

Chemical Characterization of an Envelope B/D Sample from Hanford Tank 241-AZ-102

by

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DOE Contract No. DE-AC09-96SR18500

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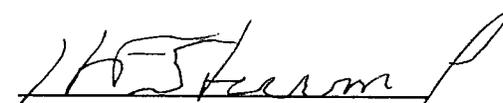
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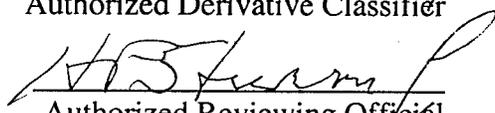


**Chemical Characterization of an Envelope B/D Sample from
Hanford Tank 241-AZ-102**

July 31, 2000

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Authorized Derivative Classifier


Authorized Reviewing Official

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SUMMARY

A sample from Hanford waste tank 241-AZ-102 was received at the Savannah River Technology Center (SRTC) and chemically characterized. The sample containing supernate and a small amount of sludge solids was analyzed as-received. The filtered supernatant liquid, the total dried solids of the sample, and the washed insoluble solids obtained from filtration of the sample were analyzed.

A mass balance calculation of the three fractions of the sample analyzed indicate the analytical results appear relatively self-consistent for major components of the sample. However, some inconsistency was observed between results were more than one method of determination was employed and for species present in low concentrations. The actinides isotopes, plutonium, americium, and curium, present analytical challenges due to the low concentration of these species and the potential for introduction of small amounts of contamination during sampling handling resulting in large uncertainties. A direct comparison to previous analyses of material from tank 241-AZ-102 showed good agreement with the filtered supernatant liquid. However, the comparison of solids data showed poor agreement. The poor agreement shown between the current results for the solids samples and previous analyses most likely results from the uncertainties associated with obtaining small solids samples from a large non-homogenized waste tank.

INTRODUCTION

The BNFL River Protection Project contracted SRTC to provide pretreatment development and testing services to support the BNFL mission to treat Hanford tank waste. As part of the program, SRTC received radioactive Hanford tank waste samples to allow testing of the pretreatment processes with actual waste samples. The first step in this program entails detailed characterization of the radioactive waste samples. The characterization data provides a basis for rational development of pretreatment processes, determination of reagent requirements, and development of physical design parameters for the pretreatment plant.

The characterization portion of the STRC program was conducted under an approved task and quality assurance plan.^{1,2,3} The analytical results and associated uncertainties presented provide a description of the sample received at SRTC. The highly radioactive nature of the samples adds complexity to the analysis. Sub-sampling, large dilutions, and remote handling potentially add error to the analytical accuracy. Replicate sample analysis and submission of standards allow some definition of the magnitude of this error. However, the error associated with obtaining small samples from large non-homogenized waste tanks will be significant. Recent experience at SRS indicates the combined sampling and analytical error associated with obtaining small samples from a well mixed waste tank is on the order of 15 - 20%.⁴

The data presented in this report documents the chemical characterization of a 3.75 L sample of Hanford waste tank 241-AZ-102.

EXPERIMENTAL

Sample History

A total of 8 samples of AZ-102 were received at SRTC on 3-1-99. Table 1 lists the sample jar labels from the 8 samples received at SRTC. Tables 2 shows other sample tracking information.

After loading the samples into the Shielded Cells facility, the 8 samples were composited. The total volume of the composited samples was ~3750 mL. The composite contained a small amount of dark sludge solids. After settling for a day the volume of the settled solids was approximately 250 mL.

The as-received composite sample was thoroughly mixed by vigorously shaking the bottle and filtered through a 0.45 μ nylon disposable filter. The sludge solids (designated Envelope D) on the filter were washed at ambient temperature (~25 °C) with two 50 mL portions of 0.01 M NaOH and dried. The mass of dried sludge solids obtained was ~53 grams. The filtered supernatant liquid (designated Envelope B) was sent for use in ion exchange studies. Archive samples of the as-received composite sample and the filtered supernatant liquid were obtained.

