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Tank 16H Residual Sample Analysis Report

L. N. Oji
D. P. Diprete
C. J. Coleman
M. S. Hay
E. P. Shine

October 2014

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REVIEWS AND APPROVALS

AUTHORS:

L. N. Oji, Author, Advanced Characterization & Processing	Date
D. P. Diprete, Co-author, Analytical Development	Date
C. J. Coleman, Co-author, Analytical Development	Date
M. S. Hay, Co-author, Advanced Characterization & Processing	Date
E. P. Shine, Co-author, Analytical Development	Date

TECHNICAL REVIEW:

S. H. Reboul, Advanced Characterization & Processing *Reviewed per Manual E7 Procedure 2.60*	Date
T. B. Edwards, Process Technology Programs *Reviewed per Manual E7 Procedure 2.60*	Date

APPROVALS:

F. M. Pennebaker, Manager, Advanced Characterization & Processing	Date
R. S. Lee, Manager, Analytical Development -Statistics	Date
S. L. Marra, Manager, Environmental & Chemical Process Technology Research Programs	Date
M. J. Mahoney, Closure & Waste Disposal Authority	Date
J. E. Occhipinti, Manager, SRR Engineering, Waste Removal and Tank Closure	Date

LIST OF REVISIONS		
Revision Number	**Summary of Changes**	**Date**
0	None	August 2014
1	Analysis results for Tc-99 on Tank 16H Annulus Composite Sample 1, run 3 and Tank 16H Annulus Composite Sample 2 run 1 were corrected. Values should read, respectively, as 2.39E-01µCi/g and 2.04E-01 µCi/g. These changes will affect the averages and standard deviations for Tc-99 in Tables 24 and 25. Figure 4, Insert A: "As-received" Tank 16H Primary Liner sample picture was put back in position.	October 2014
1	Appendix D: Revised reference for Baker and others [2014] and references to Singh and Singh [2013]. Changes to table numbering.	October 2014
1	Appendix E: (Primary Vessel) Changes to section and table numbering. Change to remarks for I-129, fixed decimal format smallest MDC for Cs-137, and % std dev's for Eu-154 and Pu-240 in summary Table E16. Change to footnote of Table E19. Change to remarks in Table E20. Change to summary statistics for I-129 in Table E25.	October 2014
1	Appendix F: (Annulus) Changes to section and table numbering. Changes to measurements for Run 3 of Sample 1 and Run 1 for Sample 2 for Tc-99 and associated revisions to Tables F17 and F19. Change Dixon low outlier result to "SNS".	October 2014

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The author extends thanks to the multiple members of the SRNL Shielded Cells and AD organization members who processed the samples and provided analytical results, specifically, L. Bush, K. Reid, C. Black, R. Sullivan, M. Lee, M. Jones, C. Diprete and L. Brown.

EXECUTIVE SUMMARY

Characterization Summary

The Savannah River National Laboratory (SRNL) was requested by Savannah River Remediation (SRR) to provide sample preparation and analysis of the Tank 16H final characterization samples to determine the residual tank inventory prior to grouting. Three Primary Liner Tank 16H residual samples from areas on the floor of the tank and eleven residual Tank 16H Annulus sample were collected and delivered to SRNL between May and November of 2013. Four Tank 16H Annulus samples previous collected in 2011 were also included in the batch of Tank 16H samples for processing and eventual characterization for a total of fifteen annulus samples.

The fifteen Tank 16H Annulus samples were homogenized and combined into three composite samples based on a proportional compositing scheme and the resulting composite samples along with each of the three discrete Primary Liner samples taken from the floor of the tank interior were analyzed for radiological, chemical and elemental components. Additional measurements performed on the Tank 16H Annulus composite samples and Primary Liner Samples include bulk density and water leaching of the solids to account for water soluble components. In general, these analyses were performed and reported in triplicate where possible.

Sufficient standards and blanks were utilized to demonstrate adequate quality assurance for the characterization of the Tank 16H samples as specified in the technical task request document. While many of the target detection limits were met for the species characterized for Tank 16H (Primary Liner and Annulus composite samples) some were not met. The isotopes whose target detection limits were not met in all cases for both the Primary Liner and Annulus composite Tank 16H samples included non-routine analytical species like Zr-93, Cl-36, Pa-231 and Cm-244. For these four radionuclides the detection limits were at least one or two orders of magnitude higher than the target detection limits.

Several of these radionuclide detection limits, especially for the routine analytes in the Tank 16H Primary Liner samples, were not met because the samples themselves did not contain quantifiable amounts of reference materials, such as U-238, from which the radionuclides could be back calculated. Thus, most of these radionuclide analytical results were near their detection limits or about the same order of magnitude as those of the target detection limit.

SRNL, in conjunction with the customer, reviewed all of these cases and determined that the impacts of not meeting the target detection limits were acceptable. The target detection limits for most of the routine radionuclides were met most of the time.

Statistical Review Summary

A statistical analysis of the Tank 16H samples from the primary vessel and the annulus has been completed. Analytes with all less-than-MDC (minimum detectable concentrations) were summarized by their minimum and maximum MDC's. Analytes with measurements on only a single sample were also summarized in the same fashion. Analytes with measurements on at least two of the three samples were summarized by their mean, standard deviation, percent standard deviation, and their 95% upper confidence limit (UCL95) for the mean concentration.

A significant feature of the sample data was the distinct difference between many of the radionuclide concentrations for Sample 2-P and those for Samples 1-P and 3-P. Nearly all of the radionuclide concentration results for Sample 2-P were reported to be less-than-MDC's. Since no discernible statistical distribution could be identified for the radionuclide concentrations, a conservative nonparametric UCL95 was established for all analytes that fell within this class.

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LIST OF ABBREVIATIONS

AD	Analytical Development
COC	Chain-of-Custody
CVAA-Hg	Cold Vapor Mercury by Atomic Absorption
DNR	Data not reported
DWPF	Defense Waste Processing Facility
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
NCSA	Nuclear Criticality Safety Assessment
PHA	Pulse Height Analysis
SRNL	Savannah River National Laboratory
SRR	Savannah River Remediation
TTA	Thenoyltrifluoroacetone
TTQAP	Task Technical and Quality Assurance Plan
TTR	Technical Task Request
XRD	X-Ray Diffraction
XRF	X-Ray Fluorescence

1.0 INTRODUCTION

Savannah River Remediation LLC (SRR) is preparing Tank 16H for closure. The Savannah River National Laboratory (SRNL) was requested by SRR to provide sample preparation and analysis of the Tank 16H final characterization samples for use in determining the Tank 16H residuals inventory. In all, three Tank 16H Primary Liner samples and fifteen Tank 16H Annulus samples were provided by SRR. Figure 1 shows the location of Tank 16H relative to other H-Area tanks. Figure 2 shows Tank 16H primary tank risers (access ports into the tank) and the targeted initial and alternate sampling locations.

Tank 16H Primary Liner and Annulus sample locations for composite sample creation are shown in Figures 2 and 3, respectively. Photographs of some of the "as-received" Tank 16H Primary Liner samples and the Annulus samples are shown in Figure 4 (insert A), Figure 5 (all inserts) and Figure 6 (inserts A and C). With four Tank 16H samples (16-W-1, 16-E-2, 16-N-1, 16-S-2,) being the exceptions, the other 11 Tank 16H Annulus samples (1-AD, 2-A, 3-A, 4-AD, 5-AD, 6-AR, 7-AD, 8-AR, 9-AD, 10-A ,11-AD) were collected by SRR and made available to SRNL between August 2013 and November 2013. The other four Tank 16H Annulus samples were collected and delivered to SRNL in the 2011 Tank 16H sample campaign. The acceptability of these 2011 samples for use in composite sample creation was determined and documented in the Tank 16H Sample Location Determination Report[i]. n all, these two sample sets (eleven annulus samples collected in 2013 and the four archived 2011 samples) formed the basis for designing the three Tank 16H Annulus composite sample materials.

In designing the three Tank 16H Annulus composite sample materials, the volume of residual material in each of the Tank 16H Annulus regions 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 annulus sample for each composite sample creation[ii]. This is based on the methodology described in the Liquid Waste Tank Residuals Sampling and Analysis Program Plan (LWTRSAPP).[iii] Thus, each of the three Tank 16H Annulus composite samples was derived from five individual Tank 16H annulus materials as summarized in Tables 1 and 6; with each composite representing the entire tank. Hence, a complete characterization of the Tank 16H residuals involves analytical data from the three Tank 16H Primary Liner Samples and the three Tank 16H Annulus composite samples (Tank 16H-Composite sample # 1, Tank 16H- Composite sample # 2 and Tank 16H- Composite sample # 3).

The Tank 16H Primary Liner Samples and the three Tank 16H Annulus composite samples were analyzed in accordance with Technical Task Request (TTR) provided by SRR,[iv] Task Technical and Quality Assurance Plan (TTQAP) for the Analysis of the Tank 16H[v] and Tank 16H Sampling and Analysis Plan[vi] and the Liquid Waste Tank Residuals Sampling – Quality Assurance Program Plan (LWTRQAPP).[vii]

2.0 TANK 16H SAMPLE RECEIPT AND PREPARATIONS FOR CHARACTERIZATION

2.1 Primary Liner Tank 16H samples

Tank 16H Primary Liner samples (1-P, 2-P and 3-P) were collected by SRR and delivered to SRNL between May 14 and May 23, 2013 (See attached COC forms in the Appendix C and Table 1). The planned Primary Liner sampling locations are shown in Figure 2. The weights of the "as-received" Primary Liners samples (1-P, 2-P and 3-P) were 253 g, 272 g and 155 g, respectively. Thus, adequate material was collected from the primary liner locations and it was not necessary to collect additional samples. The three samples were fairly dry and did not require extra air-drying inside the SRNL shielded cells. The "as-received" Tank 16H Primary Liner sample bulk densities and weight percent solids were determined as described in Appendix B. The "as-received" bulk densities and weight percent solids for the Tank 16H Primary Liner samples, as described in Appendix B, are provided in Table 2. Each Tank

16H Primary Liner sample was then homogenized to reduce the particle size. 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 go pass through the first time were re-ground with mortar and pestle until the particles were small enough to pass through the sieve.

Not all of the materials in the Primary Liner sample 2-P (14.7 grams of the total 272 grams of sample 2-P) could be ground fine enough to pass through the 850 micron sieve. With the insertion of a new magnetic stirring bar into the container with this small fraction of sample 2-P, it was observed that the magnet attracted a significant amount of the solid material from sample 2-P, as shown in Figure 4 insert C. This iron-rich fraction of sample 2-P is designated as the "magnetic fraction". After discussing the nature of the iron-rich fraction with SRR personnel, it was decided that the magnetic fraction would be digested and analyzed separately for Cs-137, gross alpha/beta and elemental analysis by Inductively Coupled Plasma–Atomic Emission Spectroscopy (ICP-ES). The results of the analysis would then be evaluated and compared with those of the regular sample 2-P to determine if additional analysis on sample 2-P material would be necessary to evaluate any impacts on final Tank 16H inventory.[viii] As described later in this report, both the parent 2-P sample and the magnetic fraction were similar in chemical composition.

The homogenized bulk density and weight percent solids for each sample were also determined and are presented in Table 2.

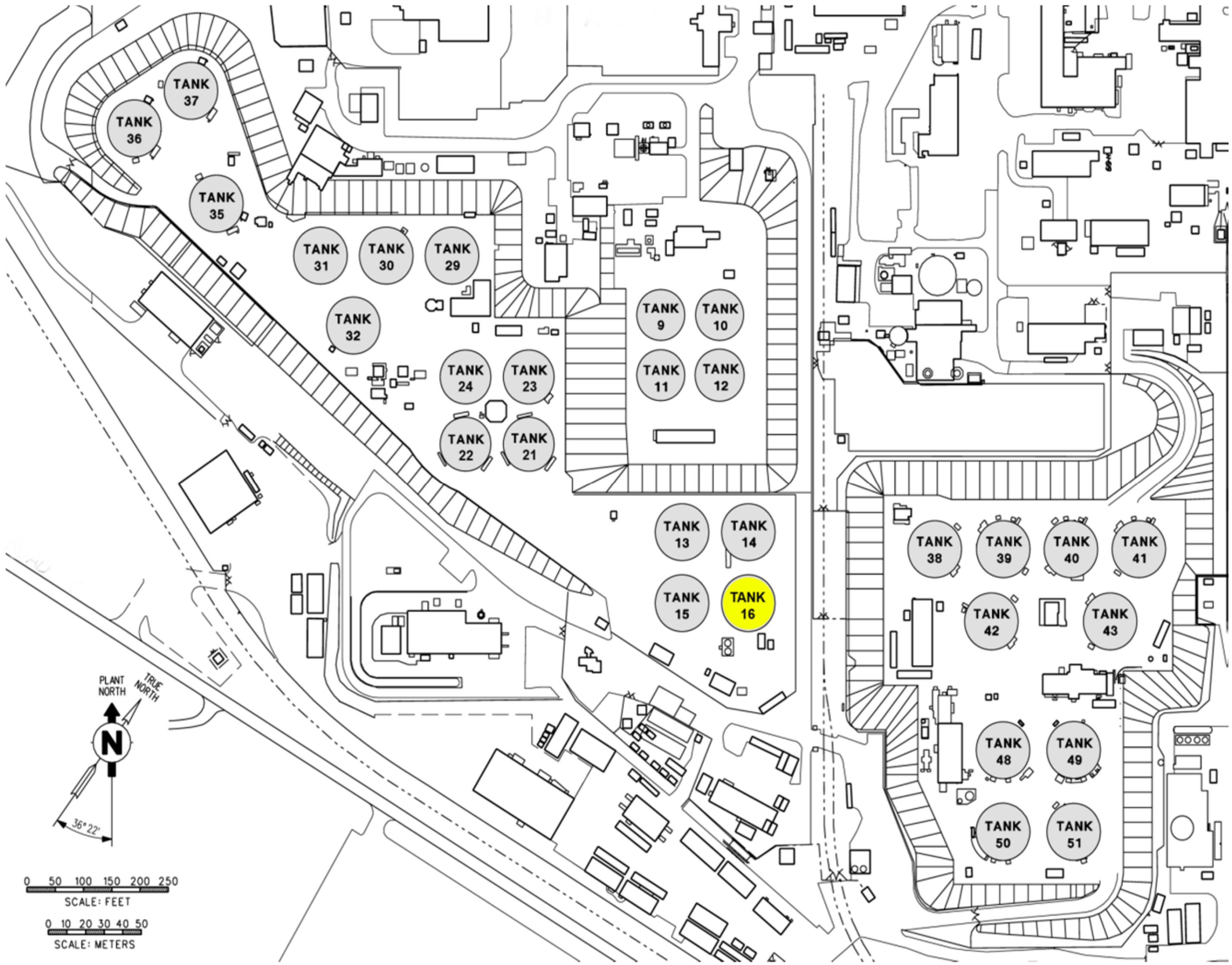

Figure 1 Location of Tank 16H in the H-Area Tank Farm

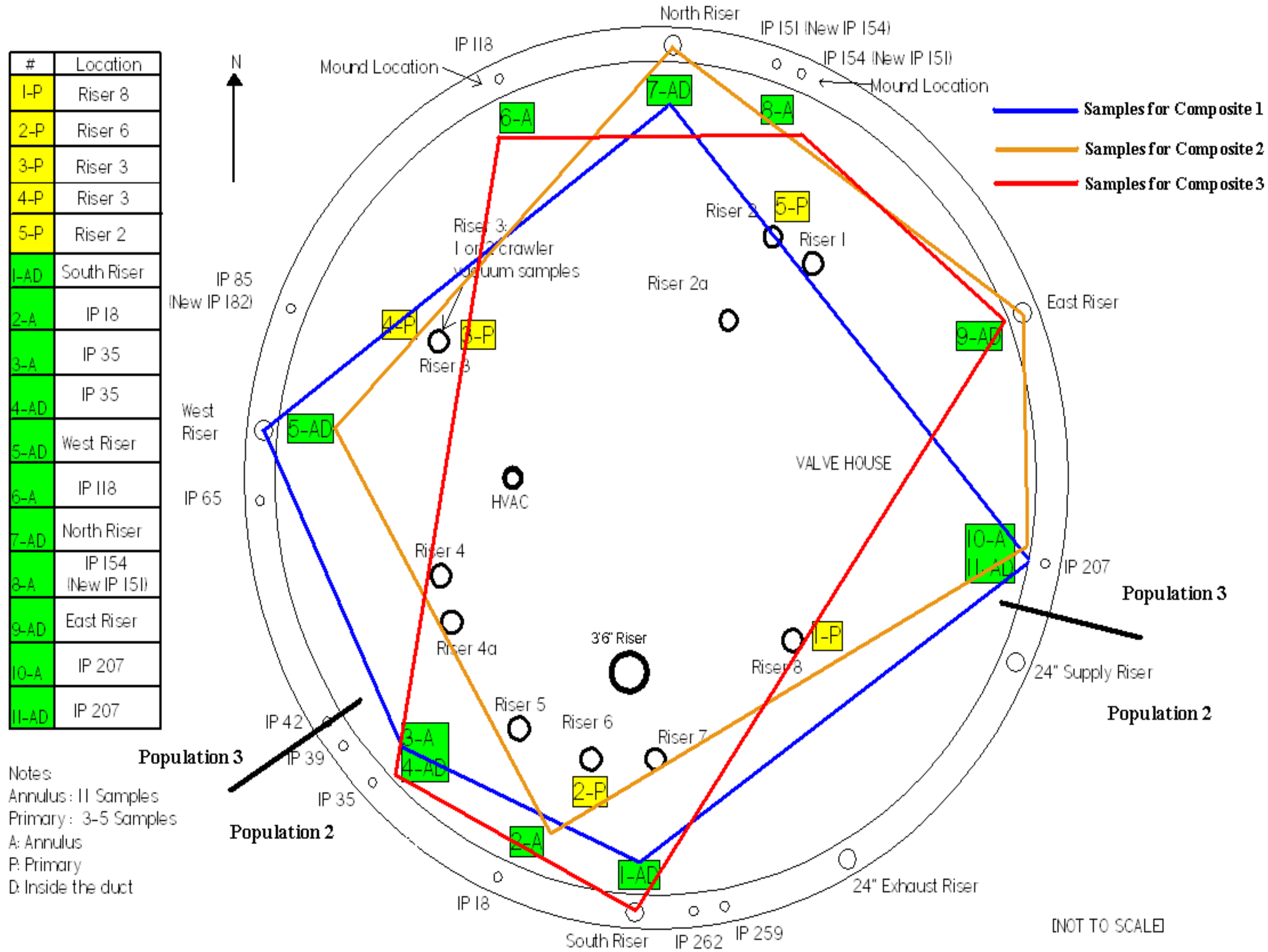

Figure 2 Locations of Tank 16H Primary Liner and 2104 Annulus Samples

#	Location
1-P	Riser 8
2-P	Riser 6
3-P	Riser 3
4-P	Riser 3
5-P	Riser 2
1-AD	South Riser
2-A	IP 18
3-A	IP 35
4-AD	IP 35
5-AD	West Riser
6-A	IP 118
7-AD	North Riser
8-A	IP 154 (New IP 151)
9-AD	East Riser
10-A	IP 207
11-AD	IP 207

Notes:
Annulus: 11 Samples
Primary: 3-5 Samples
A: Annulus
P: Primary
D: Inside the duct

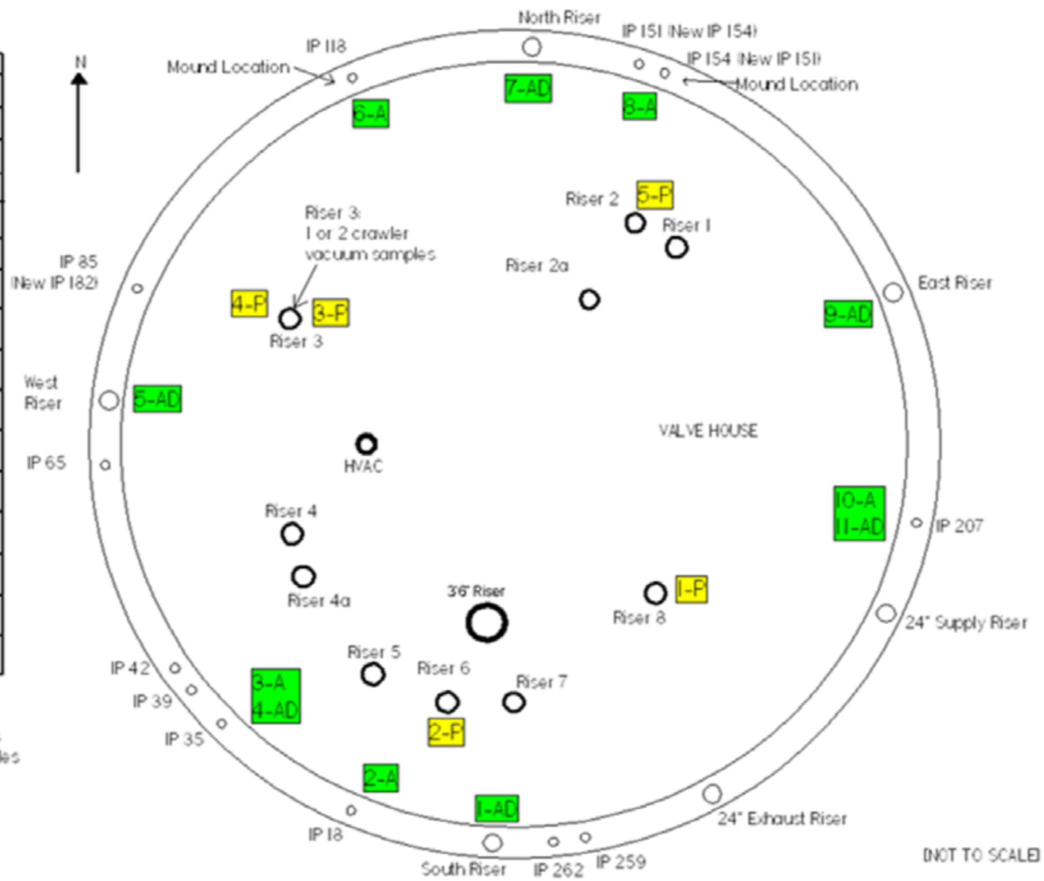

Figure 3 Tank 16 Annulus Sample Compositing Arrays

Table 1 Summary information on Tank 16H Primary Liner Sample and Annulus Sample Delivery to SRNL

Sample ID	Sample Location Riser (See Figs. 2 & 3)	Date Sample Received at SRNL	As-received weight, g	*Weight of Air-dried Sample, g
1-P	Riser 8	5/14/13	253	NA
2-P	Riser 6	5/21/13	272	NA
3-P	Riser 3	5/29/13	155	NA
1-AD	South Riser	8/19/13	NA	77.1
2-A	IP-18	8/19/13	NA	134.9
3-A	IP-35	8/19/13	NA	233.1
4-AD	IP-35	8/19/13	NA	77.8
5-AD	West Riser	8/19/13	NA	45.1
6-AR	IP-118	11/21/13	NA	292
7-AD	North Riser	8/27/13	NA	50.7
8-AR	IP-151 (Old IP-154)	11/21/13	NA	43.4
9-AD	East Riser	8/27/13	NA	142
10-A	IP-207	8/27/13	NA	72.4
11-AD	IP-207	8/27/13	NA	55.4
16-W-1	West Riser	12/01/2011	NA	124.4
16-E-2	East Riser	11/07/2011	NA	40.0
16-N-1	North Riser	11/14/2011	NA	52.6
16-S-2	South Riser	11/21/2011	NA	100.6

*Final weight reported after air drying of some samples. The last four samples in above table were leftover samples from the 2011 Tank 16 sample campaigns.

Table 2 Tank 16H Primary Liner Sample: Bulk Density (As-received), Homogenized Sample Bulk Density and Homogenized Sample Weight Percent Solids

Sample Description	TK 16 1-P	TK 16 1-P	TK 16 1-P	Average	Stdev.
	Run 1	Run 2	Run 3		
"As -received" Bulk density, g/mL	1.20	1.32	1.21	**1.24**	*0.07*
Homogenized bulk density, g/mL	1.81	1.69	1.74	**1.75**	*0.06*
Air dried and Homogenized, wt%	99.75	99.75	99.65	**99.72**	*0.06*
	TK 16 2-P	**TK 16 P-2**	**TK 16 2-P**	**Average**	**Stdev.**
	Run 1	Run 2	Run 3		
"As -received" Bulk density, g/mL	1.43	1.35	1.36	**1.38**	*0.04*
Homogenized bulk density, g/mL	1.88	1.52	1.62	**1.67**	*0.18*
Air dried and Homogenized, wt%	99.25	98.90	99.35	**99.17**	*0.24*
	TK 16 3-P	**TK 16 3-P**	**TK 16 3-P**	**Average**	**Stdev.**
	Run 1	Run 2	Run 3		
"As -received" Bulk density, g/mL	1.37	1.08	1.22	**1.22**	*0.14*
Homogenized bulk density, g/mL	1.50	1.52	1.65	**1.56**	*0.08*
Air dried and Homogenized, wt%	99.50	98.85	99.40	**99.25**	*0.35*
10% reference NaCl solution wt%	10.20	10.08	10.16	**10.15**	*0.06*

Insert A: “As-received” Tank 16H Primary Liner samples

Insert B: Homogenized Tank 16H Primary Liner samples

No sample photo images in this quadrant.
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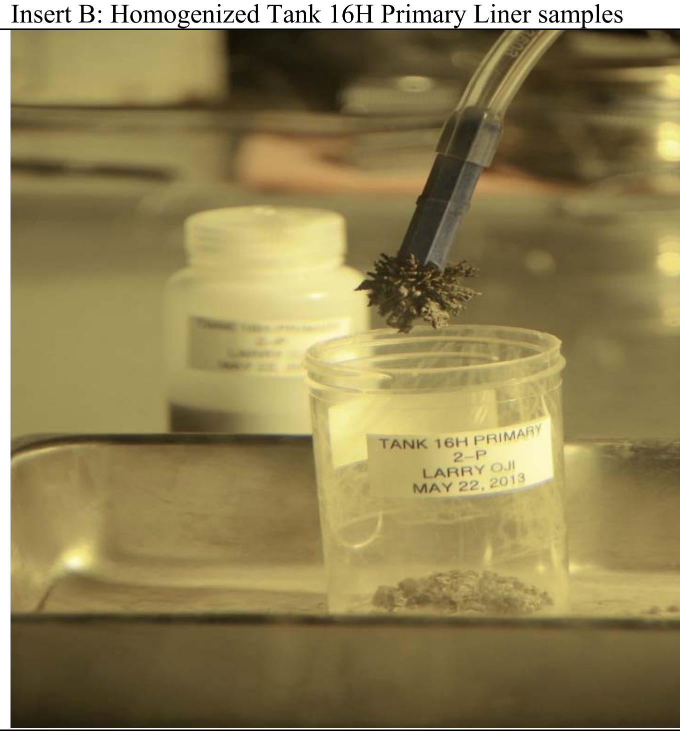


Insert C: Fraction of 2-P sample was iron-rich and magnetic

Figure 4 Tank 16H Primary Liner Samples

2.2 <u>Tank 16H Annulus Samples</u>

The sample identification and weight of air-dried Tank 16H Annulus samples are shown in Table 1. As mentioned above, the 15 Annulus samples included 4 samples from the 2011 sampling of Tank 16H.[ix] The eleven Tank 16H Annulus samples collected by SRR were delivered to SRNL between August 13 and November 18, 2013 (See attached COC forms in the Appendix C and Table 1). Initial Tank 16H Annulus sampling for samples 6-A and 8-A did not provide measurable sample quantities, so the locations were resampled and identified as samples 6-AR and 8-AR. Samples were collected using commercially available vacuum cleaners modified for this purpose.

The samples were opened in the SRNL shielded cell and the determination of the bulk densities and weight percent solids of the "as-received" materials was initiated immediately (within an hour) after opening and retrieving the sample from the vacuum used for sample collection. For sample materials that were not easily retrieved because of wet, gooey and pasty material conditions or when insufficient material was retrieved, the bulk density and/or the weight percent solids for the "as-received" sample were not determined. However, after opening the sampling vessel and exposing it to air for several days, it became possible to retrieve enough sample material. Figure 5 shows pictures of samples in the vacuum cleaner and vacuums which did not contain sample material. For these air-dried samples, the physical parameters determined were labeled as air-dried parameters.

After air-drying in the shielded cell, each Tank 16H Annulus sample was then homogenized to reduce the particle size. As described earlier, 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. The bulk density of each homogenized sample was determined followed by the blending of proportional amounts of the samples by weight to form the three Tank 16H Annulus composite samples. The bulk density of each of the three composite samples was then determined by the process described in Appendix B. A reference simulant sludge material sample, based on Tank 8 sample chemistry [See Appendix A-3], was air-dried in a clean laboratory and the resulting sludge cake ground and homogenized with a mortar and pestle. The bulk density of this ground reference Tank 8 sludge was determined inside the shielded cell along with the Tank 16H Annulus samples.

The "as-received" bulk density values may in some cases have large uncertainty values. Problems were encountered in determining the volumes of these samples because some of the samples contained large chunky pieces, which made it difficult to accurately measure the volumes. In summary, Table 3 contains the "as-received" bulk density and "as-received" weight percent solids where available, while Table 4 shows the air-dried sample bulk density, air-dried weight percent solids and homogenized bulk density- of the Tank 16H Annulus samples. The Tank 16H Annulus composite sample bulk density and weight percent solids are presented in Table 5. The compositing specifications for the three Tank 16H composite samples and their mass proportions are summarized in Table 6. Tables 3 and 4 contain the physical parameters (bulk densities and weight percent solids) for the fifteen Tank 16H Annulus samples from both the 2011 and 2013 sample collection campaigns. Samples for the 2011 sampling period include sample ID's 16-W-1, 16-E-2, 16-N-1 and 16-S-2. All other annulus samples, as shown in each table, come from the 2013 sampling period.

Figure 6 shows photographs of select "as-received" (inserts A and C) and homogenized Tank 16H Annulus samples (inserts B and D). Because of the inherent risk of cross-contamination of these samples in the shielded cells environment, actions taken to control cross-contamination in the cell included wiping down the cell (cell decontamination), covering the entire cell floor with clean stainless steel plates, changing manipulator fingers prior to initiating work and changing out the oven shelves.

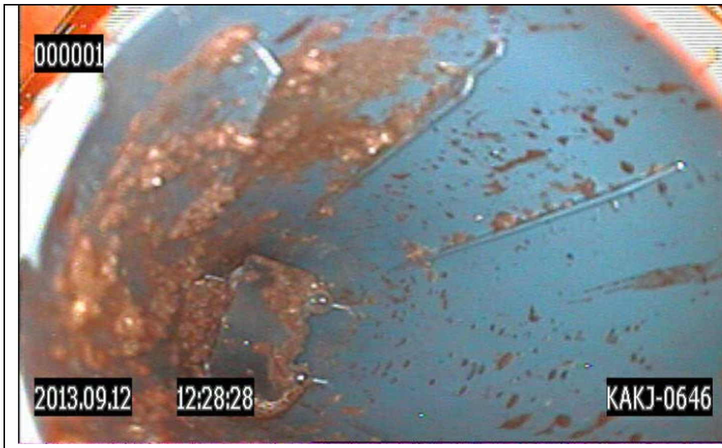
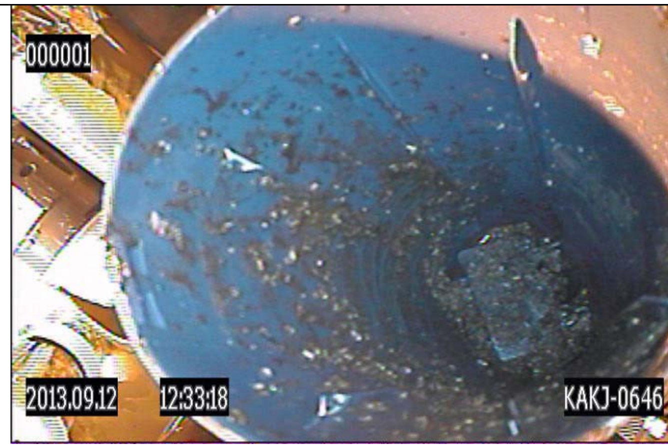
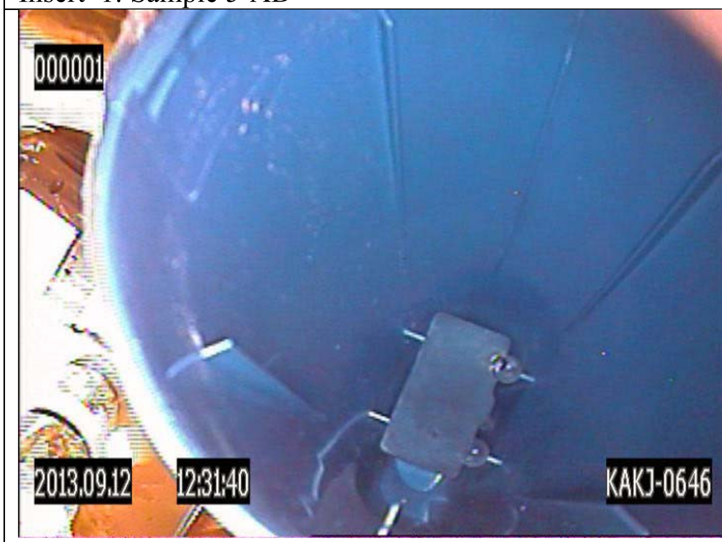
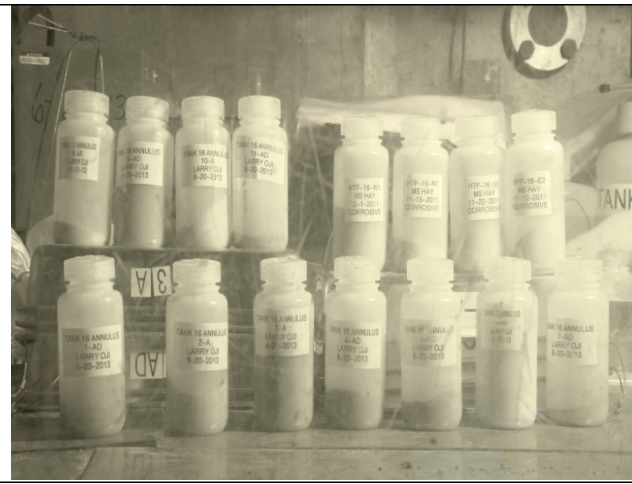
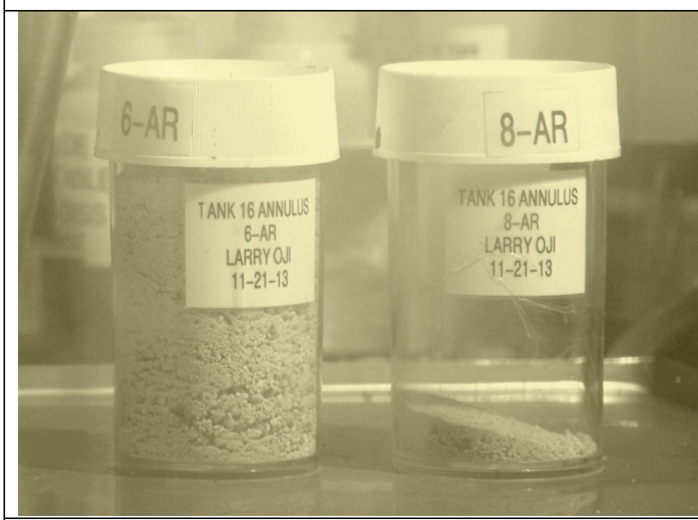
	
Insert 1: Sample 5-AD	Insert 2: Sample 11-AD
	Intentionally left blank. No picture images here.
Insert 3: Empty sample container; 8A	

Figure 5 Wet and pasty sample materials in the sample collection vacuums and empty sample collection vacuum 8A.

Insert A: Picture of Select Tank 16H Annulus samples



Insert B: Picture of all 15 -homogenized and sieved Tank 16H Annulus samples.



Insert C: Picture of Tank 16H Annulus samples 6 and 8

Insert D: Homogenized Tank 16H Annulus Composite samples

Figure 6 Photos of select "as-received" (inserts A and C) and homogenized Tank 16H Annulus samples (inserts B and D)

Table 3 "As-Received" Bulk Density and Weight Percent Solids: Tank 16H Annulus samples

	"As-Received" Bulk Density		"As-Received" wt% Solids	
Sample ID	**Average, mL/g**	Stdev.	**Average wt% solids**	Stdev.
1-AD	**1.06**	*0.07*	**75.9**	*1.0*
2-A	**0.71**	*0.02*	**70.3**	*1.9*
3-A	**0.76**	*0.01*	**71.4**	*1.0*
4-AD	**None***	*NA*	**None***	*NA*
5-AD	**None***	*NA*	**73.2**	*0.1*
6-AR	**1.01**	*0.06*	**77.7**	*1.2*
7-AD	**None***	*NA*	**None***	*NA*
8-AR	**1.35**	*0.03*	**94.3**	*0.9*
9-AD	**1.12**	*0.04*	**81.1**	*0.7*
10-A	**0.94**	*0.05*	**85.7**	*0.9*
11-AD	**None***	*NA*	**None***	*NA*
16-W-1	**None****	*NA*	**None****	*NA*
16-E-2	**None****	*NA*	**None****	*NA*
16-N-1	**None****	*NA*	**None****	*NA*
16-S-2	**None****	*NA*	**None****	*NA*

*Insufficient initial sample extracted from the vacuum sample holder.
** 2011 sample: No "as-received" sample data for bulk density and wt. % solids.

Table 4 Air-dried Sample Bulk Density, Air-dried Weight Percent Solids and Homogenized Bulk Density- Tank 16H Annulus Samples

	Air-dried bulk density		Air-dried Wt% Solids		Homogenized Bulk Density	
Sample ID	Average, mL/g	Stdev.	Average wt% solids	Stdev.	Average, mL/g	Stdev.
1-AD	1.04	0.04	80.3	2.2	0.93	0.01
2-A	0.86	0.03	78.2	1.3	0.94	0.01
3-A	0.92	0.04	76.3	1.1	0.88	0.01
4-AD	0.94	0.04	82.9	1.4	0.84	0.01
5-AD	1.08	0.04	81.2	1.4	0.84	0.01
6-AR	0.74	0.02	90.7	1.8	0.99	0.01
7-AD	1.00	0.01	81.9	0.6	0.96	0.01
8-AR	1.17	0.02	95.9	3.9	1.29	0.02
9-AD	1.04	0.01	83.6	1.2	0.91	0.01
10-A	1.02	0.04	85.0	2.8	0.88	0.01
11-AD	1.04	0.03	84.6	0.8	0.96	0.01
16-W-1	1.01	0.01	83.8	0.9	0.86	0.01
16-E-2	0.81	0.02	89.9	0.6	0.72	0.01
16-N-1	1.08	0.03	90.1	0.8	0.88	0.01
16-S-2	1.12	0.01	91.6	0.8	1.05	0.01

Table 5 Tank 16H Annulus Composite Sample Bulk Density and Weight Percent Solids

Composite Sample ID	Bulk Densities, g/mL					Weight percent solids, wt%				
	Run 1	Run 2	Run 3	Ave.	Stdev.	Run 1	Run 2	Run 3	Ave.	Stdev.
Tk 16 Annulus Composite # 1	1.08	1.03	1.04	1.05	0.03	92.1	92.0	87.1	90.4	2.9
Tk 16 Annulus Composite # 2	0.90	0.97	0.95	0.94	0.04	86.3	81.9	85.0	84.4	2.3
Tk 16 Annulus Composite # 3	0.89	0.90	0.93	0.91	0.02	88.2	88.2	83.5	86.6	2.7
5% Reference NaCl solution	NA	NA	NA	NA	NA	5.1	4.7	4.8	4.9	0.2

Table 6 Compositing Specifications and Mass Proportions for Tank 16H Annulus Samples

Composite Number	Sample Identification	Proportion of Composite Mass (%)	Material weight needed for generating 70 g of composite material (g)	Amount of material weighed out for each sample (g)
Tank 16 Annulus Composite No. 1	4-AD	10.04	7.03	7.040
	11-AD	11.10	7.77	7.785
	South Riser[a] (16-S-2)[b]	30.13	21.09	21.089
	West Riser[a] (16-W-1)[b]	22.57	15.80	15.773
	8-AR	26.15	18.31	18.307
Tank 16 Annulus Composite No. 2	1-AD	12.80	8.96	8.965
	7-AD	12.30	8.61	8.595
	2-A	30.44	21.31	21.297
	6-AR	21.23	14.86	14.861
	East Riser[a] (16-E-2)[b]	23.23	16.26	16.280
Tank 16 Annulus Composite No. 3	5-AD	11.80	8.26	8.257
	9-AD	11.41	7.99	7.988
	3-A	25.30	17.71	17.732
	North Riser[a] (16-N-1)[b]	26.47	18.53	18.536
	10-A	25.01	17.51	17.510

[a]These samples were collected in 2011 and are currently stored at SRNL. [b]Corresponding Tank 16H sample ID's. [9]

2.3 Blank Evaluations and Reference Materials

In addition to reagent blanks used by the SRNL Analytical Development (AD) Group, two types of reference matrices were used during the characterization of Tank 16H samples. The first reference material was an analyzed reference glass (ARG)[x] which was stored outside the shielded cells but processed in the shielded cells along with the samples during sample preparations. The second was an out of the cell air-dried Tank 8 simulant sludge[xi] which was exposed to the shielded cell radiological environment in which the Tank 16H radionuclide material was processed prior to analysis. The elemental chemical composition of the Tank 8 simulant sludge and ARG are presented in Appendices A-3 and A-4.

Acidified (dilute nitric acid) distilled and de-ionized water was 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 16H samples, an in-cell reference Tank 8 simulant sludge sample in a 250-mL capacity poly-bottle was placed at a strategic location in the shielded cell to ensure that the reference sample were exposed to the same cell environments as the Tank 16H samples. The simulant sludge reference sample container held about 20 grams of Tank 8 simulant sludge. The simulant container was opened when the Tank 16H 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 16H sample preparations or digestion (air-drying, aqua regia and peroxide fusion digestions), the Tank 8 simulant sludge reference material was also prepared in the same manner as the preparation of Tank 16H samples and submitted for the same analyses as the actual samples from Tank 16H.

2.4 Leaching Characterization of Tank 16H Solids

Known quantities of homogenized Tank 16H Annulus composite solids and Tank 16H Primary Liner samples were leached with distilled and de-ionized water and analyzed in triplicate. An average of 1.0 ± 0.04 grams of the composite solids was leached with an average of 30.01 ± 0.01 grams of distilled and de-ionized water. 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.

2.5 Analytical narratives and unforeseen events which may have affected Tank 16H sample characterization.

Unforeseen activities which may have negatively impacted the characterization protocols for Tank 16H include the following:

- Tank 16H Primary Liner Sample 2-P contained about 5.5%, by weight, iron-rich fraction which could not be easily ground homogenized and sieved as others. After discussing the nature of the iron-rich fraction with SRR personnel, it was decided by SRR that the iron-rich fraction (magnetic fraction) would be digested and analyzed separately for Cs-137, gross alpha/beta and elemental analysis by ICP-ES and the results of the analysis evaluated and compared with those of the regular sample 2-P to determine if additional analysis on sample 2-P material would be necessary to evaluate any impacts on the final Tank 16H inventory.[v]

- The Tank 16H Primary Liner samples, especially sample 2-P, did not show much activity for the radionuclides being characterized. The lack of typical elements utilized during radiochemical separations limited the ability to detect lower activities. Additionally, since the level of trace common radionuclides in the blanks, such as U-238, was comparable to their levels in the actual samples meeting the target detection limits for these radionuclides in the samples became more challenging.

- All work related to Tank 16H characterizations were ordered stopped by SRR for about two weeks in October 2013 due to funding constraints and SRR furlough. As a result of the suspension, samples preparation activities such as Am/Cm and peroxide fusion digestions were discarded, and restarted latter. All AD and Shielded Cell sample processing and analysis were also stopped and later re-started.

The presence of mass 93 impurity material in the Tank 8 simulant sludge posed a unique problems when analyzing for Zr-93 using this reference material. It became difficult to accurately measure for the Zr-93 activity by Inductively Coupled Plasma – Mass Spectrometry (ICP-MS).

A summary of issues that arose from sample matrices of Tank 16H characterization is presented in Appendix B1.

3.0 RESULTS

Laboratory analyses were performed on three discrete samples from the floor of Tank 16H and three composite samples generated from fifteen tank annulus samples. A combination of routine dissolution/measurement techniques and "tailor-made" digestion/isolation/analysis methods were used to quantify twenty-seven stable constituents and thirty-nine radionuclides.

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. See individual LIMS reports for analysis completion dates. Details of most of the analytical methodologies including weight percent solids and density determinations applied in Tank 16H sample characterizations are summarized in Appendix B. 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 16H residual sample characterization results presented below, values preceded by "<" (less than sign) indicate values were below minimum detection limits, 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 or method detection 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 preceding the average value. The standard deviation values were calculated only for values that were 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 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 or upper limit values as indicated by the analytical method. For example, under the one sigma percent uncertainty column for the isotope Co-60 in Table 24, the 23.3/MDA designation implies that the one sigma percent uncertainty for Co-60 in run 1 is reported with values above the detection limit and thus has a one sigma percent uncertainty of 23.3 percent. The measurements (runs 2 and 3) for Co-60 which were below the detection limit are assigned an MDA. Similarly, in the analysis result for Am-243 (run 1, Table 25), the percent uncertainty is designated as UL (upper limit due to spectral interference) and since the third and second run results of <1.90E-03 and <2.14E-03 µCi/g are considered as less than the MDA the result is only reported as an MDA. Thus, the one sigma percent uncertainty for that set of runs for Am-243 is presented as UL/MDA.

All analyses have been performed in accordance with the quality assurance protocols identified in the Liquid Waste Tank Residuals Sampling – Quality Assurance Program Plan (LWRTS-QAPP).[7] Associated raw data and corresponding quality assurance data have been captured in data packages (data "wallets") generated and controlled by the AD section and retained as records. Independent technical verification (ITV) checklists are a separate deliverable of this task (separate from this report) and can be accessed through the AD section.[xii, xiii, xiv, xv, xvi, xvii]

To monitor potential sample contamination during processing in the SRNL Shielded cell, analytical blanks (reagent blanks, ARG and Tank 8 simulant sludge) were analyzed as well as the Tank 16H samples. Blank quality control analyses results can be accessed through SRNL AD section data packages as specified in the LWRTS-QAPP.[7] Although analyses of the ARG (Analysis Reference Glass) and Tank 8 simulant solids blanks both provided valid measures of potential radionuclide contamination, results for the Tank 8 simulant solids blank were judged more appropriate for two primary reasons: 1) the Tank 8 simulant solids aliquots were carried through the entire series of Shielded Cells preparation and digestion steps, just like the tank samples (while the ARG aliquots were prepared outside of the Shielded Cells and then only digested in the Shielded Cells); and 2) the dilution factors for the Tank 8 simulant solids aliquots were consistent with those of the tank samples (while the dilution factors for the ARG aliquots were approximately four times those of the tank samples). However, there are other problems which have been recently encountered in the use of Tank 8 simulant. This Tank 8 simulant material also contains an impurity material with mass 93 and is suspected to be niobium-93 as well as K-40 and thorium. The presence of these trace impurities in the reference sample may have interfered with the quantification for these elements in the actual samples. Thus, going forward, this Tank 8 simulant will not be used as reference blanks in the shielded cells for Zr-93, K-40 and thorium analysis. Suggested blank materials to be used in the shielded cell or to take the place of Tank 8 simulant includes synthetic nitrated sodalite (aluminosilicate material) sodium nitrate or sodium carbonate powders.

For both the Tank 16H Primary Liner and Annulus composite sample analyses, most of the sample analytical replicates in digestions for the inorganics, anions and radionuclides show good precision, giving a %RD of less than 20 %, indicating the solids composition were reasonably homogenous. The reporting units for all radionuclides including peroxide fusion(PF) and aqua-regia (AQR) digestion analytical results are presented per gram of composite Tank 16H sample. Correction for water content as determined by sub-sample drying at 110 ºC, if required (original "as received" basis to dry basis), can be accomplished through the use of the dry solid weight percent (wt %) values as shown in Table 2 for the Tank 16H Primary Liner samples and Table 5 for each Tank 16H Annulus composite sample. For example, µCi/g dried solids = [x µCi/g of "as-received solids * (100 g of "as-received solids)/90.4g dried solids]; using composite sample 1 in Table 5. Here x µCi/g represents the unknown activity of the "as-received" solids.

The one sigma analytical measurement uncertainty value for all of the anions and transition metals reported here is 20%. Leaching results are presented per gram of the homogenized Tank 16H Primary Liner sample or the homogenized Tank 16H Annulus composite samples.

Tables 7 through 10 and Tables 18 through 20, respectively, contains inorganic constituent analytical results for the Primary Liner samples (1-P, 2-P and 3-P) and the three Tank 16H Annulus composite samples, while Tables 11 through 13 and Tables 21 through 23, respectively, show the water soluble anion constituents for the Tank 16H Primary Liner Samples and the three Tank 16H Annulus composite samples. Tables 14 through 17 and Tables 24 through 26, respectively, show the analytical results for the standard radiological constituents for the three Tank 16H Primary Liner Samples (1-P, 2-P and 3-P) and the three Tank 16H Annulus composite samples.

Table 9 shows the elemental constituent comparison in Tank 16H Primary Liner sample 2-P and the iron-rich magnetic fraction from sample 2-P, while Table 16 shows the U-238 and Cs-137 comparison in Tank 16H Primary Liner sample 2-P and the iron-rich magnetic fraction from sample 2-P.

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 PUTTA (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, radiochemistry is typically selected due to better sensitivity and precision.

In this data presentation, the analysis detection limit 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 result for radionuclides, cations and anions were very close to the target detection limit because they were about the same order of magnitude as the target limits, although some of these were about a factor of 4 higher than the target detection limit. 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. However, when the analytical detection limit is one or more orders of magnitude above the target detection limit, the detection limits is definitely considered unmet. Thus, in this report the emphases of not meeting the desired target detection limits 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 minimum detection limits (MDLs), as specified in the TTR and TTQAP were met for the **routine** radionuclide species characterized for Tank 16H Annulus composite samples and Tank 16H Primary Liner samples, some were not met. Most of the radionuclide analyses results which failed to meet the minimum target detection limits were, however, of the same order of magnitude as the minimum target detection limits and were a factor of 4 higher. This class of routine radionuclides analysis results for the Tank 16H Primary Liner samples included U-233, U-234 for samples 2-P and 3-P, Pu-238 and Pu-240 for sample 2-P, Am-242m and Am-243 for sample 2-P and 3-P (See Tables 14 through 17). Analyses results for Ni-59, Ni-63, Pu-239 and Sr-90/Y-90 for Tank 16H Primary Liner samples were the main routine radionuclide constituents whose analysis results failed to meet the minimum target detection limits (Ni-59 and Ni-63 analyses in samples 1-P, Ni-63 in sample 3-P and Pu-239 for sample 2-P). Analysis for Sr-90/Y-90 in sample 2-P was several orders of magnitude higher than the minimum target detection limit as shown in Table 27 summary.

In summary, the minimum target detection limits for the following radionuclides were not consistently met for the following radionuclides K-40, Ni-59, Ni-63, Cs-137, Ba-137m, Pu-238 and Pu-240 in the Primary Liner samples. In all these cases, the difference between the target detection limits and the measured analytical limits were one or more order of magnitude higher. In some Primary Liner samples the detection limit for radionuclides like Pu-238, Pu-240, Cs-137, Ba-137m and Nb-94 were the same order of magnitude as the target detection limit and less than a factor of 4 higher. In cases like this, the minimum target detection limit is considered met.

With the exception of Co-60 (Tank 16H Annulus composite sample 1), Ni-63, U-233, Am-242m, Am-243 and Cf-251 analysis results for all composite samples, the target detection limit for the **routine** radionuclides were met. As shown in Tables 24 through 26, analysis results for Ni-63, U-233, Am-242m, Am-243 and Cf-251 in Tank 16H Annulus composite samples were the same order of magnitude as the target detection limits and less than a factor of 4 higher and thus the minimum detection limit is considered met in these cases.

The Tank 16H Annulus composite sample 1 reagent blank for Co-60 had a Co-60 value which was greater than 10% of the sample value. As a result, all sample Co-60 measured values were assigned upper limit values although these values seemed quantitative enough. Even with this upper value assignment, the detection limit for Co-60 in this sample was still the same order of magnitude as the target minimum detection limit.

Several of the radionuclide detection limits for the routine analytes in the Tank 16H Primary Liner samples were not met because the samples themselves did not contain quantifiable amounts of the reference materials from which the radionuclides could be back calculated such as U-238. Most of the radionuclide analytical results were near their detection limits.

The magnetic fraction from sample 2-P was also analyzed for routine radionuclides and the analysis result compared with those from the parent sample 2-P as shown in Table 16. With the exception of U-238, there are less uranium isotopes in the magnetic fraction than in the parent sample 2-P and similarly, there seem to be more Pu-239 and Pu-240 in the parent 2-P sample than in the magnetic fraction. Other than these minor differences between the magnetic fraction and the parent 2-P sample both samples have identical chemical constituents.

Routine radionuclide analytical results are also compared between different methods used for characterization of Tank 16H Annulus composite samples and Tank 16H Primary Liner samples, specifically comparing results from inductively coupled plasma-mass spectrometer (ICP-MS) with results from other methods of analyzing for the routing 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 counting for Pu-239/240. Similarly, analytical results for Tc-99 can also be obtained through ICP-MS and through counting techniques, which involves 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 16H 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 two methods, in order words, determine if the data obtained by the two different methods are fairly in good agreement.

Appendices A-6 to A-8 show the Tc-99 analytical results for the Tank 16H Primary liner samples (1-P, 2-P and 3-P) by two methods (ICP-MS and LSC). The average percent relative deviation (%RD) between ICP-MS and LCS analytical results for Tc-99 in samples 1-P and 3-P are 22.39 ± 17.93 and 20.82 ±13.58, respectively. The analytical results from these two methods for Tc-99 are within the acceptable analytical error margins of 20% for Tc-99 analysis. Since sample 2-P does not show much activity for routine radionuclides, including Tc-99, analysis results for Tc-99 in sample 2-P by ICP-MS provides only the detection limits for Tc-99 by ICP-MS; hence only less than values are presented. On the other hand, Tc-99 by counting (LSC) is a better method with far better sensitivity and precision and thus better detection limit for Tc-99 than ICP-MS method. The Tc-99 analysis result by counting averaged 9.88 E-04 µCi/g, which points in the same direction as the ICP-MS average analytical result of <2.10E-02 µCi/g for the same sample set.

Appendices A-9 and A-10 also show a summary of the comparison data for Pu-239 and 240 analytical results by two different methods for both Tank 16H annulus composite samples and Tank 16H Primary Liner samples; ICP-MS and Pu extraction followed by counting. The ICP-MS result for Pu-239 and Pu-240 for the Tank 16H annulus composites 1 and 2 analytical results are about the same order of magnitude as the LSC data for these select routine radionuclides (Appendices A-9 and A-10). The average %RD for the two methods for Pu-239 and Pu-240 are, respectively, 23.13 ± 5.29 and 27.42 ± 13.84. Given that the LSC method is in general a better method for Pu-239 and Pu-240 analyses and with such large uncertainty, as measured by the standard deviation, the analytical results from the two methods are fairly comparable.

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

The non-radioactive reference materials used for the elemental analyses results presented in Tables 7 through 10 for the Tank 16H Primary Liner samples and Tables 18 through 20 for the Tank 16H Annulus composite samples were a reference glass standard (ARG) and dried Tank 8 simulant sludge samples. Appendices A-3 and A-4 contain the elemental analytical results for the two reference materials in comparison to their known reference values.[vi, vii] In the reference ARG samples, elements (Ba, Cr, Cu, Sr and Zn) with concentrations less than 0.1 wt% were not included in Appendices A-4 because their concentrations could be influenced by trace reagent impurities. Similarly, for the Tank 8 simulants, K was not included in Appendix A-3.

A comparison of the laboratory results for the cations present in the simulant sludge shows that the laboratory analytical results are in reasonable agreement with the expectations based on the nominal sludge simulant recipe. The typical percent relative deviation (%RD defined as [difference/mean]*100) is 20% or less, which is good considering the recipe may not be completely representative of the simulant composition. Similarly, looking at the analytical results for the 12 select elemental constituents of the ARG reference sample, Appendix A-4, the percent relative deviation for each of the 12 constituents was below 10%.

Analytical elemental results were also compared between different methods used for characterization of Tank 16H Annulus composite samples and Tank 16H Primary Liner samples, specifically comparing results from ICP-MS with results from inductively coupled plasma-emission spectroscopy (ICP-ES). The concentrations of select cations (Ba and Co) 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-5 for Ba, and Co. The average percent relative deviation between ICP-MS and ICP-ES analytical results for Co and Ba were, respectively, <10% and <5%. These comparison results are summarized in Appendix A-5 and show that ICP-ES analytical results are about the same order of magnitude as the ICP-MS data for these select metals.

Because of the low iodine concentration in the Tank 16H leachate samples, analysis for iodine by mass spectroscopy was preferred over analyses by ion chromatography (IC). Leached Tank 16H Annulus composite and Primary Liner sample analyses for iodine by mass spectroscopy for stable iodine, assuming 100% iodine natural abundance, was based on the assumption that all other elements with mass 127 (Xe-127, Sn-127, Cs-127, Ba-127, La-127, In-127 etc.) have relatively short half-lives ranging from milliseconds to a few days. Thus, the total stable iodine reported in Tables 11 through 13 and Tables 21 through 23 for elemental iodine is based on mass spectroscopy data for mass-127. The sum of iodine in each Tank 16H Annulus composite sample is approximated by adding mass 127 stable iodine results with mass 129 radioactive iodine data. The anion analysis detection limits for both the Primary Liner and Tank 16H Annulus composite sample leachates were met in all cases as shown in Tables 11 through 13 and Tables 21 through 23.

The target detection limits for all the elemental constituents of Tank 16H Primary Liner and Annulus composite samples were met with the exception of the following metals B, Mo and U for the Primary liner samples and B, Mo, U, Co, Zn and Pb for the Annulus composite samples. The detection limits for B, and Mo in the primary Liner samples are about the same order of magnitude as their individual target detection limits and so the target minimum detection limits are considered met. It is worth noting that ICP-ES is not the preferred method for analyzing for total uranium and so one does not expect to meet the target minimum detection limit by this method of analysis.

The detection limits for Mo, Co, Zn and Pb in the Tank 16H Annulus composite samples are all one or two orders of magnitude higher than the target minimum detection limits and the detection limits are thus considered unmet. The detection limits for anion analysis for both the Primary Liner and Annulus composite samples are all within the target detection limits with analysis results for PO_4^{-3} and F^{-1} being the exceptions. The minimum detection limits for these two anions are about the same order of magnitude as their individual target detection limits and so the target minimum detection limits are considered met.

Table 9 shows a summary of the elemental constituent comparison between Tank 16H Primary Liner sample 2-P and the magnetic fraction which came from the parent sample, 2-P. In general, the elemental composition in the magnetic fraction is similar to that of the parent sample, 2-P. The Fe, Mn, Co, Pb and Zn compositions are of the same order of magnitude in the parent 2-P sample and the magnetic fraction although there is more Al in the magnetic fraction and more Cr, Cu and Ba in the parent 2-P sample.

3.3 Data Quality and Presentations for Non-Routine Radionuclide Analytes.

Most of the **non-routine** radionuclides are not present in easily measurable concentrations in the Tank 16H samples, especially the Primary Liner sample 2-P and in some cases there were significant sample matrix effects as in the cases of Zr-93, Pa-231, and Cm-244. Thus, existing standard methods are not sufficient in attaining the requested minimum detection limits for these non-routine radionuclides. The analysis for Zr-93 presents another special case when the standard Tank 8 simulant sludge is used as the reference material. During this Tank 16H characterization it was confirmed that the standard Tank 8 simulant sludge contains an elemental component with mass 93, which happens to be Nb-93. The presence of mass 93 impurity material in the Tank 8 simulant sludge poses a unique problem when analyzing for Zr-93 using this reference material. It becomes difficult to accurately account for the Zr-93 concentration. These cases involving characterizations for Cl-36, Zr-93 and Pa-231 may require new method development to meet the low detection limit requirements and to minimize spectral interferences.

There were quality control problems associated with matrix interferences and low Ra-226 activities in the analysis of Tank 16H Primary Liner samples for Ra-226. As a result, no analytical results are reported for sample 2-P, run 1 and sample 3-P runs 1 and 3 (Tables 15 and 17). Analysis for Ra-226 for the three Tank 16H Annulus composite samples did not present any special problems. Thorium-230 analysis blanks for all three Tank 16H Primary Liner samples and three Annulus composite samples showed, as expected, “no-yields” for Th-230. A no-yield implies that there was no activity observed in the sample. The Tank 16H samples (Primary Liner and Annulus composite samples) themselves did, however, show activity for Th-230.

Tables 27 and 28 show, respectively, summary information for the Tank 16H Primary Liner samples unmet radionuclide detection limits and information for the Tank 16H Annulus composite samples radionuclide unmet detection limits. The analytical detection limits in the cases summarized in Tables 27 and 28 are one or more orders of magnitude above the minimum target detection limit and thus the detection limits are considered unmet. The target analytical limits for any of the Tank 16H Primary Liner samples were not met for the **non-routine** radionuclides Zr-93, Pa-231 and Cm-244.

The target detection limits for the **non-routine** radionuclides Cl-36, Zr-93 and Cm-244 were not met for any of the Tank 16H Annulus composite samples. The MDL for Pa-231 in the Tank 16H Annulus composite sample was not consistently met.

Because some of these analytical results for Tank 16H Primary Liner and Annulus composite samples did not quite meet the required minimum target detection limits, SRNL consulted with SRR who reviewed the available data at that time on the Tank 16H samples and determined that the impacts of not meeting the target detection limits were acceptable.[xviii]

3.3.1 Tank 16H Primary Liner Samples

Table 7 Elemental Constituents in Tank 16H Primary Liner Sample 1-P (Ground)

Analyte	Tank 16H 1-P, Run 1, wt%	Tank 16H 1-P, Run 2, wt%	Tank 16H 1-P, Run 3, wt%	Average wt%	Stdev.	Target Detection Limit (wt %)
Ag	<1.32E-03	<1.33E-03	<1.31E-03	**<1.32E-03**		7.0E-03
Al	5.23E-01	3.72E-01	5.60E-01	**4.85E-01**	*9.96E-02*	1.0E+00
B	<1.91E-02	<1.91E-02	<1.88E-02	**<1.90E-02**		≤1.6E-02
Ba	1.22E-02	5.34E-03	1.26E-02	**1.00E-02**	*4.08E-03*	6.0E-03
Cd	2.22E-03	1.95E-03	2.34E-03	**2.17E-03**	*2.00E-04*	1.0E-02
Co	4.87E-03	5.18E-03	4.49E-03	**4.85E-03**	*3.46E-04*	8.0E-04
Cr	2.45E-02	2.57E-02	2.66E-02	**2.56E-02**	*1.05E-03*	3.0E-02
Cu	5.63E-02	4.65E-02	6.33E-02	**5.54E-02**	*8.44E-03*	3.0E-02
Fe	6.08E+01	6.03E+01	6.16E+01	**6.09E+01**	*6.56E-01*	3.0E-02
Mn	3.65E-01	3.69E-01	3.81E-01	**3.72E-01**	*8.33E-03*	2.0E-02
Mo	<8.12E-03	<8.16E-03	<8.03E-03	**<8.10E-03**		2.0E-03
Na	2.30E-02	8.57E-03	1.90E-02	**1.69E-02**	*7.45E-03*	NA
Ni	3.00E-02	2.94E-02	3.26E-02	**3.07E-02**	*1.70E-03*	4.5E+00
Pb	1.02E-02	<9.64E-03	1.26E-02	**≤1.08E-02**		2.0E-02
Sb	<2.83E-02	<2.85E-02	<2.80E-02	**<2.83E-02**		≤1.0E-01
Si	6.64E-01	5.97E-01	6.34E-01	**6.32E-01**	*3.36E-02*	NA
Sr	2.07E-02	1.03E-02	2.22E-02	**1.77E-02**	*6.48E-03*	3.0E-02
U	<2.15E-01	<2.16E-01	<2.13E-01	**<2.15E-01**		4.0E-03
Zn	7.07E-02	6.25E-02	7.69E-02	**7.00E-02**	*7.22E-03*	8.0E-03
As	3.40E-03	3.57E-03	3.38E-03	**3.45E-03**	*1.04E-04*	≤5.4E-04
Hg	1.39E-01	1.94E-01	1.33E-01	**1.55E-01**	*3.36E-02*	2.0E-01
Se	<5.38E-04	<5.41E-04	<5.32E-04	**<5.37E-04**		≤1.0E-03

Table 8 Elemental Constituents in Tank 16H Primary Liner Sample 2-P (Ground)

Analyte	Tank 16H 2-P, Run 1, wt%	Tank 16H 2-P, Run 2, wt%	Tank 16H 2-P, Run 3, wt%	Average wt%	Stdev.	Target Detection Limit (wt %)
Ag	<1.33E-03	<1.31E-03	<1.30E-03	**<1.31E-03**		7.0E-03
Al	6.30E-02	7.46E-02	4.88E-02	**6.21E-02**	*1.29E-02*	1.0E+00
B	<1.91E-02	<1.88E-02	<1.87E-02	**<1.89E-02**		≤1.6E-02
Ba	1.81E-03	1.64E-03	1.38E-03	**1.61E-03**	*2.17E-04*	6.0E-03
Cd	2.59E-03	2.65E-03	2.43E-03	**2.56E-03**	*1.14E-04*	1.0E-02
Co	4.82E-03	5.01E-03	4.74E-03	**4.86E-03**	*1.39E-04*	8.0E-04
Cr	3.68E-02	4.97E-02	1.93E-02	**3.53E-02**	*1.53E-02*	3.0E-02
Cu	1.56E-02	1.68E-02	1.66E-02	**1.63E-02**	*6.43E-04*	3.0E-02
Fe	6.02E+01	6.06E+01	6.22E+01	**6.10E+01**	*1.06E+00*	3.0E-02
Mn	5.27E-01	5.30E-01	5.24E-01	**5.27E-01**	*3.00E-03*	2.0E-02
Mo	<8.15E-03	<8.03E-03	<7.97E-03	**<8.05E-03**		2.0E-03
Na	<6.49E-03	<6.40E-03	<6.35E-03	**<6.41E-03**		NA
Ni	2.18E-02	2.86E-02	1.28E-02	**2.11E-02**	*7.93E-03*	4.5E+00
Pb	6.11E-01	6.25E-01	5.11E-01	**5.82E-01**	*6.22E-02*	2.0E-02
Sb	<2.84E-02	<2.80E-02	<2.78E-02	**<2.81E-02**		≤1.0E-01
Si	4.85E-01	6.12E-01	5.36E-01	**5.44E-01**	*6.39E-02*	NA
Sr	<9.82E-05	<9.68E-05	<9.61E-05	**<9.70E-05**		3.0E-02
U	<2.16E-01	<2.13E-01	<2.11E-01	**<2.13E-01**		4.0E-03
Zn	4.09E-02	4.39E-02	3.17E-02	**3.88E-02**	*6.36E-03*	8.0E-03
As	3.70E-03	3.51E-03	3.93E-03	**3.71E-03**	*2.10E-04*	≤5.4E-04
Hg	3.84E-03	4.05E-03	6.21E-03	**4.70E-03**	*1.31E-03*	2.0E-01
Se	<5.40E-04	<5.32E-04	<5.28E-04	**<5.33E-04**		≤1.0E-03

Table 9 Elemental Constituent Comparison of Tank 16H Primary Liner Sample 2-P (Ground) and- Magnetic Fraction (Unground) from Sample 2-P

Analyte	Magnetic Fraction-1 (Unground) wt%	Magnetic Fraction-2 (Unground) wt%	Magnetic Fraction-3 (Unground) wt%	Magnetic Fraction (Unground) Average wt%	Sample 2-P (Ground) Average wt%
Ag	<1.30E-03	<1.32E-03	<1.26E-03	**<1.29E-03**	**<1.31E-03**
Al	9.85E-01	7.23E-02	8.12E-02	**3.80E-01**	**6.21E-02**
B	<1.87E-02	<1.91E-02	<1.82E-02	**<1.87E-02**	**<1.89E-02**
Ba	9.12E-04	6.75E-04	1.13E-03	**9.06E-04**	**1.61E-03**
Cd	<5.85E-03	<5.97E-03	<5.70E-03	**<5.84E-03**	**2.56E-03**
Co	3.81E-03	1.88E-03	2.52E-03	**2.74E-03**	**4.86E-03**
Cr	9.82E-03	6.65E-03	1.02E-02	**8.89E-03**	**3.53E-02**
Cu	9.06E-03	3.00E-03	3.21E-03	**5.09E-03**	**1.63E-02**
Fe	6.28E+01	4.16E+01	5.83E+01	**5.42E+01**	**6.10E+01**
Mn	5.71E-01	3.98E-01	5.67E-01	**5.12E-01**	**5.27E-01**
Mo	<7.97E-03	<8.12E-03	<7.76E-03	**<7.95E-03**	**<8.05E-03**
Na	<6.34E-03	<6.47E-03	<6.18E-03	**<6.33E-03**	**<6.41E-03**
Ni	<1.29E-02	<1.32E-02	<1.26E-02	**<1.29E-02**	**2.11E-02**
Pb	2.88E-01	4.24E-01	4.63E-01	**3.92E-01**	**5.82E-01**
Sb	<2.78E-02	<2.83E-02	<2.71E-02	**<2.77E-02**	**<2.81E-02**
Sr	<9.60E-05	<9.78E-05	<9.35E-05	**<9.58E-05**	**<9.70E-05**
U	<2.11E-01	<2.15E-01	<2.06E-01	**<2.11E-01**	**<2.13E-01**
Zn	1.31E-02	1.74E-02	2.39E-02	**1.81E-02**	**3.88E-02**

Table 10 Elemental Constituents in Tank 16H Primary Liner Sample 3-P (Ground)

Analyte	Tank 16H 3-P, Run 1, wt%	Tank 16H 3-P, Run 2, wt%	Tank 16H 3-P, Run 3, wt%	Average wt%	Stdev.	Target Detection Limit (wt %)
Ag	<1.26E-03	<1.31E-03	<1.32E-03	**<1.30E-03**		7.0E-03
Al	4.14E-01	3.92E-01	4.15E-01	**4.07E-01**	*1.30E-02*	1.0E+00
B	<1.82E-02	<1.90E-02	<1.91E-02	**<1.88E-02**		≤1.6E-02
Ba	7.74E-03	7.03E-03	6.76E-03	**7.18E-03**	*5.06E-04*	6.0E-03
Cd	2.72E-03	2.18E-03	2.15E-03	**2.35E-03**	*3.21E-04*	1.0E-02
Co	8.07E-03	7.43E-03	7.72E-03	**7.74E-03**	*3.20E-04*	8.0E-04
Cr	1.70E-02	1.69E-02	1.53E-02	**1.64E-02**	*9.54E-04*	3.0E-02
Cu	4.58E-02	4.31E-02	4.13E-02	**4.34E-02**	*2.26E-03*	3.0E-02
Fe	6.07E+01	6.11E+01	6.09E+01	**6.09E+01**	*2.00E-01*	3.0E-02
Mn	3.13E-01	3.10E-01	3.22E-01	**3.15E-01**	*6.24E-03*	2.0E-02
Mo	<7.75E-03	<8.08E-03	<8.12E-03	**<7.98E-03**		2.0E-03
Na	1.46E-02	1.30E-02	1.32E-02	**1.36E-02**	*8.72E-04*	NA
Ni	1.96E-02	2.03E-02	1.99E-02	**1.99E-02**	*3.51E-04*	4.5E+00
Pb	7.51E-02	8.22E-02	7.92E-02	**7.88E-02**	*3.56E-03*	2.0E-02
Sb	<2.70E-02	<2.82E-02	<2.83E-02	**<2.78E-02**		≤1.0E-01
Si	2.87E-01	3.50E-01	2.55E-01	**2.97E-01**	*4.83E-02*	NA
Sr	1.31E-02	1.18E-02	1.10E-02	**1.20E-02**	*1.06E-03*	3.0E-02
U	<2.05E-01	<2.14E-01	<2.15E-01	**<2.11E-01**		4.0E-03
Zn	1.97E-02	2.44E-02	2.22E-02	**2.21E-02**	*2.35E-03*	8.0E-03
As	4.19E-03	4.11E-03	4.13E-03	**4.14E-03**	*4.16E-05*	≤5.4E-04
Hg	1.54E-01	1.66E-01	1.75E-01	**1.65E-01**	*1.05E-02*	2.0E-01
Se	<5.14E-04	<5.36E-04	<5.38E-04	**<5.29E-04**		≤1.0E-03

Table 11 Anions Leached per gram of Tank 16H Primary Liner Sample 1-P (Ground)

