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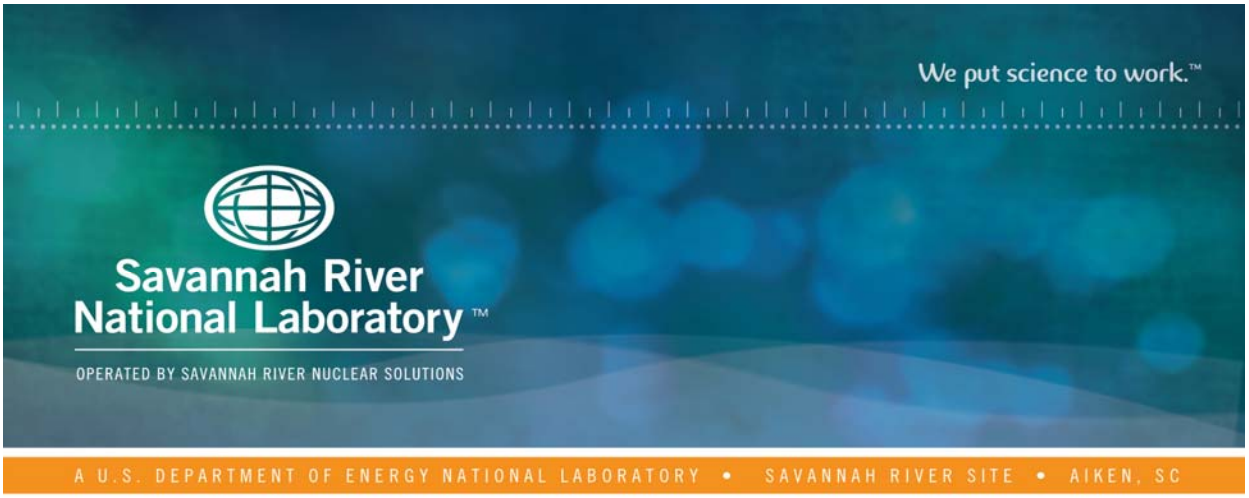

Tank 12H Residuals Sample Analysis Report

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June 2015

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LIST OF REVISIONS		
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EXECUTIVE SUMMARY

Tank 12H Characterization Summary

The Savannah River National Laboratory (SRNL) was requested by Savannah River Remediation (SRR) to provide sample preparation and analysis of the Tank 12H final characterization samples to determine the residual tank inventory prior to grouting. Eleven Tank 12H floor and mound residual material samples and three cooling coil scrape samples were collected and delivered to SRNL between May and August of 2014.

The eleven floor and mound samples were homogenized and combined into three composite samples based on a proportional compositing scheme and the resulting composite samples were analyzed for an extensive suite of radiological and elemental components. Additional measurements performed on the Tank 12H composite samples included bulk densities, weight percent solids, and water leaching of the solids to determine water soluble components. In general, these analyses were performed and reported in triplicate where possible. Where analytical methods yielded additional analytes other than those requested by the customer, the results were also reported.

The cooling coil scrape samples and the combined liquid fraction separated from the floor samples were characterized for a limited suite of analytes providing scoping information.

Sufficient standards and blanks were utilized to provide quality assurance for the characterization of the Tank 12H composite samples as specified in the technical task request document. The target detection limits for all the analyses were based on customer desired detection limits as also specified in the technical task request document. While many of the target detection limits were met for the species characterized, some were not met. The isotopes and anions with target detection limits not met in all cases included: Th-229, Th-230, Pa-231, U-233, Cm-243, Cm-244, Am-242m, fluoride, and phosphate. For these analytes, the detection limits were typically one to two orders of magnitude higher than the target detection limits. The target detection limits for the majority of the routine radionuclides and elemental constituents were met most of the time. SRR reviewed all of these cases and determined that the impacts of not meeting some of the target detection limits were acceptable.

Statistical Review Summary

Statistical characterizations of analyte concentrations in the residual solids material on the floor of Tank 12H were performed on 36 radionuclides, 22 elemental constituents, and 7 anions. The statistical methods and application of them to these analytes are described in Appendix D. Data and summary tables for these statistical results are presented in Appendix E.

The mean, the standard deviation, the percent standard deviation, and the upper 95% confidence limit (UCL95) for the mean concentration were computed for each analyte that had all results above the minimum detection limits. The smallest and largest detection limits are used to summarize the concentrations for each analyte that had all results below the minimum detection limits.

Most analytes had statistically significant sampling variances, but none of the analytes had heterogeneous measurement variances across the composite samples. The analytical data were also screened for potential outliers. Only Am-243 on Composite Sample 2 was determined to have a potential outlier result. The UCL95 was computed for Am-243 with and without the potential outlier, and the larger of the two UCL95 results was reported.

Two isotopes, Pa-231 and Am-242m, that did not meet target detection limits, had sufficient numbers of results above the minimum detection limits to support estimates of their mean concentrations and UCL95 values for their means. Besides Pa-231 and Am-242m, the radionuclides Am-243 and Cm-245 and the

elemental constituent molybdenum have mixtures of results that were above and below the minimum detection limits. An estimate of the mean concentration and a UCL95 were also computed for Am-243 and molybdenum, but Cm-245, having only two results above the minimum detection limits, did not render a UCL95. Cm-245 was summarized by its smallest and largest detection limits.

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LIST OF ABBREVIATIONS

AD	Analytical Development
AQR	Aqua Regia Digestion
ARG	Analyzed Reference Glass
COC	Chain-of-Custody
CRGS	Cesium Removed Gamma Spectra
CVAA-Hg	Cold Vapor Mercury by Atomic Absorption
DL	Detection Limit
DNR	Data Not Reported
HLW	High-Level Waste
ICP-ES	Inductively Coupled Plasma–Atomic Emission Spectroscopy
ICP-MS	Inductively Coupled Plasma – Mass Spectrometry
LIMS	Laboratory Information Management System
LSC	Liquid Scintillation Counting
MDA	Minimum Detectable Activity
MDC	Minimum Detectable Concentrations
PF	Sodium Peroxide/Hydroxide Fusion
PHA	Pulse Height Analysis
PMP	Polymethyl Pentene
RD	Relative Deviation
SRNL	Savannah River National Laboratory
SRR	Savannah River Remediation, LLC
TDL	Target Detection Limit
TTA	Thenoyltrifluoroacetone
TTQAP	Task Technical and Quality Assurance Plan
TTR	Technical Task Request
UCL95	Upper 95% Confidence Limit
UL	Upper Limit
XRD	X-Ray Diffraction
XRF	X-Ray Fluorescence

1.0 Introduction

Savannah River Remediation (SRR) is preparing Tank 12H for closure. The Savannah River National Laboratory (SRNL) was requested by SRR to provide sample preparation and analysis of the Tank 12H final characterization samples for use in determining the Tank 12H residual inventory. An assortment of Tank 12H floor, mound, and cooling coil scape samples were provided by SRR as summarized in Table 1. Figures 1 and 2, respectively, show the location of Tank 12H relative to other tanks in the H-Tank Farm system, and the individual Tank 12H floor and mound sampling locations. Three sample composites were generated from the various floor and mound materials.

For compositing the Tank 12H sample materials, the volume of residual material in each of the Tank 12H sampling segments was determined by SRR Engineering and used to estimate the strata volumes in the tank. These strata volumes were converted into volumetric proportions, and subsequently to the mass of residual material to be used from each sample for each composite sample creation. This is based on the methodology described in the Liquid Waste Tank Residuals Sampling and Analysis Program Plan.[1]

Thus, each of the three Tank 12H sample composites was derived from multiple individual Tank 12H floor and mound samples, with all three composites representing the sum of Tank 12H floor and mound residual material. Hence, a complete characterization of the Tank 12H residuals involves analytical data from three composite Tank 12H samples (Tank 12H-Composite sample 1, Tank 12H composite sample 2 and Tank 12H Composite sample 3), which are based on the five Tank 12H floor samples and the six Tank 12H mound samples.

The processing of the five Tank 12H floor sample solid fractions, the six Tank 12H mound samples and the analytical characterization of the Tank 12H-composite samples (Tank 12H composite sample 1, Tank 12H Composite sample 2 and Tank 12H composite sample 3) were performed in accordance with the Technical Task Request (TTR) provided by SRR,[2] the Task Technical and Quality Assurance Plan (TTQAP) prepared by SRNL,[3] and the Tank 12H Sample Location Document and Sampling and Analysis Plan[4, 5].

2.0 Tank 12H Sample Receipt and Preparations for Characterizations

2.1 Tank 12H Coil Samples

Two coil sample containers, labelled T12-R8-C-low and T12-R8-C-mid, were received at SRNL on July 21, 2014 and the contents of the stainless steel containers were transferred into clear polymethyl pentene (PMP) containers and then photographed. The weights of the two coil samples, T12-R8-C-Low and T12-R8-C-Mid, were respectively, 21 and 47 g. The volumes of the two coil samples were relatively small, which meant the density of the material is larger than expected for a typical sludge. The third Tank 12 coil sample, T12-R8-C-High, was delivered to SRNL on July 23, 2014 and then opened and transferred into a PMP beaker in the cell on July 24, 2014 (Figure 3). The weight of this third coil sample was 26 g. The "as-received" bulk densities of the three cooling coils samples (T12-R8-C-Low, T12-R8-C-Mid and T12-R8-C-High) were determined and documented as soon as the samples were delivered to SRNL (Appendix C-b).

The X-ray diffraction (XRD) spectrum of one of the Tank 12H coils samples, T12-R8-C-Mid, is shown in Figure 4. The XRD spectra show that the coil samples contain significant amounts of mercuric oxide. The weight percent solids of each of the three coils samples was then determined followed by the grinding/sieving/homogenizing of the sample material, and then the bulk density of each homogenized material was determined again (Appendix C-b). Only the basic scoping analyses on Tank12H cooling

coil samples presented in Appendix C-b were performed. No further characterization analysis on the three Tank 12H cooling coils samples was performed.

2.2 Tank 12 Floor Samples

The first set of Tank 12 floor samples (T12-F-1, T12-F-2, T12-F-3, T12-F-4, T12-F-5 and T12-F-6) were received at SRNL Shielded Cell Operations on August 11, 2014. Representative pictures of some Tank 12H floor samples are shown in Figure 5. Because of the belief that insufficient solid material had been collected, a resample for sample T12-F-1, T12-F-2 and T12-F-3 was performed by SRR. The three resamples were labelled as T12-F-1R, T12-F-2R, and T12-F-3R. These resamples of the Tank 12H floor material were received at SRNL on August 18, 2014 and opened on August 19, 2014. Tank 12H floor samples, with the exception of two samples (T12-F-5 and T12-F-1R), contained a sufficient amount of liquid fraction and the solid fraction was more like a black silt which was quite pasty and sticky to work with. A 0.45 micron Nalgene filter membrane was used to perform a solid liquid separation on all the samples with sufficient liquid portions and the resulting liquids were combined to form the "Tank 12H combined liquid fraction" of approximately 167mL. Scoping analyses performed on the combined liquid fractions are presented in Appendix C-b. The dry weights of the Tank 12H floor samples, after 146 hours air-drying, are presented in Table 2.

Because most of the floor samples contained appreciable amounts of liquid fraction, no "as-received" physical characterization (bulk density or weight percent solids) was performed. However, after the liquid/solids separations, each solid fraction (T12-F-1R, T12-F-2R, T12-F-3R, T12-F-4, and T12-F-5) was air-dried for about 146 hours and then the bulk densities and weight percent solids were determined. Again, please note that no appreciable solids were recovered from samples T12-F-1, T12-F-2, T12-F-3 and T12-F-6. The five air-dried Tank 12H floor sample solids were then ground/sieved/homogenized (Figure 6, insert A).

Homogenizing each sample involved grinding with a new mortar and pestle and then passing the powder through a sieve with 850 micron openings (mesh 20). Materials which did not pass through the sieve were re-ground until they were small enough to pass through the sieve. The bulk density and weight percent solids for each of the five homogenized air-dried Tank 12H floor samples were then determined as shown in Table 2. The bulk densities of the ground, sieved and homogenized resamples (T12-F-1R, T12-F-2R and T12-F-3R) were lower than the bulk densities of the other three ground, sieved and homogenized floor samples (T12-F-4, T12-F-5 and T12-F-6).

2.3 Tank 12 Mound Sample

The six Tank 12H mound samples (T12-M-H1, T12-M-H2, T12-M-H3, T12-M-L1, T12-M-L2, T12-M-L3) were received at SRNL on August 27, 2014. The stainless steel containers for these samples were each almost 95% full with very wet sludge materials.

The six Tank 12H mound samples did not contain any appreciable amount of free liquid fraction and therefore no liquid/solid separation was needed, although the wet cake materials were all very sticky and difficult to work with. When these samples were delivered to SRNL, it was not easy to recover all the sample solids from the stainless steel sampling containers because of the wet and pasty nature of the solids. However, it was possible to transfer appreciable amounts of each sample (60-70%) into a secondary capped PMP clear container, which was appropriately labelled and capped. The samples which were stored in these PMP containers were later used to determine the first weight percent solids for the mound samples but not the "as received" bulk densities because these samples were still wet and sticky.

Each of the six samples in the PMP container was air-dried for 24 hours and then the bulk density was determined. After this, all the mound samples were air-dried, including those which were in the original sampling vessels, for 150 hours. The bulk densities and weight percent solids were then determined again as shown in Table 3.

Tank 12H mound sample bulk densities and weight percent solids were determined as described in Appendix B. The bulk densities and weight percent solids for the Tank 12H mound samples are provided in Table 3. Each Tank 12H air-dried mound sample was then homogenized to reduce the particle size. As described above, homogenizing each sample involved grinding each sample separately with a new mortar and pestle and then passing the powder through a sieve with 850 micron openings (mesh 20). Materials which did not pass through the sieve were re-ground until they were small enough to pass through the sieve.

The bulk densities of the ground, sieved and homogenized lower mound samples (T12 M-L-1, T12 M-L-2 and T12 M-L-3) averaged 0.58 g/mL and were lower than the bulk densities of the ground, sieved and homogenized upper mound samples (T12 M-H-1, T12 M-H-2 and T12 M-H-3), which averaged 0.92 g/mL. Pictures of the resulting powdered Tank 12H homogenized mound samples are shown in Figure 6, insert B.

2.4 Tank 12H Composite Samples

The three Tank 12H composite samples were created by physically blending of six Tank 12H mound samples and five floor samples as shown in Tables 1 and 4. The composite samples were designated as Tank 12H Composite 1, Tank 12H Composite 2 and Tank 12H Composite 3. See Table 4 for a summary of the Tank 12H compositing sample construction and sample weights as specified in the TTR[2].

In generating the three Tank 12H composite samples, the volume of residual material in each strata was used. These strata volumes were converted into volumetric proportions, and subsequently to the mass of residual material to be used from each sample for each composite sample creation. In summary, eleven Tank 12H samples (five floor and six mound samples) were homogenized and combined into three composite samples as shown in Figure 7.[2, 6] Additional compositing details are found in the Tank 12H TTR[2]. The bulk density and other physical parameters of each of the three Tank 12H composite samples were then determined by the processes described in Appendix B and data shown in Table 5. The average bulk density for each composite sample was different from those of the others (Table 5).

2.5 Blank Evaluations and Reference Materials

In addition to reagent blanks used by the SRNL Analytical Development (AD) Group, one solid-phase reference material and one solid-phase matrix blank were used during the characterization of Tank 12H samples. The solid-phase reference material was an analyzed reference glass (ARG) which was stored outside the shielded cells, but processed in the shielded cells along with the samples during sample preparations. The elemental chemical composition of the ARG is presented in Appendix A-2.

The solid-phase matrix blank was an in-cell synthetic sodalite aluminosilicate mineral. Synthetic sodalite mineral is not a reference material, but is a non-radioactive material introduced into the cell environment where the Tank 12H materials were processed to be used as a monitoring material in case of cell contaminations. This material was processed and analyzed as the Tank 12H samples. However, a comparison of the laboratory results for the elements (Al, Si and Na) present in the synthetic sodalite mineral shows that the laboratory analytical results are in reasonable agreement with the expectations based on nominal synthetic sodalite recipe.

Dilute nitric acid and distilled and de-ionized water were used as the liquid reagent media and blanks for digestions performed in the Shielded Cells. The absence of radionuclides in these reference materials allowed the materials to additionally be utilized as blanks for radiochemical analyses.

Prior to the processing of the Tank 12H samples, sodalite aluminosilicate material in a 250-mL capacity polybottle was placed at a strategic location in the shielded cell to ensure that the material was exposed to the same cell environments as the Tank 12H samples. The sodalite aluminosilicate container held about 20 grams of the material. The container was opened when the Tank 12H samples were being air-dried or processed and closed at the end of each day of work in the Shielded Cells. At the end of each Tank 12H sample preparations or digestion (air-drying, aqua regia and peroxide fusion digestions), the sodalite aluminosilicate material was also prepared in the same manner as the preparation of Tank 12H samples and submitted for the same analyses as the actual samples from Tank 12H.

2.6 Leaching Characterization of Tank 12H Solids

Known quantities of homogenized Tank 12H composite solids were leached with distilled and de-ionized water and analyzed in triplicate. An average of 0.50 ± 0.01 grams of the composite solids was leached with 30.02 ± 0.02 grams of distilled and de-ionized water (average phase ratio of 60.04 mL/g). In this process, each solid fraction was thoroughly mixed with the given amount of distilled and de-ionized water, and the mixture was hand-agitated (Shielded Cell manipulator) for approximately five minutes and left to stand overnight before another agitation and filtering of the mixture using a 0.45 micron Nalgene filter unit. The filtrate was analyzed in triplicate for the requested anions. Thus, only surface-bound and water soluble constituents are assumed to be measured in the leachate analyses.

2.7 Analytical Narratives and Unforeseen Events Which May Have Affected Tank 12H Characterization.

Unforeseen activities which may have negatively impacted the characterization protocols for Tank 12H include the presence of relatively large amounts of Th-232 in the Tank 12H samples. This affected the analytical approaches used in the resin separations and Inductively Coupled Plasma–Mass Spectrometry (ICP-MS) analysis for radionuclides such as U-233, Th-230, and Th-229. As a result, the MDLs for these radionuclides were not met.

For the ICP-MS method, the issue of non-spectroscopic matrix effects was of concern due to the high levels of mercury in the Tank 12H digested samples. These matrix effects in the ICPMS plasma and sample introduction were noted through the evaluation of internal standard drift during analysis. The minimization of the drift through the implementation of extensive rinse cycles specifically on the mercury masses was an indication that mercury was an interference. The ability to apply lower dilution factors to achieve the best target limits for the customer was limited as a consequence of these effects.

Table 1. Summary Information on Tank 12H Sample Delivery to SRNL

Sample Location	Sample Identification	Date Sample Received at SRNL	Chain-of-Custody Number*	Comments
Floor	**T12-F-1**	August 11, 2014	Tk12–29/07/14	No appreciable material recovered
Floor	**T12-F-2**	August 11, 2014	Tk12–29/07/14	No appreciable material recovered
Floor	**T12-F-3**	August 11, 2014	Tk12–29/07/14	No appreciable material recovered
Floor	**T12-F-4**	August 11, 2014	Tk12–29/07/14	Adequate mass collected
Floor	**T12-F-5**	August 11, 2014	Tk12–29/07/14	Adequate mass collected
Floor	**T12-F-6**	August 11, 2014	Tk12–29/07/14	No appreciable material recovered
Floor	**T12-**F-1R	August 18, 2014	Tk12–13/08/14	Replacement sample; adequate mass collected
Floor	**T12-**F-2R	August 18, 2014	Tk12–13/08/14	Replacement sample; adequate mass collected
Floor	**T12-**F-3R	August 18, 2014	Tk12–29/07/14	Replacement sample; adequate mass collected
Mound	**T12-**M-L-1	August 27, 2014	Tk12–20/08/14	Adequate mass collected
Mound	**T12-**M-L-2	August 27, 2014	Tk12–20/08/14	Adequate mass collected
Mound	**T12-**M-L-3	August 27, 2014	Tk12–20/08/14	Adequate mass collected
Mound	**T12-**M-H-1	August 27, 2014	Tk12–20/08/14	Adequate mass collected
Mound	**T12-**M-H-2	August 27, 2014	Tk12–20/08/14	Adequate mass collected
Mound	**T12-**M-H-3	August 27, 2014	Tk12–20/08/14	Adequate mass collected
Cooling Coil	T12-R8-C-HIGH	July 23, 2014	Tk12–23/07/14	Upper elevation cooling coil sample Adequate mass collected
Cooling Coil	T12-R8-C-MID	July 21, 21014	Tk12– 7/18/14	Middle elevation cooling coil sample Adequate mass collected
Cooling Coil	T12-R8-C-LOW	July 21, 21014	Tk12–7/18/14	Lower end cooling coil sample Adequate mass collected

*Samples were transported and received in the SRNL Shielded Cell Operations under chain-of-Custody.

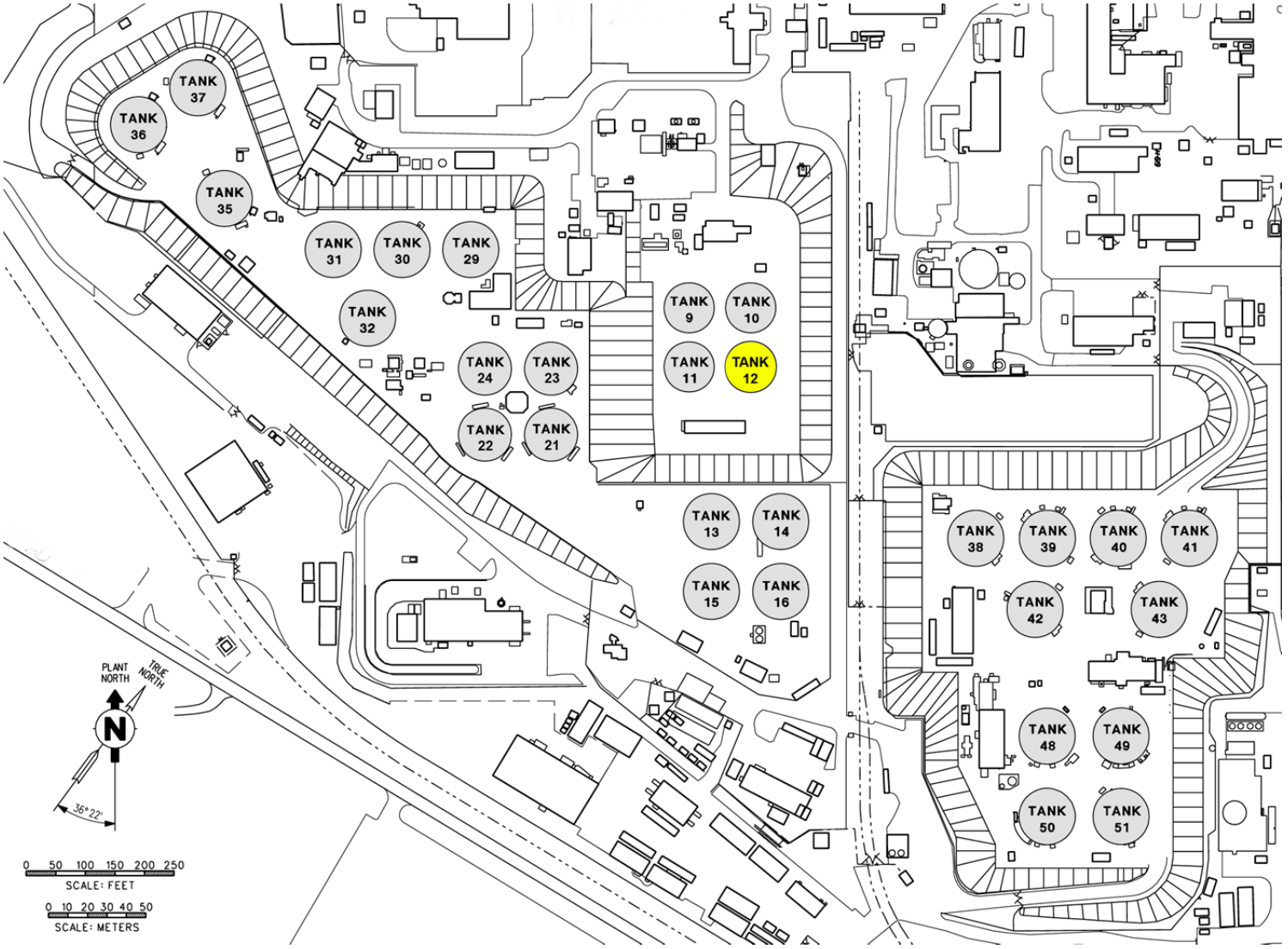


Figure 1. Location of Tank 12H Relative to Other Tanks in the H-Tank Farm

Note: For the Sample Locations, L and H prefixes indicate Mound samples, R suffixes indicate a Resample.

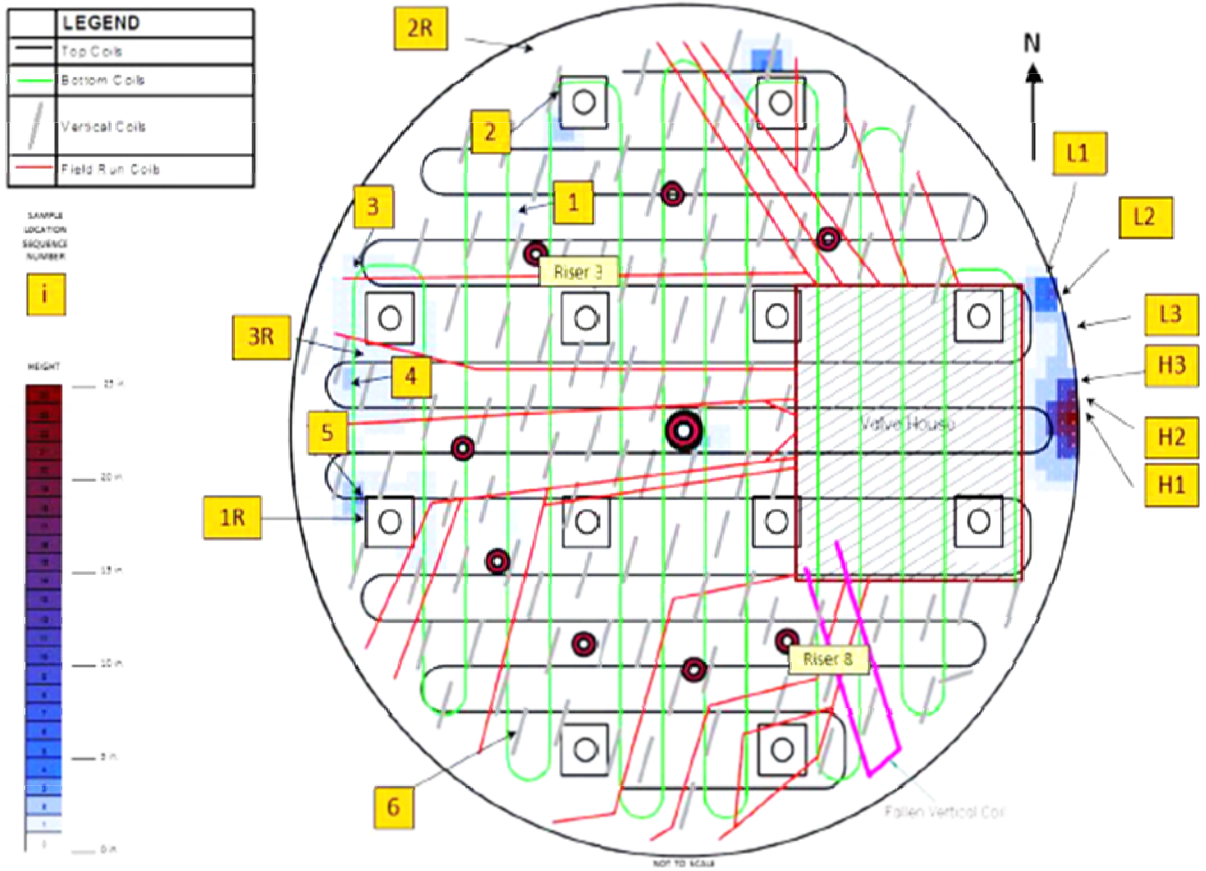


Figure 2. Tank 12H Sample Locations

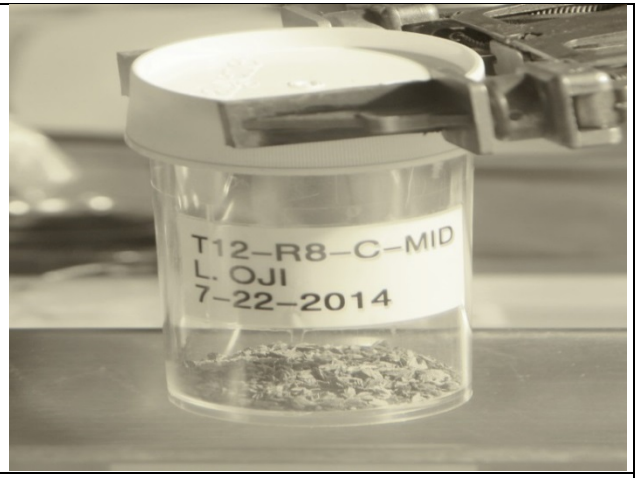
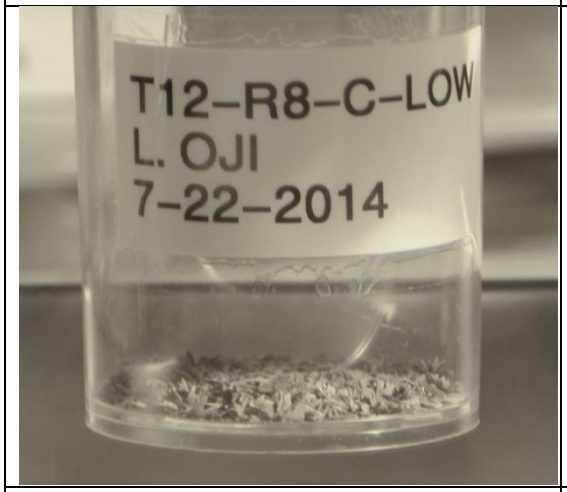
		
Insert A: Scale on a typical cooling coil pipe surface.	Insert B: Scraped scale sample from Tank 12H cooling coil-top portion of a cooling coil.	Insert C: Scraped scale sample from Tank 12H cooling coil-mid portion of a cooling coil.
	No image here.	
Insert D: Scraped scale sample from Tank 12H cooling coil-bottom portion of a cooling coil.		Insert E: All three cooling coil scale samples from high, mid and low portions of a typical cooling coil.

Figure 3. Cooling Coil Scrape Samples

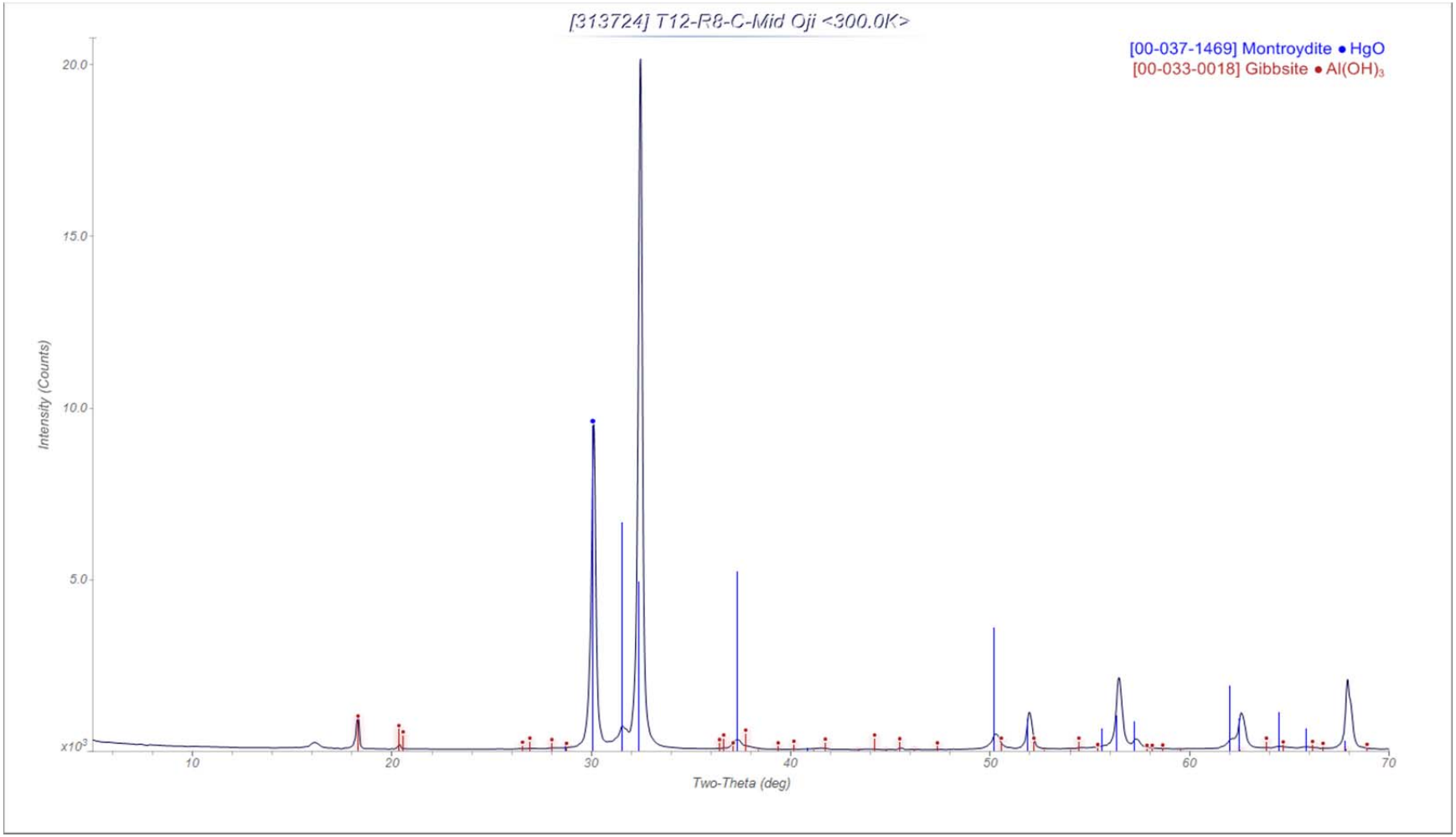


Figure 4 XRD Spectrum of T12-R8-C Mid Coil Scrape-Sample Material

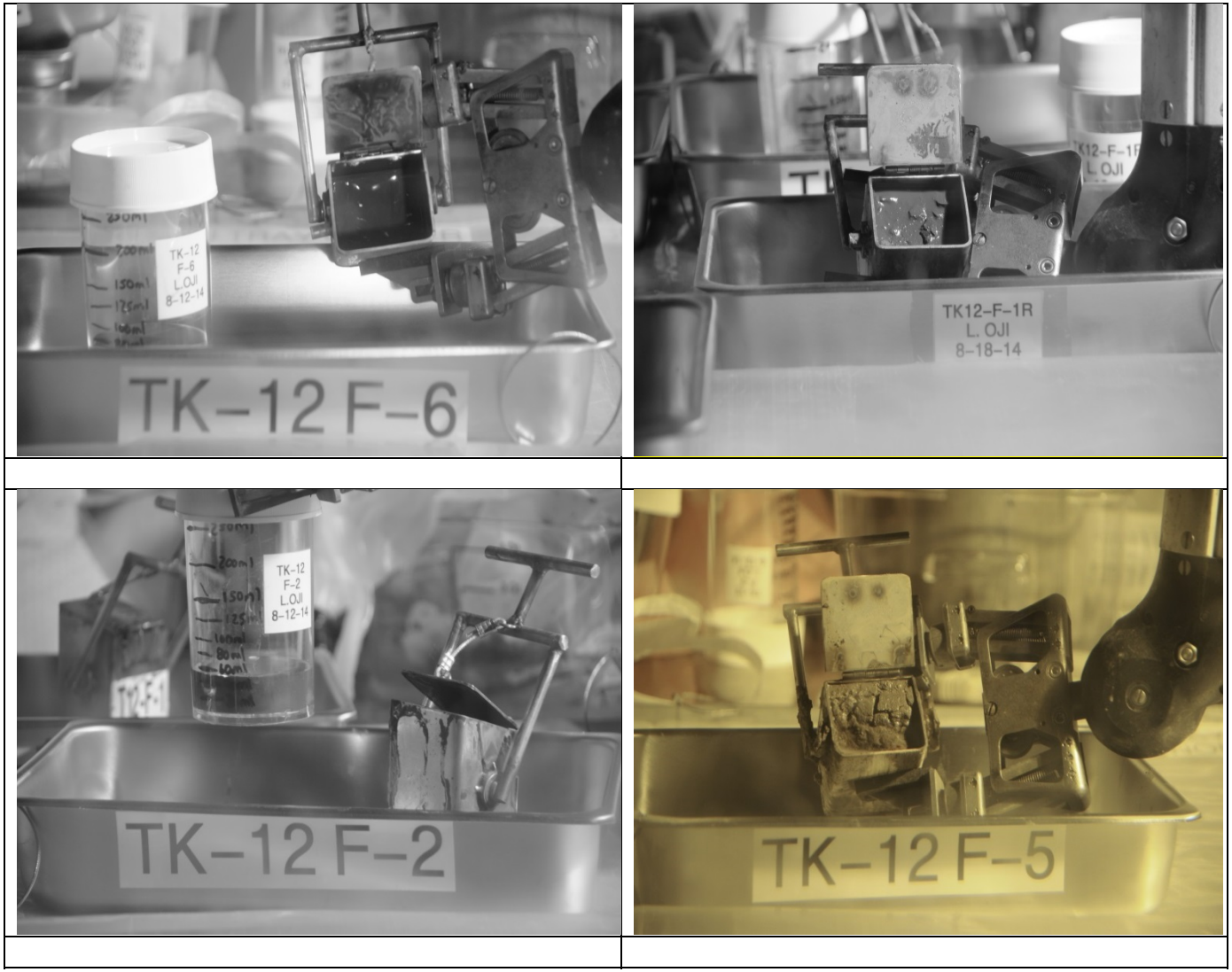


Figure 5 Photo Images of Select Tank 12H Floor Samples as Delivered to SRNL

A: Ground, homogenized and sieved Tank 12H floor solid fraction samples

B: Ground, homogenized and sieved Tank 12H Mound sample solids

Figure 6. Images of Ground, Homogenized and Sieved Tank 12H Floor and Mound Sample Solids

Figure 7. Ground, Homogenized and Sieved Tank 12H Sample Composites

Table 2. Tank 12 Floor Sample Solid Fractions

Sample ID	Total sample wt. after air-drying for 146 hr., g	Bulk density after air-drying for 146 hr., g/mL	Wt.% solids after air-drying for 146 hr.
T12-F-1	Insufficient sample material	Insufficient sample material	Insufficient sample material
T12-F-2	Insufficient sample material	Insufficient sample material	Insufficient sample material
T12-F-3	Insufficient sample material	Insufficient sample material	Insufficient sample material
T12-F-1R	148.2	1.68 ± 0.05	84.9 ± 2.9
T12-F-2R	19.6	0.93± 0.01	83.2 ± 3.6
T12-F-3R	67.9	1.11± 0.09	81.9 ± 2.0
T12-F-4	129.7	1.62 ± 0.06	85.5 ± 3.6
T12-F-5	170.2	1.97 ± 0.07	81.1 ± 1.5
T12-F-6	Insufficient sample material	Insufficient sample material	Insufficient sample material
		G-S-H[@] Sample Bulk density, g/mL	
T12-F-1R	Not Applicable	1.84 ± 0.04	
T12-F-2R	Not Applicable	1.08 ± 0.09	
T12-F-3R	Not Applicable	1.33 ± 0.08	
T12-F-4	Not Applicable	1.96 ± 0.03	
T12-F-5	Not Applicable	2.02 ± 0.05	
T12-F-6	Not Applicable	Insufficient sample material	

@ Ground, Sieved and Homogenized

Table 3. Tank 12 Mound Samples

Sample ID	Total sample wt. after air-drying for 150 hr., g	Bulk density after air-drying, g/mL		Wt.% solids (as-received)	Wt.% solids after 150 hr. air-drying
		After 24 hr.	After 150 hr.		
T12-M-L1	55.3	0.54 ± *0.09*	0.50± *0.06*	45.9 ± *1.6*	94.7 ± *1.6*
T12-M-L2	52.9	0.58 ± *0.05*	0.57 ± *0.06*	52.0 ± *3.0*	92.5 ± *1.5*
T12-M-L3	69.5	0.62 ± *0.06*	0.51 ± *0.04*	49.0 ± *0.8*	92.0 ± *0.9*
T12-M-H1	78.9	0.71 ± *0.06*	0.78 ± *0.07*	59.2 ± *2.6*	91.4 ± 2.3
T12-M-H2	97.4	0.68 ± *0.07*	0.59 ± *0.06*	50.6 ± *0.1*	94.5 ± 0.5
T12-M-H3	88.2	0.75 ± *0.08*	0.68 ± *0.03*	52.7 ± 2.4	92.5± *0.8*
	G-S-H@ Sample Bulk Density, g/mL				
T12-M-L1	0.55 ± 0.*04*	Not applicable	Not applicable	Not applicable	Not applicable
T12-M-L2	0.58 ± 0.*03*	Not applicable	Not applicable	Not applicable	Not applicable
T12-M-L3	0.60 ± 0.*02*	Not applicable	Not applicable	Not applicable	Not applicable
T12-M-H1	0.96 ± 0.*02*	Not applicable	Not applicable	Not applicable	Not applicable
T12-M-H2	0.82 ± 0.*04*	Not applicable	Not applicable	Not applicable	Not applicable
T12-M-H3	0.97 ± 0.*02*	Not applicable	Not applicable	Not applicable	Not applicable

@ Ground, Sieved and Homogenized

Table 4 Table Compositing Specifications and Mass Proportions for Tank 12H Samples

Composite Number	Sample Identification	Mass per Composite Volume	Approximate weight needed for generating 60*/70 g of composite material (g)	Amount of material weighed out for each sample (g)
Tank 12 Composite 1	M-L-1	0.09162	4.59	4.583
	M-H-1	0.12047	6.03	6.028
	F-4	0.53517	26.80	26.815
	F-5	0.65079	32.58	32.576
			Σ = 70	**Σ = 70.002**
Tank 12 Composite 2	M-L-2	0.13524	9.68	9.688
	M-H-2	0.15856	11.35	11.344
	F-2R	0.24815	17.76	17.760
	F-3R	0.29618	21.20	21.199
			Σ = 60*	**Σ = 59.991**
Tank 12 Composite 3	M-L-3	0.10887	5.81	5.819
	M-H-3	0.13169	7.03	7.040
	F-1R	0.54501	29.09	29.082
	F-4	0.52554	28.06	28.056
			Σ = 70.0	**Σ = 69.997**

* Only a 60 g of composite #2 sample mass could be created because of the limited amount of sample F-2R recovered.

Table 5. Tank 12H Composite Sample Bulk Density and Weight Percent Solids

Sample Composite ID	Bulk Densities, g/mL					Weight percent solids, wt%				
	Run 1	Run 2	Run 3	Ave.	Std. Dev.	Run 1	Run 2	Run 3	Ave.	Std. Dev.
Tank 12 Composite 1	1.41	1.38	1.40	**1.40**	*0.02*	84.6	86.8	86.4	**85.9**	*1.2*
Tank 12 Composite 2	1.08	1.10	1.13	**1.10**	*0.03*	85.6	89.5	87.3	**87.5**	*2.0*
Tank 12 Composite 3	1.34	1.32	1.33	**1.33**	*0.01*	85.2	87.5	85.8	**86.2**	*1.2*
5% Reference NaCl solution	NA	NA	NA	NA	NA	4.84	4.82	4.91	4.85	*0.05*

3.0 Results

Tank 12H Composite Sample Analysis

Laboratory analyses were performed on the three Tank 12H sample composites. A combination of routine dissolution/measurement techniques and "tailor-made" digestion/isolation/analysis methods were used to quantify several stable constituents (elements and anions) and about thirty-six radionuclides. Details of most of the analytical methodologies including weight percent solids and density determinations are summarized in Appendix B. Tank 12H elemental constituents, anions and radionuclides are presented in Tables 6-8, Tables 9-11, and Tables 12-14, respectively.