Weight percent solids and density measurements were made on the as-received composite sample and the filtered supernatant liquid. Due to the low sludge solids content of the as-received composite sample a direct measurement of the weight percent insoluble solids was necessary. Samples of the filtered supernatant liquid, the total dried solids of the as-received sample, and dried insoluble solids were prepared for analysis.

Sample Preparation

A 25-fold dilution with deionized, distilled water was generally necessary to lower the radiation levels on filtered supernatant samples before submittal for analysis. Standards were not submitted with the supernatant samples, however, the Analytical Development Section periodically measures standards and blanks to check the calibration and background of the instruments. The total dried solids for the sample were obtained by thoroughly mixing the sample and any insoluble solids present by vigorously shaking the bottle, removing an aliquot of the sample, and drying the aliquot at 100 °C to constant weight to remove free water. The dried insoluble solids were obtained by filtering a portion of the sample through a 0.45 μ filter and washing the solids obtained with a small amount of 0.01 M NaOH to remove interstitial supernatant liquid. The washed insoluble solids were then dried at 100 °C to constant weight to remove free water. Dissolution of samples of total dried solids and dried insoluble solids were performed in triplicate by contacting with aqua-regia or by fusion with sodium peroxide. The digested solids samples were diluted to 250 mL with deionized, distilled water before analysis. Quality control included dissolving a glass standard of known composition concurrently with the

dried solids samples. The glass standard indicates potential problems with sample contamination or incomplete dissolution during the digestion procedure and systematic problems with the analytical procedures. Unless otherwise noted the glass standards showed a successful dissolution and accurate analyses.

Analytical Methods

Analytical Development Section (ADS) performed all analytical measurements with the exception of the weight percent solids and density measurements conducted in the Shielded Cells. ADS uses the following analytical methods for determination of specific species. Nitrate, nitrite, sulfate, oxalate, phosphate, formate, chloride, and fluoride were measured by ion chromatography (IC). Chloride and fluoride were also determined by the ion selective electrode (ISE) method. Aluminate, carbonate, and hydroxide were measured using a titration method employing BaCl_2 to precipitate carbonate allowing the determination of all three species. Sodium, aluminum, and iron, as well as other metallic elements, were measured using inductively-coupled plasma-emission spectroscopy (ICP-ES). Potassium and mercury were measured using atomic absorption spectroscopy (AA) with mercury determined using the cold-vapor technique (CV). Gamma emitting fission products were measured using gamma spectroscopy. Actinides were determined by inductively-coupled plasma mass spectrometry (ICP-MS) and alpha counting spectroscopy. Sr^{90} was determined from beta liquid scintillation counting. Tc^{99} was measured by ICP-MS and ICP-ES.

Weight Percent Solids Analysis

The weight percent of total solids in the samples were measured using a conventional drying oven at 100 °C and stainless steel or polymethylpentene beakers. The weight percent of 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 of the sample and the weight percent dissolved solids in the filtered supernate. Obtaining the weight percent solids analysis of samples in this manner avoids difficulties associated with reproducibly measuring the insoluble solids directly. For samples with less than 3 wt % insoluble solids a direct measurement is required. Equations 1 and 2 allow calculation of the weight percent of insoluble and soluble solids from the total solids and dissolved solids measurements. The weight percent of 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. A 15 wt % NaCl standard solution was measured concurrently during the analysis of the samples. All measurements of the 15 wt % NaCl standard solution were within 5 % of the expected value.

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)

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

Eq. 1

$$W_{SS} = W_{TS} - W_{IS}$$

Eq. 2

Density Measurements

Density measurements were made on both the total sample and the filtered supernate using a pipette tip with the small end heat-sealed. After heat sealing, these pipette tips provide a reproducible volume of 8.25 mL. The sample does not wet the pipette tips eliminating problems with entrained air bubbles when filling a narrow cylinder with thick slurries.