Analyte	Run-1 (wt %)	Run-2 (wt %)	Run-3 (wt %)	Average (wt %)	Std. Dev.	Target Detection Limit (wt %)
Fluoride, F^{-1}	<4.87E-03	<4.86E-03	<4.70E-03	**<4.81E-03**		1.0E-02
Formate, CHO_2^{-1}	<4.87E-03	<4.86E-03	<4.70E-03	**<4.81E-03**		NA
Chloride , Cl^{-1}	<4.87E-03	<4.86E-03	<4.70E-03	**<4.81E-03**		4.0E-02
Nitrite , NO_2^{-1}	<4.87E-03	<4.86E-03	<4.70E-03	**<4.81E-03**		2.0E-01
Bromide, Br^{-1}	<4.87E-03	<4.86E-03	<4.70E-03	**<4.81E-03**		NA
Nitrate , NO_3^{-1}	1.46E-02	9.72E-03	1.41E-02	**1.28E-02**	*2.68E-03*	7.0E-01
Phosphate, PO_4^{-3}	<4.87E-03	<4.86E-03	<4.70E-03	**<4.81E-03**		1.0E-02
Sulfate, SO_4^{-2}	<4.87E-03	<4.86E-03	<4.70E-03	**<4.81E-03**		9.0E-02
Oxalate, $C_2O_4^{2-}$	<4.87E-03	<4.86E-03	<4.70E-03	**<4.81E-03**		NA
Iodine, I-127	<6.38E-07	<6.37E-07	<6.15E-07	**<6.30E-07**		8.0E-01
Iodine, I-129	1.13E-04	9.62E-05	1.75E-04	**1.28E-04**	*4.15E-05*	See I-129
Total Iodine	1.13E-04	9.62E-05	1.75E-04	**1.28E-04**	*4.15E-05*	NA

Table 12 Anions Leached per gram of Tank 16H Primary Liner Sample 2-P (Ground)

Analyte	Run-1 (wt %)	Run-2 (wt %)	Run-3 (wt %)	Average (wt %)	Std. Dev.	Target Detection Limit (wt %)
Fluoride, F^{-1}	<4.86E-03	<4.91E-03	<4.70E-03	**<4.82E-03**		1.0E-02
Formate, CHO_2^{-1}	<4.86E-03	<4.91E-03	<4.70E-03	**<4.82E-03**		NA
Chloride , Cl^{-1}	<4.86E-03	<4.91E-03	<4.70E-03	**<4.82E-03**		4.0E-02
Nitrite , NO_2^{-1}	<4.86E-03	<4.91E-03	<4.70E-03	**<4.82E-03**		2.0E-01
Bromide, Br^{-1}	<4.86E-03	<4.91E-03	<4.70E-03	**<4.82E-03**		NA
Nitrate , NO_3^{-1}	9.72E-03	9.82E-03	9.39E-03	**9.64E-03**	*2.24E-04*	7.0E-01
Phosphate, PO_4^{-3}	<4.86E-03	<4.91E-03	<4.70E-03	**<4.82E-03**		1.0E-02
Sulfate, SO_4^{-2}	<4.86E-03	<4.91E-03	<4.70E-03	**<4.82E-03**		9.0E-02
Oxalate, $C_2O_4^{2-}$	<4.86E-03	<4.91E-03	<4.70E-03	**<4.82E-03**		NA
Iodine, I-127	<6.37E-07	<6.43E-07	<6.15E-07	**<6.32E-07**		8.0E-01
Iodine, I-129	<4.13E-06	<3.09E-06	4.80E-06	**≤4.01E-06**		See I-129
Total Iodine	<4.77E-06	<3.73E-06	<5.42E-06	**<4.64E-06**		NA

Table 13 Anions Leached per gram of Tank 16H Primary Liner Sample 3-P (Ground)

Analyte	Run-1 (wt %)	Run-2 (wt %)	Run-3 (wt %)	Average (wt %)	Std. Dev.	Target Detection Limit (wt %)
Fluoride, F^{-1}	<4.66E-03	<4.84E-03	<4.91E-03	**<4.81E-03**		1.0E-02
Formate, CHO_2^{-1}	<4.66E-03	<4.84E-03	<4.91E-03	**<4.81E-03**		NA
Chloride , Cl^{-1}	<4.66E-03	<4.84E-03	<4.91E-03	**<4.81E-03**		4.0E-02
Nitrite , NO_2^{-1}	<4.66E-03	<4.84E-03	<4.91E-03	**<4.81E-03**		2.0E-01
Bromide, Br^{-1}	<4.66E-03	<4.84E-03	<4.91E-03	**<4.81E-03**		NA
Nitrate , NO_3^{-1}	4.66E-02	4.36E-02	4.42E-02	**4.48E-02**	*1.62E-03*	7.0E-01
Phosphate, PO_4^{-3}	<4.66E-03	<4.84E-03	<4.91E-03	**<4.81E-03**		1.0E-02
Sulfate, SO_4^{-2}	<4.66E-03	<4.84E-03	<4.91E-03	**<4.81E-03**		9.0E-02
Oxalate, $C_2O_4^{2-}$	<4.66E-03	<4.84E-03	<4.91E-03	**<4.81E-03**		NA
Iodine, I-127	<6.11E-07	<6.34E-07	<6.43E-07	**<6.29E-07**		8.0E-01
Iodine, I-129	1.60E-04	1.60E-04	1.83E-04	**1.68E-04**	*1.33E-05*	See I-129
Total Iodine	1.60E-04	1.60E-04	1.83E-04	**1.68E-04**	*1.33E-05*	NA

Table 14 Radiological Constituents for Tank 16H Primary Liner Sample 1-P, μCi/g (Ground)

Analytes	Run 1	Run 2	Run 3	Average	Stdev.	One Sigma %Uncert.	Detection Limits
C-14	<7.12E-04	<7.39E-04	<1.00E-03	**<8.18E-04**		MDA	1.0E-01
Cl-36	<3.47E-05	<4.73E-06	<1.06E-05	**<1.67E-05**		UL	9.0E-05
K-40	<5.77E-03	<6.40E-03	<9.05E-03	**<7.07E-03**		MDA	4.0E-05
Ni-59	<2.18E-01	<1.81E+00	<1.90E-02	**<6.83E-01**		UL	5.0E-02
Ni-63	<1.43E+00	<1.25E+01	<3.95E-01	**<4.78E+00**		UL	1.0E-01
Co-60	<3.09E-03	<2.89E-03	<2.85E-03	**<2.94E-03**		MDA	1.0E-03
Sr-90	3.43E+03	2.41E+03	2.57E+03	**2.80E+03**	*5.52E+02*	10.02	1.0E-03
Y-90	3.43E+03	2.41E+03	2.57E+03	**2.80E+03**	*5.52E+02*	10.02	1.0E-03
Zr-93	<2.89E-02	<3.25E-02	<2.68E-02	**<2.94E-02**		UL	1.0E-04
Nb-94	<8.15E-03	<6.62E-03	<3.80E-03	**<6.19E-03**		MDA	3.0E-03
Tc-99	1.28E-01	1.05E-01	8.74E-02	**1.07E-01**	*2.03E-02*	8.24	1.0E-03
I-129	1.99E-04	1.70E-04	3.08E-04	**2.26E-04**	*7.30E-05*	5.07	9.0E-06
Cs-135	<1.34E-04	<1.24E-04	<8.96E-05	**<1.16E-04**		UL	1.0E-04
Cs-137	<2.64E-01	<3.05E-01	<2.76E-01	**<2.82E-01**	-	UL+	1.0E-03
Ba-137m	<2.50E-01	<2.89E-01	<2.61E-01	**<2.67E-01**	-	UL+	1.0E-03
Eu-154	1.99E-01	1.63E-01	1.43E-01	**1.68E-01**	*2.84E-02*	5	NA
Ra-226	<1.95E-03	<3.15E-04	<5.27E-04	**<9.31E-04**		MDA	9.0E-04
Th-230	<4.64E-04	<3.84E-04	<6.67E-04	**<5.05E-04**		UL	1.0E-03
Pa-231	<2.66E-03	<3.20E-03	<2.95E-03	**<2.94E-03**		DL	9.0E-05
U-233	1.16E-04	8.92E-05	1.09E-04	**1.05E-04**	*1.38E-05*	20	1.0E-03
U-234	1.67E-04	1.59E-04	1.55E-04	**1.60E-04**	*6.15E-06*	20	1.0E-03
U-235	6.31E-07	5.86E-07	5.54E-07	**5.90E-07**	*3.85E-08*	20	1.0E-05
U-236	1.69E-06	1.68E-06	1.59E-06	**1.65E-06**	*5.63E-08*	20	NA
U-238	1.97E-06	2.03E-06	1.84E-06	**1.95E-06**	*9.93E-08*	20	5.0E-05
Np-237	<6.89E-04	<6.93E-04	<6.82E-04	**<6.88E-04**		MDA	1.0E-03
Pu-238	< 9.95E-01	< 9.41E-01	1.27E+00	**≤ 1.07E+00**		UL/5.79	1.0E-03
Pu-239	4.31E-02	4.07E-02	4.06E-02	**4.14E-02**	*1.41E-03*	20	1.0E-03
Pu-240	1.91E-02	1.80E-02	1.81E-02	**1.84E-02**	*6.38E-04*	20	1.0E-03
Pu-239/240	<7.70E-02	1.00E-01	1.37E-01	**≤1.05E-01**		UL/9.94	NA
Pu-241	< 2.40E-01	< 2.61E-01	< 3.18E-01	**< 2.73E-01**		UL	1.0E-03
Pu-242	<4.25E-05	<3.74E-05	<3.80E-05	**<3.93E-05**		DL	1.0E-03
Pu-244	<4.68E-07	<3.81E-07	<3.14E-07	**<3.88E-07**		DL	1.3E-04
Am-241	2.68E-01	3.14E-01	3.03E-01	**2.95E-01**	*2.35E-02*	10.2	1.0E-03
Am-242m	<3.00E-04	<2.93E-03	<1.15E-03	**<1.46E-03**		MDA	1.0E-03
Am-243	<3.53E-03	<8.92E-03	1.08E-02	**≤7.75E-03**		30/MDA	1.0E-03
Cf-249	<3.09E-03	<4.36E-03	<3.74E-03	**<3.73E-03**		MDA	5.0E-03
Cf-251	<9.50E-03	<1.51E-02	<1.36E-02	**<1.27E-02**		MDA	1.0E-03
Cm-242	<2.48E-04	<2.42E-03	<9.50E-04	**<1.21E-03**		MDA	NA
Cm-243	<1.05E-02	<1.72E-02	<1.57E-02	**<1.45E-02**		MDA	2.0E-02
Cm-244	<6.89E-02	<3.18E-02	<1.34E-01	**<7.82E-02**		UL	1.0E-03
Cm-245	<1.18E-04	<2.83E-04	<4.18E-04	**<2.73E-04**		UL	2.0E-02
Cm-247	<8.15E-08	<1.18E-07	<1.53E-07	**<1.17E-07**		UL	1.3E-04
Cm-248	<6.85E-06	<7.75E-06	<5.99E-06	**<6.86E-06**		UL	1.3E-04

+ Value is an Upper Limit (UL) due to blank being greater than 10% of the sample value. DL: Detection Limit

Table 15 Radiological Constituents for Tank 16H Primary Liner Sample 2-P, µCi/g (Ground)

Analytes	Run 1, µCi/g	Run 2 µCi/g	Run 3 µCi/g	Average µCi/g	Stdev.	One Sigma %Uncert.	Detection Limits
C-14	<7.21E-04	<7.03E-04	<8.20E-04	**<7.48E-04**		MDA	1.0E-01
Cl-36	<2.64E-06	<2.02E-06	<2.59E-06	**<2.42E-06**		MDA	9.0E-05
K-40	<2.23E-05	<2.69E-05	<1.75E-05	**<2.22E-05**		MDA	4.0E-05
Ni-59	<1.03E-03	<5.50E-04	<1.19E-03	**<9.22E-04**		MDA	5.0E-02
Ni-63	<1.94E-03	<7.48E-04	<2.22E-03	**<1.63E-03**		UL	1.0E-01
Co-60	<4.44E-04	<8.42E-04	<7.84E-04	**<6.90E-04**		MDA	1.0E-03
Sr-90	<5.00E+00	<4.41E+00	<5.77E+00	**<5.06E+00**		MDA	1.0E-03
Y-90	<5.00E+00	<4.41E+00	<5.77E+00	**<5.06E+00**		MDA	1.0E-03
Zr-93	<3.80E-03	<4.59E-03	<3.05E-03	**<3.82E-03**		DL	1.0E-04
Nb-94	<5.50E-03	<4.68E-03	<6.85E-03	**<5.68E-03**		MDA	3.0E-03
Tc-99	5.90E-04	1.50E-03	8.74E-04	**9.88E-04**	*4.66E-04*	9.93	1.0E-03
I-129	<7.30E-06	<5.45E-06	8.47E-06	**≤ 7.07E-06**		MDA/23.2	9.0E-06
Cs-135	<6.71E-05	<1.43E-04	<2.28E-05	**<7.76E-05**		UL	1.0E-04
Cs-137	<1.32E-02	<1.02E-02	<1.42E-02	**<1.25E-02**		UL+	1.0E-03
Ba-137m	<1.25E-02	<9.65E-03	<1.34E-02	**<1.18E-02**		UL+	1.0E-03
Eu-154	<1.72E-03	<1.85E-03	<1.64E-03	**<1.74E-03**		MDA	NA
Ra-226	NR	<3.94E-04	<2.17E-03	**<1.28E-03**		DNR/MDA	9.0E-04
Th-230	<2.84E-04	<1.54E-04	<1.02E-04	**<1.80E-04**		UL	1.0E-03
Pa-231	<3.67E-03	<3.35E-03	<2.84E-03	**<3.29E-03**		DL	9.0E-05
U-233	<4.75E-03	<4.69E-03	<4.65E-03	**<4.69E-03**		DL	1.0E-03
U-234	<3.07E-03	<3.02E-03	<3.00E-03	**<3.03E-03**		DL	1.0E-03
U-235	<1.06E-06	<1.05E-06	<1.04E-06	**<1.05E-06**		DL	1.0E-05
U-236	<3.18E-05	<3.13E-05	<3.11E-05	**<3.14E-05**		DL	NA
U-238	<4.14E-07	<4.07E-07	<4.03E-07	**<4.08E-07**		DL	5.0E-05
Np-237	<6.92E-04	<6.82E-04	<6.77E-04	**<6.84E-04**		MDA	1.0E-03
Pu-238	<6.04E-03	< 4.24E-03	<7.84E-03	**< 6.04E-03**		MDA/UL	1.0E-03
Pu-239	<6.10E-02	<6.02E-02	<5.97E-02	**<6.03E-02**		DL	1.0E-03
Pu-240	<1.13E-03	<1.58E-03	<1.27E-03	**<1.33E-03**		DL	1.0E-03
Pu-239/240	<4.21E-03	<2.55E-02	<7.66E-03	**<1.24E-02**		MDA/UL	NA
Pu-241	< 3.73E-02	< 3.80E-02	< 3.68E-02	**< 3.74E-02**		MDA/UL	1.0E-03
Pu-242	<1.97E-05	<9.64E-06	<8.42E-06	**<1.26E-05**		DL	1.0E-03
Pu-244	<9.01E-08	<1.26E-07	<1.01E-07	**<1.06E-07**		DL	1.3E-04
Am-241	<3.27E-03	<4.14E-03	<3.57E-03	**<3.66E-03**		MDA	1.0E-03
Am-242m	<2.00E-04	<1.15E-05	<1.09E-04	**<1.07E-04**		MDA/UL	1.0E-03
Am-243	<2.32E-04	<9.01E-05	<4.04E-04	**<2.42E-04**		MDA/UL	1.0E-03
Cf-249	<2.46E-04	<2.23E-05	<3.00E-04	**<1.89E-04**		MDA	5.0E-03
Cf-251	<5.32E-04	<5.59E-05	<6.98E-04	**<4.29E-04**		MDA	1.0E-03
Cm-242	<1.65E-04	<9.50E-06	<9.05E-05	**<8.85E-05**		MDA/UL	NA
Cm-243	<5.68E-04	<6.58E-05	<7.93E-04	**<4.75E-04**		MDA	2.0E-02
Cm-244	<1.25E-02	<3.73E-03	<2.13E-02	**<1.25E-02**		UL	1.0E-03
Cm-245	<2.95E-05	<2.66E-06	<3.76E-05	**<2.32E-05**		UL	2.0E-02
Cm-247	<1.55E-08	<1.30E-09	<1.27E-08	**<9.85E-09**		UL	1.3E-04
Cm-248	<8.87E-07	<5.81E-08	<5.09E-07	**<4.85E-07**		UL	1.3E-04

NR: Not reported due to quality issues
+ Value is an Upper Limit (UL) due to blank being greater than 10% of the sample value. DL: Detection Limit

Table 16 U-238 and Cs-137 Comparison in Tank 16H-Primary Liner Sample 2-P (Ground) and Magnetic Fraction (Unground) from sample 2-P, µCi/g

Analytes	Magnetic Fraction-1 (Unground)	Magnetic Fraction-2 (Unground)	Magnetic Fraction-3 (Unground)	Average	Stdev.	Parent Sample 2-P (Ground) Average
Alpha count	<2.42E-01	<2.43E-01	<2.34E-01	<2.40E-01		NA
Beta count	<9.23E-01	<5.32E-01	<9.01E-01	<7.85E-01		NA
Cs-137	<3.28E-03	<5.05E-03	<3.85E-03	<4.06E-03		**<1.25E-02**
U-233	<9.29E-04	<9.47E-04	<9.05E-04	<9.27E-04		**<4.69E-03**
U-234	<6.00E-04	<6.11E-04	<5.84E-04	<5.98E-04		**<3.03E-03**
U-235	<2.07E-07	<2.11E-07	<2.02E-07	<2.07E-07		**<1.05E-06**
U-236	<6.21E-06	<6.33E-06	<6.05E-06	<6.20E-06		**<3.14E-05**
Np-237	<1.59E-03	<1.62E-03	<1.55E-03	<1.59E-03		**<6.84E-04**
U-238	7.33E-08	1.16E-07	7.46E-08	8.79E-08	*2.41E-08*	**<4.08E-07**
Pu-239	<5.97E-03	<6.08E-03	<5.81E-03	<5.95E-03		**<6.03E-02**
Pu-240	<2.19E-02	<2.23E-02	<2.13E-02	<2.18E-02		**<1.33E-03**

Table 17 Radiological Constituents for Tank 16H Primary Liner Sample 3-P, µCi/g (Ground)

Analytes	Run 1, µCi/g	Run 2 µCi/g	Run 3 µCi/g	Average µCi/g	Stdev.	One Sigma % Uncert.	Detection Limits
C-14	<7.93E-04	<7.07E-04	<9.05E-04	**<8.02E-04**		MDA	1.0E-01
Cl-36	<1.41E-05	<1.45E-05	<2.66E-05	**<1.84E-05**		UL	9.0E-05
K-40	<3.77E-03	<4.91E-03	<3.96E-03	**<4.22E-03**		MDA	4.0E-05
Ni-59	<2.05E-02	<8.51E-02	<5.18E-01	**<2.08E-01**		UL	5.0E-02
Ni-63	<3.18E-01	<8.20E-01	<4.91E+00	**<2.02E+00**		UL	1.0E-01
Co-60	<2.88E-03	No result[∞]	<2.98E-03	**<2.93E-03**		MDA	1.0E-03
Sr-90	3.15E+03	3.18E+03	3.12E+03	**3.15E+03**	*3.38E+01*	10.47	1.0E-03
Y-90	3.15E+03	3.18E+03	3.12E+03	**3.15E+03**	*3.38E+01*	10.47	1.0E-03
Zr-93	<2.15E-02	<2.31E-02	<2.62E-02	**<2.36E-02**		UL	1.0E-04
Nb-94	<1.08E-02	<8.78E-03	<7.03E-03	**<8.86E-03**		MDA	3.0E-03
Tc-99	1.35E-01	1.73E-01	1.12E-01	**1.40E-01**	*3.12E-02*	7.89	1.0E-03
I-129	2.82E-04	2.82E-04	3.23E-04	**2.95E-04**	*2.37E-05*	5.16	9.0E-06
Cs-135	<1.27E-04	<1.93E-04	<1.82E-04	**<1.67E-04**		UL	1.0E-04
Cs-137	8.20E-01	6.62E-01	6.58E-01	**7.13E-01**	*9.24E-02*	5	1.0E-03
Ba-137m	7.76E-01	6.26E-01	6.22E-01	**6.75E-01**	*8.74E-02*	5	1.0E-03
Eu-154	1.70E-01	1.83E-01	1.91E-01	**1.81E-01**	*1.02E-02*	14.41	NA
Ra-226	NR	<5.27E-04	NR	**<5.27E-04**		DNR/MDA	9.0E-04
Th-230	<5.05E-04	<3.43E-04	<7.66E-04	**<5.38E-04**		UL	1.0E-03
Pa-231	<4.11E-03	<6.04E-02	<4.77E-03	**<2.31E-02**		DL	9.0E-05
U-233	1.82E-04	2.33E-04	1.77E-04	**1.97E-04**	*3.14E-05*	20	1.0E-03
U-234	1.74E-04	1.93E-04	1.53E-04	**1.73E-04**	*2.03E-05*	20	1.0E-03
U-235	7.79E-07	7.84E-07	6.62E-07	**7.42E-07**	*6.90E-08*	20	1.0E-05
U-236	2.01E-06	2.01E-06	1.74E-06	**1.92E-06**	*1.57E-07*	20	NA
U-238	2.87E-06	3.02E-06	2.54E-06	**2.81E-06**	*2.46E-07*	20	5.0E-05
Np-237	<6.58E-04	<6.87E-04	<6.89E-04	**<6.78E-04**		MDA	1.0E-03
Pu-238	1.17E+00	1.03E+00	1.01E+00	**1.07E+00**	*8.78E-02*	5.87	1.0E-03
Pu-239	4.18E-02	4.59E-02	5.05E-02	**4.61E-02**	*4.30E-03*	20	1.0E-03
Pu-240	1.82E-02	2.05E-02	2.11E-02	**2.00E-02**	*1.55E-03*	20	1.0E-03
Pu-239/240	9.23E-02	<7.43E-02	8.96E-02	**≤8.54E-02**		UL/11.06	NA
Pu-241	< 2.86E-01	< 2.35E-01	< 2.36E-01	**< 2.52E-01**		UL	1.0E-03
Pu-242	5.27E-05	5.72E-05	6.35E-05	**5.78E-05**	*5.43E-06*	20	1.0E-03
Pu-244	<6.04E-07	<5.54E-07	<8.33E-07	**<6.64E-07**		DL	1.3E-04
Am-241	3.19E-01	3.53E-01	3.86E-01	**3.53E-01**	*3.36E-02*	10.8	1.0E-03
Am-242m	<3.49E-03	<4.68E-04	<1.76E-03	**<1.90E-03**		MDA	1.0E-03
Am-243	<3.81E-03	<9.95E-03	<6.04E-03	**<6.60E-03**		MDA	1.0E-03
Cf-249	<3.70E-03	<4.73E-03	<5.68E-03	**<4.70E-03**		MDA	5.0E-03
Cf-251	<1.05E-02	<1.49E-02	<1.62E-02	**<1.39E-02**		MDA	1.0E-03
Cm-242	<2.88E-03	<3.86E-04	<1.45E-03	**<1.57E-03**		MDA	NA
Cm-243	<1.18E-02	<1.62E-02	<1.81E-02	**<1.53E-02**		MDA	2.0E-02
Cm-244	<7.34E-02	<9.46E-02	<8.20E-02	**<8.33E-02**		UL	1.0E-03
Cm-245	<3.45E-04	<4.50E-04	<5.18E-04	**<4.38E-04**		UL	2.0E-02
Cm-247	<2.04E-07	<2.32E-07	<2.95E-07	**<2.43E-07**		UL	1.3E-04
Cm-248	<1.32E-05	<9.59E-06	<1.25E-05	**<1.18E-05**		UL	1.3E-04

[∞]Analysis was not performed on this aliquot because it exceeded dose limits.
NR: Not reported due to quality issues.
DL: Detection Limit

3.3.2 Tank 16H Annulus Composite Samples

Table 18 Elemental Constituents in Tank 16H-Annulus Composite Sample 1

Analytes	Composite-1 Run-1; wt%	Composite-1 Run-2; wt%	Composite-1 Run-3; wt%	Average; wt%	Stdev.	Detection Limit (wt %)
Ag	<3.27E-03	<3.31E-03	<3.23E-03	**<3.27E-03**		7.0E-03
Al	6.12E+00	5.99E+00	6.03E+00	**6.05E+00**	*6.66E-02*	1.0E+00
B	<4.72E-02	<4.78E-02	<4.66E-02	**<4.72E-02**		≤1.6E-02
Ba	1.75E-02	1.66E-02	1.77E-02	**1.73E-02**	*5.86E-04*	6.0E-03
Be	<2.42E-04	<2.45E-04	<2.40E-04	**<2.42E-04**		NA
Ca	<2.85E-01	<2.83E-01	<2.85E-01	**<2.84E-01***		NA
Cd	<3.39E-03	<3.44E-03	<3.35E-03	**<3.39E-03**		1.0E-02
Ce	<2.74E-02	<2.78E-02	<2.71E-02	**<2.74E-02**		NA
Co	<2.77E-02	<3.25E-02	<3.30E-02	**<3.11E-02***		8.0E-04
Cr	1.50E-02	1.46E-02	1.47E-02	**1.48E-02**	*2.08E-04*	3.0E-02
Cu	2.14E-01	7.93E-02	1.57E-02	**1.03E-01**	*1.01E-01*	3.0E-02
Fe	1.98E+00	1.74E+00	1.82E+00	**1.85E+00**	*1.22E-01*	3.0E-02
Gd	<1.39E-02	1.55E-02	1.39E-02	**≤1.44E-02**		NA
K	<4.51E-01	<4.43E-01	<4.81E-01	**<4.58E-01***		NA
La	<1.11E-02	<1.08E-02	<1.09E-02	**<1.09E-02***		NA
Li	<4.43E-02	<4.48E-02	<4.38E-02	**<4.43E-02**		NA
Mg	2.03E-01	1.71E-01	1.91E-01	**1.88E-01**	*1.62E-02*	NA
Mn	1.96E-02	1.96E-02	2.08E-02	**2.00E-02**	*6.93E-04*	2.0E-02
Mo	<3.62E-02	<3.66E-02	<3.58E-02	**<3.62E-02**		2.0E-03
Na	1.27E+01	1.27E+01	1.31E+01	**1.28E+01**	*2.31E-01*	NA
Ni	<5.10E-02	<5.30E-02	<5.20E-02	**<5.20E-02**		4.5E+00
P	<8.60E-02	<8.71E-02	<8.50E-02	**<8.60E-02**		NA
Pb	<1.28E-01	<1.30E-01	<1.26E-01	**<1.28E-01**		2.0E-02
S	<1.94E+00	<1.96E+00	<1.92E+00	**<1.94E+00**		NA
Sb	<7.02E-02	<7.11E-02	<6.94E-02	**<7.02E-02**		≤1.0E-01
Si	1.99E+01	1.94E+01	1.99E+01	**1.97E+01**	*2.89E-01*	NA
Sn	<2.46E-01	<2.49E-01	<2.43E-01	**<2.46E-01**		NA
Sr	<7.22E-03	<6.90E-03	<7.00E-03	**<7.04E-03***		3.0E-02
Th	<2.31E-02	<2.34E-02	<2.29E-02	**<2.31E-02**		NA
Ti	1.08E-02	1.16E-02	1.57E-02	**1.27E-02**	*2.63E-03*	NA
U	<2.36E-01	<2.39E-01	<2.33E-01	**<2.36E-01**		4.0E-03
V	<1.72E-03	<1.74E-03	<1.70E-03	**<1.72E-03**		NA
Zn	<4.80E-02	<5.55E-02	<4.47E-02	**<4.94E-02***		8.0E-03
Zr	3.28E-02	3.32E-02	3.26E-02	**3.29E-02**	*3.06E-04*	NA
As	<2.61E-04	<2.70E-04	<2.66E-04	**<2.66E-04**		≤5.4E-04
Hg	1.90E-01	2.04E-01	1.94E-01	**1.96E-01**	*7.21E-03*	2.0E-01
Se	<5.23E-04	<5.40E-04	<5.32E-04	**<5.32E-04**		≤1.0E-03

* **Upper limit since reagents were known to contain appreciable elemental impurities and/or blank was greater than 10% of sample value.**

Table 19 Elemental Constituents in Tank 16H- Annulus Composite Sample 2

Analytes	Composite-2 Run-1; wt%	Composite-2 Run-2; wt%	Composite-2 Run-3; wt%	Average; wt%	Stdev.	Detection Limit, wt %
Ag	<3.33E-03	<3.30E-03	<3.31E-03	**<3.31E-03**		7.0E-03
Al	5.95E+00	5.56E+00	5.99E+00	**5.83E+00**	*2.38E-01*	1.0E+00
B	<4.80E-02	<4.76E-02	<4.78E-02	**<4.78E-02**		≤1.6E-02
Ba	1.88E-02	1.74E-02	1.91E-02	**1.84E-02**	*9.07E-04*	6.0E-03
Be	2.96E-04	2.69E-04	2.94E-04	**2.86E-04**	*1.50E-05*	NA
Ca	<6.74E-01	<6.45E-01	<6.52E-01	**<6.57E-01***		NA
Cd	<3.45E-03	<3.42E-03	<3.43E-03	**<3.43E-03**		1.0E-02
Ce	3.17E-02	2.92E-02	3.42E-02	**3.17E-02**	*2.50E-03*	NA
Co	<4.94E-02	<5.87E-02	<5.11E-02	**<5.31E-02***		8.0E-04
Cr	2.66E-02	2.50E-02	2.65E-02	**2.60E-02**	*8.96E-04*	3.0E-02
Cu	1.59E-02	1.88E-02	1.41E-02	**1.63E-02**	*2.37E-03*	3.0E-02
Fe	2.87E+00	2.84E+00	2.97E+00	**2.89E+00**	*6.81E-02*	3.0E-02
Gd	1.61E-02	<1.40E-02	1.41E-02	**≤1.47E-02**		NA
K	<3.69E-01	<3.67E-01	<3.21E-01	**<3.52E-01***		NA
La	<1.57E-02	<1.48E-02	<1.57E-02	**<1.54E-02***		NA
Li	<4.51E-02	<4.47E-02	<4.48E-02	**<4.49E-02**		NA
Mg	1.41E-01	9.56E-02	1.15E-01	**1.17E-01**	*2.28E-02*	NA
Mn	3.02E-02	2.91E-02	3.19E-02	**3.04E-02**	*1.41E-03*	2.0E-02
Mo	<3.68E-02	<3.65E-02	<3.66E-02	**<3.66E-02**		2.0E-03
Na	1.73E+01	1.78E+01	1.77E+01	**1.76E+01**	*2.65E-01*	NA
Ni	<5.00E-02	<5.00E-02	<5.40E-02	**<5.13E-02**		4.5E+00
P	<8.75E-02	<8.67E-02	<8.70E-02	**<8.71E-02**		NA
Pb	<1.30E-01	<1.29E-01	<1.30E-01	**<1.30E-01**		2.0E-02
S	<1.97E+00	<1.96E+00	<1.96E+00	**<1.96E+00**		NA
Sb	<7.15E-02	<7.08E-02	<7.10E-02	**<7.11E-02**		≤1.0E-01
Si	1.04E+01	9.29E+00	1.03E+01	**1.00E+01**	*6.14E-01*	NA
Sn	<2.50E-01	<2.48E-01	<2.49E-01	**<2.49E-01**		NA
Sr	<9.59E-03	<9.07E-03	<9.59E-03	**<9.42E-03***		3.0E-02
Th	<2.35E-02	<2.33E-02	<2.34E-02	**<2.34E-02**		NA
Ti	1.54E-02	1.40E-02	1.56E-02	**1.50E-02**	*8.72E-04*	NA
U	<2.40E-01	<2.38E-01	<2.39E-01	**<2.39E-01**		4.0E-03
V	<1.75E-03	<1.74E-03	<1.74E-03	**<1.74E-03**		NA
Zn	<5.82E-02	<5.73E-02	<5.56E-02	**<5.70E-02***		8.0E-03
Zr	3.95E-02	3.63E-02	3.97E-02	**3.85E-02**	*1.91E-03*	NA
As	<2.53E-04	<2.57E-04	<2.74E-04	**<2.61E-04**		≤5.4E-04
Hg	2.34E-01	2.18E-01	2.34E-01	**2.29E-01**	*9.24E-03*	2.0E-01
Se	<5.06E-04	<5.14E-04	<5.49E-04	**<5.23E-04**		≤1.0E-03

* **Upper limit since reagents were known to contain appreciable elemental impurities and/or blank was greater than 10% of sample value.**

Table 20 Elemental Constituents in Tank 16H Annulus Composite Sample 3

Analytes	Composite-3 Run-1; wt%	Composite-3 Run-2; wt%	Composite-3 Run-3; wt%	Average wt%	Stdev	Detection Limit (wt %)
Ag	<3.29E-03	<3.28E-03	<3.25E-03	<3.27E-03		7.0E-03
Al	6.93E+00	6.88E+00	6.86E+00	6.89E+00	3.61E-02	1.0E+00
B	<4.74E-02	<4.72E-02	<4.69E-02	<4.72E-02		≤1.6E-02
Ba	1.25E-02	1.23E-02	1.22E-02	1.23E-02	1.53E-04	6.0E-03
Be	<2.44E-04	2.67E-04	<2.41E-04	≤2.51E-04		NA
Ca	<2.63E-01	<2.75E-01	<2.89E-01	<2.76E-01*		NA
Cd	<3.41E-03	<3.40E-03	<3.37E-03	<3.39E-03		1.0E-02
Ce	<2.76E-02	<2.75E-02	<2.73E-02	<2.75E-02		NA
Co	<5.11E-02	<4.03E-02	<5.25E-02	<4.80E-02*		8.0E-04
Cr	1.68E-02	1.77E-02	1.74E-02	1.73E-02	4.58E-04	3.0E-02
Cu	1.37E-02	1.53E-02	7.62E-02	3.51E-02	3.56E-02	3.0E-02
Fe	2.32E+00	2.64E+00	2.50E+00	2.49E+00	1.60E-01	3.0E-02
Gd	<5.73E-03	<5.70E-03	<5.66E-03	<5.70E-03		NA
K	<3.93E-01	<3.98E-01	<4.03E-01	<3.98E-01*		NA
La	<6.19E-03	<5.85E-03	<5.73E-03	<5.92E-03*		NA
Li	<4.45E-02	<4.43E-02	<4.40E-02	<4.43E-02		NA
Mg	3.36E-02	3.39E-02	3.76E-02	3.50E-02	2.23E-03	NA
Mn	2.06E-02	2.14E-02	2.14E-02	2.11E-02	4.62E-04	2.0E-02
Mo	<3.64E-02	<3.62E-02	<3.60E-02	<3.62E-02		2.0E-03
Na	1.69E+01	1.56E+01	1.56E+01	1.60E+01	7.51E-01	NA
Ni	<5.20E-02	<5.10E-02	<5.10E-02	<5.13E-02		4.5E+00
P	<8.64E-02	<8.61E-02	<8.54E-02	<8.60E-02		NA
Pb	<1.29E-01	<1.28E-01	<1.27E-01	<1.28E-01		2.0E-02
S	<1.95E+00	<1.94E+00	<1.93E+00	<1.94E+00		NA
Sb	<7.06E-02	<7.03E-02	<6.97E-02	<7.02E-02		≤1.0E-01
Si	1.10E+01	1.11E+01	1.14E+01	1.12E+01	2.08E-01	NA
Sn	<2.47E-01	<2.46E-01	<2.44E-01	<2.46E-01		NA
Sr	<6.35E-03	<6.22E-03	<6.18E-03	<6.25E-03*		3.0E-02
Th	<2.32E-02	<2.31E-02	<2.30E-02	<2.31E-02		NA
Ti	2.35E-02	2.37E-02	2.46E-02	2.39E-02	5.86E-04	NA
U	<2.37E-01	<2.36E-01	<2.34E-01	<2.36E-01		4.0E-03
V	<1.73E-03	<1.72E-03	<1.71E-03	<1.72E-03		NA
Zn	<5.69E-02	<5.97E-02	<6.29E-02	<5.98E-02*		8.0E-03
Zr	2.68E-02	2.62E-02	2.62E-02	2.64E-02	3.46E-04	NA
As	<2.66E-04	<2.58E-04	<2.61E-04	<2.62E-04		≤5.4E-04
Hg	1.94E-01	1.83E-01	1.83E-01	1.87E-01	6.35E-03	2.0E-01
Se	9.68E-04	9.40E-04	7.73E-04	8.94E-04	1.05E-04	≤1.0E-03

. * **Upper limit since reagents were known to contain appreciable elemental impurities and/or blank was greater than 10% of sample value.**

Table 21 Anions Leached per gram of Tank 16H Annulus Composite Sample 1; wt %

Analytes	Run-1	Run-2	Run-3	Average	Std. Dev.	Target Detection Limit
Fluoride, F^{-1}	<2.84E-02	<2.89E-02	<2.84E-02	**<2.86E-02**		1.0E-02
Formate, CHO_2^{-1}	<2.84E-02	<2.89E-02	<2.84E-02	**<2.86E-02**		NA
Chloride , Cl^{-1}	4.26E-02	3.47E-02	3.41E-02	**3.72E-02**	*4.74E-03*	4.0E-02
Nitrite , NO_2^{-1}	6.45E+00	6.11E+00	6.23E+00	**6.26E+00**	*1.74E-01*	2.0E-01
Bromide, Br^{-1}	<2.84E-02	<2.89E-02	<2.84E-02	**<2.86E-02**		NA
Nitrate , NO_3^{-1}	5.31E+00	5.21E+00	5.29E+00	**5.27E+00**	*5.49E-02*	7.0E-01
Phosphate, PO_4^{-3}	<2.84E-02	<2.89E-02	<2.84E-02	**<2.86E-02**		1.0E-02
Sulfate, SO_4^{-2}	4.55E-01	4.23E-01	4.47E-01	**4.41E-01**	*1.67E-02*	9.0E-02
Oxalate, $C_2O4_2^{-}$	6.82E-02	6.37E-02	6.26E-02	**6.48E-02**	*2.98E-03*	NA
Iodine, I-127	<1.71E-05	<1.74E-05	<1.71E-05	**<1.72E-05**		8.0E-01
Iodine, I-129	4.82E-04	3.80E-04	4.57E-04	**4.40E-04**	*5.31E-05*	See I-129
Total Iodine	<4.99E-04	<3.97E-04	<4.74E-04	<**4.57E-04**		NA

Table 22 Anions Leached per gram of Tank 16H Annulus Composite Sample 2; wt %

Analytes	Run-1	Run-2	Run-3	Average	Std. Dev.	Target Detection Limit
Fluoride, F^{-1}	<3.04E-02	<2.69E-02	<3.02E-02	**<2.92E-02**		1.0E-02
Formate, CHO_2^{-1}	<3.04E-02	<2.69E-02	<3.02E-02	**<2.92E-02**		NA
Chloride , Cl^{-1}	6.39E-02	5.11E-02	6.03E-02	**5.85E-02**	*6.58E-03*	4.0E-02
Nitrite , NO_2^{-1}	6.33E+00	5.55E+00	6.18E+00	**6.02E+00**	*4.17E-01*	2.0E-01
Bromide, Br^{-1}	<3.04E-02	<2.69E-02	<3.02E-02	**<2.92E-02**		NA
Nitrate , NO_3^{-1}	4.96E+00	4.52E+00	4.92E+00	**4.80E+00**	*2.41E-01*	7.0E-01
Phosphate, PO_4^{-3}	<3.04E-02	<2.69E-02	<3.02E-02	**<2.92E-02**		1.0E-02
Sulfate, SO_4^{-2}	8.00E-01	7.51E-01	8.06E-01	**7.86E-01**	*3.01E-02*	9.0E-02
Oxalate, $C_2O4_2^{-}$	7.30E-02	6.73E-02	6.03E-02	**6.69E-02**	*6.35E-03*	NA
Iodine, I-127	<1.83E-05	<1.62E-05	<1.81E-05	**<1.75E-05**		8.0E-01
Iodine, I-129	3.68E-04	5.00E-04	4.31E-04	**4.33E-04**	*6.64E-05*	See I-129
Total Iodine	<3.86E-04	<5.16E-04	<4.49E-04	<**4.50E-04**		NA

Table 23 Anions Leached per gram of Tank 16H Annulus Composite Sample 3; wt %

Analytes	Run-1	Run-2	Run-3	Average	Std. Dev.	Target Detection Limit
Fluoride, F^{-1}	<2.95E-02	<2.86E-02	<2.92E-02	**<2.91E-02**		1.0E-02
Formate, CHO_2^{-1}	<2.95E-02	<2.86E-02	<2.92E-02	**<2.91E-02**		NA
Chloride , Cl^{-1}	2.95E-02	3.71E-02	3.50E-02	**3.39E-02**	*3.94E-03*	4.0E-02
Nitrite , NO_2^{-1}	6.11E+00	5.94E+00	6.04E+00	**6.03E+00**	*8.18E-02*	2.0E-01
Bromide, Br^{-1}	<2.95E-02	<2.86E-02	<2.92E-02	**<2.91E-02**		NA
Nitrate , NO_3^{-1}	4.90E+00	4.86E+00	4.90E+00	**4.89E+00**	*2.38E-02*	7.0E-01
Phosphate, PO_4^{-3}	<2.95E-02	<2.86E-02	<2.92E-02	**<2.91E-02**		1.0E-02
Sulfate, SO_4^{-2}	7.11E-01	7.12E-01	7.09E-01	**7.10E-01**	*1.42E-03*	9.0E-02
Oxalate, $C_2O4_2^{-}$	6.79E-02	8.57E-02	8.17E-02	**7.84E-02**	*9.37E-03*	NA
Iodine, I-127	<1.77E-05	<1.71E-05	<1.75E-05	**<1.74E-05**		8.0E-01
Iodine, I-129	5.28E-04	3.57E-04	9.42E-04	**6.09E-04**	3.00E-04	See I-129
Total Iodine	<5.46E-04	<3.74E-04	<9.60E-04	**≥6.27E-04**		NA

Table 24 Radiological Constituents for Tank 16H Annulus Composite Sample 1, µCi/g.

Analytes	Run 1, µCi/g	Run 2 µCi/g	Run 3 µCi/g	Average µCi/g	Stdev	One Sigma %Uncert.	Target Detection, µCi/g
C-14	<7.52E-04	<7.57E-04	<7.52E-04	<7.54E-04		MDA	1.0E-01
Cl-36	<8.78E-04	<6.13E-04	<4.33E-04	<6.41E-04		UL	9.0E-05
K-40	<2.46E-05	<3.74E-05	<3.87E-05	<3.36E-05		MDA	4.0E-05
Co-60	2.01E-03	<3.02E-03	<2.21E-03	≤2.41E-03		23.3/MDA	1.0E-03
Ni-59	<4.64E-02	<6.22E-03	<1.36E-03	<1.80E-02		UL/MDA	5.0E-02
Ni-63	<7.16E-01	<2.91E-01	<7.57E-02	<3.61E-01		UL	1.0E-01
Sr-90	1.40E+03	1.47E+03	1.21E+03	1.36E+03	1.32E+02	13.00	1.0E-03
Y-90	1.40E+03	1.47E+03	1.21E+03	1.36E+03	1.32E+02	13.00	1.0E-03
Zr-93	<1.44E-01	<1.50E-01	<1.48E-01	<1.47E-01		UL	1.0E-04
Nb-94	<3.67E-04	<4.59E-04	<3.87E-04	<4.05E-04		MDA	3.0E-03
Tc-99	2.06E-01	2.24E-01	2.39E-01	2.23E-01	1.65E-02	5.94	1.0E-03
I-129	8.51E-04	6.71E-04	8.06E-04	7.76E-04	9.38E-05	5.00	9.0E-06
Cs-135	2.56E-03	2.61E-03	2.61E-03	2.59E-03	2.89E-05	20	1.0E-04
Cs-137	7.30E+02	7.30E+02	7.34E+02	7.31E+02	2.60E+00	5	1.0E-03
Ba-137m	6.90E+02	6.90E+02	6.95E+02	6.92E+02	2.46E+00	5	1.0E-03
Eu-154	7.57E-01	7.66E-01	7.43E-01	7.55E-01	1.13E-02	5.0	NA
Ra-226	<1.60E-03	<9.32E-04	<1.01E-03	<1.18E-03		MDA	9.0E-04
Th-230	<7.21E-05	<1.86E-04	<1.80E-04	<1.46E-04		UL	1.0E-03
Pa-231	<4.77E-04	<2.22E-04	<3.02E-04	<3.34E-04		DL	9.0E-05
U-233	<2.08E-03	<1.73E-03	<2.10E-03	<1.97E-03		DL	1.0E-03
U-234	1.50E-03	1.57E-03	1.54E-03	1.54E-03	3.15E-05	20	1.0E-03
U-235	2.55E-05	2.56E-05	2.48E-05	2.53E-05	4.09E-07	20	1.0E-05
U-236	5.27E-05	5.36E-05	5.18E-05	5.27E-05	9.01E-07	20	NA
U-238	1.06E-04	1.06E-04	1.04E-04	1.05E-04	1.45E-06	20	5.0E-05
Np-237	1.83E-03	1.80E-03	1.82E-03	1.82E-03	1.41E-05	1.74	1.0E-03
Pu-238	3.45E+00	3.36E+00	3.39E+00	3.40E+00	5.06E-02	5.53	1.0E-03
Pu-239	4.64E-01	4.30E-01	4.24E-01	4.39E-01	2.0E-02	6.04	1.0E-03
Pu-240	2.12E-01	1.97E-01	1.93E-01	2.00E-01	1.0E-02	6.06	1.0E-03
Pu-239/240	6.76E-01	6.26E-01	6.17E-01	6.40E-01	3.15E-02	6.03	NA
Pu-241	1.30E+00	1.30E+00	1.33E+00	1.31E+00	1.71E-02	15.23	1.0E-03
Pu-242	9.41E-05	8.11E-05	8.56E-05	8.69E-05	6.60E-06	7.62	1.0E-03
Pu-244	<1.12E-07	<9.95E-08	<1.12E-07	<1.08E-07		DL	1.3E-04
Am-241	7.52E-01	8.24E-01	7.12E-01	7.63E-01	5.70E-02	5.79	1.0E-03
Am-242m	<3.14E-03	<1.14E-02	<1.16E-03	<5.22E-03		MDA	1.0E-03
Am-243	<3.55E-03	<2.12E-03	<1.13E-03	<2.26E-03		UL/MDA	1.0E-03
Cm-242	<2.59E-03	<9.41E-03	<9.59E-04	<4.32E-03		MDA	NA
Cm-243	<4.59E-03	<4.91E-03	<3.90E-03	<4.47E-03		MDA	2.0E-02
Cm-244	<3.83E-01	<1.52E-01	<1.50E-01	<2.29E-01		UL	1.0E-03
Cm-245	<3.23E-05	<1.48E-05	<1.45E-05	<2.06E-05		UL	2.0E-02
Cm-247	<1.72E-09	<1.36E-09	<1.28E-09	<1.45E-09		UL	1.3E-04
Cm-248	<1.15E-06	<1.25E-06	<9.91E-07	<1.13E-06		UL	1.3E-04
Cf-249	<1.40E-03	<1.50E-03	<1.19E-03	<1.36E-03		MDA	5.0E-03
Cf-251	<3.79E-03	<4.00E-03	<3.16E-03	<3.65E-03		MDA	1.0E-03

Table 25 Radiological Constituents for Tank 16H Annulus Composite Sample 2, µCi/g.

Analytes	Run 1, µCi/g	Run 2 µCi/g	Run 3 µCi/g	Average µCi/g	Stdev.	One Sigma %Uncert.	Target Detection, µCi/g
C-14	<8.33E-04	<7.57E-04	<7.57E-04	**<7.82E-04**		MDA	1.0E-01
Cl-36	<5.59E-04	<4.55E-04	NR	**<5.07E-04**		UL	9.0E-05
K-40	<2.48E-05	<3.78E-05	<3.56E-05	**<3.27E-05**		MDA	4.0E-05
Co-60	2.56E-03	2.80E-03	3.18E-03	**2.85E-03**	*3.16E-04*	26.80	1.0E-03
Ni-59	<1.83E-03	<2.23E-02	<1.89E-02	**<1.43E-02**		UL	5.0E-02
Ni-63	<1.71E-01	<4.36E-01	<2.94E-01	**<3.00E-01**		UL	1.0E-01
Sr-90	2.02E+03	2.13E+03	1.96E+03	**2.04E+03**	*8.46E+01*	13.15	1.0E-03
Y-90	2.02E+03	2.13E+03	1.96E+03	**2.04E+03**	*8.46E+01*	13.15	1.0E-03
Zr-93	<1.91E-01	<1.61E-01	<1.86E-01	**<1.80E-01**		UL	1.0E-04
Nb-94	<4.48E-04	<4.31E-04	<3.86E-04	**<4.22E-04**		MDA	3.0E-03
Tc-99	2.04E-01	2.63E-01	2.91E-01	**2.53E-01**	*4.44E-02*	6.13	1.0E-03
I-129	6.49E-04	8.83E-04	7.61E-04	**7.64E-04**	*1.17E-04*	5.00	9.0E-06
Cs-135	2.70E-03	2.47E-03	2.66E-03	**2.61E-03**	*1.25E-04*	20	1.0E-04
Cs-137	7.48E+02	6.71E+02	7.39E+02	**7.19E+02**	*4.19E+01*	5	1.0E-03
Ba-137m	7.07E+02	6.35E+02	6.99E+02	**6.80E+02**	*3.96E+01*	5	1.0E-03
Eu-154	1.09E+00	9.77E-01	1.08E+00	**1.05E+00**	*6.31E-02*	5.0	NA
Ra-226	<8.15E-04	<9.01E-04	<8.83E-04	**<8.66E-04**		MDA	9.0E-04
Th-230	<4.18E-04	<8.11E-05	<1.10E-04	**<2.03E-04**		UL	1.0E-03
Pa-231	<9.86E-04	<3.86E-04	<6.89E-04	**<6.87E-04**		DL	9.0E-05
U-233	<1.81E-03	<2.29E-03	<1.72E-03	**<1.94E-03**		DL	1.0E-03
U-234	1.75E-03	1.57E-03	1.78E-03	**1.70E-03**	*1.01E-04*	20	1.0E-03
U-235	2.47E-05	2.27E-05	2.51E-05	**2.42E-05**	*1.03E-06*	20	1.0E-05
U-236	5.41E-05	5.09E-05	5.63E-05	**5.38E-05**	*1.96E-06*	20	NA
U-238	1.03E-04	9.46E-05	1.05E-04	**1.01E-04**	*5.08E-06*	20	5.0E-05
Np-237	2.59E-03	2.66E-03	2.50E-03	**2.58E-03**	*8.50E-05*	0.78	1.0E-03
Pu-238	4.27E+00	3.79E+00	4.50E+00	**4.19E+00**	*3.65E-01*	5.77	1.0E-03
Pu-239	5.59E-01	4.95E-01	6.22E-01	**5.59E-01**	*6.0E-02*	6.20	1.0E-03
Pu-240	2.55E-01	2.26E-01	2.82E-01	**2.54E-01**	*3.0E-02*	6.20	1.0E-03
Pu-239/240	8.15E-01	7.21E-01	9.05E-01	**8.14E-01**	*9.24E-02*	6.18	NA
Pu-241	1.85E+00	1.45E+00	1.71E+00	**1.67E+00**	*2.04E-01*	15.20	1.0E-03
Pu-242	1.09E-04	9.46E-05	1.17E-04	**1.07E-04**	*1.14E-05*	7.59	1.0E-03
Pu-244	<1.02E-07	<1.29E-07	<1.04E-07	**<1.12E-07**		DL	1.3E-04
Am-241	9.37E-01	9.10E-01	1.05E+00	**9.67E-01**	*7.66E-02*	5.0	1.0E-03
Am-242m	<1.26E-03	<1.56E-03	<6.53E-04	**<1.16E-03**		MDA	1.0E-03
Am-243	1.19E-02	<1.90E-03	<2.14E-03	**≤5.33E-03**		UL	1.0E-03
Cm-242	<1.04E-03	<1.29E-03	<5.41E-04	**<9.58E-04**		MDA	NA
Cm-243	<5.00E-03	<4.12E-03	<4.95E-03	**<4.69E-03**		MDA	2.0E-02
Cm-244	<1.41E+00	<1.13E-01	<1.82E-01	**<5.67E-01**		UL	1.0E-03
Cm-245	<1.21E-04	<1.05E-05	<1.55E-05	**<4.91E-05**		UL	2.0E-02
Cm-247	<5.45E-09	<6.76E-10	<1.19E-09	**<2.44E-09**		UL	1.3E-04
Cm-248	<9.86E-07	<7.61E-07	<1.34E-06	**<1.03E-06**		UL	1.3E-04
Cf-249	<1.48E-03	<1.25E-03	<1.51E-03	**<1.41E-03**		MDA	5.0E-03
Cf-251	<3.98E-03	<3.28E-03	<3.95E-03	**<3.74E-03**		MDA	1.0E-03

NR: Not reported because data did not meet quality assurance requirements.

Table 26 Radiological Constituents for Tank 16H Annulus Composite Sample 3, µCi/g.

Analytes	Run 1, µCi/g	Run 2 µCi/g	Run 3 µCi/g	Average µCi/g	Stdev.	One Sigma %Uncert.	Target Detection, µCi/g
C-14	<7.57E-04	<8.29E-04	<6.94E-04	**<7.60E-04**		MDA	1.0E-01
Cl-36	<6.71E-04	<5.45E-04	<6.40E-04	**<6.19E-04**		UL	9.0E-05
K-40	<2.56E-05	<3.92E-05	<2.41E-05	**<2.96E-05**		MDA	4.0E-05
Co-60	1.73E-03	1.70E-03	2.27E-03	**1.90E-03**	*3.23E-04*	17.82	1.0E-03
Ni-59	<1.32E-03	<8.78E-03	<1.91E-03	**<4.01E-03**		MDA/UL	5.0E-02
Ni-63	<5.81E-02	<3.98E-01	<7.93E-02	**<1.78E-01**		UL	1.0E-01
Sr-90	1.23E+03	1.33E+03	1.38E+03	**1.31E+03**	*7.82E+01*	12.35	1.0E-03
Y-90	1.23E+03	1.33E+03	1.38E+03	**1.31E+03**	*7.82E+01*	12.35	1.0E-03
Zr-93	<1.33E-01	<1.32E-01	<1.23E-01	**<1.29E-01**		UL	1.0E-04
Nb-94	<5.23E-04	<5.00E-04	<5.90E-04	**<5.38E-04**		MDA	3.0E-03
Tc-99	2.19E-01	3.00E-01	3.00E-01	**2.73E-01**	*4.66E-02*	6.06	1.0E-03
I-129	9.32E-04	6.31E-04	1.66E-03	**1.08E-03**	*5.30E-04*	12	9.0E-06
Cs-135	2.37E-03	2.19E-03	2.29E-03	**2.29E-03**	*8.81E-05*	20	1.0E-04
Cs-137	6.22E+02	6.17E+02	6.13E+02	**6.17E+02**	*4.50E+00*	5	1.0E-03
Ba-137m	5.88E+02	5.84E+02	5.80E+02	**5.84E+02**	*4.26E+00*	5	1.0E-03
Eu-154	7.70E-01	7.12E-01	7.21E-01	**7.34E-01**	*3.15E-02*	5.0	NA
Ra-226	<9.10E-04	<1.49E-04	<6.13E-04	**<5.57E-04**		MDA	9.0E-04
Th-230	<7.52E-05	<5.23E-05	<1.16E-04	**<8.12E-05**		UL	1.0E-03
Pa-231	<2.90E-04	<2.49E-04	<4.15E-04	**<3.18E-04**		DL	9.0E-05
U-233	<4.59E-03	<1.94E-03	<1.61E-03	**<2.72E-03**		DL	1.0E-03
U-234	1.57E-03	1.46E-03	1.40E-03	**1.48E-03**	*8.70E-05*	20	1.0E-03
U-235	2.40E-05	2.26E-05	2.25E-05	**2.30E-05**	*8.48E-07*	20	1.0E-05
U-236	5.45E-05	5.18E-05	5.18E-05	**5.27E-05**	*1.56E-06*	20	NA
U-238	9.41E-05	9.19E-05	9.19E-05	**9.26E-05**	*1.30E-06*	20	5.0E-05
Np-237	1.58E-03	1.48E-03	1.48E-03	**1.51E-03**	*5.70E-05*	1.55	1.0E-03
Pu-238	2.88E+00	2.65E+00	2.65E+00	**2.73E+00**	*1.33E-01*	5.03	1.0E-03
Pu-239	3.51E-01	3.28E-01	3.38E-01	**3.39E-01**	*1.0E-02*	5.76	1.0E-03
Pu-240	1.60E-01	1.50E-01	1.56E-01	**1.56E-01**	5.03E-03	5.85	1.0E-03
Pu-239/240	5.14E-01	4.77E-01	4.95E-01	**4.95E-01**	*1.80E-02*	5.75	NA
Pu-241	1.02E+00	9.05E-01	9.05E-01	**9.43E-01**	*6.50E-02*	11.50	1.0E-03
Pu-242	6.94E-05	6.85E-05	7.03E-05	**6.94E-05**	*9.00E-07*	6.68	1.0E-03
Pu-244	<1.43E-07	<1.02E-07	<1.67E-07	**<1.37E-07**		DL	1.3E-04
Am-241	6.89E-01	5.23E-01	5.50E-01	**5.87E-01**	*8.94E-02*	5.0	1.0E-03
Am-242m	<1.56E-03	<5.86E-04	<2.74E-03	**<1.63E-03**		MDA/UL	1.0E-03
Am-243	<1.20E-02	<1.92E-03	<4.82E-03	**<6.24E-03**		UL	1.0E-03
Cm-242	<1.29E-03	<4.82E-04	<2.27E-03	**<1.35E-03**		MDA/UL	NA
Cm-243	<4.19E-03	<3.05E-03	<3.66E-03	**<3.63E-03**		MDA	2.0E-02
Cm-244	<8.02E-01	<1.71E-01	<1.47E-01	**<3.73E-01**		UL	1.0E-03
Cm-245	<6.71E-05	<1.45E-05	<1.48E-05	**<3.21E-05**		UL	2.0E-02
Cm-247	<3.23E-09	<8.02E-10	<9.46E-10	**<1.66E-09**		UL	1.3E-04
Cm-248	<9.73E-07	<8.60E-07	<8.83E-07	**<9.05E-07**		UL	1.3E-04
Cf-249	<1.16E-03	<9.28E-04	<1.16E-03	**<1.08E-03**		MDA	5.0E-03
Cf-251	<3.21E-03	<2.45E-03	<2.87E-03	**<2.84E-03**		MDA	1.0E-03

Table 27 Primary Liner Sample: Unmet Detection Limit Summary

Radionuclide	Sample 1-P	Sample 2-P	Sample 3-P
K-40	x		x
Ni-59	x		
Ni-63	x		x
Sr-90/Y-90		x	
Zr-93	x	x	x
Cs-137	x	x	
Ba-137m	x	x	
Pa-231	x	x	x
Pu-238	x		
Pu-239		x	
Pu-241	x	x	x
Cm-244	x	x	x
Cf-251	x		x

x: Detection limit not met because analytical results are one or more orders of magnitude higher than target detection limits.

Table 28 Tank 16H Annulus Composite Sample: Unmet Detection Limit Summary

Radionuclide	Composite 1	Composite 2	Composite 3
Cl-36	x	x	x
Zr-93	x	x	x
Pa-231		x	
Cm-244	x	x	x

x: Detection limit not met because analytical results are one or more orders of magnitude higher than target detection limits.

4.0 CONCLUSIONS

Tank 16H (Annulus composite and Primary Liner Samples) samples were analyzed for radiological, elemental and chemical constituents. Where analytical methods yielded additional analytes other than those requested by the customer, these results are also reported.

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 target detection limits were met for the species characterized for Tank 16H (Primary Liner and Annulus composite samples) some were not met. The isotopes whose target detection limits were not met in all cases for both the Primary Liner and Annulus composite Tank 16H samples included non-routine analytical species Zr-93, Cl-36, Pa-231 and Cm-244. For these four radionuclides the detection limits were at least one or two orders of magnitude higher than the target detection limits. In this Tank 16H characterizations, the detection limit for several radionuclides, both non-routine and routine, were about the same order of magnitude as those of the target detection limit. However, for a few of the other non-routine radionuclides the target detection limits were not consistently met even within the same analytical sample groups. It is worth pointing out the Tank 16H Primary Liner Samples did not show much activity and thus radionuclides analysis results were just at their limits of detection. SRNL, in conjunction with the customer, reviewed all of these cases and determined that the impacts of not meeting the target detection limits were acceptable.[xviii] The target detection limits for most of the routine radionuclides were met most of the time.

It is also recommended that other non-radioactive materials such as synthetic nitrated sodalite powder or sodium nitrate salt be considered as potential blanks to be used in the SRNL shielded cell, in place of Tank 8 simulant sludge, as trace radionuclide scavenger in SRNL Shielded Cell air environment during tank sample processing.

A statistical analysis of the Tank 16H samples from the primary vessel and the annulus has been completed. Analytes with all less-than-MDC (minimum detectable concentrations) were summarized by their minimum and maximum MDC's. Analytes with measurements on only a single sample were also summarized in the same fashion. Analytes with measurements on at least two of the three samples were summarized by their mean, standard deviation, percent standard deviation, and their 95% upper confidence limit (UCL95) for the mean concentration.

A significant feature of the sample data was the distinct difference between many of the radionuclide concentrations for Sample 2-P and those for Samples 1-P and 3-P. Nearly all of the radionuclide concentration results for Sample 2-P were reported to be less-than-MDC's. Since no discernible statistical distribution could be identified for the radionuclide concentrations, a conservative nonparametric UCL95 was established for all analytes that fell within this class.

5.0 QUALITY ASSURANCE

The Task Technical and Quality Assurance Plan details the planned activities and associated quality assurance implementing procedures for the characterization of Tank 16H (TTQAP, SRNL-RP-2013-00290, Rev. 1, Jan. 2014). Laboratory Notebook SRNL-NB-2013-00031, L5575-00080 SRNL Electronic Notebook (Production); SRNL, Aiken, SC 29808 (2014) and various AD notebooks contain the analytical/experimental data. Other relevant QA documents include the Technical Task Request (HLE-TTR-2013-00002, Rev 1, December. 30, 2013), Tank 16H Sampling and Analysis Plan-SRR-LWE-2013-00057, Revision 1, May 2013, Tank 16 Annulus-Sample Compositing Determinations- SRR-CWDA-2014-00001, Rev. 0., Liquid Waste Tank Residuals Sampling and Analysis Program Plan, SRR-CWDA-2011-00050, Rev. 2, July 31, 2013 and Liquid Waste Tank Residuals Sampling – Quality Assurance Program Plan, SRR-CWDA-2011-00117 Rev. 1, July 31, 2013.

6.0 REFERENCES

[i] J. P. Pavletich, "*Tank 16 Sample Location Determination Report*." SRR-CWDA-2013-00018, Rev. 0, May 2013.

[ii] J. P. Pavletich,"Tank 16 Annulus-Sample Compositing Determinations" SRR-CWDA-2014-00001, Rev. 0. Jan. 3, 2014.

[iii] Liquid Waste Tank Residuals Sampling and Analysis Program Plan (LWTRSAPP), SRR-CWDA-2011-00050, Rev. 2, July 31, 2013.

[iv] C. D. Walters, "Analyses of the Residual Material Samples Collected from the Tank 16H Primary Tank Floor and Annulus," HLE-TTR-2013-00002, Rev. 1, 12/30/2013.

[v] L. N. Oji, D. P. Diprete, E. P. Shine, C. J. Coleman, "Task Technical and Quality Assurance Plan for Tank 16H Residual Sample Analyses," SRNL-RP-2013-00290, Rev. 1, January, 2014.

[vi] J. P. Pavletich, "Tank 16 Sampling and Analysis Plan" SRR-LWE-2013-00057, Revision 1, May 2013.

[vii] Liquid Waste Tank Residuals Sampling – Quality Assurance Program Plan, SRR-CWDA-2011-00117 Rev. 1, July 31, 2013.

[viii] J. Pavletich, "Analysis of Iron-rich material in Tank 16 Sample 2-P", SRR-CWDA-2013-00089, Rev. 0, June 19, 2013.

[ix] M. S. Hay, S. H. Reboul "Characterization of Tank 16H Annulus Samples" SRNL-STI-2012-00178 Revision 0, April 2012.

[x] 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

[xi] D. Koopman, "Tank 8, Drum 1, sludge simulant, SRTC mobile Lab. ID #20000616," July 26, 2000.

[xii] A0169-00027 (Tom White) SRNL Electronic Notebook (Production)

[xiii] A4300-00063 and A4300-00112 (Mark Jones) SRNL Electronic Notebook (Production)

[xiv] D6971-00059 (Boyd Wiedenman) SRNL Electronic Notebook (Production)

[xv] F7480-00130 and F7480-00143 (Cecilia DiPrete) SRNL Electronic Notebook (Production)

[xvi] L3743-00054 (Leigh Brown) SRNL Electronic Notebook (Production)

[xvii] T6035-00069 (John Young) SRNL Electronic Notebook (Production)

[xviii] Katie-Dara Dixon, "Tank 16 Primary and Annulus Residual Sample Characterization Data - Requested Minimum Target Detection Limits Exceeded" SRR-CWDA-2014-00083, Rev. 0, Augusta 15, 2014.

APPENDIX A-1: Tank 16H Characterization AD Tracking Numbers (LIMS)

Analytes	Method (s)	SRNL AD Tracking Number (LIMS) Primary Liner Sample	SRNL AD Tracking Number (LIMS) Tank 16H-Annulus Composite Sample
Cl-36	Cl-36	300308669-300308678	300311656-300311666
K-40	K-40	300309641-300309651	300311005-300311014
Sr-90	Sr-90	300306859-300306870	300308615-300308621; 300308625-300308626; 300308880-300308885
Pu-238	Pu-238/241	300306859-300306870	Same as for Sr-90
Pu-241	Pu-238/241	300306859-300306870	Same as for Sr-90
Cs-137	GAMMA SPEC	300305186-300305197	Same as for Sr-90
U-233	U-233, U-234, U-235, U-236	300305201-300305203; 300305207-300305209; 300306859-300306862; 300306866-300306870	Same as for Sr-90
U-234	U-233, U-234, U-235, U-236	Same as for U-233	Same as for Sr-90
U-235	U-233, U-234, U-235, U-236	Same as for U-233	Same as for Sr-90
U-236	U-233, U-234, U-235, U-236	Same as for U-233	Same as for Sr-90
U-238	ICP-MS	Same as for U-233	Same as for Sr-90
Co-60	GAMMA SPEC Cs REMOVED	300305186-300305197	Same as for Sr-90
Eu-154	GAMMA SPEC Cs REMOVED	300305186-300305197	Same as for Sr-90
Am-241	Gamma Spec	300305186-300305197	Same as for Sr-90
Pu-239	Pu-242/244	300306859-300306870	Same as for Sr-90
Pu-240	Pu-242/244	300306859-300306870	Same as for Sr-90
Pu-242	Pu-242/244	300306859-300306870	Same as for Sr-90
Pu-244	Pu-242/244	300306859-300306870	Same as for Sr-90
Pu-239/240	Pu-TTA	300306859-300306870	Same as for Sr-90
Ni-59	Ni-59,63	300305198-300305209	300308798-300308809
Ni-63	Ni-59,63	300305198-300305209	300308798-300308809
Tc-99	Tc-99	300305771-300305780	300309578-300309587
I-129	I-129	300305815-300305824	300310588-300310598
I-127	ICP-MS	300305198-300305209	300309089-300309100
Cs-135	Cs-135	300306859-300306870	Same as for Sr-90
C-14	C-14	300309857-300309867	300310039-300310049
Zr-93	Zr-93	300306859-300306870	Same as for Sr-90
Nb-94	Nb-94	300306859-300306870	Same as for Sr-90
Am/Cm	Am/Cm	300307426-300307435	300309747-300309759; 300309761
Ra-226	Ra-226	300309162-300309180	300310741-300310760
Th-230	Th-229/230	300309466-300309484	300310600-300310619
Pa-231	Pa-231	300308299-300308317	300311473-300311491
Np-237	ICP-MS	300305198-300305209	300308798-300308809
Hg	CVAA Hg	300305198-300305209	300308798-300308809
Se	AASe	300305198-300305209	300308798-300308809
As	AASe	300305198-300305209	300308798-300308809
Cations	ICP-MS-PF digestions	300306859-300306870	Same as for Sr-90
Cations	ICP-MS-AQR digestions	300305198-300305209	300308798-300308809
Cations	ICP-ES-AQR digestions	300305198-300305209	300308798-300308809
Cations	ICP-ES-PF digestions	300305189-300305197	Same as for Sr-90
Anions	IC- Leachate analysis	300305210-300305224	300309089-300309100

APPENDIX A-2: Chemical Composition of Analyzed Reference Glass

	Analytical Results for Reference Glass (ARG)	Nominal Recipe for Reference Glass (ARG)#	Percent Relative Deviation
			%RD
Constituent	wt. %	wt. %	
Al	2.53	**2.50**	1.20
B	2.57	**2.69**	4.56
*Ca	1.04	**1.02**	1.94
Fe	9.72	**9.79**	7.20
Li	1.51	**1.49**	1.33
*K	2.27	**2.26**	0.44
Mg	0.53	**0.52**	2.47
Mn	1.43	**1.46**	2.08
*Na	8.53	**8.52**	0.12
*Ni	0.830	**0.827**	0.36
Si	22.9	**22.4**	2.21
Ti	0.68	**0.69**	1.02

* Aqua regia digestion data; all other data from Peroxide fusion.
#Reference values for ARG are reported to the number of digits given in the original citation.

APPENDIX A-3 Chemical Composition for Reference Tank 8 Simulant Sludge

	Analytical Results for Tank 8 Simulant Sludge	Nominal Recipe for Tank 8 Simulant Sludge	Percent Relative Deviation
			%RD
Constituent	wt. %	wt. %	
Al	8.33	**9.28**	10.8
Ba	0.22	**0.20**	9.5
Cr	0.22	**0.22**	0
Cu	0.11	**0.13**	16.7
Fe	21.80	**26.23**	18.4
Mn	2.35	**2.55**	8.2
Ni	2.34	**2.81**	18.3
Si	0.71	**0.89**	22.5
Sr	0.08	**0.09**	11.8
Zn	0.24	**0.27**	11.8

APPENDIX A-4 Barium analyses comparison by two methods (ICP-MS vs. ICP-ES)

Analysis Method for Barium/LIMS #	TK16H 1-P (AQR)	TK16H 2-P (AQR)	TK16H 3-P (AQR)
ICP-MS, Masses 136, 137, 138, ug/g	98.00 ± 40.58	15.28 ± 1.67	70.94 ± 5.74
300305198-300305200			
300305201-300305203			
300305204-300305206			
ICP-ES, ug/g	100.47 ± 40.81	16.10± 2.17	71.77 ± 1.78
300305198-300305200			
300305201-300305203			
300305204-300305206			
%RD	**2.49**	**5.23**	**1.4**

The average percent relative deviation (%RD) for barium concentration by both ICP-ES and ICP-MS methods averages less than 5%.

APPENDIX A-5 Cobalt analyses comparison by two methods (ICP-MS vs. ICP-ES)

Analysis Method for Barium/LIMS #	TK16H 1-P (AQR)	TK16H 2-P (AQR)	TK16H 3-P(AQR)
ICP-MS, Mass 59, ug/g	44.83 ±1.5	44.33 ±3.65	72.63 ±3.25
300305198-300305200			
300305201-300305203			
300305204-300305206			
ICP-ES, ug/g	48.47 ±3.46	48.57 ±1.39	77.40 ±3.20
300305198-300305200			
300305201-300305203			
300305204-300305206			
%RD	**7.80**	**9.13**	**6.36**

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 standard deviation (%RD) for cobalt concentration by both ICP-MS and ICP-ES methods averages less than 10%.

APPENDIX A-6 Technetium-99 analyses comparison by two methods (ICP-MS vs. LSC)

Analysis Method for Tc-99/ LIMS #	TK16H 1- P-1 (PF)	TK16H 1-P-2 (PF)	TK16H 1-P-3 (PF)	Average
ICP-MS : Mass 99, uCi/g	1.30E-01	1.49E-01	1.19E-01	**1.33E-01**
300306860				
300306861				
300306862				
LSC-Tc-99, uCi/g	1.28E-01	1.05E-01	8.74E-02	**1.07E-01**
300305771				
300305772				
300305773				
%RD	1.82	34.75	30.61	**22.39 ± *17.93***

APPENDIX A-7 Technetium-99 analyses comparison by two methods (ICP-MS vs. LSC)

Analysis Method for Tc-99/ LIMS #	TK16H 2- P-1 (PF)	TK16H 2-P-2 (PF)	TK16H 2-P-3 (PF)	Average
ICP-MS : Mass 99, uCi/g	<2.10E-02	<2.12E-02	<2.08E-02	
300306863				
300306864				
300306865				
LSC-Tc-99, uCi/g	5.90E-04	1.50E-03	8.74E-04	**9.88E-04**
300305774				
300305775				
300305776				
%RD	**Not applicable**	**Not applicable**	**Not applicable**	

APPENDIX A-8 Technetium-99 analyses comparison by two methods (ICP-MS vs. LSC)

Analysis Method for Tc-99/ LIMS #	TK16H 3- P-1 (PF)	TK16H 3-P-2 (PF)	TK16H 3-P-3 (PF)	Average
ICP-MS : Mass 99, uCi/g	1.15E-01	1.20E-01	1.24E-01	**1.20E-01**
300306866				
300306867				
300306868				
LSC-Tc-99, uCi/g	1.35E-01	1.73E-01	1.12E-01	**1.40E-01**
300305777				
300305778				
300305779				
%RD	15.92	36.17	10.37	**20.82 ± _13.58_**

APPENDIX A-9 Pu-239 analyses comparison by two methods (ICP-MS vs. Separations)

Analysis Method: Pu-239/ LIMS #	TK 16 ANN. COMP.1-1	TK 16 ANN. COMP.1-2	TK 16 ANN. COMP.1-3	Average
ICP-MS : Mass 239, uCi/g	3.46E-01	3.52E-01	3.46E-01	**3.48E-01**
300308798				
300308800				
300308801				
Separations Pu-239, uCi/g	4.64E-01	4.30E-01	4.24E-01	**4.39E-01**
300308616				
300308617				
300308618				
%RD	29.25	19.82	20.37	**23.13 ± _5.29_**

APPENDIX A-10 Pu-240 analyses comparison by two methods (ICP-MS vs. Separations)

Analysis Method: Pu-240/ LIMS #	TK 16 ANN. COMP.2-1	TK 16 ANN. COMP.2-2	TK 16 ANN. COMP.2-3	Average
ICP-MS : Mass 240, uCi/g	1.90E-01	1.99E-01	1.88E-01	**1.92E-01**
300308801				
300308802				
300308803				
Separations Pu-240, uCi/g	2.55E-01	2.26E-01	2.82E-01	**2.54E-01**
300308619				
300308620				
300308621				
%RD	29.30	12.73	40.22	**27.42 ± *13.84***

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 16H Annulus composite or Primary Liner samples 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 aqua regia.

