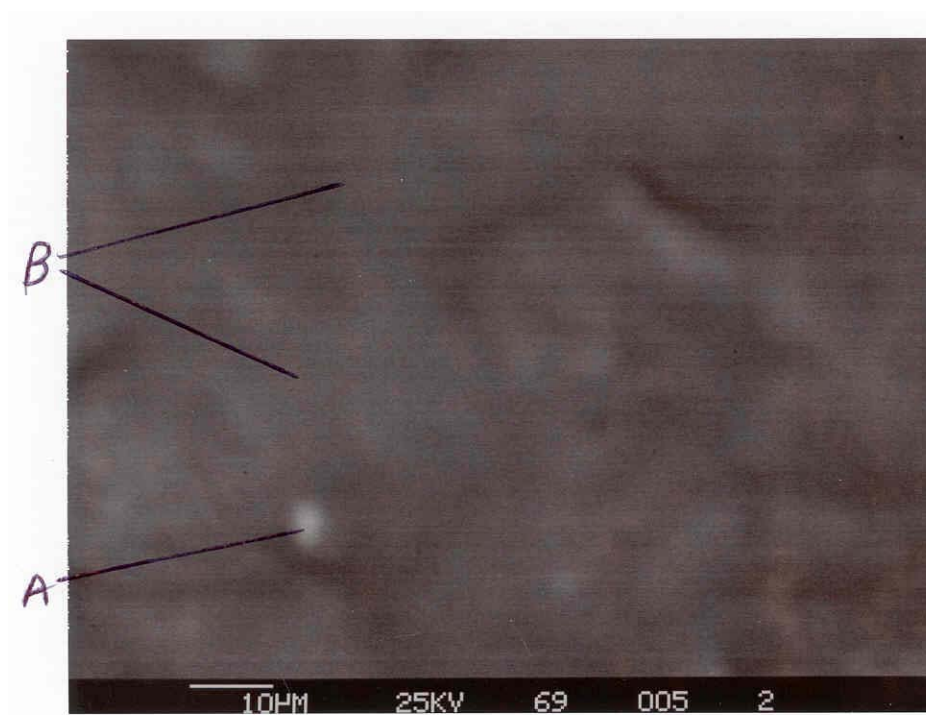


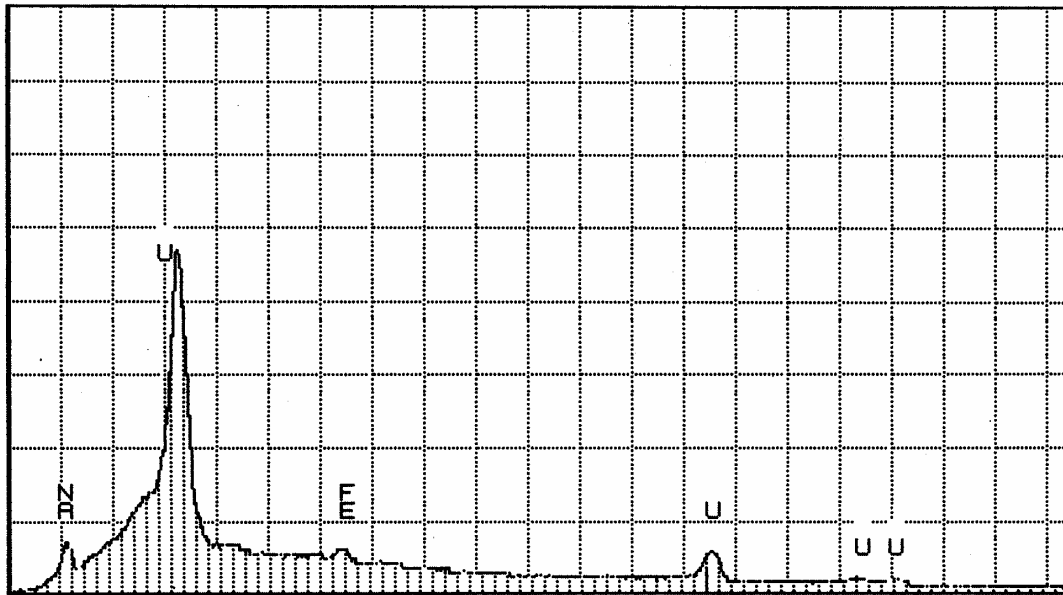
Figure B- 2. AN-107 Solid 1 1000x Magnification

TN-5502 WSRC CSEM.