RESULTS

The weight percent solids and density data in Table 3 indicate the 241-AZ-102 sample received at SRTC consists of a relatively dilute supernatant with low insoluble solids content. The low insoluble solids content of the sample require a direct determination of the weight percent insoluble solids. A measurement made on a 100 mL sample yielded a value of 0.48 wt %. Another measurement made when the bulk sample was filtered produced a value of 1.2 weight percent insoluble solids. Due to problems with filter plugging and the presence of a white residue in the dried insoluble solids, the value for the weight percent insoluble solids obtained from the bulk filtration shows a high bias due to the presence of soluble salts.

The analytical results for the filtered supernatant liquid in Table 4 agree with the weight percent solids and density data indicating a relatively dilute salt solution with a sodium concentration of 2.77 M. The supernatant contains low levels of other metals such as aluminum and phosphorus but a relatively high concentration of chromium. The supernatant also contains a relatively high concentration of sulfate. A poor correlation exists between the TIC and the carbonate values with TIC being ~30% higher. The aluminate and carbonate measured by titration after treatment with $SrCl_2$ may be subject to potential error due to precipitation of other anions along with the carbonate or incomplete precipitation of the carbonate. The precipitation of aluminate and other anions along with the carbonate would lower the observed aluminate concentration and produce a high bias for the carbonate measurement. The method dilutes the sample by ~40X which should reduce the potential of significant precipitation of other anions.

Precipitation of aluminate has not been observed with a standard containing 0.5 M aluminate, carbonate, and free hydroxide using the $SrCl_2$ method. The precipitation of aluminate will also potentially reduce the free hydroxide concentration. The TOC value exceeds the organic carbon derived from the sum of the formate and oxalate by nearly an order of magnitude. The TOC results indicate the potential presence of a significant organic carbon source besides the formate and oxalate. The data in Table 4 also shows a poor cation/anion balance with the sum of the cations exceeding the sum of the anions by nearly 20%. Substituting the TIC result converted to a carbonate concentration into the sum of the anions reduces the difference in the cation anion balance to 8%. The

cation/anion balance provides an indication of whether any significant species were missed or whether bias was present in the analytical methods for cations versus anions.

The data for the analysis of radioactive species in the filtered supernatant liquid, shown in Table 5, indicate a high concentration of Cs^{137} and a relatively high concentration of Tc^{99} . The agreement between the values obtained from ICP-MS and other methods appear fairly good. Although the Tc^{99} value from the ICP-MS exceeds the Tc value from ICP-ES by 30%, the mass 137 value agrees to within 5% of the Cs^{137} value obtained from gamma spectroscopy. These comparisons between different analytical methods provide some level of assurance as to the quality of the data. Some of the error in the analysis of the supernatant can be attributed to the large dilutions required due to the high Cs^{137} content of this sample. Gamma spectroscopy detected no other gamma emitters.

The analytical results for non-radioactive species of the total dried solids of the 241-AZ-102 sample listed in Table 6 show a high sodium content. The high sodium content results from the inclusion of the dissolved salts and low weight percent insoluble solids present in the sample. The total dried solids includes the insoluble solids present in the sample as well as all of the dissolved salts. The insoluble solids were collected by filtration of the sample and washed with small portions of 0.01 M NaOH to remove interstitial supernatant liquid. For most of the elements listed in Tables 7 and 8 the values represent the average of six replicate samples, three from the aqua-regia dissolution and three from the sodium peroxide fusion method. In some cases, only aqua-regia data or the data from the sodium peroxide fusion method were used. For example, only the sodium peroxide fusion data was used to calculate an average silicon value because silicon does not dissolve well with the aqua-regia method. Sodium data cannot be obtained from the sodium peroxide fusion method so only the aqua-regia data was used to calculate an average value. A standard glass of known composition dissolved along with the sample provides an indication of the quality of the dissolution and the presence of contamination introduced during the dissolution procedure. Other metals with significant concentrations in the total dried solids include potassium, iron, aluminum, chromium, and silicon. In contrast, iron and aluminum dominate the composition of the dried insoluble sludge solids. The insoluble solids also consist of lesser amounts of cadmium, zirconium, nickel, sodium, and silicon.