Appendix A-1 contains the SRNL Analytical Development Laboratory Information Management System (LIMS) numbers for tracking the analytical data presented in this report. The sample analysis completion dates are tracked in LIMS. Many digestion methods were performed in the SRNL Shielded Cells prior to taking representative sample aliquots out of the cells for analyses. Additionally, many of the initial separations for challenging radionuclide characterizations were performed in the Shielded Cells.

In the Tank 12H residual sample characterization results presented below, values preceded by "<" (less than sign) indicate values were below minimum detection limits (MDLs), and values proceeded by "≤" (less than or equal to sign) indicate that for replicates, at least one of the analysis values was above the instrument detection limit or MDL and at least one of the analysis values was below the detection limit or was an upper limit. Thus, where replicate analyses were both above and below the detection limit, the average of all replicates above and below the detection limit is given and a "≤ " sign precedes the average value. The standard deviations were calculated only for values that were all above the detection limits. The minimum detectable activity (MDA) is defined as the value above which instrument signal can be considered quantitative relative to the signal-to-noise ratio and the upper limit (UL) is defined as activity observed but biased high due to spectral interference or blank contamination. The detection limit (DL) as used in mass spectrometer or Inductively Coupled Plasma–Atomic Emission Spectroscopy (ICP-ES) analyses is equivalent to three times the standard deviation of the blank measurements.

The one sigma percent uncertainty for each radionuclide reported in the tables is based on the pooled estimate derived from the individual uncertainties for each replicate measurement for that radionuclide using an excel function, SQRT((SUMSQ(x_i)/n)), where n is the number of replicates and x_i is the individual uncertainty associated with each radionuclide for each run. Here it is assumed that the radio-analytical processes, be it counting or other techniques, are of the same precision for each individual measurement.

Occasionally, situations were encountered where the samples prepared and analyzed in triplicate gave mixed results with one or two of the triplicate analyses results being less than the MDA. In these cases, the reporting of the one sigma percent uncertainty is presented in a slightly different format. In this situation, the individual percent uncertainty associated with each run for that radionuclide is reported along with MDA, upper limit values or the detection limit (DL) values as indicated by the analytical method. For example, under the one sigma percent uncertainty column for the isotope Pa-231 in Table 1, the DL/10.1 designation implies that the one sigma percent uncertainty for Pa-231 in run 3 is reported with values above the detection limit and thus has a one sigma percent uncertainty of 10.1 percent. The measurements (runs 1 and 2) for Pa-231, which were below the detection limit, are assigned a DL. Similarly, in the analysis result for Cm-243 (runs 1, 2 and 3 in Table 14), the percent uncertainty is designated as MDA) since the three run results <1.22E-02, <1.88E-02 and <1.62E-02 μCi/g are considered less than the target MDA.

In some cases the preparations for analysis would involve the digestion of samples by both Aqua Regia Digestion (AQR) and Sodium Peroxide/Hydroxide Fusion (PF) digestion methods. The analyses results, however, occasionally are quite similar in magnitude. Thus, the data reported is based on the method with the least uncertainty in the results reported. In most cases the PF digestions have lower uncertainty values and are thus preferred and reported.

3.1 Data Quality and Presentations for Routine Radionuclide Constituents

The ICP-MS results are given for each atomic mass and in most cases each mass number represents only one isotope. An example of an exception is mass 238, since both uranium and plutonium are represented by this mass number. However, since the mass contribution of U-238 is significantly greater than that of Pu-238, the 238 signal is used to quantify U-238, not Pu-238. For this reason, Pu-238 was determined by the PUTTA method (chemical separation coupled with alpha spectroscopy). See Appendix B for summaries of the methods. In cases where ICP-MS and radiochemistry data give similar results for a species, the radiochemical method is typically selected due to better sensitivity, accuracy, and precision.

In this data presentation, the analysis DL for any analyte is considered met when the magnitude of the analytical result is less than or equal to that of the target detection limit as specified in the TTR. Typically, several of the analysis results for radionuclides, elements and anions were very close to the target detection limit because they were about the same order of magnitude as the target limits. For example, a detection limit of 4.0E-04 µCi/g is a factor of 4 higher than a desired target detection limit of 1.0E-04 µCi/g, but is considered as having the same order of magnitude. The analysis result for Am-242m in Tank 12H is a good example of the case described above (Table 12). In this case Am-242m analytical results for composite 1 runs 2 and 3, the results have the same order of magnitude as the detection limit but are 3 times higher than the detection limit.

However, when the analytical detection limit is one or more orders of magnitude above the target detection limit, the detection limit is not considered met. Thus, in this report the emphasis of not meeting the desired target detection limit has been put on those instances when the analytical results are one or more orders of magnitude above the target detection limit.

While many of the target MDLs, specified in the TTR and TTQAP were met for the routine radionuclide species characterized for Tank 12H composite samples, some were not met. The analytical results for U-233 and Cm-243 in all three Tank 12H composite samples, as shown in Tables 12-14, averaged one order of magnitude higher than the customer requested detection limits (1.0E-03 µCi/g for U-233 and 1.0E-03 µCi/g for Cm-243). Analytical results for Th-230 in composite sample 2 run 2 and composite sample 3 runs 2 and 3 also averaged about an order of magnitude higher than the requested detection limit of 9.0E-04 µCi/g.

The analytical results for Th-229 were typically one to two orders of magnitude higher than the target detection limit of 8.0E-05 µCi/g, and the analytical results for Cm-244 for two to three orders of magnitude higher than the target detection limit of 1.0E-03 µCi/g. In contrast, the target detection limit for Pa-231 was not met for most of the analyses; the detection limits average about an order of magnitude higher than the requested target of 9.0E-05 µCi/g.

In summary, the minimum target detection limits for Pa-231 and Th-230 were not consistently met while those for U-233, Cm-243, Th-229 and Cm-244 were not met in any of the three Tank 12H composite analyses.

Routine radionuclide analytical results were also compared between different methods used for characterization of Tank 12H composite samples, specifically comparing results from ICP-MS with

results from other methods of analyzing for the routine radionuclide. For example, analytical results for Pu-239 and Pu-240 can be obtained from ICP-MS and from a better analytical technique for these plutonium isotopes using Pu-tracer and plutonium extraction with thenoyltrifluoroacetone (TTA) followed by alpha spectrometry for Pu-239/240 then subsequent isotope quantification based on the Pu-239:Pu-240 ratio determined via ICP-MS. Similarly, analytical results for Tc-99 can also be obtained through ICP-MS and through counting techniques, which involve acid digestion of the sample and spiking of the sample with Tc-99m and extraction of the technetium species from the matrix using an Aliquat-336 based solid phase extractant. The extracted Tc-99 concentrations are then measured by liquid scintillation counting (LSC).

Using this dual analytical method approach, the analytical results for select Tank 12H composite routine radionuclide analytes (Tc-99, Pu-239 and Pu-240) have been summarized in Appendices A-6 through A-12 and the %RD for the values by the two different methods used to compare the quality of the data obtained by the two methods. These comparisons provide a basis for determining if the data obtained by the two different methods are in general agreement.

Appendix A-6 shows the Tc-99 analytical results for the three Tank 12H composite samples by ICP-MS and LSC. The analytical results cannot be compared directly, in terms of %RD, because the results from ICP-MS are all less than values while the LSC results for Tc-99 are all measurable values. However, it is worth pointing out that the ICP-MS analytical results point in the same direction as the more accurate analytical results by separations and LSC analysis. For example, the Tc-99 analytical result by LSC, 4.06E-03 μCi/g, is less than the ICP-MS reported result of <2.10E-02 μCi/g and points in the same direction as the ICP-MS analytical result for this sample (sample TK 12H Composite 1 run 1; LIMS # 300314633). Thus, the analytical results from these two methods for Tc-99 are consistent with one another.

Appendices A-7 and A-9 also show a summary of the comparison data for Pu-239 and Pu-240 analytical results by two different methods; raw ICP-MS and a hybrid method utilizing separations and combinations of ICP-MS and counting measurements. The raw ICP-MS results for Pu-239 and Pu-240 are about the same order of magnitude as the separations data for these radionuclides. The average %RD (%RD defined as [difference/mean]*100) for the two methods for Pu-239 and Pu-240 in the three Tank 12H composite analyses results are, respectively, 7.4 ± 4.7 and 7.3 ± 4.5. Given that the separations method typically provides higher quality Pu-239 and Pu-240 data with relatively small uncertainties (as measured by the standard deviation), the analytical results from the two methods are consistent. Above all, the analytical results from these two methods for both Pu-239 and Pu-240 are within the SRNL-AD acceptable analytical error margins of 20% for these analytes by the two methods.

3.2 Data Quality and Presentations for Elemental Constituents (Cations and Anions)

Analytical results for the elemental analyses of the ARG reference material were compared against the known reference values[7] in Appendix A-2. Elements with concentrations less than 0.1 wt% (Ba, Cr, Cu, Sr and Zn) were not included in Appendix A-2 because their concentrations could be influenced by trace reagent impurities. Looking at the analytical results for the 12 select elemental constituents of the ARG reference sample, the average relative deviation (RD) between the measured and reference values was less than 10%.

Elemental analytical results were also compared between different methods used for characterization of Tank 12H composite samples, specifically comparing results from ICP-MS with results from ICP-ES. The concentrations of select elements (Ba, Co and La) were calculated from ICP-MS information and the resulting concentration values compared with the ICP-ES corresponding results presented in this report. Typical calculations are shown in Appendix A-3 for Ba, Appendix A-4 for Co and Appendix A-5 for La.

The average RD between ICP-MS and ICP-ES analytical results for Ba, Co and La were, respectively, 14.9 ± 5.8%, 4.1 ± 2.7% and 2.0 ± 0.1.7 %. These comparison results are summarized in Appendix A-3 through A-5 and show that ICP-ES analytical results are quite comparable to the corresponding ICP-MS data for these select metals since the RDs are all below the 20% margin of error expected from any of these analytical tools.

Analysis for stable iodine (I-127) by mass spectroscopy was preferred over analyses by ion chromatography (IC). Tank 12H sample composite leachates were analyzed for I-127 by mass spectroscopy, assuming that I-127 dominated the isotope 127 mass signal. (This was based on the expectation that all other elements with mass 127 [Xe-127, Sn-127, Cs-127, Ba-127, La-127, In-127 etc.] have short half-lives ranging from milliseconds to a few days). Thus, the total stable iodine reported in Tables 9-11 is based on mass spectroscopy data for mass-127. The total stable and radioactive iodine in each Tank 12H composite sample is approximated by adding the mass 127 stable iodine concentrations to the mass 129 radioactive iodine concentrations.

With the exception of analyses results for fluoride and phosphate in the leachates, the anion analysis detection limits for Tank 12H composite sample leachates were met in all cases as shown in Tables 9-11. Analysis results for phosphate in all the Tank 12H composite sample leachates were about an order of magnitude higher than the target detection limit of 9.0E-04 µCi/g. Fluoride analysis results for all Tank 12H leachates were about the same order of magnitude (<2.99E-03 to <3.01E-03 µCi/g) as the target detection limit, but a factor of three higher than the desired target detection limit of 1.0E-03 µCi/g.

Elemental silver (Ag) data presented in the report, in Tables 6-8, are based on ICP-MS data (sum of masses 107 and 109) for PF digestions with LIMS numbers: 300314633-635, 300314636-638 and 300314639-641. Similarly, elemental antimony (Sb) data presented in the report, in Tables 6-8, are based on ICP-MS data (sum of masses 121 and 123) for AQR digestions with LIMS numbers: 300314645-647, 300314648-650 and 300314651-653.

3.3 Statistical Analysis

Statistical characterization of analyte concentrations in the Tank 12H residual solids were performed on 36 radionuclides, 22 elemental constituents, and 7 anions. The statistical methods and their applications to these analytes are described in Appendix D. Data and summary tables for these statistical results are presented in Appendix E.

Table 6 Elemental Constituents in Tank 12H Composite 1 Sample

Analytes	Composite-1 Run-1; wt%	Composite-1 Run-2; wt%	Composite-1 Run-3; wt%	Average wt%	Std. Dev.	Target Detection Limit (wt %)
Ag*	5.33E-02	5.54E-02	5.34E-02	**5.40E-02**	*1.18E-03*	5.0E-04
Al	6.22E+00	5.29E+00	6.12E+00	**5.88E+00**	*5.11E-01*	1.0E-01
B	1.03E-01	1.10E-01	1.10E-01	**1.08E-01**	*4.04E-03*	5.0E-03
Ba	6.33E-02	6.67E-02	6.80E-02	**6.60E-02**	*2.43E-03*	4.0E-03
Be	6.39E-05	7.71E-05	8.19E-05	**7.43E-05**	*9.32E-06*	Not specified
Ca	1.07E-01	1.12E-01	1.16E-01	**1.12E-01**	*4.51E-03*	Not specified
Cd	2.67E-03	2.84E-03	2.74E-03	**2.75E-03**	*8.54E-05*	1.0E-03
Ce	1.48E-01	1.67E-01	1.54E-01	**1.56E-01**	*9.71E-03*	NA
Co	3.71E-03	3.99E-03	4.05E-03	**3.92E-03**	*1.81E-04*	6.0E-05
Cr	5.72E-02	6.15E-02	5.82E-02	**5.90E-02**	*2.25E-03*	4.0E-03
Cu	1.33E-01	1.39E-01	1.43E-01	**1.38E-01**	*5.03E-03*	2.0E-03
Fe	3.22E+01	3.22E+01	3.27E+01	**3.24E+01**	*2.89E-01*	3.0E-01
K	<1.26E-02	<1.22E-02	<1.23E-02	**<1.24E-02**	-	Not specified
La	9.10E-02	9.70E-02	9.16E-02	**9.32E-02**	*3.31E-03*	Not specified
Li	8.54E-04	1.08E-03	9.82E-04	**9.72E-04**	*1.13E-04*	Not specified
Mg	9.24E-03	9.96E-03	1.10E-02	**1.01E-02**	*8.85E-04*	Not specified
Mn	1.17E+00	1.17E+00	1.19E+00	**1.18E+00**	*1.15E-02*	6.0E-02
Mo	1.52E-03	<1.33E-03	<1.34E-03	**≤1.40E-03**	-	3.0E-03
Na	6.57E-01	4.56E-01	4.86E-01	**5.33E-01**	*1.08E-01*	Not specified
Ni	7.27E-01	7.22E-01	7.36E-01	**7.28E-01**	*7.09E-03*	6.0E-03
P	5.36E-02	5.41E-02	5.68E-02	**5.48E-02**	*1.72E-03*	Not specified
Pb	3.46E-02	3.57E-02	3.64E-02	**3.56E-02**	*9.07E-04*	5.0E-03
S	<3.59E-01	<3.47E-01	<3.51E-01	**<3.52E-01**	-	Not specified
Sb[@]	<2.00E-04	<1.93E-04	<1.95E-04	**<1.96E-04**	-	6.0E-04
Si	1.15E-01	1.28E-01	1.44E-01	**1.29E-01**	*1.45E-02*	Not specified
Sn	<7.62E-03	<7.35E-03	<7.44E-03	**<7.47E-03**	-	Not specified
Sr	4.48E-02	4.74E-02	4.79E-02	**4.67E-02**	*1.66E-03*	5.0E-04
Th	3.47E+00	3.36E+00	3.48E+00	**3.44E+00**	*6.66E-02*	Not specified
Ti	9.77E-03	1.03E-02	1.04E-02	**1.02E-02**	*3.39E-04*	Not specified
U	4.11E-02	4.67E-02	4.40E-02	**4.39E-02**	*2.80E-03*	9.0E-03
Zn	3.43E-02	3.66E-02	3.53E-02	**3.54E-02**	*1.15E-03*	1.0E-03
Zr	9.87E-02	1.02E-01	1.08E-01	**1.03E-01**	*4.71E-03*	Not specified
As	2.04E-04	1.85E-04	1.93E-04	**1.94E-04**	*9.54E-06*	2.5E-04
Hg	2.01E+01	1.94E+01	1.86E+01	**1.94E+01**	*7.51E-01*	1.0E-03
Se	<2.99E-04	<2.89E-04	<2.92E-04	**<2.93E-04**	-	5.0E-04

*Ag wt% values were derived from ICP-MS measurements (mass 107 +109) and may be biased high by the presence of Pd-107 fission product.
[@] Sb wt% values were derived from ICP-MS measurements (mass 121 + 123).

Table 7. Elemental Constituents in Tank 12H Composite 2 Sample

Analytes	Composite-2 Run-1; wt%	Composite-2 Run-2; wt%	Composite-2 Run-3; wt%	Average wt%	Std. Dev.	Target Detection Limit (wt %)
***Ag**	8.98E-02	8.87E-02	9.24E-02	**9.03E-02**	*1.90E-03*	5.0E-04
Al	1.02E+01	9.82E+00	8.44E+00	**9.49E+00**	*9.26E-01*	1.0E-01
B	6.43E-02	6.58E-02	7.01E-02	**6.67E-02**	*3.01E-03*	5.0E-03
Ba	6.85E-02	7.37E-02	7.35E-02	**7.19E-02**	*2.95E-03*	4.0E-03
Be	<1.53E-05	<1.59E-05	<1.57E-05	**<1.56E-05**	-	Not specified
Ca	1.04E-01	1.08E-01	1.12E-01	**1.08E-01**	*4.00E-03*	Not specified
Cd	1.86E-03	1.72E-03	1.91E-03	**1.83E-03**	*9.85E-05*	1.0E-03
Ce	2.95E-01	2.83E-01	3.03E-01	**2.94E-01**	*1.01E-02*	Not specified
Co	5.56E-03	6.15E-03	6.07E-03	**5.93E-03**	*3.20E-04*	6.0E-05
Cr	3.17E-02	3.23E-02	3.35E-02	**3.25E-02**	*9.17E-04*	4.0E-03
Cu	1.75E-01	1.90E-01	1.88E-01	**1.84E-01**	*8.14E-03*	2.0E-03
Fe	2.26E+01	2.14E+01	2.23E+01	**2.21E+01**	*6.24E-01*	3.0E-01
K	<1.21E-02	<1.25E-02	<1.24E-02	**<1.23E-02**	-	Not specified
La	1.39E-01	1.39E-01	1.46E-01	**1.41E-01**	*4.04E-03*	Not specified
Li	1.38E-03	1.71E-03	1.71E-03	**1.60E-03**	*1.91E-04*	Not specified
Mg	<3.30E-03	<3.42E-03	<3.38E-03	**<3.37E-03**	-	Not specified
Mn	1.59E+00	1.61E+00	1.55E+00	**1.58E+00**	*3.06E-02*	6.0E-02
Mo	1.39E-03	<1.37E-03	1.65E-03	**≤1.47E-03**	-	3.0E-03
Na	7.06E-01	6.45E-01	6.11E-01	**6.54E-01**	*4.81E-02*	Not specified
Ni	1.06E+00	1.03E+00	1.01E+00	**1.03E+00**	*2.52E-02*	6.0E-03
P	9.48E-02	9.94E-02	1.01E-01	**9.84E-02**	*3.22E-03*	Not specified
Pb	3.01E-02	3.05E-02	3.39E-02	**3.15E-02**	*2.09E-03*	5.0E-03
S	<3.45E-01	<3.58E-01	<3.54E-01	**<3.52E-01**	-	Not specified
Sb[@]	<1.92E-04	<1.99E-04	<1.96E-04	**<1.96E-04**	-	6.0E-04
Si	1.54E-01	1.44E-01	1.51E-01	**1.50E-01**	*5.13E-03*	Not specified
Sn	<7.32E-03	<7.59E-03	<7.50E-03	**<7.47E-03**	-	Not specified
Sr	3.65E-02	3.80E-02	3.92E-02	**3.79E-02**	*1.35E-03*	5.0E-04
Th	8.18E+00	8.40E+00	7.95E+00	**8.18E+00**	*2.25E-01*	Not specified
Ti	5.24E-03	5.28E-03	5.68E-03	**5.40E-03**	*2.43E-04*	Not specified
U	1.41E-01	1.60E-01	1.51E-01	**1.51E-01**	*9.50E-03*	9.0E-03
Zn	3.19E-02	3.37E-02	3.45E-02	**3.34E-02**	*1.33E-03*	1.0E-03
Zr	1.43E-01	1.52E-01	1.58E-01	**1.51E-01**	*7.55E-03*	Not specified
As	2.65E-04	2.50E-04	2.24E-04	**2.46E-04**	*2.07E-05*	2.5E-04
Hg	1.18E+01	1.17E+01	1.16E+01	**1.17E+01**	*1.00E-01*	1.0E-03
Se	<2.88E-04	<2.98E-04	<2.95E-04	**<2.94E-04**	-	5.0E-04

*Ag wt% values were derived from ICP-MS measurements (mass 107 +109) and may be biased high by the presence of Pd-107 fission product.
[@] Sb wt% values were derived from ICP-MS measurements (mass 121 + 123).

Table 8. Elemental Constituents in Tank 12H Composite 3 Sample

Analytes	Composite-3 Run-1; wt%	Composite-3 Run-2; wt%	Composite-3 Run-3; wt%	Average wt%	Std. Dev.	Target Detection Limit (wt %)
***Ag**	3.68E-02	3.83E-02	3.70E-02	**3.74E-02**	*8.14E-04*	5.0E-04
Al	5.73E+00	5.69E+00	6.25E+00	**5.89E+00**	*3.12E-01*	1.0E-01
B	1.12E-01	1.22E-01	1.13E-01	**1.16E-01**	*5.51E-03*	5.0E-03
Ba	6.70E-02	7.03E-02	6.85E-02	**6.86E-02**	*1.65E-03*	4.0E-03
Be	1.30E-04	2.07E-04	1.33E-04	**1.57E-04**	*4.36E-05*	Not specified
Ca	1.18E-01	1.23E-01	1.20E-01	**1.20E-01**	*2.52E-03*	Not specified
Cd	2.86E-03	3.02E-03	2.89E-03	**2.92E-03**	*8.50E-05*	1.0E-03
Ce	1.26E-01	1.31E-01	1.22E-01	**1.26E-01**	*4.51E-03*	Not specified
Co	3.11E-03	3.16E-03	3.44E-03	**3.24E-03**	*1.78E-04*	6.0E-05
Cr	5.71E-02	6.21E-02	5.68E-02	**5.87E-02**	*2.98E-03*	4.0E-03
Cu	1.36E-01	1.42E-01	1.38E-01	**1.39E-01**	*3.06E-03*	2.0E-03
Fe	3.48E+01	3.63E+01	3.61E+01	**3.57E+01**	*8.14E-01*	3.0E-01
K	<1.24E-02	<1.21E-02	<1.24E-02	**<1.23E-02**	**-**	Not specified
La	7.71E-02	8.24E-02	8.01E-02	**7.99E-02**	*2.66E-03*	Not specified
Li	9.15E-04	9.64E-04	9.81E-04	**9.53E-04**	*3.43E-05*	Not specified
Mg	1.74E-02	1.93E-02	1.58E-02	**1.75E-02**	*1.75E-03*	Not specified
Mn	1.33E+00	1.26E+00	1.40E+00	**1.33E+00**	*7.00E-02*	6.0E-02
Mo	1.53E-03	<1.32E-03	1.41E-03	**≤1.42E-03**	-	3.0E-03
Na	4.04E-01	3.97E-01	4.67E-01	**4.23E-01**	*3.86E-02*	Not specified
Ni	5.56E-01	5.34E-01	5.76E-01	**5.55E-01**	*2.10E-02*	6.0E-03
P	5.36E-02	5.56E-02	5.44E-02	**5.45E-02**	*1.01E-03*	Not specified
Pb	3.55E-02	3.76E-02	3.53E-02	**3.61E-02**	*1.27E-03*	5.0E-03
S	<3.55E-01	<3.46E-01	<3.53E-01	**<3.51E-01**	-	Not specified
Sb[@]	<1.97E-04	<1.92E-04	<1.96E-04	**<1.95E-04**	-	6.0E-04
Si	1.75E-01	1.71E-01	1.80E-01	**1.75E-01**	*4.51E-03*	Not specified
Sn	<7.53E-03	<7.33E-03	<7.49E-03	**<7.45E-03**	-	Not specified
Sr	4.74E-02	5.13E-02	4.82E-02	**4.90E-02**	*2.06E-03*	5.0E-04
Th	3.30E+00	3.07E+00	3.42E+00	**3.26E+00**	*1.78E-01*	Not specified
Ti	1.24E-02	1.31E-02	1.16E-02	**1.24E-02**	*7.51E-04*	Not specified
U	6.96E-02	6.67E-02	6.97E-02	**6.87E-02**	*1.70E-03*	9.0E-03
Zn	2.94E-02	3.09E-02	3.15E-02	**3.06E-02**	*1.08E-03*	1.0E-03
Zr	1.01E-01	1.06E-01	9.91E-02	**1.02E-01**	*3.56E-03*	Not specified
As	1.95E-04	2.19E-04	2.12E-04	**2.09E-04**	*1.23E-05*	2.5E-04
Hg	1.54E+01	1.68E+01	1.50E+01	**1.57E+01**	*9.45E-01*	1.0E-03
Se	<2.96E-04	<2.88E-04	<2.94E-04	**<2.93E-04**	-	5.0E-04

*Ag wt% values were derived from ICP-MS measurements (mass 107 +109) and may be biased high by the presence of Pd-107 fission product.
[@] Sb wt% values were derived from ICP-MS measurements (mass 121 + 123).

Table 9. Anions Leached Per Gram of Tank 12H Composite 1 Sample; wt %

Analytes	Run-1	Run-2	Run-3	Average	Std. Dev.	Target Detection Limit, (TDL) (wt%)
Fluoride, F^{-1}	<3.04E-03	<3.06E-03	<2.94E-03	**<3.01E-03**	-	1.0E-03
Formate, CHO_2^{-1}	3.65E-03	3.37E-03	3.41E-03	**3.48E-03**	*1.54E-04*	Not specified
Chloride , Cl^{-1}	1.64E-02	1.72E-02	1.76E-02	**1.71E-02**	*5.96E-04*	1.0E-02
Nitrite , NO_2^{-1}	4.63E-03	4.29E-03	4.17E-03	**4.36E-03**	*2.37E-04*	3.0E-01
Bromide, Br^{-1}	<3.04E-03	<3.06E-03	<2.94E-03	**<3.01E-03**	-	Not specified
Nitrate , NO_3^{-1}	1.46E-02	1.59E-02	1.41E-02	**1.49E-02**	*9.43E-04*	3.0E-02
Phosphate, PO_4^{-3}	<3.04E-03	<3.06E-03	<2.94E-03	**<3.01E-03**	-	9.0E-04
Sulfate, SO_4^{-2}	5.30E-02	4.96E-02	4.88E-02	**5.04E-02**	*2.23E-03*	4.0E-03
Oxalate, $C_2O_4^{-2}$	4.75E-01	4.53E-01	4.41E-01	**4.56E-01**	*1.73E-02*	Not specified
Iodine, I-127	1.22E-04	1.36E-04	1.07E-04	**1.22E-04**	*1.45E-05*	Not specified
Iodine, I-129	2.78E-03	2.78E-03	2.51E-03	**2.69E-03**	*1.56E-04*	Not specified
Total Iodine	2.90E-03	2.92E-03	2.62E-03	**2.81E-03**	*1.69E-04*	5.0E-05

Table 10. Anions Leached Per Gram of Tank 12H Composite 2 Sample; wt %

Analytes	Run-1	Run-2	Run-3	Average	Std. Dev.	Target Detection Limit, (wt%)
Fluoride, F^{-1}	<3.02E-03	<3.06E-03	<2.94E-03	**<3.01E-03**	-	1.0E-03
Formate, CHO_2^{-1}	5.56E-03	5.21E-03	5.05E-03	**5.27E-03**	*2.63E-04*	Not specified
Chloride , Cl^{-1}	1.75E-02	1.65E-02	1.53E-02	**1.64E-02**	*1.14E-03*	1.0E-02
Nitrite , NO_2^{-1}	8.95E-03	9.19E-03	8.40E-03	**8.84E-03**	*4.05E-04*	3.0E-01
Bromide, Br^{-1}	<3.02E-03	<3.06E-03	<2.94E-03	**<3.01E-03**	-	Not specified
Nitrate , NO_3^{-1}	1.51E-02	1.84E-02	1.47E-02	**1.61E-02**	*2.02E-03*	3.0E-02
Phosphate, PO_4^{-3}	<3.02E-03	<3.06E-03	<2.94E-03	**<3.01E-03**	-	9.0E-04
Sulfate, SO_4^{-2}	8.47E-02	9.19E-02	8.22E-02	**8.62E-02**	*5.02E-03*	4.0E-03
Oxalate, $C_2O_4^{-2}$	5.93E-01	6.37E-01	5.81E-01	**6.04E-01**	*2.94E-02*	Not specified
Iodine, I-127	2.51E-04	2.23E-04	2.03E-04	**2.26E-04**	*2.41E-05*	Not specified
Iodine, I-129	3.39E-03	2.63E-03	2.52E-03	**2.85E-03**	*4.74E-04*	Not specified
Total Iodine	3.64E-03	2.85E-03	2.72E-03	**3.07E-03**	*4.97E-04*	5.0E-05

Table 11. Anions Leached Per Gram of Tank 12H Composite 3 Sample; wt %

Analytes	Run-1	Run-2	Run-3	Average	Std. Dev.	Target Detection Limit, (wt%)
Fluoride, F^{-1}	<3.06E-03	<2.97E-03	<2.93E-03	**<2.99E-03**	-	1.0E-03
Formate, CHO_2^{-1}	3.18E-03	4.04E-03	3.16E-03	**3.46E-03**	*5.04E-04*	Not specified
Chloride , Cl^{-1}	1.47E-02	1.49E-02	1.58E-02	**1.51E-02**	*6.20E-04*	1.0E-02
Nitrite , NO_2^{-1}	3.91E-03	4.52E-03	3.99E-03	**4.14E-03**	*3.32E-04*	3.0E-01
Bromide, Br^{-1}	<3.06E-03	<2.97E-03	<2.93E-03	**<2.99E-03**	-	Not specified
Nitrate , NO_3^{-1}	1.34E-02	1.25E-02	1.29E-02	**1.29E-02**	*4.80E-04*	3.0E-02
Phosphate, PO_4^{-3}	<3.06E-03	<2.97E-03	<2.93E-03	**<2.99E-03**	-	9.0E-04
Sulfate, SO_4^{-2}	9.78E-02	1.13E-01	8.79E-02	**9.95E-02**	*1.26E-02*	4.0E-03
Oxalate, $C_2O_4^{-2}$	5.87E-01	6.42E-01	5.57E-01	**5.95E-01**	*4.33E-02*	Not specified
Iodine, I-127	7.73E-05	8.72E-05	8.30E-05	**8.25E-05**	*4.97E-06*	Not specified
Iodine, I-129	2.71E-03	2.05E-03	1.82E-03	**2.19E-03**	*4.62E-04*	Not specified
Total Iodine	2.79E-03	2.14E-03	1.90E-03	**2.28E-03**	*4.58E-04*	5.0E-05

Table 12 Radiological Constituent for Tank 12H Composite 1 Sample

Radionuclide	Run 1, µCi/g	Run 2 µCi/g	Run 3 µCi/g	Average µCi/g	Std. Dev.	One Sigma %Uncert.	T DL, µCi/g
C-14	<5.86E-04	<5.45E-04	<5.05E-04	**<5.45E-04**	-	mda	1.0E-01
Co-60	7.12E-02	6.85E-02	6.94E-02	**6.97E-02**	*1.38E-03*	7.6	Not specified
Ni-59	1.81E-01	1.66E-01	1.76E-01	**1.74E-01**	*7.57E-03*	10.0	5.0E-02
Ni-63	1.78E+01	1.83E+01	1.84E+01	**1.82E+01**	*3.00E-01*	10.0	1.0E-01
Sr-90	1.71E+04	1.58E+04	1.81E+04	**1.70E+04**	*1.15E+03*	12.6	1.0E-03
Y-90	1.71E+04	1.58E+04	1.81E+04	**1.70E+04**	*1.15E+03*	12.6	1.0E-03
Zr-93	3.74E-01	3.47E-01	3.78E-01	**3.67E-01**	*1.72E-02*	20	1.0E-03
Nb-94	<9.41E-04	<1.05E-03	<8.74E-04	**<9.55E-04**	-	mda	3.0E-03
Tc-99	4.06E-03	3.27E-03	4.95E-03	**4.09E-03**	*8.45E-04*	8.9	1.0E-03
Sn-126	1.58E-02	1.81E-02	1.73E-02	**1.71E-02**	*1.17E-03*	17.0	1.0E-03
Sn-121m	1.01E+00	9.14E-01	1.17E+00	**1.03E+00**	*1.27E-01*	9.5	Not specified
I-129	4.91E-03	4.91E-03	4.43E-03	**4.75E-03**	*2.76E-04*	5.0	1.0E-05
Cs-135	<1.00E-05	<2.89E-05	<5.90E-05	**<3.26E-05**	-	UL	1.0E-04
Cs-137	4.86E+00	5.77E+00	6.58E+00	**5.74E+00**	*8.56E-01*	7.6	1.0E-03
Ba-137m	4.60E+00	5.45E+00	6.22E+00	**5.43E+00**	*8.10E-01*	7.6	1.0E-03
Eu-152	6.58E-01	6.94E-01	6.85E-01	**6.79E-01**	*1.88E-02*	5.0	Not specified
Eu-154	8.06E+00	8.60E+00	8.38E+00	**8.35E+00**	*2.72E-01*	5.0	Not specified
Eu-155	<9.14E-01	<3.56E-01	<5.54E-01	**<6.08E-01**	-	UL	Not specified
Ra-226	<7.75E-04	<4.68E-04	<7.25E-04	**<6.56E-04**	-	mda	9.0E-04
Ra-228	3.78E-03	3.84E-03	3.43E-03	**3.68E-03**	*2.24E-04*	8.5	1.0E-03
Th-229	<1.77E-03	<4.10E-03	<4.13E-04	**<2.09E-03**	-	UL	8.0E-05
Th-230	<3.52E-04	<5.68E-04	<4.15E-04	**<4.45E-04**	-	UL/mda	9.0E-04
Th-232	3.87E-03	4.27E-03	3.99E-03	**4.04E-03**	*2.02E-04*	1.7	5.0E-05
Pa-231	<2.21E-03	<2.22E-03	1.55E-03	**≤1.99E-03**	-	DL/10.1	9.0E-05
U-232	3.36E-03	2.79E-03	2.76E-03	**2.97E-03**	*3.37E-04*	25.9	1.0E-04
U-233	<3.39E-02	<3.89E-02	<3.40E-02	**<3.56E-02**	-	UL	1.0E-03
U-234	4.15E-03	4.53E-03	4.55E-03	**4.41E-03**	*2.25E-04*	20.0	1.0E-03
U-235	1.73E-05	1.94E-05	1.77E-05	**1.81E-05**	*1.12E-06*	20.0	1.0E-04
U-236	6.93E-05	7.35E-05	7.09E-05	**7.12E-05**	*2.12E-06*	20.0	Not specified
U-238	2.21E-04	2.38E-04	2.21E-04	**2.27E-04**	*9.81E-06*	20.0	1.0E-03
Np-237	8.39E-03	8.46E-03	8.32E-03	**8.39E-03**	*7.05E-05*	0.8	1.0E-03
Pu-238	6.94E+01	6.80E+01	6.49E+01	**6.74E+01**	*2.31E+00*	5.4	1.0E-03
Pu-239	2.98E+00	2.92E+00	2.83E+00	**2.91E+00**	*7.52E-02*	20.0	1.0E-03
Pu-240	1.05E+00	1.03E+00	9.77E-01	**1.02E+00**	*3.58E-02*	20.0	1.0E-03
Pu-239/240	4.02E+00	3.95E+00	3.81E+00	**3.93E+00**	*1.09E-01*	5.6	Not specified
Pu-241	1.63E+01	1.43E+01	1.24E+01	**1.43E+01**	*1.96E+00*	15.1	1.0E-03
Pu-242	1.26E-03	1.31E-03	1.27E-03	**1.28E-03**	*2.74E-05*	20.0	Not specified
Pu-244	<2.23E-07	<1.55E-07	<1.41E-07	**<1.73E-07**	-	DL	Not specified
Am-241	1.03E+01	1.04E+01	1.01E+01	**1.03E+01**	*1.58E-01*	5.0	1.0E-03
Am-242m	4.27E-03	<3.89E-03	<2.24E-03	**≤3.46E-03**	-	27.2/mda	1.0E-03
Am-243	1.79E-02	1.92E-02	1.51E-02	**1.74E-02**	*2.07E-03*	11.3	1.0E-03
Cm-242	3.53E-03	<3.21E-03	<1.85E-03	**≤2.86E-03**	-	27.2/mda	Not specified
Cm-243	<1.53E-02	<1.59E-02	<1.46E-02	**<1.53E-02**	-	mda	1.0E-03
Cm-244	<4.18E-01	<4.55E-01	<4.00E-01	**<4.24E-01**		UL	1.0E-03
Cm-245	<7.48E-05	<8.15E-05	<6.67E-05	**<7.43E-05**	-	UL	1.0E-03

Table 13 Radiological Constituent for Tank 12H Composite 2 Sample

Radionuclide	Run 1, µCi/g	Run 2 µCi/g	Run 3 µCi/g	Average µCi/g	Std. Dev.	One Sigma %Uncert.	TDL, µCi/g
C-14	<7.79E-04	<7.03E-04	<6.98E-04	**<7.27E-04**	-	mda	1.0E-01
Co-60	1.06E-01	1.04E-01	1.13E-01	**1.08E-01**	*4.77E-03*	5.2	Not specified
Ni-59	2.75E-01	2.73E-01	2.62E-01	**2.70E-01**	*6.92E-03*	10.0	5.0E-02
Ni-63	2.73E+01	2.64E+01	2.64E+01	**2.67E+01**	*5.72E-01*	10.0	1.0E-01
Sr-90	1.28E+04	1.10E+04	1.38E+04	**1.26E+04**	*1.42E+03*	13.2	1.0E-03
Y-90	1.28E+04	1.10E+04	1.38E+04	**1.26E+04**	*1.42E+03*	13.2	1.0E-03
Zr-93	5.77E-01	6.04E-01	5.36E-01	**5.72E-01**	*3.40E-02*	20	1.0E-03
Nb-94	<7.88E-04	<9.82E-04	<1.17E-03	**<9.80E-04**	-	mda/UL	3.0E-03
Tc-99	4.91E-03	3.91E-03	4.39E-03	**4.40E-03**	*4.98E-04*	10.3	1.0E-03
Sn-126	1.55E-02	1.41E-02	1.41E-02	**1.46E-02**	*7.94E-04*	17.5	1.0E-03
Sn-121m	9.82E-01	7.84E-01	7.39E-01	**8.35E-01**	*1.29E-01*	9.5	Not specified
I-129	5.99E-03	4.64E-03	4.45E-03	**5.03E-03**	*8.42E-04*	5.0	1.0E-05
Cs-135	<6.89E-05	<4.46E-05	<5.36E-05	**<5.57E-05**	-	UL	1.0E-04
Cs-137	9.77E+00	9.46E+00	1.10E+01	**1.01E+01**	*8.09E-01*	5.1	1.0E-03
Ba-137m	9.25E+00	8.95E+00	1.04E+01	**9.53E+00**	*7.65E-01*	5.1	1.0E-03
Eu-152	7.21E-01	7.84E-01	7.70E-01	**7.58E-01**	*3.32E-02*	5.0	Not specified
Eu-154	1.32E+01	1.35E+01	1.40E+01	**1.36E+01**	*3.69E-01*	5.0	Not specified
Eu-155	<7.34E-01	6.89E-01	<7.52E-01	**≤7.25E-01**		18.0/UL	Not specified
Ra-226	<6.62E-04	<6.35E-04	<6.31E-04	**<6.43E-04**	-	mda	9.0E-04
Ra-228	6.13E-03	6.98E-03	7.39E-03	**6.83E-03**	*6.44E-04*	6.1	1.0E-03
Th-229	<3.38E-03	<2.07E-03	<2.04E-04	**<1.88E-03**	-	UL	8.0E-05
Th-230	<5.59E-04	<1.62E-03	<4.68E-04	**<8.83E-04**	-	UL	9.0E-04
Th-232	8.53E-03	8.68E-03	9.63E-03	**8.95E-03**	*5.96E-04*	1.1	5.0E-05
Pa-231	1.13E-03	2.49E-03	1.95E-03	**1.85E-03**	*6.85E-04*	15.1	9.0E-05
U-232	3.57E-03	4.86E-03	5.18E-03	**4.54E-03**	*8.55E-04*	25.7	1.0E-04
U-233	<7.52E-02	<7.77E-02	<8.53E-02	**<7.94E-02**		UL	1.0E-03
U-234	7.81E-03	7.54E-03	7.90E-03	**7.75E-03**	*1.87E-04*	20.0	1.0E-03
U-235	4.64E-05	4.38E-05	4.87E-05	**4.63E-05**	*2.45E-06*	20.0	1.0E-04
U-236	9.31E-05	9.26E-05	9.50E-05	**9.36E-05**	*1.27E-06*	20.0	Not specified
U-238	7.83E-04	7.28E-04	8.20E-04	**7.77E-04**	*4.63E-05*	20.0	1.0E-03
Np-237	2.14E-02	2.04E-02	2.19E-02	**2.12E-02**	*7.54E-04*	1.8	1.0E-03
Pu-238	1.32E+02	1.21E+02	1.50E+02	**1.35E+02**	1.48E+01	6.5	1.0E-03
Pu-239	5.68E+00	5.32E+00	6.31E+00	**5.77E+00**	*5.02E-01*	20.0	1.0E-03
Pu-240	2.10E+00	1.96E+00	2.32E+00	**2.13E+00**	*1.79E-01*	20.0	1.0E-03
Pu-239/240	7.75E+00	7.30E+00	8.65E+00	**7.90E+00**	*6.88E-01*	6.6	Not specified
Pu-241	3.16E+01	2.75E+01	3.70E+01	**3.20E+01**	*4.79E+00*	15.4	1.0E-03
Pu-242	2.77E-03	2.58E-03	3.14E-03	**2.83E-03**	*2.86E-04*	20.0	Not specified
Pu-244	<1.07E-07	<8.24E-08	<1.82E-07	**<1.24E-07**	-	DL	Not specified
Am-241	1.84E+01	1.82E+01	1.86E+01	**1.84E+01**	*2.03E-01*	5.0	1.0E-03
Am-242m	4.68E-03	4.95E-03	3.51E-03	**4.38E-03**	*7.66E-04*	26.2	1.0E-03
Am-243	2.40E-02	2.29E-02	<4.49E-03	**≤1.71E-02**	-	10.1/mda	1.0E-03
Cm-242	3.87E-03	4.09E-03	2.91E-03	**3.62E-03**	*6.32E-04*	26.2	Not specified
Cm-243	<1.61E-02	<1.26E-02	<1.86E-02	**<1.58E-02**	-	mda	1.0E-03
Cm-244	<6.17E-01	<6.98E-01	<6.71E-01	<6.62E-01	-	UL	1.0E-03
Cm-245	1.16E-04	1.21E-04	<1.15E-04	**≤1.17E-04**	-	20.6/UL	1.0E-03

Table 14 Radiological Constituent for Tank 12H Composite 3 Sample

Radionuclide	Run 1, µCi/g	Run 2 µCi/g	Run 3 µCi/g	Average µCi/g	Std. Dev.	One Sigma %Uncert.	TDL, µCi/g
C-14	<8.83E-04	<5.90E-04	<6.40E-04	**<7.04E-04**	-	mda	1.0E-01
Co-60	4.95E-02	6.13E-02	4.64E-02	**5.24E-02**	*7.83E-03*	10.4	Not specified
Ni-59	1.35E-01	1.04E-01	1.42E-01	**1.27E-01**	*2.03E-02*	10.0	5.0E-02
Ni-63	1.38E+01	1.37E+01	1.54E+01	**1.43E+01**	*9.37E-01*	10.0	1.0E-01
Sr-90	1.60E+04	1.75E+04	1.57E+04	**1.64E+04**	*9.88E+02*	12.9	1.0E-03
Y-90	1.60E+04	1.75E+04	1.57E+04	**1.64E+04**	*9.88E+02*	12.9	1.0E-03
Zr-93	4.05E-01	4.50E-01	4.37E-01	**4.31E-01**	*2.34E-02*	20	1.0E-03
Nb-94	<1.11E-03	<1.23E-03	<4.36E-04	**<9.28E-04**	-	mda	3.0E-03
Tc-99	3.58E-03	3.69E-03	4.30E-03	**3.86E-03**	*3.86E-04*	7.7	1.0E-03
Sn-126	1.92E-02	2.17E-02	2.02E-02	**2.04E-02**	*1.23E-03*	17.4	1.0E-03
Sn-121m	1.19E+00	1.05E+00	1.10E+00	**1.11E+00**	*6.88E-02*	9.5	Not specified
I-129	4.77E-03	3.61E-03	3.22E-03	**3.87E-03**	*8.08E-04*	5.0	1.0E-05
Cs-135	<5.45E-05	<8.06E-05	<7.75E-05	**<7.09E-05**	-	UL	1.0E-04
Cs-137	9.59E+00	9.32E+00	1.21E+01	**1.03E+01**	*1.54E+00*	5.0	1.0E-03
Ba-137m	9.08E+00	8.82E+00	1.15E+01	**9.79E+00**	*1.46E+00*	5.0	1.0E-03
Eu-152	5.50E-01	6.08E-01	5.99E-01	**5.86E-01**	*3.15E-02*	5.0	Not specified
Eu-154	7.03E+00	7.57E+00	7.21E+00	**7.27E+00**	*2.75E-01*	5.0	Not specified
Eu-155	<5.86E-01	<7.93E-01	<6.89E-01	**<6.89E-01**	-	UL	Not specified
Ra-226	<5.00E-04	<5.05E-04	<5.81E-04	**<5.29E-04**	-	mda	9.0E-04
Ra-228	3.50E-03	2.87E-03	4.37E-03	**3.58E-03**	*7.53E-04*	7.5	1.0E-03
Th-229	No Data	<4.48E-04	<1.72E-03	**<1.08E-03**	-	UL/mda	8.0E-05
Th-230	<4.59E-04	<1.08E-03	<1.57E-03	**<1.04E-03**	-	UL/mda	9.0E-04
Th-232	3.37E-03	3.67E-03	3.14E-03	**3.39E-03**	*2.70E-04*	2.1	5.0E-05
Pa-231	<4.18E-03	<1.14E-03	<8.29E-04	**<2.05E-03**	-	DL	9.0E-05
U-232	2.23E-03	1.77E-03	3.93E-03	**2.64E-03**	*1.14E-03*	36.3	1.0E-04
U-233	<3.24E-02	<3.52E-02	<3.23E-02	**<3.33E-02**	-	UL	1.0E-03
U-234	4.85E-03	5.37E-03	4.63E-03	**4.95E-03**	*3.80E-04*	20.0	1.0E-03
U-235	2.32E-05	2.36E-05	2.09E-05	**2.26E-05**	*1.46E-06*	20.0	1.0E-04
U-236	7.05E-05	7.09E-05	6.58E-05	**6.91E-05**	*2.84E-06*	20.0	Not specified
U-238	3.28E-04	3.35E-04	3.03E-04	**3.22E-04**	*1.68E-05*	20.0	1.0E-03
Np-237	7.97E-03	8.18E-03	7.89E-03	**8.01E-03**	*1.47E-04*	1.6	1.0E-03
Pu-238	6.44E+01	6.89E+01	6.62E+01	**6.65E+01**	*2.27E+00*	7.1	1.0E-03
Pu-239	2.69E+00	2.69E+00	2.75E+00	**2.71E+00**	*3.64E-02*	20.0	1.0E-03
Pu-240	9.64E-01	9.55E-01	9.95E-01	**9.71E-01**	*2.13E-02*	20.0	1.0E-03
Pu-239/240	3.65E+00	3.64E+00	3.75E+00	**3.68E+00**	*5.74E-02*	7.5	Not specified
Pu-241	1.52E+01	1.58E+01	1.51E+01	**1.54E+01**	*3.67E-01*	15.7	1.0E-03
Pu-242	1.23E-03	1.35E-03	1.33E-03	**1.30E-03**	*6.05E-05*	20.0	Not specified
Pu-244	<2.21E-07	<2.41E-07	<1.75E-07	**<2.12E-07**	-	DL	Not specified
Am-241	7.97E+00	9.32E+00	8.56E+00	**8.62E+00**	*6.78E-01*	5.0	1.0E-03
Am-242m	2.62E-03	1.82E-03	1.31E-03	**1.91E-03**	*6.59E-04*	54.0	1.0E-03
Am-243	1.15E-02	1.73E-02	1.53E-02	**1.47E-02**	*2.93E-03*	13.0	1.0E-03
Cm-242	2.16E-03	1.50E-03	1.09E-03	**1.58E-03**	*5.43E-04*	54.0	Not specified
Cm-243	<1.22E-02	<1.88E-02	<1.62E-02	**<1.57E-02**	-	mda	1.0E-03
Cm-244	<3.34E-01	<4.91E-01	<3.58E-01	**<3.94E-01**	-	UL	1.0E-03
Cm-245	<4.82E-05	<6.35E-05	<6.40E-05	**<5.86E-05**	-	UL	1.0E-03

4.0 Quality Assurance

Requirements for performing reviews of technical reports and the extent of review are established in manual E7 2.60. SRNL documents the extent and type of review using the SRNL Technical Report Design Checklist contained in WSRC-IM-2002-00011, Rev. 2.