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ROI (0) 0.040: 0.060=0/sec



0.000 ES-99 VFS = 8192 20.480

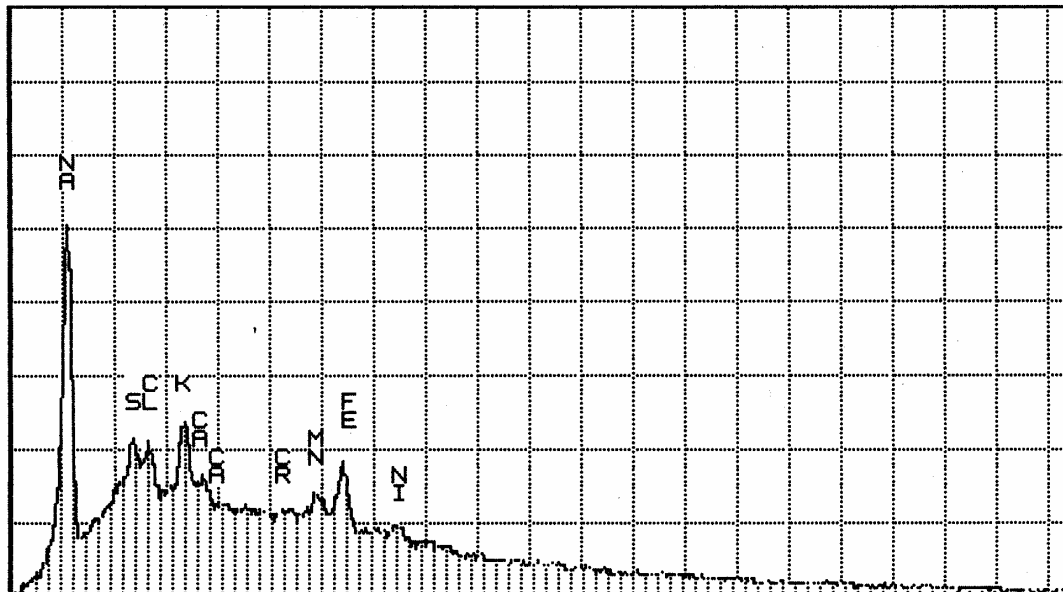
30 185569 AN107 SOLID1 MARTIN PHOTO#5 SPOT-A

TN-5502 WSRC CSEM.

MON 30-SEP-02 10:33

Cursor: 0.000keV = 0

ROI (0) 0.040: 0.060=0/sec



0.000 VFS = 2048 20.480

30 185569 AN107 SOLID1 MARTIN PHOTO#5 SPOT-B

Figure B- 3. AN-107 Solid 1 Spot-A (top) and Spot-B (bottom)