In Table 7, the results for the radioactive components of the total dried solids sample and the insoluble solids sample both show high Cs^{137} and Sr^{90} concentrations. The uranium isotopics between the two samples show good agreement with ~99.1% U^{238} , ~0.07% U^{236} , and ~0.8% U^{235} on a weight basis. However, the plutonium isotopics do not agree well with the total solids showing 1.5% Pu^{238} , 84.1% Pu^{239} , and 14.4% Pu^{240} versus the insoluble solids percentages of 0.03%, 92.7%, and 7.2% on a weight basis. The Tc^{99} as detected in the ICP-MS for both samples does not agree with the Tc value by ICP-ES reported in Table 6. The Tc values obtained by ICP-ES exceed the ICP-MS values by nearly an order of magnitude for the total solids sample and 2 orders of magnitude for the insoluble solids sample. Cm^{244} was detected in the total dried solids but appears to be due to contamination during sample preparation as the glass standard contained Cm^{244} of approximately the same magnitude. However, in the insoluble solids although a

detectable amount of Cm²⁴⁴ contamination was found in the glass standard dissolved concurrently with the sample the level is three orders of magnitude smaller.

A check on the self-consistency of the data, shown in Table 8, indicates relatively good agreement for the major components of the samples. The table was generated by converting the filtered supernatant data to a total dried solids concentration basis followed by adding the converted filtered supernatant and insoluble solids data together based on the weight percent solids and density data. Theoretically, the sum of these two samples should be equal to the total dried solids analysis minus any material leached from the insoluble solids during washing with 0.01 M NaOH. The radionuclide data, with the exception of Cs¹³⁷, Pu²³⁹, and Tc⁹⁹, show poor agreement. The calculation of the sum of the filtered supernatant data and the insoluble solids data was found to be very sensitive to the value of the weight percent insoluble solids for some components. Obviously, species concentrated in the insoluble solids show the greatest sensitivity to value for the weight percent insoluble solids used in the calculation.

The notes below Table 3 indicate the measurement of another value for the weight percent insoluble solids from a small sample of 241-AZ-102. Reducing the insoluble solids value used in the calculation from 1.2 wt % to the 0.5 wt % measurement significantly decreases the percent difference for many of the radionuclides in Table 8. For example, the percent difference between the Sr⁹⁰ in the total dried solids analysis versus the sum of the filtered supernatant and insoluble solids data drops from 136% to 1%. The percent differences for Cs¹³⁷ and Pu²³⁸ remain approximately the same while the Am²⁴¹ percent difference drops from 282% to 59%. The percent difference for the aluminum drops from 48% to 1% while most of the other ICP-ES elements remain approximately the same. For the ICP-MS data in Table 8, the percent difference in the U²³⁵ values drops from 121% to 8% and the U²³⁸ from 139% to 2%. The percent difference in the Pu²³⁹ and Pu²⁴⁰ increase to 53% and 79% respectively while the Tc⁹⁹ percent difference remains approximately the same.

COMPARISON TO PREVIOUS ANALYSES OF 241-AZ-102 SAMPLES

The samples from 241-AZ-102 sent to SRTC were from a core composite taken in 1998. The composition of the 1998 samples from Core 254 have not yet been published in the tank characterization report for tank 241-AZ-102.⁶ Data values prior to 1987 were not used for comparison to the sample received at SRTC since the tank was pumped dry in 1986. Results for tank samplings in August and October of 1987 consist mainly of anion data for the supernate. Characterization of the sludge in 241-AZ-102 was conducted on a core sample taken in 1989.⁷ However, this core sample was spit into layers or washed and remixed in various ways to form composites prior to analysis. Results from the layered analysis vary considerably and thus were not used for the comparison.

Only the results from the February 1995 sampling were compared to the current data.⁸ Three supernate samples and one solid grab sample were obtained from the tank and found to consist of 90% supernate. The reference provides minimal results for the as-

received supernate and the solids with and without washing. Only small differences were found for the composition of the solids with and without washing. The sodium and Cs¹³⁷ concentrations dropped significantly while the chromium concentration dropped approximately 50%. All other analytical results reported for the two samples were nearly identical. Even though the SRTC insoluble solids from 241-AZ-102 were washed on the filter with two small portions of 0.01 M NaOH, the solids appear to be more comparable to the unwashed solids from the reference based on the sodium and Cs¹³⁷ concentrations.