The TTQAP details the planned activities and associated quality assurance implementing procedures for the characterization of Tank 12H (TTQAP, SRNL-RP-2014-00718, Rev. 1, October 29, 2014). The documents referenced in the TTQAP include the following: Laboratory Notebook SRNL-NB-2013-00031, L5575-00080-03 SRNL Electronic Notebook (Production); SRNL, Aiken, SC 29808 (2014) and various AD notebooks contain the analytical/experimental data. Other applicable QA documents dealing with Tank 12H sampling and compositing determinations include the Technical Task Request (G-TTR-H-00008, Rev 2, October. 27, 2014 and G-TTR-H-00009, Rev 1, April. 20, 2015), Liquid Waste Tank Residuals Sampling and Analysis Program Plan, SRR-CWDA-2011-00050, Revision. 2, July 2013 and the Liquid Waste Tank Residuals Sampling-Quality Assurance Program Plan, SRR-CWDA-2011-00117, Revision 1, July 2013.

5.0 Conclusions

Tank 12H samples were analyzed for radiological, elemental and anionic constituents. The Tank 12H cooling coil scrape samples and the combined liquid fraction were characterized for a limited suite of analytes to provide scoping information. Where analytical methods yielded additional analytes than those requested by the customer, these results were also reported.

Sufficient standards and blanks were utilized to provide quality assurance for the characterization of the Tank 12H samples. The target detection limits for all the analyses were based on customer desired detection limits as specified in the technical task request document. While many of the TDLs were met for the species characterized for Tank 12H, some were not met. The isotopes and anions with target detection limits not met in all cases were Th-229, Th-230, Pa-231, U-233, Cm-243, Cm-244, Am-242m, fluoride, and phosphate.

For these analytes, the detection limits were at typically one to two orders of magnitude higher than the target detection limits. In the Tank 12H characterizations, the detection limits for several radionuclides were about the same order of magnitude as those of the target detection limit. However, for a few radionuclides the target detection limits were not consistently met even within the same analytical sample groups. SRR reviewed all of these cases and determined that the impacts of not meeting the target detection limits were acceptable.[8] The target detection limits for most radionuclides were met most of the time.

Statistical analyses of the Tank 12H composite sample results have been completed. Analytes with all results below the MDLs were summarized by the smallest and largest MDLs. Analytes with all results above the minimum detection limits on only a single composite sample were summarized in the same fashion. Analytes with all results above the MDLs on at least two of the three composite samples were summarized by their mean, standard deviation, percent standard deviation, and their 95% upper confidence limit (UCL95) for the mean concentration.

6.0 References

1. J. P. Pavletich, "Liquid Waste Tank Residuals Sampling and Analysis Program Plan," SRR-CWDA-2011-00050, Revision. 2, July 2013.
2. T. L. Chandler, "Tank 12- Waste Characterization & Sample Analysis Report," G-TTR-H-00008, Rev. 2, Appendix D, October, 27, 2014.
3. S. H. Reboul, L. N. Oji, D. P. Diprete, C. J. Coleman and E. P. Shine, "Task Technical and Quality Assurance Plan for Tank 12H Residual Material Sample Analyses," SRNL-RP-2014-00718,Rev. 1, October 29, 2014.
4. G. M. Grim, T. L. Chandler, and G. C. Arthur, "Tank 12H Sample Location Verification Document" SRR-LWE-2014-00083, Rev. 0.
5. J. P. Pavletich, "Tank 12 Sampling and Analysis Plan," SRR-LWE-2014-00074, Rev 0. July 2014.
6. L. N. Oji, "Field Notes Documenting Completion of Tank 12H Sample Compositing". SRNL-L3100-2014-00255, Rev. 0, November 05, 2014.
7. C. J. Coleman, R. A. Dewberry, M. F. Bryant and J. J Gemmill," SRL's Performance in Round Robin #6-Analyses of Simulated Defense Waste Glass', WSRC-TR-91-187, Rev. 0, May 31, 1991.
8. M. H. Layton, "Detection Limit Acceptability for Tank 12H Residual Sample Analyses," SRR-CWDA-2015-00058, April 27, 2015.

Appendix A

Appendix A-1: Tank 12H Characterization AD Tracking Numbers (LIMS)

Analytes	Method (s)	SRNL AD Tracking Number (LIMS) Tank 12H- Composite Samples
Sr-90	Sr-90	300314633-300314644
Y-90	Sr-90	300314633-300314644
Pu-238	Pu-238/241	300314633-300314644
Pu-241	Pu-238/241	300314633-300314644
Cs-137	GAMMA SPEC-PF	300314633-300314644
Ba-137m	GAMMA SPEC-PF	300314633-300314644
U-232	U-232 ALPHA PHA	300314633-300314641
U-233	U-233, U-234, U-235, U-236	300314633-300314644
U-234	U-233, U-234, U-235, U-236	300314633-300314644
U-235	U-233, U-234, U-235, U-236	300314633-300314644
U-236	U-233, U-234, U-235, U-236	300314633-300314644
U-238	U-233, U-234, U-235, U-236	300314633-300314644
Co-60	GAMMA SPEC Cs REMOVED-PF	300314633-300314644
Eu-152	GAMMA SPEC Cs REMOVED-PF	300314633-300314644
Eu-154	GAMMA SPEC Cs REMOVED-PF	300314633-300314644
Eu-155	GAMMA SPEC Cs REMOVED-PF	300314633-300314644
Am-241	GAMMA SPEC Cs REMOVED-PF	300314633-300314644
Am-242m	Am/Cm	300314923-300314932
Am-243	Am/Cm	300314923-300314932
Cm-242	Am/Cm	300314923-300314932
Cm-243	Am/Cm	300314923-300314932
Cm-244	Am/Cm	300314923-300314932
Cm-245	Am/Cm	300314923-300314932
Pu-239	Pu-242/244	300314633-300314644
Pu-240	Pu-242/244	300314633-300314644
Pu-242	Pu-242/244	300314633-300314644
Pu-244	Pu-242/244	300314633-300314644
Pu-239/240	Pu-238/Pu-241	300314633-300314644
Ni-59	Ni-59,63	300314645-300314656
Ni-63	Ni-59,63	300314645-300314656
Tc-99	Tc-99	300314825-300314833
I-129	I-129	300314713-300314722
I-127	ICP-MS	300314657-300314667
Cs-135	Cs-135	300314633-300314644
C-14	C-14	300315715-300315724
Zr-93	Zr-93	300315701-300315712
Nb-94	Nb-94	300314633-300314644
Sn-121m	Sn-126/Sn-121m	300314633-300314644
Sn-126	Sn-126/Sn-121m	300314633-300314644
Ra-226	Ra-226/ Ra-228	300315384-300315401
Ra-228	Ra-226/ Ra-228	300315384-300315401
Th-229	Th-229/Th-230	300315701- 300315712
Th-230	Th-229/Th-230	300315701- 300315712
Th-232	ICP-MS-PF	300314633-300314644
Pa-231	Pa-231	300315137-300315155
Np-237	ICP-MS-PF	300314633-300314644
Hg	CVAA Hg	300314645-300314656
Se	AASe	300314645-300314656
As	AAAs	300314645-300314656
Ag	ICP-MS-PF digestions	300314633-300314644
Sb	ICP-MS-AQR digestions	300314645-300314656
Anions	IC- Leachate analysis	300314657-300314667
Ce, La, and Si	ICP-ES-PF digestions	300314633-300314644
All other ICP-ES elements	ICP-ES-AQR digestions	300314647-300314656

Appendix A-2: Chemical Composition of Analyzed Reference Glass

	Analytical Results for Reference Glass (ARG)	Nominal Recipe for Reference Glass (ARG)#	Percent Relative Deviation
Constituent	**wt. %**	**wt. %**	**%RD**
Al	2.53	**2.50**	1.2
B	2.58	**2.69**	4.2
***Ca**	1.02	**1.02**	0.0
Fe	9.93	**9.79**	1.4
Li	1.63	**1.49**	9.0
***K**	2.27	**2.26**	0.4
Mg	0.531	**0.52**	2.1
***Mn**	1.46	**1.46**	0.0
***Na**	8.52	**8.52**	0.0
***Ni**	0.828	**0.827**	0.1
Si	22.7	**22.4**	1.3
Ti	0.612	**0.69**	12.0

* Aqua regia digestion data (AQR: LIMS # 300314655); all other data from Peroxide fusion (PF: LIMS # 300314643).
# Reference values for ARG are reported to the number of digits given in the original citation.

Appendix A-3: Barium Analysis Comparison by Two Methods (ICP-MS vs. ICP-ES)

LIMS #	Tank 12H sample ID	ICP- MS* (Mass 138, 137, 136), µg/g	ICP-ES, µg/g	%RD
300314645	TK 12 COMP. 1_1	7.36E+02	6.33E+02	1.50E+01
300314646	TK 12 COMP. 1_2	7.22E+02	6.67E+02	7.9E+00
300314647	TK 12 COMP. 1_3	7.44E+02	6.80E+02	9.0E+00
300314648	TK 12 COMP. 2_1	8.84E+02	6.85E+02	2.54E+01
300314649	TK 12 COMP. 2_2	8.87E+02	7.37E+02	1.85E+01
300314650	TK 12 . COMP. 2_3	8.93E+02	7.35E+02	1.94E+01
300314651	TK 12 COMP. 3_1	7.79E+02	6.70E+02	1.50E+01
300314652	TK 12 COMP. 3_2	7.77E+02	7.03E+02	1.00E+01
300314653	TK 12. COMP. 3_3	7.88E+02	6.85E+02	1.40E+01

The average percent relative deviation (%RD) for barium concentration by both ICP-ES and ICP-MS methods is 14.9 %.

Appendix A-4: Cobalt Analysis Comparison by Two Methods (ICP-MS vs. ICP-ES)

LIMS #	Tank 12H sample ID	ICP-MS, mass 59, µg/g	ICP-ES, µg/g	%RD
300314645	TK 12 COMP. 1_1	3.88E+01	3.71E+01	4.5
300314646	TK 12 COMP. 1_2	3.80E+01	3.99E+01	4.9
300314647	TK 12 COMP. 1_3	3.92E+01	4.05E+01	3.3
300314648	TK 12 COMP. 2_1	6.10E+01	5.56E+01	9.3
300314649	TK 12 COMP. 2_2	6.37E+01	6.15E+01	3.5
300314650	TK 12 .COMP. 2_3	6.20E+01	6.07E+01	2.1
300314651	TK 12 COMP. 3_1	3.14E+01	3.11E+01	1.0
300314652	TK 12 .COMP. 3_2	2.92E+01	3.16E+01	7.9
300314653	TK 12. COMP. 3_3	3.43E+01	3.44E+01	0.3

Isotope 59 is applicable to stable cobalt, which is assumed to be the primary contributor of cobalt mass. The mass contribution of Co-60, due to its short half-life, is assumed to be minor. The average percent relative deviation (%RD) for cobalt concentration by both ICP-MS and ICP-ES methods is 4.1 %.

Appendix A-5: Lanthanum Analysis Comparison by Two Methods (ICP-MS vs. ICP-ES)

LIMS #	Tank 12H sample ID	ICP- MS, mass 139, µg/g	ICP-ES , µg/g	%RD
300314633	TK 12H .COMP. 1_1	9.32E+02	9.10E+02	2.4
300314634	TK 12H COMP. 1_2	9.57E+02	9.70E+02	1.3
300314634	TK 12H COMP. 1_3	9.13E+02	9.16E+02	0.3
300314636	TK 12H COMP. 2_1	1.32E+03	1.39E+03	5.2
300314637	TK 12H COMP. 2_2	1.36E+03	1.39E+03	2.2
300314638	TK 12H COMP. 2_3	1.45E+03	1.46E+03	0.7
300314639	TK 12H COMP. 3_1	7.39E+02	7.71E+02	4.2
300314640	TK 12H COMP. 3_2	8.13E+02	8.24E+02	1.3
300314641	TK 12H COMP. 3_3	8.04E+02	8.01E+02	0.4

The average percent relative deviation (%RD) for lanthanum concentration by both ICP-ES and ICP-MS methods is 2.0%.

Appendix A-6: Technetium-99 Analysis Comparison by Two Methods (ICP-MS vs. LSC)

LIMS #	Tank 12H sample ID	ICP- MS, mass 99, µCi/g	LSC, µCi/g	%RD[#]
300314633	TK 12H .COMP. 1_1	<2.10E-02	4.06E-03	N/A
300314634	TK 12H COMP. 1_2	<2.02E-02	3.27E-03	N/A
300314635	TK 12H COMP. 1_3	<2.05E-02	4.95E-03	N/A
300314636	TK 12H COMP. 2_1	<2.05E-02	4.91E-03	N/A
300314637	TK 12H COMP. 2_2	<2.08E-02	3.91E-03	N/A
300314638	TK 12H COMP. 2_3	<2.03E-02	4.39E-03	N/A
300314639	TK 12H COMP. 3_1	<2.05E-02	3.58E-03	N/A
300314640	TK 12H COMP. 3_2	<2.10E-02	3.69E-03	N/A
300314641	TK 12H COMP. 3_3	<2.07E-02	4.30E-03	N/A

[#]ICP-MS analytical results point in the right direction as the more accurate analytical results by separations and LSC analysis.

Appendix A-7: Pu-239 Analysis Comparison by ICP-MS vs. Hybrid Separation Method

LIMS #	Tank 12H sample ID	Raw ICP- MS, Pu-239, µCi/g	Hybrid Method , µCi/g	%RD[#]
300314633	TK 12H .COMP. 1_1	3.17E+00	2.98E+00	6.2
300314634	TK 12H COMP. 1_2	3.34E+00	2.92E+00	13.4
300314635	TK 12H COMP. 1_3	3.20E+00	2.83E+00	12.1
300314636	TK 12H COMP. 2_1	6.28E+00	5.68E+00	10.0
300314637	TK 12H COMP. 2_2	5.94E+00	5.32E+00	11.1
300314638	TK 12H COMP. 2_3	6.40E+00	6.31E+00	1.5
300314639	TK 12H COMP. 3_1	2.80E+00	2.69E+00	3.9
300314640	TK 12H COMP. 3_2	2.90E+00	2.69E+00	7.6
300314641	TK 12H COMP. 3_3	2.74E+00	2.75E+00	0.6

[#] The average percent relative deviation (%RD) for Pu-239 concentration by both ICP-MS and Separations and hybrid methods is 7.4%.

Appendix A-8: Pu-240 Analysis Comparison by ICP-MS vs. Hybrid Separation Method

LIMS #	Tank 12H sample ID	Raw ICP- MS, Pu-240, µCi/g	Hybrid Method; µCi/g	%RD
300314633	TK 12H .COMP. 1_1	1.12E+00	1.05E+00	6.8
300314634	TK 12H COMP. 1_2	1.17E+00	1.03E+00	12.9
300314635	TK 12H COMP. 1_3	1.11E+00	9.77E-01	12.7
300314636	TK 12H COMP. 2_1	2.30E+00	2.10E+00	9.2
300314637	TK 12H COMP. 2_2	2.15E+00	1.96E+00	9.4
300314638	TK 12H COMP. 2_3	2.35E+00	2.32E+00	1.2
300314639	TK 12H COMP. 3_1	1.02E+00	9.64E-01	5.3
300314640	TK 12H COMP. 3_2	1.04E+00	9.55E-01	8.5
300314641	TK 12H COMP. 3_3	9.96E-01	9.95E-01	0.1

The average percent relative deviation (%RD) for Pu-240concentration by both ICP-MS and Separations and hybrid method is 7.3%.

Appendix B: Summary of Analytical Methods

Aqua Regia Digestions (AQR)

Samples were digested according to procedure L16.1, ADS-2226. In a typical digestion, ~0.5 g of Tank 12H composite sample was placed into a Teflon® digestion vessel. Then, 9 mL (hydrochloric acid) HCl, and 3 mL (nitric acid) HNO_3 were added to the Teflon® vessel. The Teflon® vessel was sealed and heated for a period of no more than 4 hours at 115 ºC. The sample was then cooled and diluted to 50 mL. Three samples, in total, from each composite sample were digested by AQR.

Sodium Peroxide/Hydroxide Fusions (PF)

Samples were digested according to procedure L16.1, ADS 2502. In a typical digestion, ~2 grams of Tank 12H composite material was placed into a nickel (Ni) crucible with a known weight. The material in the crucible was dried until two consecutive weights were within ±0.02 grams. The remaining material in the crucible was fused at 675 ºC using a mixture of sodium peroxide (6.0 grams) and sodium hydroxide (4.0 grams). After the sample was cooled, water was added to dissolve the fused material and the solution was acidified by the addition of 25 mL HCl. The sample was diluted to 100 mL. Three samples, in total, from each composite sample were digested by PF.

Inductively Coupled Plasma – Atomic Emission Spectroscopy (ICP-ES)

Digested sample liquids were diluted as necessary to bring analytes within the instrument range. A scandium internal standard is added to all samples after dilution at a concentration of 2 mg/L. The instrument is calibrated daily with a blank and two standards: 5 and 10 mg/L National Institute of Standards and Technology (NIST) traceable multi-element standards in dilute acid. Background and internal standard correction were applied to the results.

Ion Chromatography for Anions (IC-Anions)

For IC Anions, samples were diluted with a carbonate/bicarbonate diluent as necessary to bring analytes to within instrument calibration. A 3-point calibration curve is run daily on the instrument with concentrations of 10, 25 and 50 µg/mL.

Atomic Absorption Spectroscopy (AA)

Arsenic, selenium, and mercury are 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 instrument calibration is performed daily with a blank and two or three point standard. The standard is run for each element at the beginning of the day, after each five sample runs and at the end of the day.

Inductively Coupled Plasma – Mass Spectroscopy (ICP-MS)

Samples are diluted as necessary to bring analytes within the instrument range. An internal standard with bismuth and indium is added to all samples after dilution. The instrument is calibrated daily with a blank and a minimum of four calibration standards that are NIST traceable multi-element standards in dilute acid. Background and internal standard correction were applied to the results.

Ni-59, Ni-63

Aliquots of Tank 12 aqua regia dissolution were aliquoted and spiked with an elemental nickel carrier. The nickel species were extracted from the matrix using dimethylglyoxime (DMG) based extractant. Ni-59 concentrations were measured using low energy photon/x-ray, thin-windowed, semi-planar high purity germanium spectrometers. Ni-63 concentrations were measured by liquid scintillation analysis.

Elemental nickel carrier yields were measured by ICP-ES, and were used to correct the radioactive nickel species' analyses for any nickel losses from the radiochemical separations. Cell reagent blanks, nitrated synthetic sodalite (sodalite), ARG glass, laboratory reagent blanks, a Ni-63 standard and a Ni-59 standard were run as controls.

Cs-137

Aliquots of Tank 12 PF dissolution and AQR were analyzed by coaxial high purity germanium gamma-ray spectrophotometers to measure Cs-137. Cell reagent blanks, sodalite and ARG glass (also based on PF and AQR digestions), and laboratory reagent blanks were run as controls.

Sr-90

Aliquots of Tank 12H peroxide fusion dissolutions were spiked with an elemental strontium carrier. The strontium species were extracted from the matrix using a crown-ether-based solid phase extractant. Sr-90 concentrations were measured by liquid scintillation analysis. Elemental strontium carrier yields were measured by neutron activation analysis, and were used to correct the Sr-90 analyses for any strontium losses from the radiochemical separations. Cell reagent blanks, sodalite, and ARG glass (also based on PF and AQR digestions) and laboratory reagent blanks and a Sr-90 standard were run as controls.

Co-60, Am-241 (Cs-removed gamma analysis)

Aliquots of Tank 12H peroxide fusion dissolution were subjected to a Cs-removal process utilizing Bio Rad AMP-1 resin. The Cs-removed solutions were analyzed by coaxial high purity germanium spectrophotometers to measure the gamma-emitting radionuclides listed above.

Pu-238, 239/240, 241

Aliquots of Tank 12H PF were spiked with Pu-236 tracer. The plutonium was extracted from the matrix using thenoyltrifluoroacetone (TTA) following a series of oxidation-state adjustments. The TTA extracts were mounted on stainless steel counting plates and counted for Pu-238 and Pu-239/240 using passivated, implanted, planar silicon (PIPs) detectors. Each separation was traced based on the Pu-236 recovery. Aliquots of sample were also subjected to Cs-removal with Bio-Rad Ammonium Molybdophosphate (AMP) resin and extracted using TEVA columns (TEVA Brand name for one of Eichrom's resins). The Pu-containing extracts were measured by liquid scintillation analysis to determine Pu-241 concentrations. Cell reagent blanks, sodalite, and ARG glass (also based on PF and AQR digestions), and laboratory reagent blanks and a Pu-238 standard were run as controls.

Pu-239, 240, 242, 244

The plutonium from aliquots of Tank 12H peroxide fusion dissolutions were extracted using TEVA columns (TEVA Brand name for one of Eichrom's resins). The Pu-containing extracts were then analyzed by ICP-MS to determine Pu-239, Pu-240, Pu-242, and Pu-244 isotopes. The Tank 12 samples were yielded as is typical from the Pu-239/240 result of the TTA analysis. Cell reagent blanks, tank simulant, ARG glass, and laboratory reagent blanks were run as controls.

Am-242m, 243, Cm-242, 243, 244, 245

Tank 12H material samples were digested using a PF. Additionally, a matrix blank and matrix blank spiked with Am-241 and Cm-244 were prepared. The americium and curium species were extracted from aliquots of peroxide fusion using a carbamoylmethylphosphine oxide tributyl phosphate (CMPO/TBP) based solid phase extractant and purified further with an HDEHP based solid phase extractant. Am-241, 243, Cm-243, and 245 concentrations were measured using low energy photon/x-ray, thin-windowed, semi-planar high purity germanium spectrometers. Am-242m, Cm-242, and 244 concentrations were measured using PIPs alpha spectrometers. Cm-245 ratios to Am-241 were measured using ICP-MS and

were applied to the previously quantified Am-241. Am-241 quantities had been measured from the cesium removed gamma analyses, Am, and Cm results were traced with the Am-241 present in the sample matrix. Cell reagent blanks, tank simulant, and laboratory reagent blanks were also run as controls.

Tc-99

Tank 12H composite samples were digested in a combination of concentrated nitric and hydrochloric acids. Several matrix blanks were prepared using sodalite spiked with a Tc-99 standard. The dissolutions were subjected to a number of resin treatments to reduce dose prior to removal from the shielded cells. The treated samples were then spiked with Tc-99m and the technetium species were extracted from the matrix using an Aliquat-336 based solid phase extractant. Tc-99 concentrations were measured by liquid scintillation analysis. Tc-99m yields were measured with a NaI-well gamma spectrometer, and were used to correct the Tc-99 analyses for any technetium losses from the radiochemical separations. The average recovery of the Tc-99 spiked matrix blank was applied to the entire set of samples to correct for any losses from the decontamination steps used in the Shielded Cells.

Ra-226, Ra-228

Tank 12H composite samples were digested using a PF. Each replicate was prepared in duplicate with the duplicate containing a Ra-224 tracer. Additionally, a matrix blank was prepared using sodalite. The Ra-226 was extracted from the matrix using a combination of resin decontamination and ion exchange. The purified Ra-226 was sealed in polypropylene tubes and stored for several daughter Rn-222 half-lives. The Ra-226 progeny daughter isotope Pb-214 was then analyzed for using a high purity germanium well gamma ray spectrophotometer and results were corrected for the tracer Ra-224 recoveries. The Ra-228 progeny daughter isotope Ac-228 was also measured from the high purity germanium well gamma ray spectrophotometer and results were also corrected for the tracer Ra-224 recoveries A simulant blank traced with Ra-224 and spiked with Ra-226 was run through the process to serve as a calibration standard. A simulated blank sample traced with Ra-224 and spiked with Ra-226 was run through the process to serve as a control standard.

Pa-231

Tank 12H composite samples were digested using a PF. Each replicate was prepared in duplicate with the duplicate containing a Pa-233 tracer. Additionally, a matrix blank and matrix spiked blank were prepared using simulated sludge. The dissolutions were decontaminated with AMP and quaternary amine based resins. Protactinium species were then extracted from the matrix using a CMPO/TBP based extractant. Pa-233 tracer concentrations were measured using high purity germanium spectrometers to determine separation yields. Pa-231 was measured using the ICP-MS. The Pa-233 tracer yields were decay corrected and then used to correct the Pa-231 analyses for any losses from the radiochemical separations.

I-129

Tank 12H composite samples were dissolved in concentrated acid with an added KI carrier. A matrix blank and matrix blank containing an I-129 spike were also prepared using sodalite. The samples were rendered caustic, and decontaminated with strikes with crystalline silicotitinate (CST) and monosodium titinate (MST) followed by a filtration step. The samples were then acidified and treated with Actinide and AMP resins to facilitate removal of interfering isotopes. Sodium sulfite was added to the material to reduce the iodine. Silver nitrate was added to the solution to precipitate the iodine as AgI, which was separated via filtration. The filtrate is analyzed for I-129 content using low energy photon/x-ray, thin-windowed, semi-planar, high purity germanium spectrometers. Elemental iodine yields were measured by neutron activation analysis, and were used to correct the I-129 analyses for any iodine losses from the radiochemical separation.

C-14

Tank 12H composite sample material was added to a mixture of sodium hydroxide, and sodium carbonate/sodium hydroxide. A series of oxidation and reduction steps designed to liberate C-14 containing carbon dioxide were carried out, which selectively trapped the C-14 in a basic solution. The basic solutions were acidified and the C-14 containing carbon dioxide was captured in Carbosorb E and measured by liquid scintillation analysis. A blank, a C-14 calibration standard and a C-14 control standard were also run through the process.

Th-229, Th-230

Tank 12H composite samples were digested using a PF digestion. Each replicate was prepared in duplicate with the duplicate containing a Th-229 tracer. Additionally, a matrix blank and matrix spiked blank were prepared using sodalite. The matrix spiked blank contained both a Th-228 and Th-229 spike. Thorium was extracted from the matrix using two stages of a quaternary amine based solid phase extraction and purified further via co-precipitation with cerium. Th-229 and Th-230 concentrations were measured using PIPs alpha spectrometers. The thorium yields were calculated by comparing Th-232 activities measured from the separated aliquots to Th-232 quantities measured directly by the ICP-MS on aliquots of sludge dissolution. The thorium yields were used to correct the various analytes analyses for any Th losses from the radiochemical separations.

Nb-94

Aliquots of Tank 12H PF were spiked with a radioactive Nb-95 tracer, and then purified by anion exchange. The purified aliquots were analyzed by high purity germanium spectrometers to measure Nb-94 and Nb-95. The niobium chemical recoveries were determined from the Nb-95 tracer measurement. The Nb-94 values were corrected with the Nb-95 recoveries. Cell reagent blanks, sodalite, ARG glass, and laboratory reagent blanks were run as controls.

Sn-126, Sn-121m

Aliquots of Tank 12H PF digestions were spiked with a radioactive Sn-113m tracer, and then purified by anion exchange. The purified aliquots were analyzed using low energy photon/x-ray, thin-windowed, semi-planar high purity germanium spectrometers. The tin chemical recoveries were determined from the Sn-113m tracer measurement. The Sn-126 and Sn-121m values were corrected with the Sn-113m recoveries. Cell reagent blanks, tank simulant, ARG glass, and laboratory reagent blanks were run as controls.

Zr-93

Aliquots of Tank 12H composite samples were dissolved using PF dissolutions in nickel crucibles. Stable Zr concentrations were measured in the dissolution using ICP-MS. Thorium was stripped from the samples so as not to interfere with the Zr extraction. The Zr-93 was then extracted from aliquots using a CMPO/TBP based solid phase extractant. Zr-93 levels were measured using the ICP-MS, and the results were yielded from sample stable Zr recoveries as measured by the ICP-MS. Cell reagent blanks, sodalite, ARG glass, and laboratory reagent blanks were run as controls.

Cs-135

Cesium 135 (Cs-135) was extracted from aliquots of Tank 12H composite solutions from PF digestions using a spherical resorcinol resin. Aliquots of the Cesium extract were analyzed by gamma spectrometry to measure the Cs-137 levels in the extract. Cs-137 activities in the extract were compared to Cs-137 activities measured directly from sludge dissolutions to determine Cs chemical recoveries. The purified Cs-containing extracts were also analyzed using ICP-MS to measure Cs-135 concentrations. The Cs

yields from the gamma analyses were applied to the Cs-135 concentrations measured by the ICP-MS to determine Cs-135 concentrations in Tank 12H composite samples.

U-232

Aliquots of PF digested Tank 12H composite sample solution were spiked with a U-233 radioactive tracer, additional aliquots were run through the method with no tracer added. Uranium was extracted from the matrix using two stages of a diamyl, amylphosphonate (DAAP)-based solid phase extraction and purified further via co-precipitation with cerium. U-232, U-233, and U-238 activities were measured using PIPS alpha spectrometers. The Tank 12 samples were so high in U-234, the U-233 tracers (which have the same alpha energy as U-234) were swamped out. U-232/U-238 activity ratios were generated and were multiplied to U-238 activities measured with the ICP-MS to determine U-232 activities in the samples.

U-233, U-234, U-235, U-236

Uranium was extracted from aliquots of Tank 12 peroxide fusion dissolution using a diamyl, amylphosphonate (DAAP)-based solid phase extraction. The uranium extract was then analyzed by ICP-MS for U-233, U-234, U-235, U-236, and U-238. The sample U-238 concentrations had been determined previously from an ICP-MS analysis directly. The U-233/238, U-234/238 U-235/238, and U-236/238 ratios measured from the ICP-MS analysis of the uranium extract was applied to the U-238 concentration quantified directly off the ICP-MS analysis to determine the sample U-233, U-234, U-235, and U-236 concentrations.

Weight Percent Solids Measurement

The weight percent total solids in each Tank 12H sample and composite samples were measured in the Shielded Cells using a conventional drying oven at 110 °C. An aliquot of each composite sample was placed in a 150-mL capacity beaker container. The container was placed in the oven. The weights of the dried sample were checked periodically over 72 hours until two consecutive weights yielded comparable results. The weight fraction solid was calculated by dividing the dry weight of the sample by the initial weight of the sample. A 5% sodium chloride salt solution prepared by dissolving 5 grams of dried sodium chloride in distilled water was used as the reference matrix for weight percent determinations as described above.

Density Measurement and Volume Measurements

The bulk density of the solids (as-received or homogenized solid particles) was determined using a constant volume cut-out bottom portion of plastic 100-mL volumetric flasks. The volumes of several of these cut-outs ranged from 13 to 21 mL capacities. The fixed volume of each cut-out was determined analytically by seating it on a 3 digit balance and filling each cut-out unit with DI water until the water reached the brim of the cut-out (cup) without overflowing. A flat spatula was moved over the top of the cup to remove excess water. This was repeated several times until water was no longer touching the spatula blade. The weight of the amount of water required to fill the fixed volume cup up to the top was measured by difference. Assuming the density of the water was 1.0 g/mL at the measuring temperature of approximately 25 ^{o}C the water mass was considered equal to cup volume.

The bulk densities of the “first measured” granular tank solids or homogenized samples were individually measured as soon as practicable using a constant volume cup described above. Using each of the pre-weighed 20 mL or 13 mL capacity cup, the solids material was loaded into the cup using a spatula (with the whole assembly seated in a secondary container to prevent contamination and sample spills). Enough solid material was put into the cup until there was a solid material overflow at the top of the cup. The cup and its content was gently tapped or shaken to ensure that much of the solid content had dispersed and seated inside the cup without cavities or gaps. A flat head spatula was moved across the top of the cup to uniformly dislodge excess material across the open cup rim. At this time the contents of the cup were

flush with the circular cup rim. The cup and contents were seated on a balance and the total weight measured and recorded. Knowing the weight of the material by difference and the volume of the cup, the bulk density of the material was calculated. The measurements were determined three times for each sample and at the end of the measurements the contents of the cup were put back into the original sample container.

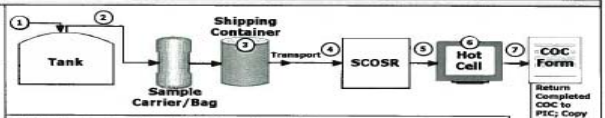
Appendix C-a: Chain-of-Custody Forms

7.18 Chain of Custody

Liquid Waste Tank Residuals Sampling: CHAIN-OF-CUSTODY # TK12 – 7/18/14 (Tank # — Date (DD/MM/YY)) Page 1 of 2

TANK # 12 | TSAP, Rev # SRR-LWE-2014-00074, R0 | Analyses in TTR, Rev # G-TTR-H-00008, R0 | Laboratory: SRNL

1. Sample identifications on the containers or sampling tool are verified against those scheduled for collection before the container(s) and retrieval basket or sampling tool are placed into the waste tank or equipment vessel.
2. Chain-of-Custody (COC) begins (Date/Time) when the collected sample is placed into the retrieval basket or sampling tool. The Person-in-Charge (PIC) at the collection time is considered the collector and the custodian until the samples are shipped to the laboratory.
3. When the samples are shipped, the PIC transfers custody to the person accompanying the samples to the Shielded Cells Operations Sample Receiving (SCOSR) facility.
4. Custody is transferred to the SCOSR Sample Custodian, and they enter the receipt into the Radioactive Material Receipt Entry Log. Those log entry numbers are added to the COC.
5. A copy of the COC is made and returned to the PIC. The original COC is retained by SCOSR for completion as described in Steps 6 and 7.
6. When the sample carrier is opened in the Hot Cell, the sample identifications are verified by SCO or Analytical Development (AD) personnel and that section of the COC is updated. The SRR Engineering Contact is immediately notified of any labeling or sample condition anomalies.
7. The SRNL personnel verifying the sample identifications will complete that section of the COC, **make a copy, and return the original COC to the PIC**.
8. The COC copy will be sent to the AD Principal Investigator (PI) for inclusion in the sample analysis report.



SRR Engineering Contact for Questions or Concerns: Greg Arthur / 507-2569 / 18634 (Name/Phone/Pager)

PIC: Mike Harrell / Closure Support / 507-5648 (Name/Organization/Phone)

Sample ID (see TSAP)	Sample Collection Date	Time	Collector	Sample Type *	Sample Container *	SCO or AD Sample ID Verifier Name, Date, Time Date	Time	ID Verifier	SRNL Radioactive Material Receipt Entry Log #
T12-RB-C-Low 7/18/14		1325	Mike E. Harrell	S	V/O				
T12-RB-C-Mid 7/18/14		1205	Mike E. Harrell	S	V/O				
			No Further Samples						

* Sample Type: S = Solids L = Liquid O = Other (describe)
Sample Container: SC = Stainless Steel Sample Cup C= Core Sampler V= Vaccum O = Other (describe)

7.18 Chain of Custody, Cont'd 2 of 2

Sample ID (see TSAP)	Sample Collection Date	Time	Collector	Sample Type *	Sample Container *	SCO or AD Sample ID Verifier Name, Date, Time Date	Time	ID Verifier	SRNL Radioactive Material Receipt Entry Log #
			No Further Samples						

	Signature	Printed Name	Organization	Date	Time
Relinquished By (PIC):		Mike E. Harrell	Closure Support	7/21/14	1105
Received By:	RC Sullivan	Rita C. Sullivan	SCO	7/21/14	1105
Relinquished By:	RC Sullivan	Rita C. Sullivan	SCO	7/22/14	0930
Received By:	Lawrence Osi	LAWRENCE OSI	SRNL	7/22/14	0930
Relinquished By:					
Received By:					
Relinquished By:					
Received By:					

Notes:
Sample T12-RB-C-Low contained 24 grams of solid materials. SRNL 24 g. 7/22/14
Sample T12-RB-C-Mid contained 50.163g

* Sample Type: S = Solids L = Liquid O = Other (describe)
Sample Container: SC = Stainless Steel Sample Cup C= Core Sampler O = Other (describe)

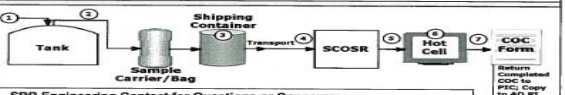
7.18 Chain of Custody

Liquid Waste Tank Residuals Sampling: CHAIN-OF-CUSTODY # TK12 29/07/14 (Tank # — Date [DD/MM/YY]) Page 1 of 2

(Waste Characterization for tanks listed in ERD Table G-6)

TANK #	TSAP, Rev #	Analyses in TTR, Rev #	Laboratory:
12	SRR-LWE-2014-00074, R.0	S-TTR-H-00008, R.0	SRNL

(3) Sample identifications on the containers or sampling tool are verified against those scheduled for collection before the container(s) and retrieval basket or sampling tool are placed into the waste tank or equipment vessel.
(2) Chain-of-Custody (COC) begins (Date/Time) when the collected sample is placed into the retrieval basket or sampling tool. The Person-in-Charge (PIC) at the collection time is considered the collector and the custodian until the samples are shipped to the laboratory.
(3) When the samples are shipped, the PIC transfers custody to the person accompanying the samples to the Shielded Cells Operations Sample Receiving (SCOSR) facility.
(4) Custody is transferred to the SCOSR Sample Custodian, and they enter the receipt into the Radioactive Material Receipt Entry Log. Those log entry numbers are added to the COC.
(5) A copy of the COC is made and returned to the PIC. The original COC is retained by SCOSR for completion as described in Steps 6 and 7.
(6) When the sample carrier is opened in the Hot Cell, the sample identifications are verified by SCO or Analytical Development (AD) personnel and that section of the COC is updated. The SRR Engineering Contact is immediately notified of any labeling or sample condition anomalies.
(7) The SRNL personnel verifying the sample identifications will complete that section of the COC, **make a copy, and return the original COC to the PIC**.
(8) The COC copy will be sent to the AD Principal Investigator (PI) for inclusion in the sample analysis report.