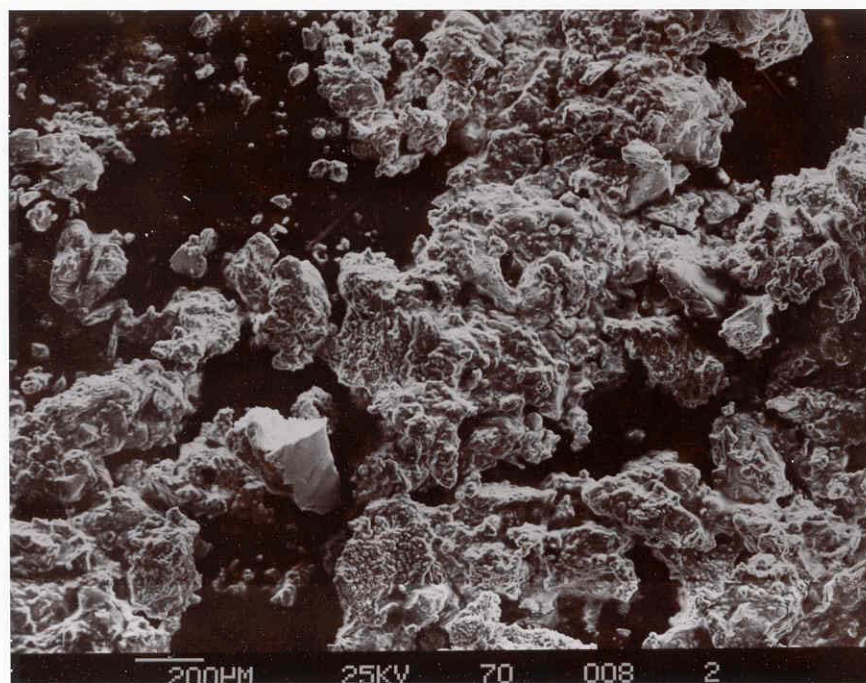


Figure B- 4. AN-107 Solid 2 50x Magnification

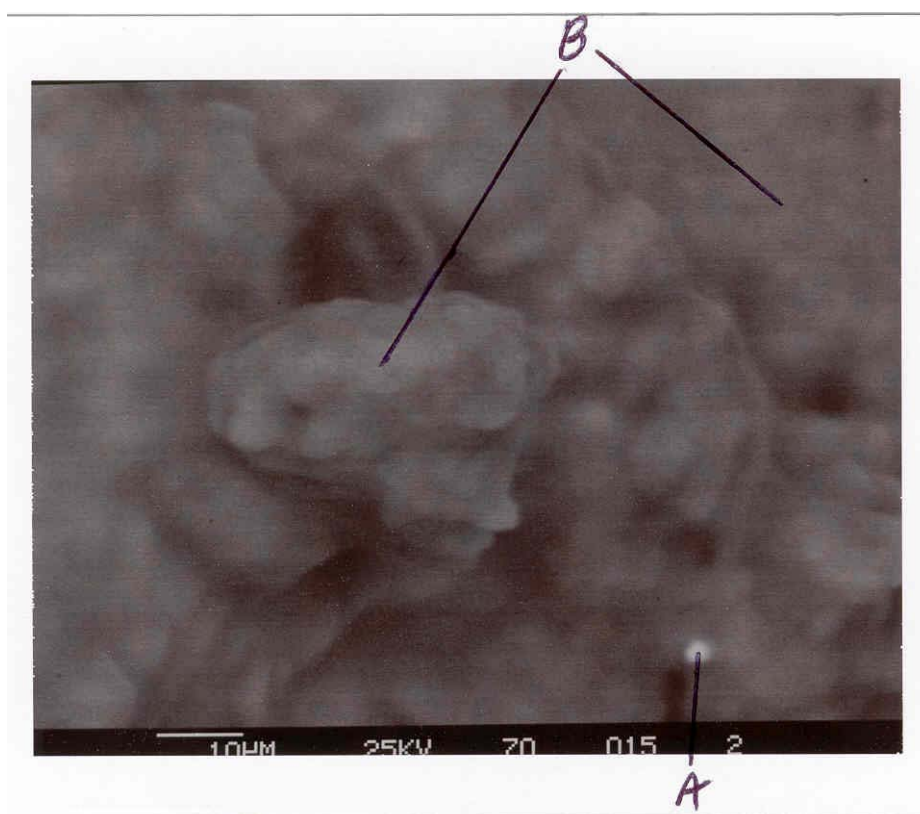


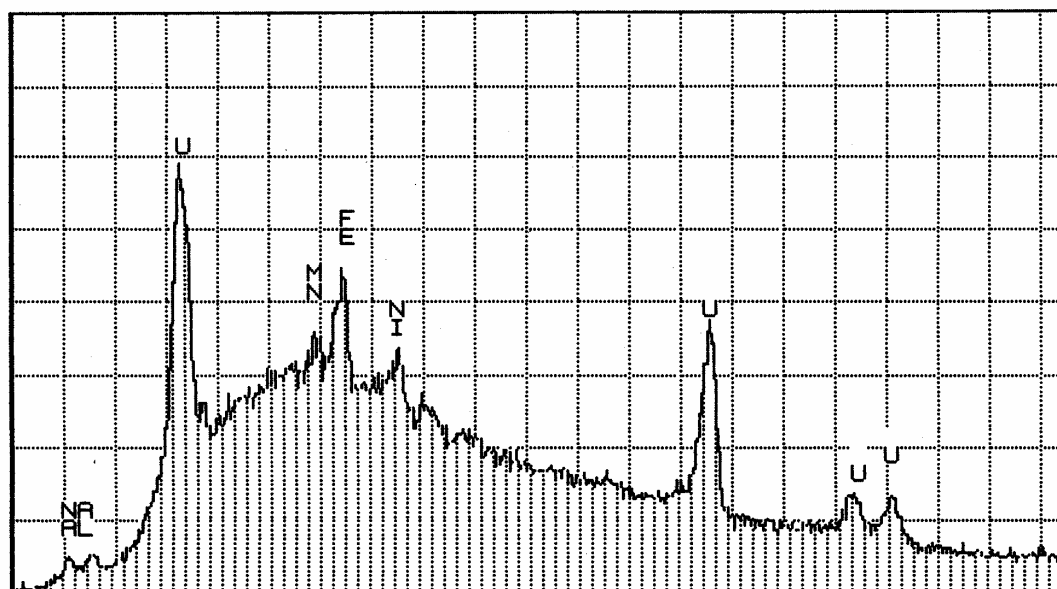
Figure B- 5. AN-107 Solid 2 1000x Magnification