Sodium Peroxide/Hydroxide Fusions (PF)

Samples were digested according to procedure L16.1, ADS 2502. In a typical digestion, ~2 grams of Tank 16H Annulus composite or Primary Liner sample 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 sodium peroxide fusion.

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

Samples are 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 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.

Gross Alpha/Gross Beta

The solid material was too concentrated to be analyzed directly. Aliquots of peroxide fusion dissolution were added to liquid scintillation cocktail and analyzed for gross alpha and gross beta activity using liquid scintillation analysis. Alpha/beta spillover was determined for each aliquot analyzed, and subsequently used for accurately determining alpha and beta activity, via the addition of a known amount of plutonium to an identical aliquot of each sample.

Ni-59, Ni-63

Aliquots of Tank 16H Primary Liner samples aqua regia dissolutions were 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, tank 8 simulant, ARG, laboratory reagent blanks, a Ni-63 standard and a Ni-59 standard were run as controls. The initial prep had interferences from Sr-90/Y-90 contamination. The protocols were adjusted with the addition of HF to ensure dissolution of any hot particles not digested by aqua regia, and the resin rinse steps were enhanced for the second analysis.

Aliquots of Tank 16H Annulus composite aqua regia dissolution were analyzed for Ni-59, and Ni-63 following the same protocols as used in the Tank 16 Primary's second analysis

Cs-137, Cs-134

Aliquots of Tank 16H Primary Liner samples peroxide fusion and aqua regia dissolutions were analyzed by coaxial high purity germanium gamma-ray spectrophotometers to measure Cs-137 and Cs-134. Due to the unexpectedly low concentrations of Cs-137 in the Tank 16 Primary samples, this method was repeated several times using ever increasing concentrations of dissolution. Cell reagent blanks, tank 8 simulant, ARG glass, and laboratory reagent blanks were run as controls.

Aliquots of Tank 16H Annulus composite peroxide fusion dissolution were analyzed for Cs-137 and Cs-134 following the same protocols as used in the Tank 16 Primary's analysis. However, the annulus sample had much higher levels of Cs-137, and did not require repetitive analyses.

Sr-90

Aliquots of Tank 16H Primary Liner samples 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. Shielded cell reagent blanks, Tank 8 simulant, ARG, laboratory reagent blanks and a Sr-90 standard were run as controls.

Aliquots of Tank 16H Annulus composite samples peroxide fusion dissolution were analyzed for Sr-90 following the same
protocols as used in the Tank 16H Primary Liner sample analysis.

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

Aliquots of Tank 16H Primary Liner samples 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. Due to the unexpectedly low concentrations of analyte in the Tank 16 Primary samples, this method was repeated using a higher concentration of dissolution. High levels of bremsstrahlung from the Sr-90/Y-90 forced a third analysis at a slightly lower concentration. One sample was never analyzed due to high worker dose rates. A hot particle drifting in the sample solution resulted in extremity rates which was as high as 28 mRem/hour.

Aliquots of Tank 16H Annulus composite samples peroxide fusion dissolution were analyzed for Co-60 and Am-241 following the same protocols as used in the Tank 16H Primary Liner sample initial analysis, no additional preparations were required.

Pu-238, 239/240, 241

Aliquots of Tank 16H Primary Liner samples peroxide fusion dissolutions 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. Shielded cell reagent blanks, Tank 8 sludge simulant, ARG, laboratory reagent blanks and a Pu-238 standard were run as controls.

Aliquots of Tank 16H Annulus composite samples peroxide fusion dissolution were analyzed for Pu-238, 239/240, 241 following the same protocols as used in the Tank 16H Primary Liner samples initial analysis.

Pu-239, 240, 242, 244

The plutonium from aliquots of Tank 16H Primary Liner samples 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 isotopics. The Tank 16 Primary samples had much lower plutonium concentrations than were expected, and as the results of the Pu separations couldn’t be yielded as is typical from the Pu-239/240 result of the TTA analysis. As a result the analysis was repeated with a more concentrated aliquot of dissolution both traced with a Pu-242 tracer as well as not traced. The resulting ICP-MS Pu-239, 240, and Pu-244 results were yielded from the Pu-242 tracer recovery (corrected for any Pu-242 contained in the sample). The Pu-242 value was determined from the analysis of the aliquot run through the procedure with no Pu-242 tracer. The measured Pu-242/Pu-239 ratio was applied to the Pu-239 result obtained from the traced analysis to calculate the Pu-242 quantity. Shielded cell reagent blanks, Tank sludge 8 simulant, ARG, and laboratory reagent blanks were run as controls.

The plutonium from aliquots of Tank 16H Annulus composite samples 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 isotopics. The Tank 16H Annulus composite samples were yielded as is typical from the Pu-239/240 result of the TTA analysis. Shielded cell reagent blanks, Tank 8 sludge simulant, ARG, and laboratory reagent blanks were run as controls.

Am-242m, 243, Cm-243, 244, 245, 247, 248, Cf-249, 251

Aliquot of Tank 16H Annulus composite and Tank 16H Primary Liner samples were digested using a sodium peroxide fusion. Additionally, a matrix blank and matrix blank spiked with Am-241 and Cm-244 were prepared using Tank 8 simulated sludge. The americium, curium and californium species were extracted from aliquots of peroxide fusion using a CMPO/tributyl phosphate commercial resin based solid phase extractant and purified further with a proprietary commercial resin called HDEHP based solid phase extractant. Am-241, 243, Cm-243, 245, 247, Cf-249 and 251 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 passivated, implanted, planar silicon (PIPS) alpha spectrometers. Cm-245, 247 and 248 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, Cm, and Cf results were traced with the Am-241 present in the sample matrix. Shielded cell reagent blanks, Tank 8 simulant, ARG, and laboratory reagent blanks were also run as controls.

Aliquots of Tank 16H Annulus composite samples peroxide fusion dissolution were analyzed for Am-242m, 243, Cm-243, 244, 245, 247, 248, Cf-249, and Cf-251 following the same protocols as used in the Tank 16H Primary Liner samples analysis.

Tc-99

Tank 16H Primary Annulus composite samples were digested in a combination of concentrated nitric and hydrochloric acids. Several matrix blanks were prepared using Tank 8 simulated sludge 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.

Sub-samples of Tank 16H Annulus composite samples were analyzed for Tc-99 following the same protocols as used in the Tank 16H Primary Liner sample analysis.

Ra-226

Tank 16H Primary Liner or Annulus composite samples were digested using a sodium peroxide fusion. Each replicate was prepared in duplicate with the duplicate containing a Ra-224 tracer. Additionally, a matrix blank was prepared using Tank 8 sludge simulant. 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. A Tank 8 sludge simulant blank sample traced with Ra-224 and spiked with Ra-226 was run through the process to serve as a calibration standard. A Tank 8 sludge simulant blank sample traced with Ra-224 and spiked with Ra-226 was run through the process to serve as a control standard.

Sub-samples of Tank 16H Annulus composite were analyzed for Ra-226 following the same protocols as used in the Tank 16 Primary's analysis.

Pa-231

Tank 16H Primary Liner or Annulus composite sub-samples were digested using a sodium peroxide fusion. 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 Tank 8 sludge simulant 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.

Sub-samples of Tank 16H Annulus composite were analyzed for Pa-231 following the same protocols as used in the Tank 16H Primary Liner sample analysis.

I-129

Tank 16H Primary Liner or Annulus 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 Tank 8 simulated sludge. The samples were rendered caustic, and decontaminated with strikes with crystalline silicotitanate (CST) and monosodium titanate (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 is added to the solution to precipitate the iodine as AgI, which is 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.

Sub-samples of Tank 16H Annulus composite samples were analyzed for I-129 using the same protocols as used in the Tank 16H Primary Liner sample analysis.

C-14

Solid Tank 16H Primary Liner or Annulus composite sample material was used for the C-14 separation and analysis. The 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.

Sub-samples of Tank 16H Annulus composite samples were analyzed for C-14 using the same protocols as used in the Tank 16H Primary sample analysis.

Th-230

Tank 16H Primary liner or Annulus composite samples were digested using a sodium peroxide fusion. 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 Tank 8 sludge simulant. 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 Th-229 tracer yields were used to correct the various analytes analyses for any thorium losses from the radiochemical separations.

Sub-samples of Tank 16H Annulus composite samples were analyzed for Th-230 using the same protocols as used in the Tank 16H Primary Liner sample analysis.

Nb-94

Aliquots of Tank 16H Primary liner samples peroxide fusion dissolution were spiked with a stable Nb carrier then purified by anion exchange. The purified aliquots were analyzed by high purity germanium spectrometers to measure Nb-94. The stable Nb recoveries were determined using ICP-MS. The Nb-94 values were corrected with the stable Nb recoveries. Shielded cell reagent blanks, Tank 8 sludge simulant, ARG, and laboratory reagent blanks were run as controls.

Aliquots of Tank 16H Annulus composite samples peroxide fusion dissolution were analyzed for Nb-94 using the same protocols as used in the Tank 16H Primary Liner sample analysis.

Zr-93

Aliquots of Tank 16H Primary Liner samples peroxide fusion dissolution were spiked with a stable Zr carrier. The Zr was then extracted from aliquots of peroxide fusion dissolution 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. Shielded cell reagent blanks, Tank 8 simulant, ARG, and laboratory reagent blanks were run as controls.

Aliquots of Tank 16H Annulus composite samples peroxide fusion dissolution were analyzed for Zr-93 using the same protocols as used in the Tank 16H Primary Liner sample analysis.

Cs-135

Aliquots of Tank 16H Primary Liner sample dissolved material (alkali fusion digestion) were purified using a solvent-solvent caustic side solvent extraction-based (CSSX) extraction system. The purified Cs-containing aliquots were analyzed using ICP-MS to measure Cs-135 masses. Cs-137 was measured in the purified Cs-containing aliquots by gamma spectrometry. Cs yields were determined by ratioing the Cs-137 concentrations measured in the purified aliquots to the Cs-137 concentrations previously measured on dissolutions of the Tank 16H Primary Liner sample. The Cs yield was applied to the Cs-135 masses measured to determine the Cs-135 mass concentrations. The Cs-135 result was then converted from ug/g to uCi/g using the specific activity of Cs-135. All results from the Tank 16H Primary Liner sample were upper limits due to the residual Ba-135 contamination observed in the blanks.

Aliquots of Tank 16H Annulus composite samples dissolved material (alkali fusion digestion) were purified using a solvent-solvent caustic side solvent extraction-based (CSSX) extraction system. The purified Cs-containing aliquots were analyzed using ICP-MS to measure Cs-135/Cs-133 mass ratios. The Cs-133 and the Ba corrected Cs-135 ratios from the aliquots of separated material were used along with the associated Cs-133 ICP-MS result from the analysis of non-separated material to obtain a value for Cs-135. The Cs-135 result was then converted from ug/g to uCi/g using the specific activity of Cs-135. Unlike the Tank 16H Primary Liner sample, all of the16H Annulus composite samples had measurable levels of Cs-135.

Cl-36

Sub-samples of Tank 16H Primary Liner sample solid material were weighed and then digested in concentrated acid. The dissolutions were subjected to numerous resin based decontamination steps. Chlorine was then separated from the non-volatile components of the matrix via AgCl precipitation. The precipitate was then counted using a gas flow proportional counter (GFPC) analysis. The AgCl precipitate was then activated by neutron activation analysis to determine Cl losses during the processes, and to correct Cl-36 results for those losses. The HCl used to initially digest the samples was used to trace Cl-36 throughout the processes.

Sub-samples of Tank 16H Annulus composite samples were analyzed for Cl-36 using the same protocols as used in the Tank 16H Primary sample analysis. However, interferences were too high in the initial Annulus Cl-36 analysis, and the analysis was repeated with enhanced decontamination steps.

K-40

Large aliquots of Tank 16H Primary liner solids samples were weighed out into 360 degree beta shielded bottles. A blank bottle of similar design was also prepared. The shielded samples were then analyzed directly on a large high purity germanium spectrometer. The spectrometer was calibrated using a K-40 standard contained in a 360 degree beta shielded bottle. For Tank 16H Primary Liner samples 1-P and 3-P, the Sr-90/Y-90 bremsstrahlung field paralyzed the detector. The samples had to be counted through photon shields constructed of tungsten shot. The Tank 16H Primary Liner 2-P sample could be counted directly on the detector.

Tank 16H Annulus composite samples were far too radioactive to be analyzed in the same fashion as the K-40 analyses on the Primary samples. Annulus composite sub-samples were digested using a sodium peroxide fusion. The dissolution was then treated with a series of decontamination steps designed to remove Cs-137, Sr-90, Y-90, Am-241 and the lanthanides. A K-40 calibration standard, a K-40 control standard, a shielded cell reagent blank and a lab reagent blank were run through the process. In the initial attempt, KI was used as the K-40 standard. The sodium peroxide fusion rendered the KI into an insoluble solid, whereas all samples dissolved completely. The process was repeated using potassium carbonate as the standard, which behaved in a similar fashion as the samples. The treated samples were then analyzed directly on a large high purity germanium spectrometer. The spectrometer was calibrated using the K-40 calibration standard.

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

Uranium was extracted from aliquots of Tank 16H Primary Liner samples 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. For the Tank 16H Primary Liner sample 2-P, no U-238 was observed in the initial ICP-MS analysis above detection limits. For sample 2-P run 2, the U-233, U-234, U-235, U-236, and U-238 detection limits from that initial ICP-MS analysis off the dissolution was reported.

Aliquots of Tank 16H Annulus composite samples peroxide fusion dissolution were analyzed for U-233, U-234, U-235, and U-236 using the same protocols as used in the Tank 16 Primary Liner sample analysis 1-P and 3-P.

Weight Percent Solids Measurement

The weight percent total solids in each Tank 16H sample were measured in the Shielded Cells using a conventional drying oven at 110 °C. An aliquot of each composite sample was placed in a 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% or 10% sodium chloride salt solution prepared by dissolving 5 or 10 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 there was not much water touching the spatula. 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} the water mass was considered equal to cup volume.

The bulk densities of the “as-received” granular tank solids or homogenized samples were individually measured 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 phase of the cup. 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 B1: **Analytical Narratives**

Tank 16 Primary	**Re-preparations due to matrix issues**	**Comments on issues from sample matrix**
Ni-59/63	y	Samples appeared to have particulate Sr-90 contamination hanging up on the column, added HF step to complete digestion and made the rinse steps more robust.
Cs-137/134	yx2	Tank samples were unexpectedly low in Cs-137. More concentrated aliquots prepared until Cs-137 was observed, unfortunately hit Cells contamination levels at that point, rendering some of the results upper limits.
Sr-90	n	Position 2 was orders of magnitude lower in activity than position 1 and 3, analysis returned a detection limit.
Cs Removed Gamma	yx2	Samples lower in activity than expected, one sample had a hot particle issue (dose rates as high as 28mRem/hr observed). That sample was discarded. A new job specific rad-worker permit (JSRWP) was written for Tank 16 analyses. Re-preparations had high dead time from Bremsstrahlung due to high levels of Sr-90/Y-90 mixed with rust in Tank 16 primary, had to dilute the 2nd preps to a degree to be able to measure anything with the HPGe detectors.
Pu-238, Pu-239/240, Pu-241	n	Samples unexpectedly low in plutonium, which resulted in large numbers of upper limit values. Plutonium data ultimately came from the Pu-242,244 run.
C-14	y	Method modified from what was done for Tank 5 and 6 to be run in labs in shielded bottles, shielded bottles too large for ziploc containment bags, J-sealed bags didn't contain gas, samples rerun with larger ziploc bags subsequently procured.
Zr-93	n	Simulants have large amounts of Nb present, causing measured tank 16 Zr-93 results to become bounding upper limit values.
Cs-135	n	Sample activity low for waste tank sample. Trace levels of Ba became an issue resulting in upper limit results ranging from below to within a factor of 2 of target. Ironically, highest value was from simulants which were high in interfering Ba.
K-40	n	As samples had little Cs-137, the usual dissolution/decontamination steps were not used. 5g Samples were weighed out into 4pi beta shielded bottles. Was adequate for position 2. Position 1 and 3 had such high levels of Sr-90 mixed with rust, large Bremsstrahlung field was generated. Detector dead times approached 100 percent. Positions 1 and 3 samples were then shielded through Tungsten and assayed. Tungsten turned out to have high K-40 background, raising detection limits beyond what was expected for the reduced efficiency, hurting the sensitivity of the analysis.
U-233, 234, 235, 236 (U Isotopics)	n	Due to the unexpectedly low levels of U in the primary, Tank 8 simulant had higher levels of U-238 than Tank 16 samples, but lower levels of U-233,4,5, and 6 than positions 1 and 3. This allowed for reporting of actual values rather than upper limits for those isotopes. Data calculated for position 2 off the straight ICP-MS run as no U-238 was observed above detection limits in the direct analysis. U-238 values are required to yield the U separation.
Pu-242, 244 (Pu Isotopics)	y	Due to the unexpectedly low levels of Pu in the primary, no Pu-239/240 above detection limits was available for tracing from the Pu-238/241 method. Data

		initially calculated for position 2 off the straight ICP-MS run as it was superior to the data from the 1st Pu-242,244 run. A New Brunswick Laboratory Pu-242 tracer solution was prepared, a second Pu-242, 244 with MS analysis was conducted using the Pu-242 tracer; data for all three positions was reported.
Tank 16 Annulus	**Re-preparations due to matrix issues**	**Comments on issues from sample matrix**
Zr-93	n	Simulants have large amounts of Nb present, causing measured tank 16 annulus Zr-93 results to become bounding upper limit values, next tank analyses will not use these simulants as the blanks for the Zr analyses
Cl-36	y	Method used for primary and for Tank 5 and 6 had to be made more robust for the annulus to clean up interferences
K-40	y	K-40 standard (KI) formed insoluble salts in peroxide fusion, prep altered to use potassium carbonate

APPENDIX C: Chain –Of-Custody Forms

CST Sample Manual

WORKING COPY
VERIFIED TO BE LATEST REVISION
BY: [signature] DATE 5/14/13

Manual:	SW11.1-SAMPLE
Section:	7.18
Revision:	0
Date	4/23/13
Page:	1 of 3

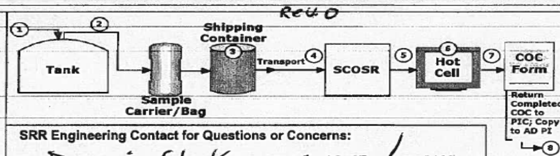
7.18 Chain of Custody

Liquid Waste Tank Residuals Sampling: CHAIN-OF-CUSTODY # 16 – 14/5/13
(Tank # — Date [DD/MM/YY])
(Waste Characterization for tanks listed in ERD Table G-6)

Page ____ of ____

TANK # 16 | **TSAP, Rev #** SRR-LWE-2013-00057 Rev. 0 | **Analyses in TTR, Rev #** HLE-TTR-2013-00002 Rev 0 | **Receiving 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:
Dennis Clark 208-1347 / 14432
(Name/Phone/Pager)

PIC: Mike Harrell / Closure Ops 2-2627 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	
i-P	5/14/13	1130	Mike Harrell	S	O	5/14/13	1345	Tank 16 1-P	13066
						N/A			

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

CST Sample Manual

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7.18 Chain of Custody, Cont'd

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	
				N/A					

	Signature	Printed Name	Organization	Date	Time
Relinquished By (PIC):		Mike E. Harrell	Closure Ops	5/14/13	1306
Received By:		Bonnie Rozier	OPS	5/14/13	1307
Relinquished By:		Bonnie Rozier	OPS	5/14/13	1440
Received By:		SCOTT REBOUL	SRNL	5/14/13	1444
Relinquished By:		SCOTT REBOUL	SRNL	5/15/13	1411
Received By:		LAWRENCE OJI	SRNL	5/15/13	1412
Relinquished By:					
Received By:					

Notes: Approximate weight of Sample is 260g (LNO)

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

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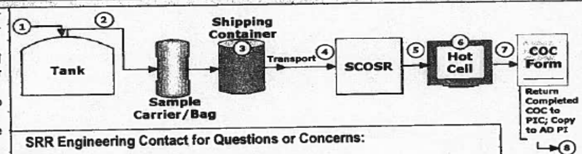
7.18 Chain of Custody

Liquid Waste Tank Residuals Sampling: CHAIN-OF-CUSTODY # 16 20/05/13 (Tank # — Date [DD/MM/YY]) Page 1 of 1

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

TANK # 16 **TSAP, Rev #** SRR-LWE-2013-00057 R0 **Analyses in TTR, Rev #** HLE-TTR-2013-00002 R0 **Receiving 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:

DENNIS CLARK 803 646 0915- 14432
(Name/Phone/Pager)

PIC: Terry Wooda?? Closure 803 645 5270
(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 #
2-P	5/20/13	1520	Terry Wooda??	S	other (vacuum)	5/22/13	14.08	Tank 16H; 2-P	13075
					N/A				

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

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7.18 Chain of Custody, Cont'd

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 #
					N/A				

	Signature	Printed Name	Organization	Date	Time
Relinquished By (PIC):		Terry Wooda??	Closure	5/21/13	1155
Received By:		LENWOOD SCOTT	INTEGRATION	5/21/13	1200
Relinquished By:		Bonnie Rozier	INTEGRATION	5/21/13	1440
Received By:		MAURICE LEE	SCO	5/21/13	1449
Relinquished By:		MAURICE LEE	SCO	5/22/13	0828
Received By:		LAWRENCE OJI	SRNL	5/22/13	0830
Relinquished By:					
Received By:					

Notes: Tank 16H, Sample 2-P, was fairly dry when opened. Amount of sample in the sampler was about 270 grams LNO.

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

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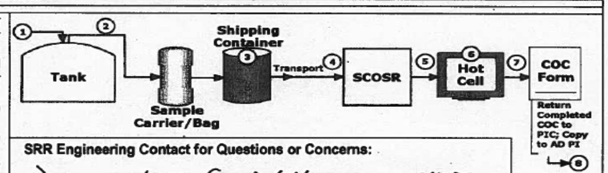
7.18 Chain of Custody

Liquid Waste Tank Residuals Sampling: CHAIN-OF-CUSTODY # 16 23/05/13 (Tank # — Date [DD/MM/YY]) Page 1 of 1
(Waste Characterization for tanks listed in ERD Table G-6)

TANK # 16 | TSAP, Rev # SRR-LWE-2013-00057 R0 | Analyses in TTR, Rev # HLE-TTR-2013-00002 R0 | Receiving 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
2. equipment vessel. 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
3. and the custodian until the samples are shipped to the laboratory. When the samples are shipped, the PIC transfers custody to the person accompanying the samples to
4. the Shielded Cells Operations Sample Receiving (SCOSR) facility. Custody is transferred to the SCOSR Sample Custodian, and they enter the receipt into the Radioactive
5. Material Receipt Entry Log. Those log entry numbers are added to the COC. A copy of the COC is made and returned to the PIC. The original COC is retained by SCOSR for
6. completion as described in Steps 6 and 7. 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
7. Contact is immediately notified of any labeling or sample condition anomalies. The SRNL personnel verifying the sample identifications will complete that section of the COC, **make a**
8. **copy, and return the original COC to the PIC.** 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: DENNIS CLARK (803) 646-0915 14432 (Name/Phone/Pager)

PIC: Terry Woodruff Closure (803)645-5290 (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 #
3-P	5/20/13	1218	Terry Woodruff	S	OTHER Vacuum	5/30/13	1040	Tank 16, 3-P	13083
					N/A				

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

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7.18 Chain of Custody, Cont'd

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 #
					N/A				

	Signature	Printed Name	Organization	Date	Time
Relinquished By (PIC)		Terry Woodruff	Closure	5/29/13	1115
Received By:		Lanwood Scott	Integration	5/29/13	1115
Relinquished By:		Lanwood Scott	Integration	5/29/13	1118
Received By:		Bonnie Rozier	Integration	5/29/13	1120
Relinquished By:		Bonnie Rozier	Integration	5/29/13	1355
Received By:		DJ Wheeler	SCO	5/29/13	1400
Relinquished By:		DJ Wheeler	SCO	5/29/13	1600
Received By:		Lawrence OJI	SRNL	5/29/13	1600

Notes: Sample is fairly dry and no flaky. wt of sample = 155 grams.

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

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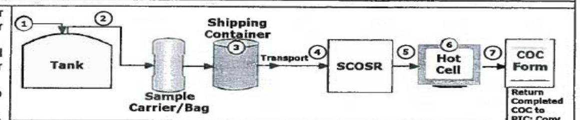
7.18 Chain of Custody

Liquid Waste Tank Residuals Sampling: CHAIN-OF-CUSTODY # 16 — 10/06/13 (Tank # — Date [DD/MM/YY]) Page 1 of 2

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

TANK # 16 | TSAP, Rev # SRR-LWE-2013-00057 Rev 0 | Analyses in TTR, Rev # HLE-TTR-2013-00002 Rev 0 | Receiving 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: DENNIS CLARK 208-1347 14432 (Name/Phone/Pager)

PIC: Terry Woodruff Closure 645-5270 (Name/Organization/Phone)

Sample ID (see TSAP)	Sample Collection Date	Time	Collector	Sample Type *	Sample Container *	SCO or AD Sample ID Verifier Date	Time	ID Verifier	SRNL Radioactive Material Receipt Entry Log #
3-A	7/8/13	1007		S	O-Vacuum	8/19/13	1430		13146
4-AD	8/6/13	1048		S	O-Vacuum	8/19/13	1430		13146
2-A	7/11/13	0825		S	O-Vacuum	8/19/13	1430		13146
1-AD	8/13/13	1123		S	O-Vacuum	8/19/13	1430		13146
5-AD	8/13/13	0932		S	O-Vacuum	8/19/13	1430		13146

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

PWB 8/19/13

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

	Signature	Printed Name	Organization	Date	Time
Relinquished By (PIC):		Terry Woodruff	Closure	8/15/13	1316
Received By:		Doug Dowos	Integration	8/15/13	1316
Relinquished By:		Doug Dowos	Integration	8/19/13	1430
Received By:		Phyllis Burkhalter	ROD	8/19/13	1431
Relinquished By:		Phyllis Burkhalter	ROD	8/20/13	0910
Received By:		LAWRENCE OJI	SRNL	8/20/13	0910
Relinquished By:					
Received By:					

Notes: Sample containers may be opened on 8/21/13. As of 8/27/13 all five Tank 16 annulus samples were opened. Sample 1-AD contained 51 grams of materials, Sample 2-A contained 147 grams of material, Sample 3-A contained 240 grams of materials, Sample container for 4-AD was empty and Sample 5-AD contained 31 grams of material.

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

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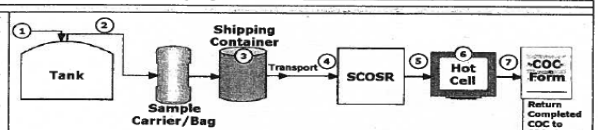
7.18 Chain of Custody

Liquid Waste Tank Residuals Sampling: CHAIN-OF-CUSTODY # 16 – 09/08/13 (Tank # — Date [DD/MM/YY]) Page 1 of 2

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

TANK #	16	TSAP, Rev #	SRR-LWP-2013-00037 Rev 0	Analyses in TTR, Rev #	HLE-TTR-2013-00002 Rev 0	Receiving 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
② equipment vessel.
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
③ and the custodian until the samples are shipped to the laboratory.
When the samples are shipped, the PIC transfers custody to the person accompanying the samples to
④ the Shielded Cells Operations Sample Receiving (SCOSR) facility.
Custody is transferred to the SCOSR Sample Custodian, and they enter the receipt into the Radioactive
⑤ Material Receipt Entry Log. Those log entry numbers are added to the COC.
A copy of the COC is made and returned to the PIC. The original COC is retained by SCOSR for
⑥ completion as described in Steps 6 and 7.
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
⑦ Contact is immediately notified of any labeling or sample condition anomalies.
The SRNL personnel verifying the sample identifications will complete that section of the COC, **make a**
⑧ **copy, and return the original COC to the PIC.**
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:

Dennis Clark 208-1347 14432
(Name/Phone/Pager)

PIC: Terry Woodruff Closure 645-5270
(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 #
8-A	8/9/13	1145	Terry Woodruff	S	O-Vacuum	9/4/13	1030	Tank ~~8-6~~ Sample 8-A	13152
7-AD	8/9/13	1146	Terry Woodruff	S	O-Vacuum	9/4/13	900	Tank16 Sample 7-AD	13152
6-A	8/14/13	1040	Terry Woodruff	S	O-Vacuum	9/4/13	825	Tank16 Sample 6-A	13152
10-A	8/20/13	1100	Terry Woodruff	S	O-Vacuum	9/9/13	1400	Tank16 Sample 10-A	13152
11-AD	8/21/13	1125	Terry Woodruff	S	O-Vacuum	9/11/13 (9/11/13)	800	Tank16 Sample 11-AD	13152

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

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7.18 Chain of Custody, Cont'd

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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 #
9-AD	8/23/13	1000	Terry Woodruff	S	O-Vacuum	9/9/13	930	Tank Sample 9-AD	13152

	Signature	Printed Name	Organization	Date	Time
Relinquished By (PIC		Terry Woodruff	Closure	8/27/13	0911
Received By:		L.M. Newman	OPS	8/27/13	0911
Relinquished By:		L.M. Newman	OPS	8/27/13	1047
Received By:		P.U. Burckhalter	PUB	8/27/13	1050
Relinquished By:		P.U. Burckhalter	PUB	8/27/13	1050
Received By:		LAWRENCE OJI	SRNL	8/27/13	1050
Relinquished By:					
Received By:					

Notes: Sample 6-A was empty. Sample container ~~#~~ 7-AD contained approx. 5 g material. Sample container 8-A is empty. Sample 9-AD contained 146 grams of materials (damp). Sample 10-A contained about 75g; Sample 11-AD contained about 50 grams (accounted after air drying).

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

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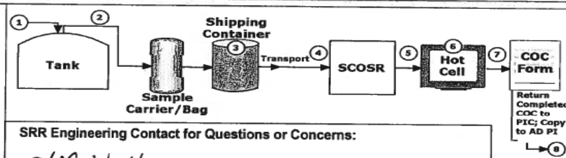
7.18 Chain of Custody

Liquid Waste Tank Residuals Sampling: CHAIN-OF-CUSTODY # 16 – 18/11/13 (Tank # — Date [DD/MM/YY])
(Waste Characterization for tanks listed in ERD Table G-6)
Page 1 of 1

TANK #	16	TSAP, Rev #	SRR-CWDA-2013-00057 Rev 0	Analyses in TTR, Rev #	HLE-TTR-2013-00002 Rev 0	Receiving Laboratory:	SRNL

① ② 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.
③ 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.
④ When the samples are shipped, the PIC transfers custody to the person accompanying the samples to the Shielded Cells Operations Sample Receiving (SCOSR) facility.
⑤ 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.
⑥ 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.
⑦ 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.
⑧ 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**.
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:
Clif Walters 208-1347 No PAGER
(Name/Phone/Pager)

PIC: Terry Woodruff OPS 645-5270
(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 #
6-AR	11/19/13	1017	Terry Woodruff	S	O-Vacuum				13184
8-AR	11/18/13	1356	Terry Woodruff	S	O-Vacuum				↓
					N/A				

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

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7.18 Chain of Custody, Cont'd

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 #
				N/A					

	Signature	Printed Name	Organization	Date	Time
Relinquished By (PIC):		Terry Woodruff	OPS	11/21/13	1300
Received By:		Justin Safford	OPS	11/21/13	1300
Relinquished By:		Justin Safford	OPS	11/21/13	1455
Received By:		Phyllis Burkhalter	SCO	11/21/13	1455
Relinquished By:		Phyllis Burkhalter	SCO	11/22/13	1400
Received By:		LAWRENCE OJI	SRNL	11/22/13	1400
Relinquished By:					
Received By:					

Notes: Tanks 16 Annulus samples: 8-AR and 6-AR (1) Wt of sample 6-AR "As-received" = 292 g total.
Date samples opened: 11/25/13 (2) wt of sample 8-AR "As-received" = 434 g total.

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

APPENDIX D: Statistical Methods for Tank 16H Samples

1.0 INTRODUCTION

Sampling has been completed for the characterization of the residual material on the floor of the primary tank and in the annulus of Tank 16H in the H-Area Tank Farm at the Savannah River Site (SRS), near Aiken, SC. The sampling was performed by Savannah River Remediation LLC (SRR), and the analytical characterization of the samples was performed by the Savannah River National Laboratory (SRNL). This appendix describes the statistical methodology used to compute summary statistics for the statistical analyses. The procedures and the summary metrics reported for each analyte depend on the type of analytical results that are reported.

2.0 OBJECTIVE AND SCOPE

The objective of this appendix is to document the methods used for a statistical analysis of the chemical and isotopic concentration results for the residual material on the floor of the primary tank and in the annulus of Tank 16H. The approach uses representative samples of the residual material from the target region of the tank (either the floor of the primary tank or the annulus) to estimate the mean concentrations of analytes in the remaining residual material. The concentration results are summarized by the means and standard deviations of the sample concentrations. Upper 95% confidence limits (UCL95s) are calculated for the actual mean concentration of each analyte.

The statistical analyses are applied to a subset of the measured analytes. Table D1 lists the analytes that have been statistically analyzed. The concentration data for the analytes are presented in Appendix E Tables E2 through E11 for the primary tank and Appendix F Tables F5 through F13 for the annulus. Each appendix begins with a summary of the statistical applications, followed by a set of data tables and a set of statistical analysis tables, and concludes with a summary.

The residual material in the primary tank and the annulus of Tank 16H were both sampled. Three discrete samples were collected from the primary tank and analyzed in triplicate. For the annulus, three composite samples, using five separate annulus samples for each composite, were also analyzed in triplicate. The same measurement error model applies to the case of measurements taken directly on discrete samples and the case of measurements taken on composite samples. Since the same discussion of statistical methods is applicable to both cases, the words "discrete" and "composite" will be dropped, and will simply be said to come from "samples" in Appendix D.

Table D1. Tank 16H Constituents by Class for Statistical Evaluation

Class	Analytes		
Physical Parameters	Air Dried & Homogenized wt%	Homogenized Bulk Density	"As Received" Bulk Density♣
Radionuclides	Am-241	Am-242m	Am-243
	Ba-137m	C-14	Cf-249
	Cf-251	Cl-36	Cm-242
	Cm-243	Cm-244	Cm-245
	Cm-247	Cm-248	Co-60
	Cs-135	Cs-137	Eu-154
	I-129	K-40	Nb-94
	Ni-59	Ni-63	Np-237
	Pa-231	Pu-238	Pu-239
	Pu-239/240	Pu-240	Pu-241
	Pu-242	Pu-244	Ra-226
	Sr-90	Tc-99	Th-230
	U-233	U-234	U-235
	U-236	U-238	Y-90
	Zr-93		
Elementals	Ag	Al	As
	B	Ba	Cd
	Co	Cr	Cu
	Fe	Hg	Mn
	Mo	Na	Ni
	Pb	Sb	Se
	Si	Sr	U
	Zn		
Anions	Bromide, Br^{-1}	Chloride , Cl^{-1}	Fluoride, F^{-1}
	Formate, CHO_2^{-1}	Iodine, I-127	Iodine, I-129
	Nitrate , NO_3^{-1}	Nitrite , NO_2^{-1}	Oxalate, $C_2O_4^{-2}$
	Phosphate, PO_4^{-3}	Sulfate, SO_4^{-2}	Total Iodine

♣ Primary tank only.

3.0 STATISTICAL METHODS

The concentration data in Appendices E and F are organized by class of analyte: physical parameters, radionuclides, elementals, and anions. Concentration results that are above their minimum detectable concentrations (MDC's) are measurements. Concentration results below their MDC's are unknown values, so that the analyte's concentration is reported as below its MDC. Results below their MDC's are commonly referred to as "less-than-detects" or "less-than-MDC's". Organization of the analytes by these types of results is important because the statistical methods and the reporting of results may differ by category. Each category is further partitioned into (as many as) three categories based on the type of analytical results:

- Analytes that have all results below their MDC's.
- Analytes that have all results above their MDC's.
- Analytes that have a mixture of results below and above their MDC's.

When an analyte has only less-than-MDC results for all of its samples, only the minimum and maximum MDC thresholds are reported. When all results are measurements, a more definitive set of summary metrics can be reported for the concentration data. These include the mean, the standard deviation and percent standard deviation (also called the coefficient of variation) for an individual analyte, and a one-sided upper 95% confidence limit (UCL95) for the actual mean concentration in the targeted area of the tank. The statistical approach to determining these summary metrics depends on the structure and distribution of the data.

The last category, where the concentration data are a mixture of below and above MDC results is considerably more difficult to handle than when all of the analytical results are of the same type. Typically the analytes in this category can be further split into two groups:

- Analytes which have at least some measurements on only one of the three samples.
- Analytes that have measurements on two or more samples.

The sample-to-sample variation is not known for the first group of analytes, since the mean concentration is only determined for one of the samples. Individual UCL95's are computed for each above MDC result (each measurement), and these UCL95's are interpreted as though they are below MDC results. Thus, the complete set of data for each of the analytes in this group is converted to a set of all less-than-MDC results, and the minimum and maximum MDC values are reported. In the most extreme case, there is only one measurement on one sample. No measure of measurement or sampling variation can be determined from this data. When this occurs, a percent standard deviation for the measurement error is assumed to be 20% in this report. This value is assumed because it is larger than nearly all of the percent standard deviations for measurement error for the analytes in this report. This percent standard deviation is used to construct a UCL95 for just the one measurement. Interpreting this UCL95 as a less-than-MDC result, leads to summarizing the results by their minimum and maximum MDC values.

The second group of analytes generally has sufficient information to determine means, standard deviations and percent standard deviations, and UCL95's. When there are at least some measurements on each of the three samples, an approach called maximum likelihood is used to establish estimates for the mean concentration of each of the three samples and provide an estimate of the measurement error standard deviation. The measurement error standard deviation is a quantitative measure of the laboratory measurement repeatability on the same sample. Usually, the normal distribution is assumed when applying the maximum likelihood procedure to the concentration data. The statistical computing procedure "Proc Reliability" in SAS statistical software [1999] is used to compute these results. Additional details can be found in the SAS help files available within the software application. The summary statistics including the UCL95 are based on these three estimated sample means, and are computed in the ProUCL Version 5.0.00 software application by Singh and Singh [2013].

When there are no measurements on one of the three samples for an analyte in the second group, this approach is likely to fail to produce results[1]. When this occurs, a conservative UCL95 can be constructed using a nonparametric Chebyshev approach. Nonparametric simply means that the approach does not assume any particular discernable form for the statistical distribution for the data. This approach attempts to produce the highest-valued Chebyshev UCL95 that is consistent with the available measurements and the pattern of less-than MDC results. The approach sets up a range of plausible values for each of the three samples using the following rules.

[1] A number of radionuclide results for the primary tank displayed this pattern of analytical results. In addition, the sample with all less-than MDC results appeared to have a considerably different concentration level than the other two samples. Therefore, a conservative approach was needed that did not have a strong reliance on any particular statistical distribution.

- If a sample has all measurements, then set the sample concentration to the mean of the three measurements.
- If a sample has all less-than-MDC results, then the sample's mean concentration can be any value between 0 and the mean of its three MDC values.
- If a sample contains measurements and MDC values, then the sample's concentration can be any value between the sum of the measurements (only) divided by 3 and the sum of the measurements and the MDC values divided by three.

Once the plausible range was determined for each of the three sample concentrations, this procedure determines the highest numerical value for the nonparametric Chebyshev UCL95 over all combinations of the plausible range of mean concentrations for the three samples. The Chebyshev UCL95 is described by Singh and Singh [2013].

The statistical analyses for analytes that have all measurements are described here. The statistical measurement error model for a concentration measurement result Y_{ij} is

$$Y_{ij} = \mu + s_i + \varepsilon_{ij}\,, \qquad (0)$$

where Y_{ij} is the *j*-th measured concentration for an analyte in sample *i*, and μ is the actual mean analyte concentration for all of the residual material in the targeted area of the tank. The random effect s_i represents the sampling error for sample *i*, the difference between the actual mean concentration in sample *i* and the actual mean concentration for all of the residual material in the targeted area of the tank. It arises from spatial heterogeneity of the residual material, and sampling, sample preparation, and volumetric proportion errors. The error term ε_{ij} is distributed with mean zero and standard deviation σ, and is the difference between concentration measurement *j* on sample *i* and the actual mean concentration in sample *i*, *i* = 1, 2, 3; *j* = 1, 2, 3.

A test for heterogeneity of measurement variance was performed prior to other analyses in order to verify the assumption that the sample material is well-mixed and the measurement variance σ^2 is the same for all samples. The test procedure is Levene's test with a Type I family-wise error rate $\alpha = 0.05$. Since the typical sample sizes for sampling residual material are small (no more than three measurement results per sample), a Bonferroni procedure, Alt [1982], is used to control for spuriously significant results by dividing the 0.05 family-wise[2] error rate by the number of comparisons for a class of analytes (physical parameters, radionuclides, elementals, and anions) to obtain the Type I error rate per comparison. The Bonferroni criteria for individual analyte tests are α = 0.05/3 = 0.0167 for physical parameters, α = 0.05/2 = 0.025 for anions, α = 0.05/13 = 0.00385 for elementals, and α = 0.05/1 =0.05 for radionuclides in the primary tank of Tank 16H and are α = 0.05/2 = 0.025 for physical parameters, α = 0.05/20 = 0.0025 for radionuclides, and α = 0.05/6 = 0.0083 for anions, and α = 0.05/9 = 0.0056 for elementals in the annulus. If the P-value for an individual constituent test is less than the Bonferroni α per compairison for its class of analytes, then it is concluded that the laboratory variances are not the same for all of the samples.

[2] 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 analytes in the set of all physical parameters, the set of all elemental constituents, the set of all radionuclides, or the set of all anions. Controlling the family-wise error rate means that the probability of making at least one Type I error for individual analytes in a family will be no more than a stated α probability.

An analysis of variance (ANOVA) F test was performed in order to determine whether the random effect s_i is warranted in Eqn (1). If the F test results indicate a statistically significant sampling error s_i at a level of significance $\alpha = 0.05$, then Eqn (1) becomes the basis for estimating the true mean concentration in the residual material; if the ANOVA F test result is not statistically significant at $\alpha = 0.05$, then the random effect s_i is not needed and Eqn (1) reduces to the following measurement error model:

$$Y_{ij} = \mu + \varepsilon_{ij} , \qquad (0)$$

where there is no sampling error term s_i in the model.

If all of the concentration measurements for an analyte are above their minimum detectable concentrations (MDC's), then the ANOVA F test can be performed, and a decision can be made to use the model in Eqn (1) with the random 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 is given by

$$UCL_{95\%} = \overline{Y}_{..} + t_{0.95,2df} \cdot \sqrt{\frac{MS_{Sample}}{9}}, \qquad (3)$$

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

$$MS_{Sample} = \frac{\sum_{i=1}^{3} \frac{Y_{i\cdot}^2}{3} - \frac{Y_{..}^2}{9}}{6}, \qquad (4)$$

and $Y_{i\cdot}$ and $Y_{..}$ are the total of the three measured concentration results for sample *i, i* = 1, 2, 3 and the total of the nine measured concentration results for all three samples, respectively. The estimated standard error of the mean concentration is the square root of the (MS_{Sample}/9) when all samples have three replicate measurements. This procedure is implemented using JMP® Statistical Software from SAS Institute, Inc. [2010] on a platform called "Fit Model". The statistical procedure is called "restricted maximum likelihood analysis" (REML). The residuals, centered on the sample means, are checked for goodness-of-fit by the Wilk-Shapiro test and examined for potential outliers by the Dixon test. Both procedures are implanted in the statistical application: ProUCL Version 5.0.00, available through the U.S. Environmental Protection Agency (US EPA Website). A technical guide by Singh and Singh [2013] is available on the US EPA website. By centering the residuals on the sample means, these checks only investigate issues related to laboratory measurements. Three samples are too few to effectively test the distribution of the samples or check for potential sample outliers.

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

$$UCL_{95\%} = \overline{Y}_{..} + t_{0.95,9-1df} \cdot \sqrt{\frac{s^2}{9}}, \qquad (5)$$

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

The above procedures are appropriate if the data or a transform of the data approximately follow the normal distribution. Figure 7 presents a sequence of goodness-of-fit tests to identify a distribution consistent with the measurement results and to select an estimation method for the mean, standard deviation, and UCL95. Studies by Singh, Singh, and Englehardt [1997] demonstrated 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 a level of significance $\alpha = 5\%$. If the W-S statistic is less than the W-S critical value, then normality is rejected; if there is no statistically significant departure from normality, the mean, 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 skewed distribution. This report adopts the strategy in Singh, and Singh [2013] to test for the gamma distribution prior to the lognormal distribution. The gamma distribution assumption is tested using 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, 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, and UCL95 are estimated assuming a gamma distribution, according to Singh and Singh [2013].

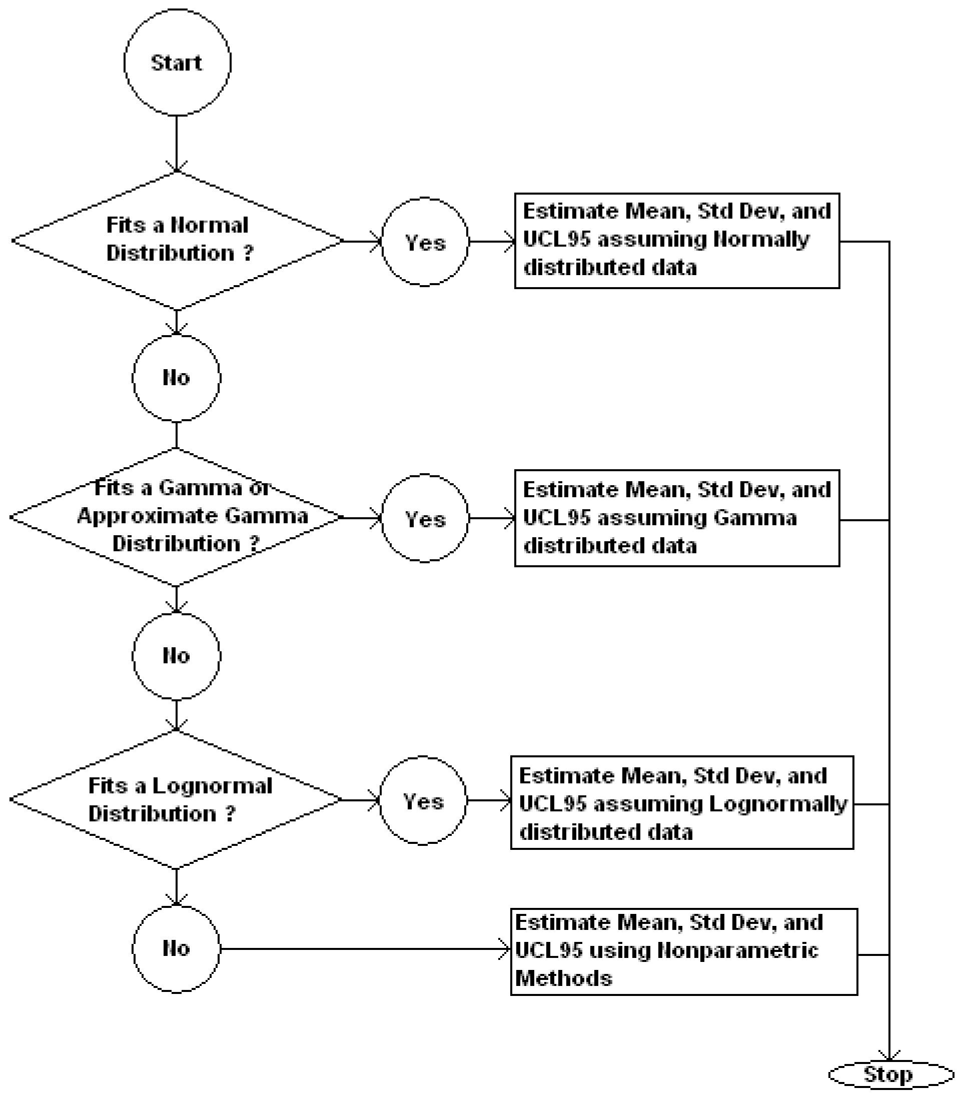


Figure 7. Sequence of Goodness-of-Fit Tests to Identify a Distribution and Select an Estimation Method

Finally, 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 W-S statistic is less than the W-S critical value, then the lognormal assumption is vacated and a nonparametric approach to estimation is adopted; if the W-S test determines that the lognormal distribution is plausible, then the lognormal distribution is adopted. Appropriate UCL95's based on the lognormal distribution and the nonparametric Chebyshev UCL95 for use when the lognormal distribution is rejected are documented by Singh, Singh, and Englehardt [1997].
Heterogeneity and ANOVA tests were performed in SAS JMP® 11.1.1 software from SAS Institute, Inc.[15], and distribution plotting, goodness-of-fit tests, and parameter estimation were performed in ProUCL 5.0.00 software developed by Singh and Singh [2013]. Software validation and verification for SAS JMP® 11.1.1 and ProUCL 5.0.00 are documented by Baker and Others [2014].

The examination of the data for outliers is highly important. This can be done visually by examining graphs, but a statistical test can provide a good 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, Steel and Torrie [1980], applied to the concentration data. The Dixon Q test was performed by the ProUCL 5.0.00 software application written by Singh and Singh, A.K. [2013]. 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. When the model contains a sampling term, the Dixon test is applied to the Studentized residuals from the sampling model.

4.0 APPLICATION TO THE ANALYTICAL DATA

Application of these statistical methods to the Tank 16H primary sample results is presented in Appendix E, and application of these statistical methods to the Tank 16H annulus results is provided in Appendix F. The statistical software applications used in this report have been reviewed for quality and the results have been documented in a report by Baker and Others [2014].

5.0 REFERENCES FOR THE STATISTICAL APPENDIX

1. [1982] Alt, F.B. “Bonferroni Inequalities and Intervals,” in Kotz, S. and Johnson, N.L. **Encyclopedia of Statistical Sciences**, John Wiley and Sons, NY.
2. [2014] Baker, R.A.; Edwards, T.B.; Harris, S.P.; Shine, E.P.; and Watson, H.L. “Verification and Validation for Commercial Statistical Packages Utilized by Applied Computational engineering and Statistics,” B-VVR-A-00002, Revision 2 (April).
3. [2010] SAS Institute, Inc. **Basic Analysis and Graphing**, JMP 11.1.1 On-line Help Books, Cary, NC.
4. [1997] Singh, A.K.; Singh, A; and Englehardt, M. “The Lognormal Distribution in Environmental Applications,” EPA/600/S-97/006 (December).
5. [2013] Singh, A. and Singh, A.K. “ProUCL Version 5.0.00 Technical Guide” US EPA/600/R-07/041 (September).
6. [1980] Steel, R.D. and Torrie, J.H. **Principles and Procedures of Statistics: a Biometrical Approach**, Second Edition, McGraw-Hill Publishers.
7. [1999] SAS Institute, Inc. The SAS System for Windows Software, Release 8.2, Cary, NC.

APPENDIX E: Statistical Analyses for Tank 16H Primary Tank Samples

1.0 INTRODUCTION

The goal of the Tank 16H characterization is to document the physical, chemical, and radiological characteristics of the residual sludge material remaining in the Tank 16H primary tank based on samples of the material. The primary statistical analyses objective is to establish an upper bound for the mean concentrations of the chemical and radiological characteristics. Appendix D describes the statistical basis for the computations. The statistical analyses use the analytical results presented in Appendix E: Table E2 for the physical parameters, Tables E3 through E5 for the radionuclides, Tables E6 through E8 for the elementals, and Tables E9 through E11 for the anions. The analytical results are either measurements, results that are above their minimum detectable concentrations (MDC's); or censored values, results that are only known to be less than their MDC's. Measurements are listed in Tables E2 through E11 in black font, while censored results, listed as less-than-MDC values (< MDC) are set off in red font. The existence of censored values leads to partitioning the characteristics into three separate classes for statistical analyses:

- Characteristics with all results below their MDC's.
- Characteristics with all results above their MDC's.
- Characteristics with a mixture of results that are above and below their MDC's.

These classes allow more uniform reporting of results, as analytes within any particular class tend to have similar statistical analyses. The upper bounds for the mean concentrations are 95% upper confidence limits (UCL95's) when all or most of the results are above their MDCs. When all or nearly all results are below their MDC's the upper bounds for the mean concentrations are represented by the minimum and maximum reported MDC's.

The sampling plan for the residual material remaining in the Tank 16H primary tank was based on three primary samples obtained from the tank floor. Each of the three primary samples was measured three times for a total of nine analytical results for each analyte of interest.

2.0 STATISTICAL ANALYSIS OF TANK 16H PRIMARY TANK SAMPLES

The following subsections apply the statistical methods described in Appendix D to characterize the concentrations of constituents in the residual material remaining in the Tank 16H primary tank.

2.1 ANALYSIS OF PHYSICAL PARAMETERS

Three physical parameters were in the data set to be statistically analyzed: the "as received" bulk density (g/mL), the homogenized bulk density (g/mL), and the weight percent solids (wt %). All 3 physical parameters had a complete set of 9 measurements: 3 measurements on each of three primary samples.
Beginning with the sampling variance model, Levene's test for heterogeneity of variance was applied to all 3 physical parameters with family-wise $\alpha = 0.05/3 = 0.0167$. Referring to Table E12, the Levene's test is not statistically significant (P-value > α) for any of the physical parameters. This means that the measurement error variances appear to be uniform across the primary samples. Therefore, tests to determine whether there is variance among the primary samples were performed using an analysis of variance (ANOVA) which assumes a constant measurement error variance. The ANOVA F-test for a sampling variance was not statistically significant at $\alpha = 0.05$ for any of the 3 physical parameters.

Therefore, the non-sampling error model was adopted for the 3 physical parameters. Subsequently, the Wilk-Shapiro goodness-of-fit test for a normal distribution and the Dixon's test for an outlier were applied to each set of sample results: none of the goodness-of-fit tests or the Dixon's tests were statistically significant at $\alpha = 0.05$. These results demonstrated that there was no significant lack of fit from a normal distribution or potential measurement outliers for either physical parameter. Subsequently, separate UCL95's were computed for the 3 physical parameters using a one-sided upper Student's t confidence interval with 8 degrees of freedom (df). The summary of the results for the physical parameters, including UCL95's, is given in Table E12 with supporting information in Table E13.

2.2 ANALYSIS OF RADIONUCLIDES

Forty-two radionuclides plus the Pu-239/Pu-240 ratio were statistically analyzed. Twenty-four radionuclides, Am-242m, C-14, Cf-249, Cf-251, Cl-36, Cm-242, Cm-243, Cm-244, Cm-245, Cm-247, Cm-248, Co-60, Cs-135, K-40, Nb-94, Ni-59, Ni-63, Np-237, Pa-231, Pu-241, Pu-244, Ra-226, Th-230, and Zr-93 had all of their results below their MDC's; only one radionculide, Tc-99 had all measurements; and 17 radionuclides, Am-241, Am-243, Ba-137m, Cs-137, Eu-154, I-129, Pu-238, Pu-239, Pu-240, Pu-242, Sr-90, U-233, U-234, U-235, U-236, U-238, and Y-90 and the Pu-239/Pu-240 ratio had a mixture of measurements above and below their MDC's.

Just Tc-99 had all measurement results and was analyzed on the logarithm of it's concentrations. Using a sampling model, Levene's test for homogeneous measurement error variance on the log concentrations was not statistically significant at $\alpha = 0.05$. This means that the measurement error variances appear to be uniform across the samples in the log metric. The subsequent ANOVA F-test for a sampling variance on the log concentrations was statistically significant at $\alpha = 0.05$. The sample mean-centered residuals for the log concentrations were subjected to the Wilk-Shapiro goodness-of-fit test for normality and examined for potential outliers with Dixon's outlier test. There was no statistically significant lack of fit from a normal distribution using the Wilk-Shapiro goodness-of-fit test at $\alpha = 0.05$. No potential outliers were identified by Dixon's test at $a = 0.05$. The 3 sample means were obtained by a transformation to the normal scale. The UCL95 was determined from the lognormal distribution in ProUCL Version 5.

Seventeen radionuclides and the Pu-239/Pu-240 ratio had a mixture of results above and below their MDC's. Of these 18 characteristics, Am-243, Ba-137m, Cs-137, and Pu-242 had all less-than-MDC results for two samples. Consequently, there was no estimate of the sampling variance for these 4 radionuclides. The available measurements on one sample were used to construct individual UCL95's for their runs. These UCL95's were interpreted as though they were MDC values and replaced their associated measurements. The concentrations for Am-243, Ba-137m, Cs-137, and Pu-242 were subsequently analyzed like other analytes that had all less-than-MDC results.

The remaining 13 radionuclides and the Pu-239/Pu-240 ratio had sufficient measurments spread across their primary samples to obtain UCL95's for their mean concentrations. The UCL95's for all of these radionculides, except I-129, were obtained using an approach to minimize the risk of a UCL95 failing to bound the actual mean concentration of the analyte in the primary tank. A nonparametric Chebyshev UCL95 was adopted since there was no discernable distribution for the concentration distributions on the original (concentration) scale. If all results for a sample were measurements, then the sample mean was fixed to the mean of the 3 measurements. Each less-than-MDC result for a run was considered to represent an unknown measurement value between 0 and the MDC value. From this, an interval of plausible measurement values was established for each sample that had at least one less-than-MDC result. The low end of each interval was the mean of 0 for any less-than-MDC results and measurements (if there were any), the high end of each interval was the mean of the MDC results and measurements (if there were any). Then the largest value of Chebyshev's UCL95 was obtained over the range of these concentration values that were consistent with the available measurements and less-than-MDC results. The associated values of the mean concentrations for the samples were identified, and the mean and total standard deviation of an individual result were computed from those values.

I-129 was analyzed on the logarithm scale, and the UCL95 was subsequently computed in ProUCL Version 5 from the three sample means back in the original units.

The minimum and maximum MDC's for the radionuclides with all results below their MDC's are listed in Table E14. The UCL95 for Tc-99, the only radionculide with all measurements, is summarized in Table E15. Table E16 contains the UCL95's for the radionculides with a mixture of results above and below their MDC's. The first portion of Table E16 summarizes the analytes that had sufficient measurements to support a UCL95. The final (detached) portion of Table E16 summarizes the minimum and maximum MDC's for Am-243, Ba-137m, Cs-137, and Pu-242. Table E17 provides supporting information for each of the radionuclides summarizedd in Tables E14, E15, and E16.

2.3. ANALYSIS OF ELEMENTALS

Six elementals, Ag, B, Mo, Sb, Se, and U displayed all less-than-MDC results and are summarized in Table E18. Thirteen elementals, Al, As, Ba, Cd, Co, Cr, Cu, Fe, Hg, Mn, Ni, Si, and Zn had all measurements, and Na, Pb, and Sr had a mixture of above and below MDC results.

Refer to Table E1 for a breakdown of elementals with all measurement results by sampling variance model (or not) and the identification of outliers (or not). For the 13 elementals with all measurements, Levene's test for homogeneous measurement error variance was never observed to be statistically significant at α = 0.0038. The subsequent ANOVA F-test for a sampling variance was statistically significant at α = 0.05 for Al, As, Ba, Co, Cu, Hg, Mn, Si, and Zn. The sample mean-centered residuals were examined for lack-of-normality by the Wilk-Shapiro test and for potential outliers with Dixon's outlier test. None of the Wilk-Shapiro tests or the Dixon's tests were statistically significant at α = 0.05 for sets of mean-centered residuals. UCL95's were subsequently constructed for these analytes using Student's t UCL95 for a sampling model.

Table E1. Classification of the Elementals with All Measurement Results by Sampling/Non-Sampling Model and Whether the Data Exhibited Potential Outliers

Statistically Significant Sampling Variance (SS-SV)		Statistically Non-significant Sampling Variance (SNS-SV)	
Statistically Significant Outlier(s) (SS-OT)	**No Statistically Significant Outliers (SNS-OT)**	**Statistically Significant Outlier(s) (SS-OT)**	**No Statistically Significant Outliers (SNS-OT)**
<None>	Al As Ba Co Cu Hg Mn SI Zn	<**None**>	Cd Cr Fe Ni

The analytes Cd, Cr, Fe, and Ni did not have a statistically significant sampling variance at $\alpha = 0.05$. UCL95's were obtained for them using Student's t UCL95 for a model without a sampling effect. The minimum and maximum MDC's for the elementals with all results below their MDC's are listed in Table E18. The UCL95's for elementals that have all measurements are summarized in Table E19. Table E20 contains the UCL95's for Na, Pb, and Sr, which had a mixture of above and below MDC results. Table E21 contains supporting details for the statistical analyses of the elementals.

2.4 ANALYSIS OF ANIONS

Nine anions, Bromide, Chloride, Fluoride, Formate, Iodine I-127, Nitrite, Oxalate, Phosphate, and Sulfate did not display any results above their MDCs. Only Nitrate had all measurements. Iodine I-129 and Total Iodine had a mixture of above and below MDC results.

Using a sampling model for Nitrate, the Levene's test for homogeneous measurement error variance was statistically significant at $\alpha = 0.05/2 = 0.025$ (Total Iodine was also tested for homogeneous measurement variances). The ANOVA F-test for a sampling variance was statistically signifcant at $\alpha = 0.05$. Using the mean-centered residuals, the Wilk-Shapiro test for goodness-of-fit to a normal distribution was not statistically significant at $\alpha = 0.05$, and Dixon's outlier test did not identifying any potential outliers at $\alpha = 0.05$. The UCL95 was determined using Student's t.

Both Iodine I-129 and total Iodine had a mixture of above and below MDC results and their UCL95's were based on nonparametric Chebyshev UCL95's.

3.0 SUMMARY OF STATISTICAL ANALYSES FOR THE RESIDUAL MATERIAL TANK 16H PRIMARY TANK

A key feature of the statistical analysis of the Tank 16H annulus concentration data was a potential sampling outlier, Primary Sample 2. This was especially evident for the radionuclides. Many of the radionuclides had all less-than-MDC results for Sample 2, and mostly measurements for Samples 1 and 3. For some of these radionuclides the 2-P sample concentrations were considerably different than the other two samples. No discernable distribution could be assumed when only two samples had estimates of their mean concentrations. Therefore, a conservative method was adopted to produce UCL95's for these radionculides. This approach produced the largest possible value for the nonparametric Chebyshev UCL95 that was consistent with all of the less-than-MDC results being between 0 and their MDC values.
The results are reported in tables in the following section. Each type of constituent, physical parameter, radionuclide, elemental, and anion is broken down into separate tables for reporting results based on whether all results are less than their MDC's, all results are measurements, or the results are a mixture of measurements and below MDC values. Summary tables for each type of constituent are followed by extensive supporting tables. A list of the tables in Appendix E follows.

Appendix E: Statistical Analyses for Tank 16H Primary Tank Samples.

Classification Tables by Type of Statistical Model and Potential Outliers **Page**

Appendix E: Tables of Concentration Data used in Statistical Analyses

Table E2. Physical Parameters

Physical Parameters	Sample 1-P (Ground)			Sample 2-P (Ground)			Sample 3-P (Ground)		
"As received" Bulk Density, g/mL	1.20	1.32	1.21	1.43	1.35	1.36	1.37	1.08	1.22
Homogenized Bulk Density, g/mL	1.81	1.69	1.74	1.88	1.52	1.62	1.50	1.52	1.65
Air Dried and Homogenized, wt %	99.75	99.75	99.65	99.25	98.90	99.35	99.50	98.85	99.40

Appendix E: Tables of Concentration Data used in Statistical Analyses

Table E3. Radionuclides with All Results below their MDCs

Radionuclide Constituent, μCi/g	Sample 1-P (Ground)			Sample 2-P (Ground)			Sample 3-P (Ground)		
Am-242m	<3.00E-04	<2.93E-03	<1.15E-03	<2.00E-04	<1.15E-05	<1.09E-04	<3.49E-03	<4.68E-04	<1.76E-03
C-14	<7.12E-04	<7.39E-04	<1.00E-03	<7.21E-04	<7.03E-04	<8.20E-04	<7.93E-04	<7.07E-04	<9.05E-04
Cf-249	<3.09E-03	<4.36E-03	<3.74E-03	<2.46E-04	<2.23E-05	<3.00E-04	<3.70E-03	<4.73E-03	<5.68E-03
Cf-251	<9.50E-03	<1.51E-02	<1.36E-02	<5.32E-04	<5.59E-05	<6.98E-04	<1.05E-02	<1.49E-02	<1.62E-02
Cl-36	<3.47E-05	<4.73E-06	<1.06E-05	<2.64E-06	<2.02E-06	<2.59E-06	<1.41E-05	<1.45E-05	<2.66E-05
Cm-242	<2.48E-04	<2.42E-03	<9.50E-04	<1.65E-04	<9.50E-06	<9.05E-05	<2.88E-03	<3.86E-04	<1.45E-03
Cm-243	<1.05E-02	<1.72E-02	<1.57E-02	<5.68E-04	<6.58E-05	<7.93E-04	<1.18E-02	<1.62E-02	<1.81E-02
Cm-244	<6.89E-02	<3.18E-02	<1.34E-01	<1.25E-02	<3.73E-03	<2.13E-02	<7.34E-02	<9.46E-02	<8.20E-02
Cm-245	<1.18E-04	<2.83E-04	<4.18E-04	<2.95E-05	<2.66E-06	<3.76E-05	<3.45E-04	<4.50E-04	<5.18E-04
Cm-247	<8.15E-08	<1.18E-07	<1.53E-07	<1.55E-08	<1.30E-09	<1.27E-08	<2.04E-07	<2.32E-07	<2.95E-07
Cm-248	<6.85E-06	<7.75E-06	<5.99E-06	<8.87E-07	<5.81E-08	<5.09E-07	<1.32E-05	<9.59E-06	<1.25E-05
Co-60	<3.09E-03	<2.89E-03	<2.85E-03	<4.44E-04	<8.42E-04	<7.84E-04	<2.88E-03	NR/N	<2.98E-03
Cs-135	<1.34E-04	<1.24E-04	<8.96E-05	<6.71E-05	<1.43E-04	<2.28E-05	<1.27E-04	<1.93E-04	<1.82E-04
K-40	<5.77E-03	<6.40E-03	<9.05E-03	<2.23E-05	<2.69E-05	<1.75E-05	<3.77E-03	<4.91E-03	<3.96E-03
Nb-94	<8.15E-03	<6.62E-03	<3.80E-03	<5.50E-03	<4.68E-03	<6.85E-03	<1.08E-02	<8.78E-03	<7.03E-03
Ni-59	<2.18E-01	<1.81E+00	<1.90E-02	<1.03E-03	<5.50E-04	<1.19E-03	<2.05E-02	<8.51E-02	<5.18E-01
Ni-63	<1.43E+00	<1.25E+01	<3.95E-01	<1.94E-03	<7.48E-04	<2.22E-03	<3.18E-01	<8.20E-01	<4.91E+00
Np-237	<6.89E-04	<6.93E-04	<6.82E-04	<6.92E-04	<6.82E-04	<6.77E-04	<6.58E-04	<6.87E-04	<6.89E-04
Pa-231	<2.66E-03	<3.20E-03	<2.95E-03	<3.67E-03	<3.35E-03	<2.84E-03	<4.11E-03	<6.04E-02	<4.77E-03
Pu-241	<2.40E-01	<2.61E-01	<3.18E-01	<3.73E-02	<3.80E-02	<3.68E-02	<2.86E-01	<2.35E-01	<2.36E-01
Pu-244	<4.68E-07	<3.81E-07	<3.14E-07	<9.01E-08	<1.26E-07	<1.01E-07	<6.04E-07	<5.54E-07	<8.33E-07
Ra-226	<1.95E-03	<3.15E-04	<5.27E-04	NR/Q	<3.94E-04	<2.17E-03	NR/Q	<5.27E-04	NR/Q
Th-230	<4.64E-04	<3.84E-04	<6.67E-04	<2.84E-04	<1.54E-04	<1.02E-04	<5.05E-04	<3.43E-04	<7.66E-04
Zr-93	<2.89E-02	<3.25E-02	<2.68E-02	<3.80E-03	<4.59E-03	<3.05E-03	<2.15E-02	<2.31E-02	<2.62E-02

NR/Q: Not reported due to quality issues.
NR/N: No result. Analysis was not performed on this aliquot because it exceeded dose limits.

Appendix E: Tables of Concentration Data used in Statistical Analyses

Table E4. Radionuclides with All Results above their MDCs

Radionuclide Constituent, μCi/g	Sample 1-P (Ground)			Sample 2-P (Ground)			Sample 3-P (Ground)		
Tc-99	1.28E-01	1.05E-01	8.74E-02	5.90E-04	1.50E-03	8.74E-04	1.35E-01	1.73E-01	1.12E-01

Table E5. Radionuclides with a Mixture of Results above and below their MDCs

Radionuclide Constituent, μCi/g	Sample 1-P (Ground)			Sample 2-P (Ground)			Sample 3-P (Ground)		
Am-241	2.68E-01	3.14E-01	3.03E-01	<3.27E-03	<4.14E-03	<3.57E-03	3.19E-01	3.53E-01	3.86E-01
Am-243	<3.53E-03	<8.92E-03	1.08E-02	<2.32E-04	<9.01E-05	<4.04E-04	<3.81E-03	<9.95E-03	<6.04E-03
Ba-137m	<2.50E-01+	<2.89E-01+	<2.61E-01+	<1.25E-02+	<9.65E-03+	<1.34E-02+	7.76E-01	6.26E-01	6.22E-01
Cs-137	<2.64E-01+	<3.05E-01+	<2.76E-01+	<1.32E-02+	<1.02E-02+	<1.42E-02+	8.20E-01	6.62E-01	6.58E-01
Eu-154	1.99E-01	1.63E-01	1.43E-01	<1.72E-03	<1.85E-03	<1.64E-03	1.70E-01	1.83E-01	1.91E-01
I-129	1.99E-04	1.70E-04	3.08E-04	<7.30E-06	<5.45E-06	8.47E-06	2.82E-04	2.82E-04	3.23E-04
Pu-238	<9.95E-01	<9.41E-01	1.27E+00	<6.04E-03	<4.24E-03	<7.84E-03	1.17E+00	1.03E+00	1.01E+00
Pu-239	4.31E-02	4.07E-02	4.06E-02	<6.10E-02	<6.02E-02	<5.97E-02	4.18E-02	4.59E-02	5.05E-02
Pu-240	1.91E-02	1.80E-02	1.81E-02	<1.13E-03	<1.58E-03	<1.27E-03	1.82E-02	2.05E-02	2.11E-02
Pu-239/240	<7.70E-02	1.00E-01	1.37E-01	<4.21E-03	<2.55E-02	<7.66E-03	9.23E-02	<7.43E-02	8.96E-02
Pu-242	<4.25E-05	<3.74E-05	<3.80E-05	<1.97E-05	<9.64E-06	<8.42E-06	5.27E-05	5.72E-05	6.35E-05
Sr-90	3.43E+03	2.41E+03	2.57E+03	<5.00E+00	<4.41E+00	<5.77E+00	3.15E+03	3.18E+03	3.12E+03
U-233	1.16E-04	8.92E-05	1.09E-04	<4.75E-03	<4.69E-03	<4.65E-03	1.82E-04	2.33E-04	1.77E-04
U-234	1.67E-04	1.59E-04	1.55E-04	<3.07E-03	<3.02E-03	<3.00E-03	1.74E-04	1.93E-04	1.53E-04
U-235	6.31E-07	5.86E-07	5.54E-07	<1.06E-06	<1.05E-06	<1.04E-06	7.79E-07	7.84E-07	6.62E-07
U-236	1.69E-06	1.68E-06	1.59E-06	<3.18E-05	<3.13E-05	<3.11E-05	2.01E-06	2.01E-06	1.74E-06
U-238	1.97E-06	2.03E-06	1.84E-06	<4.14E-07	<4.07E-07	<4.03E-07	2.87E-06	3.02E-06	2.54E-06
Y-90	3.43E+03	2.41E+03	2.57E+03	<5.00E+00	<4.41E+00	<5.77E+00	3.15E+03	3.18E+03	3.12E+03

+ Value is an upper limit due to the "blank" concentration being greater than 10% of the sample value.