Table 9 shows a comparison of the filtered supernatant liquid from the sample received at SRTC to the average values of the supernatant liquid from the 1995 sample. With the exception of the aluminum and TOC results the values show good agreement. Aluminum at these high concentrations precipitates readily with small changes in pH or temperature which could explain the large difference in concentrations between the 1995 sample and the 1998 sample. Neither the supernatant liquid from the sample received at SRTC nor that from the 1995 sample were diluted and should therefore be directly comparable.

The comparison of the solids composition in Table 10 do not show as good agreement. In all cases except sodium and Cs¹³⁷, the current results exceed the values obtained from the 1995 sample. Higher variability should be expected from sampling solids from a non-homogenized waste tank compared to the supernatant liquid.

CONCLUSIONS

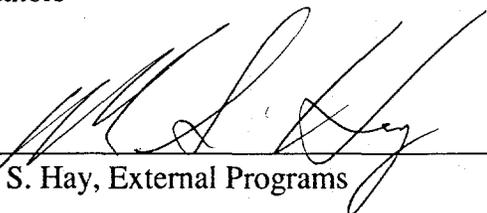
The data obtained for the sample of 241-AZ-102 received at SRTC appears relatively self-consistent for major components of the sample. Some inconsistency was observed between results were more than one method of determination was employed. Results for radionuclides present in low concentrations, namely the actinides, generally exhibit larger errors due to the difficulties in analyzing species present in low concentrations and the potential for introduction of small amounts of contamination during sample handling causing large uncertainties. The comparison to previous analyses of supernatant liquid samples from 241-AZ-102 indicates relatively good agreement with the current analytical results. The poor agreement shown between the current results for the solids samples and previous analyses most likely results from the uncertainties associated with obtaining small solids samples from a large non-homogenized waste tank.

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APPROVALS

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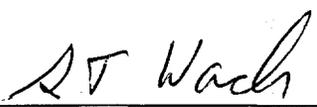

S. T. Wach, Manager, External Programs 8/2/00
Date

Table 1. Listing of Sample Jar Labels from Bottles of AZ-102 Sample Received at SRTC.

Jar No.	Jar Label
Jar 1	16085
Jar 2	16087
Jar 3	16088
Jar 4	16089
Jar 5	16090
Jar 6	16100
Jar 7	16104
Jar 8	16105

Table 2. Descriptions of Source Material for the Jar ID.

Jar ID	Laboratory ID	Group I*	Sample Date	Type Sample Shipped
16085	S99T000213	98000550	10/5/98	Core 254 Composite
16087	S99T000214	98000550	10/5/98	Core 254 Composite
16088	S99T000212	98000550	10/5/98	Core 254 Composite
16089	S99T000215	98000550	10/5/98	Core 254 Composite
16090	S99T000217	98000550	10/5/98	Core 254 Composite
16100	S99T000216	98000550	10/5/98	Core 254 Composite
16104	S99T000219	98000550	10/5/98	Core 254 Composite
16105	S99T000218	98000550	10/5/98	Core 254 Composite

Table 3. Results of Weight Percent Solids and Density Measurements for 241-AZ-102 Sample As-Received.

	As Received 241-AZ-102
wt % Total Solids	16.4 (1)
wt % Dissolved Solids	16.1 (1)
wt % Insoluble Solids	1.2*
wt % Soluble Solids	15.2**
Density of Supernatant, g/mL	1.15
Density of Slurry, g/mL	1.16

Percent relative standard deviation for triplicate measurements are shown in parentheses.

*Result of a single direct measurement by filtration of the bulk sample and weighing the dried solids collected. Due to filter plugging the solids potentially contain some soluble salts giving the value high bias. A small amount of white residue consistent with the presence of soluble salts was observed on the dried solids. Another measurement on 100 mL of the sample yielded a weight percent insoluble solids value of 0.48.