SRR Engineering Contact for Questions or Concerns:
Greg Arthur / 507-2569 / 18634
(Name/Phone/Pager)

PIC: Mike Harrell / Closure / 507-5648
(Name/Organization/Phone)

Sample ID (see TSAP)	Sample Collection Date	Time	Collector	Sample Type *	Sample Container *	SCO or AD Sample ID Verifier Name, Date, Time Date	Time	ID Verifier	SRNL Radioactive Material Receipt Entry Log #
T12-F-1	8/4/14	1144	Mike Harrell	O	SC	8-11-14	10:37	Linda L Bush	14228
T12-F-2	8/4/14	1320	Mike Harrell	O	SC	8-11-14	10:37	Linda L Bush	
T12-F-3	8/5/14	0831	Mike Harrell	O	SC	8-11-14	10:37	Linda L Bush	
T12-F-4	8/6/14	1033	Mike Harrell	O	SC	8-11-14	10:37	Linda L Bush	
T12-F-5	8/7/14	0833	Mike Harrell	O	SC	8-11-14	10:37	Linda L Bush	

* Sample Type: S = Solids L = Liquid O = Other (describe)
Sample Container: SC = Stainless Steel Sample Cup C= Core Sampler V= Vaccum O = Other (describe)

7.18 Chain of Custody, Cont'd

2 of 2

Sample ID (see TSAP)	Sample Collection Date	Time	Collector	Sample Type *	Sample Container *	SCO or AD Sample ID Verifier Name, Date, Time Date	Time	ID Verifier	SRNL Radioactive Material Receipt Entry Log #
T12-F-6	8/7/14	1425	Mike Harrell	O	SC	8-11-14	10:37	Linda L Bush	14228

	Signature	Printed Name	Organization	Date	Time
Relinquished By (PIC):	[illegible]	Mike Harrell	SRR, Closure	8/11/14	1030
Received By:	[illegible]	Linda L Bush	SRNL	8-11-14	10:50
Relinquished By:	[illegible]	Linda L Bush	SRNL	8-11-14	1400
Received By:	[illegible]	LAWRENCE OSL	SRNL	8-11-14	1400
Relinquished By:					
Received By:					
Relinquished By:					
Received By:					

Samples opened 8/12/14.

Notes: Sample F-4 contained a pasty solid with ~ 5mL free liquid.
Sample F-5 was almost full with pasty solid material with little free liquid.
Sample F-6 was mostly free liquid in bottom and some little pasty material on bottom.

* Sample Type: S = Solids L = Liquid O = Other (describe)
Sample Container: SC = Stainless Steel Sample Cup C= Core Sampler O = Other (describe)
Sample F-1 contained no solid sample, just about 5mL free liquid (F-1) black/dk liquid.
Sample F-2 contained mainly free liquid, no solids ~ 40 mL dirty black liquid.
Sample F-3; mainly dark/black liquid (fluid) ~ 60 mL.

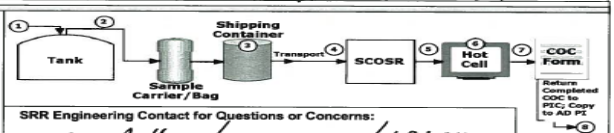
7.18 Chain of Custody

Liquid Waste Tank Residuals Sampling: CHAIN-OF-CUSTODY # TK12 - 23/07/14
(Tank # — Date [DD/MM/YY])
Page 1 of 2

(Waste Characterization for tanks listed in ERD Table G-6)

TANK #	TSAP, Rev #	Analyses in TTR, Rev #	Laboratory:
12	SRR-LWE-2014-00074, R.0	G-TTR-H-00008, R.0	SRNL

1. Sample identifications on the containers or sampling tool are verified against those scheduled for collection before the container(s) and retrieval basket or sampling tool are placed into the waste tank or equipment vessel.
2. Chain-of-Custody (COC) begins (Date/Time) when the collected sample is placed into the retrieval basket or sampling tool. The Person-in-Charge (PIC) at the collection time is considered the collector and the custodian until the samples are shipped to the laboratory.
3. When the samples are shipped, the PIC transfers custody to the person accompanying the samples to the Shielded Cells Operations Sample Receiving (SCOSR) facility.
4. Custody is transferred to the SCOSR Sample Custodian, and they enter the receipt into the Radioactive Material Receipt Entry Log. Those log entry numbers are added to the COC.
5. A copy of the COC is made and returned to the PIC. The original COC is retained by SCOSR for completion as described in Steps 6 and 7.
6. When the sample carrier is opened in the Hot Cell, the sample identifications are verified by SCO or Analytical Development (AD) personnel and that section of the COC is updated. The SRR Engineering Contact is immediately notified of any labeling or sample condition anomalies.
7. The SRNL personnel verifying the sample identifications will complete that section of the COC, **make a copy, and return the original COC to the PIC.**
8. The COC copy will be sent to the AD Principal Investigator (PI) for inclusion in the sample analysis report.



SRR Engineering Contact for Questions or Concerns:
Greg Arthur / 507-2569 / 18634
(Name/Phone/Page-I)

PIC: Mike Harrell / Closure Support / 507.5648
(Name/Organization/Phone)

Sample ID (see TSAP)	Sample Collection Date	Time	Collector	Sample Type *	Sample Container *	SCO or AD Sample ID Verifier Name, Date, Time Date	Time	ID Verifier	SRNL Radioactive Material Receipt Entry Log #
TK12-RB-C-High	7/23/14	1054	Mike E. Harrell	S	V/O				

* Sample Type: S = Solids L = Liquid O = Other (describe)
Sample Container: SC = Stainless Steel Sample Cup C= Core Sampler V= Vaccum O = Other (describe)

7.18 Chain of Custody, Cont'd

2 of 2

Sample ID (see TSAP)	Sample Collection Date	Time	Collector	Sample Type *	Sample Container *	SCO or AD Sample ID Verifier Name, Date, Time Date	Time	ID Verifier	SRNL Radioactive Material Receipt Entry Log #

	Signature	Printed Name	Organization	Date	Time
Relinquished By (PIC):	[signature]	Mike E. Harrell	Closure Support	7/23/14	1510
Received By:	[signature]	Rita C. Sullivan	SCO	7/23/14	1510
Relinquished By:	[signature]	Rita C. Sullivan	SCO	7/24/14	1400
Received By:	[signature]	LAWRENCE OTT	SRNL	7/24/14	1400
Relinquished By:					
Received By:					
Relinquished By:					
Received By:					

Notes:
wt of sample is 28 g

* Sample Type: S = Solids L = Liquid O = Other (describe)
Sample Container: SC = Stainless Steel Sample Cup C= Core Sampler O = Other (describe)

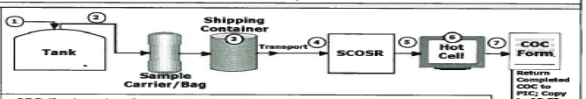
7.18 Chain of Custody

Liquid Waste Tank Residuals Sampling: CHAIN-OF-CUSTODY # TK 12 13/08/14 (Tank # — Date [DD/MM/YY]) Page 1 of 2

(Waste Characterization for tanks listed in ERD Table G-6) R0

TANK #	12	TSAP, Rev #	SRR-LWE-2014-00074	Analyses in TTR, Rev #	S-TTR-H-00008, R0	Laboratory:	SRNL

1. Sample identifications on the containers or sampling tool are verified against those scheduled for collection before the container(s) and retrieval basket or sampling tool are placed into the waste tank or equipment vessel.
2. Chain-of-Custody (COC) begins (Date/Time) when the collected sample is placed into the retrieval basket or sampling tool. The Person-in-Charge (PIC) at the collection time is considered the collector and the custodian until the samples are shipped to the laboratory.
3. When the samples are shipped, the PIC transfers custody to the person accompanying the samples to the Shielded Cells Operations Sample Receiving (SCOSR) facility.
4. Custody is transferred to the SCOSR Sample Custodian, and they enter the receipt into the Radioactive Material Receipt Entry Log. Those log entry numbers are added to the COC.
5. A copy of the COC is made and returned to the PIC. The original COC is retained by SCOSR for completion as described in Steps 6 and 7.
6. When the sample carrier is opened in the Hot Cell, the sample identifications are verified by SCO or Analytical Development (AD) personnel and that section of the COC is updated. The SRR Engineering Contact is immediately notified of any labeling or sample condition anomalies.
7. The SRNL personnel verifying the sample identifications will complete that section of the COC, **make a copy, and return the original COC to the PIC.**
8. The COC copy will be sent to the AD Principal Investigator (PI) for inclusion in the sample analysis report.



SRR Engineering Contact for Questions or Concerns:
Greg Arthur / 507-2569
(Name/Phone/Pager)

PIC: Mike Harrell / Closure / 507-5648
(Name/Organization/Phone)

Sample ID (see TSAP)	Sample Collection Date	Time	Collector	Sample Type *	Sample Container *	SCO or AD Sample ID Verifier Name, Date, Time Date	Time	ID Verifier	SRNL Radioactive Material Receipt Entry Log #
T12-F-1R	8/14/14	1226	Mike Harrell	O	SC				8/15/14 [illegible]
T12-F-2R	8/14/14	1330	Mike Harrell	O	SC				8/14/14 [illegible]
T12-F-3R	8/14/14	1050	Mike Harrell	O	SC				
T12-F-2R	8/14/14	1236	Mike Harrell	O	SC				
T12-F-1R	8/15/14	1100	Mike Harrell	O	SC				

* Sample Type: S = Solids L = Liquid O = Other (describe)
Sample Container: SC = Stainless Steel Sample Cup C= Core Sampler V= Vaccum O = Other (describe)

★ Performed Resample in Sample Location 1R + 2R in order to obtain more solids. [illegible] 8/14/14

7.18 Chain of Custody, Cont'd

2 of 2

Sample ID (see TSAP)	Sample Collection Date	Time	Collector	Sample Type *	Sample Container *	SCO or AD Sample ID Verifier Name, Date, Time Date	Time	ID Verifier	SRNL Radioactive Material Receipt Entry Log #

[illegible] 8/18/14

	Signature	Printed Name	Organization	Date	Time
Relinquished By (PIC):	[signature]	Mike Harrell	Closure	8/18/14	1350
Received By:	[signature]	Monica Jenkins	SRNL, SCO	8-18-14	1359
Relinquished By:	[signature]	Monica Jenkins	SRNL, SCO	8-18-14	1402
Received By:	[signature]	LAWRENCE COX	SRNL.	8/18/14	1404
Relinquished By:					
Received By:					
Relinquished By:					
Received By:					

Notes: Sample T12-F-1R ~~was~~ contains about 90% wet Cake; little free liquid
Sample T12-F-2R: 25% wet Cake with about 60 mL liquid fraction (est % liquid fraction = 59.7%)
Sample T12-F-3R: 50% wet cake with 15 ml of liquid fraction.

* Sample Type: S = Solids L = Liquid O = Other (describe)
Sample Container: SC = Stainless Steel Sample Cup C= Core Sampler O = Other (describe)

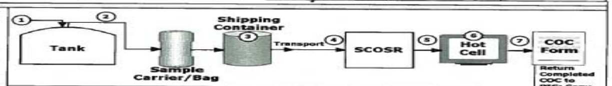
7.18 Chain of Custody

Liquid Waste Tank Residuals Sampling: CHAIN-OF-CUSTODY # TK12 20/08/14 (Tank # — Date [DD/MM/YY])
(Waste Characterization for tanks listed in ERD Table G-6)

Page 1 of 2

TANK # 12 **TSAP, Rev #** ERR-LWE-2014-00074, Rev. 0 **Analyses in TTR, Rev #** G-TTR-H-00008, Rev 0 **Laboratory:** SRNL

1. Sample identifications on the containers or sampling tool are verified against those scheduled for collection before the container(s) and retrieval basket or sampling tool are placed into the waste tank or equipment vessel.
2. Chain-of-Custody (COC) begins (Date/Time) when the collected sample is placed into the retrieval basket or sampling tool. The Person-in-Charge (PIC) at the collection time is considered the collector and the custodian until the samples are shipped to the laboratory.
3. When the samples are shipped, the PIC transfers custody to the person accompanying the samples to the Shielded Cells Operations Sample Receiving (SCOSR) facility.
4. Custody is transferred to the SCOSR Sample Custodian, and they enter the receipt into the Radioactive Material Receipt Entry Log. Those log entry numbers are added to the COC.
5. A copy of the COC is made and returned to the PIC. The original COC is retained by SCOSR for completion as described in Steps 6 and 7.
6. When the sample carrier is opened in the Hot Cell, the sample identifications are verified by SCO or Analytical Development (AD) personnel and that section of the COC is updated. The SRR Engineering Contact is immediately notified of any labeling or sample condition anomalies.
7. The SRNL personnel verifying the sample identifications will complete that section of the COC, **make a copy, and return the original COC to the PIC**.
8. The COC copy will be sent to the AD Principal Investigator (PI) for inclusion in the sample analysis report.



SRR Engineering Contact for Questions or Concerns: Greg Arthur / 803-507-2569 (Name/Phone/Pager)

PIC: Mike Harrell / Closure / 803-507-5648 (Name/Organization/Phone)

Sample ID (see TSAP)	Sample Collection					SCO or AD Sample ID Verifier Name, Date, Time			SRNL Radioactive Material Receipt Entry Log #
	Date	Time	Collector	Sample Type *	Sample Container *	Date	Time	ID Verifier	
T12-M-H1	8/23/14	1100	Mike Harrell	S	SC				
T12-M-H2	8/23/14	0946	Mike Harrell	S	SC				
~~T12-M-H3~~				S	SC				~~———~~ 8/22/14
T12-M-L1	8/23/14	1200	Mike Harrell	S	SC				
T12-M-H3	8/26/14	1313	Mike Harrell	S	SC				

* Sample Type: S = Solids L = Liquid O = Other (describe)
Sample Container: SC = Stainless Steel Sample Cup C= Core Sampler V= Vaccum O = Other (describe)

Sample labeled T12-M-H2 taken from location T12-M-L1. 8/23/14
Sample labeled T12-M-H1 taken ~~from location T12-M-L2.~~
Sample labeled T12-M-L1 taken from Location T12-M-L3 (H2) (H2)

8/26/14
Sample labeled T12-M-L3 taken from location T12-M-H1
Sample labeled T12-M-H3 taken from location T12-M-H2
Sample labeled T12-M-L2 taken from location T12-M-H

7.18 Chain of Custody, Cont'd

2 of 2

Sample ID (see TSAP)	Sample Collection					SCO or AD Sample ID Verifier Name, Date, Time			SRNL Radioactive Material Receipt Entry Log #
	Date	Time	Collector	Sample Type *	Sample Container *	Date	Time	ID Verifier	
T12-M-L2	8/26/14	1343	Mike Harrell	S	SC				
T12-M-L3	8/26/14	1230	Mike Harrell	S	SC				

	Signature	Printed Name	Organization	Date	Time
Relinquished By (PIC):	[signature]	Mike E. Harrell	Closure	8/27/14	1408
Received By:	P Burkhalter	P Burkhalter	SCO	8/27/14	1409
* Relinquished By:	P Burkhalter	P Burkhalter	SCO	8/28/14	959
Received By:	Lawrence Oji	LAWRENCE OJI	SRNL	8/28/14	10:00
Relinquished By:					
Received By:					
Relinquished By:					
Received By:					

Notes: Sample containers opened 8/29/14 and matched according to Chandler's e-mail of 8/28/14. Pictures were also taken. The Six Sample containers were each filled with sludge and no visible liquid fraction

* Sample Type: S = Solids L = Liquid O = Other (describe)
Sample Container: SC = Stainless Steel Sample Cup C= Core Sampler O = Other (describe)

Appendix C-b: Tank 12H Cooling Coil and Floor Liquid Fraction Sample Scoping Analysis Data

Scoping Analysis Data for Tank 12H Cooling Coil and Floor Liquid Fraction

SRR initially requested processing and analysis of Tank 12H cooling coil scrape and floor samples as detailed in Appendix C of the Liquid Waste (LW) Technical Task Request (TTR), ["Tank 12H- Waste Characterization & Sample Analysis Report", G-TTR-H-00008, Rev. 1, Sept. 10, 2014].

The bulk densities of the three cooling coil scrape samples (T12-R8-C-Low, T12-R8-C-Mid and T12-R8-C-High) were determined in triplicate as soon as the samples were delivered to SRNL as shown in Table C-B1 [i]. Because of the high bulk densities of the coil scrape samples, SRR requested X-Ray Florescence (XRF) and X-Ray Diffraction (XRD) characterizations on coil scrape sample T12-R8-C-Mid. The Mid coil scrape sample was selected for the initial Tank 12H cooling coil scoping analysis because it had the most mass of the three cooling coil scrape samples.

Using the ground and homogenized material from the Mid coil sample, acid digestions (Aqua Regia [AQR] and Peroxide Fusion [PF] digestions) were performed and the resulting acid digestion solutions were analyzed for:

a) Routine elemental constituents by Inductively Coupled Plasma – Emission Spectroscopy (ICP-ES);
b) Mercury by Cold Vapor Atomic Absorption (CVAA);
c) Co-60, Cs-134, Cs-137, Eu-154, and Am-241 by Gamma Spectroscopy (GS) and Cesium-Removed Gamma Spectroscopy (CRGS);
d) Total alpha and non-volatile beta by Liquid Scintillation Counting (LSC); and
e) Tc-99, I-129, Pu-238, and Pu-239/240 by nuclide-specific analytical methods.

In these scoping tests, all analytical measurements were determined only once (no replicates).

The first three floor samples (T12-F-1, T12-F-2 and T12-F-3) and the sixth (T12-F-6) contained mostly liquids without recoverable solid fractions. The first three locations were re-sampled and identified as T12-F-1R, T12-F-2R and T12-F-3R. Samples T12-F-4 and T12-F-5 contained mostly wet and pasty solids without significant liquid fractions. At SRR's request, the liquid portions of all the floor samples were filtered (liquid solids/separation) using a Nalgene filter unit (0.45 micron nylon membrane). The resulting liquid was combined in one container and had a final volume of approximately 167 mL. The combined liquid fraction was analyzed for:

a) Anions by Ion Chromatography (IC);
b) Routine elemental constituents by ICP-ES;
c) Mercury by CVAA;
d) Co-60, Cs-137, Eu-154, and Am-241 by GS and CSGS;
e) Total alpha and non-volatile beta by LSC; and
f) Tc-99, I-129, Pu-238, and Pu-239/240 by nuclide-specific analytical methods.

Figures and tables below contain the summarized scoping analysis results for the T12-R8-C-Mid cooling coil sample and the combined floor sample. Figures C-B1A and C-B1B, show respectively, the XRF and XRD spectra for the T12-R8-C-Mid cooling coil scrape solids. These results show that the coil scrape solids contain mainly mercuric oxide and a small amount of aluminum hydroxide material (possibly gibbsite). This characterization result is supported by the CVAA Hg analysis and the ICP-ES analyses which show that the coil scrape sample contains 98.2 weight percent mercury and only 0.233 weight percent aluminum (Table C-B2).

The ICP-ES analytical results for the coil scrape solids also show other detectable elemental constituents present at low concentrations including: barium, calcium, chromium, iron, magnesium, manganese, sodium, nickel, zinc, and zirconium (Table C-B2).

The characterizations for Cs-134, Cs-137, Eu-154 and Am-241 from PF and AQR digestion are presented in Table C-B3. The CRGS results for Co-60, Eu-154, and Am-241 are presented in Table C-B4. Table C-B4 also presents the analytical results for total alpha, total non-volatile beta, Tc-99, I-129, Pu-238, and Pu-239/240. As shown in these tables, all of the radiological constituents were present at detectable concentrations, with exception of short-lived Cs-134, and total alpha, which had a high minimum detection limit due to the high beta content. A better estimate of the total alpha concentration can be determined by summing the measured Pu-238, Pu-239/240, and Am-241 concentrations (sum = 9.14 μCi/g).

IC analysis results on the combined floor sample liquid fraction show measurable concentrations of anions, mostly chlorides, nitrites, nitrates and sulfates as shown in Table C-B5.

The ICP-ES and CVAA analysis results for the Tank 12H combined liquid fraction are presented in Table C-B6. Detectable constituents included: aluminum, calcium, chromium, iron, potassium, magnesium, manganese, sodium, silicon, strontium, and uranium.

As summarized in Table C-B7, radionuclides in the Tank 12H combined liquid fraction included Cs-137, which was the dominant radionuclide identified, and lower but measurable concentrations of Pu-238, Pu-239/240, Eu-154, Tc-99, and I-129.

Table C-B1 Tank 12H Cooling Coil Scrape Sample Bulk Densities

Sample ID	Sample wt., g	As-received Bulk density, g/mL	Bulk density after homogenizing, g/mL	Wt.% solids
T12-R8-C-Low	21	3.05 ± 0.02	4.26 ± 0.01	98.1 ± 0.4
T12-R8-C-Mid	47	3.01 ± 0.14	4.81 ± 0.01	97.8 ± 0.7
T12-R8-C-High	26	3.18 ± 0.18	4.82 ± 0.01	98.0 ± 0.3

[i]L. N. Oji, "Bulk Density and Weight Percent Solids for Tank 12H Cooling Coil, Floor, and Mound Samples" SRNL-L3100-2014-00245, Revision 0, October 20, 2014.

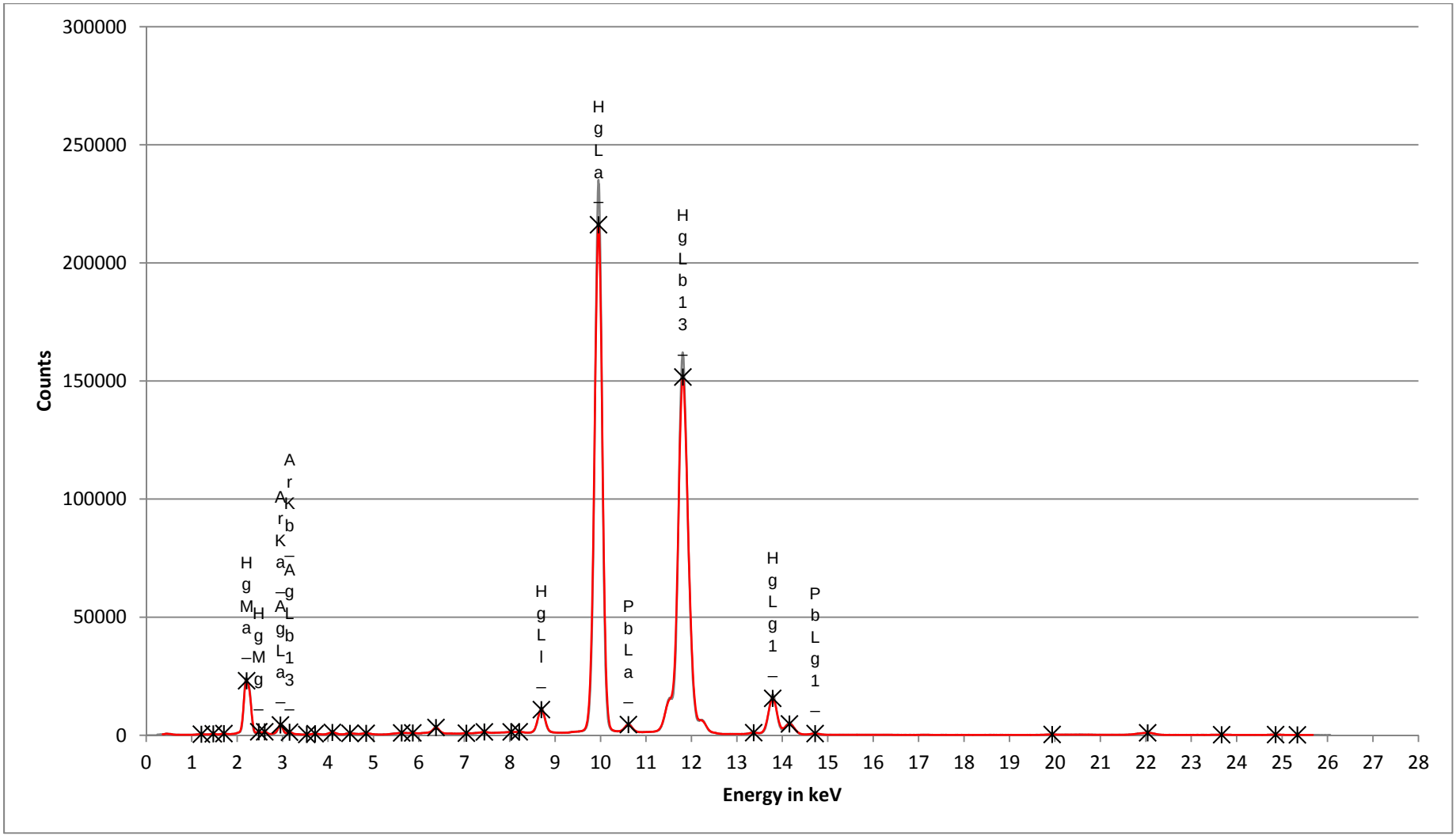


Figure C-B1A: XRF for Tank 12 Coil Sample T12-R8-C-Mid

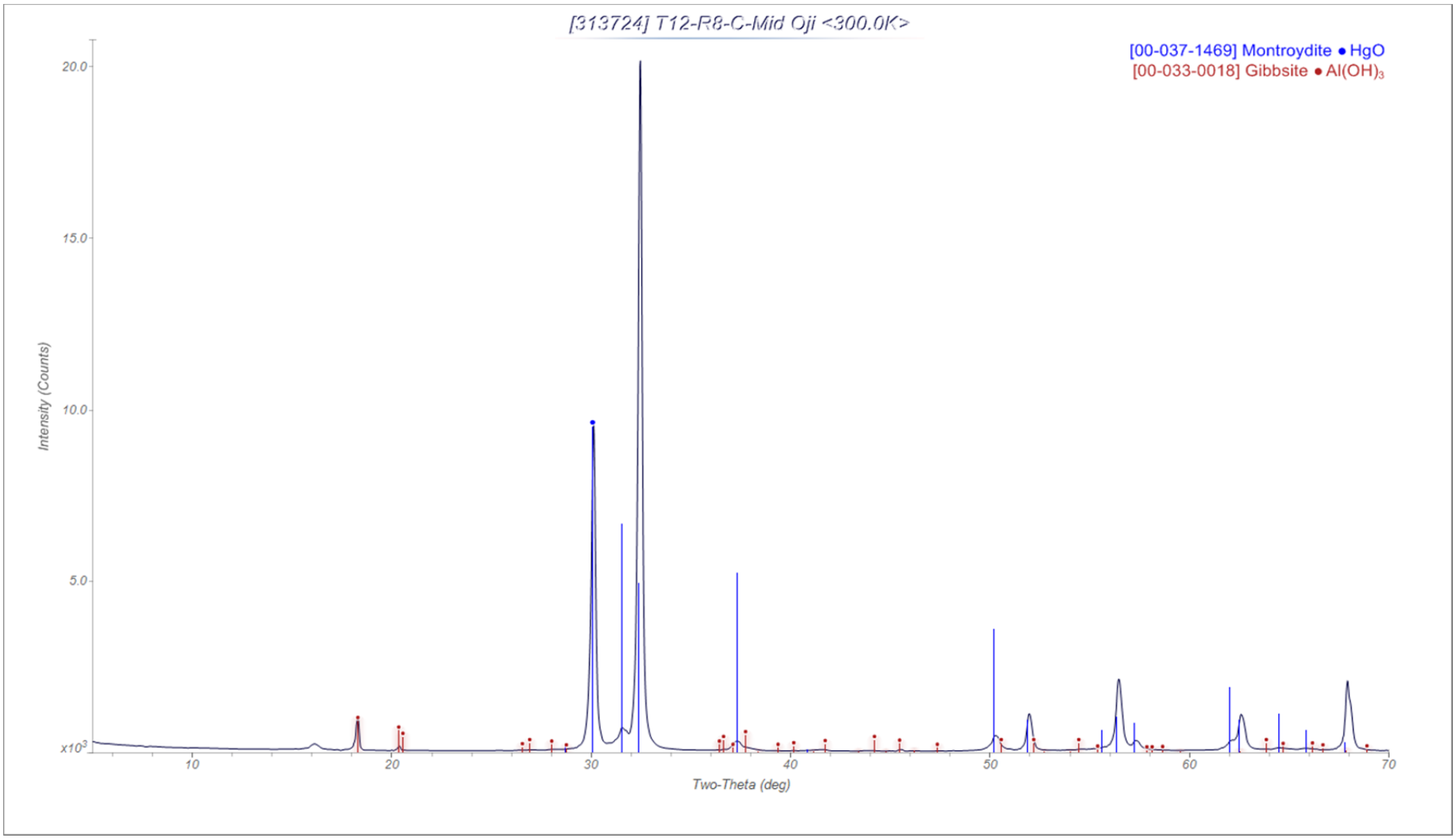


Figure C-B1B: XRD for Tank 12 Coil Sample T12-R8-C-Mid

Table C-B2 Tank 12H Coil Sample T12-R8-C-Mid:
AQR digestion (ICP-ES and CVAA Results: LIMS # 300313866)

Component	Wt%
Ag	<1.00E-02
Al	2.33E-01
B	<1.69E-02
Ba	1.96E-03
Be	<1.09E-04
Ca	≤7.03E-03*
Cd	<9.88E-04
Ce	<8.72E-03
Co	<1.36E-03
Cr	2.68E-03
Cu	<2.75E-03
Fe	8.07E-02
Gd	<3.46E-03
K	<3.04E-02
La	<1.53E-03
Li	<9.87E-03
Mg	≤1.97E-03*
Mn	2.71E-02
Mo	<1.40E-02
Na	≤1.26E-01*
Ni	6.35E-03
P	<3.68E-02
Pb	<1.01E-01
S	<9.34E-01
Sb	<3.20E-02
Si	<1.32E-02
Sn	<7.24E-02
Sr	<1.00E-03
Th	<9.03E-03
Ti	<7.24E-04
U	<5.44E-02
V	<5.37E-04
Zn	1.47E-03
Zr	2.45E-03
Hg	9.82E+01

*These results are considered upper limits since blank concentrations were greater than 10% of the sample concentrations

Table C-B3 Tank 12H Coil sample T12-R8-C-Mid:

Component	PF digestion/ GAMMA SPEC (LIMS #300313868)	AQR digestion/GAMMA SPEC (LIMS #300313866)
Cs-134, µCi/g	<5.59E-03	Not detected
Cs-137, µCi/g	1.25E+00	1.34E+00
Eu-154, µCi/g	1.28E-01	1.85E-01
Am-241, µCi/g	3.50E-01	5.23E-01

Table C-B4 Tank 12H Coil sample T12-R8-C-Mid:

Component	Result	Method/ LIMS #
Co-60, µCi/g	1.88E-03	Cesium-Removed Gamma Spec (LIMS #300313866)
Eu-154, µCi/g	1.95E-01	Cesium-Removed Gamma Spec (LIMS #300313866)
Am-241, µCi/g	5.77E-01	Cesium-Removed Gamma Spec (LIMS #300313866)
Total Alpha, µCi/g	<6.35E+00	Total Alpha (LIMS #300313866)
Non-vol. Beta, µCi/g	1.83E+02*	Total Alpha and Non-Volatile Beta (LIMS #300313866)
Tc-99, µCi/g	9.64E-03	Tc-99 (LIMS #300314152)
I-129, µCi/g	1.29E-03	I-129 (LIMS #300314152)
Pu-238, µCi/g	8.38E+00	Pu-238 and Pu-239/240 (LIMS #300313868)
Pu-239/240, µCi/g	1.84E-01	Pu-238 and Pu-239/240 (LIMS #300313868)

*Sum of cesium-removed non-volatile beta and Cs-137

Table C-B5 Tank 12H Combined Floor Liquid Fraction:

IC-Anions (LIMS #300313925)

Component	Result
Fluoride, µg/mL	<1.0E+01
Formate, µg/mL	<1.0E+01
Chloride, µg/mL	3.0E+01
Nitrite, µg/mL	2.0E+01
Bromide, µg/mL	<1.0E+01
Nitrate, µg/mL	1.13E+02
Phosphate, µg/mL	<1.0E+01
Sulfate, µg/mL	5.7E+01
Oxalate, µg/mL	<1.0E+01

Table C-B6 Tank 12H Combined Floor Liquid Fraction: ICP-ES and CVAA (LIMS #300313925)

Component	mg/L
Ag	<3.90E-02
Al	1.12E+00
B	<4.35E-01
Ba	<2.00E-02
Be	<3.00E-03
Ca	5.01E-01
Cd	<2.50E-02
Ce	<2.24E-01
Co	<3.50E-02
Cr	1.68E-01
Cu	<7.10E-02
Fe	1.04E+00
Gd	<8.90E-02
K	1.59E+01
La	<3.90E-02
Li	<2.54E-01
Mg	1.01E-02
Mn	9.05E-02
Mo	<3.59E-01
Na	1.17E+03
Ni	<1.02E-01
P	<7.15E-01
Pb	<2.60E+00
S	<2.40E+01
Sb	<8.22E-01
Si	8.72E-02
Sn	<1.86E+00
Sr	1.17E-02
Th	<1.00E+00
Ti	<1.90E-02
U	3.78E+01
V	<1.40E-02
Zn	<2.10E-02
Zr	<1.20E-02
Hg	2.21E+01

Table C-B7 Tank 12H Combined Floor Liquid Fraction: Radionuclides (LIMS #300313925)

Component	Result	Units
Pu-238	8.33E-01	µCi/L
Pu-239/240	6.49E-02	µCi/L
Co-60*	<4.08E-03	µCi/L
Eu-154*	4.55E-02	µCi/L
Am-241*	<7.97E-02	µCi/L
Total Alpha	<3.17E+00	µCi/L
Non-vol. Beta	9.28E+02	µCi/L
Cs-137**	2.17E+02	µCi/L
Eu-154**	<6.71E+00	µCi/L
Am-241**	<1.30E+01	µCi/L
Tc-99	2.25E-02	µCi/L
I-129	8.83E-03	µCi/L

*These results determined by cesium-removed gamma spec
**These results determined by routine gamma spec (without cesium removal)

Appendix D: Statistical Summary of the Analytical Results

Appendix D describes the statistical methods used to summarize the analytical results in Section D.1 and the application of these methods to the concentration results for the analytes in the residual solids in Tank 12H in Section D.2. Summary tables of the analytical results are provided in Appendices E-1 through E-8, and statistical summaries are provided in Appendices E-9 through E-20. All of the analytical results for statistical consideration were communicated by Oji[D-1].

D.1 STATISTICAL METHODS

This section describes the statistical methods used in this report. It is partitioned into the following subsections. Subsection D.1.1 describes the statistical methods when all results are above the minimum detectable concentrations (above-MDC). Above-MDC results are measurements. Subsection D.1.2 describes the statistical methods when all results are below the minimum detectable concentrations (below-MDC). These are results without detection of an analyte. Subsection D.1.3 describes the statistical methods when there is a mixture of above-MDC and below-MDC results.

Refer to Figure D-1. When all analytical results are above-MDC then the statistical summary consists of an estimate and an upper 95% confidence limit (UCL95) for the mean of the analyte concentration in the Tank 12H residuals. Estimates are given for the standard deviation for a composite sample measured once and its associated percent standard deviation. When all results are below-MDC, no estimates of the mean and standard deviation can be made. The results are summarized by the smallest and largest MDC. When some analytical results are reported as above-MDC and other analytical results are reported as below-MDC, then the methods for the statistical analyses are more complex, but the interpretation of the results is similar to those two cases. Figure D-1 shows that when a sufficient number[a] of above-MDC results exist on at least two of the composite samples, then the mean, standard deviation, percent standard deviation, and UCL95 estimates are reported. When a sufficent number of above-MDC results do not

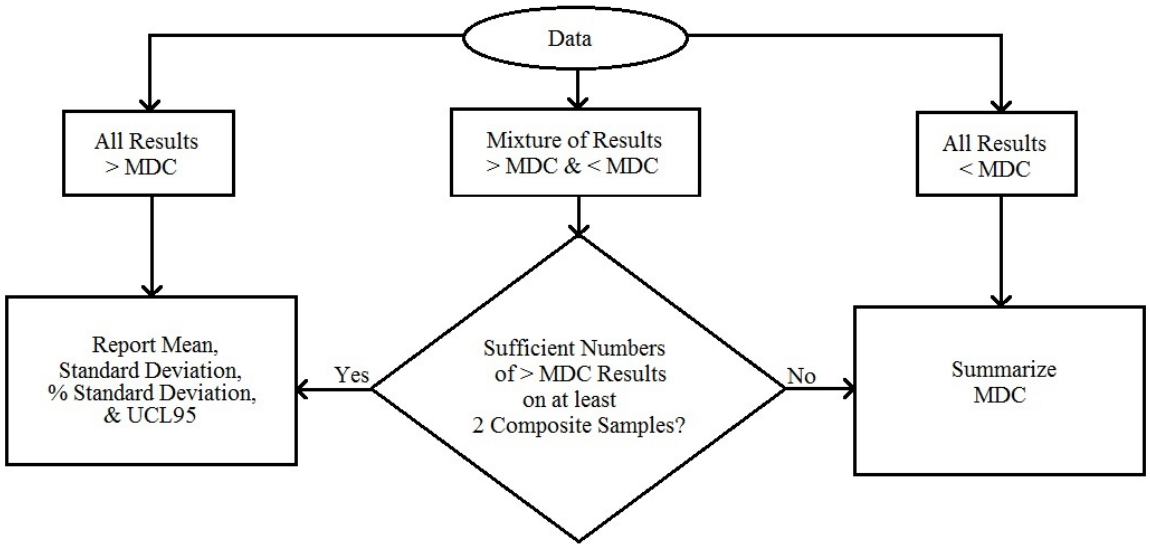


Figure D-1. The Relationship between the Type of Data Reported and the Statistical Results

[a] When the concentration data for an analyte are a mixture of above-MDC results and below-MDC results, there are no closed-form formulas: the procedure is iterative. So a "sufficient number" of above-MDC results is operationally defined as the number of above-MDC results needed for the algorithm to converge to a solution.

exist on at least two composite samples, then the sampling variance cannot be estimated from the analytical data, nor can a test be performed to distinguish between models with and without sampling effects. Those results are summarized by MDC.

D.1.1 Methods when all Results are above-MDC

The material in each composite sample is considered to be a representative sample of all of the residual material on the floor of Tank 12H, and thus the measured concentration for any analyte in a composite sample is considered to be an estimate of the actual mean concentration of the analyte in the residual material on the entire tank floor. Three replicate concentration measurements, j = 1, 2, 3, were performed for each analyte on each of three composite samples, i =1, 2, and 3. When all nine analytical results are above-MDC, that is, all nine results are measurements, the statistical measurement error model for concentration measurement Y_{ij} is

$$Y_{ij} = \mu + s_i + \varepsilon_{ij} . \quad (1)$$

The actual mean analyte concentration for all of the residual material on the floor of Tank 12H is μ. The random sampling error for Composite Sample i is the random effect s_i which is distributed with mean zero and standard deviation σ_s. The sampling error s_i is the difference between the actual mean concentration in Composite Sample i and the actual mean concentration for all of the residual material on the tank floor. Typically, this error can arise from spatial heterogeneity of the analyte concentration in the residual material in Tank 12H, sampling errors, sample material preparation errors, and volumetric proportion errors. The random measurement error for replicate measurement j for Composite Sample i is ε_{ij}, and it is distributed with mean zero and standard deviation σ_ε. The measurement error ε_{ij} is the difference between concentration measurement j for Composite Sample i and the actual mean concentration for Composite Sample i. Typically, a measurement error can arise from spatial heterogeneity of the analyte concentration within the sampled material, aliquoting errors, and the errors in the measurement process itself.

A test for heterogeneity of measurement variance σ_ε^2 was performed prior to other analyses in order to verify the assumptions that the composite sample material is well-mixed and the measurement variance σ_ε^2 is the same for all composite samples, and to determine whether an analysis of variance (ANOVA) F test or a Welch's F test should be used to test[a] the null hypothesis $\sigma_s = 0$. The test procedure is the Levene's test with a Type I[b] family-wise[c] error rate α_{family} = 0.05. Since the sample sizes are small (no more than three measurement results per composite sample), a Bonferroni procedure, see Alt[D-2] for a general discussion, is used to control for spuriously significant results by dividing the 0.05 family-wise Type I error rate by the number of comparisons in order to obtain the Type I error rate per comparison α. The Bonferroni criteria for individual analyte tests are α = 0.05/23 = 0.002174 for the set of 23 radionuclides that had all of the results above-MDC, α = 0.05/19 = 0.002632 for the set of 19 elemental constituents that had all results above-MDC, and α = 0.05/5 = 0.01 for the set of 5 anions that had all

[a] The F test assumes that the measurement error standard deviation is the same for each composite sample. If it is not the same, the Welch's test is used instead.

[b] A Type I error is defined as incorrectly rejecting the null hypothesis, in this case, incorrectly stating that the measurement error standard deviation differs among the composite samples.

[c] A family-wise error rate refers to the error rate of making at least one Type I error (rejecting the null hypothesis when it is true) in a prescribed family or set of tests, where "family" refers to all of the analytes that have all above-MDC results in the set of radionuclides, in the set of elemental constituents, or in the set of anions. Controlling the family-wise error rate means that the probability of making at least one Type I error for an analyte in a family will be no more than a stated probability: 0.05 in this instance.

results above-MDC. If the Levene's P-value for an individual analyte test is less than the Bonferroni α criterion, then it is concluded that the measurement error variances are not the same for all of the composite samples for this analyte.

When Levene's test is not statistically significant, an analysis of variance (ANOVA) F test is performed in order to determine whether the random effects s_i , i = 1, 2, and 3, are warranted in Eqn (1). The null hypothesis is $\sigma_s = 0$. If the F test indicates a statistically significant sampling standard deviation at a level of significance $\alpha = 0.05$, then Eqn (1) with the sampling effect s_i becomes the basis for estimating the true mean concentration in the residual material; if the ANOVA F test result is not statistically significant, then the random effect s_i is not needed and Eqn (1) reduces to Eqn (2).

$$Y_{ij} = \mu + \varepsilon_{ij} \tag{2}$$

When Levene's test is statistically significant, a Welch's ANOVA test is performed instead of the ANOVA F test.

If all of the concentration measurements for an analyte are above-MDC, then the ANOVA F test can be performed, and a decision is made to use the model in Eqn (1) with the sampling effect if $F \geq F_{0.95,2,6} = 5.14325,$ and to use the model in Eqn (2) without the random effect if $F < F_{0.95,2,6} = 5.14325.$ When $F \geq F_{0.95,2,6} = 5.14325,$ the UCL95 for the actual mean tank concentration μ is given by

$$UCL_{95\%} = \bar{Y}_{\bullet\bullet} + t_{0.95,2df} \cdot \sqrt{\frac{MS_{Sample}}{9}}, \tag{3}$$

where $\bar{Y}_{\bullet\bullet}$ is the sample mean concentration of the nine concentration measurement results, and MS_{Sample} is the estimate of the mean square for the sampling effect s_i in the model in Eqn (1), where

$$MS_{Sample} = \frac{\sum_{i=1}^{3} \frac{Y_{i\bullet}^2}{3} - \frac{Y_{\bullet\bullet}^2}{9}}{2}, \tag{4}$$

and $Y_{i\bullet}$ and $Y_{\bullet\bullet}$ are the total of the three measured concentration results for Composite Sample *i*, i = 1, 2, 3, and the total of the nine measured concentration results for all three composite samples, respectively. The estimated standard error of the mean concentration is the square root of $MS_{Sample}/9$ when all composite samples have three measurements.