Appendix E: Tables of Concentration Data used in Statistical Analyses

Table E6. Elemental Constituents with All Results below their MDCs

Elemental Constituent, wt %	Sample 1-P (Ground)			Sample 2-P (Ground)			Sample 3-P (Ground)		
Ag	<1.32E-03	<1.33E-03	<1.31E-03	<1.33E-03	<1.31E-03	<1.30E-03	<1.26E-03	<1.31E-03	<1.32E-03
B	<1.91E-02	<1.91E-02	<1.88E-02	<1.91E-02	<1.88E-02	<1.87E-02	<1.82E-02	<1.90E-02	<1.91E-02
Mo	<8.12E-03	<8.16E-03	<8.03E-03	<8.15E-03	<8.03E-03	<7.97E-03	<7.75E-03	<8.08E-03	<8.12E-03
Sb	<2.83E-02	<2.85E-02	<2.80E-02	<2.84E-02	<2.80E-02	<2.78E-02	<2.70E-02	<2.82E-02	<2.83E-02
Se	<5.38E-04	<5.41E-04	<5.32E-04	<5.40E-04	<5.32E-04	<5.28E-04	<5.14E-04	<5.36E-04	<5.38E-04
U	<2.15E-01	<2.16E-01	<2.13E-01	<2.16E-01	<2.13E-01	<2.11E-01	<2.05E-01	<2.14E-01	<2.15E-01

Table E7. Elemental Constituents with All Results above their MDCs

Elemental Constituent, wt %	Sample 1-P (Ground)			Sample 2-P (Ground)			Sample 3-P (Ground)		
Al	5.23E-01	3.72E-01	5.60E-01	6.30E-02	7.46E-02	4.88E-02	4.14E-01	3.92E-01	4.15E-01
As	3.40E-03	3.57E-03	3.38E-03	3.70E-03	3.51E-03	3.93E-03	4.19E-03	4.11E-03	4.13E-03
Ba	1.22E-02	5.34E-03	1.26E-02	1.81E-03	1.64E-03	1.38E-03	7.74E-03	7.03E-03	6.76E-03
Cd	2.22E-03	1.95E-03	2.34E-03	2.59E-03	2.65E-03	2.43E-03	2.72E-03	2.18E-03	2.15E-03
Co	4.87E-03	5.18E-03	4.49E-03	4.82E-03	5.01E-03	4.74E-03	8.07E-03	7.43E-03	7.72E-03
Cr	2.45E-02	2.57E-02	2.66E-02	3.68E-02	4.97E-02	1.93E-02	1.70E-02	1.69E-02	1.53E-02
Cu	5.63E-02	4.65E-02	6.33E-02	1.56E-02	1.68E-02	1.66E-02	4.58E-02	4.31E-02	4.13E-02
Fe	6.08E+01	6.03E+01	6.16E+01	6.02E+01	6.06E+01	6.22E+01	6.07E+01	6.11E+01	6.09E+01
Hg	1.39E-01	1.94E-01	1.33E-01	3.84E-03	4.05E-03	6.21E-03	1.54E-01	1.66E-01	1.75E-01
Mn	3.65E-01	3.69E-01	3.81E-01	5.27E-01	5.30E-01	5.24E-01	3.13E-01	3.10E-01	3.22E-01
Ni	3.00E-02	2.94E-02	3.26E-02	2.18E-02	2.86E-02	1.28E-02	1.96E-02	2.03E-02	1.99E-02
Si	6.64E-01	5.97E-01	6.34E-01	4.85E-01	6.12E-01	5.36E-01	2.87E-01	3.50E-01	2.55E-01
Zn	7.07E-02	6.25E-02	7.69E-02	4.09E-02	4.39E-02	3.17E-02	1.97E-02	2.44E-02	2.22E-02

Table E8. Elementals with a Mixture of Results above and below their MDCs

Elemental Constituent, wt %	Sample 1-P (Ground)			Sample 2-P (Ground)			Sample 3-P (Ground)		
Pb	1.02E-02	<9.64E-03	1.26E-02	6.11E-01	6.25E-01	5.11E-01	7.51E-02	8.22E-02	7.92E-02
Na	2.30E-02	8.57E-03	1.90E-02	<6.49E-03	<6.40E-03	<6.35E-03	1.46E-02	1.30E-02	1.32E-02
Sr	2.07E-02	1.03E-02	2.22E-02	<9.82E-05	<9.68E-05	<9.61E-05	1.31E-02	1.18E-02	1.10E-02

Appendix E: Tables of Concentration Data used in Statistical Analyses

Table E9. Anions with All Results above their MDCs

Anion Constituent wt %	Sample 1-P (Ground)			Sample 2-P (Ground)			Sample 3-P (Ground)		
Nitrate , NO_3^{-1}	1.46E-02	9.72E-03	1.41E-02	9.72E-03	9.82E-03	9.39E-03	4.66E-02	4.36E-02	4.42E-02

Table E10. Anions with All Results below their MDCs

Anion Constituent wt %	Sample 1-P (Ground)			Sample 2-P (Ground)			Sample 3-P (Ground)		
Bromide, Br^{-1}	<4.87E-03	<4.86E-03	<4.70E-03	<4.86E-03	<4.91E-03	<4.70E-03	<4.66E-03	<4.84E-03	<4.91E-03
Chloride , Cl^{-1}	<4.87E-03	<4.86E-03	<4.70E-03	<4.86E-03	<4.91E-03	<4.70E-03	<4.66E-03	<4.84E-03	<4.91E-03
Fluoride, F^{-1}	<4.87E-03	<4.86E-03	<4.70E-03	<4.86E-03	<4.91E-03	<4.70E-03	<4.66E-03	<4.84E-03	<4.91E-03
Formate, CHO_2^{-1}	<4.87E-03	<4.86E-03	<4.70E-03	<4.86E-03	<4.91E-03	<4.70E-03	<4.66E-03	<4.84E-03	<4.91E-03
Iodine, I-127	<6.38E-07	<6.37E-07	<6.15E-07	<6.37E-07	<6.43E-07	<6.15E-07	<6.11E-07	<6.34E-07	<6.43E-07
Nitrite , NO_2^{-1}	<4.87E-03	<4.86E-03	<4.70E-03	<4.86E-03	<4.91E-03	<4.70E-03	<4.66E-03	<4.84E-03	<4.91E-03
Oxalate, $C_2O_4^{-2}$	<4.87E-03	<4.86E-03	<4.70E-03	<4.86E-03	<4.91E-03	<4.70E-03	<4.66E-03	<4.84E-03	<4.91E-03
Phosphate, PO_4^{-3}	<4.87E-03	<4.86E-03	<4.70E-03	<4.86E-03	<4.91E-03	<4.70E-03	<4.66E-03	<4.84E-03	<4.91E-03
Sulfate, SO_4^{-2}	<4.87E-03	<4.86E-03	<4.70E-03	<4.86E-03	<4.91E-03	<4.70E-03	<4.66E-03	<4.84E-03	<4.91E-03

Table E11. Anions with a Mixture of Results above and below their MDCs

Anion Constituent wt %	Sample 1-P (Ground)			Sample 2-P (Ground)			Sample 3-P (Ground)		
Iodine, I-129	1.13E-04	9.62E-05	1.75E-04	<4.13E-06	<3.09E-06	4.80E-06	1.60E-04	1.60E-04	1.83E-04
Total Iodine	1.13E-04	9.62E-05	1.75E-04	<4.77E-06	<3.73E-06	<5.42E-06	1.60E-04	1.60E-04	1.83E-04

Appendix E: Statistical Analyses of Physical Parameters

Table E12. Statistical Summary for the Physical Parameters

Physical Parameters	N	Mean (g/mL)	Std Dev (g/mL)	% Std Dev	UCL95 (g/mL)	Goodness-of-Fit/Confidence Limit Remarks♠
“As received” Bulk density (g/mL)	9	1.2822	1.1088E-1	8.648%	1.3510	SNS-VH; SNS-SV; SNS-WS; SNS-OT; Use Student’s t UCL95
Air Dried and Homogenized Wt % Solids (%)	9	9.9378E+1	3.3365E-1	0.3357%	9.9585E+1	SNS-VH; SNS-SV; SNS-WS; SNS-OT; Use Student’s t UCL95
Homogenized bulk density (g/mL)	9	1.6589	1.3448E-1	8.107%	1.7422	SNS-VH; SNS-SV; SNS-WS; SNS-OT; Use Student’s t UCL95

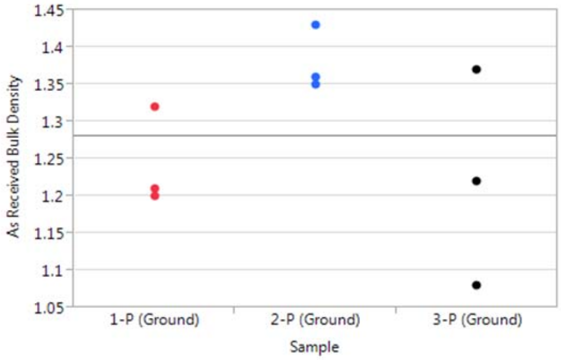
SS-VH/SNS-VH: Statistically significant /Statistically non-significant Levene’s test of variance heterogeneity at $\alpha = 0.0167$.
SS-SV/SNS-SV: Statistically significant /Statistically non-significant sampling variance $\alpha = 0.05$.
SS-WS/SNS-WS: Statistically significant /Statistically non-significant Wilk-Shapiro test statistic for testing normality $\alpha = 0.05$.
SS-OT/SNS-OT: Statistically significant /Statistically non-significant Dixon’s test for outliers $\alpha = 0.05$. This test assumes the normal distribution.

Appendix E: Statistical Analyses of Physical Parameters

Table E13. Supporting Results for Physical Parameters

"As Received" Bulk Density (g/mL)

All Results (circles) above MDCs



Means and Standard Deviations

Sample	N	Mean	Std Dev	Std Err Mean
1-P	3	1.2433	6.6583E-2	3.8442E-2
2-P	3	1.3800	4.3589E-2	2.5166E-2
3-P	3	1.2233	1.4503E-1	8.3732E-2
Overall	9	1.2822	1.1088E-1	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	1.2371	2	6	0.3550
Brown-Forsythe	1.0219	2	6	0.4150
Levene	1.3400	2	6	0.3303
Bartlett	1.1905	2		0.3041

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	4.3622E-2	2.1811E-2	2.391	0.1723
Error	6	5.4733E-2	9.1222E-3		
C. Total	8	9.8356E-2			

Statistically non-significant sampling variance.

Diagnostics for Concentrations

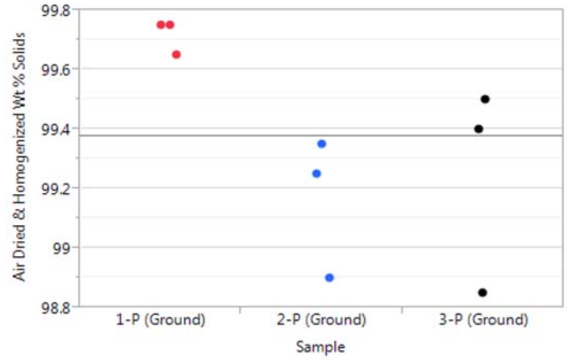
Shapiro-Wilk	0.935 > 0.829 crit. value->SNS
Dixon High Outlier	Run 1 of Sample 2 -> SNS
Dixon Low Outlier	Run 2 of Sample 3 -> SNS

Normal Statistics

Statistic	Estimate
Mean	1.2822E+0
Std.Dev.	1.1088E-1
Student's t UCL95	1.3510E+0

Air Dried & Homogenized Wt % Solids

All Results (circles) above MDCs



Means and Standard Deviations

Sample	N	Mean	Std Dev	Std Err Mean
1-P	3	9.9717E+1	5.7735E-2	3.3333E-2
2-P	3	9.9167E+1	2.3629E-1	1.3642E-1
3-P	3	9.9250E+1	3.5000E-1	2.0207E-1
Overall	9	9.9378E+1	3.3365E-1	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	1.0490	2	6	0.4067
Brown-Forsythe	0.6370	2	6	0.5612
Levene	4.5714	2	6	0.0622
Bartlett	1.8624	2		0.1553

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	5.2722E-1	2.6361E-1	4.353	0.0679
Error	6	3.6333E-1	6.0556E-2		
C. Total	8	8.9056E-1			

Statistically non-significant sampling variance.

Diagnostics for Concentrations

Shapiro-Wilk	0.907 > 0.829 crit. value->SNS
Dixon High Outlier	Runs 1 & 2 of Sample 1 -> SNS
Dixon Low Outlier	Run 2 of Sample 3 -> SNS

Normal Statistics

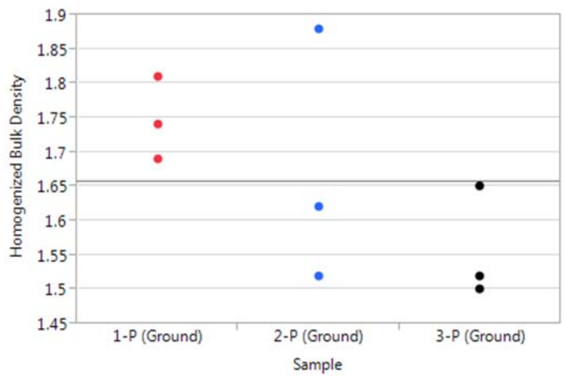
Statistic	Estimate
Mean	9.9378E+1
Std.Dev.	3.3365E-1
Student's t UCL95	9.9585E+1

Appendix E: Statistical Analyses of Physical Parameters

Table E13 Supporting Results for Physical Parameters

Homogenized Bulk Density (g/mL)

All Results (circles) above MDCs



Means and Standard Deviations

Sample	N	Mean	Std Dev	Std Err Mean
1-P	3	1.7467	6.0277E-2	3.4801E-2
2-P	3	1.6733	1.8583E-1	1.0729E-1
3-P	3	1.5567	8.1445E-2	4.7022E-2
Overall	9	1.6589	1.3448E-1	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	1.2392	2	6	0.3544
Brown-Forsythe	0.7308	2	6	0.5200
Levene	2.9141	2	6	0.1305
Bartlett	1.1345	2		0.3216

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	5.5089E-2	2.7544E-2	1.844	0.2375
Error	6	8.9600E-2	1.4933E-2		
C. Total	8	1.4469E-1			

Statistically non-significant sampling variance.

Diagnostics for Concentrations

Shapiro-Wilk	0.936 > 0.829 crit. value->SNS
Dixon High Outlier	Run 1 of Sample 2 -> SNS
Dixon Low Outlier	Run 1 of Sample 3 -> SNS

Normal Statistics

Component	Estimate
Mean	1.6589E+0
Std.Dev.	1.3448E-1
Student's t UCL95	1.7422E+0

Appendix E: Statistical Analyses of Radionuclides

Table E14. Statistical Summary for the Radionuclides – All Results below their MDCs

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
Am-242m	9	0.0000115	1.15E-5	0.00349	3.49E-3
C-14	9	0.000703	7.03E-4	0.001	1.00E-3
Cf-249	9	0.0000223	2.23E-5	0.00568	5.68E-3
Cf-251	9	0.0000559	5.59E-5	0.0162	1.62E-2
Cl-36	9	0.00000202	2.02E-6	0.0000347	3.47E-5
Cm-242	9	0.0000095	9.50E-6	0.00288	2.88E-3
Cm-243	9	0.0000658	6.58E-5	0.0181	1.81E-2
Cm-244	9	0.00373	3.73E-3	0.134	1.34E-1
Cm-245	9	0.00000266	2.66E-6	0.000518	5.18E-4
Cm-247	9	0.0000000013	1.30E-9	0.000000295	2.95E-7
Cm-248	9	0.0000000581	5.81E-8	0.0000132	1.32E-5
Co-60	8	0.000444	4.44E-4	0.00309	3.09E-3
Cs-135	9	0.0000228	2.28E-5	0.000193	1.93E-4
K-40	9	0.0000175	1.75E-5	0.00905	9.05E-3
Nb-94	9	0.0038	3.80E-3	0.0108	1.08E-2
Ni-59	9	0.00055	5.50E-4	1.81	1.81E+0
Ni-63	9	0.000748	7.48E-4	12.5	1.25E+1
Np-237	9	0.000658	6.58E-4	0.000693	6.93E-4
Pa-231	9	0.00266	2.66E-3	0.0604	6.04E-2
Pu-241	9	0.0368	3.68E-2	0.318	3.18E-1
Pu-244	9	0.0000000901	9.01E-8	0.000000833	8.33E-7
Ra-226	6	0.000315	3.15E-4	0.00217	2.17E-3
Th-230	9	0.000102	1.02E-4	0.000766	7.66E-4
Zr-93	9	0.00305	3.05E-3	0.0325	3.25E-2

Appendix E: Statistical Analyses of Radionuclides

Table E15. Statistical Summary for the Radionuclides – All Results above their MDCs

Constituent	N	Mean (µCi/g)	Std Dev (µCi/g)	% Std Dev	UCL95 (µCi/g)	Goodness-of-Fit/Confidence Limit Remarks
Tc-99	9	8.2789E-2	7.5925E-2	91.709%	7.2277E-1	SNS-WS (on Log Conc's); SS-SV (on Log Conc's); Chebyshev UCL95

SS-VH/SNS-VH: Statistically significant /Statistically non-significant Levene's test of variance heterogeneity.
SS-SV/SNS-SV: Statistically significant /Statistically non-significant sampling variance.
SS-WS/SNS-WS: Statistically significant /Statistically non-significant Wilk-Shapiro test statistic for testing normality.
SS-OT/SNS-OT: Statistically significant /Statistically non-significant Dixon's test for outliers. This test assumes the lonormal distribution.
The lognormal distribution was used because for the following reasons: (1) The standard deviation of replicate measurements on a sample was considerably more consistent from sample to sample for the logarithm of the response than the response itself. (2) The coefficient of variation (percent standard deviation) was nearly 100%.
The three sample means and the pooled standard error for a sample mean were estimated for the logarithm of the response, and then transformed back to the original scale using the relationships between the moments of the lognormal and normal distributions. These three sample means constituted a data set that was input to ProUCL version 5.0 in order to compute a UCL95 using the lognormal distribution. The mean in this table is the arithmetic average of the three sample means. Additional details are provided for the Tc-99 entry in Table E17: Supporting Results for Radionuclides.

Appendix E: Statistical Analyses of Radionuclides

Table E16. Statistical Summary for the Radionuclides –Results above and below their MDCs

Radioactive Constituent (μCi/g)	N*	Mean (μCi/g)	Std Dev (μCi/g)	% Std Dev	UCL95 (μCi/g)	Goodness-of-Fit/Confidence Limit Remarks♠
Am-241	9 (0, 3, 0)	2.1589E-1	1.9067E-1	88.317%	6.9197E-1	Maximum Nonparametric Chebyshev UCL95; See Am-241 entry in Table E17 for details.
Eu-154	9 (0, 3, 0)	1.1656E-1	1.0265E-1	88.066%	3.7111E-1	Maximum Nonparametric Chebyshev UCL95; See Eu-154 entry in Table E17 for details.
I-129	9 (0, 2, 0)	1.7725E-4	1.5319E-4	86.426%	5.6278E-4	Nonparametric Chebyshev UCL95
Pu-238	9 (2, 3, 0)	7.1289E-1	6.2147E-1	87.176%	2.2666	Maximum Nonparametric Chebyshev UCL95; See Pu-238 entry in Table E17 for details.
Pu-239	9 (0, 3, 0)	2.9178E-2	2.5511E-2	87.432%	9.3033E-2	Maximum Nonparametric Chebyshev UCL95; See Pu-239 entry in Table E17 for details.
Pu-239/240	9 (1, 3, 1)	6.3356E-2	5.9499E-2	91.912%	2.0355E-1	Maximum Nonparametric Chebyshev UCL95; See Pu-239/240 entry in Table E17 for details.
Pu-240	9 (0, 3, 0)	1.2778E-2	1.1133E-2	87.126%	4.0655E-2	Maximum Nonparametric Chebyshev UCL95; See Pu-240 entry in Table E17 for details.
Sr-90	9 (0, 3, 0)	1.9844E+3	1.7562E+3	88.500%	6.3314E+3	Maximum Nonparametric Chebyshev UCL95; See Sr-90 entry in Table E17 for details.

*N: Overall number of measurements on all of the samples, and (X, Y, Z), where X = Number below MDC for Sample 1-P, Y = Number below MDC for Sample 2-P, and Z = Number below MDC for Sample 3-P.

Note that the summary for radionuclides Am-243, Ba-137m, Cs-137, and Pu-242 (with a mixture of above and below MDC results) is provided at the bottom of the next page since their summaries follow different format than the above radionuclides.

Table E16 continues on the following page.

Appendix E: Statistical Analyses of Radionuclides

Table E16 Continued. Statistical Summary for the Radionuclides –Results above and below their MDCs

Radioactive Constituent (μCi/g)	N*	Mean (μCi/g)	Std Dev (μCi/g)	% Std Dev	UCL95 (μCi/g)	Goodness-of-Fit/Confidence Limit Remarks♠
U-233	9 (0, 3, 0)	1.6662E-3	2.6249E-3	157.538%	8.2779E-3	Maximum Nonparametric Chebyshev UCL95; See U-233 entry in Table E17 for details.
U-234	9 (0, 3, 0)	1.1212E-3	1.6531E-3	147.440%	5.2813E-3	Maximum Nonparametric Chebyshev UCL95; See U-234 entry in Table E17 for details.
U-235	9 (0, 3, 0)	4.4400E-7	3.9454E-7	88.860%	1.4302E-6	Maximum Nonparametric Chebyshev UCL95; See U-235 entry in Table E17 for details.
U-236	9 (0, 3, 0)	1.1658E-5	1.7098E-5	146.663%	5.4686E-5	Maximum Nonparametric Chebyshev UCL95; See U-236 entry in Table E17 for details.
U-238	9 (0, 3, 0)	1.5856E-6	1.4474E-6	91.284%	5.2079E-6	Maximum Nonparametric Chebyshev UCL95; See U-238 entry in Table E17 for details.
Y-90	9 (0, 3, 0)	1.9844E+3	1.7562E+3	88.500%	6.3314E+3	Maximum Nonparametric Chebyshev UCL95; See Y-90 entry in Table E17 for details.

*N: Overall number of measurements on all of the samples, and (X, Y, Z), where X = Number of Runs below MDC for Sample 1-P, Y = Number of Runs below MDC for Sample 2-P, and Z = Number of Runs below MDC for Sample 3-P.

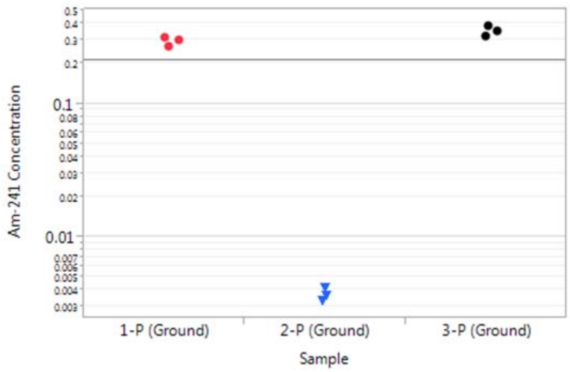
The following is a continuation of the radionuclides with a mixture of above and below MDC results. However, in the following portion of Table E16 the radionuclides had measurements for only one sample. Therefore, individual UCL95's were computed for each Run of Sample 3 and interpreted as MDC's. Thus, the final summary is based on the reporting format for radionuclides with all less-than-MDC results. Details for individual radionuclides are given in Table E17 of supporting information.

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
Am-243	9 (2, 3, 3)	0.0000901	9.01E-5	0.0171	1.71E-2
Ba-137m	9 (3, 3, 0)	0.00965	9.65E-3	1.03	1.03E+0
Cs-137	9 (3, 3, 0)	0.0102	1.02E-2	1.09	1.09E+0
Pu-242	9 (3, 3, 0)	0.00000842	8.42E-06	0.0000793	7.93E-05

Appendix E: Supporting Tables for Statistical Analyses of Radionuclides

Table E17. Supporting Results for Radionuclides

Am-241 (μCi/g)
Mixture of Results (circles) above MDCs
and (triangles) below MDCs



Means and Standard Deviations

Sample	N	Mean	Std Dev	Std Err Mean
1-P	3	2.9500E-01	2.4021E-02	1.3870E-02
3-P	3	3.5267E-01	3.3501E-02	1.9340E-02

Sample	Run 1 MDC	Run 2 MDC	Run 3 MDC
2-P	<3.27E-03	<4.14E-03	<3.57E-03

The mean concentration for Sample 2-P is assumed to be between 0 and 3.66E-03 μCi/g (the mean of the MDC's for the 3 runs for Sample 2-P). The UCL95 for the data set of sample means is {2.9500E-01, X, 3.5267E-01}, where X is the mean for Sample 2-P. The non-parameteric Chebyshev (MVUE) UCL95 is maximized when the mean concentration is set to 0. The Maximum Chebyshev UCL95 is 6.9197E-01 μCi/g.

The associated mean and standard deviation of the data set of means {2.9500E-01, 0, 3.5267E-01} are 2.1589E-01 μCi/g and 1.8918E-01 μCi/g, respectively.

$$S_{Total} = \sqrt{S^2_{3\,Sample\,Means} - S^2_{MeasErr(Mean)} + S^2_{MeasErr(One\,Result)}}$$

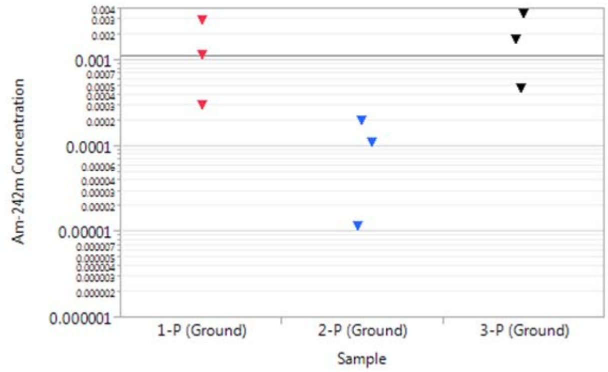
$$= \sqrt{(1.8918E\text{-}1)^2 - (1.6829E\text{-}2)^2 + 3\cdot(1.6829E\text{-}2)^2} = 1.9067E\text{-}1,$$

where the pooled measurement error standard deviation, 2.9149E-2 μCi/g, is based on Samples 1-P and 3-P only.

Nonparametric Statistics

Component	Estimate
Mean	2.1589E-1
Std.Dev.	1.9067E-1
(Nonparametric) Chebyshev UCL95	6.9197E-1

Am-242m (μCi/g)
All Results (triangles) below MDCs



Minimum Detection Concentrations (MDCs)

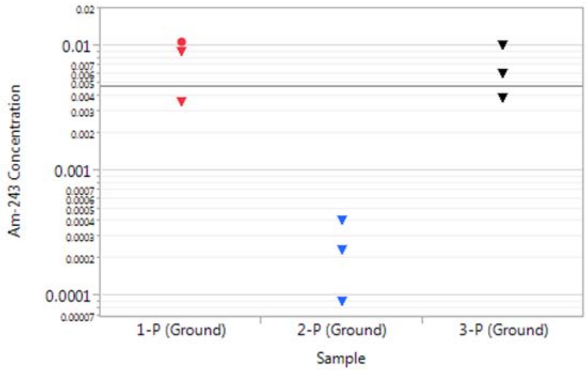
Sample	Number	Minimum MDC	Maximum MDC
1-P	3	3.00E-4	2.93E-3
2-P	3	1.15E-5	2.00E-4
3-P	3	4.68E-4	3.49E-3
Overall	9	1.15E-5	3.49E-3

Appendix E: Supporting Tables for Statistical Analyses of Radionuclides

Table E17. Supporting Results for Radionuclides

Am-243 (μCi/g)

Mixture of Results (circles) above MDCs
and (triangles) below MDCs



Means and Standard Deviations

Sample	N	Mean	Std Dev	Std Err Mean
3-P	1	1.08E-2	undetermined	undetermined

Minimum Detection Concentrations (MDCs)
(omitting the measurement on Run 3 of Sample 1-P)

Sample	N	Minimum MDC	Maximum MDC
1-P	2	3.53E-3	8.92E-3
2-P	3	9.01E-5	4.04E-4
3-P	3	3.81E-3	9.95E-3
Overall	8	9.01E-5	8.92E-3

There was only 1 measurement, 1.08E-2 μCi/g (Run 3 of Sample 1-P), in the 9 analytical results. There is no effective way to determine an overall mean or UCL95 for the mean. Samples 2-P and 3-P have all less-than MDC results. All 8 less-than-MDC results have lower MDC's than the 1 measurement on Sample 1-P.

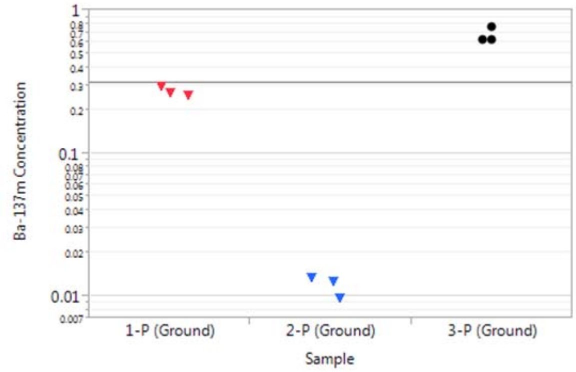
Examining the percent standard deviation for the measurement error over all samples of radionuclides with 3 measurements, a value of 20% for the measurement error percent standard deviation is larger than all but the percent standard deviation for Sample 1-P of I-129. Adopting a 20% percent standard deviation for Am-243, an individual Student's t UCL95 can be computed for Run 3 of Sample 1-P for Am-243 by $UCL95_{Run} = Measured\ Result_{Run} + t_{.95,2df} \cdot Std\ Dev$, where the $Std\ Dev_{Run} = 0.2 \cdot MeasuredResult_{Run}$ and Student's t quantile is $t_{.95,2df} = 2.92$. The UCL95 is 1.7107E-2. This UCL95 is interpreted as the MDC value for Run 3 of Sample 1 for Am-243. The MDC values for Runs 1 and 2 of Sample 1-P are 3.53E-3 and 8.92E-3, respectively. The minimum and maximum of these 2 MDC values and the computed MDC value of 1.71E-2 for Run 3 of Sample 1-P were used to produce the final Sample 1-P results in the following table.

Final Minimum Detection Concentrations (MDCs)

Sample	N	Minimum MDC	Maximum MDC
1-P	3	3.53E-3	1.71E-2
2-P	3	9.01E-5	4.04E-4
3-P	3	3.81E-3	9.95E-3
Overall	9	9.01E-5	1.71E-2

Ba-137m (μCi/g)

Mixture of Results (circles) above MDCs
and (triangles) below MDCs



Minimum Detection Concentrations (MDCs)

Sample	N	Minimum MDC	Maximum MDC
1-P	3	2.50E-1	2.89E-1
2-P	3	9.65E-3	1.34E-2

Means and Standard Deviations

Sample	N	Mean	Std Dev	Std Err Mean
3-P	3	6.74667E-1	8.7780E-2	5.068E-2

Samples 1-P and 2-P have all less-than detection results summarized in the first table (above). Only Sample 3 has 3 measurements summarized in the second table (above). The standard deviation in the above table represents only the run-to-run (measurement error) variation for Sample 3-P. There is no effective way to determine the sampling variance since measurements exist for only one sample.

UCL95's for individual Runs on Sample 3-P are computed by $UCL95_{Run} = Measured\ Result_{Run} + t_{.95,2df} \cdot Std\ Dev$, where the Student's t quantile $t_{.95,2df} = 2.92$. The results are in the following table, and are interpreted as MDC values for each of the 3 Runs from Sample 3-P. The last table provides minimum and maximum MDC values for each sample and overall for Ba-137m based on all of the less-than-MDC information from Samples 1-P and 2-P and the computed MDC's from Sample 3-P.

Individual Student's t UCL95's for Sample 3 by Run

Sample	Run 1	Run 2	Run 3
3-P	1.0323E+0	8.8232E-1	8.7832E-1

Final Minimum Detection Concentrations (MDCs)

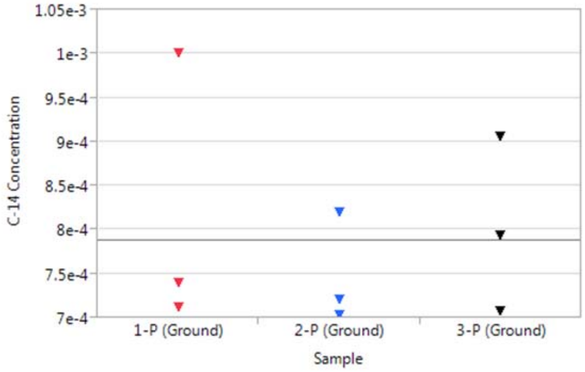
Sample	N	Minimum MDC	Maximum MDC
1-P	3	2.50E-1	2.89E-1
2-P	3	9.65E-3	1.34E-2
3-P	3	8.78E-1	1.03E+0
Overall	9	9.65E-3	1.03E+0

Appendix E: Supporting Tables for Statistical Analyses of Radionuclides

Table E17. Supporting Results for Radionuclides

C-14 (μCi/g)

All Results (triangles) below MDCs

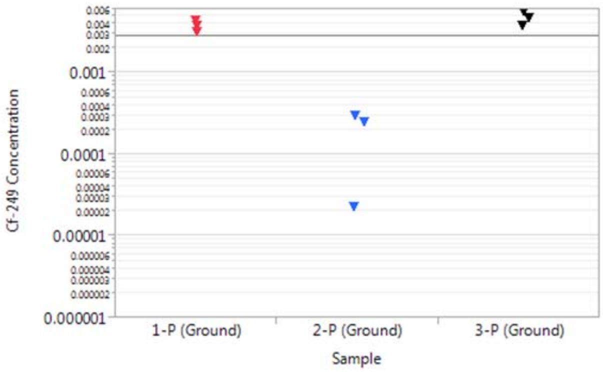


Minimum Detection Concentrations (MDCs)

Sample	Number	Minimum MDC	Maximum MDC
1-P	3	7.12E-4	1.00E-3
2-P	3	7.03E-4	8.20E-4
3-P	3	7.07E-4	9.05E-4
Overall	9	7.03E-4	1.00E-3

Cf-249 (μCi/g)

All Results (triangles) below MDCs



Minimum Detection Concentrations (MDCs)

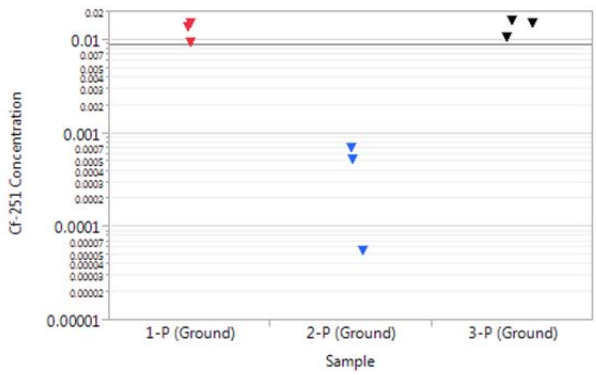
Sample	Number	Minimum MDC	Maximum MDC
1-P	3	3.09E-3	4.36E-3
2-P	3	2.23E-5	3.00E-4
3-P	3	3.70E-3	5.68E-3
Overall	9	2.23E-5	5.68E-3

Appendix E: Supporting Tables for Statistical Analyses of Radionuclides

Table E17. Supporting Results for Radionuclides

Cf-251 (μCi/g)

All Results (triangles) below MDCs

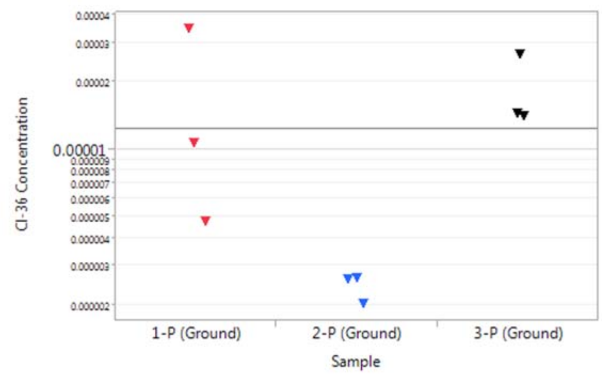


Minimum Detection Concentrations (MDCs)

Sample	Number	Minimum MDC	Maximum MDC
1-P	3	9.50E-3	1.51E-2
2-P	3	5.59E-5	6.98E-4
3-P	3	1.05E-2	1.62E-2
Overall	9	5.59E-5	1.62E-2

Cl-36 (μCi/g)

All Results (triangles) below MDCs



Minimum Detection Concentrations (MDCs)

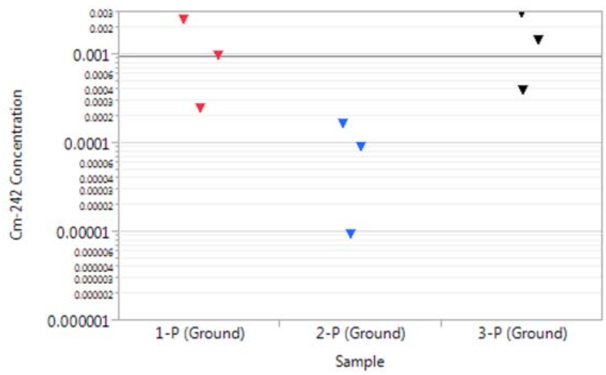
Sample	Number	Minimum MDC	Maximum MDC
1-P	3	4.73E-6	3.47E-5
2-P	3	2.02E-6	2.64E-6
3-P	3	1.41E-5	2.66E-5
Overall	9	2.02E-6	3.47E-5

Appendix E: Supporting Tables for Statistical Analyses of Radionuclides

Table E17. Supporting Results for Radionuclides

Cm-242 (μCi/g)

All Results (triangles) below MDCs

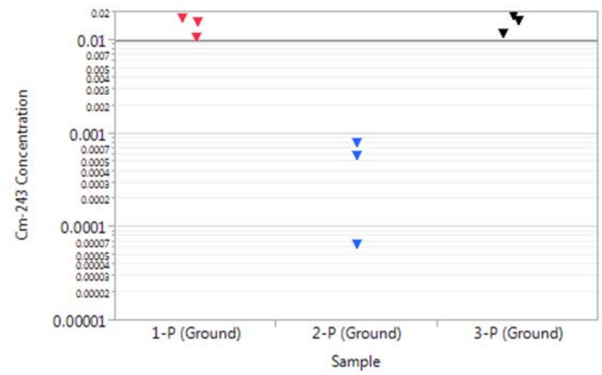


Minimum Detection Concentrations (MDCs)

Sample	Number	Minimum MDC	Maximum MDC
1-P	3	2.48E-4	2.42E-3
2-P	3	9.50E-6	1.65E-4
3-P	3	3.86E-4	2.88E-3
Overall	9	9.50E-6	2.88E-3

Cm-243 (μCi/g)

All Results (triangles) below MDCs



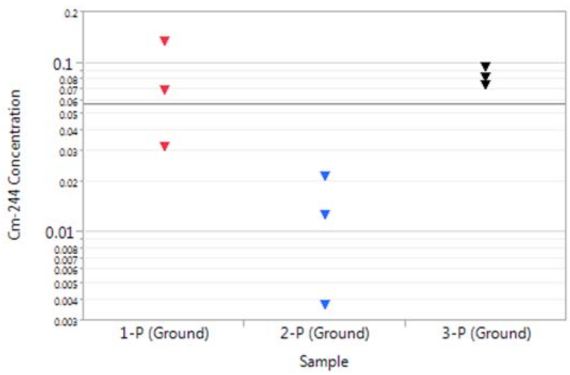
Minimum Detection Concentrations (MDCs)

Sample	Number	Minimum MDC	Maximum MDC
1-P	3	1.05E-2	1.72E-2
2-P	3	6.58E-5	7.93E-4
3-P	3	1.18E-2	1.81E-2
Overall	9	6.58E-5	1.81E-2

Appendix E: Supporting Tables for Statistical Analyses of Radionuclides

Table E17. Supporting Results for Radionuclides

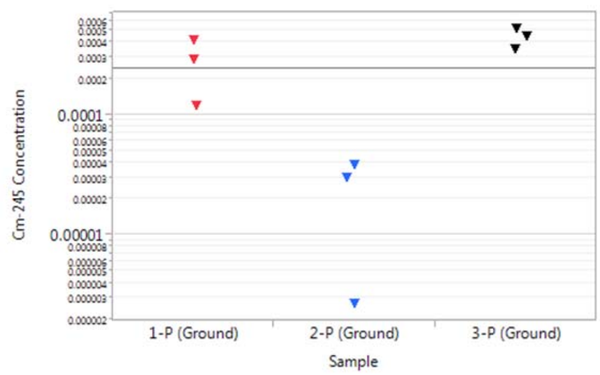
Cm-244 (μCi/g)
All Results (triangles) below MDCs



Minimum Detection Concentrations (MDCs)

Sample	Number	Minimum MDC	Maximum MDC
1-P	3	3.18E-2	1.34E-1
2-P	3	3.73E-3	2.13E-2
3-P	3	7.34E-2	9.46E-2
Overall	9	3.73E-3	1.34E-1

Cm-245 (μCi/g)
All Results (triangles) below MDCs



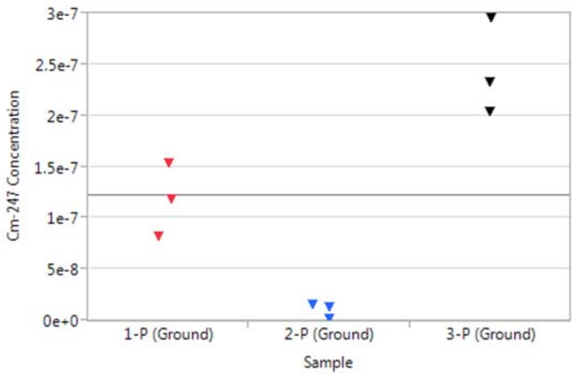
Minimum Detection Concentrations (MDCs)

Sample	Number	Minimum MDC	Maximum MDC
1-P	3	1.18E-4	4.18E-4
2-P	3	2.66E-6	3.76E-5
3-P	3	3.45E-4	5.18E-4
Overall	9	2.66E-6	5.18E-4

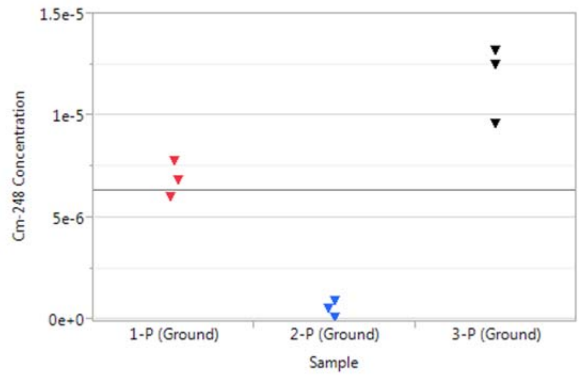
Appendix E: Supporting Tables for Statistical Analyses of Radionuclides

Table E17. Supporting Results for Radionuclides

Cm-247 (μCi/g)
All Results (triangles) below MDCs



Cm-248 (μCi/g)
All Results (triangles) below MDCs



Minimum Detection Concentrations (MDCs) (Cm-247)

Sample	Number	Minimum MDC	Maximum MDC
1-P	3	8.15E-8	1.53E-7
2-P	3	1.30E-9	1.55E-8
3-P	3	2.04E-7	2.95E-7
Overall	9	1.30E-9	2.95E-7

Minimum Detection Concentrations (MDCs) (Cm-248)

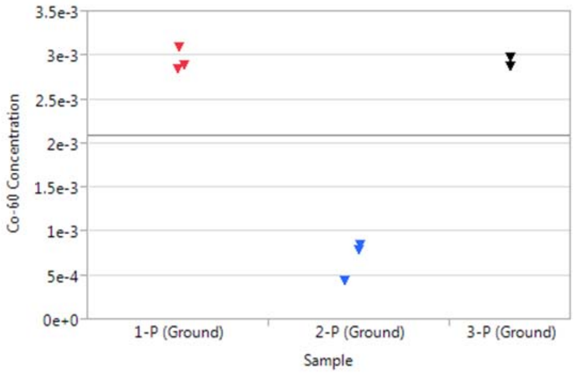
Sample	Number	Minimum MDC	Maximum MDC
1-P	3	5.99E-6	7.75E-6
2-P	3	5.81E-8	8.87E-7
3-P	3	9.59E-6	1.32E-5
Overall	9	5.81E-8	1.32E-5

Appendix E: Supporting Tables for Statistical Analyses of Radionuclides

Table E17. Supporting Results for Radionuclides

Co-60

All Results (triangles) below MDCs

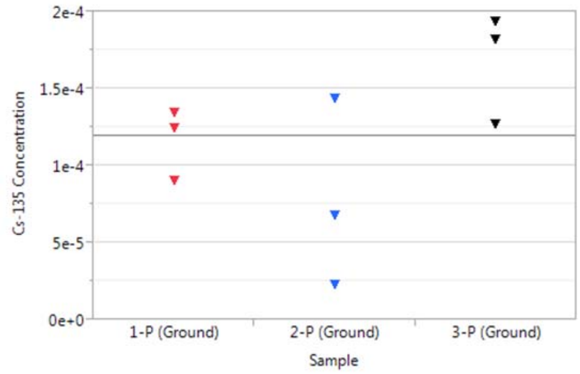


Minimum Detection Concentrations (MDCs)

Sample	Number	Minimum MDC	Maximum MDC
1-P	3	2.85E-3	3.09E-3
2-P	3	4.44E-4	8.42E-4
3-P	2	2.88E-3	2.98E-3
Overall	8	4.44E-4	3.09E-3

Cs-135

All Results (triangles) below MDCs



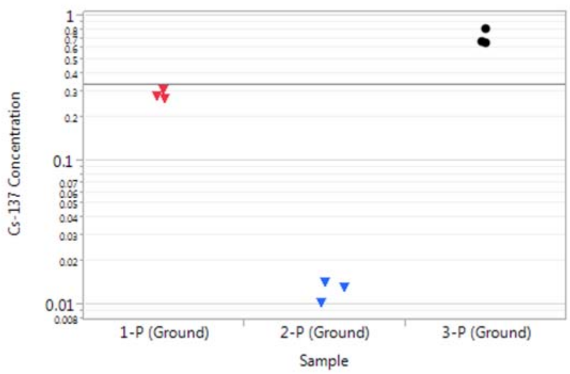
Minimum Detection Concentrations (MDCs)

Sample	Number	Minimum MDC	Maximum MDC
1-P	3	8.96E-5	1.34E-4
2-P	3	2.28E-5	1.43E-4
3-P	3	1.27E-4	1.93E-4
Overall	9	2.28E-5	1.93E-4

Appendix E: Supporting Tables for Statistical Analyses of Radionuclides

Table E17. Supporting Results for Radionuclides

Cs-137 (μCi/g)
Mixture of Results (circles) above MDCs
and (triangles) below MDCs



Minimum Detection Concentrations (MDCs)

Sample	N	Minimum MDC	Maximum MDC
1-P	3	2.64E-1	3.05E-1
2-P	3	1.02E-2	1.42E-2

Means and Standard Deviations

Sample	N	Mean	Std Dev	Std Err Mean
3-P	3	7.1333E-1	9.2398E-2	5.3346E-2

Samples 1-P and 2-P have all less-than detection results summarized in the first table (above). Only Sample 3 has 3 measurements summarized in the second table (above). The standard deviation in the above table represents only the run-to-run (measurement error) variation for Sample 3-P. There is no effective way to determine the sampling variance since measurements exist for only one sample.

UCL95's for individual Runs on Sample 3-P are computed by $UCL95_{Run} = Measured\ Result_{Run} + t_{95,2df} \cdot Std\ Dev$, where the Student's t quantile $t_{95,2df} = 2.92$.. The results are in the following table, and are interpreted as MDC values for each of the Runs from Sample 3-P. The last table provides minimum and maximum MDC values for each sample and overall for Cs-137 based on all of the less-than-MDC information from Samples 1-P and 2-P and the computed MDC's from Sample 3-P.

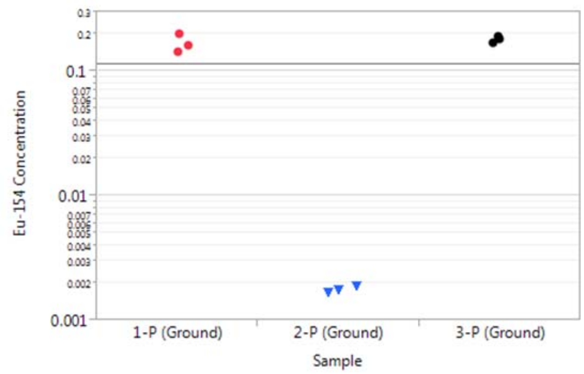
Individual Student's t UCL95's for Sample 3 by Run

Sample	Run 1	Run 2	Run 3
3-P	1.0898E+0	9.3180E-1	9.2780E-1

Final Minimum Detection Concentrations (MDCs)

Sample	N	Minimum MDC	Maximum MDC
1-P	3	2.64E-1	3.05E-1
2-P	3	1.02E-2	1.42E-2
3-P	3	9.28E-1	1.09E+0
Overall	9	1.02E-2	1.09E+0

Eu-154 (μCi/g)
Mixture of Results (circles) above MDCs
and (triangles) below MDCs



Means and Standard Deviations

Sample	N	Mean	Std Dev	Std Err Mean
1-P	3	1.6833E-1	2.8378E-2	1.6384E-2
3-P	3	1.8133E-1	1.0599E-2	6.1192E-3

Sample	Run 1 MDC	Run 2 MDC	Run 3 MDC
2-P	<1.72E-3	<1.85E-3	<1.64E-3

The mean concentration for Sample 2-P is assumed to be between 0 and 1.74 μCi/g (the mean of the MDC's for the 3 runs for Sample 2-P). The UCL95 for the data set of sample means is {1.6833E-1, X, 1.8133E-1}, where X is the mean for Sample 2-P. The non-parameteric Chebyshev (MVUE) UCL95 is maximized when the mean concentration is set to X = 0. The Maximum Chebyshev UCL95 is 3.7111E-01 μCi/g.

The associated mean and standard deviation of the data set of means {1.6833E-1, 0, 1.8133E-1} are 1.1656E-1 μCi/g and 1.0115E-1 μCi/g, respectively.

$$S_{Total} = \sqrt{S^2_{3\,Sample\,Means} - S^2_{MeasErr(Mean)} + S^2_{MeasErr(One\,Result)}}$$

$$= \sqrt{(1.1656E\text{-}1)^2 - (1.2367E\text{-}2)^2 + 3 \cdot (1.2367E\text{-}2)^2} = 1.0265E\text{-}1,$$

Where the pooled measurement error standard deviation, 2.1420E-2, is based on Samples 1-P and 3-P only.

Nonparametric Statistics

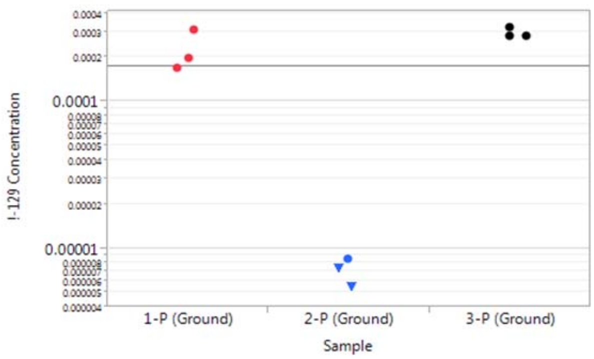
Component	Estimate
Mean	1.1656E-1
Std.Dev.	1.0265E-1
(Nonparametric) Chebyshev UCL95	3.7111E-1

Appendix E: Supporting Tables for Statistical Analyses of Radionuclides

Table E17. Supporting Results for Radionuclides

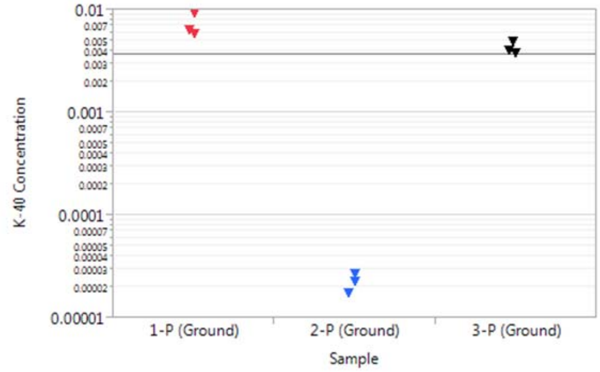
I-129 (μCi/g)

Mixture of Results (circles) above MDCs and (triangles) below MDCs



K-40 (μCi/g)

All Results (triangles) below MDCs



Means and Standard Deviations

Sample	N	Mean	Std Dev	Std Err Mean
1-P	3	2.2567E-04	7.2762E-05	4.2009E-05
3-P	3	2.9567E-04	2.3671E-05	1.3667E-05

Sample	Run 1	Run 2	Run 3
2-P	<7.30E-06	<5.45E-06	8.47E-06

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	1.4056	1	4	0.3014
Brown-Forsythe	0.8370	1	4	0.4120
Levene	4.8165	1	4	0.0932
Bartlett	1.6972	1		0.1927

Statistically non-significant measurement variance heterogeneity. Comparison of Samples 1-P and 3-P only.

Fixed Effect Model from SAS: Normal

Parameter	Estimate Effects (Std Error)	Estimate Sample Mean
Intercept	0.2957 (0.0237)	
Sample 1-P	-0.0700 (0.0335)	2.257E-1
Sample 2-P	-0.3196 (0.0390)	-2.39E-2
Sample 3-P	0.0000 (0.0000)	2.957E-1
Scale	SD=0.0410 (0.0110)	

Reject normal model due to negative mean (red) for Sample 2-P mean.

Fixed Effect Model from SAS: Lognormal

Parameter	Estimate Effects (Std Error) on Log Scale	Estimate Sample Means on Original Scale
Intercept	-8.1283 (0.1241)	2.2353E-04
Sample 1-P	-0.3008 (0.1756)	6.2521E-06
Sample 2-P	-3.8774 (0.1962)	3.0197E-04
Sample 3-P	0.0000 (0.0000)	
Overall Mean		1.7725E-4
Scale	SD=0.2150 (0.2150)	CV=21.8%

Use sample means (red) in ProUCL to obtain an estimate of UCL95.

Nonparametric Statistics

Component	Estimate
Mean	1.7725E-4
Std.Dev.	1.5319E-4
(Nonparametric) Chebyshev UCL95	5.6278E-4

Minimum Detection Concentrations (MDCs)

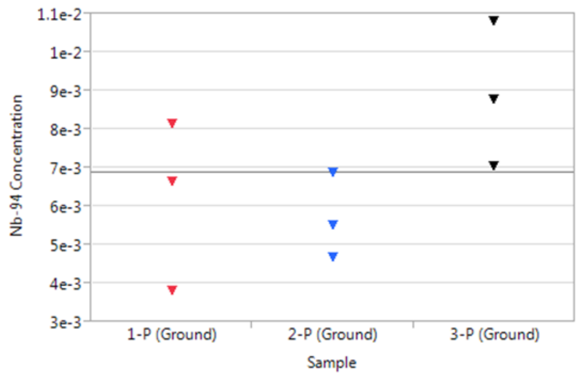
Sample	N	Minimum MDC	Maximum MDC
1-P	3	5.77E-3	9.05E-3
2-P	3	1.75E-5	2.69E-5
3-P	3	3.77E-3	4.91E-3
Overall	9	1.75E-5	9.05E-3

Appendix E: Supporting Tables for Statistical Analyses of Radionuclides

Table E17. Supporting Results for Radionuclides

Nb-94 (μCi/g)

All Results (triangles) below MDCs

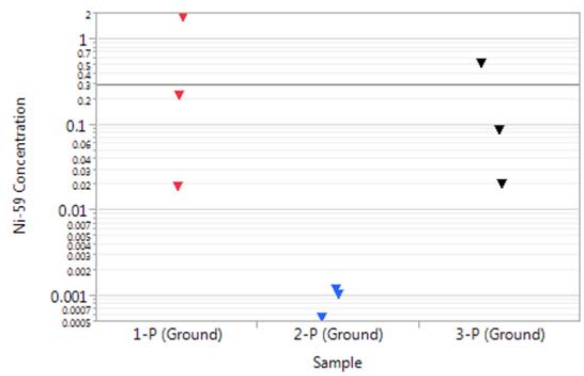


Minimum Detection Concentrations (MDCs)

Sample	Number	Minimum MDC	Maximum MDC
1-P	3	3.80E-3	8.15E-3
2-P	3	4.68E-3	6.85E-3
3-P	3	7.03E-3	1.08E-2
Overall	9	3.80E-3	1.08E-2

Ni-59 (μCi/g)

All Results (triangles) below MDCs



Minimum Detection Concentrations (MDCs)

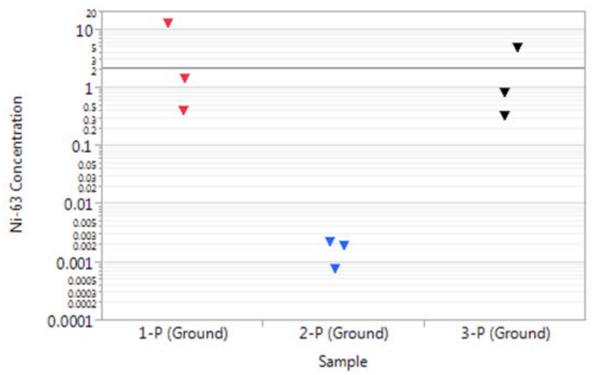
Sample	Number	Minimum MDC	Maximum MDC
1-P	3	1.90E-2	1.81E+0
2-P	3	5.50E-4	1.19E-3
3-P	3	2.05E-2	5.18E-1
Overall	9	5.50E-4	1.81E+0

Appendix E: Supporting Tables for Statistical Analyses of Radionuclides

Table E17. Supporting Results for Radionuclides

Ni-63 (μCi/g)

All Results (triangles) below MDCs

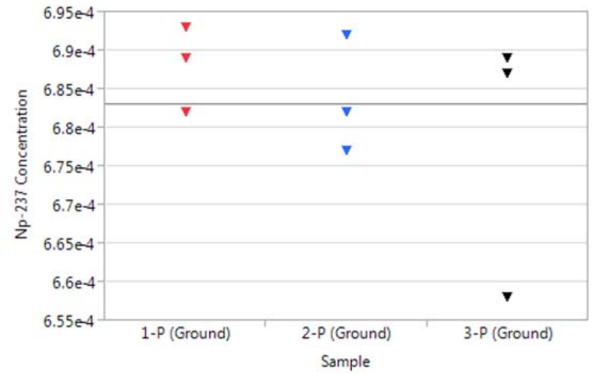


Minimum Detection Concentrations (MDCs)

Sample	Number	Minimum MDC	Maximum MDC
1-P	3	3.95E-1	1.25E+1
2-P	3	7.48E-4	2.22E-3
3-P	3	3.18E-1	4.91E+0
Overall	9	7.48E-4	1.25E+1

Np-237 (μCi/g)

All Results (triangles) below MDCs



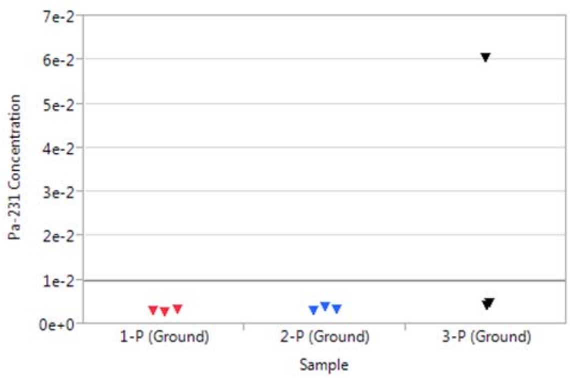
Minimum Detection Concentrations (MDCs)

Sample	Number	Minimum MDC	Maximum MDC
1-P	3	6.82E-4	6.93E-4
2-P	3	6.77E-4	6.92E-4
3-P	3	6.58E-4	6.89E-4
Overall	9	6.58E-4	6.93E-4

Appendix E: Supporting Tables for Statistical Analyses of Radionuclides

Table E17. Supporting Results for Radionuclides

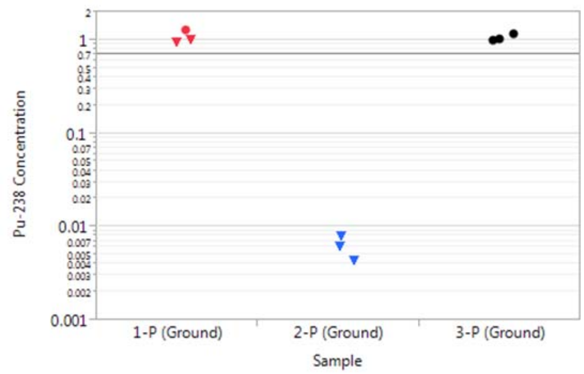
Pa-231 (μCi/g)
All Results (triangles) below MDCs



Minimum Detection Concentrations (MDCs)

Sample	Number	Minimum MDC	Maximum MDC
1-P	3	2.66E-3	3.20E-3
2-P	3	2.84E-3	3.67E-3
3-P	3	4.11E-3	6.04E-2
Overall	9	2.66E-3	6.04E-2

Pu-238 (μCi/g)
Mixture of Results (circles) above MDCs
and (triangles) below MDCs



Mean and Standard Deviation

Sample	N	Mean	Std Dev	Std Err Mean
3-P	3	1.07000	0.087178	0.05033

Sample	Run 1	Run 2	Run 3
1-P	<9.95E-1	<9.41E-1	1.27
2-P	<6.04E-3	<4.24E-3	<7.84E-3

The mean concentration for Sample 1-P is assumed to be between 1.27/3 = 4.2333E-1 and 1.0687 μCi/g, and the mean concentration of Sample 2-P is assumed to be between 0 and 6.04e-3 μCi/g. The nonparametric Chebyshev (MVUE) UCL95 in maximized over the data set of sample means {X, Y, 1.0700}, where the pair {X, Y} is the Cartesian product: [4.2333E-01, 1.0687] x [0, 9.6800E-01], and 1.0700 is the Sample 3 mean concentration. The nonparametric Chebyshev (MVUE) UCL95 has been found to be maximized when the mean concentrations for Samples 1 and 2 are 1.0687 and 0. The Maximum Chebyshev UCL95 is 2.2666 μCi/g.

The associated mean and standard deviation of the data set of means {1.0687, 0, 1.0700} are 7.1289E-01 μCi/g and 6.1738E-01 μCi/g, respectively.

$$S_{Total} = \sqrt{S^2_{3\,Sample\,Means} - S^2_{MeasErr(Mean)} + S^2_{MeasErr(One\,Result)}}$$

$$= \sqrt{(7.1289E\text{-}1)^2 - (5.0332E\text{-}2)^2 + 3\cdot(5.0332E\text{-}2)^2} = 6.2147E\text{-}1,$$

where the measurement standard error of the mean from Sample 3 is 8.7178E-02 μCi/g.

Nonparametric Statistics

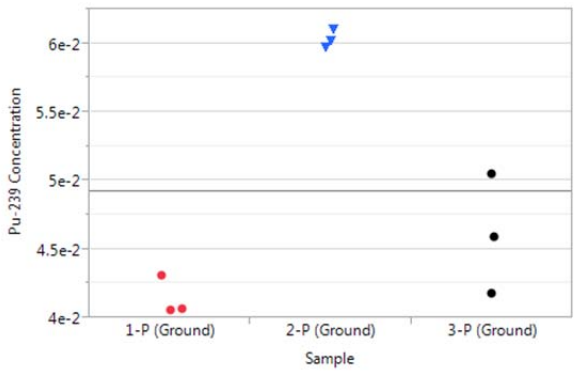
Component	Estimate
Mean	7.1289E-01
Std.Dev.	6.2147E-01
(Nonparametric) Chebyshev UCL95	2.2666

Appendix E: Supporting Tables for Statistical Analyses of Radionuclides

Table E17. Supporting Results for Radionuclides

Pu-239 (μCi/g)

Mixture of Results (circles) above MDCs
and (triangles) below MDCs



Means and Standard Deviations

Sample	N	Mean	Std Dev	Std Err Mean
1-P	3	4.1467E-2	1.4154E-3	8.1718E-4
3-P	3	4.6067E-2	4.3524E-3	2.5129E-3

Sample	Run 1 MDC	Run 2 MDC	Run 3 MDC
2-P	<6.10E-02	<6.02E-02	<5.97E-02

The mean concentration for Sample 2-P is assumed to be between 0 and 6.03E-02 μCi/g, the mean of the MDC's for the 3 runs for Sample 2-P. The data set of sample mean concentrations is {4.1467E-2, X, 4.6067E-2}, where X is the mean for Sample 2-P. The non-parameteric Chebyshev (Minimum Variance Unbiased Estimator or MVUE) UCL95 is maximized when the mean concentration for Sample 2-P is set to X = 0. The Maximum Chebyshev UCL95 is 9.3033E-2 μCi/g.

The associated mean and standard deviation of the data set of means {4.1467E-2, 0, 4.6067E-2} are 2.9178E-2 μCi/g and 2.5373E-2 μCi/g, respectively.

$$S_{Total} = \sqrt{S^2_{3\,Sample\,Means} - S^2_{MeasErr(Mean)} + S^2_{MeasErr(One\,Result)}}$$

$$= \sqrt{(2.5373E\text{-}2)^2 - (1.8685E\text{-}3)^2 + 3\cdot(1.8685E\text{-}3)^2} = 2.5511E\text{-}2,$$

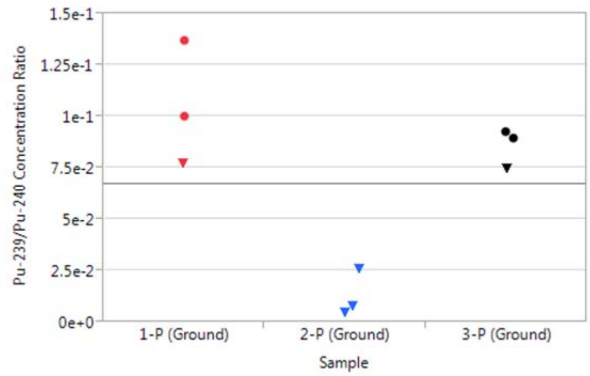
where the pooled standard deviation, 3.2359E-03 μCi/g, is based on Samples 1-P and 3-P only.

Nonparametric Statistics

Component	Estimate
Mean	2.9178E-2
Std.Dev.	2.5511E-2
(Nonparametric) Chebyshev UCL95	9.3033E-2

Pu-239/Pu-240 (dimensionless)

Mixture of Results (circles) above MDCs
and (triangles) below MDCs



Minimum Detection Concentrations (MDCs)

Sample	Number	Minimum MDC	Maximum MDC
2-P	3	0.00421	0.0255

Fixed Effect Model from SAS: Normal

Parameter	Estimate Effects (Std Error)	Estimate Sample Mean
Intercept	79.1555	
Sample 1-P	20.8611	100.0166
Sample 3-P	0	79.1555
Scale	25.5687	

Only the Scale (measurement error standard deviation) estimate is used to compute the total standard deviation, and it is not used to compute the UCL95. The mean for Sample 1-P is assumed to be between 7.9000E-2 and 1.0467E-1 μCi/g, the mean for Sample 2-P was assumed to be in the interval 0 to 1.2457E-2 μCi/g, and the mean for Sample 3-P was assumed to be in the interval 6.0633E-2 to 8.5400E-2 μCi/g. The data set of plausible sample means is {X, Y, Z}, where X (Sample 1-P Mean), Y (Sample 2-P Mean), and Z (Sample 3-P Mean) took on values in their respective intervals. The non-parametric Chebyshev (Minimum Variance Unbiased Estimator or MVUE) UCL95 is maximized when the mean concentration for Sample 2-P is set to X (Sample 1 Mean) = 1.0467E-1, Y (Sample 2 Mean) = 0, and Z (Sample 3 Mean) = 8.5400E-2 μCi/g. The Maximum Chebyshev UCL95 is 2.0355E-01 μCi/g.

The associated mean and standard deviation of the data set of means {1.0467E-1, 0, 8.5400E-2} are 6.3356E-2 μCi/g and 5.5707E-2 μCi/g, respectively.

$$S_{Total} = \sqrt{S^2_{3\,Sample\,Means} - S^2_{MeasErr(Mean)} + S^2_{MeasErr(One\,Result)}}$$

$$= \sqrt{(6.3356E\text{-}2)^2 - (1.4780E\text{-}2)^2 + 3\cdot(1.4780E\text{-}2)^2} = 5.9499E\text{-}2,$$

where the pooled standard deviation, 2.5600E-2 μCi/g, is based on the SAS output for Samples 1-P and 3-P only.

Nonparametric Statistics

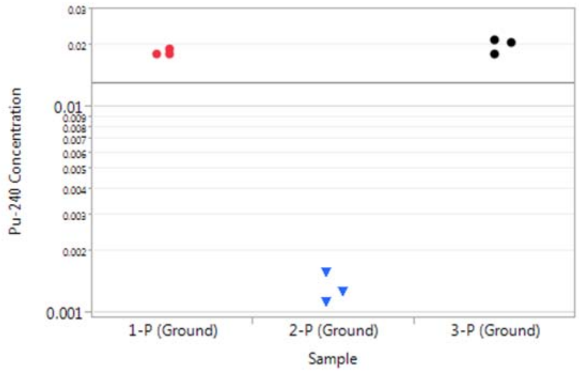
Component	Estimate
Mean	6.3356E-2
Std.Dev.	5.9499E-2
(Nonparametric) Chebyshev UCL95	2.0355E-1

Appendix E: Supporting Tables for Statistical Analyses of Radionuclides

Table E17. Supporting Results for Radionuclides

Pu-240 (μCi/g)

Mixture of Results (circles) above MDCs
and (triangles) below MDCs



Means and Standard Deviations

Sample	N	Mean	Std Dev	Std Err Mean
1-P	3	1.8400E-02	6.0828E-04	3.5119E-04
3-P	3	1.9933E-02	1.5308E-03	8.8380E-04

Sample	Run 1 MDC	Run 2 MDC	Run 3 MDC
2-P	<1.13e-3	<1.58e-3	<1.27e-3

The mean concentration for Sample 2-P is assumed to be between 0 and 1.33E-03 μCi/g, the mean of the MDC's for the 3 runs for Sample 2-P. The data set of sample mean concentrations is {1.8400E-02, X, 1.9900E-02}, where X is the mean for Sample 2-P. The non-parametric Chebyshev (Minimum Variance Unbiased Estimator or MVUE) UCL95 is maximized when the mean concentration for Sample 2-P is set to X = 0. The Maximum Chebyshev UCL95 is 4.0655E-02 μCi/g.

The associated mean and standard deviation of the data set of means {1.8400E-02, 0, 1.9900E-02} are 1.2778E-02 μCi/g and 1.1092E-02 μCi/g, respectively.

$$S_{Total} = \sqrt{S^2_{3\,Sample\,Means} - S^2_{MeasErr(Mean)} + S^2_{MeasErr(One\,Result)}}$$

$$= \sqrt{(1.2778E\text{-}2)^2 - (6.7247E\text{-}4)^2 + 3\cdot(1.1648E\text{-}3)^2} = 1.1133E\text{-}2,$$

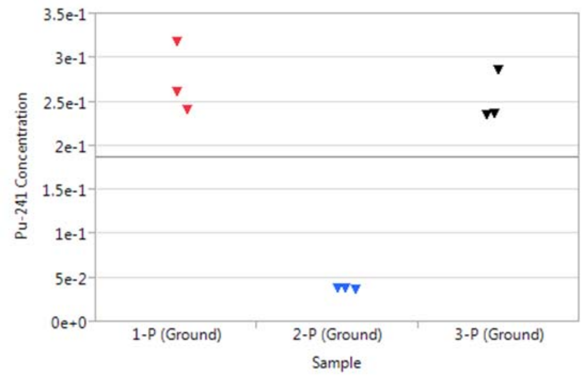
where the pooled standard deviation, 1.1648E-03 μCi/g, is based on Samples 1-P and 3-P only.

Nonparametric Statistics

Component	Estimate
Mean	1.2778E-2
Std.Dev.	1.1133E-2
(Nonparametric) Chebyshev UCL95	4.0655E-2

Pu-241 (μCi/g) below

All Results (triangles) below MDCs



Minimum Detection Concentrations (MDCs)

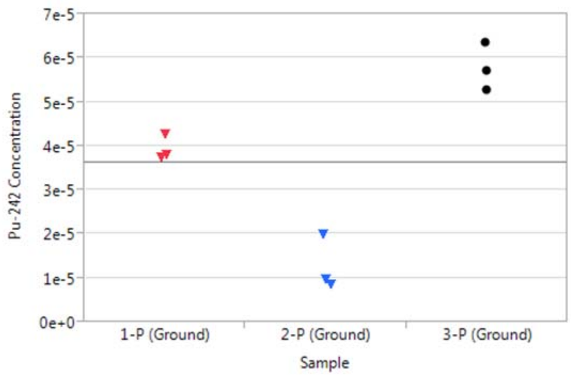
Sample	Number	Minimum MDC	Maximum MDC
1-P	3	2.40E-1	3.18E-1
2-P	3	3.68E-2	3.80E-2
3-P	3	2.35E-1	2.86E-1
Overall	9	3.68E-2	3.18E-1

Appendix E: Supporting Tables for Statistical Analyses of Radionuclides

Table E17. Supporting Results for Radionuclides

Pu-242 (μCi/g)

Mixture of Results (circles) above MDCs and (triangles) below MDCs



Minimum Detection Concentrations (MDCs)

Sample	N	Minimum MDC	Maximum MDC
1-P	3	3.74E-05	4.25E-05
2-P	3	8.42E-06	1.97E-05

Means and Standard Deviations

Sample	N	Mean	Std Dev	Std Err Mean
3-P	3	5.7800E-05	5.4249E-06	3.1321E-06

Samples 1-P and 2-P have all less-than MDC results summarized in the first table (above). Only Sample 3 has 3 measurements summarized in the second table (above). The standard deviation in the above table represents only the run-to-run (measurement error) variation for Sample 3-P. There is no effective way to determine the sampling variance since measurements exist for only one sample.