TN-5502 WSRC CSEM.

MON 30-SEP-02 14:26

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ROI (0) 0.040: 0.060=0/sec



0.000

VFS = 1024 20.480

30

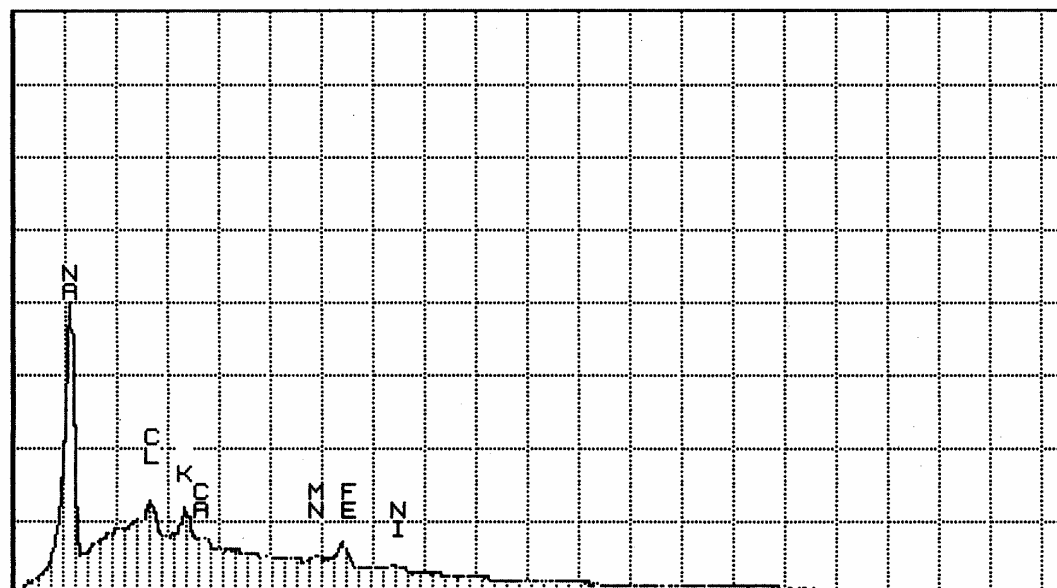
185570 AN107 SOLID2 MARTIN PHOTO#15 SPOT-A

TN-5502 WSRC CSEM.

MON 30-SEP-02 14:28

Cursor: 0.000keV = 0

ROI (0) 0.040: 0.060=0/sec



0.000

VFS = 8192 20.480

30

185570 AN107 SOLID2 MARTIN PHOTO#15 SPOT-B

Figure B- 6. AN-107 Solid 2 Spot-A (top) and Spot-B (bottom)