**Calculated from the difference between the wt % total solids and wt % insoluble solids.

Table 4. Concentration of Non-Radioactive Species in Filtered Supernatant Liquid of 241-AZ-102.

Element	Average Concentration mg/L	Average Concentration M	% Relative Standard Deviation
Al	7.53E+02	2.79E-02	4.8
B	4.71E+00	4.36E-04	6.5
Ba	<5.00E-01	<3.64E-06	
Ca	1.08E+02	2.70E-03	12.8
Cd	<7.50E-01	<6.67E-06	
Co	<1.50E+00	<2.55E-05	
Cr	7.68E+02	1.48E-02	1.7
Cu	<1.25E+00	<1.97E-05	
Fe	<1.00E+00	<1.79E-05	
Hg	<2.50E-01	<1.25E-06	
K	3.15E+03	8.07E-02	1.6
La	<5.25E+00	<3.78E-05	
Li	<1.00E+00	<1.44E-04	
Mg	<5.00E-01	<2.06E-05	
Mn	<7.50E-01	<1.37E-05	
Mo	5.86E+01	6.11E-04	1.0
Na	6.37E+04	2.77E+00	2.2
Ni	<1.75E+00	<2.98E-05	
P	1.68E+02	5.42E-03	5.2
Pb	<8.25E+00	<3.98E-05	
Ru	<1.23E+01	<1.21E-04	
Si	<6.97E+00	<2.48E-04	
Sn	<4.50E+00	<3.79E-05	
Sr	<2.50E-01	<2.85E-06	
Tc	1.07E+01	1.08E-04	7.6
Ti	<1.00E+00	<2.09E-05	
V	<1.25E+00	<2.45E-05	
Zn	<1.00E+00	<1.53E-05	
Zr	<2.00E+00	<2.19E-05	

Percent relative standard deviations are for analysis of three replicate samples.

Table 4. Concentration of Non-Radioactive Species in Filtered Supernatant Liquid of 241-AZ-102. (Continued)

Analyte	Average Concentration mg/L	Average Concentration M	% Relative Standard Deviation
[NO ₃]	1.69E+04	2.73E-01	2.3
[NO ₂]	3.03E+04	6.59E-01	2.4
[PO ₄ ³⁻]	<2.50E+02	<2.63E-03	
[SO ₄ ²⁻]	1.65E+04	1.72E-01	2.5
[C ₂ O ₄ ²⁻]	2.83E+03	3.21E-02	17
[Cl ⁻]	<5.00E+01	<1.41E-03	
[F ⁻]	9.67E+02	5.09E-02	10
[CHO ₂ ⁻]	<2.50E+02	<5.56E-03	
[OH ⁻] free	9.03E+03	1.09E-01	17
[CO ₃ ²⁻]	2.35E+04	3.92E-01	5.8
[AlO ₂ ⁻]	<2.95E+03	<5.00E-02	
TIC	6.14E+03	*	0.2
TOC	6.04E+03	*	0.8

Percent relative standard deviations are for analysis of three replicate samples.

*Cannot calculate molar concentrations without knowing the specific compounds represented by the analytical result.

Table 5. Concentration of Radioactive Species in Filtered Supernatant Liquid of 241-AZ-102.

Analyte	Average Concentration mg/L	Average Concentration μ Ci/mL	% Relative Standard Deviation
Sr ⁹⁰	1.51E-02	2.07E+00	7.9
Tc ⁹⁹ *	1.57E+01	2.67E-01	1.4
Cs ¹³³ *	9.05E+00	stable	12
Cs ¹³⁵ *	5.86E+00	6.74E-03	2.9
Cs ¹³⁷ *	1.25E+01	1.08E+03	1.5
Cs ¹³⁷	1.31E+01	1.14E+03	1.8
Np ²³⁷ *	2.51E-01	1.77E-04	5.3
U ²³⁸ *	6.94E+00	2.33E-06	3.3
Pu ²³⁸ /Am ²⁴¹	**	1.11E-03	47
Pu ²³⁹	1.19E-01	7.28E-03	31
Am ²⁴¹	<2.03E-04	<1.64E-02	
Cm ²⁴⁴	<8.52E-04	<2.91E-03	

Percent relative standard deviations are for analysis of three replicate samples.