UCL95's for individual Runs on Sample 3-P are computed by $UCL95_{Run} = Measured\ Result_{Run} + t_{.95,2df} \cdot Std\ Dev$, where the Student's t quantile $t_{.95,2df} = 2.92$.. The results are in the following table, and are interpreted as MDC values for each of the Runs from Sample 3-P. The last table provides minimum and maximum MDC values for each sample and overall for Pu-242 based on all of the less-than-MDC information from Samples 1-P and 2-P and the computed MDC's from Sample 3-P.

Individual Student's t UCL95's for Sample 3-P by Run

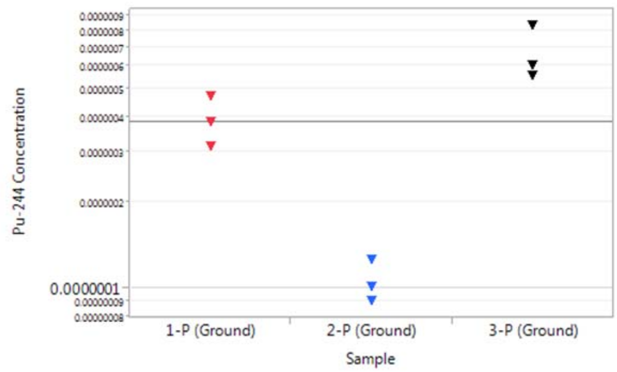
Sample	Run 1	Run 2	Run 3
3-P	6.8541E-05	7.3041E-05	7.9341E-05

Final Minimum Detection Concentrations (MDCs)

Sample	N	Minimum MDC	Maximum MDC
1-P	3	3.74E-05	4.25E-05
2-P	3	8.42E-06	1.97E-05
3-P	3	6.85E-05	7.93E-05
Overall	9	8.42E-06	7.93E-05

Pu-244 (μCi/g)

All Results (triangles) below MDCs



Minimum Detection Concentrations (MDCs)

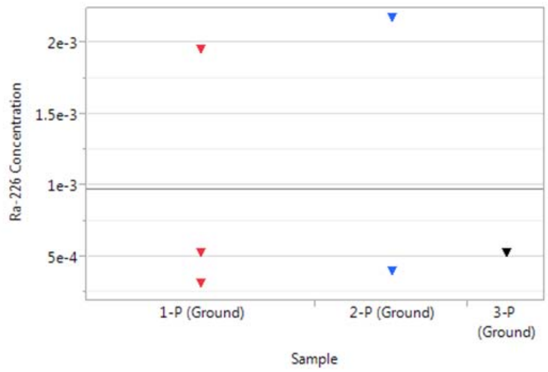
Sample	Number	Minimum MDC	Maximum MDC
1-P	3	3.14E-7	4.68E-7
2-P	3	9.01E-8	1.26E-7
3-P	3	5.54E-7	8.33E-7
Overall	9	9.01E-8	8.33E-7

Appendix E: Supporting Tables for Statistical Analyses of Radionuclides

Table E17. Supporting Results for Radionuclides

Ra-226 (µCi/g)

All Results (triangles) below MDCs

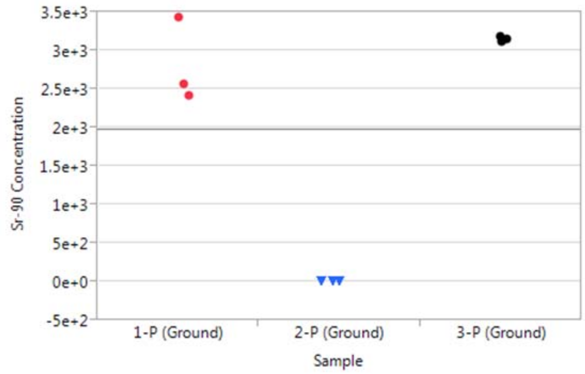


Minimum Detection Concentrations (MDCs)

Sample	Number	Minimum MDC	Maximum MDC
1-P	3	3.15E-4	1.95E-3
2-P	2	3.94E-4	2.17E-3
3-P	1	5.27E-4	5.27E-4
Overall	6	3.15E-4	2.17E-3

Sr-90 (µCi/g)

Mixture of Results (circles) above MDCs and (triangles) below MDCs



Means and Standard Deviations

Sample	N	Mean	Std Dev	Std Err Mean
1-P	3	2,803.33	548.574	316.72
3-P	3	3,150.00	30.000	17.32

Sample	Run 1 MDC	Run 2 MDC	Run 3 MDC
2-P	<5.00	<4.41	<5.77

The mean concentration for Sample 2-P is assumed to be between 0 and 5.06 µCi/g, the mean of the MDC's for the 3 runs for Sample 2-P. The data set of sample mean concentrations is {2,803.33; X; 3,150.00}, where X is the mean for Sample 2-P. The non-parameteric Chebyshev (Minimum Variance Unbiased Estimator or MVUE) UCL95 is maximized when the mean concentration for Sample 2-P is set to X = 0. The Maximum Chebyshev UCL95 is 6,331.4 µCi/g.

The associated mean and standard deviation of the data set of means {2,803.33; 0; 3,150.00} are 1,984.4 µCi/g and 1,727.3 µCi/g, respectively.

$$S_{Total} = \sqrt{S^2_{3\,Sample\,Means} - S^2_{MeasErr(Mean)} + S^2_{MeasErr(One\,Result)}}$$

$$= \sqrt{(1{,}727.3)^2 - (224.29)^2 + 3\cdot(224.29)^2} = 1{,}756.2,$$

where the pooled standard deviation, 3.8848E+02 □Ci/g, is based on Samples 1-P and 3-P only.

Nonparametric Statistics

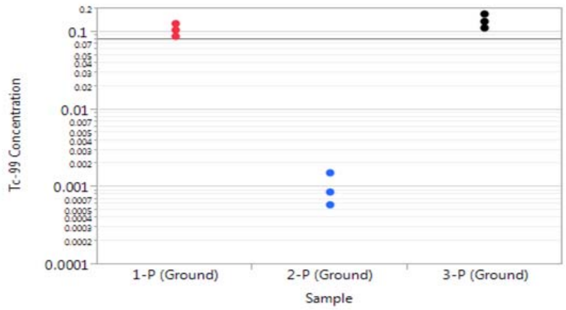
Component	Estimate
Mean	1.9844E+3
Std.Dev.	1.7562E+3
(Nonparametric) Chebyshev UCL95	6.3314E+3

Appendix E: Supporting Tables for Statistical Analyses of Radionuclides

Table E17. Supporting Results for Radionuclides

Tc-99 (μCi/g)

All Results (circles) above MDCs



Means and Standard Deviations (Response: Log Conc)

Sample	N	$Mean_{Log(Conc)}$	$SD_{Log(Conc)}$	Sample Mean in Orig. Units
1-P	3	-2.2489	0.19081	0.107306
2-P	3	-6.9934	0.46848	0.000934
3-P	3	-1.9821	0.21811	0.140127

Overall Mean	$s_{Pooled\ Log(Conc)}$	$Std.Err.Mean_{Pooled\ Log(Conc)}$
0.082789	0.31804	0.18362

$$Sample\ Mean_{Original\ Units} = e^{Sample\ Mean_{Log(Conc)} + \frac{(S_{Pooled\ Log(Conc)})^2}{2}}$$

Note that the 3 Sample Means in the Original Units were input to ProUCL using the lognormal distribution. $S_{3SampleMeans} = 0.072763$. UCL95 (Chebyshev lognormal data) = 0.74765.

$$S_{Total} = \sqrt{S^2_{3\,Sample\,Means} - S^2_{MeasErr(Mean)} + S^2_{MeasErr(One\,Result)}}$$

$$= \sqrt{(5.2945E\text{-}3) - (2.3504E\text{-}4) + 3 \cdot (2.3504E\text{-}4)} = 7.5925E\text{-}2$$

Tests for Equal Measurement Variances (Log Conc)

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	1.0852	2	6	0.3960
Brown-Forsythe	0.8663	2	6	0.4672
Levene	1.2757	2	6	0.3454
Bartlett	0.8194	2		0.4407

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance (Response: Log of Concentration)

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	4.7694E+1	2.3847E+1	235.754	<0.0001
Error	6	6.0691E-1	1.0115E-1		
C. Total	8	4.8301E+1			

Statistically Significant Sampling Variance.

Diagnostics for Sampling Model Residuals of Log{Conc}

Shapiro-Wilk	0.977> 0.829 Critical Value->SNS
Dixon High Outlier	Run 2 of Sample 2 -> SNS
Dixon Low Outlier	Run 1 of Sample 2 -> SNS

Continuation of Tc-99 (μCi/g)

Statistics for Tc-99

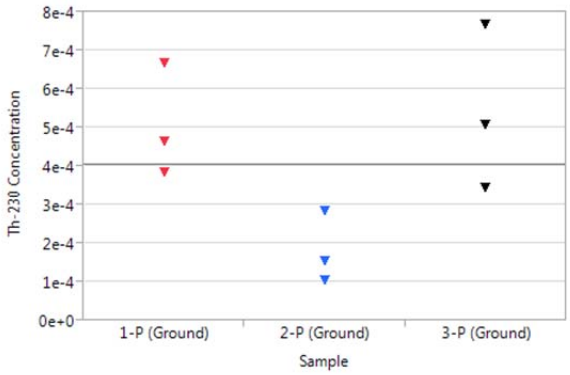
Component	Estimate
Mean	8.2789E-2
Std.Dev.	7.5925E-2
(Lognormal) Chebyshev UCL95	7.2277E-1

Appendix E: Supporting Tables for Statistical Analyses of Radionuclides

Table E17. Supporting Results for Radionuclides

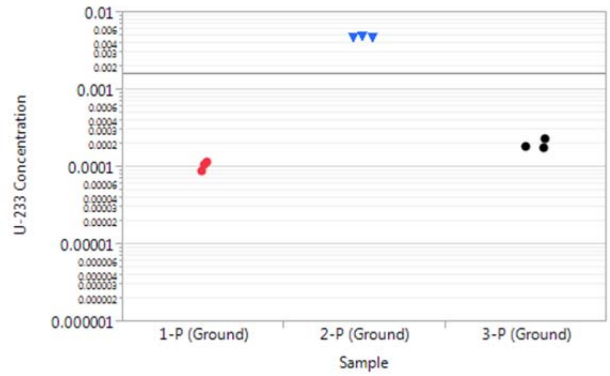
Th-230 (μCi/g)

All Results (triangles) below MDCs



U-233 (μCi/g)

Mixture of Results (circles) above MDCs and (triangles) below MDCs



Minimum Detection Concentrations (MDCs)

Sample	Number	Minimum MDC	Maximum MDC
1-P	3	3.84E-4	6.67E-4
2-P	3	1.02E-4	2.84E-4
3-P	3	3.43E-4	7.66E-4
Overall	9	1.02E-4	7.66E-4

Means and Standard Deviations

Sample	N	Mean	Std Dev	Std Err Mean
1-P	3	1.0473E-04	1.3900E-05	8.0252E-06
3-P	3	1.9733E-04	3.0989E-05	1.7892E-05

Sample	Run 1 MDC	Run 2 MDC	Run 3 MDC
2-P	<4.75E-03	<4.69E-03	<4.65E-03

The mean concentration for Sample 2-P is assumed to be between 0 and 4.70E-03 μCi/g, the mean of the MDC's for the 3 runs for Sample 2-P. The data set of sample mean concentrations is {1.0473E-04, X, 1.9733E-04}, where X is the mean for Sample 2-P. The non-parameteric Chebyshev (Minimum Variance Unbiased Estimator or MVUE) UCL95 is maximized when the mean concentration for Sample 2-P is set to X = 4.70E-03. The Maximum Chebyshev UCL95 is 8.2779E-03 μCi/g.

The associated mean and standard deviation of the data set of means {1.0473E-04, 4.70E-03, 1.9733E-04} are 1.6662E-03 μCi/g and 2.6248E-03 μCi/g, respectively.

$$S_{Total} = \sqrt{S^2_{3\,Sample\,Means} - S^2_{MeasErr(Mean)} + S^2_{MeasErr(One\,Result)}}$$

$$= \sqrt{(2.6248E\text{-}3)^2 - (1.3866E\text{-}5)^2 + 3 \cdot (1.3866E\text{-}5)^2} = 2.6249E\text{-}3,$$

where the pooled standard deviation, 2.4016E-05 μCi/g, is based on Samples 1-P and 3-P only.

Nonparametric Statistics

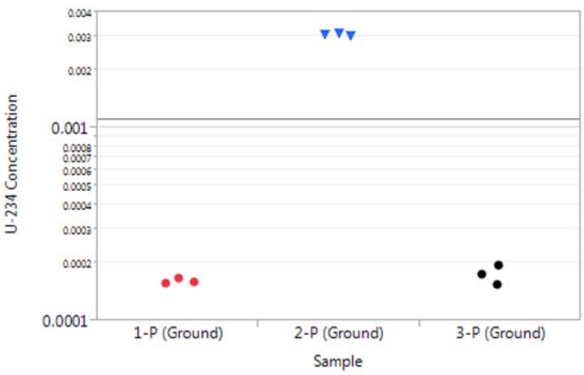
Component	Estimate
Mean	1.6662E-3
Std.Dev.	2.6249E-3
(Nonparametric) Chebyshev UCL95	8.2779E-3

Appendix E: Supporting Tables for Statistical Analyses of Radionuclides

Table E17. Supporting Results for Radionuclides

U-234 (μCi/g)

Mixture of Results (circles) above MDCs
and (triangles) below MDCs



Means and Standard Deviations

Sample	N	Mean	Std Dev	Std Err Mean
1-P	3	1.6033E-04	6.1101E-06	3.5277E-06
3-P	3	1.7333E-04	2.0008E-05	1.1552E-05

Sample	Run 1 MDC	Run 2 MDC	Run 3 MDC
2-P	<3.07E-03	<3.02E-03	<3.00E-03

The mean concentration for Sample 2-P is assumed to be between 0 and 3.03E-03 μCi/g, the mean of the MDC's for the 3 runs for Sample 2-P. The data set of sample mean concentrations is {1.6033E-04, X, 1.7333E-04}, where X is the mean for Sample 2-P. The non-parameteric Chebyshev (Minimum Variance Unbiased Estimator or MVUE) UCL95 is maximized when the mean concentration for Sample 2-P is set to X = 3.03E-03. The Maximum Chebyshev UCL95 is 5.2813E-03 μCi/g.

The associated mean and standard deviation of the data set of means {1.6033E-04, 3.03E-03, 1.7333E-04} are 1.1212E-03 μCi/g and 1.6531E-03 μCi/g, respectively.

$$S_{Total} = \sqrt{S^2_{3\,Sample\,Means} - S^2_{MeasErr(Mean)} + S^2_{MeasErr(One\,Result)}}$$

$$= \sqrt{(1.6531E\text{-}3)^2 - (8.5408E\text{-}6)^2 + 3\cdot(8.5408E\text{-}6)^2} = 1.6531E\text{-}3,$$

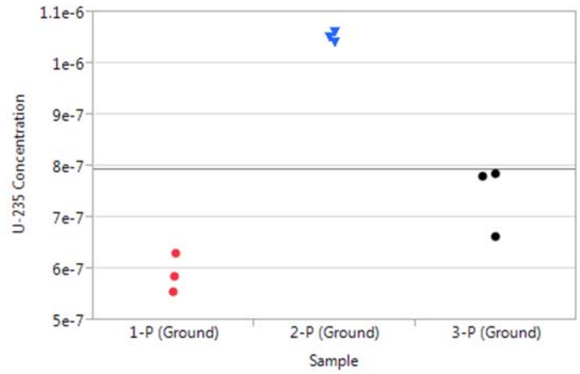
where the pooled standard deviation, 1.4793E-05 μCi/g, is based on Samples 1-P and 3-P only.

Nonparametric Statistics

Component	Estimate
Mean	1.1212E-3
Std.Dev.	1.6531E-3
(Nonparametric) Chebyshev UCL95	5.2813E-3

U-235 (μCi/g)

Mixture of Results (circles) above MDCs
and (triangles) below MDCs



Means and Standard Deviations

Sample	N	Mean	Std Dev	Std Err Mean
1-P	3	5.9033E-07	3.8682E-08	2.2333E-08
3-P	3	7.4167E-07	6.9039E-08	3.9859E-08

Sample	Run 1 MDC	Run 2 MDC	Run 3 MDC
2-P	<1.06E-06	<1.05E-06	<1.04E-06

The mean concentration for Sample 2-P is assumed to be between 0 and 1.05E-06 μCi/g, the mean of the MDC's for the 3 runs for Sample 2-P. The data set of sample mean concentrations is {5.9033E-07, X, 7.4167E-07}, where X is the mean for Sample 2-P. The non-parameteric Chebyshev (Minimum Variance Unbiased Estimator or MVUE) UCL95 is maximized when the mean concentration for Sample 2-P is set to X = 0. The Maximum Chebyshev UCL95 is 1.4302E-06 μCi/g.

The associated mean and standard deviation of the data set of means {5.9033E-07, 0, 7.4167E-07} are 4.4400E-07 μCi/g and 3.9189E-07 μCi/g, respectively.

$$S_{Total} = \sqrt{S^2_{3\,Sample\,Means} - S^2_{MeasErr(Mean)} + S^2_{MeasErr(One\,Result)}}$$

$$= \sqrt{(3.9189E\text{-}7)^2 - (3.2308E\text{-}8)^2 + 3\cdot(3.2308E\text{-}8)^2} = 3.9454E\text{-}7,$$

where the pooled standard deviation, 5.5958E-08 μCi/g, is based on Samples 1-P and 3-P only.

Nonparametric Statistics

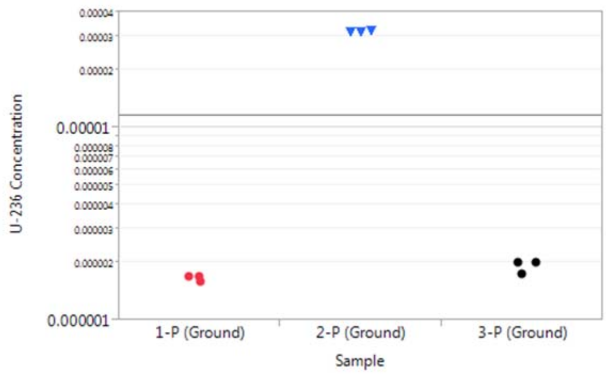
Component	Estimate
Mean	4.4400E-7
Std.Dev.	3.9454E-7
(Nonparametric) Chebyshev UCL95	1.4302E-6

Appendix E: Supporting Tables for Statistical Analyses of Radionuclides

Table E17. Supporting Results for Radionuclides

U-236 (μCi/g)

Mixture of Results (circles) above MDCs
and (triangles) below MDCs



Means and Standard Deviations

Sample	N	Mean	Std Dev	Std Err Mean
1-P	3	1.6533E-06	5.5076E-08	3.1798E-08
3-P	3	1.9200E-06	1.5588E-07	9.0000E-08

Sample	Run 1 MDC	Run 2 MDC	Run 3 MDC
2-P	<3.18E-05	<3.13E-05	<3.11E-05

The mean concentration for Sample 2-P is assumed to be between 0 and 3.14E-05 μCi/g, the mean of the MDC's for the 3 runs for Sample 2-P. The data set of sample mean concentrations is {1.6533E-06, X, 1.9200E-06}, where X is the mean for Sample 2-P. The non-parameteric Chebyshev (Minimum Variance Unbiased Estimator or MVUE) UCL95 is maximized when the mean concentration for Sample 2-P is set to X = 3.14E-05. The Maximum Chebyshev UCL95 is 5.4686E-05 μCi/g.

The associated mean and standard deviation of the data set of means {1.6533E-06, 3.14E-05, 1.9200E-06} are 1.1658E-05 μCi/g and 1.7098E-05 μCi/g, respectively.

$$S_{Total} = \sqrt{S^2_{3 Sample Means} - S^2_{MeasErr(Mean)} + S^2_{MeasErr(One Result)}}$$

$$= \sqrt{(1.7098E\text{-}5)^2 - (6.7495E\text{-}8)^2 + 3 \cdot (6.7495E\text{-}8)^2} = 1.7098E\text{-}5,$$

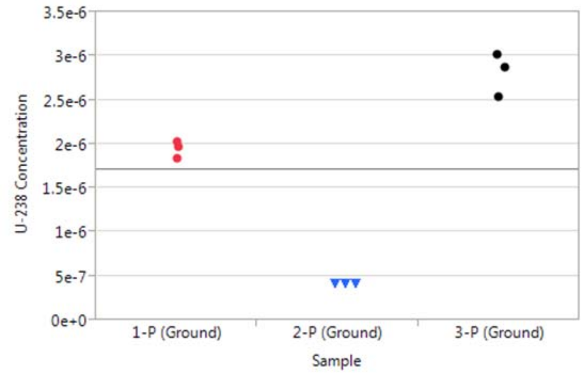
where the pooled standard deviation, 1.1690E-07 μCi/g, is based on Samples 1-P and 3-P only.

Nonparametric Statistics

Component	Estimate
Mean	1.1658E-5
Std.Dev.	1.7098E-5
(Nonparametric) Chebyshev UCL95	5.4686E-5

U-238 (μCi/g)

Mixture of Results (circles) above MDCs
and (triangles) below MDCs



Means and Standard Deviations

Sample	N	Mean	Std Dev	Std Err Mean
1-P	3	1.9467E-06	9.7125E-08	5.6075E-08
3-P	3	2.8100E-06	2.4556E-07	1.4177E-07

Sample	Run 1 MDC	Run 2 MDC	Run 3 MDC
2-P	<4.14E-07	<4.07E-07	<4.03E-07

The mean concentration for Sample 2-P is assumed to be between 0 and 4.08E-07 μCi/g, the mean of the MDC's for the 3 runs for Sample 2-P. The data set of sample mean concentrations is {1.9467E-06, X, 2.8100E-06}, where X is the mean for Sample 2-P. The non-parameteric Chebyshev (Minimum Variance Unbiased Estimator or MVUE) UCL95 is maximized when the mean concentration for Sample 2-P is set to X = 0. The Maximum Chebyshev UCL95 is 5.2079E-06 μCi/g.

The associated mean and standard deviation of the data set of means {1.9467E-06, 0, 2.8100E-06} are 1.5856E-06 μCi/g and 1.4394E-06 μCi/g, respectively.

$$S_{Total} = \sqrt{S^2_{3 Sample Means} - S^2_{MeasErr(Mean)} + S^2_{MeasErr(One Result)}}$$

$$= \sqrt{(1.4394E\text{-}6)^2 - (1.0781E\text{-}7)^2 + 3 \cdot (1.0781E\text{-}7)^2} = 1.4474E\text{-}6,$$

where the pooled standard deviation, 1.8673E-07 μCi/g, is based on Samples 1-P and 3-P only.

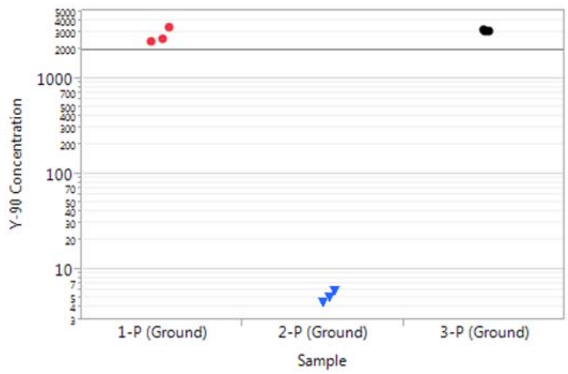
Nonparametric Statistics

Component	Estimate
Mean	1.5856E-6
Std.Dev.	1.4474E-6
(Nonparametric) Chebyshev UCL95	5.2079E-6

Appendix E: Supporting Tables for Statistical Analyses of Radionuclides

Table E17. Supporting Results for Radionuclides

Y-90 (μCi/g)
Mixture of Results (circles) above MDCs
and (triangles) below MDCs



Means and Standard Deviations

Sample	N	Mean	Std Dev	Std Err Mean
1-P	3	2,803.33	548.574	316.72
3-P	3	3,150.00	30.000	17.32

Sample	Run 1 MDC	Run 2 MDC	Run 3 MDC
2-P	<5.00	<4.41	<5.77

The mean concentration for Sample 2-P is assumed to be between 0 and 5.06 μCi/g, the mean of the MDC's for the 3 runs for Sample 2-P. The data set of sample mean concentrations is {2,803.33; X; 3,150.00}, where X is the mean for Sample 2-P. The non-parametric Chebyshev (Minimum Variance Unbiased Estimator or MVUE) UCL95 is maximized when the mean concentration for Sample 2-P is set to X = 0. The Maximum Chebyshev UCL95 is 6,331.4 μCi/g.

The associated mean and standard deviation of the data set of means {2,803.33; 0; 3,150.00} are 1,984.4 μCi/g and 1,727.3 μCi/g, respectively.

$$S_{Total} = \sqrt{S^2_{3 Sample Means} - S^2_{MeasErr(Mean)} + S^2_{MeasErr(One Result)}}$$

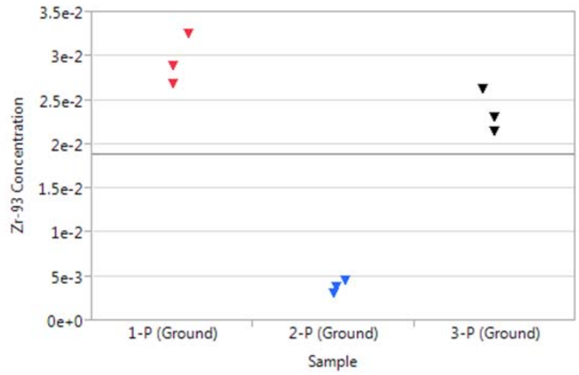
$$= \sqrt{(1.7273E+3)^2 - (2.2429E+2)^2 + 3 \cdot (2.2429E+2)^2} = 1{,}756.2,$$

where the pooled standard deviation, 3.8848E+02 μCi/g, is based on Samples 1-P and 3-P only.

Nonparametric Statistics

Component	Estimate
Mean	1.9844E+3
Std.Dev.	1.7562E+3
(Nonparametric) Chebyshev UCL95	6.3314E+3

Zr-93 (μCi/g)
All Results (triangles) below MDCs



Minimum Detection Concentrations (MDCs)

Sample	Number	Minimum MDC	Maximum MDC
1-P	3	2.68E-2	3.25E-2
2-P	3	3.05E-3	4.59E-3
3-P	3	2.15E-2	2.62E-2
Overall	9	3.05E-3	3.25E-2

Appendix E: Statistical Analyses of Elementals

Table E18. Statistical Summary for the Elemental Constituents – All Results below their MDCs

Elemental Constituent (*wt %*)	N	Smallest Minimum Detectable Concentration (*wt %*)♠		Largest Minimum Detectable Concentration (*wt %*)♠	
		Fixed Decimal Format	Scientific Format	Fixed Decimal Format	Scientific Format
Ag	9	0.00126	1.26E-03	0.00133	1.33E-03
B	9	0.0182	1.82E-02	0.0191	1.91E-02
Mo	9	0.00775	7.75E-03	0.00816	8.16E-03
Sb	9	0.027	2.70E-02	0.0285	2.85E-02
Se	9	0.000514	5.14E-04	0.000541	5.41E-04
U	9	0.205	2.05E-01	0.216	2.16E-01

Table E19. Statistical Summary for the Elemental Constituents – All Results above their MDCs

Elemental Constituent (*wt %*)	N	Mean (*wt %*)	Std Dev (*wt %*)♠	% Std Dev	UCL95 (*wt %*)	Goodness-of-Fit/Confidence Limit Remarks
Al	9	3.1804E-1	2.3004E-1	72.331%	6.9741E-1	SNS-VH; SS-SV; SNS-WS; SNS-OT; Student's t UCL95
As	9	3.7689E-3	3.6760E-4	9.754%	4.3589E-3	SNS-VH; SS-SV; SNS-WS; SNS-OT; Student's t UCL95
Ba	9	6.2778E-3	4.7084E-3	75.001%	1.3509E-2	SNS-VH; SS-SV; SNS-WS; SNS-OT; Student's t UCL95
Cd	9	2.3589E-3	2.5887E-4	10.974%	2.5193E-3	SNS-VH; SNS-SV; SNS-WS; SNS-OT; Student's t UCL95
Co	9	5.8144E-3	1.6836E-3	28.956%	8.6258E-3	SNS-VH; SS-SV; SNS-WS; SNS-OT; Student's t UCL95
Cr	9	2.5756E-2	1.1201E-2	43.489%	3.2698E-2	SNS-VH; SNS-SV; SNS-WS; SNS-OT; Student's t UCL95
Cu	9	3.8367E-2	2.0420E-2	53.223%	7.2080E-2	SNS-VH; SS-SV; SNS-WS; SNS-OT; Student's t UCL95
Fe	9	6.0933E+1	6.3246E-1	1.038%	6.1325E+1	SNS-VH; SNS-SV; SNS-WS; SNS-OT; Student's t UCL95
Hg	9	1.0834E-1	9.1412E-2	84.375%	2.5988E-1	SNS-VH; SS-SV; SNS-WS; SNS-OT; Student's t UCL95
Mn	9	4.0456E-1	1.0988E-1	27.160%	5.8959E-1	SNS-VH; SS-SV; SNS-WS; SNS-OT; Student's t UCL95
Ni	9	2.3889E-2	6.5222E-3	27.302%	2.7932E-2	SNS-VH; SNS-SV; SNS-WS; SNS-OT; Student's t UCL95
Si	9	4.9111E-1	1.7817E-1	36.279%	7.8345E-1	SNS-VH; SS-SV; SNS-WS; SNS-OT; Student's t UCL95
Zn	9	4.3656E-2	2.4772E-2	56.744%	8.4669E-2	SNS-VH; SS-SV; SNS-WS; SNS-OT; Student's t UCL95

SS-VH/SNS-VH: Statistically Significant/Statistically non-significant Levene's test of variance heterogeneity at a Bonferroni $\alpha = 0.05/16 = 0.003125$. There are 13 tests of variance heterogeneity for elementals in Table E19, and 3 tests for variance heterogeneity for elements in Table E20 (16 tests in all).

SS-SV/SNS-SV: Statistically Significant/Statistically non-significant (statistically significant) sampling variance at $\alpha = 0.05$.

SS-WS/SNS-WS: Statistically Significant/Statistically non-significant Wilk-Shapiro test statistic for testing normality at $\alpha = 0.05$.

SS-OT/SNS-OT: Statistically Significant/Statistically non-significant Dixon's outlier test statistic for identifying potential outliers at $\alpha = 0.05$.

Appendix E: Statistical Analyses of Elementals

Table E20. Statistical Summary for the Elemental Constituents – Mixture of Results above and below the MDCs

Elemental Constituent (*wt %*)	N	Mean (wt %)	Std Dev (wt %)	% Std Dev	UCL95 (wt %)	Goodness-of-Fit/Confidence Limit Remarks♥
Na	9 (0, 3, 0)	1.0152E-02	9.9351E-03	97.863%	3.2655E-02	Modified UCL95; Refer to Table E21
Pb	9 (1, 0, 0)	2.2415E-01	3.1446E-01	140.290%	1.0103	Modified UCL95; Refer to Table E21
Sr	9 (0, 3, 0)	9.9000E-03	9.8080E-03	99.071%	3.2664E-02	Modified UCL95; Refer to Table E21

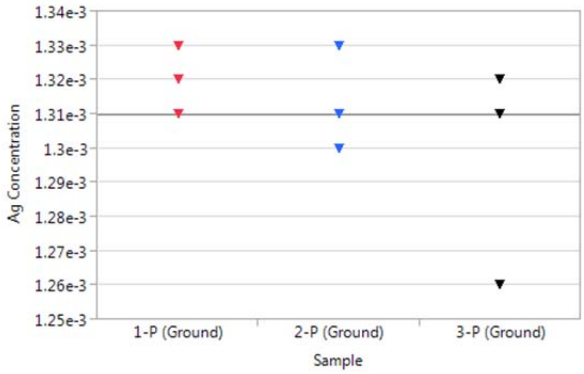
N = Number of analytical results. The number of less-than-MDC results per sample are given in the parentheses (X, Y, Z), where X = the number of less-than-MDC results for Sample 1, Y = the number of less-than MDC results for Sample 2, and Z = the number of less-than-MDC results for Sample 3.

Appendix E: Supporting Tables for Statistical Analyses of Elementals

Table E21. Supporting Results for Elementals

Ag (*wt %*)

All Results (triangles) below MDCs

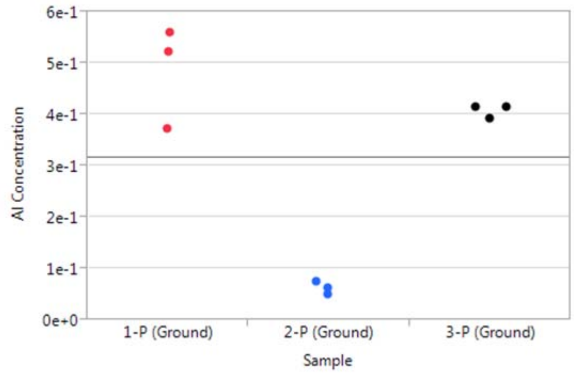


Minimum Detection Concentrations (MDCs)

Sample	N	Minimum MDC	Maximum MDC
1-P	3	1.31E-3	1.33E-3
2-P	3	1.30E-3	1.33E-3
3-P	3	1.26E-3	1.32E-3
Overall	9	1.26E-3	1.33E-3

Al (*wt %*)

All Results (circles) above MDCs



Means and Standard Deviations

Sample	N	Mean	Std Dev	Std Err Mean
1-P	3	4.8500E-1	9.9594E-2	5.7501E-2
2-P	3	6.2133E-2	1.2922E-2	7.4604E-3
3-P	3	4.0700E-1	1.3000E-2	7.5056E-3
Overall	9	3.1804E-1	2.0135E-1	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	1.7171	2	6	0.2572
Brown-Forsythe	1.3930	2	6	0.3185
Levene	8.8390	2	6	0.0163
Bartlett	4.0588	2		0.0173

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	3.0383E-1	1.5192E-1	44.442	0.0003
Error	6	2.0510E-2	3.4183E-3		
C. Total	8	3.2434E-1			

Statistically Significant Sampling Variance.

Diagnostics for Sampling Model Centered Residuals

Shapiro-Wilk	0.929 > 0.829 crit. value->SNS
Dixon High Outlier	Run 1 of Sample 1 -> SNS
Dixon Low Outlier	Run 1 of Sample 2 -> SNS

Variance Components

Component	Variance Comp	Std Dev
Samples	4.9499E-2	2.2248E-1
Measurements	3.4183E-3	5.8466E-2
Total	5.2918E-2	2.3004E-1
Mean Concentration	1.6880E-2	1.2992E-1

Student's t UCL95

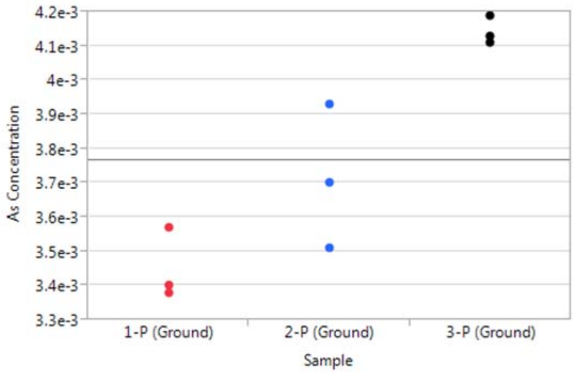
UCL95	6.9741E-1

Appendix E: Supporting Tables for Statistical Analyses of Elementals

Table E21. Supporting Results for Elementals

As (*wt %*)

All Results (circles) above MDCs



Means and Standard Deviations

Sample	N	Mean	Std Dev	Std Err Mean
1-P	3	3.4500E-3	1.0440E-4	6.0277E-5
2-P	3	3.7133E-3	2.1031E-4	1.2143E-4
3-P	3	4.1433E-3	4.1633E-5	2.4037E-5
Overall	9	3.7689E-3	3.2571E-4	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	1.2836	2	6	0.3435
Brown-Forsythe	1.2203	2	6	0.3592
Levene	2.0031	2	6	0.2156
Bartlett	1.7165	2		0.1797

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	7.3496E-7	3.6748E-7	19.3863	0.0024
Error	6	1.1373E-7	1.8956E-8		
C. Total	8	8.4869E-7			

Statistically Significant Sampling Variance.

Diagnostics for Sampling Model Centered Residuals

Shapiro-Wilk	0.929 > 0.829 crit. value->SNS
Dixon High Outlier	Run 1 of Sample 1 -> SNS
Dixon Low Outlier	Run 1 of Sample 2 -> SNS

Variance Components

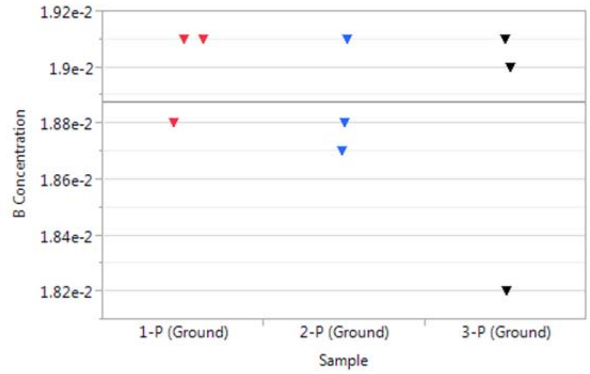
Component	Variance Comp	Std Dev
Samples	1.1617E-7	3.4084E-4
Measurements	1.8956E-8	1.3768E-4
Total	1.3513E-7	3.6760E-4
Mean Concentration	4.0831E-8	2.0207E-4

Student's t UCL95

UCL95	4.3589E-3

B (*wt %*)

All Results (triangles) below MDCs



Minimum Detection Concentrations (MDCs)

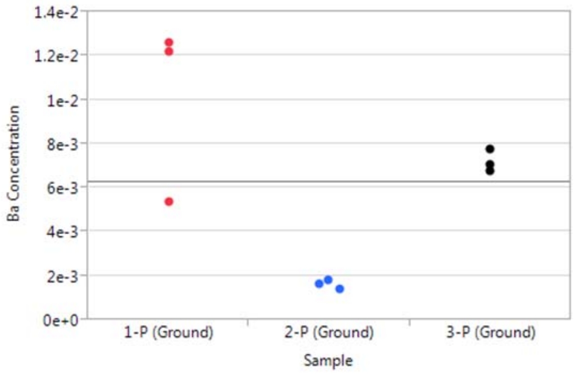
Sample	Number	Minimum MDC	Maximum MDC
1-P	3	1.88E-2	1.91E-2
2-P	3	1.87E-2	1.91E-2
3-P	3	1.82E-2	1.91E-2
Overall	9	1.82E-2	1.91E-2

Appendix E: Supporting Tables for Statistical Analyses of Elementals

Table E21. Supporting Results for Elementals

Ba (*wt %*)

All Results (circles) above MDCs



Means and Standard Deviations

Sample	N	Mean	Std Dev	Std Err Mean
1-P	3	1.0047E-2	4.0810E-3	2.356E-3
2-P	3	1.6100E-3	2.1656E-4	1.250E-4
3-P	3	7.1767E-3	5.0619E-4	2.922E-4
Overall	9	6.2778E-3	4.2473E-3	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	1.7454	2	6	0.2527
Brown-Forsythe	0.9617	2	6	0.4342
Levene	12.8088	2	6	0.0068
Bartlett	5.5678	2		0.0038

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	1.1040E-4	5.5201E-5	9.766	0.0130
Error	6	3.3915E-5	5.6526E-6		
C. Total	8	1.4432E-4			

Statistically Significant Sampling Variance.

Diagnostics for Sampling Model Centered Residuals

Shapiro-Wilk	0.929 > 0.829 crit. value->SNS
Dixon High Outlier	Run 1 of Sample 1 -> SNS
Dixon Low Outlier	Run 1 of Sample 2 -> SNS

Variance Components

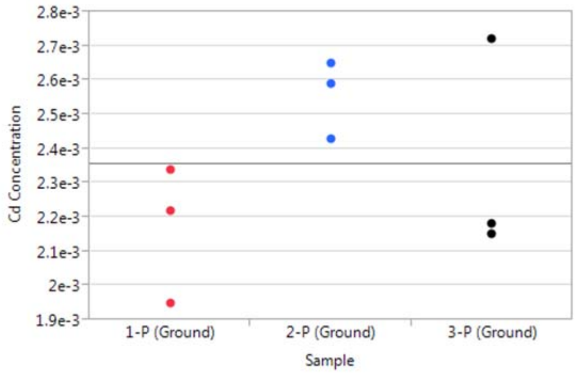
Component	Variance Comp	Std Dev
Samples	1.6516E-5	4.0640E-3
Measurements	5.6526E-6	2.3775E-3
Total	2.2169E-5	4.7084E-3
Mean Concentration	6.1334E-6	2.4766E-3

Student's t UCL95

UCL95	1.3509E-2

Cd (*wt %*)

All Results (circles) above MDCs



Means and Standard Deviations

Sample	N	Mean	Std Dev	Std Err Mean
1-P	3	2.1700E-3	1.9975E-4	1.1533E-4
2-P	3	2.5567E-3	1.1372E-4	6.5659E-5
3-P	3	2.3500E-3	3.2078E-4	1.8520E-4
Overall	9	2.3589E-3	2.5887E-4	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	0.9207	2	6	0.4480
Brown-Forsythe	0.2620	2	6	0.7779
Levene	2.8033	2	6	0.1381
Bartlett	0.7925	2		0.4527

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	2.2462E-7	1.1231E-7	2.164	0.1961
Error	6	3.1147E-7	5.1911E-8		
C. Total	8	5.3609E-7			

Statistically Non-significant Sampling Variance.

Diagnostics for Concentrations

Shapiro-Wilk	0.936 > 0.829 crit. value->SNS
Dixon High Outlier	Run 1 of Sample 2 -> SNS
Dixon Low Outlier	Run 1 of Sample 3 -> SNS

Normal Statistics

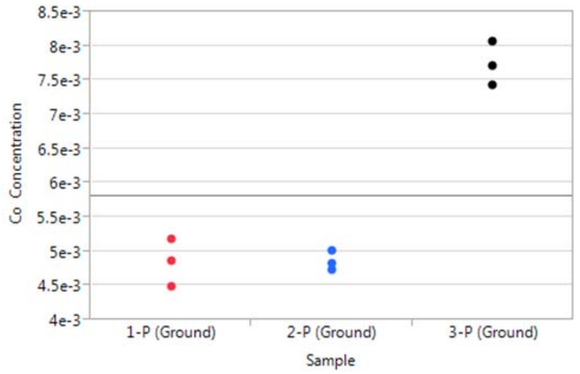
Component	Estimate
Mean	2.3589E-3
Std.Dev.	2.5887E-4
Student's t UCL95	2.5193E-3

Appendix E: Supporting Tables for Statistical Analyses of Elementals

Table E21. Supporting Results for Elementals

Co (*wt %*)

All Results (circles) above MDCs



Means and Standard Deviations

Sample	N	Mean	Std Dev	Std Err Mean
1-P	3	4.8467E-3	3.4559E-4	1.9953E-4
2-P	3	4.8567E-3	1.3868E-4	8.0069E-5
3-P	3	7.7400E-3	3.2047E-4	1.8502E-4
Overall	9	5.8144E-3	1.4649E-3	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	0.6102	2	6	0.5738
Brown-Forsythe	0.6190	2	6	0.5696
Levene	0.7153	2	6	0.5265
Bartlett	0.6479	2		0.5231

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	1.6685E-5	8.3425E-6	103.691	<.0001
Error	6	4.8273E-7	8.0456E-8		
C. Total	8	1.7168E-5			

Statistically Significant Sampling Variance.

Diagnostics for Sampling Model Centered Residuals

Shapiro-Wilk	0.929 > 0.829 crit. value->SNS
Dixon High Outlier	Run 1 of Sample 1 -> SNS
Dixon Low Outlier	Run 1 of Sample 2 -> SNS

Variance Components

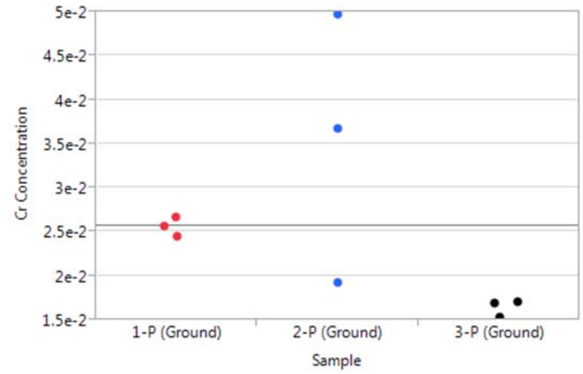
Component	Variance Comp	Std Dev
Samples	2.7540E-6	1.6595E-3
Measurements	8.0456E-8	2.8365E-4
Total	2.8345E-6	1.6836E-3
Mean Concentration	9.2695E-7	9.6278E-4

Student's t UCL95

UCL95	8.6258E-3

Cr (*wt %*)

All Results (circles) above MDCs



Means and Standard Deviations

Sample	N	Mean	Std Dev	Std Err Mean
1-P	3	2.5600E-2	1.0536E-3	6.0828E-4
2-P	3	3.5267E-2	1.5258E-2	8.8091E-3
3-P	3	1.6400E-2	9.5394E-4	5.5076E-4
Overall	9	2.5756E-2	1.1201E-2	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	1.7623	2	6	0.2500
Brown-Forsythe	3.2431	2	6	0.1110
Levene	4.6589	2	6	0.0601
Bartlett	6.2349	2		0.0020

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	5.3404E-4	2.6702E-4	3.411	0.1025
Error	6	4.6965E-4	7.8274E-5		
C. Total	8	1.0037E-3			

Statistically Non-significant Sampling Variance.

Diagnostics for Concentrations

Shapiro-Wilk	0.936 > 0.829 crit. value->SNS
Dixon High Outlier	Run 1 of Sample 2 -> SNS
Dixon Low Outlier	Run 1 of Sample 3 -> SNS

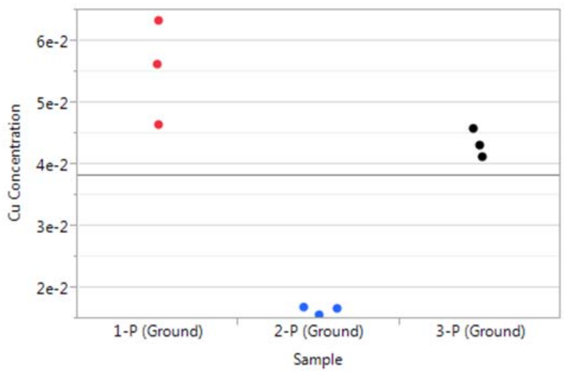
Normal Statistics

Component	Estimate
Mean	2.5756E-2
Std.Dev.	1.1201E-2
Student's t UCL95	3.2698E-2

Appendix E: Supporting Tables for Statistical Analyses of Elementals

Table E21. Supporting Results for Elementals

Cu (*wt %*)
All Results (circles) above MDCs



Means and Standard Deviations

Sample	N	Mean	Std Dev	Std Err Mean
1-P	3	5.5367E-2	8.4388E-3	4.8721E-3
2-P	3	1.6333E-2	6.4291E-4	3.7118E-4
3-P	3	4.3400E-2	2.2650E-3	1.3077E-3
Overall	9	3.8367E-2	1.7864E-2	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	1.6394	2	6	0.2704
Brown-Forsythe	2.4445	2	6	0.1673
Levene	3.6647	2	6	0.0912
Bartlett	3.8527	2		0.0212

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	2.3994E-3	1.1997E-3	46.890	0.0002
Error	6	1.5351E-4	2.5586E-5		
C. Total	8	2.5530E-3			

Statistically significant sampling variance.

Diagnostics for Sampling Model Centered Residuals

Shapiro-Wilk	0.929 > 0.829 crit. value->SNS
Dixon High Outlier	Run 1 of Sample 1 -> SNS
Dixon Low Outlier	Run 1 of Sample 2 -> SNS

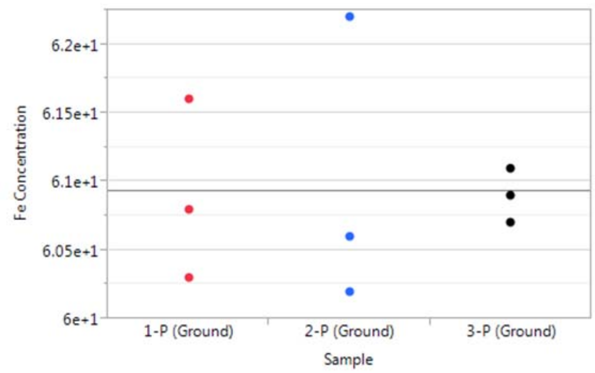
Variance Components

Component	Variance Comp	Std Dev
Samples	3.9137E-4	1.9783E-2
Measurements	2.5586E-5	5.0582E-3
Total	4.1696E-4	2.0420E-2
Mean Concentration	1.3330E-4	1.1546E-2

Student's t UCL95

UCL95	7.2080E-2

Fe (*wt %*)
All Results (circles) above MDCs



Means and Standard Deviations

Sample	N	Mean	Std Dev	Std Err Mean
1-P	3	6.0900E+1	6.5574E-1	3.7859E-1
2-P	3	6.1000E+1	1.0583E+0	6.1101E-1
3-P	3	6.0900E+1	2.0000E-1	1.1547E-1
Overall	9	6.0933E+1	6.325E-1	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	1.1071	2	6	0.3897
Brown-Forsythe	0.7395	2	6	0.5163
Levene	3.6145	2	6	0.0933
Bartlett	1.6731	2		0.1877

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	2.0000E-2	1.0000E-2	0.019	0.9814
Error	6	3.1800E+0	5.3000E-1		
C. Total	8	3.2000E+0			

Statistically non-significant sampling variance.

Diagnostics for Concentrations

Shapiro-Wilk	0.936 > 0.829 crit. value->SNS
Dixon High Outlier	Run 1 of Sample 2 -> SNS
Dixon Low Outlier	Run 1 of Sample 3 -> SNS

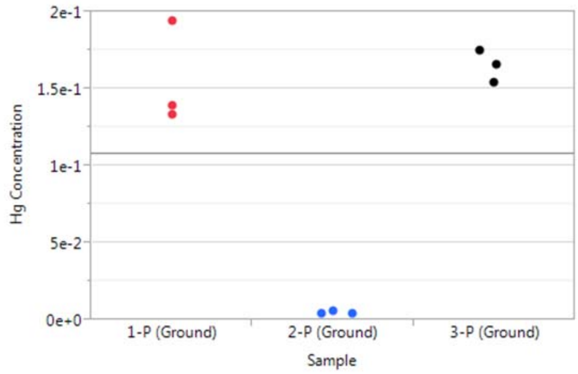
Normal Statistics

Component	Estimate
Mean	6.0933E+1
Std.Dev.	6.3246E-1
Student's t UCL95	6.1325E+1

Appendix E: Supporting Tables for Statistical Analyses of Elementals

Table E21. Supporting Results for Elementals

Hg (*wt %*)
All Results (circles) above MDCs



Means and Standard Deviations

Sample	N	Mean	Std Dev	Std Err Mean
1-P	3	1.5533E-1	3.3620E-2	1.9411E-2
2-P	3	4.7000E-3	1.3119E-3	7.5743E-4
3-P	3	1.6500E-1	1.0536E-2	6.0828E-3
Overall	9	1.0834E-1	7.9817E-2	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	1.6019	2	6	0.2770
Brown-Forsythe	0.9439	2	6	0.4401
Levene	9.0829	2	6	0.0153
Bartlett	4.7433	2		0.0087

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	4.8480E-2	2.4240E-2	58.501	0.0001
Error	6	2.4861E-3	4.1435E-4		
C. Total	8	5.0966E-2			

Statistically significant sampling variance.

Diagnostics for Sampling Model Centered Residuals

Shapiro-Wilk	0.929 > 0.829 crit. value->SNS
Dixon High Outlier	Run 1 of Sample 1 -> SNS
Dixon Low Outlier	Run 1 of Sample 2 -> SNS

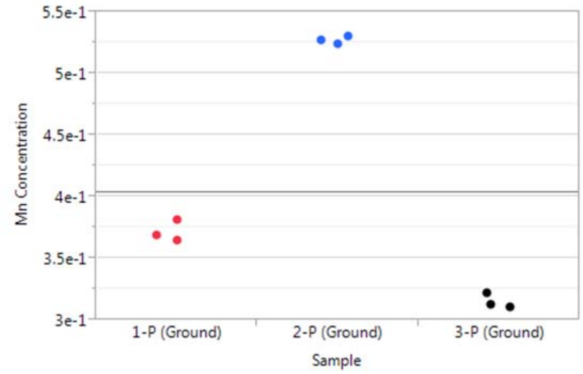
Variance Components

Component	Variance Comp	Std Dev
Samples	7.9419E-3	8.9117E-2
Measurements	4.1435E-4	2.0356E-2
Total	8.3562E-3	9.1412E-2
Mean Concentration	2.6933E-3	5.1897E-2

Student's t UCL95

UCL95	2.5988E-1

Mn (*wt %*)
All Results (circles) above MDCs



Means and Standard Deviations

Sample	N	Mean	Std Dev	Std Err Mean
1-P	3	3.7167E-1	8.3267E-3	4.8074E-3
2-P	3	5.2700E-1	3.0000E-3	1.7321E-3
3-P	3	3.1500E-1	6.2450E-3	3.6056E-3
Overall	9	4.0456E-1	9.5209E-2	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	0.7573	2	6	0.5090
Brown-Forsythe	0.4130	2	6	0.6791
Levene	1.9928	2	6	0.2169
Bartlett	0.7360	2		0.4790

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	7.2284E-2	3.6142E-2	924.080	<.0001
Error	6	2.3467E-4	3.9111E-5		
C. Total	8	7.2518E-2			

Statistically significant sampling variance.

Diagnostics for Sampling Model Centered Residuals

Shapiro-Wilk	0.929 > 0.829 crit. value->SNS
Dixon High Outlier	Run 1 of Sample 1 -> SNS
Dixon Low Outlier	Run 1 of Sample 2 -> SNS

Variance Components

Component	Variance Comp	Std Dev
Samples	1.2034E-2	1.0970E-1
Measurements	3.9111E-5	6.2539E-3
Total	1.2073E-2	1.0988E-1
Mean Concentration	4.0158E-3	6.3370E-2

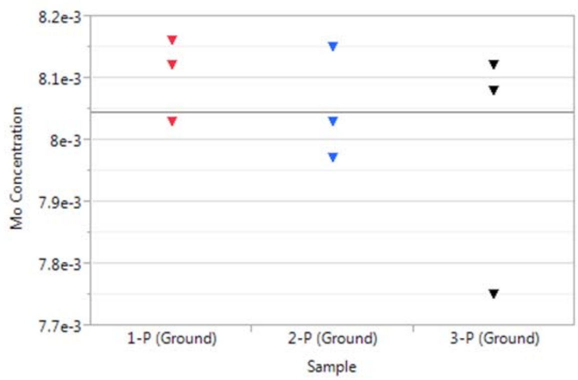
Student's t UCL95

UCL95	5.8959E-1

Appendix E: Supporting Tables for Statistical Analyses of Elementals

Table E21. Supporting Results for Elementals

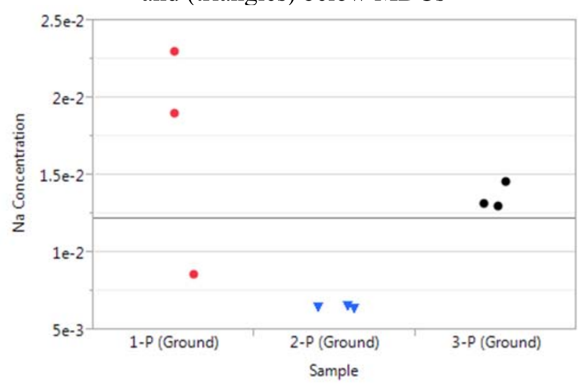
Mo (*wt %*)
All Results (triangles) below MDCs



Minimum Detection Concentrations (MDCs)

Sample	Number	Minimum MDC	Maximum MDC
1-P	3	8.03E-3	8.16E-3
2-P	3	7.97E-3	8.15E-3
3-P	3	7.75E-3	8.12E-3
Overall	9	7.75E-3	8.16E-3

Na (*wt %*)
Mixture of Results (circles) above MDCs
and (triangles) below MDCs



Means and Standard Deviations

Sample	N	Mean	Std Dev	Std Err Mean
1-P	3	1.6857E-02	7.4499E-03	4.3012E-03
3-P	3	1.3600E-02	8.7178E-04	5.0332E-04

Sample	Run 1 MDC	Run 2 MDC	Run 3 MDC
2-P	<6.49E-03	<6.40E-03	<6.35E-03

The mean concentration for Sample 2-P is assumed to be between 0 and 6.41E-3 *wt %*, the mean of the MDC's for the 3 runs for Sample 2-P. The data set of sample mean concentrations is {1.6857E-2, X, 1.3600E-2}, where X is the mean for Sample 2-P. The non-parametric Chebyshev (Minimum Variance Unbiased Estimator or MVUE) UCL95 is maximized when the mean concentration for Sample 2-P is set to X = 0. The Maximum Chebyshev UCL95 is 3.2655E-2 *wt %*.

The associated mean and standard deviation of the data set of means {1.6857E-2, 0, 1.3600E-2} are 1.0152E-2 *wt %* and 8.9416E-03 *wt %*, respectively.

$$S_{Total} = \sqrt{S^2_{3\,Sample\,Means} - S^2_{MeasErr(Mean)} + S^2_{MeasErr(One\,Result)}}$$

$$= \sqrt{(8.9416E\text{-}3)^2 - (3.0622E\text{-}3)^2 + 3\cdot(3.0622E\text{-}3)^2} = 9.9351E\text{-}3,$$

where the pooled standard error of the mean, $S_{MeasErr(Mean)} = 3.0622E\text{-}3$, is based on Samples 1-P and 3-P only.

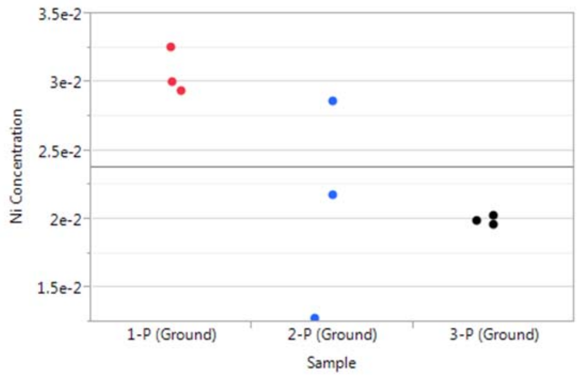
Nonparametric Statistics

Component	Estimate
Mean	1.0152E-2
Std.Dev.	9.9351E-3
(Nonparametric) Chebyshev UCL95	3.2655E-2

Appendix E: Supporting Tables for Statistical Analyses of Elementals

Table E21. Supporting Results for Elementals

Ni (*wt %*)
All Results (circles) above MDCs



Means and Standard Deviations

Sample	N	Mean	Std Dev	Std Err Mean
1-P	3	3.0667E-2	1.7010E-3	9.8206E-4
2-P	3	2.1067E-2	7.9255E-3	4.5758E-3
3-P	3	1.9933E-2	3.5119E-4	2.0276E-4
Overall	9	2.3889E-2	6.5222E-3	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	1.6924	2	6	0.2613
Brown-Forsythe	2.7396	2	6	0.1428
Levene	3.9561	2	6	0.0802
Bartlett	5.0365	2		0.0065

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	2.0865E-4	1.0432E-4	4.754	0.0579
Error	6	1.3166E-4	2.1943E-5		
C. Total	8	3.4031E-4			

Statistically Significant Sampling Variance.

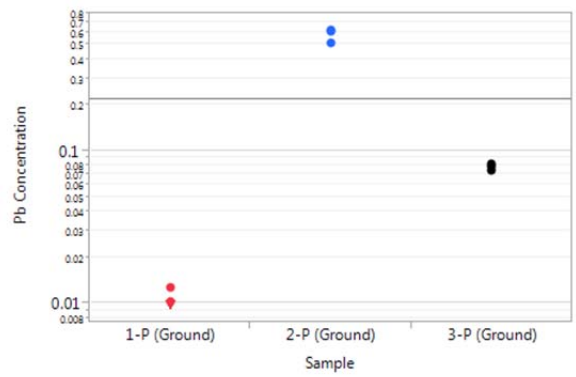
Diagnostics for Concentrations

Shapiro-Wilk	0.936 > 0.829 crit. value->SNS
Dixon High Outlier	Run 1 of Sample 2 -> SNS
Dixon Low Outlier	Run 1 of Sample 3 -> SNS

Normal Statistics

Component	Estimate
Mean	2.3889E-2
Std.Dev.	6.5222E-3
Student's t UCL95	2.7932E-2

Pb (*wt %*)
Mixture of Results (circles) above MDCs
and (triangles) below MDCs



Means and Standard Deviations (Response: Log Conc)

The following is the output from a SAS Proc Reliability fit of a lognormal distribution to a fixed one-way ANOVA, where the factor is Sample and the response is the log of the Pb concentration data which includes a MDC for Sample 1-P, Run 2.

Sample	N	$Mean_{Log(Conc)}$	Sample Mean in Original Units
1-P	3	-4.5509	0.010607
2-P	3	0.5447	0.582700
3-P	3	-2.5411	0.079144
Pooled $SD_{Log(Conc)}$: 0.183620			
Overall	9	-3.7415	

$$Sample\ Mean_{\substack{Original \\ Units}} = e^{Sample\ Mean_{Log(Conc)} + \frac{(PooledSD_{Log(Conc)})}{2}}$$

Note that the 3 Sample Means in the Original Units (displayed in red above) were input to ProUCL and evaluated with the lognormal distribution including the goodness-of-fit tests below.

Tests that the Measurement Variances are Equal Log Concentrations using Samples 2-P & 3-P only.

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	1.1923	1	4	0.3362
Brown-Forsythe	0.4025	1	4	0.5603
Levene	4.1425	1	4	0.1115
Bartlett	1.1235	1		0.2892

Statistically non-significant measurement variance heterogeneity.

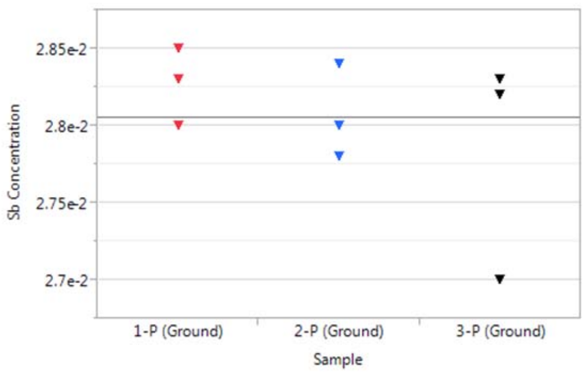
Nonparametric Statistics (UCL from ProUCL)

Component	Estimate
Mean	2.2415E-01
Std.Dev.	3.1446E-01
(Nonparametric) Chebyshev UCL95	1.0103

Appendix E: Supporting Tables for Statistical Analyses of Elementals

Table E21. Supporting Results for Elementals

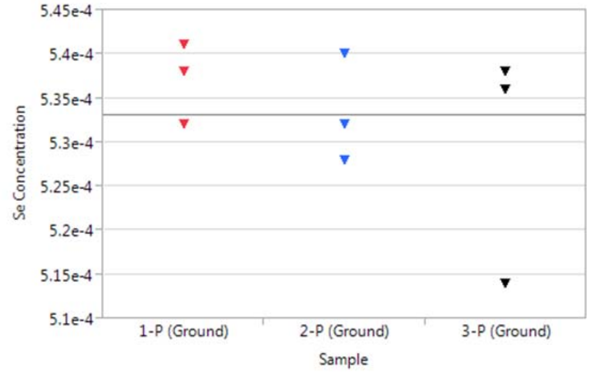
Sb (*wt %*)
All Results (triangles) below MDCs



Minimum Detection Concentrations (MDCs)

Sample	Number	Minimum MDC	Maximum MDC
1-P	3	2.80E-2	2.85E-2
2-P	3	2.78E-2	2.84E-2
3-P	3	2.70E-2	2.83E-2
Overall	9	2.70E-2	2.85E-2

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



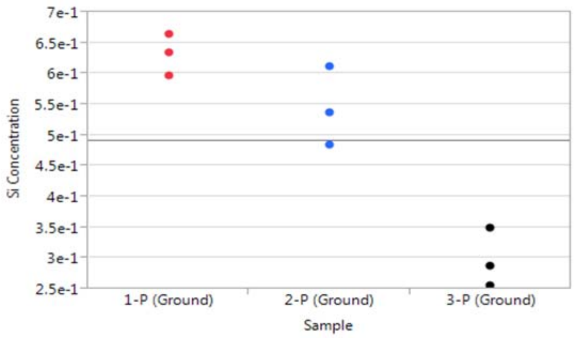
Minimum Detection Concentrations (MDCs)

Sample	Number	Minimum MDC	Maximum MDC
1-P	3	5.32E-4	5.41E-4
2-P	3	5.28E-4	5.40E-4
3-P	3	5.14E-4	5.38E-4
Overall	9	5.14E-4	5.41E-4

Appendix E: Supporting Tables for Statistical Analyses of Elementals

Table E21. Supporting Results for Elementals

Si (*wt %*)
All Results (circles) above MDCs



Means and Standard Deviations

Sample	N	Mean	Std Dev	Std Err Mean
1-P	3	6.3167E-1	3.3561E-2	1.9376E-2
2-P	3	5.4433E-1	6.3909E-2	3.6898E-2
3-P	3	2.9733E-1	4.8336E-2	2.7907E-2
Overall	9	4.9111E-1	1.5633E-1	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	0.5039	2	6	0.6277
Brown-Forsythe	0.3131	2	6	0.7425
Levene	0.5922	2	6	0.5825
Bartlett	0.3215	2		0.7251

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	1.8041E-1	9.0207E-2	35.858	0.0005
Error	6	1.5094E-2	2.5157E-3		
C. Total	8	1.9551E-1			

Statistically significant sampling variance.

Diagnostics for Sampling Model Centered Residuals

Shapiro-Wilk	0.929 > 0.829 crit. value->SNS
Dixon High Outlier	Run 1 of Sample 1 -> SNS
Dixon Low Outlier	Run 1 of Sample 2 -> SNS

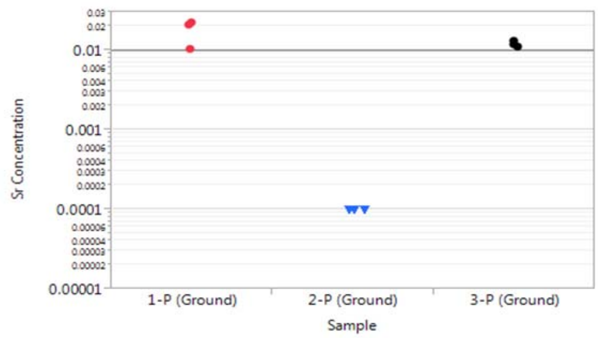
Variance Components

Component	Variance Comp	Std Dev
Samples	2.9231E-2	1.7097E-1
Measurements	2.5157E-3	5.0156E-2
Total	3.1746E-2	1.7817E-1
Mean Concentration	1.0023E-2	1.0012E-1

Student's t UCL95

UCL95	7.8345E-1

Sr (*wt %*)
Mixture of Results (circles) above MDCs
and (triangles) below MDCs



Means and Standard Deviations

Sample	N	Mean	Std Dev	Std Err Mean
1-P	3	1.7733E-02	6.4810E-03	3.7418E-03
3-P	3	1.1967E-02	1.0599E-03	6.1192E-04

Sample	Run 1 MDC	Run 2 MDC	Run 3 MDC
2-P	<9.82E-05	<9.68E-05	<9.61E-05

The mean concentration for Sample 2-P is assumed to be between 0 and 9.70E-05 *wt %*, the mean of the MDC's for the 3 runs for Sample 2-P. The data set of sample mean concentrations is {1.7733E-02, X, 1.1967E-02}, where X is the mean for Sample 2-P. The non-parametric Chebyshev (Minimum Variance Unbiased Estimator or MVUE) UCL95 is maximized when the mean concentration for Sample 2-P is set to X = 0. The Maximum Chebyshev UCL95 is 3.2664E-02 *wt %*.

The associated mean and standard deviation of the data set of means {1.7733E-02, 0, 1.1967E-02} are 9.9000E-03 *wt %* and 9.0455E-03 *wt %*, respectively.

$$S_{Total} = \sqrt{S^2_{3\,Sample\,Means} - S^2_{MeasErr(Mean)} + S^2_{MeasErr(One\,Result)}}$$

$$= \sqrt{(9.0455E\text{-}3)^2 - (2.6810E\text{-}3)^2 + 3\cdot(2.6810E\text{-}3)^2} = 9.8080E\text{-}3,$$

where the pooled standard error of the mean, $S_{MeasErr(Mean)} = 2.6810E\text{-}3$, is based on Samples 1-P and 3-P only.

Nonparametric Statistics

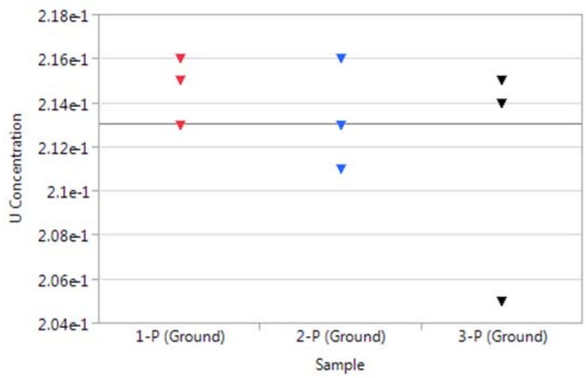
Component	Estimate
Mean	9.9000E-3
Std.Dev.	9.8080E-3
(Nonparametric) Chebyshev UCL95	3.2664E-2

Appendix E: Supporting Tables for Statistical Analyses of Elementals

Table E21. Supporting Results for Elementals

U (*wt %*)

All Results (triangles) below MDCs

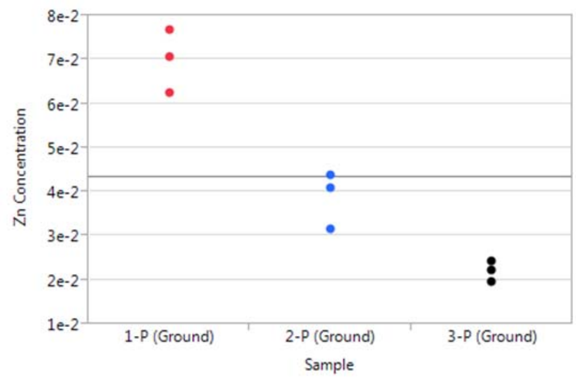


Minimum Detection Concentrations (MDCs)

Sample	Number	Minimum MDC	Maximum MDC
1-P	3	2.13E-1	2.16E-1
2-P	3	2.11E-1	2.16E-1
3-P	3	2.05E-1	2.15E-1
Overall	9	2.05E-1	2.16E-1

Zn (*wt %*)

All Results (circles) above MDCs



Means and Standard Deviations

Sample	N	Mean	Std Dev	Std Err Mean
1-P	3	7.0033E-2	7.2231E-3	4.1703E-3
2-P	3	3.8833E-2	6.3571E-3	3.6703E-3
3-P	3	2.2100E-2	2.3516E-3	1.3577E-3
Overall	9	4.3656E-2	2.1643E-2	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	0.7156	2	6	0.5264
Brown-Forsythe	0.6135	2	6	0.5722
Levene	1.4481	2	6	0.3068
Bartlett	0.8989	2		0.4070

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	3.5510E-3	1.7755E-3	54.288	0.0001
Error	6	1.9623E-4	3.2706E-5		
C. Total	8	3.7473E-3			

Statistically Significant Sampling Variance.

Diagnostics for Sampling Model Centered Residuals

Shapiro-Wilk	0.929 > 0.829 crit. value->SNS
Dixon High Outlier	Run 1 of Sample 1 -> SNS
Dixon Low Outlier	Run 1 of Sample 2 -> SNS

Variance Components

Component	Variance Comp	Std Dev
Samples	5.8094E-4	2.4103E-2
Measurements	3.2706E-5	5.7189E-3
Total	6.1365E-4	2.4772E-2
Mean Concentration	1.9728E-4	1.4046E-2

Student's t UCL95

UCL95	8.4669E-2

Appendix E: Statistical Analyses of Anions

Table E22. Statistical Summary for the Anions – All Results below their MDCs

Anion Constituent (*wt %*)	N	Smallest Minimum Detectable Concentration (*wt %*)		Largest Minimum Detectable Concentration (*wt %*)	
		Fixed Decimal Format	Scientific Format	Fixed Decimal Format	Scientific Format
Bromide, Br^{-1}	9	0.00466	4.66E-03	0.00491	4.91E-03
Chloride , Cl^{-1}	9	0.00466	4.66E-03	0.00491	4.91E-03
Fluoride, F^{-1}	9	0.00466	4.66E-03	0.00491	4.91E-03
Formate, CHO_2^{-1}	9	0.00466	4.66E-03	0.00491	4.91E-03
Iodine, I-127	9	0.000000611	6.11E-07	0.000000643	6.43E-07
Nitrite , NO_2^{-1}	9	0.00466	4.66E-03	0.00491	4.91E-03
Oxalate, $C_2O_4^{-2}$	9	0.00466	4.66E-03	0.00491	4.91E-03
Phosphate, PO_4^{-3}	9	0.00466	4.66E-03	0.00491	4.91E-03
Sulfate, SO_4^{-2}	9	0.00466	4.66E-03	0.00491	4.91E-03

Table E23. Statistical Summary for the Anions – All Results above their MDCs

Anion Constituent (*wt %*)	N	Mean (mg/g)	Std Dev (mg/g)	% Std Dev	UCL95 (mg/g)	Goodness-of-Fit/Confidence Limit Remarks♠
Nitrate, NO_3^{-1}	9	2.2417E-2	1.9505E-02	87.010%	5.5205E-2	Refer to the entry for Nitrate in Table E25.