When $F < F_{0.95,2,6} = 5.14325,$ the UCL95 for the actual mean concentration in Tank 12H is given by

$$UCL_{95\%} = \bar{Y}_{\bullet\bullet} + t_{0.95,9-1df} \cdot \sqrt{\frac{s^2}{9}}, \tag{5}$$

where *s* is the sample standard deviation of all nine measured concentration results.

The above procedures are appropriate if the data follow the normal distribution. Potential outliers can be observed from graphs. When the model in Eqn (2) without the sampling effect is adopted, there is sufficient data to run certain diagnostic tests for goodness-of-fit and outliers. Figure D-2 presents a sequence of goodness-of-fit tests to identify a distribution consistent with the measurement results and an estimation method for the mean, standard deviation, percent standard deviation, and UCL95. Studies by Singh, Singh, and Englehardt[D-3] demonstrate that using the coefficient of variation (the percent standard

deviation) is much less effective than using a formal goodness-of-fit test to determine whether the concentration measurements are consistent with a particular distribution such as the normal distribution. Consequently, the normal distribution assumption is tested by the Wilk-Shapiro (W-S) goodness-of-fit test at $\alpha = 5\%$ level of significance. If the P-Value for the W-S goodness-of-fit statistic is less than or equal to $\alpha = 0.05$, then the normality hypothesis is rejected, and if the P-Value for the W-S goodness-of-fit statistic is greater than $\alpha = 0.05$, then the normality hypothesis is adopted. If there is no statistically significant departure from normality, the mean, standard deviation, percent standard deviation, and UCL95 are estimated based on a normal distribution.

If the normal distribution assumption is rejected by the W-S test, then the measurements are tested to determine whether they are consistent with a right-skewed distribution. This report adopts the strategy in Singh, Armbya, and Singh[D-4] to test for the gamma distribution prior to the lognormal distribution. The gamma distribution assumption is tested using the Anderson-Darling (A-D) goodness-of-fit statistic. If the A-D statistic exceeds the A-D critical value then the gamma distribution assumption is rejected; if there is no statistically significant departure from the gamma distribution, the mean, standard deviation, percent standard deviation, and UCL95 are determined based on a gamma distribution. If the gamma distribution is rejected, but a plot of the concentration results versus the theoretical gamma quantiles displays a linear pattern with high correlation (over 95%), then the results are said to follow an approximate gamma distribution. The mean, standard deviation, percent standard deviation, and UCL95 are estimated assuming a gamma distribution, according to Singh, Armbya, and Singh[D-4].

If the gamma distribution is rejected and the gamma quantile plot does not exhibit high correlation (> 95%), then the W-S goodness-of-fit test is used to determine if the measurements are consistent with the lognormal distribution. If the P-value for the W-S statistic for the goodness-of-fit to a lognormal distribution is greater than $\alpha = 0.05$, then the lognormal distribution is adopted. If the P-value is less than or equal to $\alpha = 0.05$, then a nonparametric approach is used for estimation. Additional details are documented by Singh, Singh, and Englehardt[D-3].

The W-S goodness-of-fit tests were performed in JMP® Pro Software (64-bit) Version 11.2.1 from SAS® Institute[D-5], and all goodness-of-fit tests except the W-S goodness-of-fit tests and every estimate of the mean, standard deviation, and UCL95 were computed by the ProUCL Version 5.0 software application from the U.S. Environmental Protection Agency[D-4] when the model in Eqn (2) without the sampling error was adopted.

When the model in Eqn (1) with the sampling effect is adopted, there are too few measurements to provide a reasonable test for goodness-of-fit, so no formal goodness-of-fit test is routinely performed. A visual check for outliers is performed on graphs. When the model in Eqn (1) with the sampling effects has been chosen, estimates of the mean, standard deviation, percent standard deviation, and UCL95 were computed using restricted maximum likelihood estimation (REML) using the "Fit Model" platform in JMP® Pro Software (64-bit) Version 11.2.1. The REML algorithm is described by McCullagh and Nelder[D-6]. When all 9 results for an analyte are above-MDC, that is, when all 9 results are measurements, then REML estimates coincide with the formulas in Eqn (3) and Eqn (4).

The examination of the data for potential outliers is highly important. This can be done visually by examining graphs, but a statistical test can provide a better basis for deciding whether a concentration result conforms to the pattern of the rest of the data. Outliers were assessed graphically and by the Dixon Q test, described in Steel and Torrie[D-8], when the model in Eqn (2) without the sampling effect was adopted. The Dixon Q test was performed by the ProUCL 5.0 software application written by Singh, Armbya, and Singh[D-4]. The null hypothesis of the Q test is that there is no outlier. Rejecting the null hypothesis at a 5% level of significance is evidence that a concentration result does not appear to conform to the general pattern of the rest of the concentration data. If a Dixon Q test is statistically significant at

α = 0.05, then the statistical procedures are rerun omitting the potential outlier. This report adopts the convention of reporting the results associated with the larger UCL95 from the procedure run with and without the potential outlier.

An outlier test was not routinely performed when the model in Eqn (1) with the sampling effect was adopted. However, measurement variation across the composite samples was assessed by the Levene's test as previously discussed for the model in Eqn (1). Outliers were visually screened using plots provided in the supporting tables for each analyte. The plot for one analyte, Am-243, suggests that one of the results for Composite Sample 2, a below-MDC result, might be an outlier. This was checked using the Cook's D, assuming fixed sampling effects, and conservatively treating the below-MDC value as though it were above-MDC for the purpose of assessing its influence. Belsey, Kuh, and Welsch[D-9] describe Cook's test.

Software validation and verification for SAS JMP® Pro 11.2.1 and ProUCL 5.0 are documented by Baker, et al[D-7].

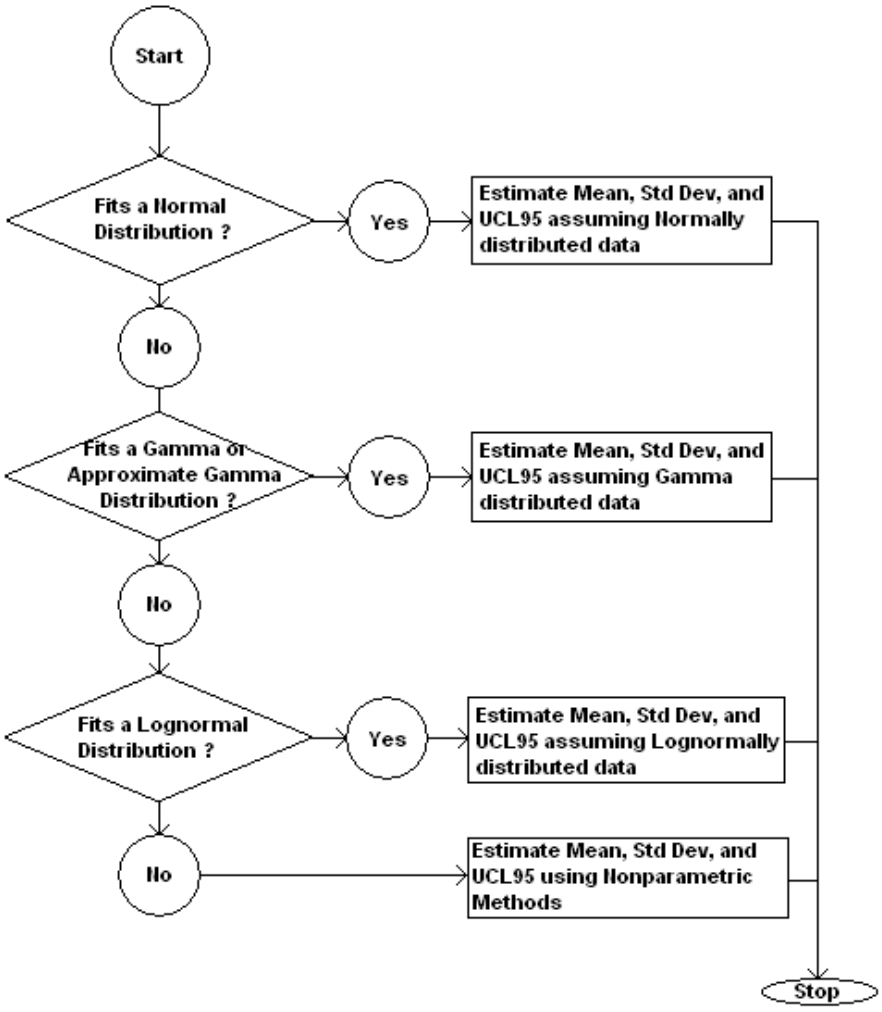


Figure D-2. Sequence of Goodness-of-Fit Tests to Identify a Distribution and Select an Estimation Method

D.1.2 Statistical Methods when All Analytical Results are less-than-MDC

When a sample has all less-than-MDC results, the analyte has not been detected in the sampled material. No mean, standard deviation, percent standard deviation, or UCL95 estimate can be computed. Each of the three composite samples is described by the smallest and the largest of the three reported MDC results for the sample. The smallest and the largest of the nine MDC results are also reported for that analyte.

D.1.3 Statistical Methods when Analytical Results Contain a Mixture of above-MDC and below-MDC Results

When a sufficient number of greater-than-MDC results are reported, then a decision must be made to use a model with sample effects similar to Eqn (1) or a model without sample effects similar to Eqn (2). Each below-MDC result is treated as an interval-censored result. An interval-censored result is one in which the concentration is only known within a range: in this case between 0 and the MDC. The software application SAS Proc Reliability®[D-10] is used to provide estimates for the parameters in a model of the form of Eqn (1) but with fixed sampling effects. An example illustrating the computations in this discussion is given in Appendix E-13 for Pa-231. The model in SAS® contains a term labeled the "Intercept" which is the mean concentration for Composite Sample 3. The sampling effects s_i are referred to as "Csamp i" in the SAS® Proc Reliability output, where Csamp i = mean concentration of Composite Sample i – mean concentration of Composite Sample 3, i = 1, 2, 3. By definition, Csamp 3 is zero. The model is assumed appropriate if at least one of the fixed sample effects Csamp i for Composite Samples 1 or 2 is more than twice its estimated standard error.

Suppose the model with sampling effects is determined to be appropriate. The procedure for determining a UCL95 under a sampling error model when the data contain below-MDC results has two steps. In the first step, a representive concentration value is determined for each composite sample using SAS® Proc Reliability. Had all the analytical results been above-MDC, an alternative approach to the one outlined in Section D.1.1, would be to compute the mean of the three measurements separately for each composite sample, then treat the three composite sample means as though they are a random sample from the population of composite sample means in order to compute the UCL95. In the similar manner, the mean concentration for Composite Sample *i* is estimated by Eqn (6).

$$Mean\{\text{Composite Sample } i\} = \text{Intercept} + \text{Csamp } i,\ i = 1, 2, 3 \qquad (6)$$

Once the mean concentration is estimated for each composite sample, the second step determines the mean of the 3 composite sample means, the standard deviation of the 3 composite sample means, and the UCL95 based on the 3 composite sample means using the JMP® Pro Version 11.2.1 software platform "Fit Distribution". The standard deviation $StdDev_{Comp.SampleMeans} = \sqrt{Var_{Comp.SampleMeans}}$ of the 3 composite sample means is based on a mean of 3 measurements per composite sample. The standard deviation of the 3 composite sample means for the analyte concentration is adjusted to reflect the uncertainty of a single measurement instead of the uncertainty from the mean of 3 measurements by the formula in Eqn (7), where $Var_{MeasError}$ is the square of the "Scale" estimate in the SAS® Proc Reliability output.

$$Std.Dev. = \sqrt{Var_{Comp.SampleMeans} - Var_{MeasError}/3 + Var_{MeasError}} \qquad (7)$$

Now suppose that a model without sampling effects such as that in Eqn (2) is appropriate. Then a model without fixed sampling effects is run in SAS® Proc Reliability. An example of this is shown in Appendix E-17 for Mo. In that table the standard errors (under the header "Std Error") of the estimates for Csamp 1 and Csamp 2 are larger than the estimates themselves. Therefore, a model without sampling

effects is adopted. The model without sampling effects has a parameter labeled "Intercept", but it is defined as the mean of all of the composite sample concentrations. The reported mean and standard deviation for the analtye are given under the header "Estimate" on the lines identified as "Intercept" and "Scale", respectively. Using the SAS® Proc Reliability output values for the "Estimate" and the "Std Error" for the "Intercept" parameter, the UCL95 is computed by Eqn (8).

$$UCL95 = Estimate + t_{0.95,DF} \cdot Std\ Error\ , \tag{8}$$

where DF equals the number of above-MDC results minus 1.

When the number of above-MDC results is insufficent for the SAS® Proc Reliability to converge for estimates in the model with sampling effects, but at least 2 above-MDC results occur on one composite sample, then SAS® Proc Reliability is used to fit the mean for that one composite sample. Consider, for example, the radionuclide Cm-245 in Appendix E-13. Cm-245 has only 2 above-MDC values on Composite Sample 2. A model with just an "Intercept" parameter was run using only the results for Composite Sample 2. The estimate of the measurement standard deviation is reported as the "Estimate" in the output for the "Scale" parameter. Then the UCL95 was computed separately for each above-MDC result x by Eqn (9), where s is the Scale estimate.

$$UCL95 = x + t_{0.95,1df} \cdot s\ . \tag{9}$$

The UCL95 replaces the above-MDC result in the working data set for the analye and is interpreted as if it is the MDC for a below-MDC result. Then the working data set consists entirely of below-MDC results, and the statistical summary follows the paradigm for analytes with all below-MDC results.

All mixtures of above-MDC and below-MDC results for Tank 12H were able to be accomodated by the preceding methods.

D.2 STATISTICAL ANALYSES FOR TANK 12H ANALYTE CONCENTRATIONS

The statistical analyses are based on the analytical results[a] presented in Appendices E-1 through E-3 for radionuclides, Appendices E-4 through E-6 for elemental constituents, and Appendices E-7 and E-8 for anions. The analytical data are also partitioned into three separate classes: analytes with all results above minimum detectable concentrations (above-MDC), that is, all measurements; analytes with all results below minimum detectable concentrations (below-MDC); and analytes with a mixture of results that are above-MDC and below-MDC. This allows more uniform reporting of results, as analytes within any particular class tend to have similar statistical analyses. Refer to Figure D-1 for a pictoral presentation of these classes. The following sections describe the application of the statistical methods described in Section D.1.

D.2.1 Analysis of Radionuclides

Analyses were performed for 36 radionuclides. There were 23 radionuclides with all of the results above-MDC, 9 radionuclides with all of the results below-MDC, and 4 radionuclides with a mixture of above-MDC and below-MDC results. Only one radionuclide, Th-229, had a missing result. However, the eight reported results for Th-229 were all below-MDC. Table D-1 partitions the radionuclides by these classes for Tank 12H.

[a] The concentration results listed in Appendices E-1 through E-8 have been rounded to three digits. Minor differences in the last decimal place between the values of the means and the standard deviations for the concentrations of individual composite samples in Appendix E and in the main body of the report (Tables 6-14) may exist due to rounding.

Table D-1. Radionuclides by Statistical Classification

Summarized by UCL95				Summarized by MDC		
All Results above-MDC			Results above-MDC & below-MDC	Results above-MDC & below-MDC	All Results below-MDC	
Data in Appendix E-1			Appendix E-3	Appendix E-3	Appendix E-2	
Statistical Summary in Appendix E-9			Appendix E-11	Appendix E-12	Appendix E-10	
Supporting Results in Appendix E-13			Appendix E-13	Appendix E-13	Appendix E-13	
Am-241	Pu-238	Th-232	Am-242m	Cm-245	C-14	Ra-226
Ba-137m	Pu-239	U-232	Am-243		Cm-243	Th-229
Co-60	Pu-240	U-234	Pa-231		Cm-244	Th-230
Cs-137	Pu-241	U-235			Cs-135	U-233
I-129	Ra-228	U-238			Nb-94	
Ni-59	Sn-126	Y-90				
Ni-63	Sr-90	Zr-93				
Np-237	Tc-99					

The statistical summary for radionuclides with all above-MDC results is presented in Appendix E-9. All of the radionuclides with all above-MDC results but I-129, Tc-99, and U-232 had a statistically significant sampling variance. There was no statistically significant heterogeneity among measurement variances across samples for any radionuclide. All UCL95 determinations were based on a normal distribution for the mean.

The nine radionuclides with all below-MDC results are summarized in Appendix E-10.

There were four radionuclides that had a mixture of above-MDC and below-MDC results: Am-242m, Am-243, Pa-231, and Cm-245. Of these Cm-245 had only two above-MDC results on Composite Sample 2. This was insufficient to compute a UCL95, and Cm-245 was summarized by MDC. Am-243 had only one below-MDC result (< 4.49E-3 μCi/g) on Measurement 3 of Composite Sample 2, and it appears to be a potential outlier based on a graphical observation and the value of Cook's D. The statistical analyses were performed with and without this result in the data set. The reported results for Am-243 were based on the concentration data without the potential outlier producer. The UCL95 was higher when the potential outlier was omitted.

D.2.2 Analysis of Elemental Constituents

Twenty-two elemental constituents were characterized. Of those, 19 have all above-MDC results, 2 have all below-MDC results, and only 1 has a mixture of above-MDC and below-MDC results. Table D-2 partitions the elemental constituents by these classes for Tank 12H.

The statistical summary for the elemental constituents with all above-MDC results is presented in Appendix E-14. Barium is the only elemental constituent with a non-significant sampling variance. There was no statistically significant heterogeneity among measurement variances across samples for any elemental constituent. All UCL95 determinations were based on a normal distribution for the mean.

The two elemental constituents with all below-MDC results, antimony and selenium, are summarized in Appendix E-15.

Only molybdenum had a mixture of above-MDC and below-MDC results, and it had a sufficient number of above-MDC results to determine a UCL95. Molybdenum did not appear to have sampling variation. The statistical summary for molybdenum is reported in Appendix E-16.

Table D-2. Elemental Constituents by Statistical Classification

Summarized by UCL95				Summarized by MDC	
All Results above-MDC			Results above-MDC & below-MDC	Results above-MDC & below-MDC	All Results below-MDC
Data in Appendix E-4			Appendix E-6		Appendix E-5
Statistical Summary in Appendix E-14			Appendix E-16		Appendix E-15
Supporting Results in Appendix E-17			Appendix E-17		Appendix E-17
Ag	Cr	Sr	Mo	none	Sb
Al	Cu	Th			Se
As	Fe	U			
B	Hg	Zn			
Ba	Mn	Zr			
Cd	Ni				
Co	Pb				

D.2.3 Analysis of Anions

Seven anions were characterized. Of those, five have all above-MDC results, and two have all below-MDC results. No anion has a mixture of above-MDC and below-MDC results. Table D-3 partitions the anions by these classes for Tank 12H.

The statistical summary for anions with all above-MDC results, chloride, nitrate, nitrite, sulfate, and total iodine, is presented in Appendix E-18. Nitrate and sulfate had statistically significant sampling variance; the others did not. There was no statistically significant heterogeneity among measurement variances across samples for any anion. All UCL95 determinations were based on a normal distribution for the mean.

Fluoride and phosphate are the only two anions with all below-MDC results, and they are summarized in Appendix E-19.

Table D-3. Anions by Statistical Classification

Summarized by UCL95			Summarized by MDC	
All Results above-MDC		Results above-MDC & below-MDC	Results above-MDC & below-MDC	All Results below-MDC
Data in Appendix E-7				Appendix E-8
Statistical Summary in Appendix E-18				Appendix E-19
Supporting Results in Appendix E-20				Appendix E-20
Chloride Cl^{-1}	Sulfate SO_4^{-2}	none	none	Fluoride
Nitrate NO_3^{-1}	Total Iodine			Phosphate
Nitrite NO_2^{-1}				

D.3 CONCLUSIONS AND RECOMMENDATIONS FOR THE STATISTICAL APPENDIX

Most of the analytes have all above-MDC results, and all of these analytes had a computed UCL95 based on the normal distribution. Only five analytes have a mixture of above-MDC and below-MDC results. Of these, only Cm-245 with two above-MDC results could not support determination of a UCL95. Cm-245 was summarized by MDC. Nine radionuclides, two elemental constituents, and two anions had no detectable concentrations (above-MDC results) in any sample. These were also summarized by MDC.

Only one analyte, Am-243, was observed to have a potential measurement outlier. The computations for the UCL95 for Am-243 were performed with and without the potential outlier, and the larger UCL95 result, based on omitting the potential outlier, is reported.

The software program JMP® Pro Software (64-bit) Version 11.2.1 from SAS Institute, Inc. was heavily used. The software applications JMP® Pro Software (64-bit) Version 11.2.1, ProUCL Version 5.0, SAS® Version 8.2, and Microsoft Excel® in Microsoft Office Professional Plus 2010 are covered by the SRNL statistics group software verification and validation program described by Baker, et al[D-7].

D.4 REFERENCES FOR APPENDIX D

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Appendix E: Statistical Analyses for Tank 12H Samples

Tables of Concentration Data for Tank 12H **Page**

Tables of Statistical Analyses for Tank 12H **Page**

Appendix E-1: Radionuclides with All Results above-MDC

Radionuclide Constituents (μCi/g)	Composite Sample 1			Composite Sample 2			Composite Sample 3		
	Run 1	Run 2	Run 3	Run 1	Run 2	Run 3	Run 1	Run 2	Run 3
Am-241	1.03E+1	1.04E+1	1.01E+1	1.84E+1	1.82E+1	1.86E+1	7.97E+0	9.32E+0	8.56E+0
Ba-137m	4.60E+0	5.45E+0	6.22E+0	9.25E+0	8.95E+0	1.04E+1	9.08E+0	8.82E+0	1.15E+1
Co-60	7.12E-2	6.85E-2	6.94E-2	1.06E-1	1.04E-1	1.13E-1	4.95E-2	6.13E-2	4.64E-2
Cs-137	4.86E+0	5.77E+0	6.58E+0	9.77E+0	9.46E+0	1.10E+1	9.59E+0	9.32E+0	1.21E+1
I-129	4.91E-3	4.91E-3	4.43E-3	5.99E-3	4.64E-3	4.45E-3	4.77E-3	3.61E-3	3.22E-3
Ni-59	1.81E-1	1.66E-1	1.76E-1	2.75E-1	2.73E-1	2.62E-1	1.35E-1	1.04E-1	1.42E-1
Ni-63	1.78E+1	1.83E+1	1.84E+1	2.73E+1	2.64E+1	2.64E+1	1.38E+1	1.37E+1	1.54E+1
Np-237	8.39E-3	8.46E-3	8.32E-3	2.14E-2	2.04E-2	2.19E-2	7.97E-3	8.18E-3	7.89E-3
Pu-238	6.94E+1	6.80E+1	6.49E+1	1.32E+2	1.21E+2	1.50E+2	6.44E+1	6.89E+1	6.62E+1
Pu-239	2.98E+0	2.92E+0	2.83E+0	5.68E+0	5.32E+0	6.31E+0	2.69E+0	2.69E+0	2.75E+0
Pu-240	1.05E+0	1.03E+0	9.77E-1	2.10E+0	1.96E+0	2.32E+0	9.64E-1	9.55E-1	9.95E-1
Pu-241	1.63E+1	1.43E+1	1.24E+1	3.16E+1	2.75E+1	3.70E+1	1.52E+1	1.58E+1	1.51E+1
Ra-228	3.78E-3	3.84E-3	3.43E-3	6.13E-3	6.98E-3	7.39E-3	3.50E-3	2.87E-3	4.37E-3
Sn-126	1.58E-2	1.81E-2	1.73E-2	1.55E-2	1.41E-2	1.41E-2	1.92E-2	2.17E-2	2.02E-2
Sr-90	1.71E+4	1.58E+4	1.81E+4	1.28E+4	1.10E+4	1.38E+4	1.60E+4	1.75E+4	1.57E+4
Tc-99	4.06E-3	3.27E-3	4.95E-3	4.91E-3	3.91E-3	4.39E-3	3.58E-3	3.69E-3	4.30E-3
Th-232	3.87E-3	4.27E-3	3.99E-3	8.53E-3	8.68E-3	9.63E-3	3.37E-3	3.67E-3	3.14E-3
U-232	3.36E-3	2.79E-3	2.76E-3	3.57E-3	4.86E-3	5.18E-3	2.23E-3	1.77E-3	3.93E-3
U-234	4.15E-3	4.53E-3	4.55E-3	7.81E-3	7.54E-3	7.90E-3	4.85E-3	5.37E-3	4.63E-3
U-235	1.73E-5	1.94E-5	1.77E-5	4.64E-5	4.38E-5	4.87E-5	2.32E-5	2.36E-5	2.09E-5
U-238	2.21E-4	2.38E-4	2.21E-4	7.83E-4	7.28E-4	8.20E-4	3.28E-4	3.35E-4	3.03E-4
Y-90	1.71E+4	1.58E+4	1.81E+4	1.28E+4	1.10E+4	1.38E+4	1.60E+4	1.75E+4	1.57E+4
Zr-93	3.74E-1	3.47E-1	3.78E-1	5.77E-1	6.04E-1	5.36E-1	4.05E-1	4.50E-1	4.37E-1

MDC: Minimum Detectable Concentration.

Appendix E-2: Radionuclides with All Results below-MDC

Radionuclide Constituents (μCi/g)	Composite Sample 1			Composite Sample 2			Composite Sample 3		
	Run 1	Run 2	Run 3	Run 1	Run 2	Run 3	Run 1	Run 2	Run 3
C-14	<5.86E-4	<5.45E-4	<5.05E-4	<7.79E-4	<7.03E-4	<6.98E-4	<8.83E-4	<5.90E-4	<6.40E-4
Cm-243	<1.53E-2	<1.59E-2	<1.46E-2	<1.61E-2	<1.26E-2	<1.86E-2	<1.22E-2	<1.88E-2	<1.62E-2
Cm-244	<4.18E-1	<4.55E-1	<4.00E-1	<6.17E-1	<6.98E-1	<6.71E-1	<3.34E-1	<4.91E-1	<3.58E-1
Cs-135	<1.00E-5	<2.89E-5	<5.90E-5	<6.89E-5	<4.46E-5	<5.36E-5	<5.45E-5	<8.06E-5	<7.75E-5
Nb-94	<9.41E-4	<1.05E-3	<8.74E-4	<7.88E-4	<9.82E-4	<1.17E-3	<1.11E-3	<1.23E-3	<4.36E-4
Ra-226	<7.75E-4	<4.68E-4	<7.25E-4	<6.62E-4	<6.35E-4	<6.31E-4	<5.00E-4	<5.05E-4	<5.81E-4
Th-229	<1.77E-3	<4.10E-3	<4.13E-4	<3.38E-3	<2.07E-3	<2.04E-4	No Data	<4.48E-4	<1.72E-3
Th-230	<3.52E-4	<5.68E-4	<4.15E-4	<5.59E-4	<1.62E-3	<4.68E-4	<4.59E-4	<1.08E-3	<1.57E-3
U-233	<3.39E-2	<3.89E-2	<3.40E-2	<7.52E-2	<7.77E-2	<8.53E-2	<3.24E-2	<3.52E-2	<3.23E-2

MDC: Minimum Detectable Concentration.

Appendix E-3: Radionuclides with a Mixture of Results above-MDC and below-MDC

Radionuclide Constituents (μCi/g)	Composite Sample 1			Composite Sample 2			Composite Sample 3		
	Run 1	Run 2	Run 3	Run 1	Run 2	Run 3	Run 1	Run 2	Run 3
Am-242m	4.27E-3	<3.89E-3	<2.24E-3	4.68E-3	4.95E-3	3.51E-3	2.62E-3	1.82E-3	1.31E-3
Am-243	1.79E-2	1.92E-2	1.51E-2	2.40E-2	2.29E-2	<4.49E-3	1.15E-2	1.73E-2	1.53E-2
Cm-245	<7.48E-5	<8.15E-5	<6.67E-5	1.16E-4	1.21E-4	<1.15E-4	<4.82E-5	<6.35E-5	<6.40E-5
Pa-231	<2.21E-3	<2.22E-3	1.55E-3	1.13E-3	2.49E-3	1.95E-3	<4.18E-3	<1.14E-3	<8.29E-4

MDC: Minimum Detectable Concentration.

Appendix E-4: Elemental Constituents with All Results above-MDC

Elemental Constituents (wt %)	Composite Sample 1			Composite Sample 2			Composite Sample 3		
	Run 1	Run 2	Run 3	Run 1	Run 2	Run 3	Run 1	Run 2	Run 3
Ag*	5.33E-2	5.54E-2	5.34E-2	8.98E-2	8.87E-2	9.24E-2	3.68E-2	3.83E-2	3.70E-2
Al	6.22E+0	5.29E+0	6.12E+0	1.02E+1	9.82E+0	8.44E+0	5.73E+0	5.69E+0	6.25E+0
As	2.04E-4	1.85E-4	1.93E-4	2.65E-4	2.50E-4	2.24E-4	1.95E-4	2.19E-4	2.12E-4
B	1.03E-1	1.10E-1	1.10E-1	6.43E-2	6.58E-2	7.01E-2	1.12E-1	1.22E-1	1.13E-1
Ba	6.33E-2	6.67E-2	6.80E-2	6.85E-2	7.37E-2	7.35E-2	6.70E-2	7.03E-2	6.85E-2
Cd	2.67E-3	2.84E-3	2.74E-3	1.86E-3	1.72E-3	1.91E-3	2.86E-3	3.02E-3	2.89E-3
Co	3.71E-3	3.99E-3	4.05E-3	5.56E-3	6.15E-3	6.07E-3	3.11E-3	3.16E-3	3.44E-3
Cr	5.72E-2	6.15E-2	5.82E-2	3.17E-2	3.23E-2	3.35E-2	5.71E-2	6.21E-2	5.68E-2
Cu	1.33E-1	1.39E-1	1.43E-1	1.75E-1	1.90E-1	1.88E-1	1.36E-1	1.42E-1	1.38E-1
Fe	3.22E+1	3.22E+1	3.27E+1	2.26E+1	2.14E+1	2.23E+1	3.48E+1	3.63E+1	3.61E+1
Hg	2.01E+1	1.94E+1	1.86E+1	1.18E+1	1.17E+1	1.16E+1	1.54E+1	1.68E+1	1.50E+1
Mn	1.17E+0	1.17E+0	1.19E+0	1.59E+0	1.61E+0	1.55E+0	1.33E+0	1.26E+0	1.40E+0
Ni	7.27E-1	7.22E-1	7.36E-1	1.06E+0	1.03E+0	1.01E+0	5.56E-1	5.34E-1	5.76E-1
Pb	3.46E-2	3.57E-2	3.64E-2	3.01E-2	3.05E-2	3.39E-2	3.55E-2	3.76E-2	3.53E-2
Sr	4.48E-2	4.74E-2	4.79E-2	3.65E-2	3.80E-2	3.92E-2	4.74E-2	5.13E-2	4.82E-2
Th	3.47E+0	3.36E+0	3.48E+0	8.18E+0	8.40E+0	7.95E+0	3.30E+0	3.07E+0	3.42E+0
U	4.11E-2	4.67E-2	4.40E-2	1.41E-1	1.60E-1	1.51E-1	6.96E-2	6.67E-2	6.97E-2
Zn	3.43E-2	3.66E-2	3.53E-2	3.19E-2	3.37E-2	3.45E-2	2.94E-2	3.09E-2	3.15E-2
Zr	9.87E-2	1.02E-1	1.08E-1	1.43E-1	1.52E-1	1.58E-1	1.01E-1	1.06E-1	9.91E-2

MDC: Minimum Detectable Concentrations.

* Ag *wt %* values were derived from ICMS measurements (mass 107 +109) and may be biased high by the presence of Pd-107 fission product.

Appendix E-5: Elemental Constituents with All Results below-MDC

Elemental Constituents (wt %)	Composite Sample 1			Composite Sample 2			Composite Sample 3		
	Run 1	Run 2	Run 3	Run 1	Run 2	Run 3	Run 1	Run 2	Run 3
Sb@	<2.00E-4	<1.93E-4	<1.95E-4	<1.92E-4	<1.99E-4	<1.96E-4	<1.97E-4	<1.92E-4	<1.96E-4
Se	<2.99E-4	<2.89E-4	<2.92E-4	<2.88E-4	<2.98E-4	<2.95E-4	<2.96E-4	<2.88E-4	<2.94E-4

MDC: Minimum Detectable Concentrations.

@ Sb *wt %* values were derived from ICMS measurements (mass 121 +123).

Appendix E-6: Elemental Constituents with a Mixture of Results above-MDC and below-MDC

Elemental Constituents (wt %)	Composite Sample 1			Composite Sample 2			Composite Sample 3		
	Run 1	Run 2	Run 3	Run 1	Run 2	Run 3	Run 1	Run 2	Run 3
Mo	1.52E-3	<1.33E-3	<1.34E-3	1.39E-3	<1.37E-3	1.65E-3	1.53E-3	<1.32E-3	1.41E-3

MDC: Minimum Detectable Concentrations.

Appendix E-7: Anions with All Results above-MDC

Anion Constituents (wt %)	Composite Sample 1			Composite Sample 2			Composite Sample 3		
	Run 1	Run 2	Run 3	Run 1	Run 2	Run 3	Run 1	Run 2	Run 3
Chloride Cl^{-1}	1.64E-2	1.72E-2	1.76E-2	1.75E-2	1.65E-2	1.53E-2	1.47E-2	1.49E-2	1.58E-2
Nitrate NO_3^{-1}	1.46E-2	1.59E-2	1.41E-2	1.51E-2	1.84E-2	1.47E-2	1.34E-2	1.25E-2	1.29E-2
Nitrite NO_2^{-1}	4.63E-3	4.29E-3	4.17E-3	8.95E-3	9.19E-3	8.40E-3	3.91E-3	4.52E-3	3.99E-3
Sulfate SO_4^{-2}	5.30E-2	4.96E-2	4.88E-2	8.47E-2	9.19E-2	8.22E-2	9.78E-2	1.13E-1	8.79E-2
Total Iodine	2.90E-3	2.92E-3	2.62E-3	3.64E-3	2.85E-3	2.72E-3	2.79E-3	2.14E-3	1.90E-3

MDC: Minimum Detectable Concentrations.

Appendix E-8: Anions with All Results below-MDC

Anion Constituents (wt %)	Composite Sample 1			Composite Sample 2			Composite Sample 3		
	Run 1	Run 2	Run 3	Run 1	Run 2	Run 3	Run 1	Run 2	Run 3
Fluoride F^{-1}	<3.04E-3	<3.06E-3	<2.94E-3	<3.02E-3	<3.06E-3	<2.94E-3	<3.06E-3	<2.97E-3	<2.93E-3
Phosphate PO_4^{-3}	<3.04E-3	<3.06E-3	<2.94E-3	<3.02E-3	<3.06E-3	<2.94E-3	<3.06E-3	<2.97E-3	<2.93E-3

MDC: Minimum Detectable Concentrations.

Appendix E-9: Statistical Summary for the Radionuclides with all Results above-MDC

Constituent	N	Mean (μCi/g)	Std Dev (μCi/g)	% Std Dev	UCL95 (μCi/g)	Remarks
Am-241	9	1.2428E+1	5.2485E+0	42.23%	2.1257E+1	SNS-VH; SS-SV; Student's t Confidence Limit (2 DF)
Ba-137m	9	8.2522E+0	2.6041E+0	31.56%	1.2388E+1	SNS-VH; SS-SV; Student's t Confidence Limit (2 DF)
Co-60	9	7.6589E-2	2.8606E-2	37.35%	1.2425E-1	SNS-VH; SS-SV; Student's t Confidence Limit (2 DF)
Cs-137	9	8.7167E+0	2.7407E+0	31.44%	1.3073E+1	SNS-VH; SS-SV; Student's t Confidence Limit (2 DF)
I-129	9	4.5478E-3	7.9578E-4	17.50%	5.0410E-3	SNS-VH; SNS-WS; SNS-DT; SNS-SV; Student's t Confidence Limit (8 DF)
Ni-59	9	1.9044E-1	7.3632E-2	38.66%	3.1326E-1	SNS-VH; SS-SV; Student's t Confidence Limit (2 DF)
Ni-63	9	1.9722E+1	6.3671E+0	32.28%	3.0418E+1	SNS-VH; SS-SV; Student's t Confidence Limit (2 DF)
Np-237	9	1.2546E-2	7.5352E-3	60.06%	2.5234E-2	SNS-VH; SS-SV; Student's t Confidence Limit (2 DF)
Pu-238	9	8.9422E+1	3.9534E+1	44.21%	1.5500E+2	SNS-VH; SS-SV; Student's t Confidence Limit (2 DF)
Pu-239	9	3.7967E+0	1.7285E+0	45.53%	6.6826E+0	SNS-VH; SS-SV; Student's t Confidence Limit (2 DF)
Pu-240	9	1.3723E+0	6.5959E-1	48.06%	2.4744E+0	SNS-VH; SS-SV; Student's t Confidence Limit (2 DF)
Pu-241	9	2.0578E+1	1.0228E+1	49.70%	3.7325E+1	SNS-VH; SS-SV; Student's t Confidence Limit (2 DF)
Ra-228	9	4.6989E-3	1.9101E-3	40.65%	7.8164E-3	SNS-VH; SS-SV; Student's t Confidence Limit (2 DF)
Sn-126	9	1.7333E-2	3.0436E-3	17.56%	2.2238E-2	SNS-VH; SS-SV; Student's t Confidence Limit (2 DF)
Sr-90	9	1.5311E+4	2.6128E+3	17.06%	1.9398E+4	SNS-VH; SS-SV; Student's t Confidence Limit (2 DF)
Tc-99	9	4.1178E-3	5.7717E-4	14.02%	4.4755E-3	SNS-VH; SNS-WS; SNS-DT; SNS-SV; Student's t Confidence Limit (8 DF)
Th-232	9	5.4611E-3	3.0531E-3	55.91%	1.0579E-2	SNS-VH; SS-SV; Student's t Confidence Limit (2 DF)
U-232	9	3.3833E-3	1.1411E-3	33.73%	4.0906E-3	SNS-VH; SNS-WS; SNS-DT; SNS-SV; Student's t Confidence Limit (8 DF)
U-234	9	5.7033E-3	1.8071E-3	31.69%	8.7259E-3	SNS-VH; SS-SV; Student's t Confidence Limit (2 DF)
U-235	9	2.9000E-5	1.5214E-5	52.46%	5.4533E-5	SNS-VH; SS-SV; Student's t Confidence Limit (2 DF)
U-238	9	4.4189E-4	2.9505E-4	66.77%	9.3770E-4	SNS-VH; SS-SV; Student's t Confidence Limit (2 DF)
Y-90	9	1.5311E+4	2.6128E+3	17.06%	1.9398E+4	SNS-VH; SS-SV; Student's t Confidence Limit (2 DF)
Zr-93	9	4.5644E-1	1.0747E-1	23.55%	6.3412E-1	SNS-VH; SS-SV; Student's t Confidence Limit (2 DF)

MDC: Minimum Detectable Concentration.
DF: degrees of freedom.
SS-VH (SNS-VH): Statistically significant (statistically non-significant) test for measurement error heterogeneity at $\alpha = 0.05/23=0.002174$.
SS-SV (SNS-SV): Statistically significant (statistically non-significant) sampling variance at $\alpha = 0.05$.
SS-WS (SNS-WS): Statistically significant (statistically non-significant) Wilk-Shapiro test for normality at $\alpha = 0.05$ (used for models without a sampling variance).
SS-DT (SNS-DT): Statistically significant (statistically non-significant) Dixon test for an outlier at $\alpha = 0.05$ (used for models without a sampling variance).

Appendix E-10: Statistical Summary for the Radionuclides with all Results below-MDC

Radionuclide Constituent (μCi/g)	N	Smallest Minimum Detectable Concentration (μCi/g)		Largest Minimum Detectable Concentration (μCi/g)	
		Fixed Decimal Format	Scientific Format	Fixed Decimal Format	Scientific Format
C-14	9	0.000505	5.05E-4	0.000883	8.83E-4
Cm-243	9	0.0122	1.22E-2	0.0188	1.88E-2
Cm-244	9	0.334	3.34E-1	0.698	6.98E-1
Cs-135	9	0.00001	1.00E-5	0.0000806	8.06E-5
Nb-94	9	0.000436	4.36E-4	0.00123	1.23E-3
Ra-226	9	0.000468	4.68E-4	0.000775	7.75E-4
Th-229[♣]	8	0.000204	2.04E-4	0.0041	4.10E-3
Th-230	9	0.000352	3.52E-4	0.00162	1.62E-3
U-233	9	0.0323	3.23E-2	0.0853	8.53E-2

MDC: Minimum Detectable Concentration.

[♣]Th-229 had 1 missing result for Run 1 of Composite Sample 3.

Appendix E-11: Statistical Summary of the UCL95 for Radionuclides with a Mixture of Results above-MDC and below-MDC

Constituent (μCi/g)	N◆	Mean (μCi/g)	Std Dev (μCi/g)	% Std Dev	UCL95 (μCi/g)	Remarks
Am-242m	9 (1,3,3)	3.0818E-3	1.3970E-3	45.33%	5.1673E-3	Student's t Confidence Limit (2 DF)
Am-243♠	9 (3,2,3)	1.8443E-2	4.7822E-3	25.93%	2.6046E-2	Student's t Confidence Limit (1.947 DF)
Pa-231	9 (1,3,0)	1.2940E-3	6.7826E-4	52.42%	2.4375E-3	Student's t Confidence Limit (2 DF)

These analytes had sufficient information to estimate the composite sample means and determine a UCL95.
MDC: Minimum Detectable Concentration.
DF: degrees of freedom.
◆ N = 9 analytical results with the number of results (measurements) above the respective MDC listed inside the parentheses for each composite sample: ♠ The Am-243 results were analyzed with and without a potential outlier. The results given here are without the potential outlier. The details are given in Appendix-E13.

Appendix E-12: Statistical Summary of the MDC for Radionuclides with a Mixture of Results above-MDC and below-MDC

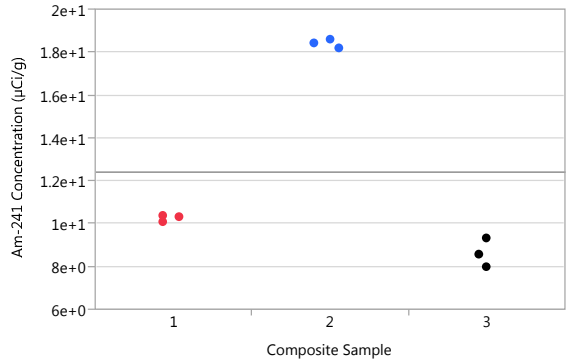
Constituent (μCi/g)	N◆	Smallest Minimum Detectable Concentration (μCi/g)		Largest Minimum Detectable Concentration (μCi/g)	
		Fixed Decimal Format	Scientific Format	Fixed Decimal Format	Scientific Format
Cm-245	9 (0,2,0)	0.0000482	4.82E-5	0.000144	1.44E-4

This analyte did not have sufficient data to estimate the means of Composite Samples 1 and 3, and a UCL95 could not be determined. Refer to Appendix-E13 for additional details for the statistical analysis for Cm-245.
MDC: Minimum Detectable Concentration.
◆ N = 9 analytical results with the number of results (measurements) above the respective MDC listed inside the parentheses for each composite sample: (# above-MDC for Composite Sample 1, # above-MDC for Composite Sample 2, # above-MDC for Composite Sample 3).