*Determined by ICP-MS

**Cannot calculate mg/L concentrations without knowing the ratio of Pu²³⁸ to Am²⁴¹.

Table 6. Concentration of Non-Radioactive Species in Total Dried Solids and Insoluble Dried Solids of 241-AZ-102 Sample.

Element	241-AZ-102 Total Solids		241-AZ-102 Insoluble Solids	
	Average Concentration Wt%	% Relative Standard Deviation	Average Concentration Wt%	% Relative Standard Deviation
Al	6.25E-01	15	1.02E+01	2.9
B	1.83E-02	30	<3.36E-02	
Ba	7.44E-03	39	1.19E-01	4.5
Ca	1.14E-01	36	8.40E-01	14
Cd	9.56E-02	11	3.30E+00	4.3
Co	9.85E-03	7.8	1.83E-02	23
Cr	3.92E-01	11	1.91E-01	5.4
Cu	1.22E-02	7.5	6.15E-02	4.9
Fe	8.33E-01	21	2.48E+01	4.2
Hg	<9.8E-04		<5.0E-04	
K	1.53E+00	11	1.31E-01	27
La	4.14E-02	35	1.08E+00	7.1
Li	1.04E-02	4.8	<1.0E-02	
Mg	1.95E-02	24	1.89E-01	9.2
Mn	2.28E-02	18	6.15E-01	4.4
Mo	2.72E-02	13	5.29E-03	12
Na	3.19E+01	1.3	1.79E+00	2.9
Ni	6.59E-02	14	1.80E+00	4.5
P	1.22E-01	36	3.43E-01	73
Pb	4.74E-02	11	2.98E-01	12
Ru	4.39E-02	2.9	3.74E-01	8.2
Si	1.59E-01	22	1.60E+00	34
Sn	2.35E-02	22	2.02E-01	67
Sr	1.93E-03	15	5.79E-02	4.1
Tc	1.65E-02	59	1.96E-02	19
Ti	1.26E-02	1.8	1.57E-02	11
V	1.11E-02	3.2	1.49E-02	21
Zn	1.76E-02	3.1	3.39E-02	59
Zr	1.32E-01	16	3.10E+00	4.7

Table 7. Concentration of Radioactive Species in Total Dried Solids and Insoluble Dried Solids of 241-AZ-102.

Isotope	241-AZ-102 Total Solids		241-AZ-102 Insoluble Solids	
	Average Concentration $\mu\text{Ci/g}$	% Relative Standard Deviation	Average Concentration $\mu\text{Ci/g}$	% Relative Standard Deviation
Sr ⁹⁰	8.54E+02	11	2.73E+04	30
Tc ⁹⁹ *	1.06E+00	18	4.05E-02	33
Cs ¹³⁷	5.23E+03	15	7.93E+02	12
Eu ¹⁵⁴	Not detected		8.92E+01	4.4
Eu ¹⁵⁵	Not detected		1.63E+02	10
U ²³³ *	Not detected		6.11E-03	**
U ²³⁴ *	Not detected		1.46E-02	**
U ²³⁵ *	1.75E-05	16	5.28E-04	7.6
U ²³⁶ *	4.82E-05	47	1.11E-03	9.1
U ²³⁸ *	3.16E-04	19	1.01E-02	12
Pu ²³⁸	2.18E+00	73	2.16E+00	47
Pu ²³⁹ *	4.24E-01	25	6.57E+00	12
Pu ²⁴⁰ *	2.67E-01	68	1.88E+00	34
Am ²⁴¹	6.43E+00	11	3.35E+02	19
Cm ²⁴⁴	6.90E+00***	115	3.14E+00	32
Np ²³⁷ *	6.70E-03	17	1.43E-01	9

*Determined by ICP-MS

**Value is from a single determination (other replicates were below detection limits)

***The Cm²⁴⁴ detected in the sample appears to be from contamination during sample preparation.

Table 8. Comparison of the Concentrations in the Total Dried Solids Sample Versus the Sum of the Filtered Supernatant and Insoluble Dried Solids Samples of the 241-AZ-102 Sample.