LTS: Levene's test for heterogeneity of measurement/sample preparation variances were statistical significant at $\alpha = 0.05$.
* UCL is based on a model with a sampling variance and a measurement/sample preparation variance.
** The measurement/sample preparation variances were as follows: Sample 1-P SD = 0.0026848 wt % or 20.96% of the Sample 1-P mean; Sample 2-P SD = 0.0002250 wt % or 2.33% of the Sample 2-P mean; and Sample 3-P SD = 0.0015875 wt % or 3.54% of the Sample 3-P mean.
The test for a sampling variance using weights was signifcant for traditional variance components, but not with REML. The sampling variance component was estimated to be over 99% of the total variation by the traditional variance componet and REML methods. Therefore, a decision is made to use the model with the sampling variance. Also, the UCL95 with the sampling variance model was higher than the model without the sampling variance.

Appendix E: Statistical Analyses of Anions

Table E24. Statistical Summary for the Anions – Mixture of Results above and below their MDCs

Anion Constituent (*wt %*)	N	Mean (*wt %*)	Std Dev (*wt %*)	% Std Dev	UCL95 (*wt %*)	Goodness-of-Fit Remarks
Iodine, I-129	9 (0, 2, 0)	1.0054E-4	8.6893E-5	86.426%	3.1922E-4	Nonparametric Chebyshev UCL95
Total Iodine	9 (0, 3, 0)	9.8667E-5	9.1355E-5	92.589%	3.1952E-4	Maximum Nonparametric Chebyshev UC See entry in Table E25 for details.

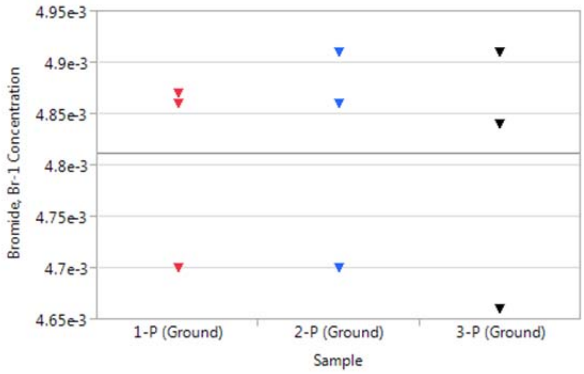
N is the overall sample size (number of analytical results), and the parentheses contain the number of less-than-MDC results for samples 1-P, 2-P, and 3-P.

Appendix E: Supporting Tables for Statistical Analyses of Anions

Table E25. Supporting Results for Anions

Bromide, Br^{-1} (wt %)

All Results (triangles) below MDCs

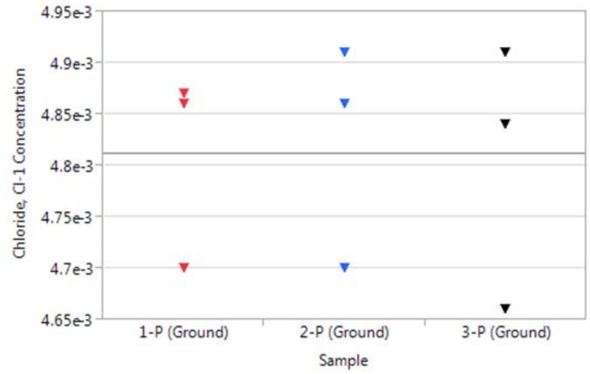


Minimum Detection Concentrations (MDCs)

Sample	N	Minimum MDC	Maximum MDC
1-P	3	4.70E-3	4.87E-3
2-P	3	4.70E-3	4.91E-3
3-P	3	4.66E-3	4.91E-3
Overall	9	4.66E-3	4.91E-3

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

All Results (triangles) below MDCs



Minimum Detection Concentrations (MDCs)

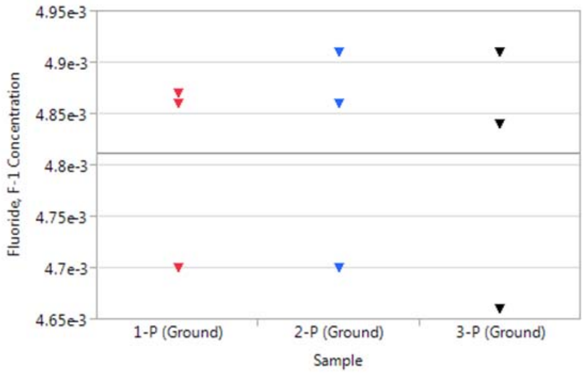
Sample	N	Minimum MDC	Maximum MDC
1-P	3	4.70E-3	4.87E-3
2-P	3	4.70E-3	4.91E-3
3-P	3	4.66E-3	4.91E-3
Overall	9	4.66E-3	4.91E-3

Appendix E: Supporting Tables for Statistical Analyses of Anions

Table E25. Supporting Results for Anions

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

All Results (triangles) below MDCs

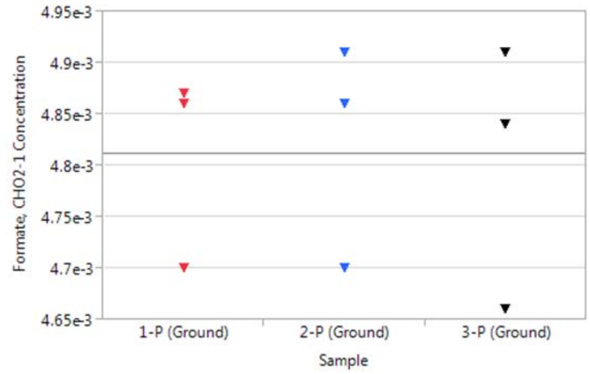


Minimum Detection Concentrations (MDCs)

Sample	Number	Minimum MDC	Maximum MDC
1-P	3	4.70E-3	4.87E-3
2-P	3	4.70E-3	4.91E-3
3-P	3	4.66E-3	4.91E-3
Overall	9	4.66E-3	4.91E-3

Formate, CHO_2^{-1} (*wt %*)

All Results (triangles) below MDCs



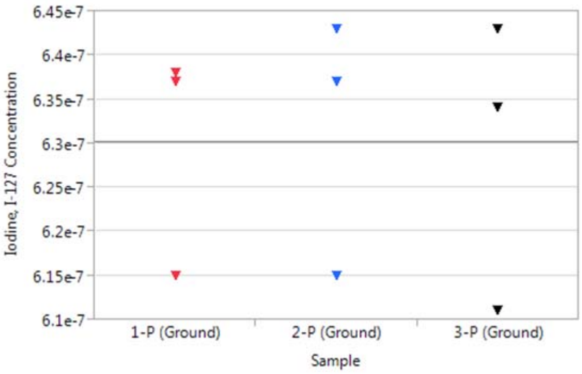
Minimum Detection Concentrations (MDCs)

Sample	Number	Minimum MDC	Maximum MDC
1-P	3	4.70E-3	4.87E-3
2-P	3	4.70E-3	4.91E-3
3-P	3	4.66E-3	4.91E-3
Overall	9	4.66E-3	4.91E-3

Appendix E: Supporting Tables for Statistical Analyses of Anions

Table E25. Supporting Results for Anions

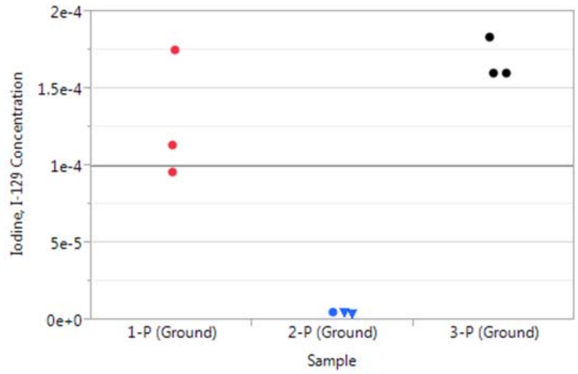
Iodine, I-127 (*wt %*)
All Results (triangles) below MDCs



Minimum Detection Concentrations (MDCs)

Sample	Number	Minimum MDC	Maximum MDC
1-P	3	6.15E-7	6.38E-7
2-P	3	6.15E-7	6.43E-7
3-P	3	6.11E-7	6.43E-7
Overall	9	6.11E-7	6.43E-7

Iodine, I-129 (*wt %*)
Mixture of Results (circles) above MDCs
and (triangles) below MDCs



The original concentrations were fit to a normal distribution and separately to a lognormal distribution in SAS Proc Reliability using a fixed effect sampling variance model. The output from that program included the 3 means of concentrations and 3 means of logged concentrations as well as the pooled standard deviation of the concentrations and the logged concentrations. The formula below was used to convert the logged estimates to 3 Sample means on the original concentration scale. These were input into ProUCL 5.0 to determine statistics. The normal fit was rejected because one of the samples calculated means was negative.

$$Sample\,Mean_{\substack{Original\\Units}} = e^{Sample\,Mean_{Log(Conc)} + \frac{(PooledSD_{Log(Conc)})^2}{2}}$$

Fixed Effect Model: Normal

Parameter	Estimate Effects (Std Error)	Estimate Sample Mean
Intercept	0.0001677 (0.0000135)	
Sample 1-P	-0.0000396 (0.0000191)	0.0001281
Sample 2-P	-0.0001813 (0.0000222)	-0.0000136
Sample 3-P	0.0000000 (0.0000000)	0.0001677
Scale	SD=0.0000234 (0.0000063)	

Fixed Effect Model: Lognormal

Parameter	Estimate Effects (Std Error) on Log Scale	Estimate Sample Means on Original Scale
Intercept	-8.6956 (0.1245)	
Sample 1-P	-0.3004 (0.1761)	0.00012682
Sample 2-P	-3.8786 (0.1967)	0.0000035415
Sample 3-P	0.0000 (0.0000)	0.00017126
Overall Mean		0.000100538
Scale	SD=0.2156 (0.0594)	CV=21.8%

Use estimates of sample means (red) from lognormal fit in ProUCL to obtain an estimate of UCL95. The Scale SD (CV) pertain to just the measurement error, where SD = standard deviation and CV = coefficient of variation (the percent standard deviation).

Nonparametric Statistics (UCL from ProUCL)

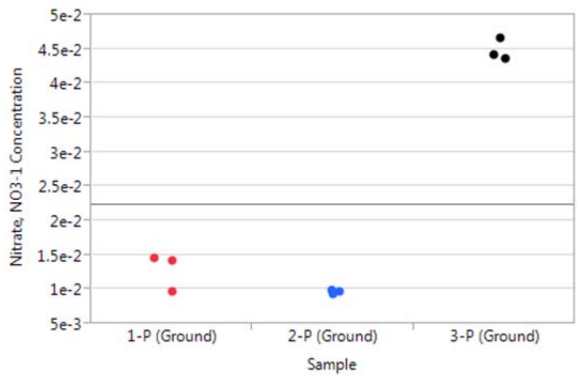
Component	Estimate
Mean	1.0054E-4
Std.Dev.	8.6893E-5
(Nonparametric) Chebyshev UCL95	3.1922E-4

Appendix E: Supporting Tables for Statistical Analyses of Anions

Table E25. Supporting Results for Anions

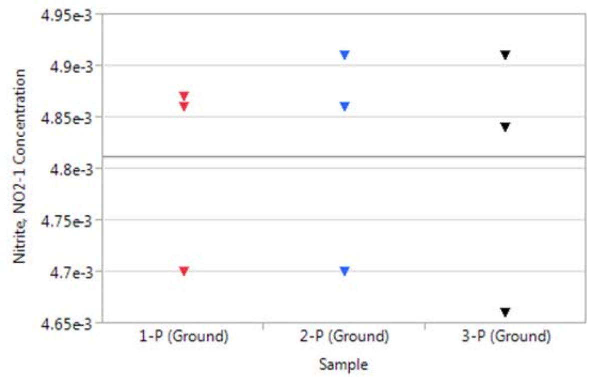
Nitrate, NO_3^{-1} (*wt %*)

All Results (circles) above MDCs



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

All Results (triangles) below MDCs



Means and Standard Deviations on Original Scale

Sample	N	Mean	Std Dev	Std Err Mean
1-P	3	1.2807E-2	2.6848E-3	1.5501E-3
2-P	3	9.6433E-3	2.2502E-4	1.2991E-4
3-P	3	4.4800E-2	1.5875E-3	9.1652E-4
Overall	9	2.2417E-2	1.6916E-2	

Minimum Detection Concentrations (MDCs)

Sample	Number	Minimum MDC	Maximum MDC
1-P	3	4.70E-3	4.87E-3
2-P	3	4.70E-3	4.91E-3
3-P	3	4.66E-3	4.91E-3
Overall	9	4.66E-3	4.91E-3

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	1.2090	2	6	0.3621
Brown-Forsythe	0.6802	2	6	0.5417
Levene	6.5754	2	6	0.0308
Bartlett	2.9688	2		0.0514
Statistically Non-significant Measurement Error heterogeneity.				

Analysis of Variance

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	2.2696E-3	1.1348E-3	348.138	<.0001
Error	6	1.9558E-5	3.2596E-6		
C. Total	8	2.2891E-3			
Statistically Significant Sampling Variance.					

Diagnostics for Sampling Model Centered Residuals

Shapiro-Wilk	0.929 > 0.829 crit. value->SNS
Dixon High Outlier	Run 1 of Sample 1 -> SNS
Dixon Low Outlier	Run 1 of Sample 2 -> SNS

Variance Components

Component	Variance Comp	Std Dev
Samples	3.7718E-04	1.9421E-02
Measurements	3.2596E-06	1.8054E-03
Total	3.8043E-04	1.9505E-02
Mean Concentration	1.2609E-04	1.1229E-02

Student's t UCL95

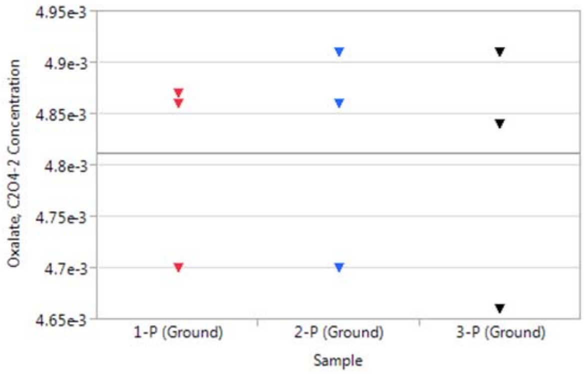
UCL95	5.5205E-2

Appendix E: Supporting Tables for Statistical Analyses of Anions

Table E25. Supporting Results for Anions

Oxalate, $C_2O_4^{2-}$ (*wt %*)

All Results (triangles) below MDCs

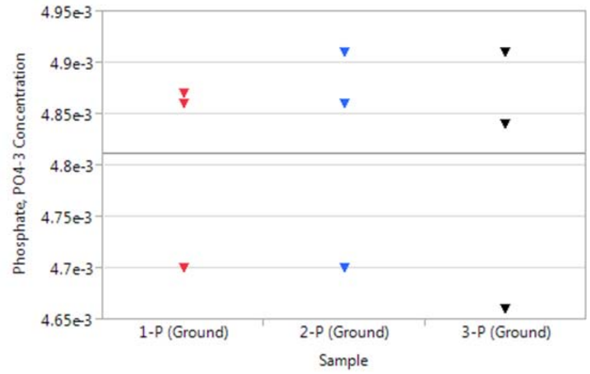


Minimum Detection Concentrations (MDCs)

Sample	Number	Minimum MDC	Maximum MDC
1-P	3	4.70E-3	4.87E-3
2-P	3	4.70E-3	4.91E-3
3-P	3	4.66E-3	4.91E-3
Overall	9	4.66E-3	4.91E-3

Phosphate, PO_4^{-3} (*wt %*)

All Results (triangles) below MDCs



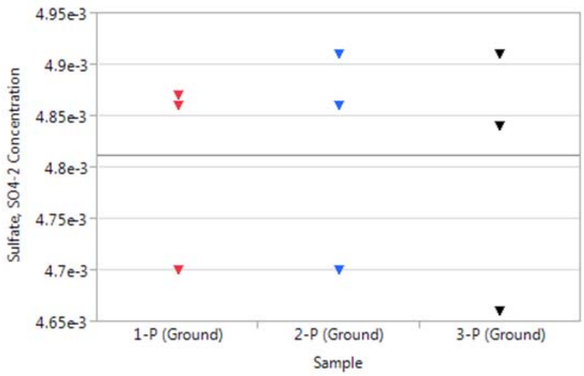
Minimum Detection Concentrations (MDCs)

Sample	Number	Minimum MDC	Maximum MDC
1-P	3	4.70E-3	4.87E-3
2-P	3	4.70E-3	4.91E-3
3-P	3	4.66E-3	4.91E-3
Overall	9	4.66E-3	4.91E-3

Appendix E: Supporting Tables for Statistical Analyses of Anions

Table E25. Supporting Results for Anions

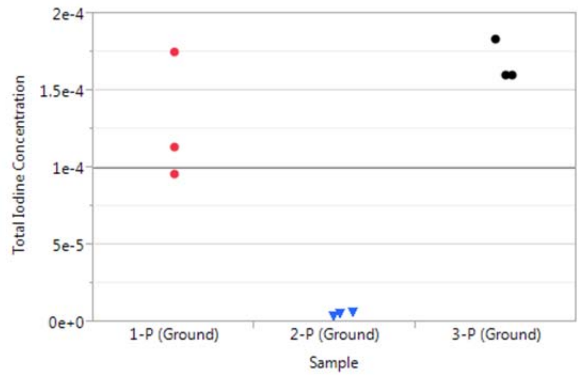
Sulfate, SO_4^{-2} (*wt %*)
All Results (triangles) below MDCs



Minimum Detection Concentrations (MDCs)

Sample	Number	Minimum MDC	Maximum MDC
1-P	3	4.70E-3	4.87E-3
2-P	3	4.70E-3	4.91E-3
3-P	3	4.66E-3	4.91E-3
Overall	9	4.66E-3	4.91E-3

Total Iodine (*wt %*)
Mixture of Results (circles) above MDCs
and (triangles) below MDCs



Sample	N	Minimum MDC	Maximum MDC
2-P	3	3.73E-6	5.42E-6

Test for Measurement Variance Heterogeneity

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	1.5556	1	4	0.2803
Brown-Forsythe	1.3062	1	4	0.3168
Levene	5.1049	1	4	0.0867
Bartlett	2.4071	1		0.1208

Comparison of Samples 1-P and 3-P only. Not Stat. Sig. at $\alpha = 0.05$.

The mean concentration for Sample 2-P is assumed to be between 0 and 4.64E-6 wt %. The nonparametric Chebyshev (MVUE) UCL95 in maximized over the data set of sample means {1.2800E-04, Y, 1.6800E-04}. The nonparametric Chebyshev (MVUE) UCL95 has been found to be maximized when the mean concentrations for Sample 2 is 0. The Maximum Chebyshev UCL95 is 3.1952E-4 wt %.

The associated mean and standard deviation of the data set of means {1.2800E-04, 0, 1.6800E-04} are 9.8667E-5 wt % and 8.7757E-5 wt %, respectively.

$$S_{Total} = \sqrt{S^2_{3\,Sample\,Means} - S^2_{MeasErr(Mean)} + S^2_{MeasErr(One\,Result)}}$$

$$= \sqrt{(8.7757E\text{-}5)^2 - (1.7949E\text{-}5)^2 + 3\cdot(1.7949E\text{-}5)^2} = 9.1355E\text{-}5,$$

where the measurement standard deviation is 3.1089E-05 wt %.

Nonparametric Statistics

Component	Estimate
Mean	9.8667E-5
Std.Dev.	9.1355E-5
(Nonparametric) Chebyshev UCL95	3.1952E-4

APPENDIX F: Statistical Analyses for Tank 16H Annulus Samples

1.0 INTRODUCTION

The goal of the Tank 16 annulus characterization is to document the physical, chemical, and radiological characteristics of the residual material remaining in the Tank 16H annulus based on samples of the annulus material. The primary statistical analyses objective is to establish an upper bound for the mean concentrations of these chemical and radionuclide analytes. Appendix D describes the statistical basis for the computations. The statistical analyses of the Tank 16H residual material are based on the analytical results presented in Appendix F: Table F5 for the physical parameters, Table F6 through Table F8 for the radionuclides, Table F9 through Table F11 for the elementals, and Table F12through Table F13 for the anions. The analytical results are either measurements, results that are above their minimum detectable concentrations (MDC's); or results that are less than their MDC's. Measurements are listed in Table F5 through Table F13 in black font, while censored results, listed as less-than-MDC values (<MDC) are set off in red font. The existence of censored values leads to partitioning the characteristics into three separate classes for statistical analyses:

- Analytes with all results below their MDC's.
- Analytes with all results above their MDC's.
- Analytes with a mixture of results that are above and below their MDC's.

These classes allow more uniform reporting of results, as analytes within any particular class tend to have similar statistical analyses. The upper bounds for the mean concentrations are 95% upper confidence limits (UCL95's) when all or most of the results are above their MDCs. When all or nearly all results are below their MDC's the upper bounds for the mean concentrations are represented by the minimum and maximum reported MDC's.

The sampling plan for the residual material remaining in the Tank 16H annulus was based on stratifying the annulus into non-overlapping sectors called strata. Fifteen samples of residual material were obtained from the Tank 16H. Five of the samples were assigned to each of three composite samples. The amount of material alliquoted from each primary sample to its assigned composite sample was based on the distribution of the relative proportion of mass of residual material across the strata. Each of the three composite samples was measured three times for a total of nine analytical results for each analyte of interest.

2.0 OBJECTIVE AND SCOPE

The following subsections apply the statistical methods described in Appendix D to characterize the concentrations of constituents in the residual material remaining in the Tank 16H annulus.

2.1 ANALYSIS OF PHYSICAL PARAMETERS

Two physical parameters were in the data set to be statistically analyzed: the homogenized composite sample bulk density (g/mL) and the weight percent solids (wt %). Both of these physical parameters had a set of 9 measurements: 3 measurements on each of three composite samples.

Refer to Table F1 for a classification of the physical parameters by sampling variance or no sampling variance model and whether potential outliers were detected. Levene's test for heterogeneity of variance was applied to both physical parameters with family-wise α = 0.025. Referring to **Table F14**, the Levene's test is not statistically significant (P-value > α) for either physical parameter. This means that the measurement error variances appear to be uniform across the composite samples. Therefore, tests to determine whether there is variance among the composite samples were performed using an analysis of variance (ANOVA) which assumes a constant measurement error variance. The ANOVA F-test for a sampling variance was statistically significant at α = 0.05 for the homogenized bulk density, but not statistically significant at α = 0.05 for wt % solids. Subsequently, the Wilk-Shapiro goodness-of-fit test for a normal distribution and the Dixon's test for an outlier were applied to the sample mean-centered residuals (since there was a statistically significant sampling variance) for the homogenized bulk density and to the wt % (since there was a non-significant sampling variance): neither test was statistically significant for either physical parameter. These results demonstrated that there was no significant lack of fit from a normal distribution or potential measurement outliers for either physical parameter. Subsequently, UCL95's were computed for the bulk density and the wt % solids using a one-sided upper Student's t confidence interval with 2 degrees of freedom (df) and 8 df, respectively. The summary of the results for the physical parameters, including UCL95's, is given in Table F14 with supporting information in Table F15.

Table F1. Classification of the Physical Parameters with All Measurements by Sampling Model and Whether the Data Exhibited Potential Outliers

Statistically Significant Sampling Variance (SS-SV)		Statistically Non-significant Sampling Variance (SNS-SV)	
Statistically Significant Outlier (SS-OT)	**No Statistically Significant Outliers (SNS-OT)**	**Statistically Significant Outlier (SS-OT)**	**No Statistically Significant Outliers (SNS-OT)**
<None>	Homogenized Bulk Density	<None>	Weight Percent Solids

2.2 ANALYSIS OF RADIONUCLIDES

Forty-two radionuclides plus the Pu-239/Pu-240 ratio were statistically analyzed. Twenty-one radionuclides, Am-242m, Am-243, C-14, Cf-249, Cf-251, Cl-36, Cm-242, Cm-243, Cm-244, Cm-245, Cm-247, Cm-248, K-40, Nb-94, Ni-59, Ni-63, Pa-231, Pu-244, Ra-226, Th-230, U-233, and Zr-93 had all of their results below their MDC's; 19 radionculides, Am-241, Ba-137m, Cs-135, Cs-137, Eu-154, I-129, Np-237, Pu-238, Pu-239, Pu-240, Pu-241, Pu-242, Sr-90, Tc-99, U-234, U-235, U-236, U-238, and Y-90 and the Pu-239/Pu-240 ratio had all measurements; and two radionuclides, Am-243 and Co-60, had a mixture of measurements and below-MDC results.

Refer to Table F2 for a breakdown of radionuclides with all measurement results by sampling variance model (or not) and the identification of outliers (or not). For the 19 radionuclides and the Pu-239/Pu-40 ratio with all measurements, Levene's test for homogeneous measurement error variance was never observed to be statistically significant at α = 0.0025. This means that the measurement error variances appear to be uniform across the composite samples. The subsequent ANOVA F-test for a sampling variance was statistically significant at α = 0.05 for all but I-129, Tc-99, U-235, and U-236. For the 15 radionuclides and the Pu-239/Pu-240 Ratio with a statistically significant sampling variance, the sample mean-centered residuals were examined for potential outliers with Dixon's outlier test, and 7 of the 15 radionuclides, Ba-137, Cs-137, Pu-238, Pu-239, Pu-240, Pu-241, and U-238 and the Pu-239/Pu-240 Ratio had a statistically significant low-side measurement outlier for Run 2 of Composite Sample 2 at α = 0.05. None of the sets of sample mean-centered residuals displayed lack of fit from a normal distribution using the Wilk-Shapiro goodness-of-fit test at α = 0.05. For I-129, Tc-99, U-235, and U-236 that did not exhibit a statistically significant sampling variance at α = 0.05, and only I-129 was identified to have a high-side outlier for Run 3 of Composite Sample 3 by the Dixon's test on the I-129 concentrations at $\alpha = 0.05$. None of these 4 sets of concentrations displayed lack of fit from a normal distribution using the Wilk-Shapiro goodness-of-fit test at $a = 0.05$.

Table F2. Classification of the Radionuclides with All Measurements by Sampling Model and Whether the Data Exhibited Potential Outliers

Statistically Significant Sampling Variance (SS-SV)		Statistically Non-significant Sampling Variance (SNS-SV)	
Statistically Significant Outlier (SS-OT)	**No Statistically Significant Outliers (SNS-OT)**	**Statistically Significant Outlier (SS-OT)**	**No Statistically Significant Outliers (SNS-OT)**
Ba-137	Am-241	I-129	Tc-99
Cs-137	Cs-135		U-235
Pu-238	Eu-154		U-236
Pu-239	Np-237		
Pu-240	Pu-242		
Pu-241	Sr-90		
U-238	U-234		
Pu-239/Pu-240 Ratio	Y-90		

Radionuclides that were identified to have a potential outlier were statistically reanalyzed without the outlier in the data set. All of these radionuclides retained the originally determined sampling variance or no sampling variance model, there were no further potential outliers identified by Dixon's test for outliers, and the measurements were all demonstrated to be consistent with a normal distribution once the outlier was removed. The UCL95's from the data sets without the potential outlier were higher than the UCL95's originally determined with the low-side outlier included for the characteristics that had a statistically significant sampling variance. The UCL95's without the outlier are recommended since they are conservatively large. The UCL95 for I-129 was smaller without the high-side outlier than with the outlier, and the higher UCL95 from the original data set with the outlieris recommended.

Two radionuclides had a mixture of results above and below their MDC's. Co-60 had two ≤MDC results for Composite Sample 1; the other 7 results were measurements. Estimates of each of the three composite sample means for Co-60 concentrations were derived using a model with a fixed sampling effect. Subsequently, these three composite sample mean estimates were used to construct a UCL95.

Am-243 had 8 of its 9 results below their MDC's. The sole measurement is larger than all but 1 of the 8 MDC values. No measure of variation can be extracted from a single measurement. It is suggested to adopt a 20% percent standard deviation as its measurement standard deviation. The 20% standard deviation represents a value larger than most of the percent standard deviations observed for radionuclides in the Tank 16H annulus residual material. A UCL95 for the Am-243 concentration for Run 1 of Composite Sample 2 with a measured value of 1.19E-2 μCi/g is 0.0119 + 2.9200 *(0.20 * 0.0119) = 1.885E-2 μCi/g, where 2.9200 is the 95% Student's t quantile with 2 df. This UCL95 for Run 1 of Composite Sample 2 can be considered to be an MDC value for Run 1 of Composite Sample 2. Replacing the measurement by this conservative UCL95 yields a data set of 9 ≤MDC results for Am-243. The Am-243 concentration can now be interpreted similarly to other radionuclides that have all less-than-MDC results.

The minimum and maximum MDC's for the radionuclides with all results below their MDC's are listed in Table F16. The UCL95's for radionuclides that have all measurements are summarized in Table F17. F18 contains the UCL95 for Co-60 and the minimum and maximum MDC's for Am-243. Table F19 contains supporting details for the statistical analyses of the radionuclides.

2.3. ANALYSIS OF ELEMENTALS

Twelve elementals, Ag, As, B, Cd, Co, Mo, Ni, Pb, Sb, Sr, U, and Zn displayed all less-than-MDC results. Nine elementals, Al, Ba, Cr, Cu, Fe, Hg, Mn, Na, and Si had all measurements, and Se had a mixture of above and below MDC results.

Refer to Table F3 for a classification of elementals with all measurement results by sampling variance model (or not) and the identification of outliers (or not). For the nine elementals with all measurements, Levene's test for homogeneous measurement error variance was never observed to be statistically significant at α = 0.0056. The subsequent ANOVA F-test for a sampling variance was statistically significant at α = 0.05 for all but Cu. For the eight elementals with a statistically significant sampling variance, the sample mean-centered residuals were examined for potential outliers with Dixon's outlier test, and only Al had a statistically significant low-side outlier for Run 2 of Composite Sample 2 at α = 0.05. The set of mean-centered residuals for Al did not exhibit a lack of fit from a normal distribution using the Wilk-Shapiro goodness-of-fit test at α = 0.05. A low-side outlier for Run 1 of Composite Sample 1 was observed for the Cu concentrations by Dixon's test at α = 0.05. The two elementals that were observed to have a potential outlier did not change their sampling variance or no sampling variance model when reanalyzed without the outlier. No further outliers were flagged by Dixon's outlier test for Al or Cu after the initial outlier was removed .

Both Al and Cu had larger UCL95 values with all of the data than with a potential outlier removed. The larger UCL95 values were adopted to be conservative.

Table F3. Classification of the Elementals with All Measurement Results by Sampling/Non-Sampling Model and Whether the Data Exhibited Potential Outliers

Statistically Significant Sampling Variance (SS-SV)		Statistically Non-significant Sampling Variance (SNS-SV)	
Statistically Significant Outlier (SS-OT)	**No Statistically Significant Outliers (SNS-OT)**	**Statistically Significant Outlier (SS-OT)**	**No Statistically Significant Outliers (SNS-OT)**
Al	B Cr Fe Hg Mn Na Si	Cu	<none>

The minimum and maximum MDC's for the elementals with all results below their MDC's are listed in Table F20. The UCL95's for elementals that have all measurements are summarized in Table F21 Table F22 contains the minimum and maximum MDC's for Se, which had a mixture of above and below MDC results. Table F23 contains supporting details for the statistical analyses of the elementals.

2.4 ANALYSIS OF ANIONS

Six anions, Bromide, Fuoride, Formate, Iodine I-127, Phosphate, and Total Iodine did not display any results above their MDCs. Six anions, Chloride, Iodine I-129, Nitrate, Nitrite, Oxalate, and Sulfate had all measurements. No anions had a mixture of above and below MDC results.

Table F4 shows the anions with all measurement results by sampling variance model (or not) and the identification of outliers (or not). For the six anions with all measurements, Levene's test for homogeneous measurement error variance was never observed to be statistically significant at $\alpha = 0.05/6 = 0.083$. The subsequent ANOVA F test for a sampling variance was statistically significant at $\alpha = 0.05$ for Nitrate, Chloride, and Sulfate. For these three anions with a statistically significant sampling variance, the sample mean-centered residuals were examined for potential outliers with Dixon's outlier test, and only Nitrate had a statistically significant low-side outlier for Run 2 of Composite Sample 2 at $\alpha = 0.05$. The set of mean-centered residuals for Nitrate did not exhibit a lack of fit from a normal distribution using the Wilk-Shapiro goodness-of-fit test at $\alpha = 0.05$. A high-side outlier for Run 3 of Composite Sample 3 was observed for the Iodine, I-129 concentrations by Dixon's test at $\alpha = 0.05$. The two anions that were observed to have a potential outlier did not change their sampling variance or no sampling variance model when reanalyzed without the outlier. No further outliers were flagged by Dixon's outlier test for Nitrate or Iodine, I-129 after the initial outlier was removed.

Both Nitrate and Iodine, I-129 had larger UCL95 values with all of the data than with a potential outlier removed. The larger UCL95 values were adopted to be conservative.

Table F4. Classification of the Anions with All Measurements by Sampling Model and Whether the Data Exhibited Potential Outliers

Statistically Significant Sampling Variance (SS-SV)		Statistically Non-significant Sampling Variance (SNS-SV)	
Statistically Significant Outlier (SS-OT)	**No Statistically Significant Outliers (SNS-OT)**	**Statistically Significant Outlier (SS-OT)**	**No Statistically Significant Outliers (SNS-OT)**
Nitrate	Chloride Sulfate	Iodine, I-129	Nitrite Oxalate

3.0 SUMMARY OF STATISTICAL ANALYSES FOR THE RESIDUAL MATERIAL TANK 16H PRIMARY TANK

Most composite samples exhibited a statistically significant sampling variance. A key feature was a potential measurement outlier, generally displayed for Run 2 of Composite Sample 2 for about eight radionuclides listed in Table F2. When a potential outlier was discovered by Dixon's outlier test, the analyses were rerun without the outlier, and the larger of the UCL95's, with or without the outlier in the data set, was adopted.

Another feature of this data set was the relatively low concentrations for many constituents. This resulted in a large number of constituents that displayed all results below their MDC values.

Several constituents had a mixture of measurements and less-than-MDC results. Co-60 had relatively few less-than-MDC results (2 of 9), so estimates of the mean concentration for each of the composite samples could be determined, and the UCL95 for the mean concentration of Co-60 in the population of residual material in the annulus was computed.

When there were relatively few measurements, an estimate of the mean concentration for each composite sample could not be determined: no UCL95 could be determined for the mean concentration of the constituent in the population of residual material in the annulus. In particular, the elemental Se had just 3 measurements, all for Composite Sample 3. An estimate of the sampling standard deviation could not be computed without any measurements on 2 of the 3 composite samples, but an estimate of the measurement standard deviation was available from the Composite Sample 3 results. Therefore, individual UCL95's could be computed for each of the runs for Composite Sample 3 and interpreted as MDC's for those runs. With all 9 analytical results for Se now considered less-than-MDC results, the Se concentration can be interpreted like any other constituent with all less-than-MDC results.

Am-243 had 1 measurement for Run 1 of Composite Sample 2: the other 8 results were less than their MDC's. No estimate of the measurement standard deviation or the sampling standard deviation could be extracted from the Am-243 results. A conservative (large) estimate of the measurement standard deviation was adopted for Am-243: 20%. An individual UCL95 was constructed for Run1 of Composite Sample 2, and it served as an MDC replacing the measurement for this run. Now with all results being less-than-MDC values, the Am-243 concentration was summarized like analytes with all less-than-MDC results.

The results are reported in tables in the following section. Each type of constituent, physical parameter, radionuclide, elemental, and anion is broken down into separate tables for the reporting results based on whether all results are less than their MDC's, all results are measurements, or the results are a mixture of measurements and below MDC values. Summary tables for each type of constituent are followed by extensive supporting tables. A list of the tables in Appendix F follows.

Appendix F: Statistical Analyses for Tank 16H Annulus Samples.

Classification Tables by Type of Statistical Model and Potential Outliers **Page**

Appendix F: Statistical Analysis of the Tank 16-H Annulus Samples

Table F5. Physical Parameters with All Results above their MDCs

Physical Parameters	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
Bulk Densities, g/mL	1.08	1.03	1.04	0.90	0.97	0.95	0.89	0.90	0.93
Wt % Solids	92.1	92.0	87.1	86.3	81.9	85.0	88.2	88.2	83.5

Table F6. Radionuclides with All Results below their MDCs

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	<3.14E-03	<1.14E-02	<1.16E-03	<1.26E-03	<1.56E-03	<6.53E-04	<1.56E-03	<5.86E-04	<2.74E-03
C-14	<7.52E-04	<7.57E-04	<7.52E-04	<8.33E-04	<7.57E-04	<7.57E-04	<7.57E-04	<8.29E-04	<6.94E-04
Cf-249	<1.40E-03	<1.50E-03	<1.19E-03	<1.48E-03	<1.25E-03	<1.51E-03	<1.16E-03	<9.28E-04	<1.16E-03
Cf-251	<3.79E-03	<4.00E-03	<3.16E-03	<3.98E-03	<3.28E-03	<3.95E-03	<3.21E-03	<2.45E-03	<2.87E-03
Cl-36	<8.78E-04	<6.13E-04	<4.33E-04	<5.59E-04	<4.55E-04	NR	<6.71E-04	<5.45E-04	<6.40E-04
Cm-242	<2.59E-03	<9.41E-03	<9.59E-04	<1.04E-03	<1.29E-03	<5.41E-04	<1.29E-03	<4.82E-04	<2.27E-03
Cm-243	<4.59E-03	<4.91E-03	<3.90E-03	<5.00E-03	<4.12E-03	<4.95E-03	<4.19E-03	<3.05E-03	<3.66E-03
Cm-244	<3.83E-01	<1.52E-01	<1.50E-01	<1.41E+00	<1.13E-01	<1.82E-01	<8.02E-01	<1.71E-01	<1.47E-01
Cm-245	<3.23E-05	<1.48E-05	<1.45E-05	<1.21E-04	<1.05E-05	<1.55E-05	<6.71E-05	<1.45E-05	<1.48E-05
Cm-247	<1.72E-09	<1.36E-09	<1.28E-09	<5.45E-09	<6.76E-10	<1.19E-09	<3.23E-09	<8.02E-10	<9.46E-10
Cm-248	<1.15E-06	<1.25E-06	<9.91E-07	<9.86E-07	<7.61E-07	<1.34E-06	<9.73E-07	<8.60E-07	<8.83E-07
K-40	<2.46E-05	<3.74E-05	<3.87E-05	<2.48E-05	<3.78E-05	<3.56E-05	<2.56E-05	<3.92E-05	<2.41E-05
Nb-94	<3.67E-04	<4.59E-04	<3.87E-04	<4.48E-04	<4.31E-04	<3.86E-04	<5.23E-04	<5.00E-04	<5.90E-04
Ni-59	<4.64E-02	<6.22E-03	<1.36E-03	<1.83E-03	<2.23E-02	<1.89E-02	<1.32E-03	<8.78E-03	<1.91E-03
Ni-63	<7.16E-01	<2.91E-01	<7.57E-02	<1.71E-01	<4.36E-01	<2.94E-01	<5.81E-02	<3.98E-01	<7.93E-02
Pa-231	<4.77E-04	<2.22E-04	<3.02E-04	<9.86E-04	<3.86E-04	<6.89E-04	<2.90E-04	<2.49E-04	<4.15E-04
Pu-244	<1.12E-07	<9.95E-08	<1.12E-07	<1.02E-07	<1.29E-07	<1.04E-07	<1.43E-07	<1.02E-07	<1.67E-07
Ra-226	<1.60E-03	<9.32E-04	<1.01E-03	<8.15E-04	<9.01E-04	<8.83E-04	<9.10E-04	<1.49E-04	<6.13E-04
Th-230	<7.21E-05	<1.86E-04	<1.80E-04	<4.18E-04	<8.11E-05	<1.10E-04	<7.52E-05	<5.23E-05	<1.16E-04
U-233	<2.08E-03	<1.73E-03	<2.10E-03	<1.81E-03	<2.29E-03	<1.72E-03	<4.59E-03	<1.94E-03	<1.61E-03
Zr-93#	<1.44E-01	<1.50E-01	<1.48E-01	<1.91E-01	<1.61E-01	<1.86E-01	<1.33E-01	<1.32E-01	<1.23E-01

NR: Not reported because data did not meet quality assurance requirements.
Zr-93 values are considered upper limits because blanks were greater than 10% of the sample value.

Appendix F: Statistical Analysis of the Tank 16-H Annulus Samples

Table F7. Radionuclides with All Results above their MDCs

Radionuclide	Composite Sample 1			Composite Sample 2			Composite Sample 3		
Constituents (μCi/g)	Run 1	Run 2	Run 3	Run 1	Run 2	Run 3	Run 1	Run 2	Run 3
Am-241	7.52E-01	8.24E-01	7.12E-01	9.37E-01	9.10E-01	1.05E+00	6.89E-01	5.23E-01	5.50E-01
Ba-137m	6.90E+02	6.90E+02	6.95E+02	7.07E+02	6.35E+02	6.99E+02	5.88E+02	5.84E+02	5.80E+02
Cs-135	2.56E-03	2.61E-03	2.61E-03	2.70E-03	2.47E-03	2.66E-03	2.37E-03	2.19E-03	2.29E-03
Cs-137	7.30E+02	7.30E+02	7.34E+02	7.48E+02	6.71E+02	7.39E+02	6.22E+02	6.17E+02	6.13E+02
Eu-154	7.57E-01	7.66E-01	7.43E-01	1.09E+00	9.77E-01	1.08E+00	7.70E-01	7.12E-01	7.21E-01
I-129	8.51E-04	6.71E-04	8.06E-04	6.49E-04	8.83E-04	7.61E-04	9.32E-04	6.31E-04	1.66E-03
Np-237	1.83E-03	1.80E-03	1.82E-03	2.59E-03	2.66E-03	2.50E-03	1.58E-03	1.48E-03	1.48E-03
Pu-238	3.45E+00	3.36E+00	3.39E+00	4.27E+00	3.79E+00	4.50E+00	2.88E+00	2.65E+00	2.65E+00
Pu-239	4.64E-01	4.30E-01	4.24E-01	5.59E-01	4.95E-01	6.22E-01	3.51E-01	3.28E-01	3.38E-01
Pu-239/240	6.76E-01	6.26E-01	6.17E-01	8.15E-01	7.21E-01	9.05E-01	5.14E-01	4.77E-01	4.95E-01
Pu-240	2.12E-01	1.97E-01	1.93E-01	2.55E-01	2.26E-01	2.82E-01	1.60E-01	1.50E-01	1.56E-01
Pu-241	1.30E+00	1.30E+00	1.33E+00	1.85E+00	1.45E+00	1.71E+00	1.02E+00	9.05E-01	9.05E-01
Pu-242	9.41E-05	8.11E-05	8.56E-05	1.09E-04	9.46E-05	1.17E-04	6.94E-05	6.85E-05	7.03E-05
Sr-90	1.40E+03	1.47E+03	1.21E+03	2.02E+03	2.13E+03	1.96E+03	1.23E+03	1.33E+03	1.38E+03
Tc-99	2.06E-01	2.24E-01	2.39E-01	2.04E-01	2.63E-01	2.91E-01	2.19E-01	3.00E-01	3.00E-01
U-234	1.50E-03	1.57E-03	1.54E-03	1.75E-03	1.57E-03	1.78E-03	1.57E-03	1.46E-03	1.40E-03
U-235	2.55E-05	2.56E-05	2.48E-05	2.47E-05	2.27E-05	2.51E-05	2.40E-05	2.26E-05	2.25E-05
U-236	5.27E-05	5.36E-05	5.18E-05	5.41E-05	5.09E-05	5.63E-05	5.45E-05	5.18E-05	5.18E-05
U-238	1.06E-04	1.06E-04	1.04E-04	1.03E-04	9.46E-05	1.05E-04	9.41E-05	9.19E-05	9.19E-05
Y-90	1.40E+03	1.47E+03	1.21E+03	2.02E+03	2.13E+03	1.96E+03	1.23E+03	1.33E+03	1.38E+03

MDCs: Minimum Detectable Concentrations

Appendix F: Statistical Analysis of the Tank 16-H Annulus Samples

Table F8. Radionuclides with a Mixture of Results above and below their MDCs

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-243	<3.55E-03	<2.12E-03	<1.13E-03	1.19E-02	<1.90E-03	<2.14E-03	<1.20E-02	<1.92E-03	<4.82E-03
Co-60	2.01E-03	<3.02E-03	<2.21E-03	2.56E-03	2.80E-03	3.18E-03	1.73E-03	1.70E-03	2.27E-03

MDCs: Minimum Detectable Concentrations

Appendix F: Statistical Analysis of the Tank 16-H Annulus Samples

Table F9. Elemental Constituents with All Results below their MDCs

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	<3.27E-03	<3.31E-03	<3.23E-03	<3.33E-03	<3.30E-03	<3.31E-03	<3.29E-03	<3.28E-03	<3.25E-03
As	<2.61E-04	<2.70E-04	<2.66E-04	<2.53E-04	<2.57E-04	<2.74E-04	<2.66E-04	<2.58E-04	<2.61E-04
B	<4.72E-02	<4.78E-02	<4.66E-02	<4.80E-02	<4.76E-02	<4.78E-02	<4.74E-02	<4.72E-02	<4.69E-02
Cd	<3.39E-03	<3.44E-03	<3.35E-03	<3.45E-03	<3.42E-03	<3.43E-03	<3.41E-03	<3.40E-03	<3.37E-03
Co*	<2.77E-02	<3.25E-02	<3.30E-02	<4.94E-02	<5.87E-02	<5.11E-02	<5.11E-02	<4.03E-02	<5.25E-02
Mo	<3.62E-02	<3.66E-02	<3.58E-02	<3.68E-02	<3.65E-02	<3.66E-02	<3.64E-02	<3.62E-02	<3.60E-02
Ni	<5.10E-02	<5.30E-02	<5.20E-02	<5.00E-02	<5.00E-02	<5.40E-02	<5.20E-02	<5.10E-02	<5.10E-02
Pb	<1.28E-01	<1.30E-01	<1.26E-01	<1.30E-01	<1.29E-01	<1.30E-01	<1.29E-01	<1.28E-01	<1.27E-01
Sb	<7.02E-02	<7.11E-02	<6.94E-02	<7.15E-02	<7.08E-02	<7.10E-02	<7.06E-02	<7.03E-02	<6.97E-02
Sr*	<7.22E-03	<6.90E-03	<7.00E-03	<9.59E-03	<9.07E-03	<9.59E-03	<6.35E-03	<6.22E-03	<6.18E-03
U	<2.36E-01	<2.39E-01	<2.33E-01	<2.40E-01	<2.38E-01	<2.39E-01	<2.37E-01	<2.36E-01	<2.34E-01
Zn*	<4.80E-02	<5.55E-02	<4.47E-02	<5.82E-02	<5.73E-02	<5.56E-02	<5.69E-02	<5.97E-02	<6.29E-02

* Upper limit data since the blank was greater than 10% of the sample value.

Table F10. Elemental Constituents with All Results above their MDCs

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
Al	6.12E+00	5.99E+00	6.03E+00	5.95E+00	5.56E+00	5.99E+00	6.93E+00	6.88E+00	6.86E+00
Ba	1.75E-02	1.66E-02	1.77E-02	1.88E-02	1.74E-02	1.91E-02	1.25E-02	1.23E-02	1.22E-02
Cr	1.50E-02	1.46E-02	1.47E-02	2.66E-02	2.50E-02	2.65E-02	1.68E-02	1.77E-02	1.74E-02
Cu	2.14E-01	7.93E-02	1.57E-02	1.59E-02	1.88E-02	1.41E-02	1.37E-02	1.53E-02	7.62E-02
Fe	1.98E+00	1.74E+00	1.82E+00	2.87E+00	2.84E+00	2.97E+00	2.32E+00	2.64E+00	2.50E+00
Hg	1.90E-01	2.04E-01	1.94E-01	2.34E-01	2.18E-01	2.34E-01	1.94E-01	1.83E-01	1.83E-01
Mn	1.96E-02	1.96E-02	2.08E-02	3.02E-02	2.91E-02	3.19E-02	2.06E-02	2.14E-02	2.14E-02
Na	1.27E+01	1.27E+01	1.31E+01	1.73E+01	1.78E+01	1.77E+01	1.69E+01	1.56E+01	1.56E+01
Si	1.99E+01	1.94E+01	1.99E+01	1.04E+01	9.29E+00	1.03E+01	1.10E+01	1.11E+01	1.14E+01

Appendix F: Statistical Analysis of the Tank 16-H Annulus Samples

Table F11. Elemental Constituents with a Mixture of Results above and below their MDCs

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
Se	<5.23E-04	<5.40E-04	<5.32E-04	<5.06E-04	<5.14E-04	<5.49E-04	9.68E-04	9.40E-04	7.73E-04

Table F12. Anions with All Results below their MDCs

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
Formate, CHO_2^{-1}	<2.84E-02	<2.89E-02	<2.84E-02	<3.04E-02	<2.69E-02	<3.02E-02	<2.95E-02	<2.86E-02	<2.92E-02
Phosphate, PO_4^{-3}	<2.84E-02	<2.89E-02	<2.84E-02	<3.04E-02	<2.69E-02	<3.02E-02	<2.95E-02	<2.86E-02	<2.92E-02
Bromide, Br^{-1}	<2.84E-02	<2.89E-02	<2.84E-02	<3.04E-02	<2.69E-02	<3.02E-02	<2.95E-02	<2.86E-02	<2.92E-02
Fluoride, F-1	<2.84E-02	<2.89E-02	<2.84E-02	<3.04E-02	<2.69E-02	<3.02E-02	<2.95E-02	<2.86E-02	<2.92E-02
Iodine, I-127	<1.71E-05	<1.74E-05	<1.71E-05	<1.83E-05	<1.62E-05	<1.81E-05	<1.77E-05	<1.71E-05	<1.75E-05
Total Iodine	<4.99E-04	<3.97E-04	<4.74E-04	<3.86E-04	<5.16E-04	<4.49E-04	<5.46E-04	<3.74E-04	<9.60E-04

MDCs: Minimum Detectable Concentrations

Table F13. Anions with All Results above their MDCs

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
Nitrate , NO_3^{-1}	5.31E+00	5.21E+00	5.29E+00	4.96E+00	4.52E+00	4.92E+00	4.90E+00	4.86E+00	4.90E+00
Nitrite , NO_2^{-1}	6.45E+00	6.11E+00	6.23E+00	6.33E+00	5.55E+00	6.18E+00	6.11E+00	5.94E+00	6.04E+00
Oxalate, $C_2O_4^{2-}$	6.82E-02	6.37E-02	6.26E-02	7.30E-02	6.73E-02	6.03E-02	6.79E-02	8.57E-02	8.17E-02
Sulfate, SO_4^{-2}	4.55E-01	4.23E-01	4.47E-01	8.00E-01	7.51E-01	8.06E-01	7.11E-01	7.12E-01	7.09E-01
Chloride , Cl^{-1}	4.26E-02	3.47E-02	3.41E-02	6.39E-02	5.11E-02	6.03E-02	2.95E-02	3.71E-02	3.50E-02
Iodine, I-129	4.82E-04	3.80E-04	4.57E-04	3.68E-04	5.00E-04	4.31E-04	5.28E-04	3.57E-04	9.42E-04

Appendix F: Statistical Analysis of the Tank 16-H Annulus Samples

Table F14. Statistical Summary for the Physical Parameters

Physical Parameters	N	Mean (g/mL)	Std Dev♠ (g/mL)	% Std Dev	UCL95 (g/mL)	Goodness-of-Fit/Confidence Limit Remarks♠
Bulk Density, g/mL	9	0.9656	0.07853	8.13%	1.092	SNS-VH; SS-SV; SNS-WS; SNS-OT; Use Student's t UCL95 (2 df)
Wt % Solids	9	87.14	3.472	3.98%	89.30	SNS-VH; SNS-SV; SNS-WS; SNS-OT; Use Student's t UCL95 (8 df)

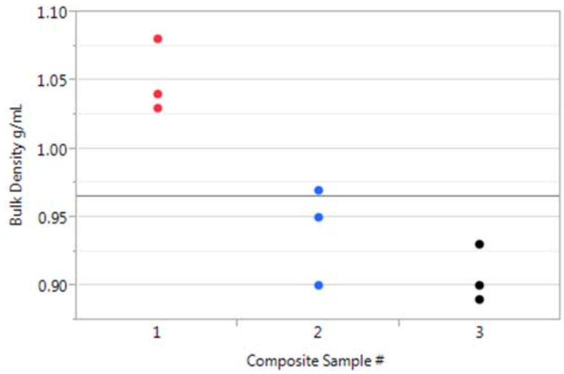
SS-VH/SNS-VH: Statistically significant/Statistically non-significant test for measurement error heterogeneity at $\alpha = 0.05/2 = 0.025$.
SS-SV / SNS-SV: Statistically significant/Statistically non-significant sampling variance at $\alpha = 0.05$.
SS-WS/ SNS-WS: Statistically significant/Statistically non-significant Wilk-Shapiro test for normality at $\alpha = 0.05$.
SS-OT/ SNS-OT: Statistically significant/Statistically non-significant Dixon's test for outliers at $\alpha = 0.05$.
♠ When the sampling variation among the composite samples is demonstrated to be statistically significant (SS-SV), the standard deviation (Std Dev) and the percent standard deviation (% Std Dev) account for the variation among the composite samples and the variation attributable to a single measurement of the composite sample: $StdDev : s_{Total} = \sqrt{s_{Samp}^2 + s_{Meas}^2}$.

Appendix F: Statistical Analysis of the Tank 16-H Annulus Samples

Table F15. Supporting Results for Physical Parameters

Bulk Densities, g/mL

All Results (circles) above MDCs



Means and Measurement Error Standard Deviations

Sample	Number	Mean	Std Dev	Std Err Mean
1	3	1.0500	0.02646	0.01528
2	3	0.9400	0.03606	0.02082
3	3	0.9067	0.02082	0.01202
Pooled	9	0.9656	0.02848	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	0.4438	2	6	0.6611
Brown-Forsythe	0.1795	2	6	0.8400
Levene	0.6972	2	6	0.5342
Bartlett	0.2475	2	.	0.7807

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	0.0337556	0.016878	20.808	0.0020*
Error	6	0.0048667	0.000811		
C. Total	8	0.0386222			

Statistically significant sampling variance.

Diagnostics for Sampling Model Centered Residuals

Wilk-Shapiro	0.929 > 0.829 crit. value->SNS
Dixon High Outlier	Run 1 of Sample 1 -> SNS
Dixon Low Outlier	Run 1 of Sample 2 -> SNS

Variance Components

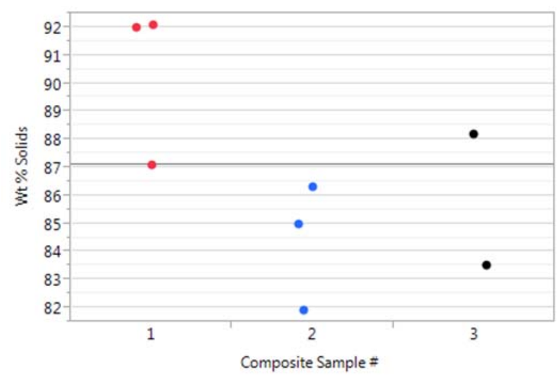
Component	Variance Comp	Std Dev
Composite Samples	5.35556E-3	7.3182E-2
Measurements	8.11111E-4	2.8480E-2
Total	6.16667E-3	7.8528E-2
Mean Concentration	1.87531E-3	4.3305E-2

Student's t UCL95

UCL95	1.09205

Wt % Solids

All Results (circles) above MDCs



Means and Measurement Error Standard Deviations

Sample	Number	Mean	Std Dev	Std Err Mean
1	3	90.40	2.858	1.650
2	3	84.40	2.261	1.305
3	3	86.63	2.714	1.567
Pooled	9	87.14	2.623	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	0.0912	2	6	0.9141
Brown-Forsythe	0.0051	2	6	0.9949
Levene	0.2667	2	6	0.7745
Bartlett	0.0476	2	.	0.9535

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	55.175556	27.5878	4.0092	0.0784
Error	6	41.286667	6.8811		
C. Total	8	96.462222			

Statistically non-significant sampling variance.

Diagnostics for Concentrations

Wilk-Shapiro	0.951 > 0.829 crit. value->SNS
Dixon High Outlier	Run 1 of Sample 1 -> SNS
Dixon Low Outlier	Run 2 of Sample 2 -> SNS

Normal Statistics

Component	Estimate
Mean (MLE)	87.144
Std.Dev. (Bias-corr. MLE)	3.4724
Student's t UCL95	89.297

Appendix F: Statistical Analysis of the Tank 16-H Annulus Samples

Table F16. Statistical Summary for the Radionuclides with All Measurements below their MDCs

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
Am-242m	9	0.000586	5.86e-4	0.0114	1.14e-2
C-14	9	0.000694	6.94e-4	0.000833	8.33e-4
Cf-249	9	0.000928	9.28e-4	0.00151	1.51e-3
Cf-251	9	0.00245	2.45e-3	0.004	4.00e-3
Cl-36	8	0.000433	4.33e-4	0.000878	8.78e-4
Cm-242	9	0.000482	4.82e-4	0.00941	9.41e-3
Cm-243	9	0.00305	3.05e-3	0.005	5.00e-3
Cm-244	9	0.113	1.13e-1	1.41	1.41e+0
Cm-245	9	0.0000105	1.05e-5	0.000121	1.21e-4
Cm-247	9	0.000000000676	6.76e-10	0.00000000545	5.45e-9
Cm-248	9	0.000000761	7.61e-7	0.00000134	1.34e-6
K-40	9	0.0000241	2.41e-5	0.0000392	3.92e-5
Nb-94	9	0.000367	3.67e-4	0.00059	5.90e-4
Ni-59	9	0.00132	1.32e-3	0.0464	4.64e-2
Ni-63	9	0.0581	5.81e-2	0.716	7.16e-1
Pa-231	9	0.000222	2.22e-4	0.000986	9.86e-4
Pu-244	9	0.0000000995	9.95e-8	0.000000167	1.67e-7
Ra-226	9	0.000149	1.49e-4	0.0016	1.60e-3
Th-230	9	0.0000523	5.23e-5	0.000418	4.18e-4
U-233	9	0.00161	1.61e-3	0.00459	4.59e-3
Zr-93	9	0.123	1.23e-1	0.191	1.91e-1

MDCs: Minimum Detectable Concentrations

Appendix F: Statistical Analysis of the Tank 16-H Annulus Samples

Table F17. Statistical Summary for the Radionuclides with All Results above their MDCs

Constituent	N	Mean♣ (μCi/g)	Std Dev♠ (μCi/g)	% Std Dev	UCL95 (μCi/g)	Goodness-of-Fit/Confidence Limit Remarks♦
Am-241	9	7.7189E-1	1.9888E-1	25.77%	1.0911E+0	SNS-VH; SS-SV; SNS-WS; SNS-OT; Student's t UCL95
Ba-137m	9	6.5200E+2	6.2061E+1	9.52%	7.5174E+2	SNS-VH; SS-SV; SNS-WS; SS-OT (Run 2 Sample 2); Student's t UCL95
	8	6.5955e+2	6.5758E+1	9.97%	7.7026E+2	Omitted a potential outlier at Run 2 from Composite Sample 2. SNS-VH; SS-SV; SNS-WS ; SNS-OT; Student's t UCL95
Cs-135	9	2.4956E-3	1.9798E-4	7.933%	2.8057E-3	SNS-VH; SS-SV; SNS-WS; SNS-OT; Student's t UCL95
Cs-137	9	6.8933E+2	6.5754E+1	9.54%	7.9494E+2	SNS-VH; SS-SV; SNS-WS ; SS-OT (Run 2 Sample 2); Student's t UCL95
	8	6.9738E+2	6.9682E+1	9.99%	8.1471E+2	Omitted a potential outlier at Run 2 from Composite Sample 2. SNS-VH; SS-SV; SNS-WS; SNS-OT; Student's t UCL95
Eu-154	9	8.4622E-1	1.7907E-1	21.16%	1.1428E+0	SNS-VH; SS-SV; SNS-WS; SNS-OT; Student's t UCL95
I-129	9	8.7156E-4×	3.1159E-4×	35.75%	1.1221E-3	SNS-VH; SNS-SV; SS-WS; SS-OT (Composite Sample 3, Run 3: 0.00166μCi/g); SNS-KS (Appr. Gamma); 95% Adjusted gamma UCL95
	8	7.7300E-4	1.1379E-4	14.72%	8.4922E-4	Omitted Run 3 from Composite Sample 3: 0.00166□ompos SNS-VH; SNS-SV; SNS-OT; SNS-WS; Student's t UCL95
Np-237	9	1.9711E-3	5.5348E-4	28.08%	2.9008E-3	SNS-VH; SS-SV; SNS-WS ; SNS-OT; Student's t UCL95
Pu-238	9	3.4378E+0	7.5334E-1	21.91%	4.6697E+0	SNS-VH; SS-SV; SNS-WS ; SS-OT: Run 2 Sample 2; Student's t UCL95
	8	3.5030E+0	8.3826E-1	23.93%	4.9094E+0	Omitted a potential outlier at Run 2 from Composite Sample 2. SNS-VH; SNS-SV; SNS-OT; SNS-WS; Student's t UCL95
Pu-239	9	4.4567E-1	1.1455E-1	25.70%	6.3106E-1	Omitted a potential outlier at Run 2 from Composite Sample 2. SNS-VH; SS-SV; SS-OT; SNS-WS; Student's t UCL95
	8	4.5598E-1	1.2794E-1	28.06%	6.6954 E-1	Omitted a potential outlier at Run 2 from Composite Sample 2. SS-VH; SNS-SV; SNS-OT; SNS-WS; Student's t UCL95

SS-VH/SNS-VH: Statistically significant/Statistically non-significant test for measurement error heterogeneity at $\alpha = 0.05/20=0.0025$.
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 $\alpha = 0.05$.
SS-KS; SNS-KS: Statistically significant/Statistically non-significant Kolmogorov-Smirnov test for gamma distribution at $\alpha = 0.05$.
SNS-OT (SS-OT): Statistically significant/Statistically non-significant Dixon's test for outliers at $\alpha = 0.05$. The results depend on normal distribution assumption.
♣ Estimate of the mean and standard deviation based on bias-corrected maximum likelihood assuming a gamma distribution.
× Note that the mean used is the REML estimate (the mean of the sample means) when the sampling variance is statistically significant and the number of measurements per sample differs.
♠ When the sampling variation among the composite samples is demonstrated to be statistically significant (SS-SV), the standard deviation (Std Dev) and the percent standard deviation (% Std Dev) account for the variation among the composite samples and the variation attributable to a single measurement of the composite sample: $StdDev: s_{Total} = \sqrt{s_{Samp}^2 + s_{Meas}^2}$.

Appendix F: Statistical Analysis of the Tank 16-H Annulus Samples

Table 17 Continued. Statistical Summary for the Radionuclides with All Results above their MDCs

Constituent	N	Mean♣ (μCi/g)	Std Dev♠ (μCi/g)	% Std Dev	UCL95 (μCi/g)	Goodness-of-Fit/Confidence Limit Remarks♦
Pu-239/240 (dimensionless)	9	6.4956E-1	1.6610E-1	25.57%	9.1828E-1	SNS-VH; SS-SV; SS-OT; SNS-WS; Student's t UCL95
	8	6.6457E-1	1.8561E-1	27.93%	9.7436 E-1	Omitted a potential outlier at Run 2 from Composite Sample 2. SNS-VH; SS-SV; SNS-OT; SNS-WS; Student's t UCL95
Pu-240	9	2.0344E-1	5.1558E-2	25.34%	2.8699E-1	SNS-VH; SS-SV; SS-OT; SNS-WS; Student's t UCL95
	8	2.0804E-1	5.753E-2	27.65%	3.0411 E-1	Omitted a potential outlier at Run 2 from Composite Sample 2. SNS-VH; SS-SV; SNS-OT; SNS-WS; Student's t UCL95
Pu-241	9	1.3078E+0	3.7712E-1	28.84%	1.9203E+0	SNS-VH; SS-SV; SS-OT; SNS-WS; Student's t UCL95
	8	1.3439E+0	4.2187E-1	31.39%	2.0512E+0	Omitted a potential outlier at Run 2 from Composite Sample 2. SNS-VH; SS-SV; SNS-OT; SNS-WS; Student's t UCL95
Pu-242	9	8.7733E-5	1.9746E-5	22.51%	1.1934E-4	SNS-VH; SS-SV; SNS-OT; SNS-WS; Student's t UCL95
Sr-90	9	1.5700E+3	4.1334E+2	26.33%	2.2525E+3	SNS-VH; SS-SV; SNS-OT; SNS-WS; Student's t UCL95
Tc-99	9	2.4956E-1	3.9778E-2	15.94%	2.7421E-1	SNS-VH; SNS-SV; SNS-WS; SNS-OT; Student's t UCL95
U-234	9	1.5711E-3	1.3473E-4	8.58%	1.7660E-3	SNS-VH; SS-SV; SNS-WS; SNS-OT; Student's t UCL95
U-235	9	2.4167E-5	1.2649E-6	5.23%	2.4951E-5	SNS-VH; SNS-SV; SNS-WS; SNS-OT; Student's t UCL95
U-236	9	5.3056E-5	1.7140E-6	3.23%	5.4118E-5	SNS-VH; SNS-SV; SNS-WS; SNS-OT; Student's t UCL95
U-238	9	9.9611E-5	6.9948E-6	7.02%	1.1047E-4	SNS-VH; SS-SV; SNS-WS; SS-OT; Student's t UCL95
	8	1.0065E-4	7.0563E-6	7.01%	1.1241E-4	Omitted a potential outlier at Run 2 from Composite Sample 2. SNS-VH; SS-SV; SNS-OT; SNS-WS; Student's t UCL95
Y-90	9	1.5700E+3	4.1334E+2	26.33%	2.2525E+3	SNS-VH; SS-SV; SNS-OT; SNS-WS; Student's t UCL95

SS-VH/SNS-VH: Statistically significant/Statistically non-significant test for measurement error heterogeneity at $\alpha = 0.05/20=0.0025$.

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 $\alpha = 0.05$.

SNS-OT (SS-OT): Statistically significant/Statistically non-significant Dixon's test for outliers at $\alpha = 0.05$. The results depend on normal distribution assumption.

♣ Note that the mean used is the REML estimate (the mean of the sample means) when the sampling variance is statistically significant and the number of measurements per sample differs.

♠ When the sampling variation among the composite samples is demonstrated to be statistically significant (SS-SV), the standard deviation (Std Dev) and the percent standard deviation (% Std Dev) account for the variation among the composite samples and the variation attributable to a single measurement of the composite sample: $StdDev: s_{Total} = \sqrt{s_{Samp}^2 + s_{Meas}^2}$.

Appendix F: Statistical Analysis of the Tank 16-H Annulus Samples

Table F18. Statistical Summary for the Radionuclides with Results above and below their MDCs

Constituent	N	Mean (μCi/g)	Std Dev (μCi/g)♠	% Std Dev	UCL95 (μCi/g)♠	Goodness-of-Fit/Confidence Limit Remarks♦
Co-60	9 (2,0,0)	2.232E-3	5.6630E-4	25.37%	3.1303E-3	Sampling variance model was used assuming normal data. See the Co-60 entry in Table F19 for additional information.

N is the number of analytical results. The numbers inside the parentheses (X, Y, Z) represent: X = Number of less-than-MDC results for Composite Sample 1, Y = number of less-than-MDC results for Composite Sample 2, and Z = number of less-than-MDC results for Composite Sample 3.

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
Am-243	9 (3,2,3)	0.00113	1.13E-3	0.0189	1.89E-2

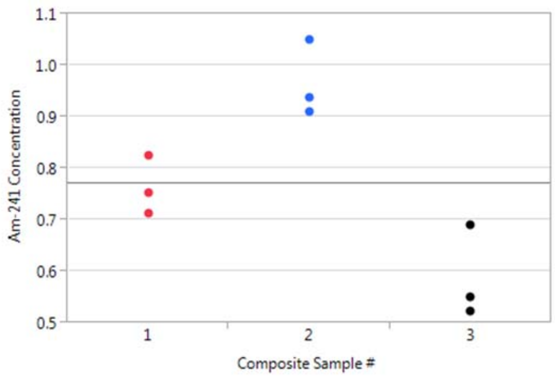
MDC: Minimum Detectable Concentration.

Both Co-60 and Am-243 fall into the class of radionuclides containing a mixture of above and below MDC results. However, 7 of the 9 Co-60 results were measurements, so that the Co-60 results contained sufficient information to construct a UCL95. In contrast, only 1 of the 9 Am-243 results was a measurement, so that a UCL95 could not be computed.

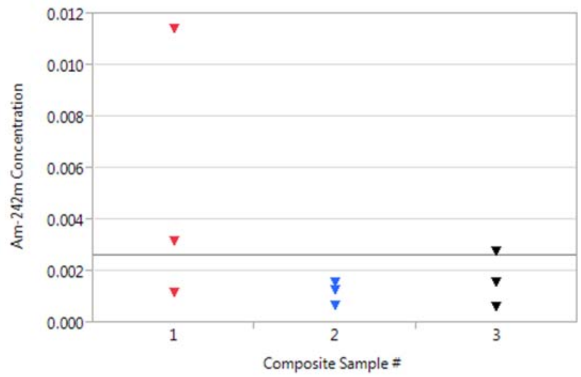
Appendix F: Statistical Analysis of the Tank 16-H Annulus Samples

Table F19. Support Results for Radionuclides

Am-241 (μCi/g)
All Results (circles) above MDCs



Am-242m (μCi/g)
All Results (triangles) below MDCs



Means and Standard Deviations by Sample

Sample	Number	Mean	Std Dev	Std Err Mean
1	3	0.762667	0.056757	0.03277
2	3	0.965667	0.074272	0.04288
3	3	0.587333	0.089075	0.05143
Pooled	9	0.771889	0.074548	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	0.2855	2	6	0.7613
Brown-Forsythe	0.0714	2	6	0.9319
Levene	0.6467	2	6	0.5567
Bartlett	0.1609	2		0.8514

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	0.21508689	0.107543	19.352	0.0024
Error	6	0.03334400	0.005557		
C. Total	8	0.24843089			

Statistically significant sampling variance.

Diagnostics for Sampling Model Centered Residuals

Wilk-Shapiro	0.845 > 0.829 crit. value->SNS
Dixon High Outlier	Run 1 of Sample 3 -> SNS
Dixon Low Outlier	Run 2 of Sample 3 -> SNS

Variance Components

Component	Variance Comp	Std Dev
Composite Samples	3.399537E-2	1.84378E-1
Measurements	5.557333E-3	7.45475E-2
Total	3.955270E-2	1.98879E-1
Mean Concentration	1.194927E-2	1.09313E-1

Student's t UCL95

UCL95	1.09108

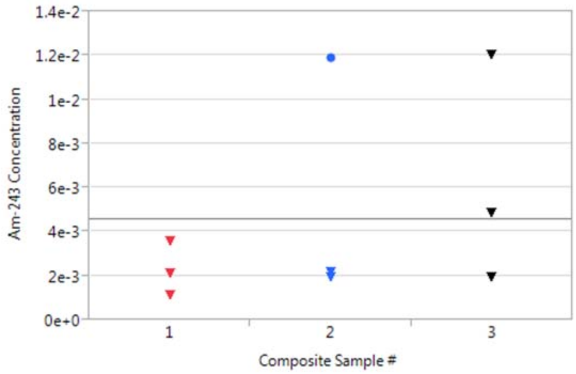
Minimum Detection Concentrations (MDCs)

Sample	N	Minimum MDC	Maximum MDC
1	3	1.16E-3	1.14E-2
2	3	6.53E-4	1.56E-3
3	3	5.86E-4	2.74E-3
Overall	9	5.86E-4	1.14E-2

Appendix F: Statistical Analysis of the Tank 16-H Annulus Samples

Table F19. Support Results for Radionuclides

Am-243 (μCi/g)
Mixture of Results (circles) above MDCs
and (triangles) below MDCs



In addition to the above 8 results less than their MDCs, 1 result was a measurement for Run 1 of Composite Sample 2: 1.19E-2 μCi/g. No measure of variation is available from this data set because it only has one measurement. A percent standard deviation of 20% is larger than most percent standard deviations for radionuclides in the annulus material, and is adopted for Am-243. A UCL95 for the individual result for Run 1 of Composite Sample 2 is then 0.0119 + 2.9200 * (0.20 * 0.0119) = 0.018850 μCi/g. This UCL95 can replace the single 0.0119 measurement as a MDC value. The minimum and maximum MDC ignoring the run1 composite Sample 2 measurement are in the following table.