Appendix E-13: Supporting Results for Radionuclides

Am-241 (μCi/g)

All Results (circles) above MDC



Means and Standard Deviations by Sample

Sample	Number	Mean	Std Dev	Std Err Mean
1	3	1.0267E+1	1.5275E-1	8.8192E-2
2	3	1.8400E+1	2.0000E-1	1.1547E-1
3	3	8.6167E+0	6.7678E-1	3.9074E-1
Overall	9	1.2428E+1	4.5501E+0	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	1.5266	2	6	0.2911
Brown-Forsythe	1.8409	2	6	0.2380
Levene	2.4703	2	6	0.1649
Bartlett	2.0519	2		0.1285

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance (ANOVA)

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	1.6459E+2	8.2294E+1	473.53	<.0001
Error	6	1.0427E+0	1.7379E-1		
C. Total	8	1.6563E+2			

Statistically significant sampling variance.

Variance Components

Component	Variance Comp	Std Dev
Composite Samples	2.7373E+1	5.2319E+0
Measurements	1.7379E-1	4.1688E-1
Total	2.7547E+1	5.2485E+0
Mean Concentration	9.1437E+0	3.0239E+0

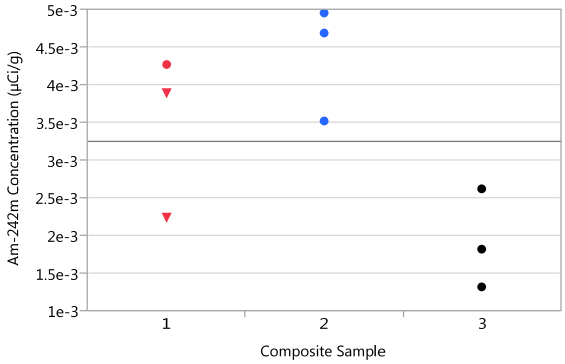
REML mean is 1.2428E+1.

Student's t UCL95

UCL95	2.1257E+1

Am-242m (μCi/g)

Results (circles) above and (triangles) below MDC



There were 7 measurements and 2 below-MDC results: the below-MDC results were both reported for Composite Sample 1.

Means and Standard Deviations by Sample

Sample	Number	Mean	Std Dev	Std Err Mean
2	3	4.3800E-3	7.6544E-4	4.4193E-4
3	3	1.9167E-3	6.6032E-4	3.8124E-4

Results from a fixed effect ANOVA in SAS® Proc Reliability, treating each below-MDC result as interval censored between 0 and its MDC. The effects Csamp i, i = 1, 2, 3, are deviations of the mean of Composite Sample i from the mean of Composite Sample 3.

Am-242m	Estimate	Std Error	Csamp Means
Intercept	1.9167E-03	4.5912E-04	
Csamp 1	1.0312E-03	7.2725E-04	2.9488E-03
Csamp 2	2.4633E-03	6.4929E-04	4.3800E-03
Csamp 3	0.0000	0.0000	1.9167E-03
Std Dev	7.9521E-04	2.1752E-04	

The 3 composite sample means under the header "Csamp Mean" in the above table were treated as a random sample of 3 results, and a Student's t UCL95 was computed with 2df. The standard deviation of a composite sample measured once was computed by the following formula.

$$Std.Dev. = \sqrt{Var_{Comp.SampleMeans} - Var_{MeasError}/3 + Var_{MeasError}}$$

$$= \sqrt{(1.2370E-3)^2 - (7.9521E-4)^2/3 + (7.9521E-4)^2}$$

$$= \sqrt{1.9517E-6} = 1.3970E-3,$$ where $Var_{Comp.SampleMeans}$ is the variance of the 3 composite sample means under the heading "Csamp Mean", and $Var_{MeasError}$ is the square of the measurement error standard deviation in the above table. The summary follows.

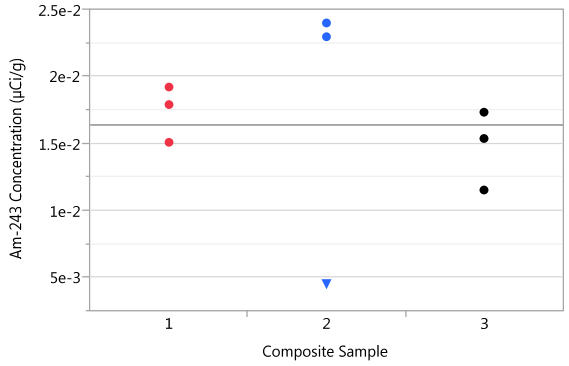
Mean	3.0818E-3
Standard Deviation	1.3970E-3
UCL95	5.1673E-3

In the "ANOVA" table, DF: degrees of freedom; SS: Sums of Squares; MS: Mean Squares. In the "Tests that the Measurement Variances are Equal" table, DFNum: Numerator DF; DFDen: Denominator DF for the F Ratio.

Appendix E-13: Supporting Results for Radionuclides

Am-243 (μCi/g)

Results (circles) above and (triangles) below MDC



There were 8 measurements. One below-MDC result was reported on Run 3 of Composite Sample 2.

Means and Standard Deviations by Sample

Sample	Number	Mean	Std Dev	Std Err Mean
1	3	1.7400E-2	2.0952E-3	1.2097E-3
3	3	1.4700E-2	2.9462E-3	1.7010E-3

Results from a fixed effect ANOVA in SAS® Proc Reliability, treating the below-MDC result as interval censored between 0 and the MDC, are given in the following table. The intercept term is the mean for Composite Sample 3.

Am-243	Estimate	Std Error	Csamp Means
Intercept	1.4700E-02	3.5088E-03	
Csamp 1	2.7000E-03	4.9622E-03	1.7400E-02
Csamp 2	1.6631E-03	5.0234E-03	1.6363E-02
Csamp 3	0	0	1.4700E-02
Std Dev	6.0774E-03	1.5859E-03	

The magnitudes of the standard errors for the fixed effects "Csamp1" and "Csamp 2", the deviations of the means of Composite Samples 1 and 2, respectively, from the mean of Composite Sample 3, are greater than their estimates, so the model was reduced to one with only an overall mean, and the results from an ANOVA in SAS® Proc Reliability, treating the below-MDC result as interval censored between 0 and the MDC, are as follows. The intercept term is the overall mean.

Am-243	Estimate	Std Error
Intercept	1.6229E-02	1.9995E-03
Std Dev	5.9847E-03	1.4449E-03

Am-243 (μCi/g)

Continued

A Student's t UCL95 (conservative at 7 DF) for the mean concentration is as follows.

Mean	1.6229E-2
Standard Deviation	5.9847E-3
UCL95	2.0017E-2

The below-MDC result (depicted by a triangle symbol on the plot to the left) was investigated for being an outlier using Cook's D statistic. The computations were performed in JMP® version 11. The below-MDC result was treated as though it was a measurement for purposes of calculating Cook's D statistic (only), and a fixed effects ANOVA model was fit with a composite sample effect. Cook's D for the potential outlier for Run 3 on Composite Sample 2 was 0.90. The magnitude of this result was sufficiently large (near to 1) to consider an alternative analysis without the result for Run 3 of Composite Sample 2.

The alternative analysis omits the result for Run 3 of Composite Sample 2. The remaining 8 results are all measurements. The F test for sampling variance under a random effect model has a P-value of 0.0235 (statistically significant at $\alpha = 0.05$).

Analysis of Variance (ANOVA)

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	9.3075E-5	4.6538E-5	8.7002	0.0235
Error	5	2.6745E-5	5.3490E-6		
C. Total	7	1.1982E-4			

Statistically significant sampling variance.

The tables for the variance components and the UCL95 for the alternative analysis without the potential outlier are given below. The alternative analysis UCL95 result is larger than the original UCL95 result in the table at the top of this column. The alternative UCL95 result, being larger, will be used.

Variance Components

Component	Variance Comp	Std Dev
Composite Samples	1.7493E-5	4.1825E-3
Measurements	5.3760E-6	2.3186E-3
Total	2.2869E-5	4.7822E-3
Mean Concentration	6.5334E-6	2.5560E-3

REML mean is 1.8443E-2.

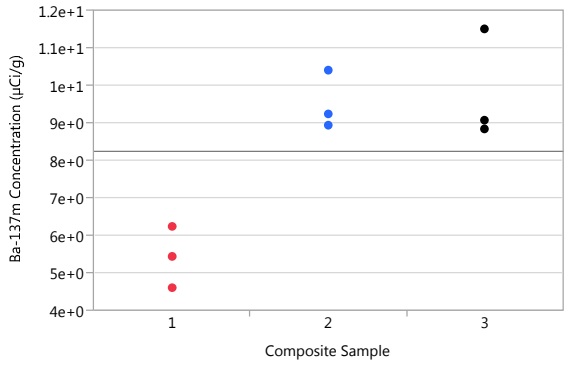
Student's t UCL95

UCL95	2.6046E-2

Appendix E-13: Supporting Results for Radionuclides

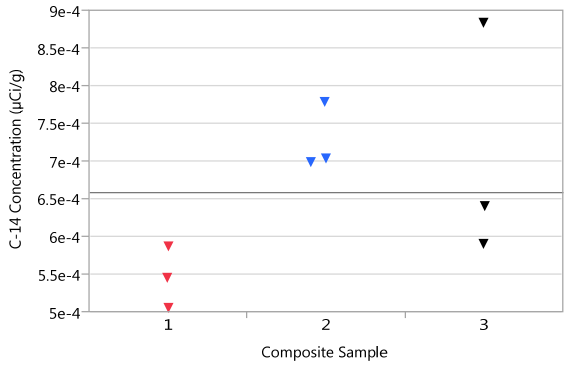
Ba-137m (μCi/g)

All Results (circles) above MDC



C-14 (μCi/g)

All Results (triangles) below MDC



Means and Standard Deviations

Sample	Number	Mean	Std Dev	Std Err Mean
1	3	5.4233	8.1033E-1	4.6784E-1
2	3	9.5333	7.6540E-1	4.4190E-1
3	3	9.8000	1.4780E+0	8.5331E-1
Overall	9	8.2522	2.3177E+0	

Minimum Detectable Concentration (MDC)

Sample	Number	Minimum MDC	Maximum MDC
1	3	5.05E-4	5.86E-4
2	3	6.98E-4	7.79E-4
3	3	5.90E-4	8.83E-4
Overall	9	5.05E-4	8.83E-4

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	0.7845	2	6	0.4981
Brown-Forsythe	0.1898	2	6	0.8319
Levene	1.7836	2	6	0.2467
Bartlett	0.4689	2		0.6257

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance (ANOVA)

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	3.6118E+1	1.8059E+1	15.81	0.0041
Error	6	6.8537E+0	1.1423E+0		
C. Total	8	4.2972E+1			

Statistically significant sampling variance.

Variance Components

Component	Variance Comp	Std Dev
Composite Samples	5.6390	2.3747
Measurements	1.1423	1.0688
Total	6.7813	2.6041
Mean Concentration	2.0066	1.4165

REML mean is 8.2522.

Student's t UCL95

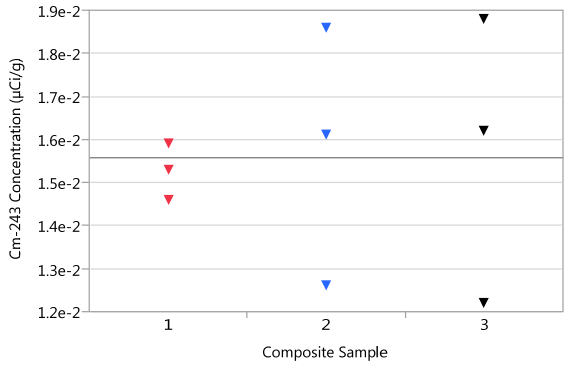
UCL95	1.2388E+1

In the "ANOVA" table, DF: degrees of freedom; SS: Sums of Squares; MS: Mean Squares. In the "Tests that the Measurement Variances are Equal" table, DFNum: Numerator DF; DFDen: Denominator DF for the F Ratio.

Appendix E-13: Supporting Results for Radionuclides

Cm-243 (μCi/g)

All Results (triangles) below MDC

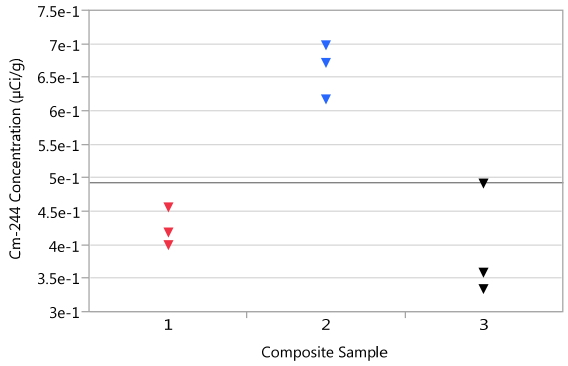


Minimum Detectable Concentration (MDC)

Sample	Number	Minimum MDC	Maximum MDC
1	3	1.46E-2	1.59E-2
2	3	1.26E-2	1.86E-2
3	3	1.22E-2	1.88E-2
Overall	9	1.22E-2	1.88E-2

Cm-244 (μCi/g)

All Results (triangles) below MDC



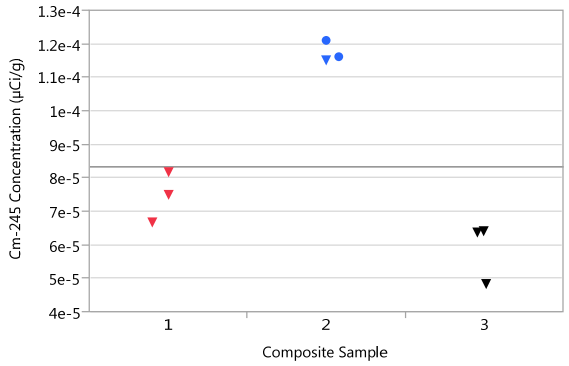
Minimum Detectable Concentration (MDC)

Sample	Number	Minimum MDC	Maximum MDC
1	3	4.00E-1	4.55E-1
2	3	6.17E-1	6.98E-1
3	3	3.34E-1	4.91E-1
Overall	9	3.34E-1	6.98E-1

Appendix E-13: Supporting Results for Radionuclides

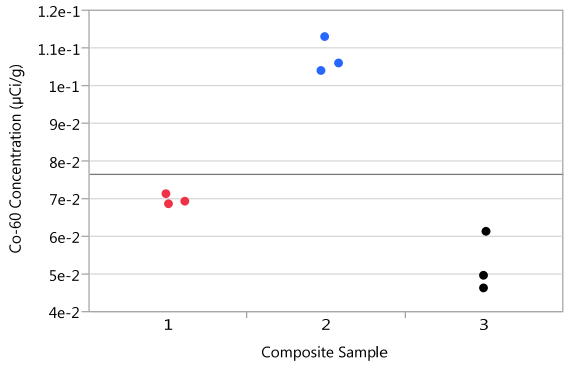
Cm-245 (μCi/g)

Results (circles) above and (triangles) below MDC



Co-60 (μCi/g)

All Results (circles) above MDC



There were 2 above-MDC results (measurements) and 7 below-MDC results for Cm-245 concentration. Both measurements were reported for the Composite Sample 2, so there is no measure of sampling variation. Consequently, a UCL95 cannot be computed. The 3 reported results for Composite Sample 2 were fit to a model with only an intercept term (the mean of Composite Sample 2). The results are reproduced in the following table.

Cm-245	Estimate	Std Error
Intercept	1.1653E-04	2.2684E-06
Scale	3.6240E-06	1.9525E-06

A separate Student's t UCL95 was constructed for Runs 1 and 2 of Composite Sample 2 using the formula $x+t_{0.95,1df}\cdot s$, where x is one of the measurements, and $s=3.6240\text{E}{-6}$ is the measurement standard deviation. The UCL95 for Run 1 of Composite Sample 2 is 1.3888E-4, and the UCL95 for Run 2 of Composite Sample 2 is 1.4388E-4. These 2 UCL95's replace their respective measurements and will be treated as MDC values. The minimum and maximum MDC values characterize the results for Cm-245 concentration.

Minimum Detectable Concentration (MDC)

Sample	Number	Minimum MDC	Maximum MDC
1	3	6.67E-5	8.15E-5
2	3	1.15E-4	1.44E-4
3	3	4.82E-5	6.40E-5
Overall	9	4.82E-5	1.44E-4

Means and Standard Deviations

Sample	Number	Mean	Std Dev	Std Err Mean
1	3	6.9700E-2	1.3748E-3	7.9373E-4
2	3	1.0767E-1	4.7258E-3	2.7285E-3
3	3	5.2400E-2	7.8619E-3	4.5391E-3
Overall	9	7.6589E-2	2.4918E-2	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	1.1447	2	6	0.3792
Brown-Forsythe	0.7265	2	6	0.5217
Levene	4.2921	2	6	0.0696
Bartlett	1.8013	2		0.1651

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance (ANOVA)

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	4.7952E-3	2.3976E-3	83.60	<.0001
Error	6	1.7207E-4	2.8678E-5		
C. Total	8	4.9672E-3			

Statistically significant sampling variance.

Variance Components

Component	Variance Comp	Std Dev
Composite Samples	7.8963E-4	2.8100E-2
Measurements	2.8678E-5	5.3552E-3
Total	8.1831E-4	2.8606E-2
Mean Concentration	2.6640E-4	1.6321E-2

REML Mean = 7.6589E-2.

Student's t UCL95

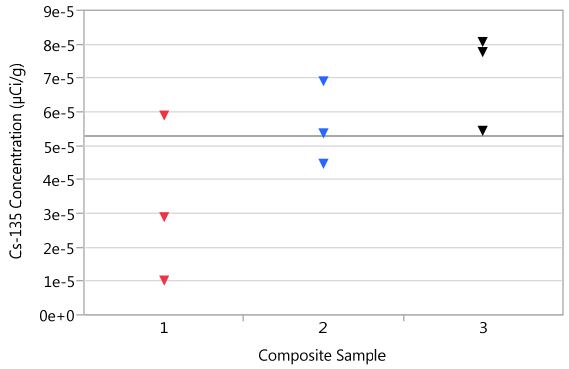
UCL95	1.2425E-1

In the "ANOVA" table, DF: degrees of freedom; SS: Sums of Squares; MS: Mean Squares. In the "Tests that the Measurement Variances are Equal" table, DFNum: Numerator DF; DFDen: Denominator DF for the F Ratio.

Appendix E-13: Supporting Results for Radionuclides

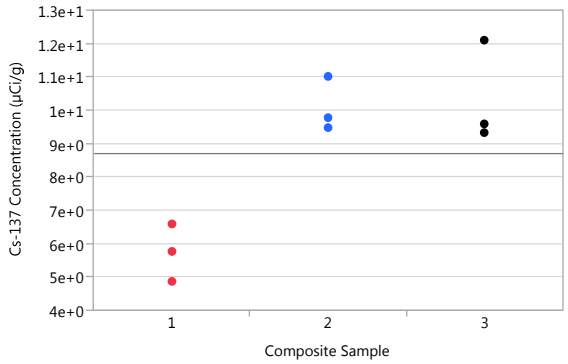
Cs-135 (μCi/g)

All Results (triangles) below MDC



Cs-137 (μCi/g)

All Results (circles) above MDC



Minimum Detectable Concentration (MDC)

Sample	Number	Minimum MDC	Maximum MDC
1	3	1.00E-5	5.90E-5
2	3	4.46E-5	6.89E-5
3	3	5.45E-5	8.06E-5
Overall	9	1.00E-5	8.06E-5

Means and Standard Deviations

Sample	Number	Mean	Std Dev	Std Err Mean
1	3	5.7367E+0	8.6048E-1	4.9680E-1
2	3	1.0077E+1	8.1451E-1	4.7026E-1
3	3	1.0337E+1	1.5330E+0	8.8511E-1
Overall	9	8.7167E+0	2.4385E+0	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	0.7429	2	6	0.5149
Brown-Forsythe	0.1756	2	6	0.8431
Levene	1.6586	2	6	0.2670
Bartlett	0.4329	2		0.6487

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance (ANOVA)

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	4.0063E+1	2.0032E+1	16.01	0.0039
Error	6	7.5082E+0	1.2514E+0		
C. Total	8	4.7571E+1			

Statistically significant sampling variance.

Variance Components

Component	Variance Comp	Std Dev
Composite Samples	6.2601	2.5020
Measurements	1.2514	1.1186
Total	7.5114	2.7407
Mean Concentration	2.2257	1.4919

REML Mean = 8.7167.

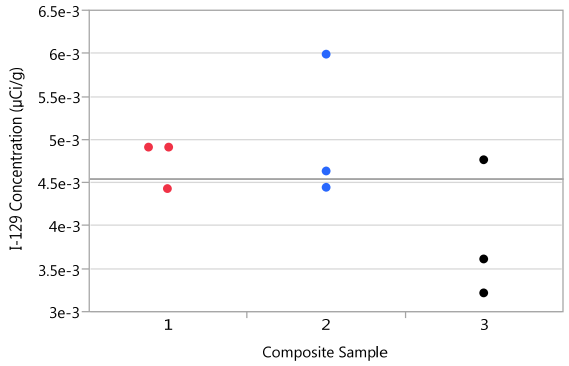
Student's t UCL95

UCL95	1.3073E+1

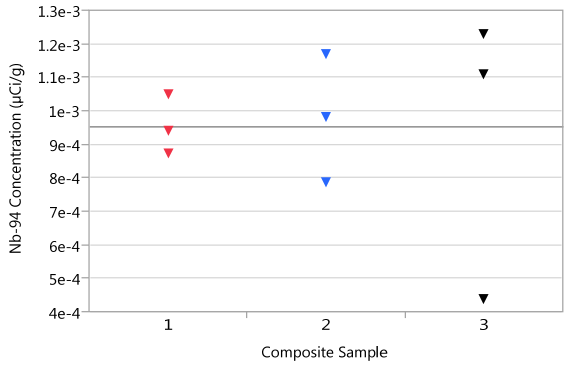
In the "ANOVA" table, DF: degrees of freedom; SS: Sums of Squares; MS: Mean Squares. In the "Tests that the Measurement Variances are Equal" table, DFNum: Numerator DF; DFDen: Denominator DF for the F Ratio.

Appendix E-13: Supporting Results for Radionuclides

I-129 (μCi/g)
All Results (circles) above MDC



Nb-94 (μCi/g)
All Results (triangles) below MDC



Means and Standard Deviations

Sample	Number	Mean	Std Dev	Std Err Mean
1	3	4.7500E-3	2.7713E-4	1.6000E-4
2	3	5.0267E-3	8.3966E-4	4.8478E-4
3	3	3.8667E-3	8.0625E-4	4.6549E-4
Overall	9	4.5478E-3	7.9578E-4	

Minimum Detectable Concentration (MDC)

Sample	Number	Minimum MDC	Maximum MDC
1	3	8.74E-4	1.05E-3
2	3	7.88E-4	1.17E-3
3	3	4.36E-4	1.23E-3
Overall	9	4.36E-4	1.23E-3

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	0.6976	2	6	0.5341
Brown-Forsythe	0.3942	2	6	0.6905
Levene	2.5143	2	6	0.1610
Bartlett	0.9228	2		0.3974

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance (ANOVA)

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	2.2024E-6	1.1012E-6	2.31	0.1806
Error	6	2.8637E-6	4.7729E-7		
C. Total	8	5.0661E-6			

Statistically non-significant sampling variance.

Diagnostics for I-129 Measurements

Shapiro-Wilk	P-Value=0.5416>α=0.05 ->SNS
Dixon High Outlier	Run 1 of Sample 2 -> SNS
Dixon Low Outlier	Run 3 of Sample 3 -> SNS

Student's t UCL95

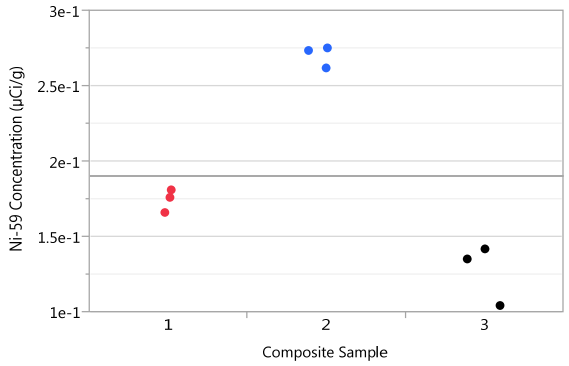
UCL95	5.0410E-3

In the "ANOVA" table, DF: degrees of freedom; SS: Sums of Squares; MS: Mean Squares. In the "Tests that the Measurement Variances are Equal" table, DFNum: Numerator DF; DFDen: Denominator DF for the F Ratio.
SNS: Statistically non-significant at $\alpha = 0.05$.

Appendix E-13: Supporting Results for Radionuclides

Ni-59 (μCi/g)

All Results (circles) above MDC



Means and Standard Deviations

Sample	Number	Mean	Std Dev	Std Err Mean
1	3	1.7433E-1	7.6376E-3	4.4096E-3
2	3	2.7000E-1	7.0000E-3	4.0415E-3
3	3	1.2700E-1	2.0224E-2	1.1676E-2
Overall	9	1.9044E-1	6.4104E-2	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	1.2975	2	6	0.3402
Brown-Forsythe	0.5963	2	6	0.5805
Levene	3.9286	2	6	0.0812
Bartlett	1.2049	2		0.2997

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance (ANOVA)

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	3.1842E-2	1.5921E-2	92.50	<.0001
Error	6	1.0327E-3	1.7211E-4		
C. Total	8	3.2874E-2			

Statistically significant sampling variance.

Variance Components

Component	Variance Comp	Std Dev
Composite Samples	5.2496E-3	7.2454E-2
Measurements	1.7211E-4	1.3119E-2
Total	5.4217E-3	7.3632E-2
Mean Concentration	1.7690E-3	4.2059E-2

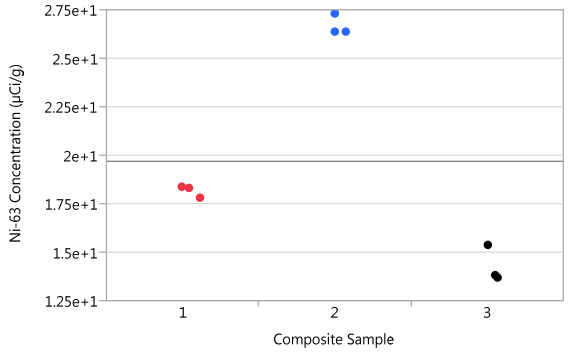
REML Mean = 1.9044E-1.

Student's t UCL95

UCL95	3.1326E-1

Ni-63 (μCi/g)

All Results (circles) above MDC



Means and Standard Deviations

Sample	Number	Mean	Std Dev	Std Err Mean
1	3	1.8167E+1	3.2146E-1	1.8559E-1
2	3	2.6700E+1	5.1962E-1	3.0000E-1
3	3	1.4300E+1	9.5394E-1	5.5076E-1
Overall	9	1.9722E+1	5.5238E+0	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	1.0609	2	6	0.4032
Brown-Forsythe	0.2828	2	6	0.7632
Levene	3.8186	2	6	0.0852
Bartlett	0.9213	2		0.3980

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance (ANOVA)

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	2.4153E+2	2.4153E+2	282.31	<.0001
Error	6	2.5667E+0	2.5667E+0		
C. Total	8	2.4410E+2			

Statistically significant sampling variance.

Variance Components

Component	Variance Comp	Std Dev
Composite Samples	4.0112E+1	6.3334E+0
Measurements	4.2778E-1	6.5405E-1
Total	4.0540E+1	6.3671E+0
Mean Concentration	1.3418E+1	3.6631E+0

REML Mean = 1.9722E+1.

Student's t UCL95

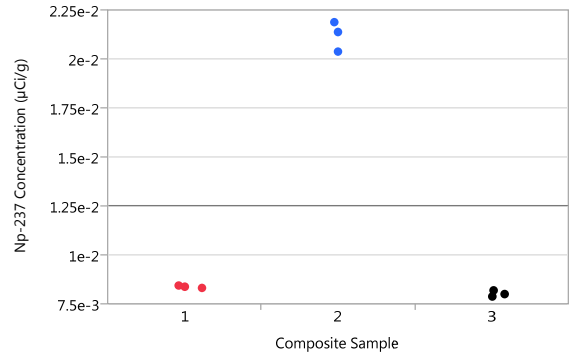
UCL95	3.0418E+1

In the "ANOVA" table, DF: degrees of freedom; SS: Sums of Squares; MS: Mean Squares. In the "Tests that the Measurement Variances are Equal" table, DFNum: Numerator DF; DFDen: Denominator DF for the F Ratio.

Appendix E-13: Supporting Results for Radionuclides

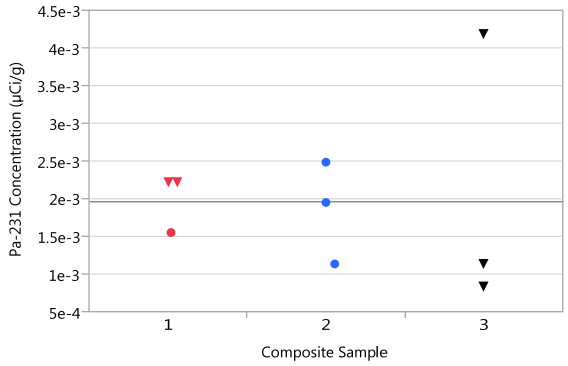
Np-237 (μCi/g)

All Results (circles) above MDC



Pa-231 (μCi/g)

Results (circles) above and (triangles) below MDC



Means and Standard Deviations

Sample	Number	Mean	Std Dev	Std Err Mean
1	3	8.3900E-3	7.0000E-5	4.0415E-5
2	3	2.1233E-2	7.6376E-4	4.4096E-4
3	3	8.0133E-3	1.4978E-4	8.6474E-5
Overall	9	1.2546E-2	6.5296E-3	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	1.6940	2	6	0.2611
Brown-Forsythe	2.1153	2	6	0.2017
Levene	5.4875	2	6	0.0442
Bartlett	3.9921	2		0.0185

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance (ANOVA)

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	3.3986E-4	1.6993E-4	834.81	<.0001
Error	6	1.2213E-6	2.0356E-7		
C. Total	8	3.4108E-4			

Statistically significant sampling variance.

Variance Components

Component	Variance Comp	Std Dev
Composite Samples	5.6576E-5	7.5217E-3
Measurements	2.0356E-7	4.5117E-4
Total	5.6779E-5	7.5352E-3
Mean Concentration	1.8881E-5	4.3452E-3

REML Mean = 1.2546E-2.

Student's t UCL95

UCL95	2.5234E-2

Minimum Detectable Concentration (MDC)

There are 4 measurements and 5 results below the MDC: 2 of the 3 results on Composite Sample 1 and all 3 results on Composite Sample 3 are below the MDC. Composite Sample 2 had 3 measurements.

Mean and Standard Deviation for Composite Sample 2

Sample	Number	Mean	Std Dev	Std Err Mean
2	3	1.8567E-3	6.8479E-4	3.9536E-4

Results from a fixed effect ANOVA in SAS® Proc Reliability, treating the below-MDC results as interval censored between 0 and the MDC.

Pa-231	Estimate	Std Error	Csamp Means
Intercept	5.4073E-4	3.3624E-4	
Csamp 1	9.4285E-4	4.9674E-4	1.4835E-3
Csamp 2	1.3159E-3	4.1012E-4	1.8566E-3
Csamp 3	0.	0.	5.4070E-4
Scale	4.0673E-4	1.1228E-4	

The 3 composite sample mean estimates in the above table were treated as a random sample of 3 mean results, and a Student's t UCL95 was computed with 2df. The standard deviation was computed by the following formula.

$$Std.Dev. = \sqrt{Var_{Comp.SampleMeans} - Var_{MeasError}/3 + Var_{MeasError}}$$

$$= \sqrt{(6.7820E-4)^2 - (4.0673E-4)^2/3 + (4.0673E-4)^2}$$

$$= \sqrt{5.7024E-7} = 7.5514E-4$$

The summary follows.

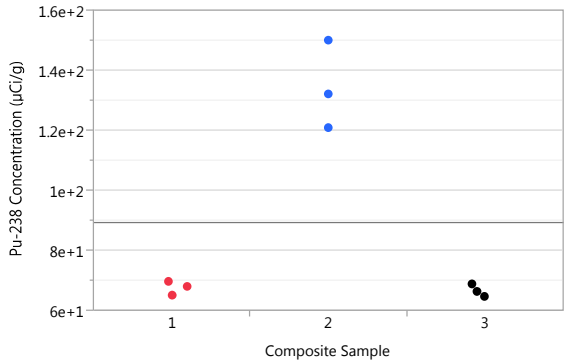
Mean	1.2936E-3
Standard Deviation	7.5514E-4
UCL95	2.4369E-3

In the "ANOVA" table, DF: degrees of freedom; SS: Sums of Squares; MS: Mean Squares. In the "Tests that the Measurement Variances are Equal" table, DFNum: Numerator DF; DFDen: Denominator DF for the F Ratio. DFNum: Numerator degrees of freedom; DFDen: Denominator degrees of freedom for the F Ratio.

Appendix E-13: Supporting Results for Radionuclides

Pu-238 (μCi/g)

All Results (circles) above MDC



Means and Standard Deviations

Sample	Number	Mean	Std Dev	Std Err Mean
1	3	6.7433E+1	2.3029E+0	1.3296E+0
2	3	1.3433E+2	1.4640E+1	8.4525E+0
3	3	6.6500E+1	2.2650E+0	1.3077E+0
Overall	9	8.9422E+1	3.4510E+1	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	1.6903	2	6	0.2617
Brown-Forsythe	2.3095	2	6	0.1804
Levene	4.3822	2	6	0.0671
Bartlett	3.5005	2		0.0302

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance (ANOVA)

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	9.0778E+3	4.5389E+3	60.58	0.0001
Error	6	4.4953E+2	7.4922E+1		
C. Total	8	9.5274E+3			

Statistically significant sampling variance.

Variance Components

Component	Variance Comp	Std Dev
Composite Samples	1.4880E+3	3.8575E+1
Measurements	7.4922E+1	8.6558E+0
Total	1.5629E+3	3.9534E+1
Mean Concentration	5.0432E+2	2.2457E+1

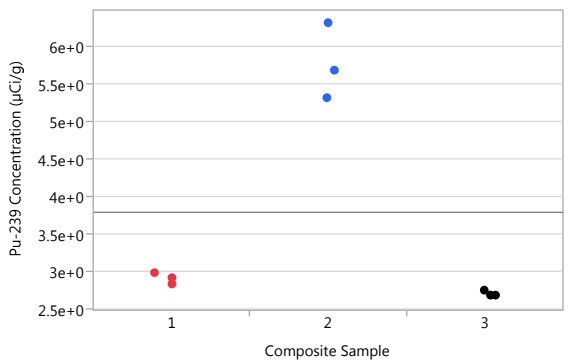
REML Mean = 8.9422E+1.

Student's t UCL95

UCL95	1.5500E+2

Pu-239 (μCi/g)

All Results (circles) above MDC



Means and Standard Deviations

Sample	Number	Mean	Std Dev	Std Err Mean
1	3	2.9100E+0	7.5498E-2	4.3589E-2
2	3	5.7700E+0	5.0110E-1	2.8931E-1
3	3	2.7100E+0	3.4641E-2	2.0000E-2
Overall	9	3.7967E+0	1.5041E+0	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	1.7288	2	6	0.2553
Brown-Forsythe	2.5494	2	6	0.1580
Levene	5.2998	2	6	0.0472
Bartlett	4.8391	2		0.0079

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance (ANOVA)

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	1.7583E+1	8.7916E+0	102.23	<.0001
Error	6	5.1600E-1	8.6000E-2		
C. Total	8	1.8099E+1			

Statistically significant sampling variance.

Variance Components

Component	Variance Comp	Std Dev
Composite Samples	2.9019E+0	1.7035E+0
Measurements	8.6000E-2	2.9326E-1
Total	2.9879E+0	1.7285E+0
Mean Concentration	9.7684E-1	9.8835E-1

REML Mean = 3.7967.

Student's t UCL95

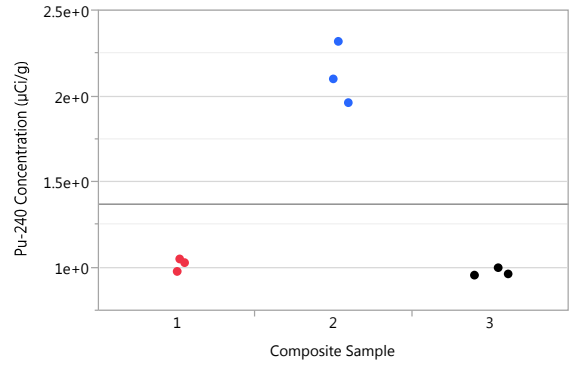
UCL95	6.6826

In the "ANOVA" table, DF: degrees of freedom; SS: Sums of Squares; MS: Mean Squares. In the "Tests that the Measurement Variances are Equal" table, DFNum: Numerator DF; DFDen: Denominator DF for the F Ratio.

Appendix E-13: Supporting Results for Radionuclides

Pu-240 (μCi/g) mixed

All Results (circles) above MDC



Means and Standard Deviations

Sample	Number	Mean	Std Dev	Std Err Mean
1	3	1.0190E+0	3.7723E-2	2.1779E-2
2	3	2.1267E+0	1.8148E-1	1.0477E-1
3	3	9.7133E-1	2.0984E-2	1.2115E-2
Overall	9	1.3723E+0	5.7376E-1	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	1.6764	2	6	0.2640
Brown-Forsythe	2.3167	2	6	0.1797
Levene	4.1643	2	6	0.0734
Bartlett	3.5392	2		0.0290

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance (ANOVA)

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	2.5640E+0	1.2820E+0	110.53	<.0001
Error	6	6.9593E-2	1.1599E-2		
C. Total	8	2.6336E+0			

Statistically significant sampling variance.

Variance Components

Component	Variance Comp	Std Dev
Composite Samples	4.2347E-1	6.5074E-1
Measurements	1.1599E-2	1.0770E-1
Total	4.3506E-1	6.5959E-1
Mean Concentration	1.4244E-1	3.7742E-1

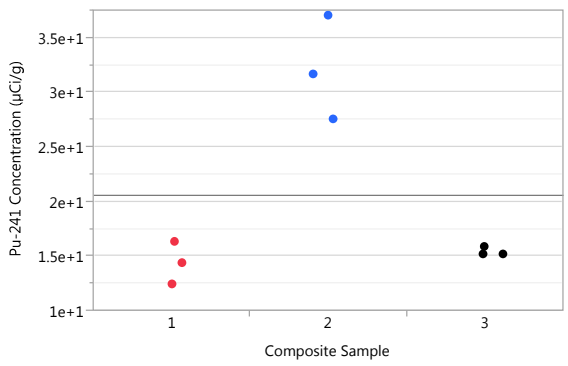
REML Mean = 1.3723.

Student's t UCL95

UCL95	2.4744

Pu-241 (μCi/g)

All Results (circles) above MDC



Means and Standard Deviations

Sample	Number	Mean	Std Dev	Std Err Mean
1	3	1.4333E+1	1.9502E+0	1.1260E+0
2	3	3.2033E+1	4.7648E+0	2.7510E+0
3	3	1.5367E+1	3.7859E-1	2.1858E-1
Overall	9	2.0578E+1	8.9822E+0	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	1.4754	2	6	0.3012
Brown-Forsythe	2.1295	2	6	0.2000
Levene	2.8353	2	6	0.1359
Bartlett	3.3028	2		0.0368

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance (ANOVA)

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	5.9216E+2	5.9214E+2	33.33	0.0006
Error	6	5.3300E+1	5.3300E+1		
C. Total	8	6.4544E+2			

Statistically significant sampling variance.

Variance Components

Component	Variance Comp	Std Dev
Composite Samples	9.5728E+1	9.7841E+0
Measurements	8.8833E+0	2.9805E+0
Total	1.0461E+2	1.0228E+1
Mean Concentration	3.2896E+1	5.7355E+0

REML Mean = 2.0578E+1.

Student's t UCL95

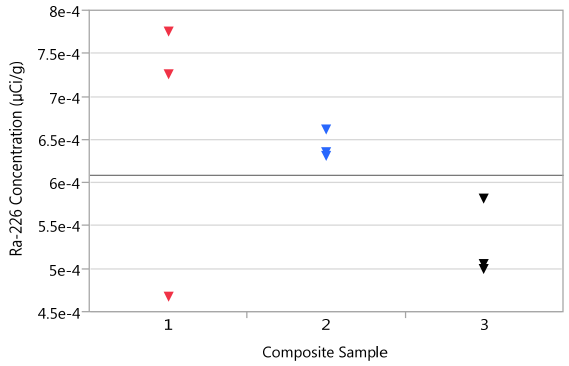
UCL95	3.7325E+1

In the "ANOVA" table, DF: degrees of freedom; SS: Sums of Squares; MS: Mean Squares. In the "Tests that the Measurement Variances are Equal" table, DFNum: Numerator DF; DFDen: Denominator DF for the F Ratio.

Appendix E-13: Supporting Results for Radionuclides

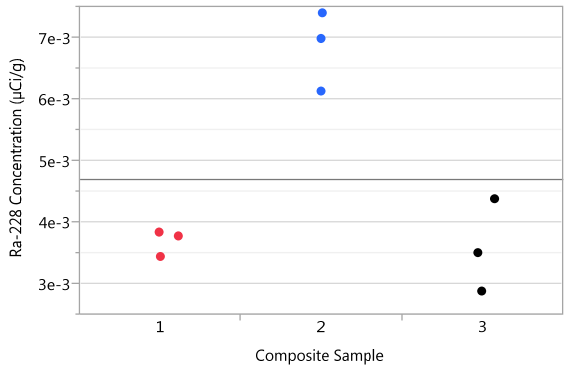
Ra-226 (μCi/g)

All Results (triangles) below MDC



Ra-228 (μCi/g)

All Results (circles) above MDC



Minimum Detectable Concentration (MDC)

Sample	Number	Minimum MDC	Maximum MDC
1	3	4.68E-4	7.75E-4
2	3	6.31E-4	6.62E-4
3	3	5.00E-4	5.81E-4
Overall	9	4.68E-4	7.75E-4

Means and Standard Deviations

Sample	Number	Mean	Std Dev	Std Err Mean
1	3	3.6833E-3	2.2143E-4	1.2785E-4
2	3	6.8333E-3	6.4268E-4	3.7105E-4
3	3	3.5800E-3	7.5319E-4	4.3486E-4
Overall	9	4.6989E-3	1.6799E-3	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	0.7633	2	6	0.5066
Brown-Forsythe	0.7855	2	6	0.4977
Levene	1.3792	2	6	0.3215
Bartlett	1.0287	2		0.3575

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance (ANOVA)

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	2.0517E-5	1.0259E-5	29.90	0.0008
Error	6	2.0587E-6	3.4312E-7		
C. Total	8	2.2576E-5			

Statistically significant sampling variance.

Variance Components

Component	Variance Comp	Std Dev
Composite Samples	3.3052E-6	1.8180E-3
Measurements	3.4312E-7	5.8577E-4
Total	3.6483E-6	1.9101E-3
Mean Concentration	1.1399E-6	1.0676E-3

REML Mean = 4.6989E-3.

Student's t UCL95

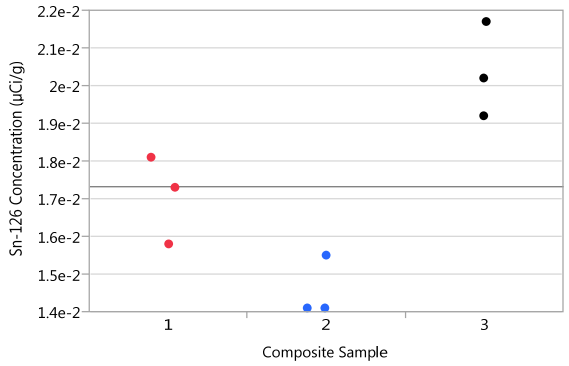
UCL95	7.8164E-3

In the "ANOVA" table, DF: degrees of freedom; SS: Sums of Squares; MS: Mean Squares. In the "Tests that the Measurement Variances are Equal" table, DFNum: Numerator DF; DFDen: Denominator DF for the F Ratio.