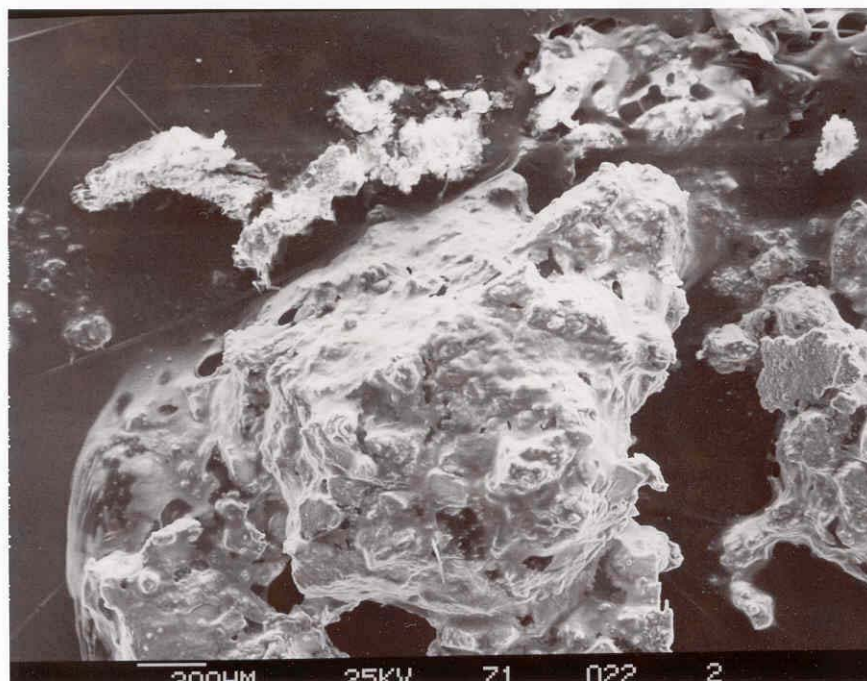


Figure B- 7. AN-107 Solid 3 50x Magnification



Figure B- 8. AN-107 Solid 3 1000x Magnification

TN-5502 WSRC CSEM.

MON 30-SEP-02 15:05

Cursor: 0.000keV = 0

ROI (0) 0.040: 0.060=0/sec

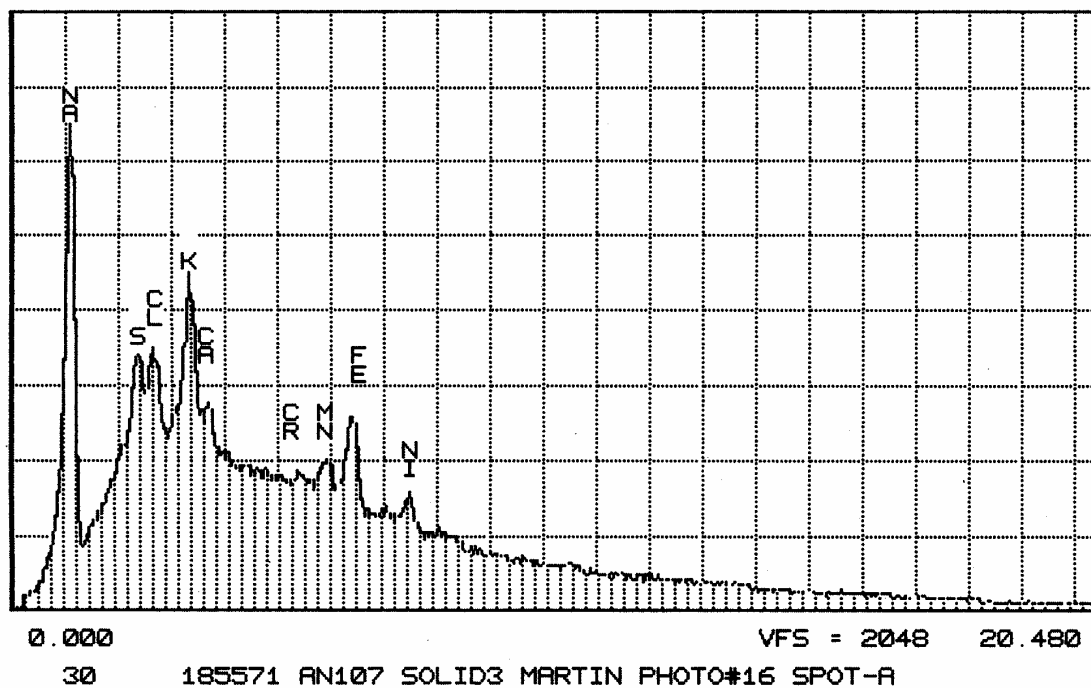


Figure B- 9. AN-107 Solid 3 Spot-A

APPENDIX C. RAW ANALYTICAL RESULTS

The large volume of raw analytical data is available upon request.

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