Minimum Detection Concentrations (MDCs) Ignoring the Measurement for Run1 of Composite Sample 2

Sample	Number	Minimum MDC	Maximum MDC
1	3	1.13E-3	3.55E-3
2	2	1.90E-3	1.19E-2
3	3	1.92E-3	1.20E-2
Overall	8	1.13E-3	1.20E-2

Minimum Detection Concentrations (MDCs) Replacing the Measurement for Run1 of Composite Sample 2 by its Computed MDC value

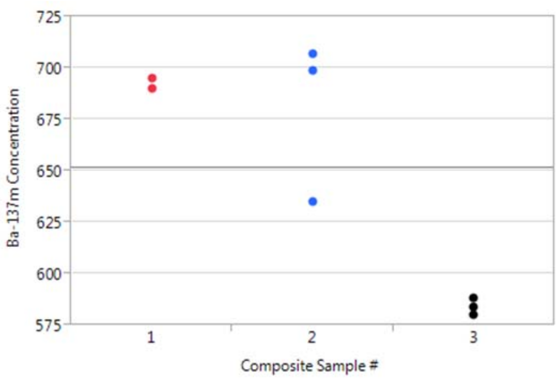
Sample	Number	Minimum MDC	Maximum MDC
1	3	1.13E-3	3.55E-3
2	2	1.90E-3	1.89E-2
3	3	1.92E-3	1.20E-2
Overall	8	1.13E-3	1.89E-2

Appendix F: Statistical Analysis of the Tank 16-H Annulus Samples

Table F19. Support Results for Radionuclides

Ba-137m (μCi/g)

All Results (circles) above MDCs



Means and Standard Deviations

Sample	Number	Mean	Std Dev	Std Err Mean
1	3	691.667	2.8868	1.667
2	3	680.333	39.4631	22.784
3	3	584.000	4.0000	2.309
Pooled	9	652.000	22.9613	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	1.7499	2	6	0.2519
Brown-Forsythe	1.1648	2	6	0.3737
Levene	11.9640	2	6	0.0081
Bartlett	5.3667	2		0.0047

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	21000.667	10500.3	19.9163	0.0022
Error	6	3163.333	527.2		
C. Total	8	24164.000			

Statistically significant sampling variance.

Diagnostics for Sampling Model Residuals

Wilk-Shapiro	0.833 > 0.829 crit. value->SNS
Dixon High Outlier	Run 1 of Sample 2 -> SNS
Dixon Low Outlier	Run 2 of Sample 2 -> SS

Variance Components

Component	Variance Comp	Std Dev
Composite Samples	3.324370E+3	57.6574
Measurements	5.272222E+2	22.9613
Total	3.851593E+3	62.0612
Mean Concentration	1.166704E+3	34.1570

Student's t UCL95

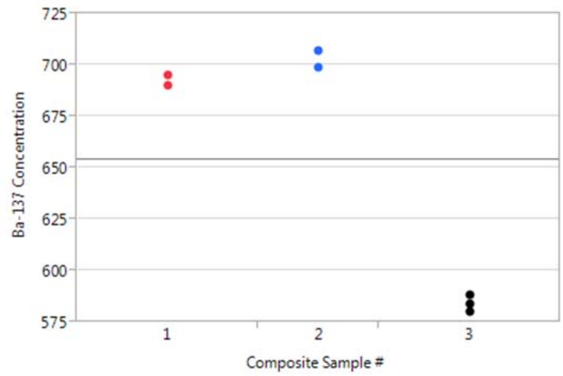
UCL95	751.7381

Repeat analysis without potential outlier at Sample 2 Run 2 (635 μCi/g).

Ba-137m (μCi/g)

All Results (circles) above MDCs

Omitted Sample 2 Run 2: 635μCi/g



Means and Standard Deviations

Sample	Number	Mean	Std Dev	Std Err Mean
1	3	691.667	2.88675	1.6667
2	2	703.000	5.65685	4.0000
3	3	584.000	4.00000	2.3094
Pooled	8	654.125	4.01663	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	0.3211	1	4	0.6012
Brown-Forsythe	0.5983	2	5	0.5848
Levene	0.7840	2	5	0.5056
Bartlett	0.2512	2		0.7779

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	23758.208	11879.1	736.308	<.0001
Error	5	80.667	16.1		
C. Total	7	23838.875			

Statistically significant sampling variance.

Diagnostics for Sampling Model Residuals

Wilk-Shapiro	0.862 > 0.829 crit. value->SNS
Dixon High Outlier	Run 1 of Sample 2 -> SNS
Dixon Low Outlier	Run 3 of Sample 2 -> SNS

Variance Components

Component	Variance Comp	Std Dev
Composite Samples	4307.934	65.6349
Measurements	16.133	4.01657
Total	4324.067	65.7576
Mean Concentration	1438.070	37.9219

REML Mean	659.5465

Student's t UCL95

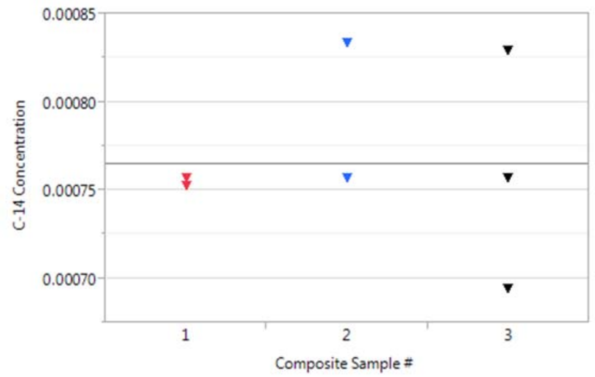
UCL95	770.2671

Appendix F: Statistical Analysis of the Tank 16-H Annulus Samples

Table F19. Support Results for Radionuclides

C-14 (μCi/g)

All Results (triangles) below MDCs



Minimum Detection Concentrations (MDCs)

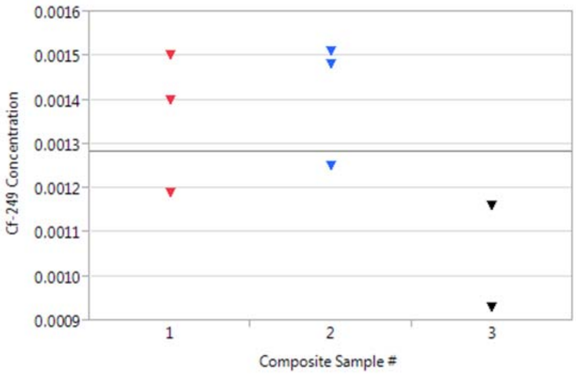
Sample	Number	Minimum MDC	Maximum MDC
1	3	7.52E-4	7.57E-4
2	3	7.57E-4	8.33E-4
3	3	6.94E-4	8.29E-4
Overall	9	6.94E-4	8.33E-4

Appendix F: Statistical Analysis of the Tank 16-H Annulus Samples

Table F19. Support Results for Radionuclides

Cf-249 (μCi/g)

All Results (triangles) below MDCs

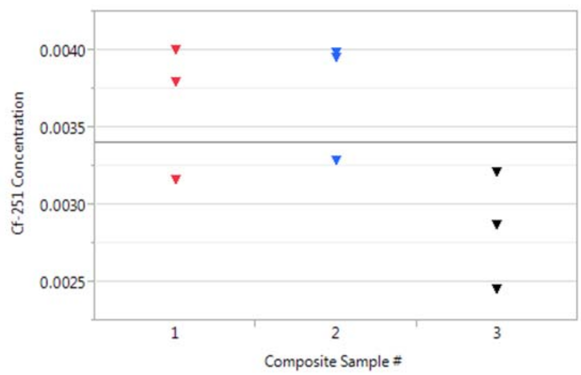


Minimum Detection Concentrations (MDCs)

Sample	Number	Minimum MDC	Maximum MDC
1	3	1.19E-3	1.50E-3
2	3	1.25E-3	1.51E-3
3	3	9.28E-4	1.16E-3
Overall	9	9.28E-4	1.51E-3

Cf-251 (μCi/g)

All Results (triangles) below MDCs



Minimum Detection Concentrations (MDCs)

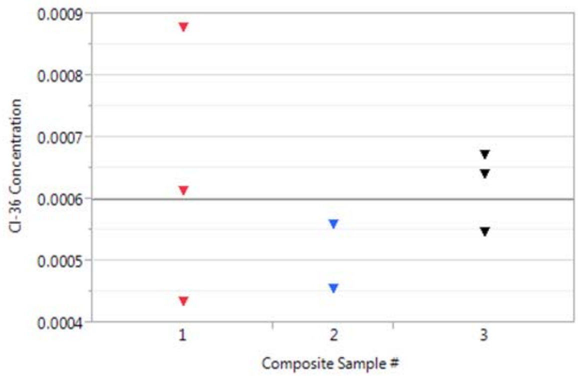
Sample	Number	Minimum MDC	Maximum MDC
1	3	3.16E-3	4.00E-3
2	3	3.28E-3	3.98E-3
3	3	2.45E-3	3.21E-3
Overall	9	2.45E-3	4.00E-3

Appendix F: Statistical Analysis of the Tank 16-H Annulus Samples

Table F19. Support Results for Radionuclides

Cl-36 (μCi/g)

All Results (triangles) below MDCs

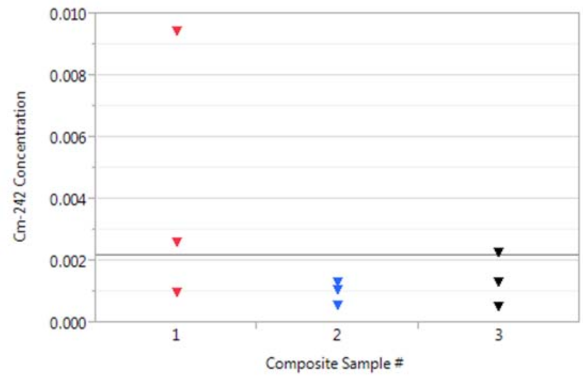


Minimum Detection Concentrations (MDCs)

Sample	Number	Minimum MDC	Maximum MDC
1	3	4.33E-4	8.78E-4
2	2	4.55E-4	5.59E-4
3	3	5.45E-4	6.71E-4
Overall	8	4.33E-4	8.78E-4

Cm-242 (μCi/g)

All Results (triangles) below MDCs



Minimum Detection Concentrations (MDCs)

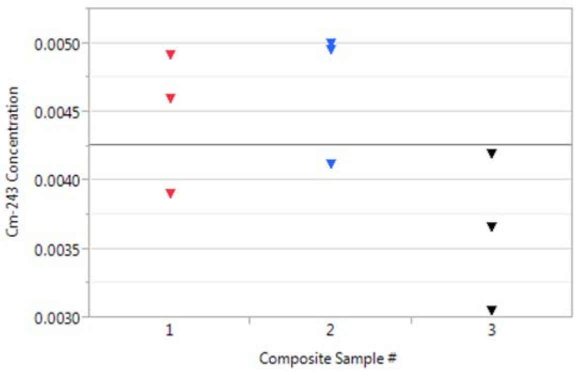
Sample	Number	Minimum MDC	Maximum MDC
1	3	9.59E-4	9.41E-3
2	3	5.41E-4	1.29E-3
3	3	4.82E-4	2.27E-3
Overall	9	4.82E-4	9.41E-3

Appendix F: Statistical Analysis of the Tank 16-H Annulus Samples

Table F19. Support Results for Radionuclides

Cm-243 (μCi/g)

All Results (triangles) below MDCs

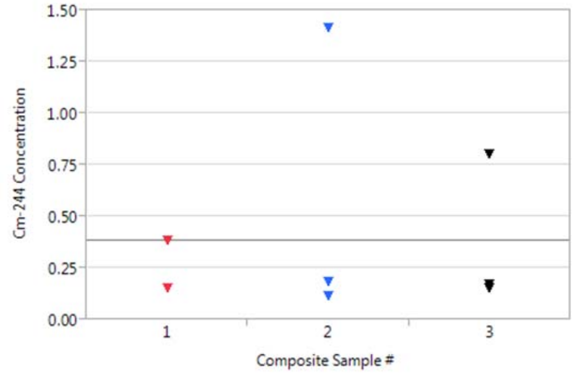


Minimum Detection Concentrations (MDCs)

Sample	Number	Minimum MDC	Maximum MDC
1	3	3.90E-3	4.91E-3
2	3	4.12E-3	5.00E-3
3	3	3.05E-3	4.19E-3
Overall	9	3.05E-3	5.00E-3

Cm-244 (μCi/g)

All Results (triangles) below MDCs



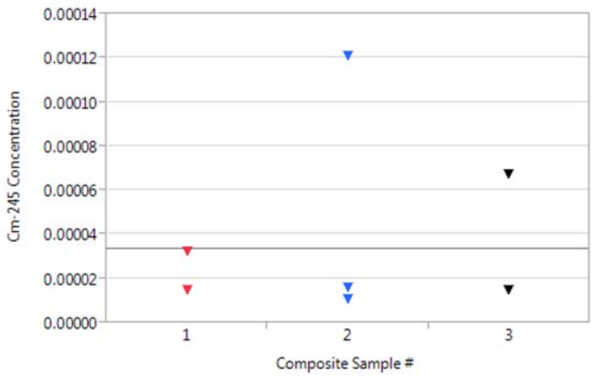
Minimum Detection Concentrations (MDCs)

Sample	Number	Minimum MDC	Maximum MDC
1	3	0.150	0.383
2	3	0.113	1.410
3	3	0.147	0.802
Overall	9	0.113	1.410

Appendix F: Statistical Analysis of the Tank 16-H Annulus Samples

Table F19. Support Results for Radionuclides

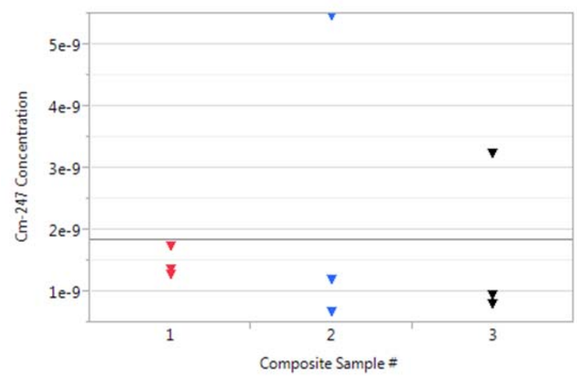
Cm-245 (µCi/g)
All Results (triangles) below MDCs



Minimum Detection Concentrations (MDCs)

Sample	Number	Minimum MDC	Maximum MDC
1	3	1.45E-5	3.23E-5
2	3	1.05E-5	1.21E-4
3	3	1.45E-5	6.71E-5
Overall	9	1.05E-5	1.21E-4

Cm-247 (µCi/g)
All Results (triangles) below MDCs



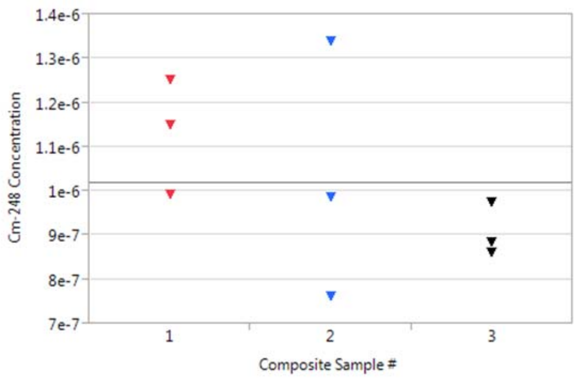
Minimum Detection Concentrations (MDCs)

Sample	Number	Minimum MDC	Maximum MDC
1	3	1.28E-9	1.72E-9
2	3	6.76E-10	5.45E-9
3	3	8.02E-10	3.23E-9
Overall	9	6.76E-10	5.45E-9

Appendix F: Statistical Analysis of the Tank 16-H Annulus Samples

Table F19. Support Results for Radionuclides

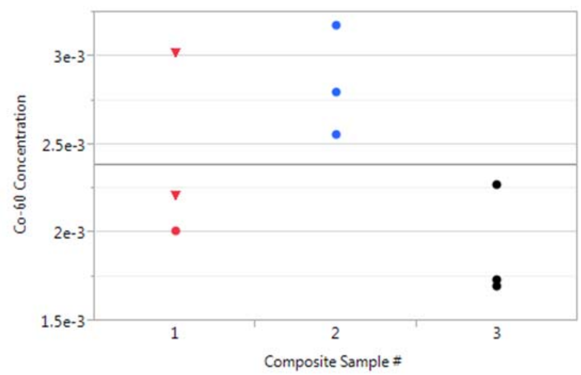
Cm-248 (μCi/g)
All Results (triangles) below MDCs



Minimum Detection Concentrations (MDCs)

Sample	Number	Minimum MDC	Maximum MDC
1	3	9.91e-7	1.25e-6
2	3	7.61e-7	1.34e-6
3	3	8.60e-7	9.73e-7
Overall	9	7.61e-7	1.34e-6

Co-60
Mixture of Results (circles) above MDCs and (triangles) below MDCs



Sample 1

Run 1	Run 2	Run 3
0.0020	<0.00302	<0.00221

Means and Standard Deviations

Sample	Number	Mean	Std Dev	Std Err Mean
2	3	2.847E-3	3.13E-4	1.805E-4
3	3	1.900E-3	3.21e-4	1.852E-4

SAS Proc Reliability accommodates censored data as well as measurements. An ANOVA model with a fixed effect sampling term was run in Proc Reliability. The following table displays the results. The Student's t UCL95 was based on the 3 sample mean estimates from this table.

SAS Proc Reliability Results

Parameters	Estimates	Std Error
Mean Sample 1	1.9506E-3	2.818E-4
Mean Sample 2	2.8467E-3	2.359E-4
Mean Sample 3	1.9000E-3	1.362E-4
Meas. Error Std Dev	2.358E-4	6.22E-5

Statistics for the 3 Sample Mean Estimates

Mean	Std Dev	UCL95
2.2324E-3	5.32572E-4	3.1303E-3

$s_{Estimates} = 5.3257E-4 = \sqrt{s_{Samp}^2 + s_{Meas}^2/3}$ and $s_{Meas} = 2.358E-4$

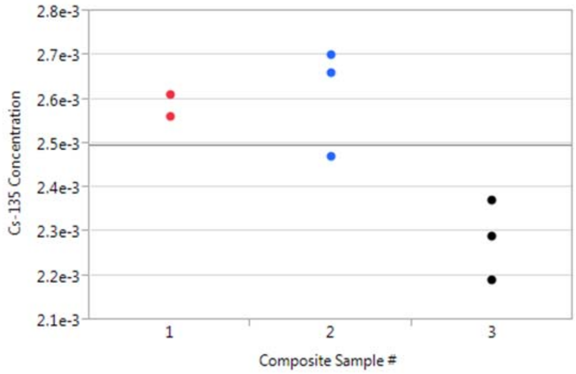
$s_{Total} = \sqrt{s_{Samp}^2 + s_{Meas}^2} = \sqrt{(5.1488E-4)^2 + (2.358E-4)^2} = 5.633E-4$

Appendix F: Statistical Analysis of the Tank 16-H Annulus Samples

Table F19. Support Results for Radionuclides

Cs-135

All Results (circles) above MDCs



Means and Standard Deviations

Sample	N	Mean	Std Dev	Std Err Mean
1	3	2.5933E-3	2.8868E-5	1.6667E-5
2	3	2.6100E-3	1.2288E-4	7.0946E-5
3	3	2.2833E-3	9.0185E-5	5.2068E-5
Overall	9	2.4956E-3	1.7721E-4	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	0.9206	2	6	0.4480
Brown-Forsythe	0.6317	2	6	0.5637
Levene	2.5691	2	6	0.1563
Bartlett	1.3241	2		0.2661

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	2.0309E-7	1.0154E-7	12.658	0.0070
Error	6	4.8133E-8	8.0222E-9		
C. Total	8	2.5122E-7			

Statistically significant sampling variance.

Diagnostics for Sampling Model Residuals

Wilk-Shapiro	0.927 > 0.829 crit. value->SNS
Dixon High Outlier	Run 1 of Sample 2 -> SNS
Dixon Low Outlier	Run 2 of Sample 2 -> SNS

Variance Components

Component	Variance Comp	Std Dev
Composite Samples	3.1174E-8	1.7656E-4
Measurements	8.0222E-9	8.9567E-5
Total	3.9196E-8	1.9798E-4
Mean Concentration	1.1283E-8	1.0622E-4

Student's t UCL95

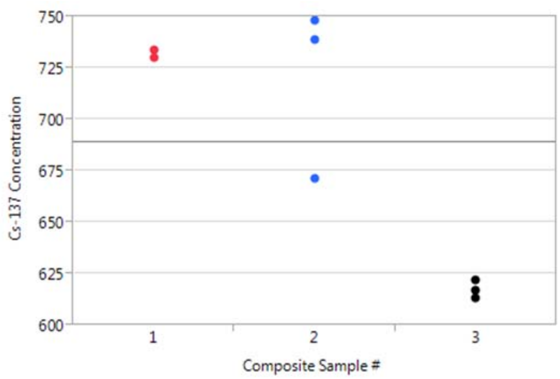
UCL95	2.8057E-3

Appendix F: Statistical Analysis of the Tank 16-H Annulus Samples

Table F19. Support Results for Radionuclides

Cs-137 (μCi/g)

All Results (circles) above MDCs



Means and Standard Deviations

Sample	Number	Mean	Std Dev	Std Err Mean
1	3	731.333	2.30940	1.33333
2	3	719.333	42.0991	24.3059
3	3	617.333	4.50925	2.60342
Pooled	9	689.333	24.4813	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	1.7520	2	6	0.2516
Brown-Forsythe	1.2080	2	6	0.3624
Levene	12.0368	2	6	0.0079
Bartlett	5.7446	2		0.0032

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	23544.000	11772.0	19.642	0.0023
Error	6	3596.000	599.3		
C. Total	8	27140.000			

Statistically significant sampling variance.

Diagnostics for Sampling Model Residuals

Wilk-Shapiro	0.832 > 0.829 crit. value->SNS
Dixon High Outlier	Run 1 of Sample 2 -> SNS
Dixon Low Outlier	Run 2 of Sample 2 -> SS

Variance Components

Component	Variance Comp	Std Dev
Composite Samples	3.72422E+03	6.1026E+01
Measurements	5.99333E+02	2.4481E+01
Total	4.32356E+03	6.5754E+01
Mean Concentration	1.30800E+03	3.6166E+01

Student's t UCL95

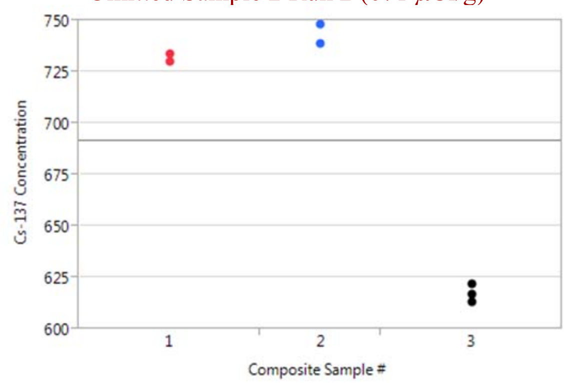
UCL95	794.9384

Repeat analysis without potential outlier at Sample 2 Run 2 (671 μCi/g).

Cs-137 (μCi/g)

All Results (circles) above MDCs

Omitted Sample 2 Run 2 (671 μCi/g)



Means and Standard Deviations

Sample	Number	Mean	Std Dev	Std Err Mean
1	3	731.333	2.30940	1.3333
2	2	743.500	6.36396	4.5000
3	3	617.333	4.50925	2.6034
Pooled	8	691.625	4.28563	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	0.9052	1	4	0.3953
Brown-Forsythe	1.2500	2	5	0.3629
Levene	1.7567	2	5	0.2643
Bartlett	0.5688	2		0.5662

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	26670.042	13335.0	726.04	<.0001
Error	5	91.833	18.4		
C. Total	7	26761.875			

Statistically significant sampling variance.

Diagnostics for Sampling Model Residuals

Wilk-Shapiro	0.901 > 0.829 crit. value->SNS
Dixon High Outlier	Run 1 of Sample 3 -> SNS
Dixon Low Outlier	Run 3 of Sample 2 -> SNS

Variance Components

Component	Variance Comp	Std Dev
Composite Samples	4.83723E+03	6.9550E+01
Measurements	1.83661E+01	4.2856E+00
Total	4.85560E+03	6.9682E+01
Mean Concentration	1.61445E+03	4.0180E+01

REML Mean	697.3792

Student's t UCL95

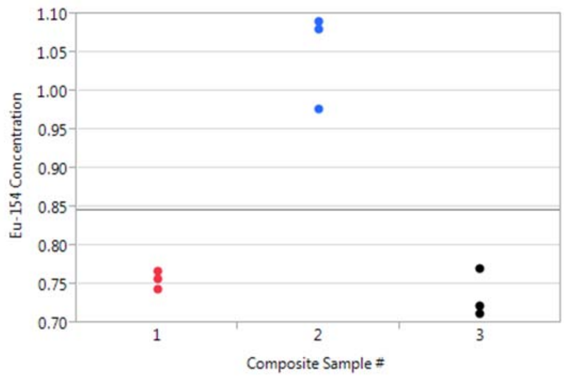
UCL95	814.7057

Appendix F: Statistical Analysis of the Tank 16-H Annulus Samples

Table F19. Support Results for Radionuclides

Eu-154 (μCi/g)

All Results (circles) above MDCs



Means and Standard Deviations

Sample	Number	Mean	Std Dev	Std Err Mean
1	3	0.75533	0.011590	0.006692
2	3	1.04900	0.062554	0.036116
3	3	0.73433	0.031214	0.018022
Pooled	9	0.84622	0.040913	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	1.2897	2	6	0.3420
Brown-Forsythe	0.5202	2	6	0.6190
Levene	5.8691	2	6	0.0387
Bartlett	1.8119	2		0.1633

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance

Source	DF	SS	MS	F Ratio	Prob >
Sample	2	0.18569622	0.092848	55.4685	0.0001
Error	6	0.01004333	0.001674		
C. Total	8	0.19573956			

Statistically significant sampling variance.

Diagnostics for Sampling Model Residuals

Wilk-Shapiro	0.923 > 0.829 crit. value->SNS
Dixon High Outlier	Run 1 of Sample 2 -> SNS
Dixon Low Outlier	Run 2 of Sample 2 -> SNS

Variance Components

Component	Variance Comp	Std Dev
Composite Samples	3.03914E-02	1.7433E-01
Measurements	1.67389E-03	4.0913E-02
Total	3.20653E-02	1.7907E-01
Mean Concentration	1.03165E-02	1.0157E-01

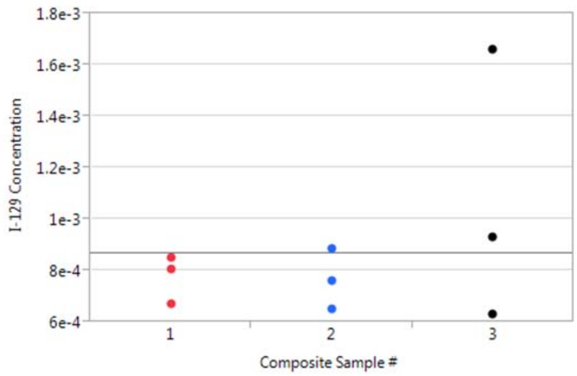
Student's t UCL95

UCL95	1.14281

Appendix F: Statistical Analysis of the Tank 16-H Annulus Samples

Table F19. Support Results for Radionuclides

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



Means and Standard Deviations

Sample	N	Mean	Std Dev	Std Err Mean
1	3	7.7600E-4	9.3675E-5	5.4083E-5
2	3	7.6433E-4	1.1704E-4	6.7571E-5
3	3	1.0743E-3	5.2906E-4	3.0555E-4
Pooled	9	8.7156E-4	3.1748E-4	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	1.6328	2	6	0.2715
Brown-Forsythe	1.5787	2	6	0.2813
Levene	5.2581	2	6	0.0479
Bartlett	2.7946	2		0.0611

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	1.85289e-7	9.262e-8	0.9189	0.4486
Error	6	6.04753e-7	1.008e-7		
C. Total	8	7.89992e-7			

Statistically non-significant sampling variance.

Diagnostics for Concentrations

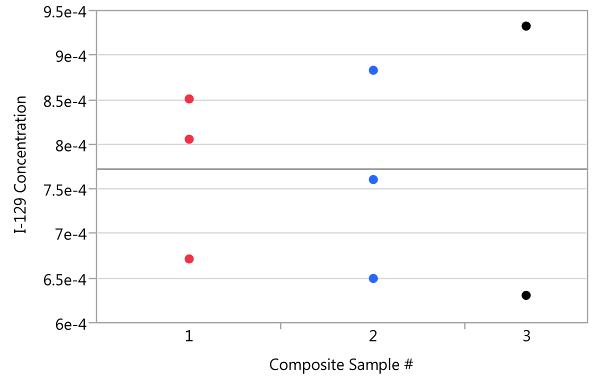
Wilk-Shapiro	0.708 < 0.829 crit. value->SS
Dixon High Outlier	Run 3 of Sample 3 -> SS
Dixon Low Outlier	Run 2 of Sample 3 -> SNS
KS (approx. Gamma)	0.261<0.279 crit. value -> SNS

Gamma Statistics

Component	Estimate
Mean (MLE)	8.7156E-4
Std.Dev. (Bias-corr. MLE)	3.1159E-4
Adjusted Gamma UCL95	1.1221E-3

Iodine, I-129 was reanalyzed in the right column after omitting a potential outlier for Run 3 of Composite Sample 3 (1.66E-3 wt %).

I-129 (μCi/g)
All Results (circles) above MDCs
Omitted Run 3 of Sample 3: 1.66E-3 μCi/g



Means and Standard Deviations

Sample	N	Mean	Std Dev	Std Err Mean
1	3	7.7600E-4	9.3675E-5	5.4083E-5
2	3	7.6433E-4	1.1704E-4	6.7571E-5
3	2	7.8150E-4	2.1284E-4	1.5050E-4
Pooled	8	7.7300E-4	1.3435E-4	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	0.1628	1	4	0.7072
Brown-Forsythe	1.4025	2	5	0.3285
Levene	1.9130	2	5	0.2416
Bartlett	0.4130	2		0.6616

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	3.9683E-10	1.984E-10	0.0110	0.9891
Error	5	9.0245E-8	1.805E-8		
C. Total	7	9.0642E-8			

Statistically non-significant sampling variance.

Diagnostics for Concentrations

Wilk-Shapiro	0.928 > 0.818 crit. value->SNS
Dixon High Outlier	Run 1 of Sample 3 -> SNS
Dixon Low Outlier	Run 2 of Sample 3 -> SNS

Normal Statistics

Component	Estimate
Mean	7.7300E-4
Standard Deviation	1.1379E-4
Student's t UCL95 with 7 df	8.4922E-4

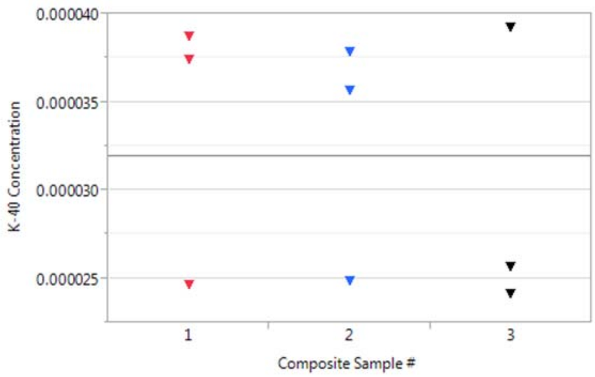
The UCL95 with all of the I-129 data is recommended over this result that omitted a measurement.

Appendix F: Statistical Analysis of the Tank 16-H Annulus Samples

Table F19. Support Results for Radionuclides

K-40 (μCi/g)

All Results (triangles) below MDCs

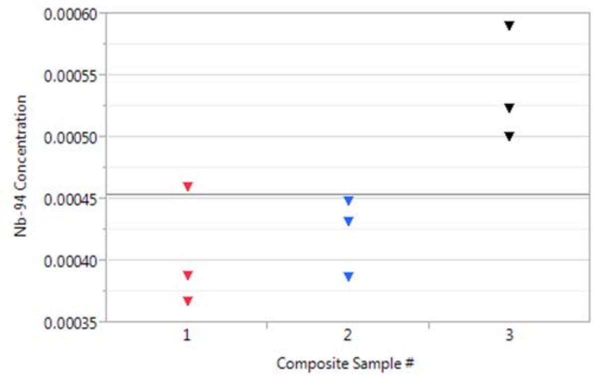


Minimum Detection Concentrations (MDCs)

Sample	Number	Minimum MDC	Maximum MDC
1	3	2.46E-5	3.87E-5
2	3	2.48E-5	3.78E-5
3	3	2.41E-5	3.92E-5
Overall	9	2.41E-5	3.92E-5

Nb-94 (μCi/g)

All Results (triangles) below MDCs



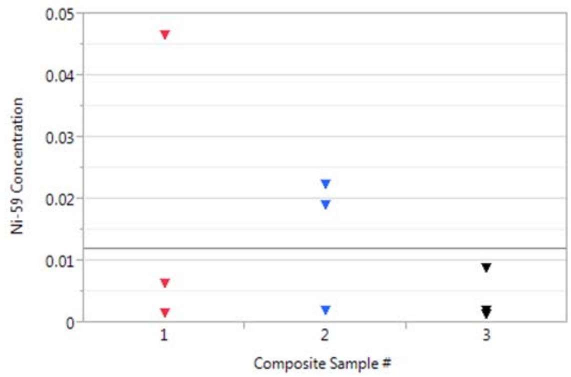
Minimum Detection Concentrations (MDCs)

Sample	Number	Minimum MDC	Maximum MDC
1	3	3.67E-4	4.59E-4
2	3	3.86E-4	4.48E-4
3	3	5.00E-4	5.90E-4
Overall	9	3.67E-4	5.90E-4

Appendix F: Statistical Analysis of the Tank 16-H Annulus Samples

Table F19. Support Results for Radionuclides

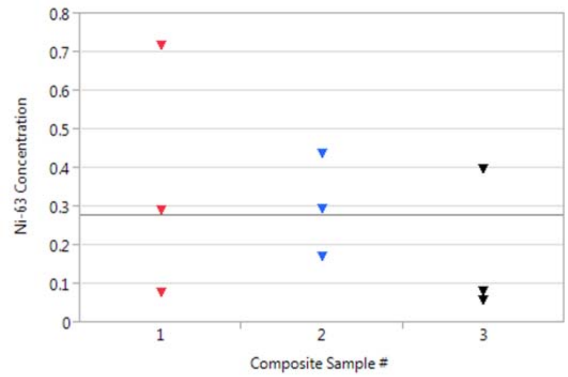
Ni-59 (μCi/g)
All Results (triangles) below MDCs



Minimum Detection Concentrations (MDCs)

Sample	Number	Minimum MDC	Maximum MDC
1	3	1.36E-3	4.64E-2
2	3	1.83E-3	2.23E-2
3	3	1.32E-3	8.78E-3
Overall	9	1.32E-3	4.64E-2

Ni-63 (μCi/g)
All Results (triangles) below MDCs



Minimum Detection Concentrations (MDCs)

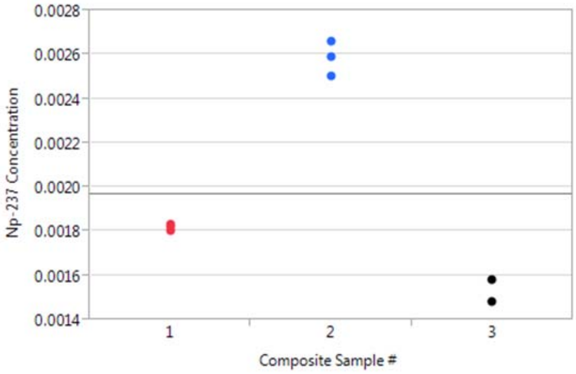
Sample	Number	Minimum MDC	Maximum MDC
1	3	7.57E-2	7.16E-1
2	3	1.71E-1	4.36E-1
3	3	5.81E-2	3.98E-1
Overall	9	5.81E-2	7.16E-1

Appendix F: Statistical Analysis of the Tank 16-H Annulus Samples

Table F19. Support Results for Radionuclides

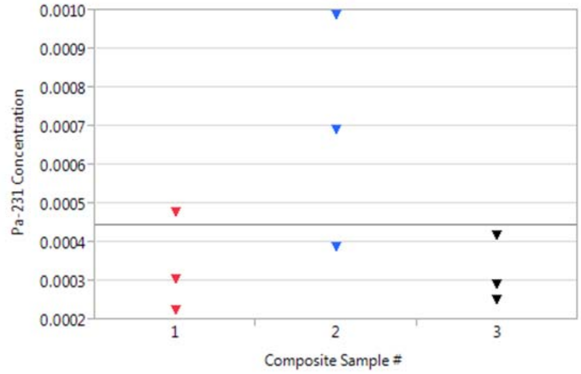
Np-237 (μCi/g)

All Results (circles) above MDCs



Pa-231 (μCi/g)

All Results (triangles) below MDCs



Means and Standard Deviations (Np-237)

Sample	Number	Mean	Std Dev	Std Err Mean
1	3	1.817E-3	1.5275e-5	8.819e-6
2	3	2.583E-3	8.0208e-5	4.631e-5
3	3	1.513E-3	5.7735e-5	3.333e-5
Pooled	9	1.971E-3	5.7735e-5	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	0.9753	2	6	0.4298
Brown-Forsythe	0.7471	2	6	0.5132
Levene	2.1667	2	6	0.1958
Bartlett	1.6378	2		0.1944

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	1.82469E-6	9.123E-7	273.703	<.0001
Error	6	2.00000E-8	3.333E-9		
C. Total	8	1.84469E-6			

Statistically significant sampling variance.

Diagnostics for Sampling Model Residuals

Wilk-Shapiro	0.953 > 0.829 crit. value->SNS
Dixon High Outlier	Run 2 of Sample 2 -> SNS
Dixon Low Outlier	Run 3 of Sample 2 -> SNS

Variance Components

Component	Variance Comp	Std Dev
Composite Samples	3.030037E-7	5.50458E-4
Measurements	3.333333E-9	5.77350E-5
Total	3.063370E-7	5.53477E-4
Mean Concentration	1.013716E-7	3.18389E-4

Student's t UCL95

UCL95	2.90080E-3

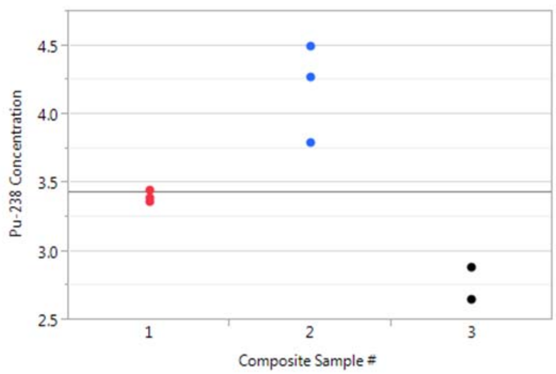
Minimum Detection Concentrations (MDCs) (Pa-231)

Sample	Number	Minimum MDC	Maximum MDC
1	3	2.22E-4	4.77E-4
2	3	3.86E-4	9.86E-4
3	3	2.49E-4	4.15E-4
Overall	9	2.22E-4	9.86E-4

Appendix F: Statistical Analysis of the Tank 16-H Annulus Samples

Table F19. Support Results for Radionuclides

Pu-238 (μCi/g)
All Results (circles) above MDCs



Means and Standard Deviations

Sample	Number	Mean	Std Dev	Std Err Mean
1	3	3.40000	0.045826	0.026458
2	3	4.18667	0.362261	0.209152
3	3	2.72667	0.132791	0.076667
Pooled	9	3.43778	0.224326	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	1.5115	2	6	0.2940
Brown-Forsythe	1.3882	2	6	0.3195
Levene	4.4105	2	6	0.0663
Bartlett	2.6727	2		0.0691

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	3.2038222	1.60191	31.8331	0.0006
Error	6	0.3019333	0.05032		
C. Total	8	3.5057556			

Statistically significant sampling variance.

Diagnostics for Sampling Model Residuals

Wilk-Shapiro	0.940 > 0.829 crit. value->SNS
Dixon High Outlier	Run 3 of Sample 2 -> SNS
Dixon Low Outlier	Run 2 of Sample 2 -> SS

Variance Components

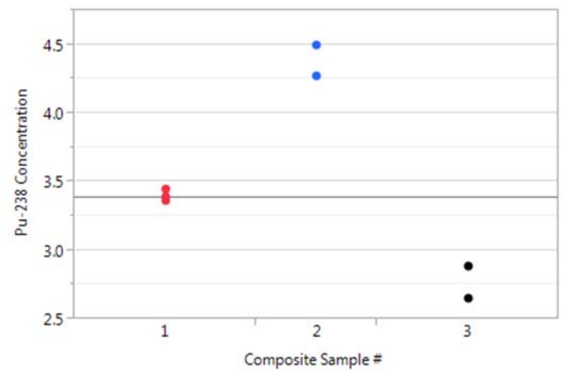
Component	Variance Comp	Std Dev
Composite Samples	0.517196	0.71916
Measurements	0.0503222	0.22433
Total	0.567519	0.75334
Mean Concentration	0.177990	0.42189

Student's t UCL95

UCL95	4.66969

Repeat analysis without potential outlier at Sample 2 Run 2: 3.79 □Ci/g.

Pu-238 (μCi/g)
All Results (circles) above MDCs
Omitted Sample 2 Run 2: 3.79 μCi/g



Means and Standard Deviations

Sample	Number	Mean	Std Dev	Std Err Mean
1	3	3.4000	0.04583	0.0265
2	2	4.3850	0.16263	0.1150
3	3	2.7267	0.13279	0.0767
Pooled	8	3.3938	0.11482	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	1.3603	1	4	0.3083
Brown-Forsythe	0.6050	2	5	0.5817
Levene	5.4666	2	5	0.0552
Bartlett	0.9216	2		0.3979

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	3.3002708	1.65014	125.168	<.0001
Error	5	0.0659167	0.01318		
C. Total	7	3.3661875			

Statistically significant sampling variance.

Diagnostics for Sampling Model Residuals

Wilk-Shapiro	0.924 > 0.818 crit. value->SNS
Dixon High Outlier	Run 1 of Sample 3 -> SNS
Dixon Low Outlier	Run 1 of Sample 2 -> SNS

Variance Components

Component	Variance Comp	Std Dev
Composite Samples	0.6894960	0.830359
Measurements	0.0131871	0.114835
Total	0.7026831	0.838262
Mean Concentration	0.2312972	0.480934

REML Mean	3.50296

Student's t UCL95

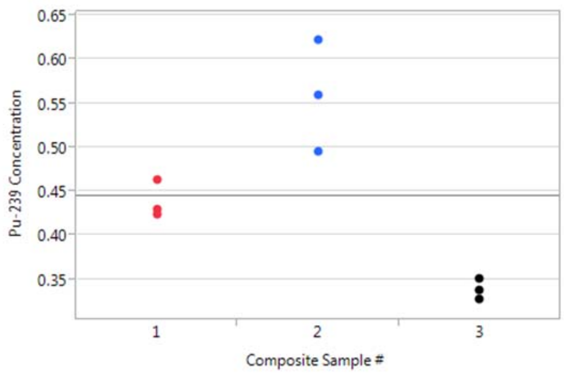
UCL95	4.90939

Appendix F: Statistical Analysis of the Tank 16-H Annulus Samples

Table F19. Support Results for Radionuclides

Pu-239 (μCi/g)

All Results (circles) above MDCs



Means and Standard Deviations

Sample	Number	Mean	Std Dev	Std Err Mean
1	3	0.4393	0.021572	0.012454
2	3	0.5587	0.063501	0.036662
3	3	0.3390	0.011533	0.006658
Pooled	9	0.4457	0.039288	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	1.5111	2	6	0.2941
Brown-Forsythe	1.8095	2	6	0.2427
Levene	2.0330	2	6	0.2118
Bartlett	2.2012	2		0.1107

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	0.07256067	0.036280	23.504	0.0015
Error	6	0.00926133	0.001544		
C. Total	8	0.08182200			

Statistically significant sampling variance.

Diagnostics for Sampling Model Residuals

Wilk-Shapiro	0.938 > 0.829 crit. value->SNS
Dixon High Outlier	Run 3 of Sample 2 -> SNS
Dixon Low Outlier	Run 2 of Sample 2 -> SS

Variance Components

Component	Variance Comp	Std Dev
Composite Samples	1.15789E-02	1.0761E-01
Measurements	1.54356E-03	3.9288E-02
Total	1.31225E-02	1.1455E-01
Mean Concentration	4.03115E-03	6.3491E-02

Student's t UCL95

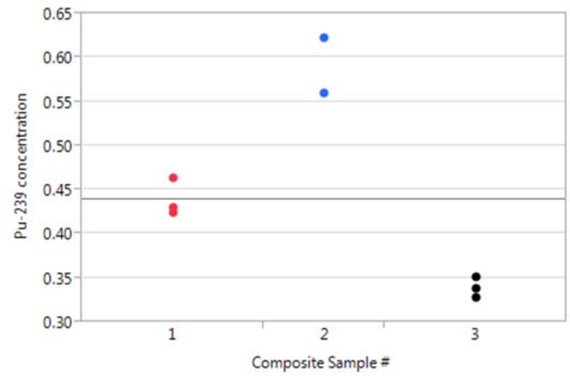
UCL95	0.63106

Repeat analysis without potential outlier at Composite Sample 2 Run 2: 0.495 □Ci/g.

Pu-239 (μCi/g)

All Results (circles) above MDCs

Omitted Sample 2 Run 2: 0.495 μCi/g



Means and Standard Deviations

Sample	Number	Mean	Std Dev	Std Err Mean
1	3	0.4393	0.02157	0.0125
2	2	0.5905	0.04455	0.0315
3	3	0.3390	0.01153	0.0067
Pooled	8	0.4395	0.02522	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	0.8383	1	4	0.4117
Brown-Forsythe	2.3614	2	5	0.1897
Levene	8.5882	2	5	0.0241
Bartlett	1.0071	2		0.3653

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	7.59028E-2	3.7951E-2	59.65	0.0003
Error	5	3.18117E-3	6.3623E-4		
C. Total	7	7.9084E-2			

Statistically significant sampling variance.

Diagnostics for Sampling Model Residuals

Wilk-Shapiro	0.960 > 0.818 crit. value->SNS
Dixon High Outlier	Run 3 of Sample 2 -> SNS
Dixon Low Outlier	Run 1 of Sample 2 -> SNS

Variance Components

Component	Variance Comp	Std Dev
Composite Samples	1.573231E-2	1.25429E-01
Measurements	6.366199E-4	2.52313E-02
Total	1.636893E-2	1.27941E-01
Mean Concentration	5.314839E-3	7.29029E-02

REML Mean	0.455981

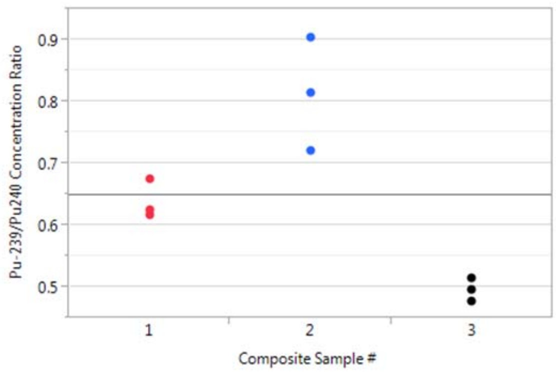
Student's t UCL95

UCL95	0.66954

Appendix F: Statistical Analysis of the Tank 16-H Annulus Samples

Table F19. Support Results for Radionuclides

Pu-239/Pu-240 (dimensionless)
All Results (circles) above MDCs



Means and Standard Deviations

Sample	Number	Mean	Std Dev	Std Err Mean
1	3	0.6397	0.031786	0.018352
2	3	0.8137	0.092007	0.053120
3	3	0.4953	0.018502	0.010682
Pooled	9	0.6495	0.057207	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	1.4897	2	6	0.2983
Brown-Forsythe	1.7223	2	6	0.2564
Levene	2.0042	2	6	0.2155
Bartlett	2.0312	2		0.1312

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	0.15244422	0.076222	23.2905	0.0015
Error	6	0.01963600	0.003273		
C. Total	8	0.17208022			

Statistically significant sampling variance.

Diagnostics for Sampling Model Residuals

Wilk-Shapiro	0.945 > 0.829 crit. value->SNS
Dixon High Outlier	Run 3 of Sample 2 -> SNS
Dixon Low Outlier	Run 2 of Sample 2 -> SS

Variance Components

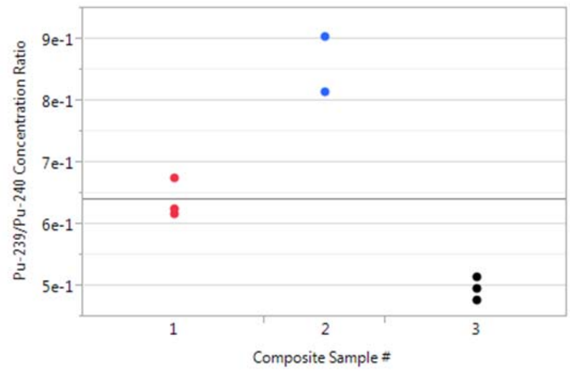
Component	Variance Comp	Std Dev
Composite Samples	2.43165E-02	1.5594E-01
Measurements	3.27267E-03	5.7207E-02
Total	2.75891E-02	1.6610E-01
Mean Concentration	8.46912E-03	9.2028E-02

Student's t UCL95

UCL95	0.91828

Repeat analysis without potential outlier at Sample 2 Run 2 (0.721).

Pu-239/Pu-240 (dimensionless)
All Results (circles) above MDCs
Omitted Sample 2 Run 2: 0.721 μCi/g



Means and Standard Deviations

Sample	Number	Mean	Std Dev	Std Err Mean
1	3	0.6397	0.03179	0.01835
2	2	0.8600	0.06364	0.04500
3	3	0.4953	0.01850	0.01068
Pooled	8	0.6406	0.03676	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	0.6971	1	4	0.4507
Brown-Forsythe	2.0344	2	5	0.2257
Levene	6.6311	2	5	0.0392
Bartlett	0.8574	2		0.4243

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	0.1595825	0.079791	59.0580	0.0003
Error	5	0.0067553	0.001351		
C. Total	7	0.1663379			

Statistically significant sampling variance.

Diagnostics for Sampling Model Residuals

Wilk-Shapiro	0.955 > 0.818 crit. value->SNS
Dixon High Outlier	Run 3 of Sample 2 -> SNS
Dixon Low Outlier	Run 1 of Sample 2 -> SNS

Variance Components

Component	Variance Comp	Std Dev
Composite Samples	3.30972E-02	1.8193E-01
Measurements	1.35190E-03	3.6768E-02
Total	3.44491E-02	1.8560E-01
Mean Concentration	1.11826E-02	1.0575E-01

REML Mean	0.664565

Student's t UCL95

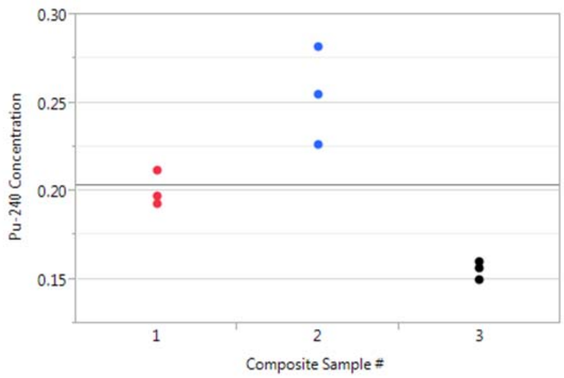
UCL95	0.97436

Appendix F: Statistical Analysis of the Tank 16-H Annulus Samples

Table F19. Support Results for Radionuclides

Pu-240 (μCi/g) mixed

All Results (circles) above MDCs



Means and Standard Deviations

Sample	Number	Mean	Std Dev	Std Err Mean
1	3	0.200667	0.010017	0.005783
2	3	0.254333	0.028006	0.016169
3	3	0.155333	0.005033	0.002906
Pooled	9	0.203444	0.017416	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	1.4906	2	6	0.2982
Brown-Forsythe	1.7902	2	6	0.2456
Levene	2.1069	2	6	0.2027
Bartlett	2.1592	2		0.1154

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	0.01473622	0.007368	24.291	0.0013
Error	6	0.00182000	0.000303		
C. Total	8	0.01655622			

Statistically significant sampling variance.

Diagnostics for Sampling Model Residuals

Wilk-Shapiro	0.947 > 0.829 crit. value->SNS
Dixon High Outlier	Run 3 of Sample 2 -> SNS
Dixon Low Outlier	Run 2 of Sample 2 -> SS

Variance Components

Component	Variance Comp	Std Dev
Composite Samples	2.35493E-03	4.8528E-02
Measurements	3.03333E-04	1.7416E-02
Total	2.65826E-03	5.1558E-02
Mean Concentration	8.18679E-04	2.8613E-02

Student's t UCL95

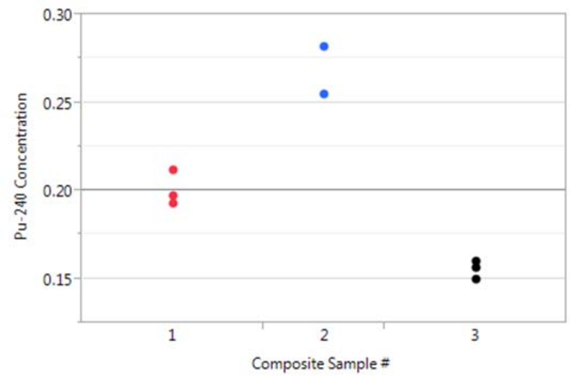
UCL95	0.2869927

Repeat analysis without potential outlier at Sample 2 Run 2 (0.226 μCi/g).

Pu-240 (μCi/g) mixed

All Results (circles) above MDCs

Omitted Sample 2 Run 2: 0.226 μCi/g



Means and Standard Deviations

Sample	Number	Mean	Std Dev	Std Err Mean
1	3	0.200667	0.010017	0.00578
2	2	0.268500	0.019092	0.01350
3	3	0.155333	0.005033	0.00291
Pooled	8	0.200625	0.011098	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	0.9338	1	4	0.3886
Brown-Forsythe	2.2638	2	5	0.1995
Levene	7.0389	2	5	0.0352
Bartlett	0.9569	2		0.3841

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	0.01536804	0.007684	62.387	0.0003
Error	5	0.00061583	0.000123		
C. Total	7	0.01598387			

Statistically significant sampling variance.

Diagnostics for Sampling Model Residuals

Wilk-Shapiro	0.962 > 0.818 crit. value->SNS
Dixon High Outlier	Run 3 of Sample 2 -> SNS
Dixon Low Outlier	Run 1 of Sample 2 -> SNS

Variance Components

Component	Variance Comp	Std Dev
Composite Samples	3.18658E-3	5.6450E-02
Measurements	1.23238E-4	1.1101E-02
Total	3.30982E-3	5.7531E-02
Mean Concentration	1.07589E-03	3.2801E-02

REML Mean	0.20804

Student's t UCL95

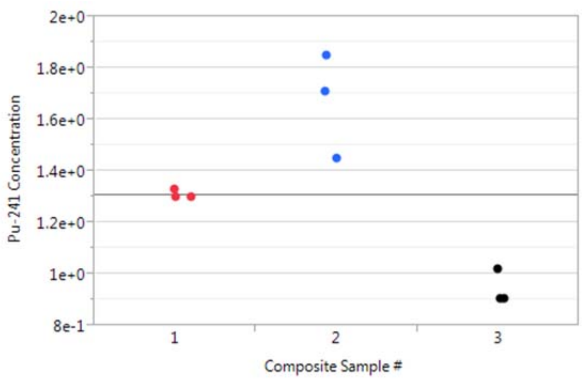
UCL95	0.304111

Appendix F: Statistical Analysis of the Tank 16-H Annulus Samples

Table F19. Support Results for Radionuclides

Pu-241 (μCi/g) below

All Results (circles) above MDCs



Means and Standard Deviations

Sample	Number	Mean	Std Dev	Std Err Mean
1	3	1.31000	0.017321	0.010000
2	3	1.67000	0.202978	0.117189
3	3	0.94333	0.066395	0.038333
Pooled	9	1.30778	0.123704	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	1.5756	2	6	0.2819
Brown-Forsythe	1.7355	2	6	0.2543
Levene	4.4947	2	6	0.0641
Bartlett	3.4250	2		

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	0.7920889	0.396044	25.8806	0.0011
Error	6	0.0918167	0.015303		
C. Total	8	0.8839056			

Statistically significant sampling variance.

Diagnostics for Sampling Model Residuals

Wilk-Shapiro	0.923 > 0.829 crit. value->SNS
Dixon High Outlier	Run 1 of Sample 2 -> SNS
Dixon Low Outlier	Run 2 of Sample 2 -> SS

Variance Components

Component	Variance Comp	Std Dev
Composite Samples	0.1269139	0.35625
Measurements	0.0153028	0.12370
Total	0.1422167	0.37712
Mean Concentration	0.0440049	0.20977

Student's t UCL95

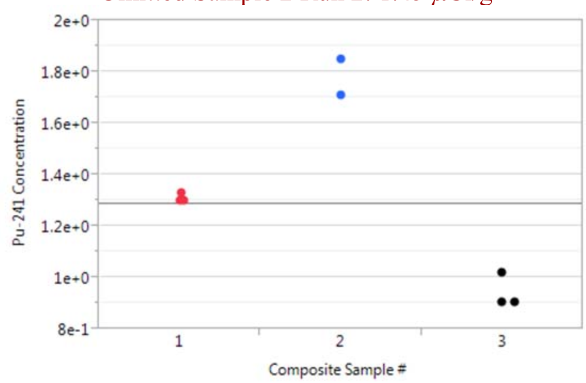
UCL95	1.920313

Repeat analysis without potential outlier at Sample 2 Run 2: 1.45 □Ci/g.

Pu-241 (μCi/g) below

All Results (circles) above MDCs

Omitted Sample 2 Run 2: 1.45 μCi/g



Means and Standard Deviations

Sample	Number	Mean	Std Dev	Std Err Mean
1	3	1.31000	0.017321	0.01000
2	2	1.78000	0.098995	0.07000
3	3	0.94333	0.066395	0.03833
Pooled	8	1.29000	0.061995	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	1.5369	1	4	0.2828
Brown-Forsythe	1.1563	2	5	0.3866
Levene	10.2301	2	5	0.0171
Bartlett	1.4963	2		

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance

Source	DF	SS	MS	F Ratio	Prob >
Sample	2	0.84193333	0.420967	109.532	<.0001
Error	5	0.01921667	0.003843		
C. Total	7	0.86115000			

Statistically significant sampling variance.

Diagnostics for Sampling Model Residuals

Wilk-Shapiro	0.923 > 0.818 crit. value->SNS
Dixon High Outlier	Run 1 of Sample 3 -> SNS
Dixon Low Outlier	Run 3 of Sample 2 -> SNS

Variance Components

Component	Variance Comp	Std Dev
Composite Samples	0.174133725	0.41729
Measurements	0.003844488	0.06200
Total	0.177978213	0.42187
Mean Concentration	0.058471740	0.24181

REML Mean	1.34392

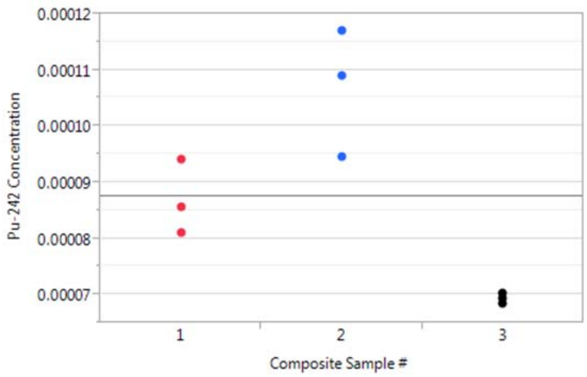
Student's t UCL95

UCL95	2.051156

Appendix F: Statistical Analysis of the Tank 16-H Annulus Samples

Table F19. Support Results for Radionuclides

Pu-242 (μCi/g)
All Results (circles) above MDCs



Means and Standard Deviations

Sample	Number	Mean	Std Dev	Std Err Mean
1	3	8.693e-5	6.6018e-6	3.8115e-6
2	3	1.069e-4	1.1351e-5	6.5537e-6
3	3	6.940e-5	9.0000e-7	5.1962e-7
Pooled	9	8.773e-5	7.5993e-6	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	1.2248	2	6	0.3581
Brown-Forsythe	1.5107	2	6	0.2942
Levene	3.3984	2	6	0.1031
Bartlett	3.0647	2		0.0467

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	2.10851e-9	1.054e-9	18.256	0.0028
Error	6	3.4649e-10	5.77e-11		
C. Total	8	2.45500e-9			

Statistically significant sampling variance.

Diagnostics for Sampling Model Residuals

Wilk-Shapiro	0.962 > 0.829 crit. value->SNS
Dixon High Outlier	Run 3 of Sample 2 -> SNS
Dixon Low Outlier	Run 2 of Sample 2 -> SNS

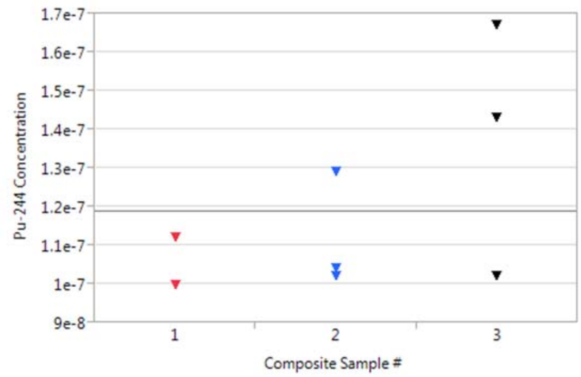
Variance Components

Component	Variance Comp	Std Dev
Composite Samples	3.32168E-10	1.8225E-05
Measurements	5.77489E-11	7.5993E-06
Total	3.89917E-10	1.9746E-05
Mean Concentration	1.17139E-10	1.0823E-05

Student's t UCL95

UCL95	1.19337E-4

Pu-244 (μCi/g)
All Results (triangles) below MDCs



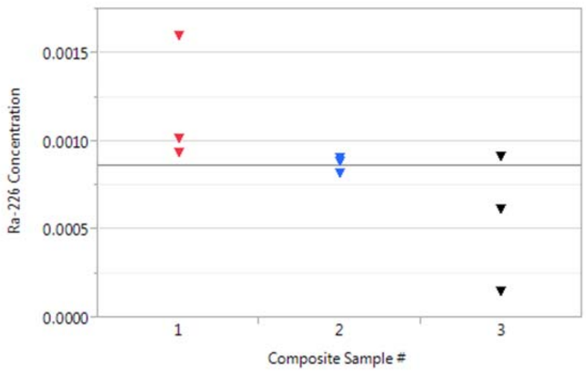
Minimum Detection Concentrations (MDCs)

Sample	Number	Minimum MDC	Maximum MDC
1	3	9.95e-8	1.12e-7
2	3	1.02e-7	1.29e-7
3	3	1.02e-7	1.67e-7
Overall	9	9.95e-8	1.67e-7

Appendix F: Statistical Analysis of the Tank 16-H Annulus Samples

Table F19. Support Results for Radionuclides

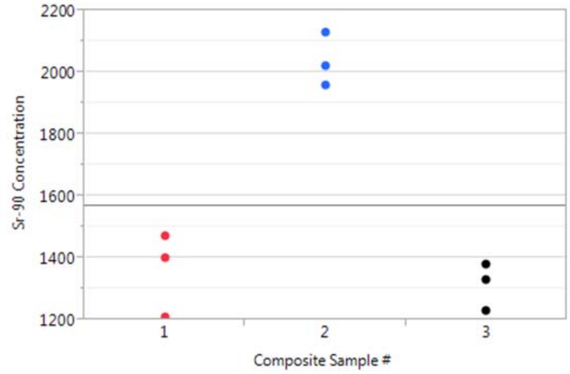
Ra-226 (μCi/g)
All Results (triangles) below MDCs



Minimum Detection Concentrations (MDCs)

Sample	Number	Minimum MDC	Maximum MDC
1	3	9.32E-4	1.60E-3
2	3	8.15E-4	9.01E-4
3	3	1.49E-4	9.10E-4
Overall	9	1.49E-4	1.60E-3

Sr-90 (μCi/g)
All Results (circles) above MDCs



Means and Standard Deviations

Sample	Number	Mean	Std Dev	Std Err Mean
1	3	1360.000	134.5362	77.6745347
2	3	2036.667	86.21678	49.7772817
3	3	1313.333	76.37626	44.0958552
Pooled	9	1570.000	102.2524	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	0.5689	2	6	0.5940
Brown-Forsythe	0.2325	2	6	0.7994
Levene	0.8720	2	6	0.4651
Bartlett	0.3076	2		0.7352

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	983266.667	491633	47.0213	0.0002
Error	6	62733.3333	10456		
C. Total	8	1046000.00			

Statistically significant sampling variance.

Diagnostics for Sampling Model Residuals

Wilk-Shapiro	0.950 > 0.829 crit. value->SNS
Dixon High Outlier	Run 2 of Sample 1 -> SNS
Dixon Low Outlier	Run 3 of Sample 1 -> SNS

Variance Components

Component	Variance Comp	Std Dev
Composite Samples	1.603926E+5	4.00490E+2
Measurements	1.045556E+4	1.02252E+2
Total	1.708482E+5	4.13338E+2
Mean Concentration	5.462593E+4	2.33722E+2

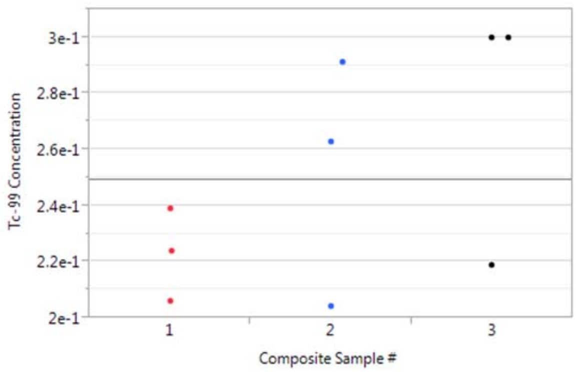
Student's t UCL95

UCL95	2252.465

Appendix F: Statistical Analysis of the Tank 16-H Annulus Samples

Table F19. Support Results for Radionuclides

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



Means and Standard Deviations

Sample	N	Mean	Std Dev	Std Err Mean
1	3	0.2230	1.6523E-2	9.5394E-3
2	3	0.2527	4.4411E-2	2.5641E-2
3	3	0.2730	4.6765E-2	2.7000E-2
Overall	9	0.2496	3.9778E-2	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	0.6704	2	6	0.5460
Brown-Forsythe	0.2780	2	6	0.7665
Levene	2.2326	2	6	0.1885
Bartlett	0.8243	2		0.4385

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	3.7936E-3	1.8968E-3	1.2838	0.3435
Error	6	8.8647E-3	1.4774E-3		
C. Total	8	1.2658E-2			

Statistically non-significant sampling variance.

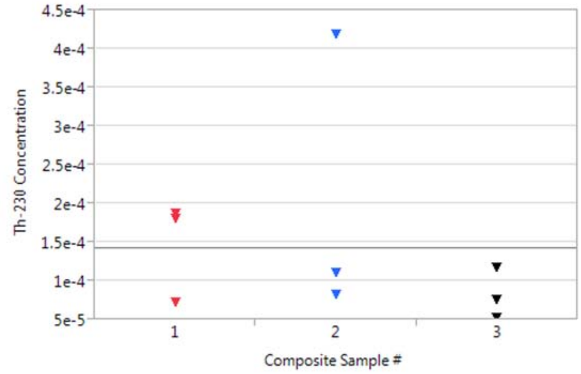
Diagnostics for Concentrations

Wilk-Shapiro	0.870 > 0.829 crit. value->SNS
Dixon High Outlier	Runs 2 & 3 of Sample 3 -> SNS
Dixon Low Outlier	Run 1 of Sample 2 -> SNS

Normal Statistics

Component	Estimate
Mean	0.249556
Std.Dev.	0.039778
Student's t UCL95	0.274212

Th-230 (μCi/g)
All Results (triangles) below MDCs



Minimum Detection Concentrations (MDCs)

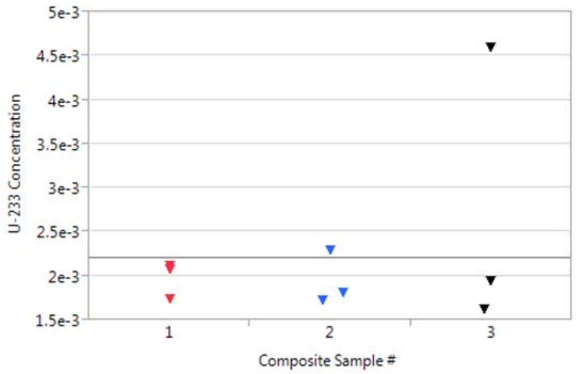
Sample	N	Minimum MDC	Maximum MDC
1	3	7.21E-5	1.86E-4
2	3	8.11E-5	4.18E-4
3	3	5.23E-5	1.16E-4
Overall	9	5.23E-5	4.18E-4

Appendix F: Statistical Analysis of the Tank 16-H Annulus Samples

Table F19. Support Results for Radionuclides

U-233 (μCi/g)

All Results (triangles) below MDCs

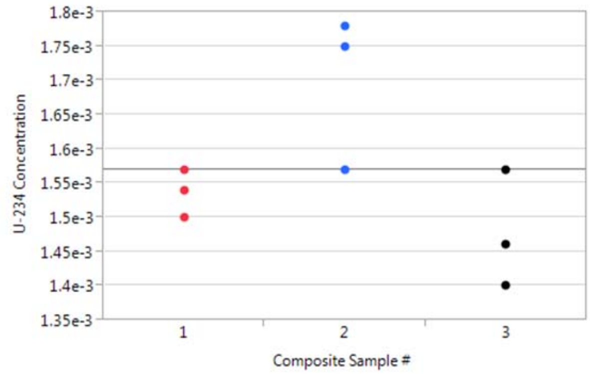


Minimum Detection Concentrations (MDCs)

Sample	N	Minimum MDC	Maximum MDC
1	3	1.73E-3	2.10E-3
2	3	1.72E-3	2.29E-3
3	3	1.61E-3	4.59E-3
Overall	9	1.61E-3	4.59E-3

U-234 (μCi/g)

All Results (circles) above MDCs



Means and Standard Deviations

Sample	N	Mean	Std Dev	Std Err Mean
1	3	1.5367E-3	3.5119E-5	2.0276E-5
2	3	1.7000E-3	1.1358E-4	6.5574E-5
3	3	1.4767E-3	8.6217E-5	4.9777E-5
Pooled	9	1.5711E-3	8.4787E-5	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	0.8142	2	6	0.4866
Brown-Forsythe	0.4073	2	6	0.6825
Levene	2.4593	2	6	0.1659
Bartlett	0.9366	2		0.3920

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	8.01556e-8	4.008e-8	5.5750	0.0428
Error	6	4.31333e-8	7.189e-9		
C. Total	8	1.23289e-7			

Statistically significant sampling variance.

Diagnostics for Sampling Model Residuals

Wilk-Shapiro	0.967 > 0.829 crit. value->SNS
Dixon High Outlier	Run 1 of Sample 3 -> SNS
Dixon Low Outlier	Run 2 of Sample 2 -> SNS

Variance Components

Component	Variance Comp	Std Dev
Composite Samples	1.096296e-8	1.0470E-04
Measurements	7.188889e-9	8.4787E-05
Total	1.815185e-8	1.3473E-04
Mean Concentration	4.45309E-09	6.6732E-05

Student's t UCL95

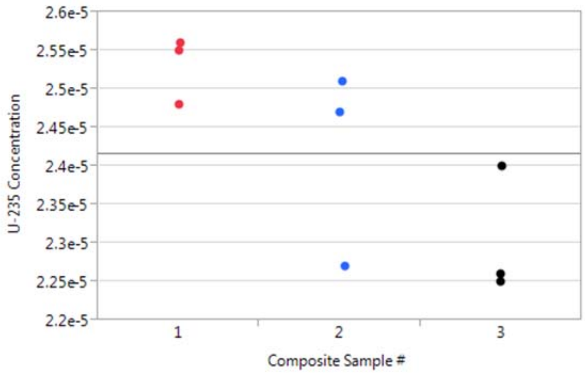
UCL95	0.001766

Appendix F: Statistical Analysis of the Tank 16-H Annulus Samples

Table F19. Support Results for Radionuclides

U-235 (μCi/g)

All Results (circles) above MDCs



Means and Standard Deviations

Sample	N	Mean	Std Dev	Std Err Mean
1	3	2.5300E-5	4.3589E-7	2.5166E-7
2	3	2.4167E-5	1.2858E-6	7.4237E-7
3	3	2.3033E-5	8.3865E-7	4.8419E-7
Pooled	9	2.4167E-5	9.2135E-7	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	0.9006	2	6	0.4550
Brown-Forsythe	0.3434	2	6	0.7224
Levene	2.8945	2	6	0.1318
Bartlett	0.8332	2		0.4346

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	7.7067e-12	3.85e-12	4.5393	0.0630
Error	6	5.0933e-12	8.49e-13		
C. Total	8	1.2800e-11			

Statistically non-significant sampling variance.