Appendix E-13: Supporting Results for Radionuclides

Sn-126 (μCi/g)

All Results (circles) above MDC



Means and Standard Deviations

Sample	Number	Mean	Std Dev	Std Err Mean
1	3	1.7067E-2	1.1676E-3	6.7412E-4
2	3	1.4567E-2	8.0829E-4	4.6667E-4
3	3	2.0367E-2	1.2583E-3	7.2648E-4
Overall	9	1.7333E-2	2.6921E-3	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	0.2629	2	6	0.7772
Brown-Forsythe	0.1907	2	6	0.8312
Levene	0.2403	2	6	0.7936
Bartlett	0.1662	2		0.8469

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance (ANOVA)

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	5.0780E-5	2.5390E-5	21.16	0.0019
Error	6	7.2000E-6	1.2000E-6		
C. Total	8	5.7980E-5			

Statistically significant sampling variance.

Variance Components

Component	Variance Comp	Std Dev
Composite Samples	8.0633E-6	2.8396E-3
Measurements	1.2000E-6	1.0954E-3
Total	9.2633E-6	3.0436E-3
Mean Concentration	2.8211E-6	1.6796E-3

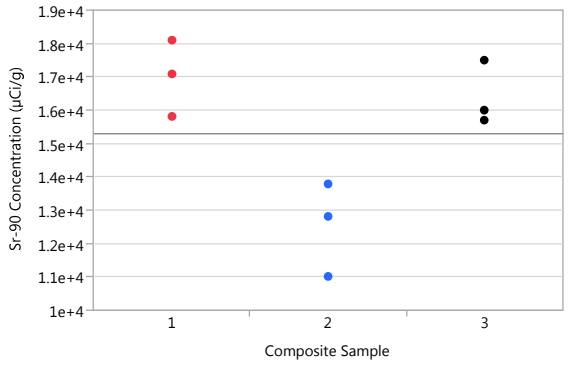
REML Mean = 1.7333E-2.

Student's t UCL95

UCL95	2.2238E-2

Sr-90 (μCi/g)

All Results (circles) above MDC



Means and Standard Deviations

Sample	Number	Mean	Std Dev	Std Err Mean
1	3	1.7000E+4	1.1533E+3	6.6583E+2
2	3	1.2533E+4	1.4189E+3	8.1921E+2
3	3	1.6400E+4	9.6437E+2	5.5678E+2
Overall	9	1.5311E+4	2.3401E+3	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	0.2393	2	6	0.7943
Brown-Forsythe	0.1311	2	6	0.8796
Levene	0.2192	2	6	0.8093
Bartlett	0.1219	2		0.8853

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance (ANOVA)

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	3.5262E+7	1.7631E+7	12.38	0.0074
Error	6	8.5467E+6	1.4244E+6		
C. Total	8	4.3809E+7			

Statistically significant sampling variance.

Variance Components

Component	Variance Comp	Std Dev
Composite Samples	5.4022E+6	2.3243E+3
Measurements	1.4244E+6	1.1935E+3
Total	6.8267E+6	2.6128E+3
Mean Concentration	1.9590E+6	1.3996E+3

REML Mean = 1.5311E+4.

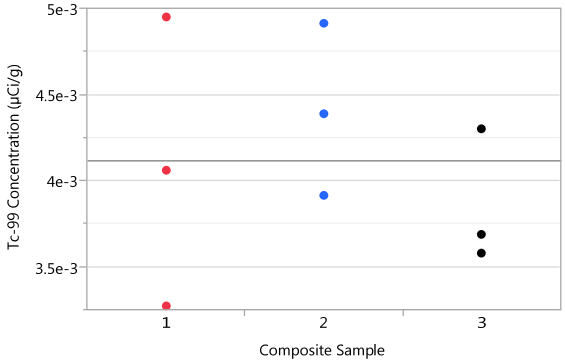
Student's t UCL95

UCL95	1.9398E+4

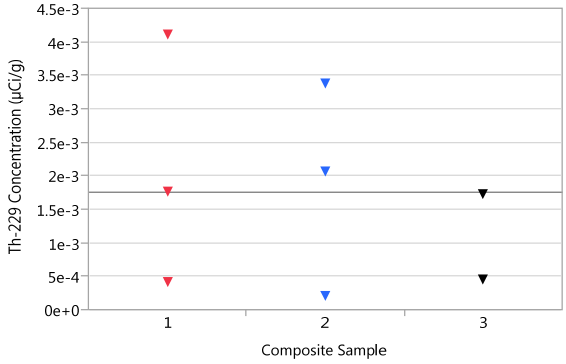
In the "ANOVA" table, DF: degrees of freedom; SS: Sums of Squares; MS: Mean Squares. In the "Tests that the Measurement Variances are Equal" table, DFNum: Numerator DF; DFDen: Denominator DF for the F Ratio.

Appendix E-13: Supporting Results for Radionuclides

Tc-99 (μCi/g)
All Results (circles) above MDC



Th-229 (μCi/g)
All Results (triangles) below MDC



Means and Standard Deviations

Sample	Number	Mean	Std Dev	Std Err Mean
1	3	4.0933E-3	8.4050E-4	4.8526E-4
2	3	4.4033E-3	5.0013E-4	2.8875E-4
3	3	3.8567E-3	3.8786E-4	2.2393E-4
Overall	9	4.1178E-3	5.7717E-4	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	0.8022	2	6	0.4912
Brown-Forsythe	0.5706	2	6	0.5931
Levene	0.6282	2	6	0.5653
Bartlett	0.5209	2		0.5940

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance (ANOVA)

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	4.5096E-7	2.2548E-7	0.61	0.5734
Error	6	2.2140E-6	3.6900E-7		
C. Total	8	2.6650E-6			

Statistically non-significant sampling variance.

Diagnostics for Tc-99 Measurements

Shapiro-Wilk	P-Value=0.7545>α=0.05 ->SNS
Dixon High Outlier	Run 3 of Sample 1 -> SNS
Dixon Low Outlier	Run 2 of Sample 1 -> SNS

Student's t UCL95

UCL95	4.4755E-3

Minimum Detectable Concentration (MDC)

Sample	Number	Minimum MDC	Maximum MDC
1	3	4.13E-4	4.10E-3
2	3	2.04E-4	3.38E-3
3	2	4.48E-4	1.72E-3
Overall	8	2.04E-4	4.10E-3

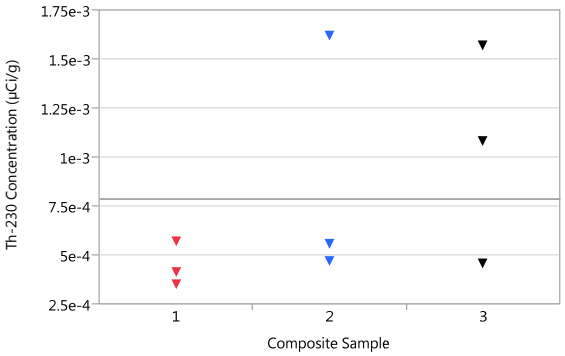
Th-229 had 1 missing result for Run 1 of Composite Sample 3.

In the "ANOVA" table, DF: degrees of freedom; SS: Sums of Squares; MS: Mean Squares. In the "Tests that the Measurement Variances are Equal" table, DFNum: Numerator DF; DFDen: Denominator DF for the F Ratio.
SNS: Statistically non-significant at $\alpha = 0.05$.

Appendix E-13: Supporting Results for Radionuclides

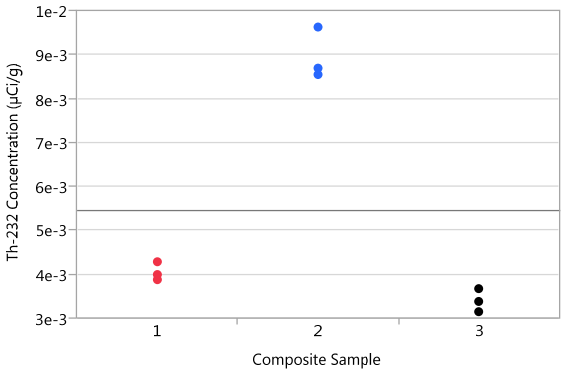
Th-230 (μCi/g)

All Results (triangles) below MDC



Th-232 (μCi/g)

All Results (circles) above MDC



Minimum Detectable Concentration (MDC)

Sample	Number	Minimum MDC	Maximum MDC
1	3	3.52E-4	5.68E-4
2	3	4.68E-4	1.62E-3
3	3	4.59E-4	1.57E-3
Overall	9	3.52E-4	1.62E-3

Means and Standard Deviations

Sample	Number	Mean	Std Dev	Std Err Mean
1	3	4.0433E-3	2.0526E-4	1.1851E-4
2	3	8.9467E-3	5.9652E-4	3.4440E-4
3	3	3.3933E-3	2.6577E-4	1.5344E-4
Overall	9	5.4611E-3	2.6515E-3	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	1.2033	2	6	0.3636
Brown-Forsythe	0.4542	2	6	0.6551
Levene	3.4843	2	6	0.0990
Bartlett	1.0478	2		0.3507

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance (ANOVA)

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	5.5305E-5	2.7652E-5	177.03	<.0001
Error	6	9.3720E-7	1.5620E-7		
C. Total	8	5.6242E-5			

Statistically significant sampling variance.

Variance Components

Component	Variance Comp	Std Dev
Composite Samples	9.1654E-6	3.0274E-3
Measurements	1.5620E-7	3.9522E-4
Total	9.3216E-6	3.0531E-3
Mean Concentration	3.0725E-6	1.7528E-3

REML Mean = 5.4611E-3.

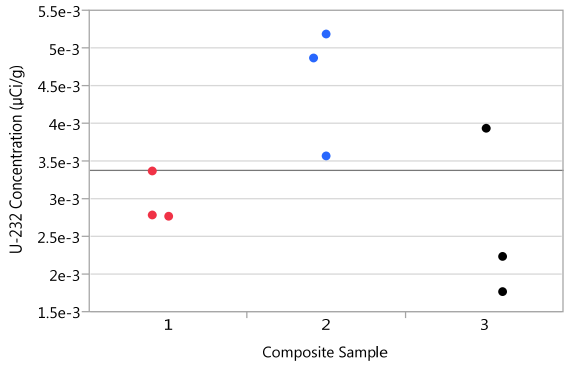
Student's t UCL95

UCL95	1.0579E-2

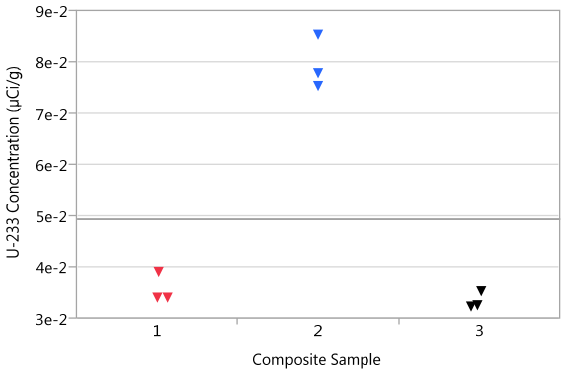
In the "ANOVA" table, DF: degrees of freedom; SS: Sums of Squares; MS: Mean Squares. In the "Tests that the Measurement Variances are Equal" table, DFNum: Numerator DF; DFDen: Denominator DF for the F Ratio.

Appendix E-13: Supporting Results for Radionuclides

U-232 (μCi/g)
All Results (circles) above MDC



U-233 (μCi/g)
All Results (triangles) below MDC



Means and Standard Deviations

Sample	Number	Mean	Std Dev	Std Err Mean
1	3	2.9700E-3	3.3808E-4	1.9519E-4
2	3	4.5367E-3	8.5231E-4	4.9208E-4
3	3	2.6433E-3	1.1378E-3	6.5690E-4
Overall	9	3.3833E-3	1.1411E-3	

Minimum Detectable Concentration (MDC)

Sample	Number	Minimum MDC	Maximum MDC
1	3	3.39E-2	3.89E-2
2	3	7.52E-2	8.53E-2
3	3	3.23E-2	3.52E-2
Overall	9	3.23E-2	8.53E-2

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	0.8383	2	6	0.4775
Brown-Forsythe	0.4716	2	6	0.6453
Levene	2.6884	2	6	0.1467
Bartlett	0.9903	2		0.3715

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance (ANOVA)

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	6.1459E-6	3.0729E-6	4.32	0.0689
Error	6	4.2705E-6	7.1176E-7		
C. Total	8	1.0416E-5			

Statistically non-significant sampling variance.

Diagnostics for U-232 Measurements

Shapiro-Wilk	P-Value=0.8235>α=0.05 ->SNS
Dixon High Outlier	Run 3 of Sample 2 -> SNS
Dixon Low Outlier	Run 2 of Sample 3 -> SNS

Student's t UCL95

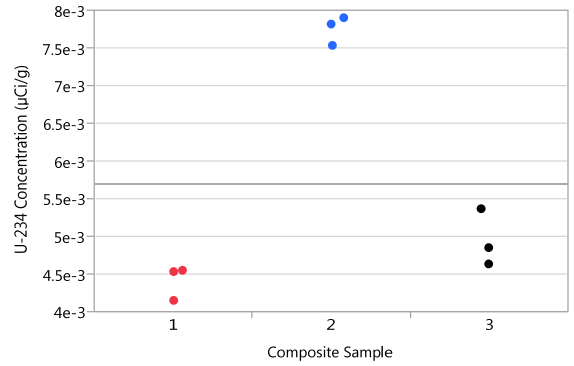
UCL95	4.0906E-3

In the "ANOVA" table, DF: degrees of freedom; SS: Sums of Squares; MS: Mean Squares. In the "Tests that the Measurement Variances are Equal" table, DFNum: Numerator DF; DFDen: Denominator DF for the F Ratio.
SNS: Statistically non-significant at $\alpha = 0.05$.

Appendix E-13: Supporting Results for Radionuclides

U-234 (μCi/g)

All Results (circles) above MDC



Means and Standard Deviations

Sample	Number	Mean	Std Dev	Std Err Mean
1	3	4.4100E-3	2.2539E-4	1.3013E-4
2	3	7.7500E-3	1.8735E-4	1.0817E-4
3	3	4.9500E-3	3.8000E-4	2.1939E-4
Overall	9	5.7033E-3	1.5711E-3	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	0.7552	2	6	0.5099
Brown-Forsythe	0.3284	2	6	0.7322
Levene	1.2589	2	6	0.3495
Bartlett	0.4611	2		0.6306

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance (ANOVA)

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	1.9287E-5	9.6436E-6	125.62	<.0001
Error	6	4.6060E-7	7.6767E-8		
C. Total	8	1.9748E-5			

Statistically significant sampling variance.

Variance Components

Component	Variance Comp	Std Dev
Composite Samples	3.1889E-6	1.7858E-3
Measurements	7.6767E-8	2.7707E-4
Total	3.2657E-6	1.8071E-3
Mean Concentration	1.0715E-6	1.0351E-3

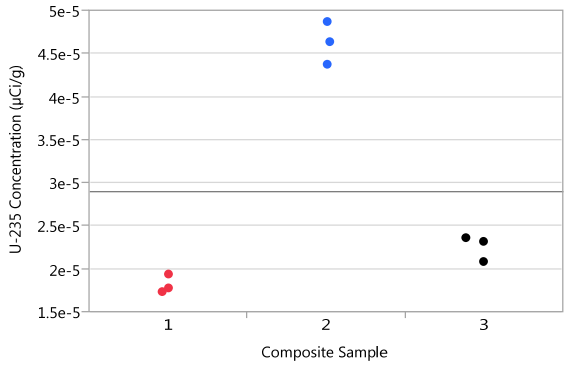
REML Mean = 5.7033E-3.

Student's t UCL95

UCL95	8.7259E-3

U-235 (μCi/g)

All Results (circles) above MDC



Means and Standard Deviations

Sample	Number	Mean	Std Dev	Std Err Mean
1	3	1.8133E-5	1.1150E-6	6.4377E-7
2	3	4.6300E-5	2.4515E-6	1.4154E-6
3	3	2.2567E-5	1.4572E-6	8.4130E-7
Overall	9	2.9000E-5	1.3205E-5	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	0.8136	2	6	0.4868
Brown-Forsythe	0.5027	2	6	0.6283
Levene	0.6920	2	6	0.5365
Bartlett	0.5356	2		0.5853

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance (ANOVA)

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	1.3763E-9	6.8814E-10	220.17	<.0001
Error	6	1.8753E-11	3.1256E-12		
C. Total	8	1.3950E-9			

Statistically significant sampling variance.

Variance Components

Component	Variance Comp	Std Dev
Composite Samples	2.2834E-10	1.5111E-5
Measurements	3.1256E-12	1.7679E-6
Total	2.3146E-10	1.5214E-5
Mean Concentration	7.6460E-11	8.7442E-6

REML Mean = 2.900E-5.

Student's t UCL95

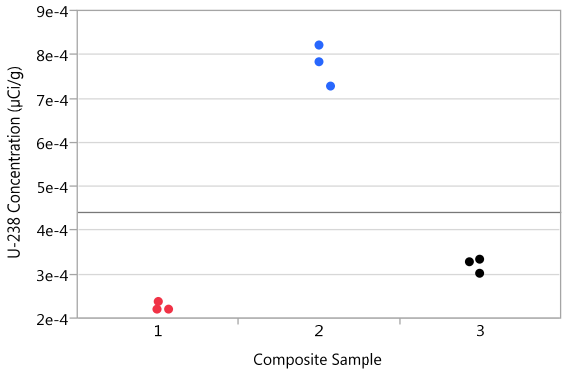
UCL95	5.4533E-5

In the "ANOVA" table, DF: degrees of freedom; SS: Sums of Squares; MS: Mean Squares. In the "Tests that the Measurement Variances are Equal" table, DFNum: Numerator DF; DFDen: Denominator DF for the F Ratio.

Appendix E-13: Supporting Results for Radionuclides

U-238 (μCi/g)

All Results (circles) above MDC



Means and Standard Deviations

Sample	Number	Mean	Std Dev	Std Err Mean
1	3	2.2667E-4	9.8150E-6	5.6667E-6
2	3	7.7700E-4	4.6293E-5	2.6727E-5
3	3	3.2200E-4	1.6823E-5	9.7125E-6
Overall	9	4.4189E-4	2.5594E-4	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	1.4587	2	6	0.3046
Brown-Forsythe	1.5014	2	6	0.2960
Levene	2.6623	2	6	0.1487
Bartlett	1.8980	2		0.1499

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance (ANOVA)

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	5.1898E-7	2.5949E-7	308.63	<.0001
Error	6	5.0447E-9	8.4078E-10		
C. Total	8	5.2403E-7			

Statistically significant sampling variance.

Variance Components

Component	Variance Comp	Std Dev
Composite Samples	8.6216E-8	2.9363E-4
Measurements	8.4078E-10	2.8996E-5
Total	8.7057E-8	2.9505E-4
Mean Concentration	2.8832E-8	1.6980E-4

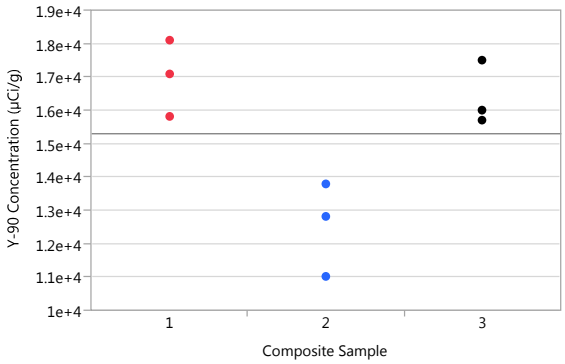
REML Mean = 4.4189E-4.

Student's t UCL95

UCL95	9.3770E-4

Y-90 (μCi/g)

All Results (circles) above MDC



Means and Standard Deviations

Sample	Number	Mean	Std Dev	Std Err Mean
1	3	1.7000E+4	1.1533E+3	6.6583E+2
2	3	1.2533E+4	1.4189E+3	8.1921E+2
3	3	1.6400E+4	9.6437E+2	5.5678E+2
Overall	9	1.5311E+4	2.3401E+3	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	0.2393	2	6	0.7943
Brown-Forsythe	0.1311	2	6	0.8796
Levene	0.2192	2	6	0.8093
Bartlett	0.1219	2		0.8853

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance (ANOVA)

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	3.5262E+7	1.7631E+7	12.38	0.0074
Error	6	8.5467E+6	1.4244E+6		
C. Total	8	4.3809E+7			

Statistically significant sampling variance.

Variance Components

Component	Variance Comp	Std Dev
Composite Samples	5.4022E+6	2.3243E+3
Measurements	1.4244E+6	1.1935E+3
Total	6.8267E+6	2.6128E+3
Mean Concentration	1.9590E+6	1.3996E+3

REML Mean = 1.5311E+4.

Student's t UCL95

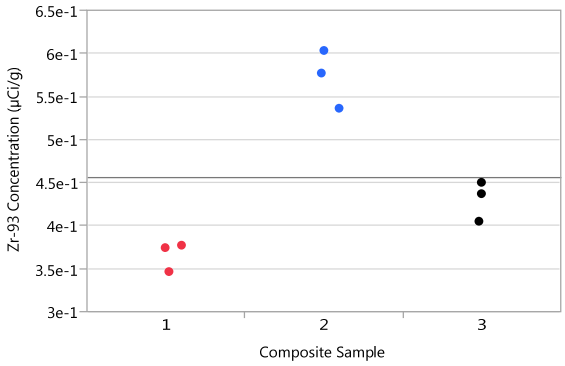
UCL95	1.9398E+4

In the "ANOVA" table, DF: degrees of freedom; SS: Sums of Squares; MS: Mean Squares. In the "Tests that the Measurement Variances are Equal" table, DFNum: Numerator DF; DFDen: Denominator DF for the F Ratio.

Appendix E-13: Supporting Results for Radionuclides

Zr-93 (μCi/g)

All Results (circles) above MDC



Means and Standard Deviations

Sample	Number	Mean	Std Dev	Std Err Mean
1	3	3.6633E-1	1.6862E-2	9.7354E-3
2	3	5.7233E-1	3.4239E-2	1.9768E-2
3	3	4.3067E-1	2.3159E-2	1.3371E-2
Overall	9	4.5644E-1	9.3962E-2	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	0.6409	2	6	0.5594
Brown-Forsythe	0.3854	2	6	0.6959
Levene	0.6955	2	6	0.5350
Bartlett	0.4048	2		0.6671

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance (ANOVA)

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	6.6644E-2	3.3322E-2	50.16	0.0002
Error	6	3.9860E-3	6.6433E-4		
C. Total	8	7.0630E-2			

Statistically significant sampling variance.

Variance Components

Component	Variance Comp	Std Dev
Composite Samples	1.0886E-2	1.0434E-1
Measurements	6.6433E-4	2.5775E-2
Total	1.1550E-2	1.0747E-1
Mean Concentration	3.7025E-3	6.0848E-2

REML Mean = 4.5644E-1.

Student's t UCL95

UCL95	6.3412E-1

In the "ANOVA" table, DF: degrees of freedom; SS: Sums of Squares; MS: Mean Squares. In the "Tests that the Measurement Variances are Equal" table, DFNum: Numerator DF; DFDen: Denominator DF for the F Ratio.

Appendix E-14: Summary for the Elemental Constituents with All Results above-MDC

Elemental Constituent	N	Mean (*wt %*)	Std Dev (*wt %*)	% Std Dev	UCL95 (*wt %*)	Remarks
Ag	9	6.0567E-2	2.7088E-2	44.72%	1.0619E-1	SNS-VH; SS-SV; Student's t Confidence Limit (2 DF)
Al	9	7.0844E+0	2.1444E+0	30.27%	1.0592E+1	SNS-VH; SS-SV; Student's t Confidence Limit (2 DF)
As	9	2.1633E-4	2.9639E-5	13.70%	2.6184E-4	SNS-VH; SS-SV; Student's t Confidence Limit (2 DF)
B	9	9.6689E-2	2.6484E-2	27.39%	1.4094E-1	SNS-VH; SS-SV; Student's t Confidence Limit (2 DF)
Ba	9	6.8833E-2	3.2989E-3	4.79%	7.0878E-2	SNS-VH; SNS-WS ; SNS-DT; SNS-SV; Student's t Confidence Limit (8 DF)
Cd	9	2.5011E-3	5.9219E-4	23.68%	3.4918E-3	SNS-VH; SS-SV; Student's t Confidence Limit (2 DF)
Co	9	4.3600E-3	1.4119E-3	32.38%	6.7180E-3	SNS-VH; SS-SV; Student's t Confidence Limit (2 DF)
Cr	9	5.0044E-2	1.5302E-2	30.58%	7.5660E-2	SNS-VH; SS-SV; Student's t Confidence Limit (2 DF)
Cu	9	1.5378E-1	2.6883E-2	17.48%	1.9839E-1	SNS-VH; SS-SV; Student's t Confidence Limit (2 DF)
Fe	9	3.0067E+1	7.1195E+0	23.68%	4.2039E+1	SNS-VH; SS-SV; Student's t Confidence Limit (2 DF)
Hg	9	1.5600E+1	3.8773E+0	24.85%	2.2065E+1	SNS-VH; SS-SV; Student's t Confidence Limit (2 DF)
Mn	9	1.3633E+0	2.0858E-1	15.30%	1.7096 E+0	SNS-VH; SS-SV; Student's t Confidence Limit (2 DF)
Ni	9	7.7233E-1	2.4253E-1	31.40%	1.1803E+0	SNS-VH; SS-SV; Student's t Confidence Limit (2 DF)
Pb	9	3.4400E-2	2.8108E-3	8.17%	3.8661E-2	SNS-VH; SS-SV; Student's t Confidence Limit (2 DF)
Sr	9	4.4522E-2	6.0117E-3	13.50%	5.4378E-2	SNS-VH; SS-SV; Student's t Confidence Limit (2 DF)
Th	9	4.9589E+0	2.7915E+0	56.29%	9.6591E+0	SNS-VH; SS-SV; Student's t Confidence Limit (2 DF)
U	9	8.7756E-2	5.6069E-2	63.89%	1.8194E-1	SNS-VH; SS-SV; Student's t Confidence Limit (2 DF)
Zn	9	3.3122E-2	2.5989E-3	7.85%	3.7184E-2	SNS-VH; SS-SV; Student's t Confidence Limit (2 DF)
Zr	9	1.1864E-1	2.8386E-2	23.93%	1.6589E-1	SNS-VH; SS-SV; Student's t Confidence Limit (2 DF)

MDC: Minimum Detectable Concentrations.
DF: degrees of freedom.
SS-VH (SNS-VH): Statistically significant (statistically non-significant) test for measurement error heterogeneity at $\alpha = 0.05/19=0.002632$.
SS-SV (SNS-SV): Statistically significant (statistically non-significant) sampling variance at $\alpha = 0.05$.
SS-WS (SNS-WS): Statistically significant (statistically non-significant) Wilk-Shapiro test for normality at $\alpha = 0.05$ (used for models without a sampling variance).
SS-DT (SNS-DT): Statistically significant (statistically non-significant) Dixon test for an outlier at $\alpha = 0.05$ (used for models without a sampling variance).

Appendix E-15: Summary for the Elemental Constituents with All Results below-MDC

Elemental Constituent	N	Smallest Minimum Detectable Concentration (*wt %*)		Largest Minimum Detectable Concentration (*wt %*)	
		Fixed Decimal Format	Scientific Format	Fixed Decimal Format	Scientific Format
Sb	9	0.000192	1.92E-4	0.0002	2.00E-4
Se	9	0.000288	2.88E-4	0.000299	2.99E-4

MDC: Minimum Detectable Concentrations

Appendix E-16: Statistical Summary of the UCL95 for the Elemental Constituent with a Mixture of Results above-MDC and below-MDC

Constituent	N◆	Mean (*wt %*)	Std Dev (*wt %*)	% Std Dev	UCL95 (*wt %*)	Remarks
Mo	9 (1,2,2)	1.3732E-3	1.7111E-4	12.46%	1.5148E-3	SNS-SV; Student's t Confidence Limit (4 DF)

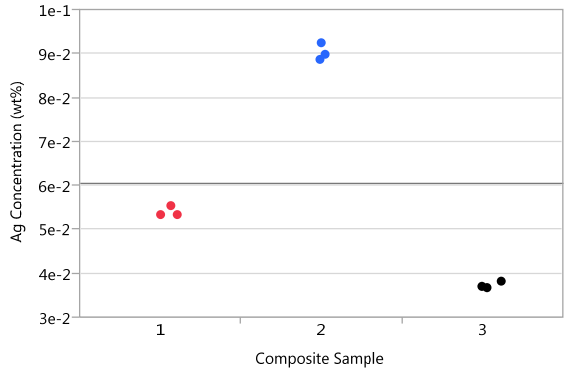
MDC: Minimum Detectable Concentrations.
DF: degrees of freedom.
SS-SV (SNS-SV): Statistically significant (statistically non-significant) sampling variance at $\alpha = 0.05$.

◆ N = 9 analytical results with the number of results (measurements) above the respective MDC listed inside the parentheses: (# above MDC for Composite Sample 1, # above MDC for Composite Sample 2, # above MDC for Composite Sample 3).

Appendix E-17: Supporting Results for Elemental Constituents

Ag (*wt %*)

All Results (circles) above MDC



Means and Standard Deviations

Sample	Number	Mean	Std Dev	Std Err Mean
1	3	5.4033E-2	1.1846E-3	6.8394E-4
2	3	9.0300E-2	1.9000E-3	1.0970E-3
3	3	3.7367E-2	8.1445E-4	4.7022E-4
Overall	9	6.0567E-2	2.3469E-2	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	0.8117	2	6	0.4876
Brown-Forsythe	0.3734	2	6	0.7033
Levene	1.5275	2	6	0.2909
Bartlett	0.5737	2		0.5634

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance (ANOVA)

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	4.3950E-3	2.1975E-3	1161.32	<.0001
Error	6	1.1353E-5	1.8922E-6		
C. Total	8	4.4063E-3			

Statistically significant sampling variance.

Variance Components

Component	Variance Comp	Std Dev
Composite Samples	7.3187E-4	2.7053E-2
Measurements	1.8922E-6	1.3756E-3
Total	7.3376E-4	2.7088E-2
Mean Concentration	2.4417E-4	1.5626E-2

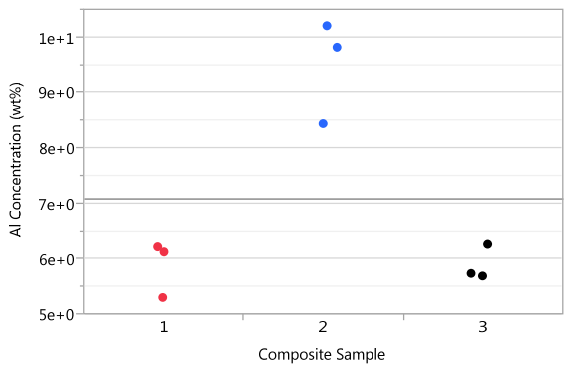
REML Mean = 6.0567E-2.

Student's t UCL95

UCL95	1.0619E-1

Al (*wt %*)

All Results (circles) above MDC



Means and Standard Deviations

Sample	Number	Mean	Std Dev	Std Err Mean
1	3	5.8767	5.1052E-1	2.9475E-1
2	3	9.4867	9.2614E-1	5.3471E-1
3	3	5.8900	3.1241E-1	1.8037E-1
Overall	9	7.0844	1.8842E+0	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	1.0504	2	6	0.4063
Brown-Forsythe	0.4737	2	6	0.6441
Levene	2.8842	2	6	0.1325
Bartlett	0.9129	2		0.4014

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance (ANOVA)

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	2.5968E+1	1.2984E+1	32.03	0.0006
Error	6	2.4319E+0	4.0532E-1		
C. Total	8	2.8400E+1			

Statistically significant sampling variance.

Variance Components

Component	Variance Comp	Std Dev
Composite Samples	4.1929E+0	2.0477E+0
Measurements	4.0532E-1	6.3665E-1
Total	4.5983E+0	2.1444E+0
Mean Concentration	1.4427E+0	1.2011E+0

REML Mean = 7.0844.

Student's t UCL95

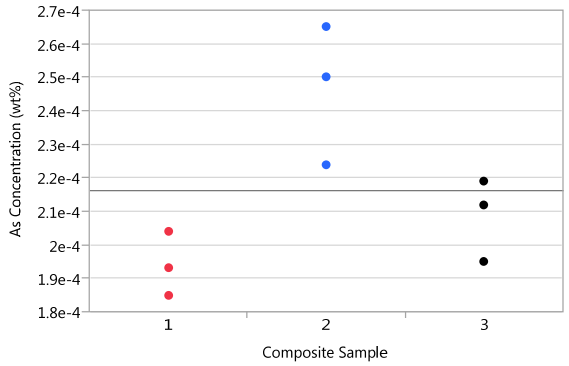
UCL95	1.0592E+1

In the "ANOVA" table, DF: degrees of freedom; SS: Sums of Squares; MS: Mean Squares. In the "Tests that the Measurement Variances are Equal" table, DFNum: Numerator DF; DFDen: Denominator DF for the F Ratio.

Appendix E-17: Supporting Results for Elemental Constituents

As (*wt %*)

All Results (circles) above MDC



Means and Standard Deviations

Sample	Number	Mean	Std Dev	Std Err Mean
1	3	1.9400E-4	9.5390E-6	5.5076E-6
2	3	2.4633E-4	2.0744E-5	1.1977E-5
3	3	2.0867E-4	1.2342E-5	7.1259E-6
Overall	9	2.1633E-4	2.6739E-5	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	0.8049	2	6	0.4902
Brown-Forsythe	0.4825	2	6	0.6393
Levene	1.0699	2	6	0.4005
Bartlett	0.5244	2		0.5919

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance (ANOVA)

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	4.3727E-9	2.1863E-9	9.74	0.0131
Error	6	1.3473E-9	2.2456E-10		
C. Total	8	5.7200E-9			

Statistically significant sampling variance.

Variance Components

Component	Variance Comp	Std Dev
Composite Samples	6.5393E-10	2.5572E-5
Measurements	2.2456E-10	1.4985E-5
Total	8.7848E-10	2.9639E-5
Mean Concentration	2.4293E-10	1.5586E-5

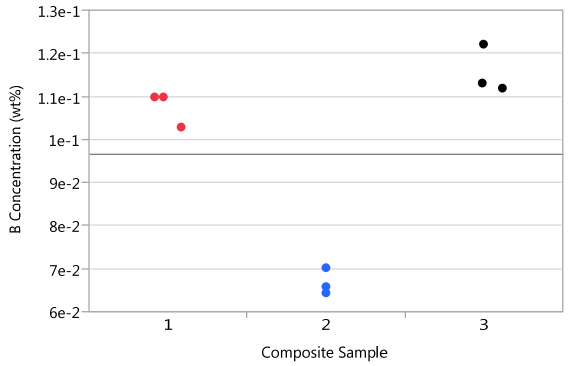
REML Mean = 2.1633E-4.

Student's t UCL95

UCL95	2.6184E-4

B (*wt %*)

All Results (circles) above MDC



Means and Standard Deviations

Sample	Number	Mean	Std Dev	Std Err Mean
1	3	1.0767E-1	4.0415E-3	2.3333E-3
2	3	6.6733E-2	3.0105E-3	1.7381E-3
3	3	1.1567E-1	5.5076E-3	3.1798E-3
Overall	9	9.6689E-2	2.3037E-2	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	0.4912	2	6	0.6345
Brown-Forsythe	0.1030	2	6	0.9037
Levene	1.2793	2	6	0.3445
Bartlett	0.2913	2		0.7473

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance (ANOVA)

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	4.1340E-3	2.0670E-3	111.27	<.0001
Error	6	1.1146E-4	1.8577E-5		
C. Total	8	4.2455E-3			

Statistically significant sampling variance.

Variance Components

Component	Variance Comp	Std Dev
Composite Samples	6.8281E-4	2.6131E-2
Measurements	1.8577E-5	4.3101E-3
Total	7.0139E-4	2.6484E-2
Mean Concentration	2.2967E-4	1.5155E-2

REML Mean = 9.6689E-2.

Student's t UCL95

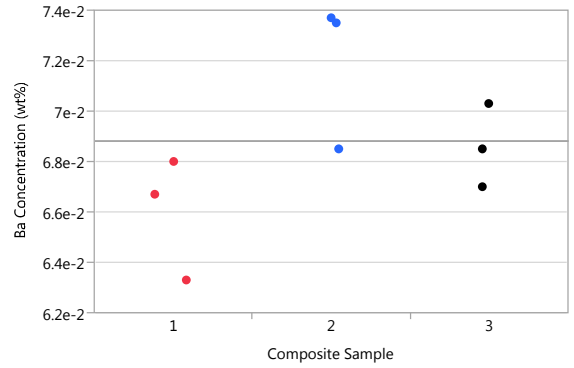
UCL95	1.4094E-1

In the "ANOVA" table, DF: degrees of freedom; SS: Sums of Squares; MS: Mean Squares. In the "Tests that the Measurement Variances are Equal" table, DFNum: Numerator DF; DFDen: Denominator DF for the F Ratio.

Appendix E-17: Supporting Results for Elemental Constituents

Ba (*wt %*)

All Results (circles) above MDC



Means and Standard Deviations

Sample	Number	Mean	Std Dev	Std Err Mean
1	3	6.6000E-2	2.4269E-3	1.4012E-3
2	3	7.1900E-2	2.9462E-3	1.7010E-3
3	3	6.8600E-2	1.6522E-3	9.5394E-4
Overall	9	6.8833E-2	3.2989E-3	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	0.4023	2	6	0.6856
Brown-Forsythe	0.0816	2	6	0.9226
Levene	1.0404	2	6	0.4094
Bartlett	0.2599	2		0.7711

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance (ANOVA)

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	5.2460E-5	2.6230E-5	4.55	0.0628
Error	6	3.4600E-5	5.7667E-6		
C. Total	8	8.7060E-5			

Statistically non-significant sampling variance.

Diagnostics for Ba Measurements

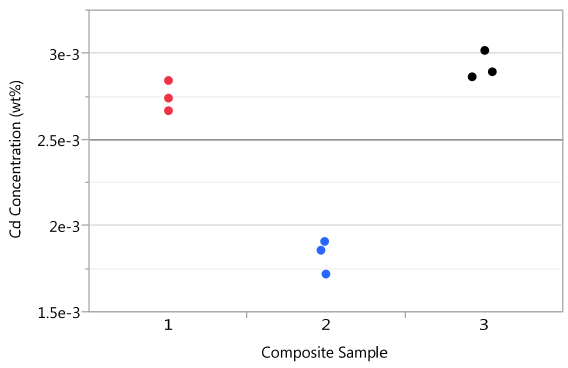
Shapiro-Wilk	P-Value=0.5440>α=0.05 ->SNS
Dixon High Outlier	Run 2 of Sample 2 -> SNS
Dixon Low Outlier	Run 1 of Sample 1 -> SNS

Student's t UCL95

UCL95	7.0878E-2

Cd (*wt %*)

All Results (circles) above MDC



Means and Standard Deviations

Sample	Number	Mean	Std Dev	Std Err Mean
1	3	2.7500E-3	8.5440E-5	4.9329E-5
2	3	1.8300E-3	9.8489E-5	5.6862E-5
3	3	2.9233E-3	8.5049E-5	4.9103E-5
Overall	9	2.5011E-3	5.1482E-4	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	0.0527	2	6	0.9491
Brown-Forsythe	0.0190	2	6	0.9813
Levene	0.0914	2	6	0.9139
Bartlett	0.0234	2		0.9768

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance (ANOVA)

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	2.0718E-6	1.0359E-6	128.24	<.0001
Error	6	4.8467E-8	8.0778E-9		
C. Total	8	2.1203E-6			

Statistically significant sampling variance.

Variance Components

Component	Variance Comp	Std Dev
Composite Samples	3.4261E-7	5.8533E-4
Measurements	8.0778E-9	8.9876E-5
Total	3.5069E-7	5.9219E-4
Mean Concentration	1.1510E-7	3.3927E-4

REML Mean = 2.5011E-3.

Student's t UCL95

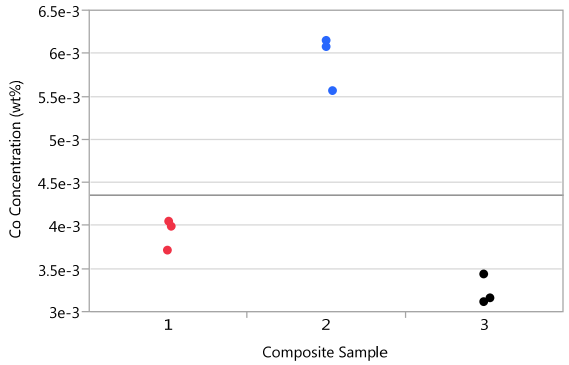
UCL95	3.4918E-3

In the "ANOVA" table, DF: degrees of freedom; SS: Sums of Squares; MS: Mean Squares. In the "Tests that the Measurement Variances are Equal" table, DFNum: Numerator DF; DFDen: Denominator DF for the F Ratio.
SNS: Statistically non-significant at $\alpha = 0.05$.

Appendix E-17: Supporting Results for Elemental Constituents

Co (*wt %*)

All Results (circles) above MDC



Means and Standard Deviations

Sample	Number	Mean	Std Dev	Std Err Mean
1	3	3.9167E-3	1.8148E-4	1.0477E-4
2	3	5.9267E-3	3.2005E-4	1.8478E-4
3	3	3.2367E-3	1.7786E-4	1.0269E-4
Overall	9	4.3600E-3	1.2284E-3	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	0.6957	2	6	0.5349
Brown-Forsythe	0.1819	2	6	0.8381
Levene	1.6340	2	6	0.2713
Bartlett	0.3929	2		0.6751

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance (ANOVA)

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	1.1739E-5	5.8693E-6	105.44	<.0001
Error	6	3.3400E-7	5.5667E-8		
C. Total	8	1.2073E-5			

Statistically significant sampling variance.

Variance Components

Component	Variance Comp	Std Dev
Composite Samples	1.9379E-6	1.3921E-3
Measurements	5.5667E-8	2.3594E-4
Total	1.9935E-6	1.4119E-3
Mean Concentration	6.5214E-7	8.0755E-4

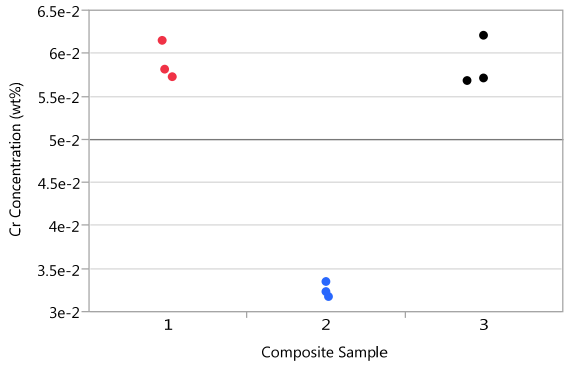
REML Mean = 4.3600E-3.