	Units	Total Dried Solids	Filtrate and Insoluble Solids	Percent Difference
Counting				
Sr ⁹⁰	uCi/g	8.54E+02	2.01E+03	136%
Cs ¹³⁷	uCi/g	5.32E+03	6.05E+03	14%
Pu ²³⁸	uCi/g	2.18E+00	1.96E-01	91%
Am ²⁴¹	uCi/g	6.43E+00	2.46E+01	282%
ICP-ES				
Al	wt%	6.25E-01	9.25E-01	48%
Ca	wt%	1.14E-01	1.19E-01	4%
Cr	wt%	3.92E-01	4.19E-01	7%
Cu	wt%	1.22E-02	5.17E-03	58%
K	wt%	1.53E+00	1.67E+00	9%
Mo	wt%	2.72E-02	3.12E-02	15%
Na	wt%	3.19E+01	3.37E+01	6%
P	wt%	1.22E-01	1.14E-01	7%
Si	wt%	1.59E-01	1.18E-01	26%
Tc	wt%	1.65E-02	7.05E-03	57%
ICP-MS				
U ²³⁵	wt%	8.11E-04	1.79E-03	121%
U ²³⁸	wt%	9.42E-02	2.25E-01	139%
Pu ²³⁹	wt%	6.92E-04	7.86E-04	14%
Pu ²⁴⁰	wt%	1.18E-04	6.09E-05	48%
Tc ⁹⁹	wt%	6.26E-03	8.37E-03	34%

Table 9. Comparison of the Supernatant from a 1995 Sample⁸ of 241-AZ-102 with the Filtered Supernatant of the 241-AZ-102 Sample Received at SRTC.

Analyte	Units	Supernatant from 1995 Sample ⁸	Filtered Supernatant (from Table 4 and 5)	Percent Difference
Cs ¹³⁷	μCi/ml	1.04E+03	1.14E+03	9%
Sr ⁹⁰	μCi/ml	1.79E+00	2.07E+00	14%
[NO ₃]	M	3.84E-01	2.73E-01	41%
[NO ₂]	M	6.00E-01	6.59E-01	9%
[SO ₄ ²⁻]	M	1.80E-01	1.72E-01	5%
[F]	M	5.29E-02	5.09E-02	4%
[OH] _{free}	M	1.11E-01	1.09E-01	1%
Al	M	5.71E-02	2.79E-02	105%
Na	M	2.31E+00	2.77E+00	16%
TIC	mg/L	5.84E+03	6.14E+03	5%
TOC	mg/L	1.50E+03	6.04E+03	75%

Table 10. Comparison of the Unwashed Solids from the 1995 Sample⁸ of 241-AZ-102 with the Insoluble Solids of the 241-AZ-102 Sample Received at SRTC.

Analyte	Units	Unwashed Solids from 1995 sample ⁸	Insoluble Solids from Tables 6 and 7	Percent Difference
Cs ¹³⁷	μCi/g	8.10E+02	7.93E+02	2%
Am ²⁴¹	μCi/g	6.78E+01	3.35E+02	80%
Pu ^{239/240}	μCi/g	1.60E+00	8.73E+00	82%
Sr ⁹⁰	μCi/g	6.97E+03	2.73E+04	74%
Al	wt%	6.51E+00	1.02E+01	36%
Ca	wt%	1.70E-01	8.40E-01	80%
Cd	wt%	1.07E+00	3.30E+00	68%
Cr	wt%	9.75E-02	1.91E-01	49%
Fe	wt%	6.66E+00	2.48E+01	73%
La	wt%	2.40E-01	1.08E+00	78%
Na	wt%	3.81E+00	1.79E+00	113%
Ni	wt%	4.90E-01	1.80E+00	73%
Zr	wt%	7.60E-01	3.10E+00	75%