Student's t UCL95

UCL95	6.7180E-3

Cr (*wt %*)

All Results (circles) above MDC



Means and Standard Deviations

Sample	Number	Mean	Std Dev	Std Err Mean
1	3	5.8967E-2	2.2502E-3	1.2991E-3
2	3	3.2500E-2	9.1652E-4	5.2915E-4
3	3	5.8667E-2	2.9771E-3	1.7188E-3
Overall	9	5.0044E-2	1.3299E-2	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	0.8190	2	6	0.4848
Brown-Forsythe	0.2931	2	6	0.7560
Levene	3.0845	2	6	0.1199
Bartlett	0.9423	2		0.3897

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance (ANOVA)

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	1.3853E-3	6.9263E-4	140.72	<.0001
Error	6	2.9533E-5	4.9222E-6		
C. Total	8	1.4148E-3			

Statistically significant sampling variance.

Variance Components

Component	Variance Comp	Std Dev
Composite Samples	2.2924E-4	1.5141E-2
Measurements	4.9222E-6	2.2186E-3
Total	2.3416E-4	1.5302E-2
Mean Concentration	7.6959E-5	8.7726E-3

REML Mean = 5.0044E-2.

Student's t UCL95

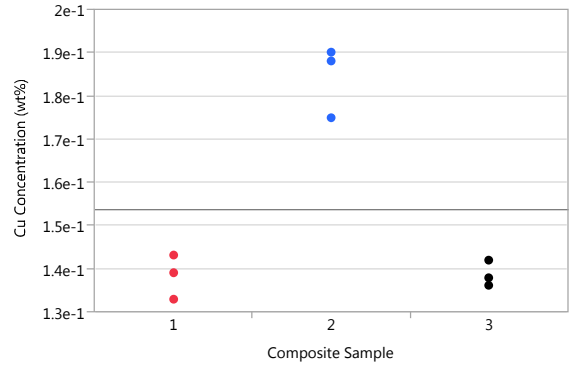
UCL95	7.5660E-2

In the "ANOVA" table, DF: degrees of freedom; SS: Sums of Squares; MS: Mean Squares. In the "Tests that the Measurement Variances are Equal" table, DFNum: Numerator DF; DFDen: Denominator DF for the F Ratio.

Appendix E-17: Supporting Results for Elemental Constituents

Cu (*wt %*)

All Results (circles) above MDC



Means and Standard Deviations

Sample	Number	Mean	Std Dev	Std Err Mean
1	3	1.3833E-1	5.0332E-3	2.9059E-3
2	3	1.8433E-1	8.1445E-3	4.7022E-3
3	3	1.3867E-1	3.0551E-3	1.7638E-3
Overall	9	1.5378E-1	2.3462E-2	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	0.8988	2	6	0.4556
Brown-Forsythe	0.3262	2	6	0.7337
Levene	2.2550	2	6	0.1861
Bartlett	0.7275	2		0.4831

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance (ANOVA)

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	4.2016E-3	2.1008E-3	62.40	<.0001
Error	6	2.0200E-4	3.3667E-5		
C. Total	8	4.4036E-3			

Statistically significant sampling variance.

Variance Components

Component	Variance Comp	Std Dev
Composite Samples	6.8904E-4	2.6250E-2
Measurements	3.3667E-5	5.8023E-3
Total	7.2270E-4	2.6883E-2
Mean Concentration	2.3342E-4	1.5278E-2

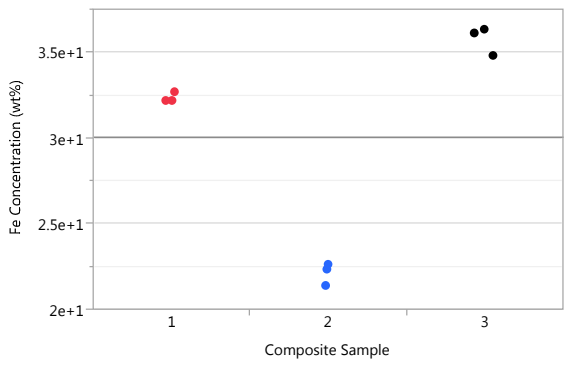
REML Mean = 1.5378E-1.

Student's t UCL95

UCL95	1.9839E-1

Fe (*wt %*)

All Results (circles) above MDC



Means and Standard Deviations

Sample	Number	Mean	Std Dev	Std Err Mean
1	3	3.2367E+1	2.8868E-1	1.6667E-1
2	3	2.2100E+1	6.2450E-1	3.6056E-1
3	3	3.5733E+1	8.1445E-1	4.7022E-1
Overall	9	3.0067E+1	6.1733	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	0.7496	2	6	0.5122
Brown-Forsythe	0.3362	2	6	0.7271
Levene	2.3580	2	6	0.1755
Bartlett	0.7572	2		0.4690

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance (ANOVA)

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	3.0261E+2	3.0261E+2	399.33	<.0001
Error	6	2.2733E+0	2.2733E+0		
C. Total	8	3.0488E+2			

Statistically significant sampling variance.

Variance Components

Component	Variance Comp	Std Dev
Composite Samples	5.0308E+1	7.0928E+0
Measurements	3.7889E-1	6.1554E-1
Total	5.0687E+1	7.1195E+0
Mean Concentration	1.6811E+1	4.1002E+0

REML Mean = 3.0067E+1.

Student's t UCL95

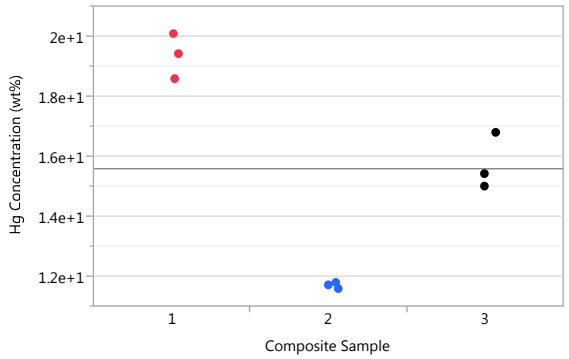
UCL95	4.2039E+1

In the "ANOVA" table, DF: degrees of freedom; SS: Sums of Squares; MS: Mean Squares. In the "Tests that the Measurement Variances are Equal" table, DFNum: Numerator DF; DFDen: Denominator DF for the F Ratio.

Appendix E-17: Supporting Results for Elemental Constituents

Hg (*wt %*)

All Results (circles) above MDC



Means and Standard Deviations

Sample	Number	Mean	Std Dev	Std Err Mean
1	3	1.9367E+1	7.5056E-1	4.3333E-1
2	3	1.1700E+1	1.0000E-1	5.7735E-2
3	3	1.5733E+1	9.4516E-1	5.4569E-1
Overall	9	1.5600E+1	3.3760	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	0.9525	2	6	0.4373
Brown-Forsythe	1.0140	2	6	0.4175
Levene	3.1627	2	6	0.1154
Bartlett	2.5732	2		0.0763

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance (ANOVA)

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	8.8247E+1	4.4123E+1	90.252	<.0001
Error	6	2.9333E+0	4.8889E-1		
C. Total	8	9.1180E+1			

Statistically significant sampling variance.

Variance Components

Component	Variance Comp	Std Dev
Composite Samples	1.4545E+1	3.8138E+0
Measurements	4.8889E-1	6.9921E-1
Total	1.5034E+1	3.8773E+0
Mean Concentration	4.9026E+0	2.2142E+0

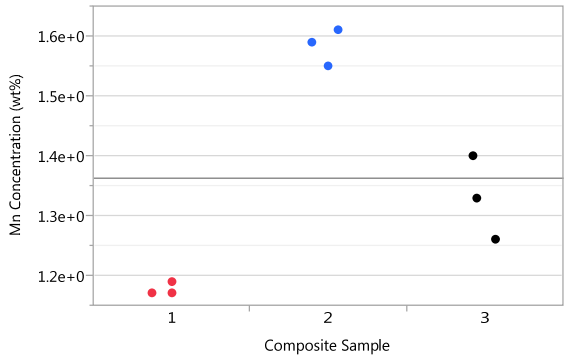
REML Mean = 1.5600E+1.

Student's t UCL95

UCL95	2.2065E+1

Mn (*wt %*)

All Results (circles) above MDC



Means and Standard Deviations

Sample	Number	Mean	Std Dev	Std Err Mean
1	3	1.1767	1.1547E-2	6.6667E-3
2	3	1.5833	3.0551E-2	1.7638E-2
3	3	1.3300	7.0000E-2	4.0415E-2
Overall	9	1.3633	1.8200E-1	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	1.3957	2	6	0.3179
Brown-Forsythe	1.7231	2	6	0.2563
Levene	1.7948	2	6	0.2449
Bartlett	2.0924	2		0.1234

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance (ANOVA)

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	2.5307E-1	1.2653E-1	63.620	<.0001
Error	6	1.1933E-2	1.9889E-3		
C. Total	8	2.6500E-1			

Statistically significant sampling variance.

Variance Components

Component	Variance Comp	Std Dev
Composite Samples	4.1515E-2	2.0375E-1
Measurements	1.9889E-3	4.4597E-2
Total	4.3504E-2	2.0858E-1
Mean Concentration	1.4059E-2	1.1857E-1

REML Mean = 1.3633.

Student's t UCL95

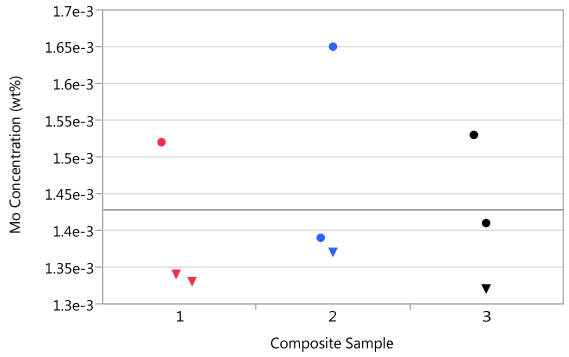
UCL95	1.7096

In the "ANOVA" table, DF: degrees of freedom; SS: Sums of Squares; MS: Mean Squares. In the "Tests that the Measurement Variances are Equal" table, DFNum: Numerator DF; DFDen: Denominator DF for the F Ratio.

Appendix E-17: Supporting Results for Elemental Constituents

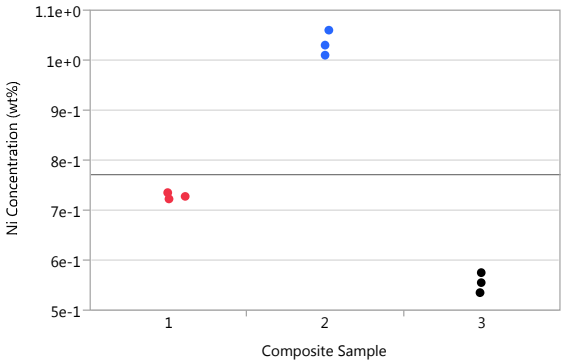
Mo (*wt %*)

Results (circles) above and (triangles) below MDC



Ni (*wt %*)

All Results (circles) above MDC



The results for Mo concentration had a pattern of 1 measurement and 2 below-MDC results on Composite Sample 1; 2 measurements and 1 below-MDC result on Composite Sample 2; and 2 measurements and 1 below-MDC result on Composite Sample 3. A fixed effects model was run in SAS® Proc Reliability treating each below-MDC result as interval censored between 0 and its MDC. The results are given in the following table. The intercept term is the mean of Composite Sample 3.

Mo	Estimate	Std Error	Csamp Means
Intercept	1.3834E-03	1.0060E-04	
Csamp 1	-8.2437E-05	1.5081E-04	1.3009E-03
Csamp 2	5.0000E-05	1.4032E-04	1.4334E-03
Csamp 3	0.0	0.0	1.3834E-03
Std Dev	1.6342E-04	5.7365E-05	

The standard errors for the fixed effects "Csamp 1" and "Csamp 2" are greater than the estimates, so an analysis without the sampling effect was run. The results from SAS® Proc Reliability are as follows. The intercept term is the overall mean.

Mo	Estimate	Std Error
Intercept	1.3732E-03	6.6442E-05
Std Dev	1.7111E-04	6.0195E-05

A Student's t confidence interval (conservative with 4 DF) was used for the UCL95.

Mean	1.3732E-3
Std Error Mean	6.6442E-5
Standard Deviation	1.7111E-4
UCL95	1.3821E-3

Means and Standard Deviations

Sample	Number	Mean	Std Dev	Std Err Mean
1	3	7.2833E-1	7.0946E-3	4.0961E-3
2	3	1.0333E+0	2.5166E-2	1.4530E-2
3	3	5.5533E-1	2.1008E-2	1.2129E-2
Overall	9	7.7233E-1	2.1026E-1	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	0.7867	2	6	0.4973
Brown-Forsythe	0.8896	2	6	0.4588
Levene	1.2459	2	6	0.3527
Bartlett	1.0811	2		0.3392

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance (ANOVA)

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	3.5144E-1	1.7572E-1	468.58	<.0001
Error	6	2.2500E-3	3.7001E-4		
C. Total	8	3.5369E-1			

Statistically significant sampling variance.

Variance Components

Component	Variance Comp	Std Dev
Composite Samples	5.8448E-2	2.4176E-1
Measurements	3.7500E-4	1.9365E-2
Total	5.8823E-2	2.4253E-1
Mean Concentration	1.9524E-2	1.3973E-1

REML Mean = 7.7233E-1.

Student's t UCL95

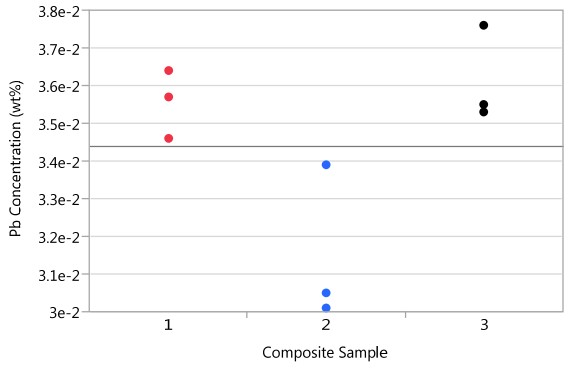
UCL95	1.5148E-3

In the "ANOVA" table, DF: degrees of freedom; SS: Sums of Squares; MS: Mean Squares. In the "Tests that the Measurement Variances are Equal" table, DFNum: Numerator DF; DFDen: Denominator DF for the F Ratio.

Appendix E-17: Supporting Results for Elemental Constituents

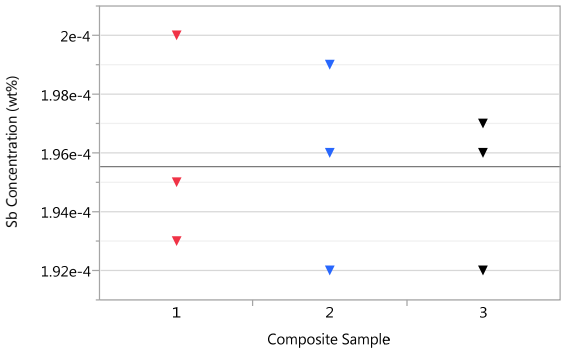
Pb (*wt %*)

All Results (circles) above MDC



Sb (*wt %*)

All Results (triangles) below MDC



Means and Standard Deviations

Sample	Number	Mean	Std Dev	Std Err Mean
1	3	3.5567E-2	9.0738E-4	5.2387E-4
2	3	3.1500E-2	2.0881E-3	1.2055E-3
3	3	3.6133E-2	1.2741E-3	7.3560E-4
Overall	9	3.4400E-2	2.5480E-3	

Minimum Detectable Concentration (MDC)

Sample	Number	Minimum MDC	Maximum MDC
1	3	1.93E-4	2.00E-4
2	3	1.92E-4	1.99E-4
3	3	1.92E-4	1.97E-4
Overall	9	1.92E-4	2.00E-4

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	0.8218	2	6	0.4837
Brown-Forsythe	0.2121	2	6	0.8147
Levene	2.3274	2	6	0.1786
Bartlett	0.5689	2		0.5662

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance (ANOVA)

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	3.8327E-5	1.9163E-5	8.446	0.0180
Error	6	1.3613E-5	2.2689E-6		
C. Total	8	5.1940E-5			

Statistically significant sampling variance.

Variance Components

Component	Variance Comp	Std Dev
Composite Samples	5.6315E-6	2.3731E-3
Measurements	2.2689E-6	1.5063E-3
Total	7.9004E-6	2.8108E-3
Mean Concentration	2.1293E-6	1.4592E-3

REML Mean = 3.4400E-2.

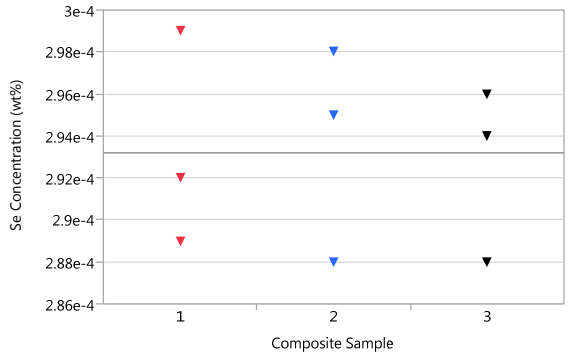
Student's t UCL95

UCL95	3.8661E-2

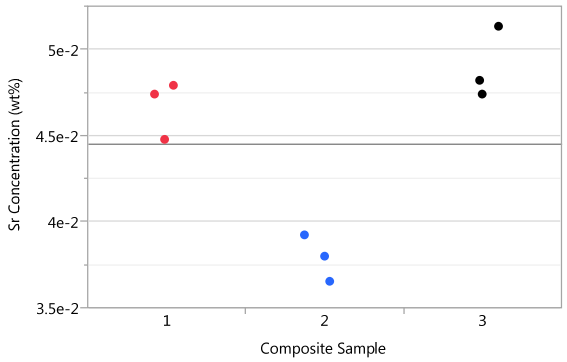
In the "ANOVA" table, DF: degrees of freedom; SS: Sums of Squares; MS: Mean Squares. In the "Tests that the Measurement Variances are Equal" table, DFNum: Numerator DF; DFDen: Denominator DF for the F Ratio.

Appendix E-17: Supporting Results for Elemental Constituents

Se (*wt %*)
All Results (triangles) below MDC



Sr (*wt %*)
All Results (circles) above MDC



Minimum Detectable Concentration (MDC)

Sample	Number	Minimum MDC	Maximum MDC
1	3	2.89E-4	2.99E-4
2	3	2.88E-4	2.98E-4
3	3	2.88E-4	2.96E-4
Overall	9	2.88E-4	2.99E-4

Means and Standard Deviations

Sample	Number	Mean	Std Dev	Std Err Mean
1	3	4.6700E-2	1.6643E-3	9.6090E-4
2	3	3.7900E-2	1.3528E-3	7.8102E-4
3	3	4.8967E-2	2.0599E-3	1.1893E-3
Overall	9	4.4522E-2	5.2765E-3	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	0.2719	2	6	0.7709
Brown-Forsythe	0.0729	2	6	0.9305
Levene	0.5816	2	6	0.5877
Bartlett	0.1429	2		0.8669

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance (ANOVA)

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	2.0505E-4	1.0252E-4	34.780	0.0005
Error	6	1.7687E-5	2.9478E-6		
C. Total	8	2.2274E-4			

Statistically significant sampling variance.

Variance Components

Component	Variance Comp	Std Dev
Composite Samples	3.3192E-5	5.7613E-3
Measurements	2.9478E-6	1.7169E-3
Total	3.6140E-5	6.0117E-3
Mean Concentration	1.1392E-5	3.3751E-3

REML Mean = 4.4522E-2.

Student's t UCL95

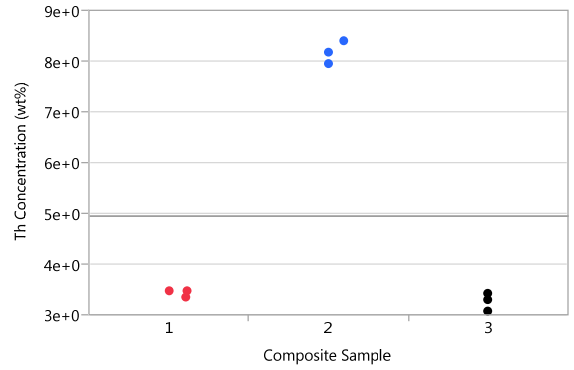
UCL95	5.4378E-2

In the "ANOVA" table, DF: degrees of freedom; SS: Sums of Squares; MS: Mean Squares. In the "Tests that the Measurement Variances are Equal" table, DFNum: Numerator DF; DFDen: Denominator DF for the F Ratio.

Appendix E-17: Supporting Results for Elemental Constituents

Th (*wt %*)

All Results (circles) above MDC



Means and Standard Deviations

Sample	Number	Mean	Std Dev	Std Err Mean
1	3	3.4367	6.6583E-2	3.8442E-2
2	3	8.1767	2.2502E-1	1.2991E-1
3	3	3.2633	1.7786E-1	1.0269E-1
Overall	9	4.9589	2.4190E+0	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	0.8024	2	6	0.4911
Brown-Forsythe	0.8463	2	6	0.4745
Levene	1.0510	2	6	0.4061
Bartlett	1.0011	2		0.3675

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance (ANOVA)

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	4.6638E+1	2.3319E+1	806.89	<.0001
Error	6	1.7340E-1	2.8900E-2		
C. Total	8	4.6812E+1			

Statistically significant sampling variance.

Variance Components

Component	Variance Comp	Std Dev
Composite Samples	7.7634	2.7863
Measurements	2.8900E-2	1.7000E-1
Total	7.7923	2.7915
Mean Concentration	2.5910	1.6097

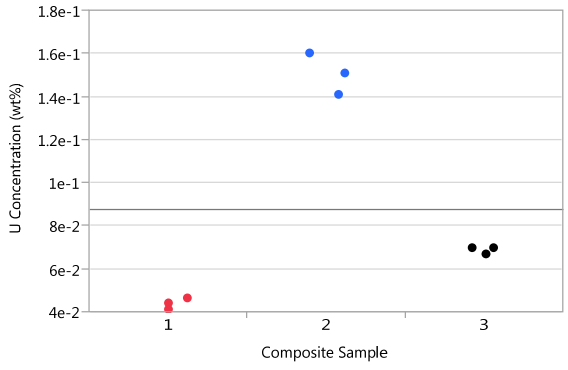
REML Mean = 4.9589.

Student's t UCL95

UCL95	9.6591

U (*wt %*)

All Results (circles) above MDC



Means and Standard Deviations

Sample	Number	Mean	Std Dev	Std Err Mean
1	3	4.3933E-2	2.8006E-3	1.6169E-3
2	3	1.5067E-1	9.5044E-3	5.4874E-3
3	3	6.8667E-2	1.7039E-3	9.8376E-4
Overall	9	8.7756E-2	4.8644E-2	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	1.5632	2	6	0.2842
Brown-Forsythe	2.0669	2	6	0.2076
Levene	2.3063	2	6	0.1807
Bartlett	2.3914	2		0.0915

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance (ANOVA)

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	1.8728E-2	9.3639E-3	277.91	<.0001
Error	6	2.0216E-4	3.3693E-5		
C. Total	8	1.8930E-2			

Statistically significant sampling variance.

Variance Components

Component	Variance Comp	Std Dev
Composite Samples	3.1101E-3	5.5768E-2
Measurements	3.3693E-5	5.8046E-3
Total	3.1438E-3	5.6069E-2
Mean Concentration	1.0404E-3	3.2256E-2

REML Mean = 8.7756E-2.

Student's t UCL95

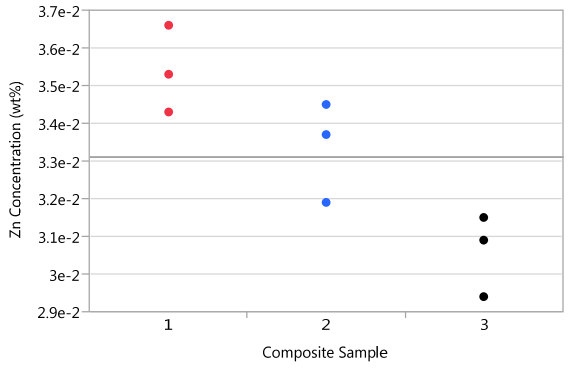
UCL95	1.8194E-1

In the "ANOVA" table, DF: degrees of freedom; SS: Sums of Squares; MS: Mean Squares. In the "Tests that the Measurement Variances are Equal" table, DFNum: Numerator DF; DFDen: Denominator DF for the F Ratio.

Appendix E-17: Supporting Results for Elemental Constituents

Zn (*wt %*)

All Results (circles) above MDC



Means and Standard Deviations

Sample	Number	Mean	Std Dev	Std Err Mean
1	3	3.5400E-2	1.1533E-3	6.6583E-4
2	3	3.3367E-2	1.3317E-3	7.6884E-4
3	3	3.0600E-2	1.0817E-3	6.2450E-4
Overall	9	3.3122E-2	2.3285E-3	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	0.0829	2	6	0.9214
Brown-Forsythe	0.0343	2	6	0.9665
Levene	0.1031	2	6	0.9036
Bartlett	0.0379	2		0.9628

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance (ANOVA)

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	3.4829E-5	1.7414E-5	12.2254	0.0076
Error	6	8.5467E-6	1.4244E-6		
C. Total	8	4.3376E-5			

Statistically significant sampling variance.

Variance Components

Component	Variance Comp	Std Dev
Composite Samples	5.3300E-6	2.3087E-3
Measurements	1.4244E-6	1.1935E-3
Total	6.7544E-6	2.5989E-3
Mean Concentration	1.9349E-6	1.3910E-3

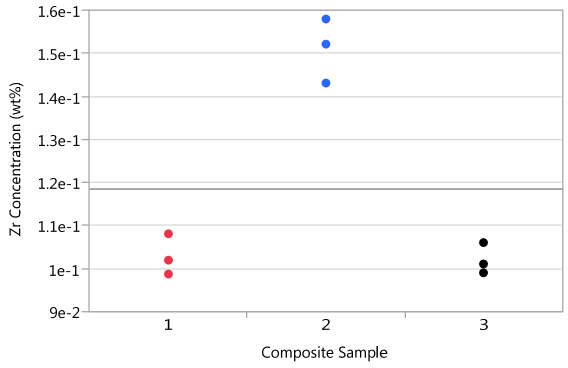
REML Mean = 3.3122E-2.

Student's t UCL95

UCL95	3.7184E-2

Zr (*wt %*)

All Results (circles) above MDC



Means and Standard Deviations

Sample	Number	Mean	Std Dev	Std Err Mean
1	3	1.0290E-1	4.7149E-3	2.7221E-3
2	3	1.5100E-1	7.5498E-3	4.3589E-3
3	3	1.0203E-1	3.5642E-3	2.0578E-3
Overall	9	1.1864E-1	2.4739E-2	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	0.7426	2	6	0.5150
Brown-Forsythe	0.4755	2	6	0.6431
Levene	0.8074	2	6	0.4892
Bartlett	0.4754	2		0.6217

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance (ANOVA)

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	4.7121E-3	2.3561E-3	76.8834	<.0001
Error	6	1.8387E-4	3.0644E-5		
C. Total	8	4.8960E-3			

Statistically significant sampling variance.

Variance Components

Component	Variance Comp	Std Dev
Composite Samples	7.7513E-4	2.7841E-2
Measurements	3.0644E-5	5.5357E-3
Total	8.0578E-4	2.8386E-2
Mean Concentration	2.6178E-4	1.6180E-2

REML Mean = 1.1864E-1.

Student's t UCL95

UCL95	1.6589E-1

In the "ANOVA" table, DF: degrees of freedom; SS: Sums of Squares; MS: Mean Squares. In the "Tests that the Measurement Variances are Equal" table, DFNum: Numerator DF; DFDen: Denominator DF for the F Ratio.

Appendix E-18: Statistical Summary for the Anions with All Measurements above-MDC

Constituent	N	Mean (wt %)	Std Dev (wt %)	% Std Dev	UCL95 (wt %)	Remarks
Chloride Cl^{-1}	9	1.6211E-2	1.1005E-3	6.79%	1.6893E-2	SNS-VH; SNS-SV; SNS-DT; SNS-SW; Student's t Confidence Limit (8 DF)
Nitrate NO_3^{-1}	9	1.4622E-2	1.7810E-3	12.18%	1.5726E-2	SNS-VH; SNS-SV; SNS-DT; SNS-SW; Student's t Confidence Limit (8 DF)
Nitrite NO_2^{-1}	9	5.7833E-3	2.6691E-3	46.15%	1.0260E-2	SNS-VH; SS-SV; Student's t Confidence Limit (2 DF)
Sulfate SO_4^{-2}	9	7.8767E-2	2.6214E-2	33.28%	1.2158E-1	SNS-VH; SS-SV; Student's t Confidence Limit (2 DF)
Total Iodine	9	2.7200E-3	4.9490E-4	18.19%	3.0268E-3	SNS-VH; SNS-SV; SNS-DT; SNS-SW; Student's t Confidence Limit (8 DF)

MDC: Minimum Detectable Concentrations.
DF: degrees of freedom.
SS-VH (SNS-VH): Statistically significant (statistically non-significant) test for measurement error heterogeneity at $\alpha = 0.05/5=0.01$.
SS-SV (SNS-SV): Statistically significant (statistically non-significant) sampling variance at $\alpha = 0.05$.
SS-WS (SNS-WS): Statistically significant (statistically non-significant) Wilk-Shapiro test for normality at $\alpha = 0.05$ (used for models without a sampling variance).
SS-DT (SNS-DT): Statistically significant (statistically non-significant) Dixon test for an outlier at $\alpha = 0.05$ (used for models without a sampling variance).

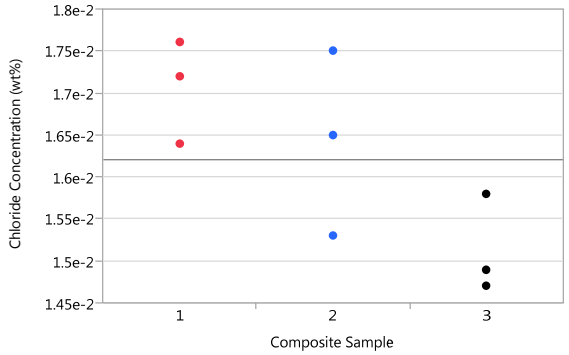
Appendix E-19: Statistical Summary for the Anions with All Measurements below-MDC

Anion Constituent (*wt %*)	N	Smallest Minimum Detectable Concentration (*wt %*)		Largest Minimum Detectable Concentration (*wt %*)	
		Fixed Decimal Format	Scientific Format	Fixed Decimal Format	Scientific Format
Fluoride F^{-1}	9	0.00293	2.93E-3	0.00306	3.06E-3
Phosphate PO_4^{-3}	9	0.00293	2.93E-3	0.00306	3.06E-3

Appendix E-20: Supporting Results for Anions

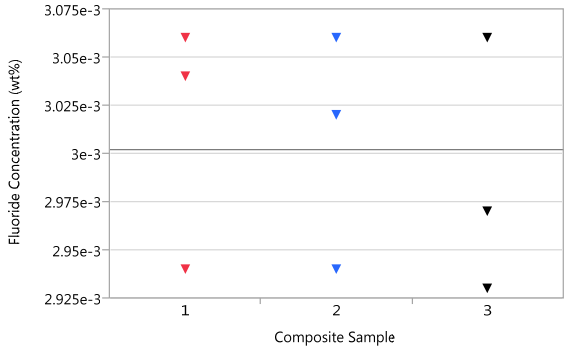
Chloride, Cl^{-1} (*wt %*)

All Results (circles) above MDC



Fluoride, F^{-1} (*wt %*)

All Results (triangles) below MDC



Means and Standard Deviations on Original Scale

Sample	Number	Mean	Std Dev	Std Err Mean
1	3	1.7067E-2	6.1101E-4	3.5277E-4
2	3	1.6433E-2	1.1015E-3	6.3596E-4
3	3	1.5133E-2	5.8595E-4	3.3830E-4
Overall	9	1.6211E-2	1.1005E-3	

Minimum Detectable Concentration (MDC)

Sample	Number	Minimum MDC	Maximum MDC
1	3	2.94E-3	3.06E-3
2	3	2.94E-3	3.06E-3
3	3	2.93E-3	3.06E-3
Overall	9	2.93E-3	3.06E-3

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	0.7522	2	6	0.5111
Brown-Forsythe	0.4644	2	6	0.6493
Levene	0.6035	2	6	0.5770
Bartlett	0.4399	2		0.6441

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance (ANOVA)

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	5.8289E-6	2.9140E-6	4.530	0.0632
Error	6	3.8600E-6	6.4330E-7		
C. Total	8	9.6889E-6			

Statistically non-significant sampling variance.

Diagnostics for Chloride Measurements

Shapiro-Wilk	P-Value=0.4333>α=0.05 ->SNS
Dixon High Outlier	Run 3 of Sample 1 -> SNS
Dixon Low Outlier	Run 1 of Sample 3 -> SNS

Student's t UCL95

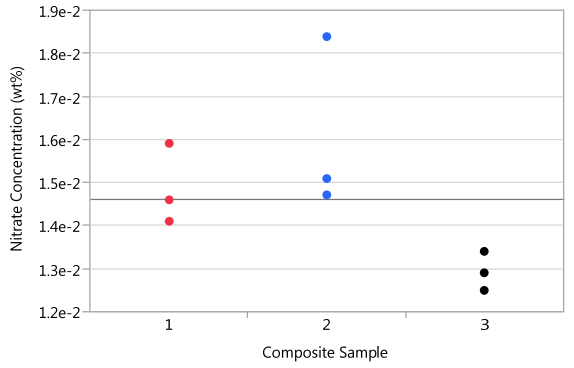
UCL95	1.6893E-2

In the "ANOVA" table, DF: degrees of freedom; SS: Sums of Squares; MS: Mean Squares. In the "Tests that the Measurement Variances are Equal" table, DFNum: Numerator DF; DFDen: Denominator DF for the F Ratio.
SNS: Statistically non-significant at $\alpha = 0.05$.

Appendix E-20: Supporting Results for Anions

Nitrate, NO_3^{-1} (*wt %*)

All Results (circles) above MDC



Means and Standard Deviations on Original Scale

Sample	Number	Mean	Std Dev	Std Err Mean
1	3	1.4867E-2	9.2916E-4	5.3645E-4
2	3	1.6067E-2	2.0306E-3	1.1724E-3
3	3	1.2933E-2	4.5092E-4	2.6034E-4
Overall	9	1.4622E-2	1.7810E-3	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	1.3207	2	6	0.3347
Brown-Forsythe	0.5459	2	6	0.6056
Levene	5.2091	2	6	0.0488
Bartlett	1.6098	2		0.1999

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance (ANOVA)

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	1.4996E-5	7.498E-6	4.334	0.0684
Error	6	1.0380E-5	1.730E-6		
C. Total	8	2.5376E-5			

Statistically non-significant sampling variance.

Diagnostics for Nitrate Measurements

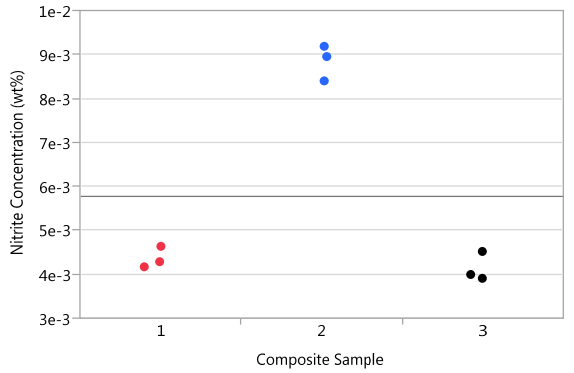
Shapiro-Wilk	P-Value=0.4187>α=0.05 ->SNS
Dixon High Outlier	Run 2 of Sample 2 -> SNS
Dixon Low Outlier	Run 2 of Sample 3 -> SNS

Student's t UCL95

UCL95	1.5726E-2

Nitrite, NO_2^{-1} (*wt %*)

All Results (circles) above MDC



Means and Standard Deviations on Original Scale

Sample	Number	Mean	Std Dev	Std Err Mean
1	3	4.3633E-3	2.3860E-4	1.3776E-4
2	3	8.8467E-3	4.0501E-4	2.3383E-4
3	3	4.1400E-3	3.3151E-4	1.9140E-4
Overall	9	5.7833E-3	2.3175E-3	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	0.3622	2	6	0.7104
Brown-Forsythe	0.1457	2	6	0.8674
Levene	0.6113	2	6	0.5733
Bartlett	0.2191	2		0.8032

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance (ANOVA)

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	4.2303E-5	2.1151E-5	191.782	<.0001
Error	6	6.6173E-7	1.1029E-7		
C. Total	8	4.2965E-5			

Statistically significant sampling variance.

Variance Components

Component	Variance Comp	Std Dev
Composite Samples	7.0137E-6	2.6483E-3
Measurements	1.1029E-7	3.3210E-4
Total	7.1240E-6	2.6691E-3
Mean Concentration	2.3502E-6	1.5330E-3

REML Mean = 5.8733E-2.

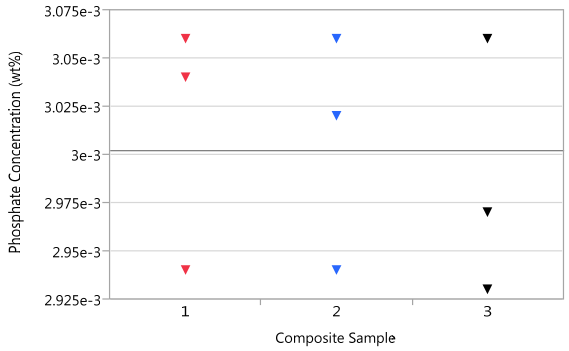
Student's t UCL95

UCL95	1.0260E-2

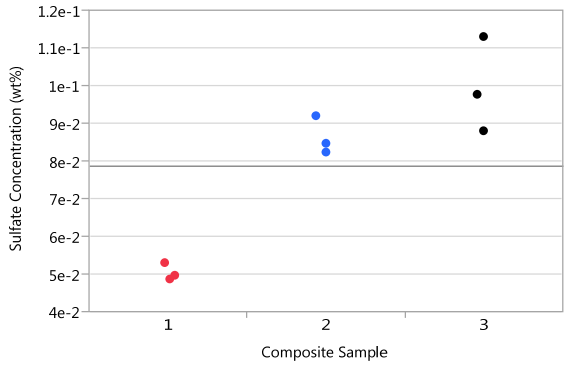
In the "ANOVA" table, DF: degrees of freedom; SS: Sums of Squares; MS: Mean Squares. In the "Tests that the Measurement Variances are Equal" table, DFNum: Numerator DF; DFDen: Denominator DF for the F Ratio.
SNS: Statistically non-significant at $\alpha = 0.05$.

Appendix E-20: Supporting Results for Anions

Phosphate, PO_4^{-3} (*wt %*)
All Results (triangles) below MDC



Sulfate, SO_4^{-2} (*wt %*)
All Results (circles) above MDC



Minimum Detectable Concentration (MDC)

Sample	Number	Minimum MDC	Maximum MDC
1	3	2.94E-3	3.06E-3
2	3	2.94E-3	3.06E-3
3	3	2.93E-3	3.06E-3
Overall	9	2.93E-3	3.06E-3

Means and Standard Deviations on Original Scale

Sample	Number	Mean	Std Dev	Std Err Mean
1	3	5.0467E-2	2.2301E-3	1.2875E-3
2	3	8.6267E-2	5.0362E-3	2.9077E-3
3	3	9.9567E-2	1.2643E-2	7.2994E-3
Overall	9	7.8767E-2	2.3048E-2	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	1.4404	2	6	0.3084
Brown-Forsythe	1.5434	2	6	0.2879
Levene	2.8390	2	6	0.1356
Bartlett	2.0753	2		0.1255
Statistically non-significant measurement variance heterogeneity.				

Analysis of Variance (ANOVA)

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	3.8693E-3	1.9347E-3	30.52	0.0007
Error	6	3.8036E-4	6.3393E-5		
C. Total	8	4.2497E-3			
Statistically significant sampling variance.					

Variance Components

Component	Variance Comp	Std Dev
Composite Samples	6.2376E-4	2.4975E-2
Measurements	6.3393E-5	7.9620E-3
Total	6.8715E-4	2.6214E-2
Mean Concentration	2.1496E-4	1.4662E-2

REML Mean = 7.8767E-2.

Student's t UCL95

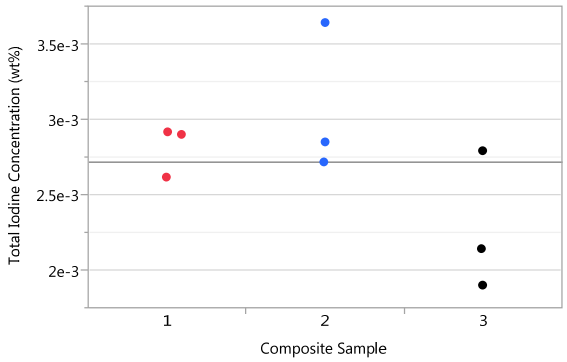
UCL95	1.2158E-1

In the "ANOVA" table, DF: degrees of freedom; SS: Sums of Squares; MS: Mean Squares. In the "Tests that the Measurement Variances are Equal" table, DFNum: Numerator DF; DFDen: Denominator DF for the F Ratio.

Appendix E-20: Supporting Results for Anions

Total Iodine (*wt %*)

All Results (circles) above MDC



Means and Standard Deviations on Original Scale

Sample	Number	Mean	Std Dev	Std Err Mean
1	3	2.8133E-3	1.6773E-4	9.6839E-5
2	3	3.0700E-3	4.9790E-4	2.8746E-4
3	3	2.2767E-3	4.6047E-4	2.6585E-4
Overall	9	2.7200E-3	4.9490E-4	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	0.6915	2	6	0.5367
Brown-Forsythe	0.3919	2	6	0.6919
Levene	2.3280	2	6	0.1785
Bartlett	0.8745	2		0.4171
Statistically non-significant measurement variance heterogeneity.				

Analysis of Variance (ANOVA)

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	9.8327E-7	4.91633E-7	3.022	0.1236
Error	6	9.7613E-7	1.62689E-7		
C. Total	8	1.9594E-6			
Statistically non-significant sampling variance.					

Diagnostics for Total Iodine Measurements

Shapiro-Wilk	P-Value=0.4207>α=0.05 ->SNS
Dixon High Outlier	Run 1 of Sample 2 -> SNS
Dixon Low Outlier	Run 3 of Sample 3 -> SNS

Student's t UCL95

UCL95	3.0268E-3

In the "ANOVA" table, DF: degrees of freedom; SS: Sums of Squares; MS: Mean Squares. In the "Tests that the Measurement Variances are Equal" table, DFNum: Numerator DF; DFDen: Denominator DF for the F Ratio.
SNS: Statistically non-significant at $\alpha = 0.05$.

Distribution:

G. C. Arthur, 241-284H
T. B. Brown, 773-A
J. R. Cantrell. 705-1C
T. L. Chandler, 241-162H
C. J. Coleman, 773-A
L. H. Connelly,773-A
G. R. Davis, 241-156H
D. P. DiPrete, 773-41A
K. D. Dixon, 705-1C
W. A. Drown, 773-42A
T. B. Edwards, 999-W
A. P. Fellinger, 773-42A
S. D. Fink, 773-A
C. M. Gregory, 773-A
M. S. Hay, 773-42A
C. C. Herman, 773-A
E. N. Hoffman, 999-W
P. R. Jackson, DOE-SR, 703-46A
M. H. Layton, 705-1C
M. J. Mahoney, 705-1C
S. L. Marra, 773-A
D. H. McGuire, 999-W
J. E. Occhipinti, 704-56H
L. N. Oji, 773-42A
J. P. Pavletich, 705-1C
F. M. Pennebaker, 773-42A
S. H. Reboul, 773-42A
A. R. Shafer, 766-H
E. P. Shine, 703-41A
R. O. Voegtlen, 241-162H
W. R. Wilmarth, 773-A
R. H. Young. 773-A
Records Administration (EDWS)