Diagnostics for Concentrations

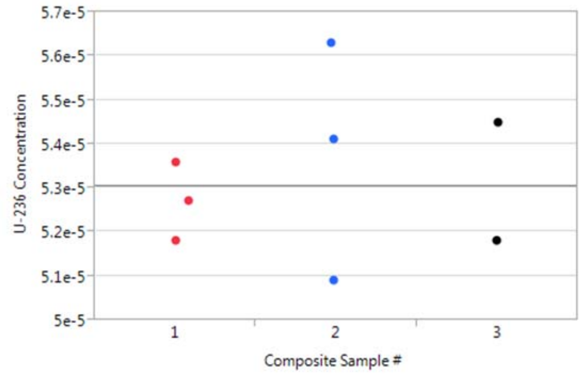
Wilk-Shapiro	0.859 > 0.829 crit. value->SNS
Dixon High Outlier	Runs 2 of Sample 1 -> SNS
Dixon Low Outlier	Run 3 of Sample 3 -> SNS

Normal Statistics

Component	Estimate
Mean	2.4167E-5
Std.Dev.	1.2649E-6
Student's t UCL95	2.4951E-5

U-236 (μCi/g)

All Results (circles) above MDCs



Means and Standard Deviations

Sample	N	Mean	Std Dev	Std Err Mean
1	3	5.270E-5	9.0000E-7	5.1962E-7
2	3	5.377E-5	2.7154E-6	1.5677E-6
3	3	5.270E-5	1.5589E-6	9.0000E-7
Pooled	9	5.306E-5	1.8809E-6	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	1.0233	2	6	0.4146
Brown-Forsythe	0.6524	2	6	0.5541
Levene	1.5923	2	6	0.2788
Bartlett	0.9126	2		0.4015

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	2.2756e-12	1.14e-12	0.3216	0.7367
Error	6	2.1227e-11	3.54e-12		
C. Total	8	2.3502e-11			

Statistically non-significant sampling variance.

Diagnostics for Concentrations

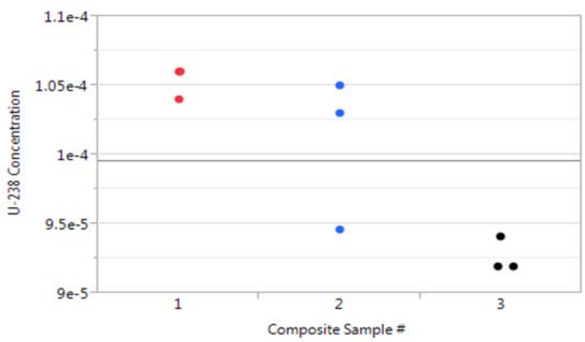
Wilk-Shapiro	0.932 > 0.829 crit. value->SNS
Dixon High Outlier	Runs 3 of Sample 2 -> SNS
Dixon Low Outlier	Run 2 of Sample 2 -> SNS

Normal Statistics

Component	Estimate
Mean	5.3056E-5
Std.Dev.	1.7140E-6
Student's t UCL95	5.4118E-5

Appendix F: Statistical Analysis of the Tank 16-H Annulus Samples
Table F19. Support Results for Radionuclides

U-238 (μCi/g)
All Results (circles) above MDCs



Means and Standard Deviations

Sample	Number	Mean	Std Dev	Std Err Mean
1	3	1.0533E-4	1.1547E-6	6.6667E-7
2	3	1.0087E-4	5.5185E-6	3.1861E-6
3	3	9.2633E-5	1.2702E-6	7.3333E-7
Pooled	9	9.9611E-5	3.3367E-6	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	1.6025	2	6	0.2769
Brown-Forsythe	1.0348	2	6	0.4110
Levene	6.8689	2	6	0.0281
Bartlett	2.4935	2		0.0826

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	2.4903e-10	1.25e-10	11.184	0.0095
Error	6	6.6800e-11	1.11e-11		
C. Total	8	3.1583e-10			

Statistically significant sampling variance.

Diagnostics for Sampling Model Residuals

Wilk-Shapiro	0.914 > 0.829 crit. value->SNS
Dixon High Outlier	Run 3 of Sample 2 -> SNS
Dixon Low Outlier	Run 2 of Sample 2 -> SS

Variance Components

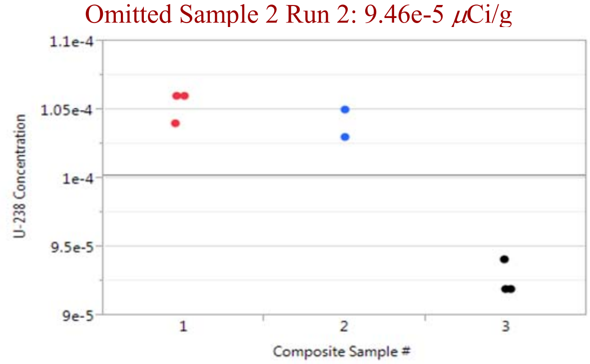
Component	Variance Comp	Std Dev
Composite Samples	3.77937e-11	6.1477E-06
Measurements	1.11333e-11	3.3367E-06
Total	4.89270e-11	6.9948E-06
Mean Concentration	1.38349E-11	3.7195E-06

Student's t UCL95

UCL95	1.104721E-4

Repeat analysis without potential outlier at Sample 2 Run 2 (9.46e-5μCi/g).

U-238 (μCi/g)
All Results (circles) above MDCs
Omitted Sample 2 Run 2: 9.46e-5 μCi/g



Means and Standard Deviations

Sample	Number	Mean	Std Dev	Std Err Mean
1	3	1.0533E-4	1.1547E-6	6.6667E-7
2	2	1.0400E-4	1.4142E-6	1.0000E-6
3	3	9.2633E-5	1.2702E-6	7.3333E-7
Pooled	8	1.0024E-4	1.2565E-6	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	0.0318	1	4	0.8671
Brown-Forsythe	0.0601	2	5	0.9423
Levene	0.0707	2	5	0.9327
Bartlett	0.0222	2		0.9780

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	2.797E-10	1.398E-10	88.58	0.0001
Error	5	7.893E-12	1.579E-12		
C. Total	7	2.876E-10			

Statistically significant sampling variance.

Diagnostics for Sampling Model Residuals

Wilk-Shapiro	0.890 > 0.818 crit. value->SNS
Dixon High Outlier	Run 1 of Sample 3 -> SNS
Dixon Low Outlier	Run 3 of Sample 1 -> SNS

Variance Components

Component	Variance Comp	Std Dev
Composite Samples	4.821282e-11	6.9435e-6
Measurements	1.577924e-12	1.2562e-6
Total	4.979074e-11	7.0563e-6
Mean Concentration	1.627538e-11	4.0343e-6

REML Mean	1.00650E-4

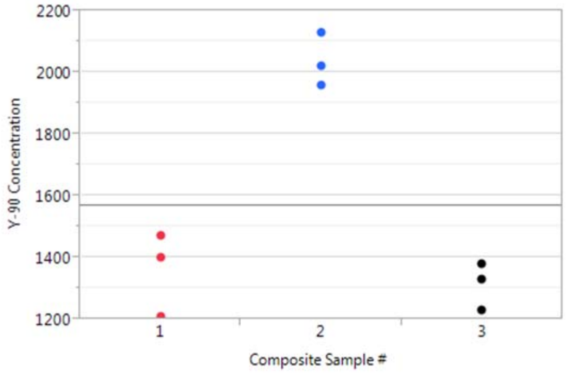
Student's t UCL95

UCL95	1.124109E-4

Appendix F: Statistical Analysis of the Tank 16-H Annulus Samples

Table F19. Support Results for Radionuclides

Y-90 (μCi/g)
All Results (circles) above MDCs



Means and Standard Deviations

Sample	Number	Mean	Std Dev	Std Err Mean
1	3	1360.000	134.5362	77.6745347
2	3	2036.667	86.21678	49.7772817
3	3	1313.333	76.37626	44.0958552
Pooled	9	1570.000	102.2524	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	0.5689	2	6	0.5940
Brown-Forsythe	0.2325	2	6	0.7994
Levene	0.8720	2	6	0.4651
Bartlett	0.3076	2		0.7352

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	983266.667	491633	47.0213	0.0002
Error	6	62733.3333	10456		
C. Total	8	1046000.00			

Statistically significant sampling variance.

Diagnostics for Sampling Model Residuals

Wilk-Shapiro	0.950 > 0.829 crit. value->SNS
Dixon High Outlier	Run 2 of Sample 1 -> SNS
Dixon Low Outlier	Run 3 of Sample 1 -> SNS

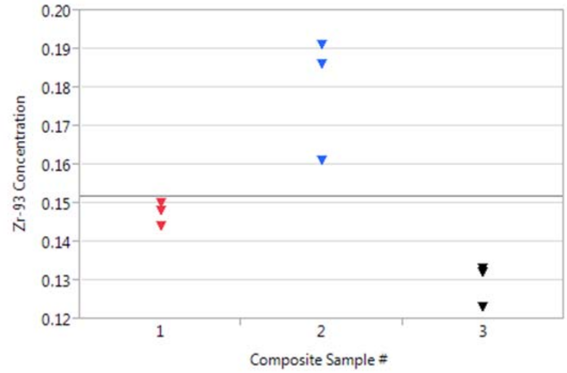
Variance Components

Component	Variance Comp	Std Dev
Composite Samples	1.603926e+5	4.0049e+2
Measurements	1.045556e+4	1.0225e+2
Total	1.708482e+5	4.1334e+2
Mean Concentration	5.462593e+4	2.3372e+2

Student's t UCL95

UCL95	2252.464574

Zr-93 (μCi/g)
All Results (triangles) below MDCs



Minimum Detection Concentrations (MDCs)

Sample	N	Minimum MDC	Maximum MDC
1	3	0.144	0.150
2	3	0.161	0.191
3	3	0.123	0.133
Overall	9	0.123	0.191

Appendix F: Statistical Analysis of the Tank 16-H Annulus Samples

Table F20. Summary for the Elemental Constituents with All Results below their MDCs

Elemental Constituent (wt %)	N	Smallest Minimum Detectable Concentration (μCi/g)♠		Largest Minimum Detectable Concentration (μCi/g)♠	
		Fixed Decimal Format	Scientific Format	Fixed Decimal Format	Scientific Format
Ag	9	0.00323	3.23e-3	0.00333	3.33e-3
As	9	0.000253	2.53e-4	0.000274	2.74e-4
B	9	0.0466	4.66e-2	0.048	4.80e-2
Cd	9	0.00335	3.35e-3	0.00345	3.45e-3
Co*	9	0.0277	2.77e-2	0.0587	5.87e-2
Mo	9	0.0358	3.58e-2	0.0368	3.68e-2
Ni	9	0.05	5.00e-2	0.054	5.40e-2
Pb	9	0.126	1.26e-1	0.13	1.30e-1
Sb	9	0.0694	6.94e-2	0.0715	7.15e-2
Sr*	9	0.00618	6.18e-3	0.00959	9.59e-3
U	9	0.233	2.33e-1	0.24	2.40e-1
Zn*	9	0.0447	4.47e-2	0.0629	6.29e-2

MDCs: Minimum Detectable Concentrations

Appendix F: Statistical Analysis of the Tank 16-H Annulus Samples

Table F21. Summary for the Elemental Constituents with All Results above their MDCs

Elemental Constituent (wt %)	N	Mean (wt %)	Std Dev (wt %)[♠]	% Std Dev	UCL95 (wt %)	Goodness-of-Fit/Confidence Limit Remarks[♦]
Al	9	6.2567	5.7099E-1	9.13%	7.1987	SNS-VH; SS-SV; SNS-WS; SS-OT (Run 2 Sample 2); Student's t Confidence Limit
	8	6.3024	5.1204E-1	8.12%	7.16292	Omitted Run 2 on Composite Sample 2: 5.56 wt %; SNS-VH; SS-SV; SNS-WS; SNS-OT; Student's t Confidence Limit
Ba	9	1.6011E-2	3.2786E-3	20.48%	2.1470E-2	SNS-VH; SS-SV; SNS-WS; SNS-OT; Student's t Confidence Limit
Cr	9	1.9367E-2	5.9306E-3	30.62%	2.9331E-2	SNS-VH; SS-SV; SNS-WS; SNS-OT; Student's t Confidence Limit
Cu	9	5.144E-2	6.6657E-2	129.58%	1.4829E-1	SNS-VH; SNS-SV; SS-WS; SS-OT (Run 1 on Composite Sample 1: 0.214 wt %; No discernable distribution; Use Chebyshev UCL95.
	8	3.1125E-2	2.8830E-2	92.63%	7.56E-2	Omitted Run 1 on Composite Sample 1: 0.214 wt %; SNS-VH; SNS-SV; SS-WS; SNS-OT; No discernable distribution; Use Chebyshev UCL95.
Fe	9	2.4089	5.3710E-1	22.30%	3.2984	SNS-VH; SS-SV; SNS-WS; SNS-OT; Student's t Confidence Limit
Hg	9	2.0378E-1	2.2932E-2	11.25%	2.4096E-1	SNS-VH; SS-SV; SNS-WS; SNS-OT; Student's t Confidence Limit
Mn	9	2.3844E-2	5.7575E-3	24.15%	3.3463E-2	SNS-VH; SS-SV; SNS-WS; SNS-OT; Student's t Confidence Limit
Na	9	1.5489E+1	2.4607	15.89%	1.9585E+1	SNS-VH; SS-SV; SNS-WS; SNS-OT; Student's t Confidence Limit
Si	9	1.3632E+1	5.3265	39.07%	2.2594E+1	SNS-VH; SS-SV; SNS-WS; SNS-OT; Student's t Confidence Limit

SS-VH/SNS-VH: Statistically significant/Statistically non-significant Levene's test of variance heterogeneity at a Bonferroni $\alpha = 0.05/9 = 0.0056$.

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 statistic for testing normality at $\alpha = 0.05$.
SS-LF/SNS-LF: Statistically significant/Statistically non-significant Lilliefors test statistic for testing normality at $\alpha = 0.05$.
SS-OT/SNS-OT: Statistically significant/Statistically non-significant) Dixon's test for outliers at $\alpha = 0.05$. The Dixon test depends on the assumption of a normal distribution.

♠ When the sampling variation among the composite samples is demonstrated to be statistically significant (SS-SV), the standard deviation (Std Dev) and the percent standard deviation (% Std Dev) account for the variation among the composite samples and the variation attributable to a single measurement of the composite sample: $StdDev: s_{Total} = \sqrt{s_{Samp}^2 + s_{Meas}^2}$.

Appendix F: Statistical Analysis of the Tank 16-H Annulus Samples

Table F22. Statistical Summary for the Elemental Constituents with a Mixture of Results above and below their MDCs

Elemental Constituent (wt %)	N◆	Mean (wt %)	Std Dev (wt %)	% Std Dev	UCL95 (wt %)	Goodness-of-Fit/Confidence Limit Remarks♥
Se	9 (3,3,0)	0.000475	0.000450	94.6%	0.00123	Largest possible Student's t UCL95; Refer to Se in Table F23 for details.

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

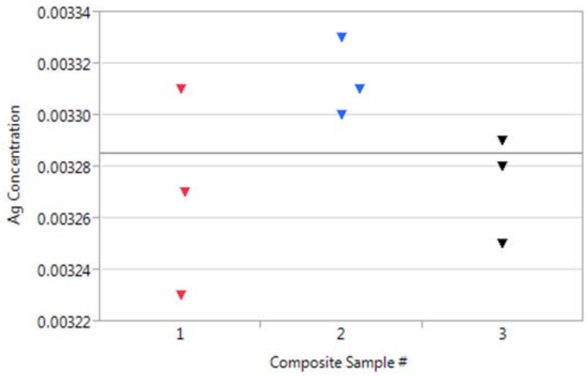
♥ Two of the three composite samples exhibit only results below MDCs for Se. Consequently, the concentration results for Se do not provide information on variability among the composite samples, only the measurement variability for Composite Sample 3. The reported values for the mean, standard deviation (Std Dev), percent standard deviation (% Std Dev), the one-sided upper 95% confidence limit for the mean (UCL95) are based on a method that produces the largest possible UCL95 consistent with the below MDC results.

Appendix F: Statistical Analysis of the Tank 16-H Annulus Samples

Table F23. Supporting Results for Elementals

Ag (*wt %*)

All Results (triangles) below MDCs



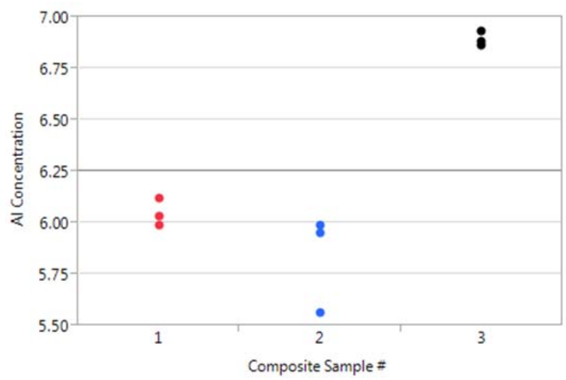
Minimum Detection Concentrations (MDCs)

Sample	Number	Minimum MDC	Maximum MDC
1	3	3.23E-3	3.31E-3
2	3	3.30E-3	3.33E-3
3	3	3.25E-3	3.29E-3
Overall	9	3.23E-3	3.33E-3

Appendix F: Statistical Analysis of the Tank 16-H Annulus Samples
Table F23. Supporting Results for Elementals

Al (*wt %*)

All Results (circles) above MDCs



Means and Standard Deviations

Sample	Number	Mean	Std Dev	Std Err Mean
1	3	6.0467	0.066583	0.038442
2	3	5.8333	0.237557	0.137154
3	3	6.8900	0.036056	0.020817
Pooled	9	6.2567	0.143952	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	1.5952	2	6	0.2783
Brown-Forsythe	0.7639	2	6	0.5064
Levene	8.2653	2	6	0.0189
Bartlett	2.7074	2		0.0667

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	1.8732667	0.936633	45.199	0.0002
Error	6	0.1243333	0.020722		
C. Total	8	1.9976000			

Statistically significant sampling variance.

Diagnostics for Sampling Model Residuals

Wilk-Shapiro	0.904 > 0.829 crit. value->SNS
Dixon High Outlier	Run 3 of Sample 2 -> SNS
Dixon Low Outlier	Run 2 of Sample 2 -> SS

Variance Components

Component	Variance Comp	Std Dev
Composite Samples	0.3053037	0.55254
Measurements	0.0207222	0.14395
Total	0.3260259	0.57099
Mean Concentration	0.1040704	0.32260

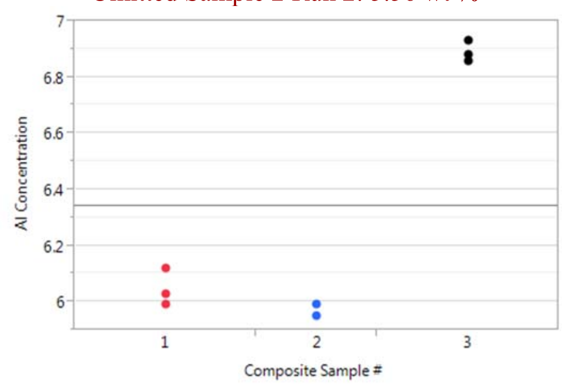
Student's t UCL95

UCL95	7.19865

Al (*wt %*)

All Results (circles) above MDCs

Omitted Sample 2 Run 2: 5.56 wt %



Means and Standard Deviations

Sample	Number	Mean	Std Dev	Std Err Mean
1	3	6.0467	0.066583	0.038442
2	2	5.9700	0.028284	0.020000
3	3	6.8900	0.036056	0.020817
Pooled	8	6.3438	0.049531	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	0.8177	1	4	0.4170
Brown-Forsythe	0.4063	2	5	0.6863
Levene	1.4041	2	5	0.3281
Bartlett	0.4644	2		0.6285

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	1.4393208	0.719660	293.34	<.0001
Error	5	0.0122667	0.002453		
C. Total	7	1.4515875			

Statistically significant sampling variance.

Diagnostics for Sampling Model Residuals

Wilk-Shapiro	0.954 > 0.818 crit. value->SNS
Dixon High Outlier	Run 1 of Sample 1 -> SNS
Dixon Low Outlier	Run 2 of Sample 1 -> SNS

Variance Components

Component	Variance Comp	Std Dev
Composite Samples	2.59727E-01	5.0963E-01
Measurements	2.45315E-03	4.9529E-02
Total	2.62180E-01	5.1204E-01
Mean Concentration	8.68482E-02	2.9470E-01

REML Mean	6.302396

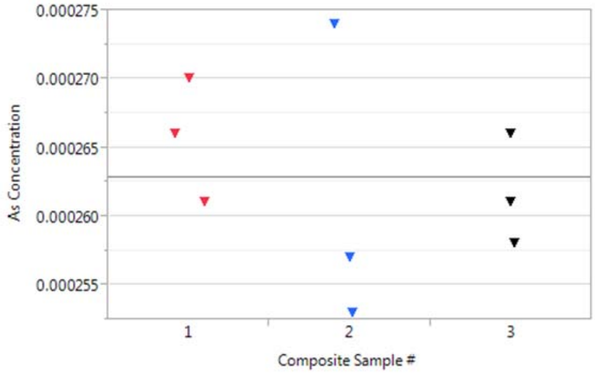
Student's t UCL95

UCL95	7.16292

Appendix F: Statistical Analysis of the Tank 16-H Annulus Samples

Table F23. Supporting Results for Elementals

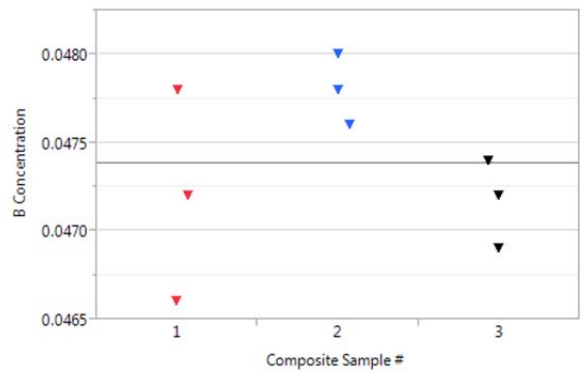
As (*wt %*)
All Results (triangles) below MDCs



Minimum Detection Concentrations (MDCs)

Sample	N	Minimum MDC	Maximum MDC
1	3	2.61E-4	2.70E-4
2	3	2.53E-4	2.74E-4
3	3	2.58E-4	2.66E-4
Overall	9	2.53E-4	2.74E-4

B (*wt %*)
All Results (triangles) below MDCs



Minimum Detection Concentrations (MDCs)

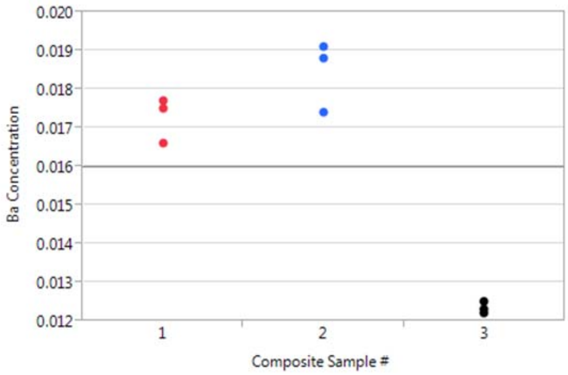
Sample	N	Minimum MDC	Maximum MDC
1	3	4.66E-2	4.78E-2
2	3	4.76E-2	4.80E-2
3	3	4.69E-2	4.74E-2
Overall	9	4.66E-2	4.80E-2

Appendix F: Statistical Analysis of the Tank 16-H Annulus Samples

Table F23. Supporting Results for Elementals

Ba (*wt %*)

All Results (circles) above MDCs



Means and Standard Deviations

Sample	N	Mean	Std Dev	Std Err Mean
1	3	0.01727	0.000586	0.0003383
2	3	0.01843	0.000907	0.0005239
3	3	0.01233	0.000153	0.0000882
Overall	9	0.01601	0.000630	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	1.0859	2	6	0.3958
Brown-Forsythe	0.6352	2	6	0.5621
Levene	4.6349	2	6	0.0607
Bartlett	1.8387	2		0.1590

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	0.00006291	0.000031	79.297	<.0001
Error	6	0.00000238	0.000000		
C. Total	8	0.00006529			

Statistically significant sampling variance.

Diagnostics for Sampling Model Residuals

Wilk-Shapiro	0.923 > 0.829 crit. value->SNS
Dixon High Outlier	Run 3 of Sample 2 -> SNS
Dixon Low Outlier	Run 2 of Sample 2 -> SNS

Variance Components

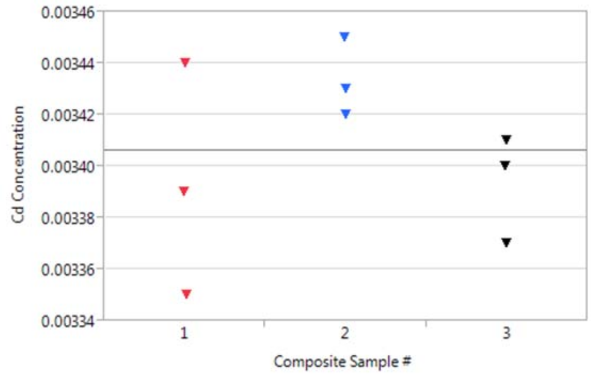
Component	Variance Comp	Std Dev
Composite Samples	1.035259E-5	3.21754E-3
Measurements	3.966667E-7	6.29815E-4
Total	1.074926E-5	3.27861E-3
Mean Concentration	3.494938E-6	1.86948E-3

Student's t UCL95

UCL95	0.02147

Cd (*wt %*)

All Results (triangles) below MDCs

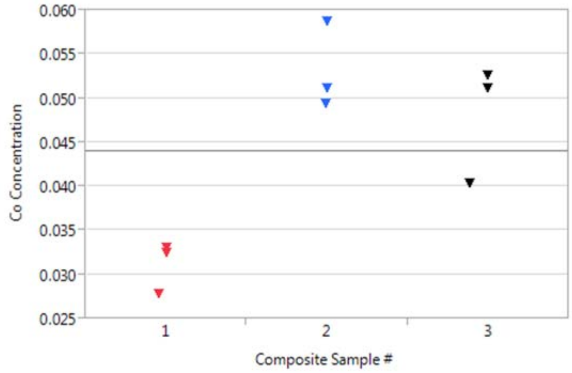


Minimum Detection Concentrations (MDCs)

Sample	N	Minimum MDC	Maximum MDC
1	3	3.35E-3	3.44E-3
2	3	3.42E-3	3.45E-3
3	3	3.37E-3	3.41E-3
Overall	9	3.35E-3	3.45E-3

Appendix F: Statistical Analysis of the Tank 16-H Annulus Samples
Table F23. Supporting Results for Elementals

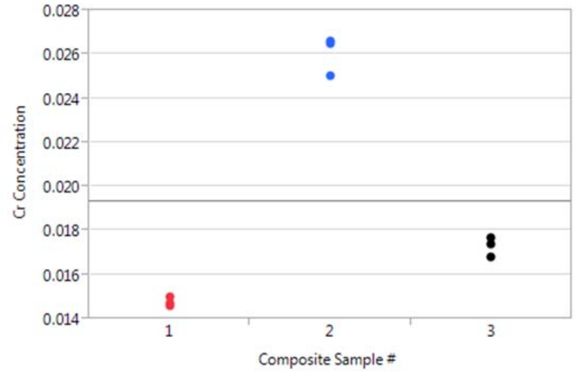
Co (*wt %*)
All Results (triangles) below MDCs



Minimum Detection Concentrations (MDCs)

Sample	Number	Minimum MDC	Maximum MDC
1	3	2.77E-2	3.30E-2
2	3	4.94E-2	5.87E-2
3	3	4.03E-2	5.25E-2
Overall	9	2.77E-2	5.87E-2

Cr (*wt %*)
All Results (circles) above MDCs



Means and Standard Deviations

Sample	Number	Mean	Std Dev	Std Err Mean
1	3	0.014767	0.000208	0.000120
2	3	0.026033	0.000896	0.000517
3	3	0.017300	0.000458	0.000265
Overall	9	0.019367	0.000594	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	1.2310	2	6	0.3565
Brown-Forsythe	0.4449	2	6	0.6604
Levene	4.6789	2	6	0.0596
Bartlett	1.4629	2		0.2316

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance

Source	DF	SS	MS	F Ratio	Prob >
Sample	2	0.00020963	0.000105	297.577	<.0001
Error	6	0.00000211	0.000000		
C. Total	8	0.00021174			

Statistically significant sampling variance.

Diagnostics for Sampling Model Residuals

Wilk-Shapiro	0.922 > 0.829 crit. value->SNS
Dixon High Outlier	Run 1 of Sample 2 -> SNS
Dixon Low Outlier	Run 2 of Sample 2 -> SNS

Variance Components

Component	Variance Comp	Std Dev
Composite Samples	3.482037E-5	5.90088E-3
Measurements	3.522222E-7	5.93483E-4
Total	3.517259E-5	5.93065E-3
Mean Concentration	1.164593E-5	3.41261E-3

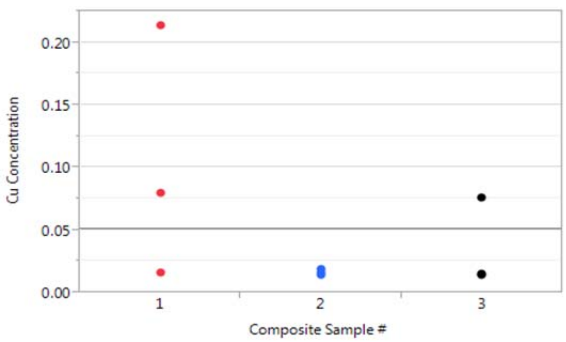
Student's t UCL95

UCL95	0.02933

Appendix F: Statistical Analysis of the Tank 16-H Annulus Samples
Table F23. Supporting Results for Elementals

Cu (*wt %*)

All Results (circles) above MDCs



Means and Standard Deviations

Sample	Number	Mean	Std Dev	Std Err Mean
1	3	0.103000	0.101252	0.05846
2	3	0.016267	0.002371	0.00137
3	3	0.035067	0.035631	0.02057
Overall	9	0.051444	0.061987	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	1.5599	2	6	0.2848
Brown-Forsythe	1.7185	2	6	0.2570
Levene	5.5438	2	6	0.0433
Bartlett	5.4433	2		0.0043

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	0.01249105	0.006246	1.6254	0.2728
Error	6	0.02305443	0.003842		
C. Total	8	0.03554548			

Statistically non-significant sampling variance.

Diagnostics for Concentrations

Wilk-Shapiro	0.645 < 0.829 crit. value->SS
Dixon High Outlier*	Run 1 of Sample 1 -> SS
Dixon Low Outlier*	Run 1 of Sample 3 -> SNS

Data do not follow a normal, gamma, or lognormal distribution. Obtain a UCL95 via a nonparametric method. Use Chebyshev's bootstrap UCL95 method.

Summary Statistics

Mean	5.1444E-2
Standard Deviation	6.6657E-2
Chebyshev UCL95	1.4829E-1

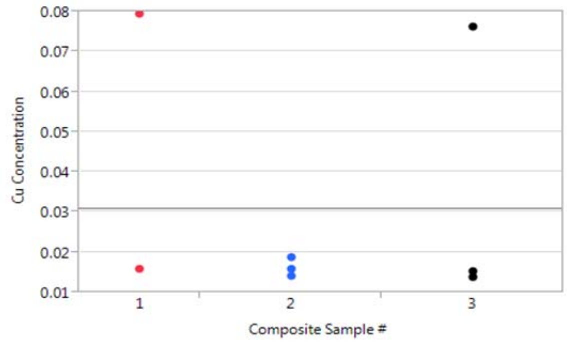
Reanalyze without the potential Run 1 Sample 1 outlier: 0.214 wt %.

* The Wilk-Shapiro goodness-of-fit test for normality was statistically significant (with or without the potential outlier in the data set). Therefore, the Dixon outlier test results are suspect, but the magnitude of the potential outlier suggests that Cu be run with and without it.

Cu (*wt %*)

All Results (circles) above MDCs

Omitted Run 1 Sample 1: 0.214 wt %



Means and Standard Deviations

Sample	Number	Mean	Std Dev	Std Err Mean
1	2	0.047500	0.044972	0.03180
2	3	0.016267	0.002371	0.00137
3	3	0.035067	0.035631	0.02057
Overall	8	0.031125	0.03024	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	1.7620	1	4	0.2551
Brown-Forsythe	1.2361	2	5	0.3663
Levene	12.5835	2	5	0.0112
Bartlett	3.3590	2		0.0348

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	0.00124520	0.000623	0.6807	0.5477
Error	5	0.00457293	0.000915		
C. Total	7	0.00581814			

Statistically non-significant sampling variance.

Diagnostics for Concentrations

Wilk-Shapiro	0.618 < 0.818 crit. value->SS
Dixon High Outlier	Run 2 of Sample 1 -> SNS
Dixon Low Outlier	Run 1 of Sample 3 -> SNS

Data do not follow a normal, gamma, or lognormal distribution. Obtain a UCL95 via a nonparametric method. Use Chebyshev's bootstrap UCL95 method.

Summary Statistics

Mean	3.1125E-2
Standard Deviation	2.8830E-2
Chebyshev UCL95	0.075555

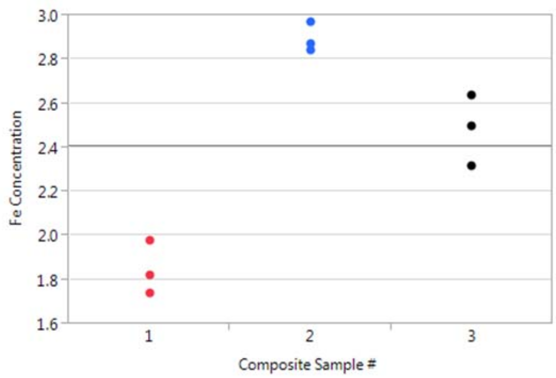
It is recommended to use the nonparametric UCL95 with all 9 data.

Appendix F: Statistical Analysis of the Tank 16-H Annulus Samples

Table F23. Supporting Results for Elementals

Fe (*wt %*)

All Results (circles) above MDCs



Means and Standard Deviations

Sample	Number	Mean	Std Dev	Std Err Mean
1	3	1.846667	0.122202	0.07055
2	3	2.893333	0.068069	0.03930
3	3	2.486667	0.160416	0.09262
Overall	9	2.408889	0.122882	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	0.6548	2	6	0.5530
Brown-Forsythe	0.5065	2	6	0.6262
Levene	0.7521	2	6	0.5111
Bartlett	0.5395	2		0.5830

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	1.67048889	0.835244	55.3142	0.0001
Error	6	0.09060000	0.015100		
C. Total	8	1.76108889			

Statistically significant sampling variance.

Diagnostics for Sampling Model Residuals

Wilk-Shapiro	0.965 > 0.829 crit. value->SNS
Dixon High Outlier	Run 2 of Sample 3 -> SNS
Dixon Low Outlier	Run 1 of Sample 3 -> SNS

Variance Components

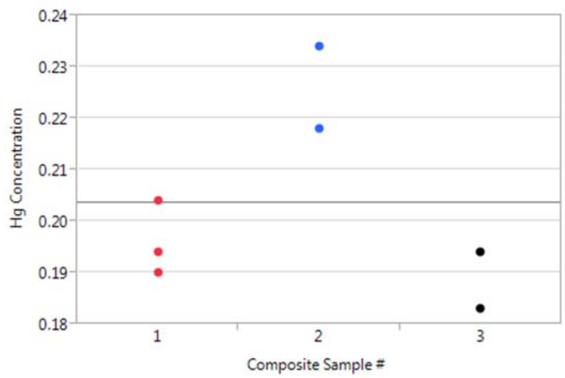
Component	Variance Comp	Std Dev
Composite Samples	0.27338148	0.522859
Measurements	0.01510000	0.122882
Total	0.28848148	0.537105
Mean Concentration	0.09280494	0.304639

Student's t UCL95

UCL95	3.29843

Hg (*wt %*)

All Results (circles) above MDCs



Means and Standard Deviations

Sample	Number	Mean	Std Dev	Std Err Mean
1	3	0.196000	0.007211	0.00416
2	3	0.228667	0.009238	0.00533
3	3	0.186667	0.006351	0.00367
Overall	9	0.203778	0.007696	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	0.2505	2	6	0.7862
Brown-Forsythe	0.0419	2	6	0.9592
Levene	0.5342	2	6	0.6116
Bartlett	0.1218	2		0.8853

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	0.00291822	0.001459	24.638	0.0013
Error	6	0.00035533	0.000059		
C. Total	8	0.00327356			

Statistically significant sampling variance.

Diagnostics for Sampling Model Residuals

Wilk-Shapiro	0.906 > 0.829 crit. value->SNS
Dixon High Outlier	Run 2 of Sample 1 -> SNS
Dixon Low Outlier	Run 2 of Sample 2 -> SNS

Variance Components

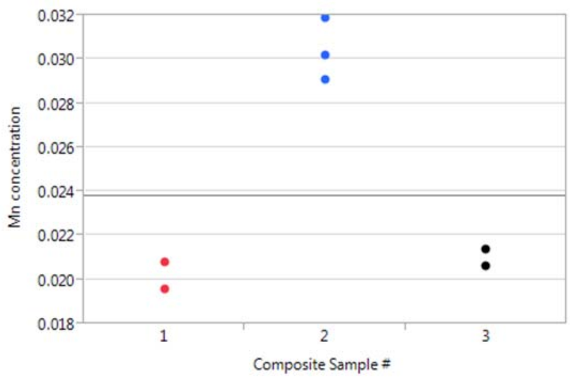
Component	Variance Comp	Std Dev
Composite Samples	4.666296E-4	2.16016E-2
Measurements	5.922222E-5	7.69560E-3
Total	5.258519E-4	2.29315E-2
Mean Concentration	1.621235E-4	1.27328E-2

Student's t UCL95

UCL95	0.24096

Appendix F: Statistical Analysis of the Tank 16-H Annulus Samples
Table F23. Supporting Results for Elementals

Mn (*wt %*)
All Results (circles) above MDCs



Means and Standard Deviations

Sample	Number	Mean	Std Dev	Std Err Mean
1	3	0.020000	0.000693	0.000400
2	3	0.030400	0.001411	0.000814
3	3	0.021133	0.000462	0.000267
Overall	9	0.023844	0.000946	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	1.1558	2	6	0.3762
Brown-Forsythe	0.7796	2	6	0.5001
Levene	1.7583	2	6	0.2506
Bartlett	1.0277	2		0.3578

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	0.00019532	0.000098	109.183	<.0001
Error	6	0.00000537	0.000001		
C. Total	8	0.00020068			

Statistically significant sampling variance.

Diagnostics for Sampling Model Residuals

Wilk-Shapiro	0.965 > 0.829 crit. value->SNS
Dixon High Outlier	Run3 of Sample 2 -> SNS
Dixon Low Outlier	Run 2 of Sample 2 -> SNS

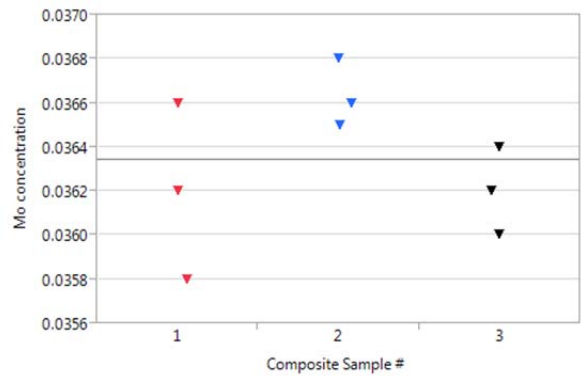
Variance Components

Component	Variance Comp	Std Dev
Composite Samples	3.225444E-5	5.67930E-3
Measurements	8.944444E-7	9.45751E-4
Total	3.314889E-5	5.75751E-3
Mean Concentration	1.085086E-5	3.29407E-3

Student's t UCL95

UCL95	0.03346

Mo (*wt %*)
All Results (triangles) below MDCs

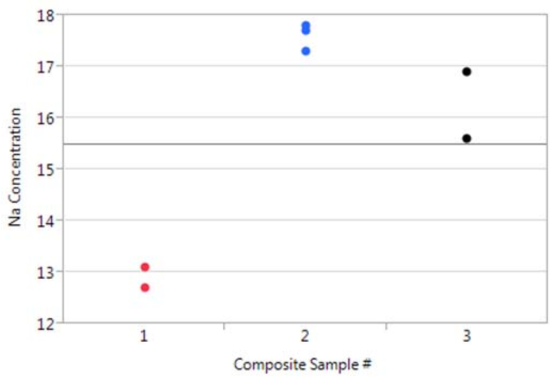


Minimum Detection Concentrations (MDCs)

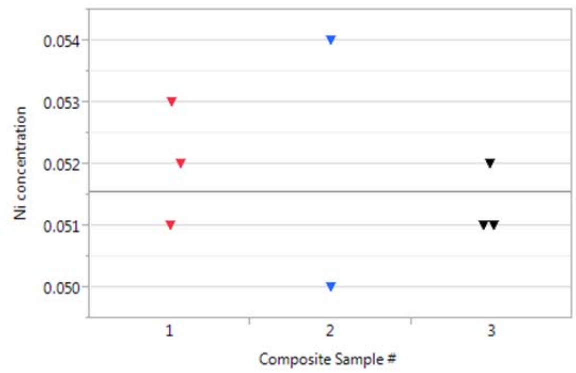
Sample	Number	Minimum MDC	Maximum MDC
1	3	3.58E-2	3.66E-2
2	3	3.65E-2	3.68E-2
3	3	3.60E-2	3.64E-2
Overall	9	3.58E-2	3.68E-2

Appendix F: Statistical Analysis of the Tank 16-H Annulus Samples
Table F23. Supporting Results for Elementals

Na (*wt %*)
All Results (circles) above MDCs



Ni (*wt %*)
All Results (triangles) below MDCs



Means and Standard Deviations

Sample	Number	Mean	Std Dev	Std Err Mean
1	3	12.8333	0.23094	0.13333
2	3	17.6000	0.26458	0.15275
3	3	16.0333	0.75056	0.43333
Overall	9	15.4889	0.47842	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	1.3774	2	6	0.3219
Brown-Forsythe	0.3687	2	6	0.7063
Levene	5.7925	2	6	0.0397
Bartlett	1.4243	2		0.2407

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	35.4155556	17.70778	77.3641	<.0001
Error	6	1.37333333	0.22889		
C. Total	8	36.7888889			

Statistically significant sampling variance.

Diagnostics for Sampling Model Residuals

Wilk-Shapiro	0.903 > 0.829 crit. value->SNS
Dixon High Outlier	Run 1 of Sample 3 -> SNS
Dixon Low Outlier	Runs 2 & 3 of Sample 3 -> SNS

Variance Components

Component	Variance Comp	Std Dev
Composite Samples	5.82629630	2.41377
Measurements	0.22888889	0.47842
Total	6.05518519	2.46073
Mean Concentration	1.96753086	1.40269

Student's t UCL95

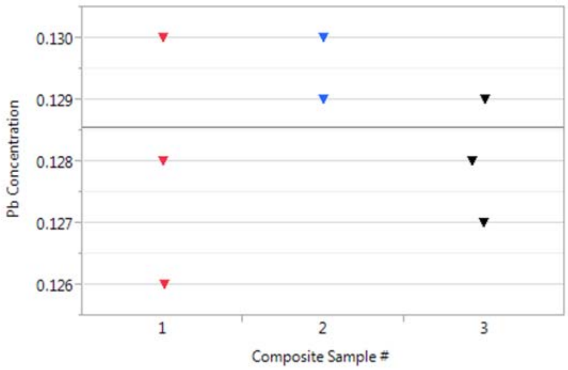
UCL95	19.58471

Minimum Detection Concentrations (MDCs)

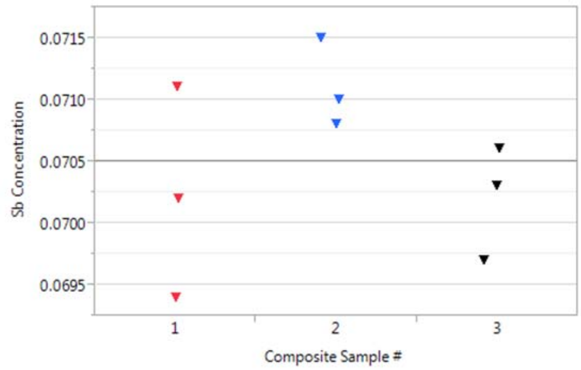
Sample	Number	Minimum MDC	Maximum MDC
1	3	5.10E-2	5.30E-2
2	3	5.00E-2	5.40E-2
3	3	5.10E-2	5.20E-2
Overall	9	5.00E-2	5.40E-2

Appendix F: Statistical Analysis of the Tank 16-H Annulus Samples
Table F23. Supporting Results for Elementals

Pb (*wt %*)
All Results (triangles) below MDCs



Sb (*wt %*)
All Results (triangles) below MDCs



Minimum Detection Concentrations (MDCs) (Pb)

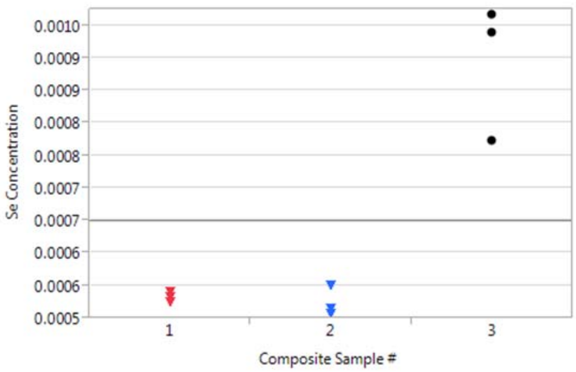
Sample	Number	Minimum MDC	Maximum MDC
1	3	0.126	0.130
2	3	0.129	0.130
3	3	0.127	0.129
Overall	9	0.126	0.130

Minimum Detection Concentrations (MDCs) (Sb)

Sample	Number	Minimum MDC	Maximum MDC
1	3	6.94E-2	7.11E-2
2	3	7.08E-2	7.15E-2
3	3	6.97E-2	7.06E-2
Overall	9	6.94E-2	7.15E-2

Appendix F: Statistical Analysis of the Tank 16-H Annulus Samples
Table F23. Supporting Results for Elementals

Se (*wt %*)
Mixture of Results (circles) above MDCs
and (triangles) below MDCs



All 3 results for composite Samples 1 and 2 were below their MDCs. All 3 results for Composite Sample 3 were above their MDCs. There is no direct information to compute the sampling variance. Mathematically, the largest possible UCL95 using Student's t UCL95 can be obtained by setting all 3 Composite Sample 1 results to their MDCs, and all 3 Composite Sample 2 results to 0. This honors the restriction on the composite sample concentrations in the following table.

Minimum Detection Concentrations (MDCs)

Sample	Run	Minimum	Maximum MDC
1	1	0	0.000523
1	2	0	0.000540
1	3	0	0.000532
2	1	0	0.000506
2	2	0	0.000514
2	3	0	0.000549

The "dataset" that produces the largest possible UCL95 includes the values in red in the above table plus the 3 concentration results for Composite Sample 3.

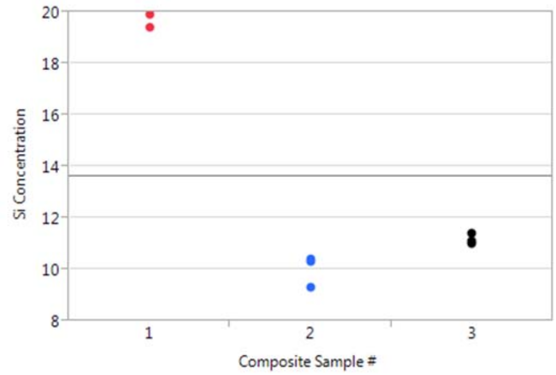
Concentration measurements for Composite Sample 3

Sample	Run	Maximum MDC
3	1	0.000968
3	2	0.00094
3	3	0.000773

The following results were obtained.

Statistic	Estimate
Mean	0.0004751
Std Dev	0.0004495
% Std Dev	94.611%
UCL95	0.001233

Si (*wt %*)
All Results (circles) above MDCs



Means and Standard Deviations

Sample	Number	Mean	Std Dev	Std Err Mean
1	3	19.73333	0.288675	0.16667
2	3	9.996667	0.614030	0.35451
3	3	11.16667	0.208167	0.12019
Overall	9	13.63222	0.409756	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	1.1729	2	6	0.3716
Brown-Forsythe	0.3547	2	6	0.7151
Levene	4.1209	2	6	0.0748
Bartlett	1.0195	2		0.3608

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	169.559356	84.77968	504.94	<.0001
Error	6	1.00740000	0.167900		
C. Total	8	170.566756			

Statistically significant sampling variance.

Diagnostics for Sampling Model Residuals

Wilk-Shapiro	0.917 > 0.829 crit. value->SNS
Dixon High Outlier	Run 1 of Sample 2 -> SNS
Dixon Low Outlier	Run 2 of Sample 2 -> SNS

Variance Components

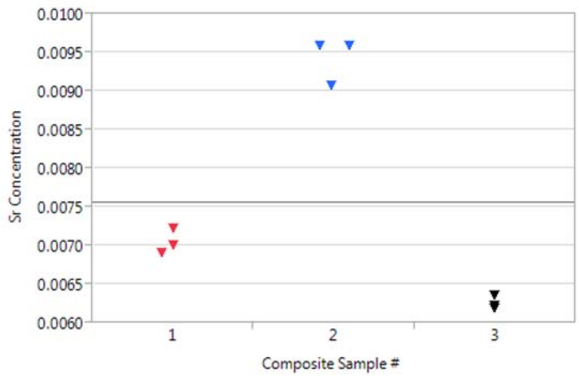
Component	Variance Comp	Std Dev
Composite Samples	28.203926	5.310737
Measurements	0.167900	0.409756
Total	28.371826	5.326521
Mean Concentration	9.419964	3.069196

Student's t UCL95

UCL95	22.59423

Appendix F: Statistical Analysis of the Tank 16-H Annulus Samples
Table F23. Supporting Results for Elementals

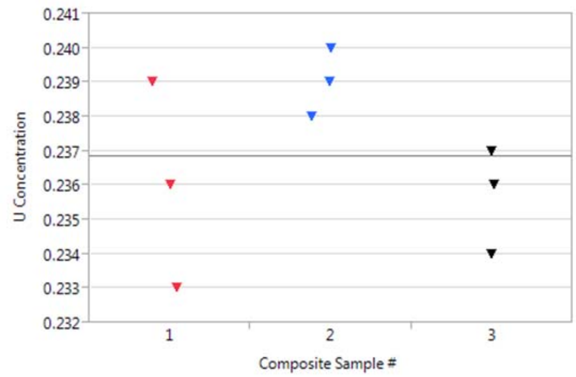
Sr (*wt %*)
All Results (triangles) below MDCs



Minimum Detection Concentrations (MDCs)

Sample	Number	Minimum MDC	Maximum MDC
1	3	6.90E-3	7.22E-3
2	3	9.07E-3	9.59E-3
3	3	6.18E-3	6.35E-3
Overall	9	6.18E-3	9.59E-3

U (*wt %*)
All Results (triangles) below MDCs



Minimum Detection Concentrations (MDCs)

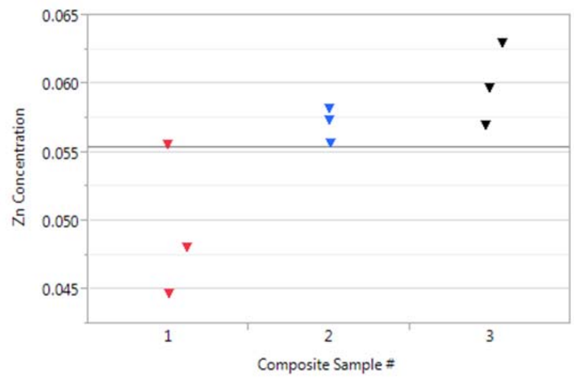
Sample	Number	Minimum MDC	Maximum MDC
1	3	0.233	0.239
2	3	0.238	0.240
3	3	0.234	0.237
Overall	9	0.233	0.240

Appendix F: Statistical Analysis of the Tank 16-H Annulus Samples

Table F23. Supporting Results for Elementals

Zn (*wt %*)

All Results (triangles) below MDCs



Minimum Detection Concentrations (MDCs)			
ample	Number	Minimum MDC	Maximum MDC
1	3	4.47E-2	5.55E-2
2	3	5.56E-2	5.82E-2
3	3	5.69E-2	6.29E-2
)verall	9	4.47E-2	6.29E-2

Appendix F: Statistical Analysis of the Tank 16-H Annulus Samples

Table F24. Statistical Summary for the Anions with All Measurements below their MDCs

Anion Constituent (*wt %*)	N	Smallest Minimum Detectable Concentration (*wt %*)		Largest Minimum Detectable Concentration (*wt %*)	
		Fixed Decimal Format	Scientific Format	Fixed Decimal Format	Scientific Format
Bromide, Br-1	9	0.0269	2.69e-2	0.0304	3.04e-2
Fluoride, F-1	9	0.0269	2.69e-2	0.0304	3.04e-2
Formate, CHO2-1	9	0.0269	2.69e-2	0.0304	3.04e-2
Iodine, I-127	9	0.0000162	1.62e-5	0.0000183	1.83e-5
Phosphate, PO4-3	9	0.0269	2.69e-2	0.0304	3.04e-2
Total Iodine	9	0.000374	3.74e-4	0.00096	9.60e-4

Appendix F: Statistical Analysis of the Tank 16-H Annulus Samples

Table F25. Statistical Summary for the Anions with All Measurements above their MDCs

Constituent	N	Mean (wt %)	Std Dev♣ (wt %)	% Std Dev	UCL95 (wt %)	Goodness-of-Fit/Confidence Limit Remarks♠
Chloride , Cl^{-1}	9	4.314E-2	1.400E-2	32.452%	6.564E-2	SNS-VH; SS-SV; SNS-WS; SNS-OT; Use Student's t UCL95
Iodine, I-129♠	9	4.9389E-4	1.7695E-4	35.828%	6.3621E-4	SNS-VH; SNS-SV; SS-WS; SS-OT (Run 3 for Sample 3); SNS-KS (approx. gamma); Use bias-corrected gamma maximum likelihood estimates of the mean and standard deviation; Adjusted Gamma UCL95
	8	4.3788E-4	6.4492E-5	14.728%	4.8107E-4	Omit Potential Outlier Run 3 for Sample 3: 0.000942 wt %; SNS-VH; SNS-SV; SNS-WS; SNS-DT; Use Student's t UCL95
Nitrate , NO_3^{-1}×	9	4.9856	2.7651E-1	5.546%	5.4072	SNS-VH; SS-SV; SS-DT; SNS-WS; Use Student's t UCL95
	8	5.0324	2.1010E-1	4.175%	5.3823	SNS-VH; SS-SV; SNS-DT; SNS-WS; Use Student's t UCL
Nitrite , NO_2^{-1}	9	6.1044	2.5749E-1	4.218%	6.2641	SNS-VH; SNS-SV; SNS-WS; SNS-DT; Use Student's t UCL95
Oxalate, $C_2O_4^{2-}$	9	7.0044E-2	8.6302E-3	12.321%	7.5394E-2	SNS-VH; SNS-SV; SNS-WS; SNS-DT; Use Student's t UCL95
Sulfate, SO_4^{-2}	9	6.4600E-1	1.8162E-1	28.115%	9.5095E-1	SNS-VH; SS-SV; SNS-WS; SNS-DT; Use Student's t UCL95

MDCs: Minimum Detectable Concentrations

SS-VH/ SNS-VH: Levene's test for heterogeneity of measurement/sample preparation variances were statistical significant at $\alpha = 0.05/6 = .0083$

SS-SV/ SNS-SV: Statistically significant/Statistically non-significant sampling variance at $\alpha = 0.05$.

SS-KS (approx. gamma)/ SNS-KS (approx. gamma): Statistically significant/Statistically non-significant Kolmogrov-Smirnoff goodness-of-fit test for a gamma distribution at $\alpha = 0.05$.

SS-WS/ SNS-WS: Statistically significant/Statistically non-significant Wilk-Shapiro test statistic for testing normality at $\alpha = 0.05$.

SS-OT / SNS-OT: Statistically significant/Statistically non-significant Dixon's test for a high outlier or for a low outlier at $\alpha = 0.05$.

♠ Iodine, I-129 was analyzed twice: first, accommodating a potential high outlier from Measurement Run 3 of Composite Sample 3 (0.000942 wt %); and then second, omitting the potential outlier. The first analysis is recommended since it admits conservatively higher values for the mean, standard deviation, and the UCL95.

× Nitrate, NO_3^{-1} was analyzed twice: first, accommodating a potential low outlier from Measurement Run 2 of Composite Sample 2 (5.55 wt %); then second, omitting the potential outlier. The first analysis is recommended since it admits conservatively higher values for the mean, standard deviation, and the UCL95.

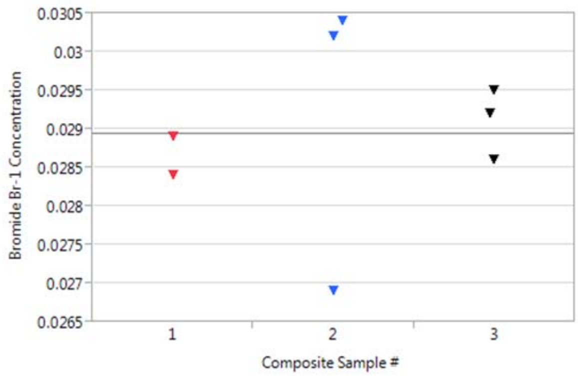
♣ When the sampling variation among the composite samples is demonstrated to be statistically significant (SS-SV), the standard deviation (Std Dev) and the percent standard deviation (% Std Dev) account for the variation among the composite samples and the variation attributable to a single measurement of the composite sample: $StdDev : s_{Total} = \sqrt{s_{Samp}^2 + s_{Meas}^2}$.

Appendix F: Statistical Analysis of the Tank 16-H Annulus Samples

Table F26. Supporting Results for Anions

Bromide, Br-1 (wt %)

All Results (triangles) below MDCs

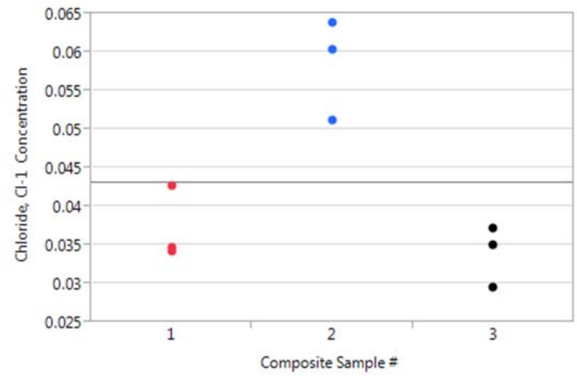


Minimum Detection Concentrations (MDCs)

Sample	Number	Minimum MDC	Maximum MDC
1	3	2.84E-2	2.89E-2
2	3	2.69E-2	3.04E-2
3	3	2.86E-2	2.95E-2
Overall	9	2.69E-2	3.04E-2

Chloride, Cl-1 (*wt %*)

All Results (circles) above MDCs



Means and Standard Deviations on Original Scale

Sample	Number	Mean	Std Dev	Std Err Mean
1	3	3.713E-2	4.744E-3	2.74E-3
2	3	5.843E-2	6.601E-3	3.81E-3
3	3	3.387E-2	3.925E-3	2.27E-3
Pooled	9	4.314E-2	5.212E-3	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	0.4333	2	6	0.6672
Brown-Forsythe	0.1591	2	6	0.8564
Levene	0.6924	2	6	0.5363
Bartlett	0.2312	2		0.7936

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	1.0679E-3	5.339E-4	19.659	0.0023
Error	6	1.6296E-4	2.716E-5		
C. Total	8	1.2308E-3			

Statistically significant sampling variance.

Diagnostics for Sampling Model Residuals

Wilk-Shapiro	0.939 < 0.829 crit. value->SNS
Dixon High Outlier	Run 1 of Sample 1 -> SNS Run 1 of Sample 2 -> SNS
Dixon Low Outlier	Run 2 of Sample 2 -> SNS

Variance Components

Component	Variance Comp	Std Dev
Composite Samples	1.6893E-04	1.2997E-02
Measurements	2.7160E-05	5.2115E-03
Total	1.9609E-04	1.4003E-02
Mean Concentration	5.9327E-05	7.7024E-03

Student's t UCL95

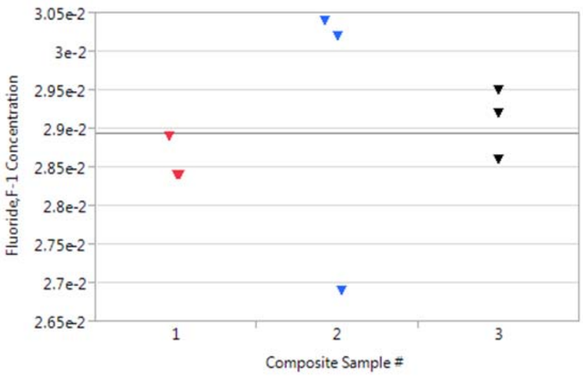
UCL95	6.5635E-2

Appendix F: Statistical Analysis of the Tank 16-H Annulus Samples

Table F26. Supporting Results for Anions

Fluoride, F-1 (*wt %*)

All Results (triangles) below MDCs

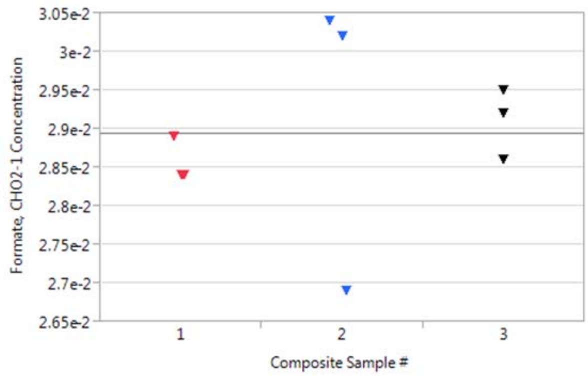


Minimum Detection Concentrations (MDCs)

Sample	N	Minimum MDC	Maximum MDC
1	3	2.84E-2	2.89E-2
2	3	2.69E-2	3.04E-2
3	3	2.86E-2	2.95E-2
Overall	9	2.69E-2	3.04E-2

Formate, CHO 2 -1 (*wt %*)

All Results (triangles) below MDCs



Minimum Detection Concentrations (MDCs)

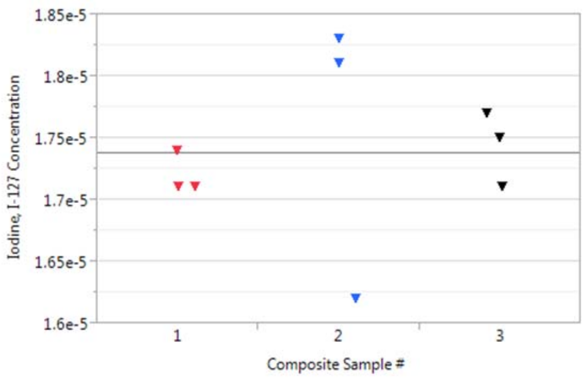
Sample	N	Minimum MDC	Maximum MDC
1	3	2.84E-2	2.89E-2
2	3	2.69E-2	3.04E-2
3	3	2.86E-2	2.95E-2
Overall	9	2.69E-2	3.04E-2

Appendix F: Statistical Analysis of the Tank 16-H Annulus Samples

Table F26. Supporting Results for Anions

Iodine, I-127 (*wt %*)

All Results (triangles) below MDCs



Minimum Detection Concentrations (MDCs)

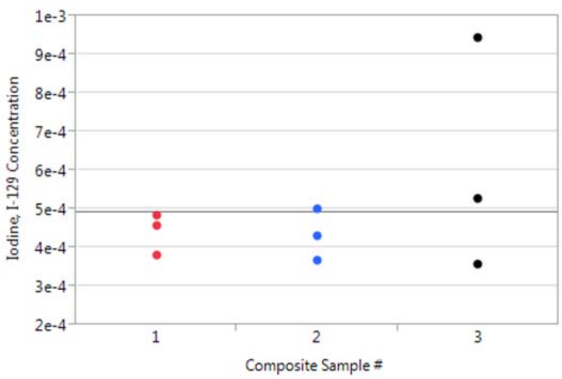
Sample	Number	Minimum MDC	Maximum MDC
1	3	1.71E-5	1.74 E-5
2	3	1.62E-5	1.83 E-5
3	3	1.71E-5	1.77 E-5
Overall	9	1.62E-5	1.83 E-5

Appendix F: Statistical Analysis of the Tank 16-H Annulus Samples

Table F26. Supporting Results for Anions

Iodine, I-129 (*wt %*)

All Results (circles) above MDCs



Means and Standard Deviations on Original Scale

Sample	Number	Mean	Std Dev	Std Err Mean
1	3	4.397E-4	5.316E-5	3.069E-5
2	3	4.330E-4	6.602E-5	3.811E-5
3	3	6.090E-4	3.008E-4	1.737E-4
Pooled	9	4.939E-4	1.804E-4	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	1.6344	2	6	0.2713
Brown-Forsythe	1.5822	2	6	0.2806
Levene	5.2810	2	6	0.0475
Bartlett	2.8083	2		0.0603

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance*

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	5.9694E-8	2.985E-8	0.9168	0.4493
Error	6	1.9533E-7	3.255E-8		
C. Total	8	2.5502E-7			

Statistically non-significant sampling variance.

Diagnostics for Concentrations

Wilk-Shapiro	0.708 < 0.829 crit. value->SS
Dixon High Outlier	Run 3 of Sample 3 -> SS
Dixon Low Outlier	Run 2 of Sample 3 -> SNS
KS (approx. Gamma)	0.261<0.279 crit. value -> SNS

Gamma Statistics

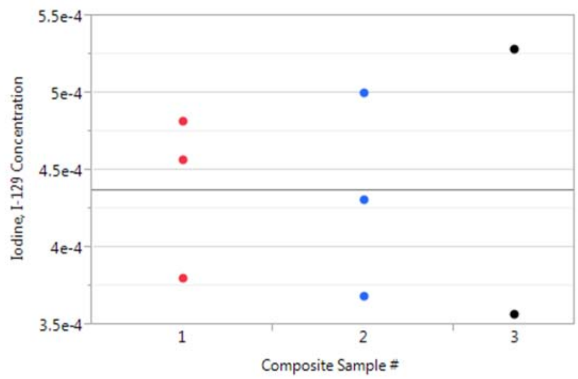
Component	Estimate
Mean (MLE)	4.9389E-4
Std.Dev. (Bias-corr. MLE)	1.7695E-4
Adjusted Gamma UCL95	6.3621E-4

Iodine, I-129 reanalyzed on following page after omitting a potential outlier for Run 3 of Composite Sample 3 (0.000942 wt %).

Iodine, I-129 (*wt %*)

All Results (circles) above MDCs

Omitted Run 3 of Composite Sample 3: 0.000942 wt %



Means and Standard Deviations on Original Scale

Sample	Number	Mean	Std Dev	Std Err Mean
1	3	4.397E-4	5.3163E-5	3.0694E-5
2	3	4.330E-4	6.6023E-5	3.8118E-5
3	2	4.425E-4	1.2092E-4	8.5500e-5
Pooled	8	4.379E-4	7.6146E-5	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	0.1547	1	4	0.7141
Brown-Forsythe	1.4120	2	5	0.3265
Levene	1.9550	2	5	0.2359
Bartlett	0.4165	2		0.6594

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	1.2371E-10	6.185E-11	0.0107	0.9894
Error	5	2.89912E-8	5.7982E-9		
C. Total	7	2.91149E-8			

Statistically non-significant sampling variance.

Diagnostics for Concentrations

Wilk-Shapiro	0.929 > 0.818 crit. value->SNS
Dixon High Outlier	Run 1 of Sample 3 -> SNS
Dixon Low Outlier	Run 2 of Sample 3 -> SNS

Normal Statistics

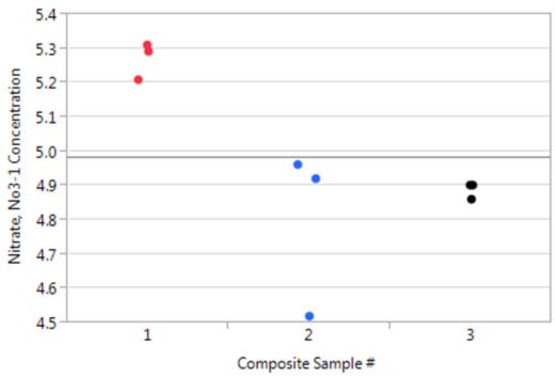
Component	Estimate
Mean (MLE)	4.3788E-4
Std.Dev. (Bias-corr. MLE)	6.4492E-5
Student's t UCL95	4.8107E-4

Appendix F: Statistical Analysis of the Tank 16-H Annulus Samples

Table F26. Supporting Results for Anions

Nitrate, NO 3 -1 (*wt %*)

All Results (circles) above MDCs



Means and Standard Deviations on Original Scale

Sample	Number	Mean	Std Dev	Std Err Mean
1	3	5.27000	5.292E-2	3.055E-2
2	3	4.80000	2.433E+0	1.405E-1
3	3	4.88667	2.309E-2	1.333E-2
Pooled	9	4.98556	1.444E-1	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	1.6772	2	6	0.2639
Brown-Forsythe	0.9160	2	6	0.4496
Levene	10.2525	2	6	0.0116
Bartlett	3.7876	2		0.0226

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	0.375356	0.18768	9.004	0.0156
Error	6	0.125067	0.02084		
C. Total	8	0.500422			

Statistically significant sampling variance.

Diagnostics for Sampling Model Residuals

Wilk-Shapiro	0.877 > 0.829 crit. value->SNS
Dixon High Outlier	Run 1 of Sample 2 -> SNS
Dixon Low Outlier	Run 2 of Sample 2 -> SS

Variance Components

Component	Variance Comp	Std Dev
Composite Samples	5.5611E-02	0.235820
Measurements	2.0844E-02	0.144376
Total	7.6456E-02	0.276506
Mean Concentration	2.0853E-02	0.144406

Student's t UCL95

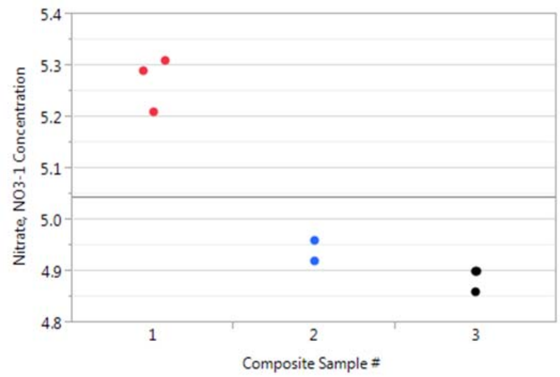
UCL95	5.40722

Nitrate, NO_3^{-1} reanalyzed on following page after omitting a potential outlier for Run 2 of Composite Sample 2 (4.52 wt %).

Nitrate, NO 3 -1 (*wt %*)

All Results (circles) above MDCs

Omitted Run 2 of Composite Sample 2: 4.52 wt %



Means and Standard Deviations on Original Scale

Sample	Number	Mean	Std Dev	Std Err Mean
1	3	5.27000	5.292E-2	3.055E-2
2	2	4.94000	2.828E-2	2.000E-2
3	3	4.88667	2.309E-2	1.333E-2
Pooled	8	5.04375	3.864E-2	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	1.1242	1	4	0.3488
Brown-Forsythe	0.3401	2	5	0.7270
Levene	2.3387	2	5	0.1919
Bartlett	0.5485	2		0.5778

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	0.249121	0.124560	83.411	0.0001
Error	5	0.007467	0.001493		
C. Total	7	0.256588			

Statistically significant sampling variance.

Diagnostics for Sampling Model Residuals

Wilk-Shapiro	0.906 > 0.818 crit. value->SNS
Dixon High Outlier	Run 1 of Sample 1 -> SNS
Dixon Low Outlier	Run 2 of Sample 1 -> SNS

Variance Components

Component	Variance Comp	Std Dev
Composite Samples	4.26512E-2	0.206522
Measurements	1.49253E-3	0.038633
Total	4.41437E-2	0.210104
Mean Concentration	1.43829E-2	0.119929

REML Mean	5.032400

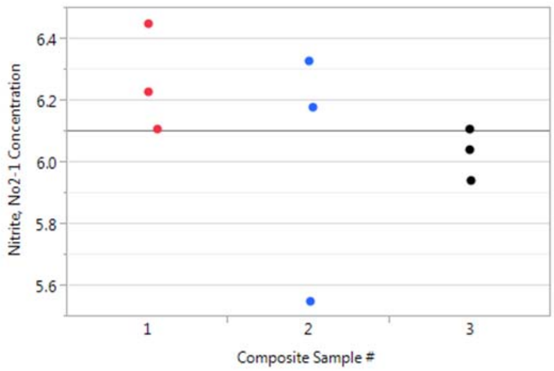
Student's t UCL95

UCL95	5.38229

Appendix F: Statistical Analysis of the Tank 16-H Annulus Samples

Table F26. Supporting Results for Anions

Nitrite, NO 2 -1 (*wt %*)
All Results (circles) above MDCs



Means and Standard Deviations on Original Scale

Sample	Number	Mean	Std Dev	Std Err Mean
1	3	6.26333	0.17243	0.09955
2	3	6.02000	0.41388	0.23896
3	3	6.03000	0.08544	0.04933
Pooled	9	6.10444	0.26352	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	1.3926	2	6	0.3186
Brown-Forsythe	0.8053	2	6	0.4900
Levene	4.8098	2	6	0.0567
Bartlett	1.7984	2		0.1656

Statistically non-significant measurement variance heterogeneity.

Analysis of Variance

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	0.1137556	0.05688	0.819	0.4847
Error	6	0.4166667	0.06944		
C. Total	8	0.5304222			

Statistically non-significant sampling variance.

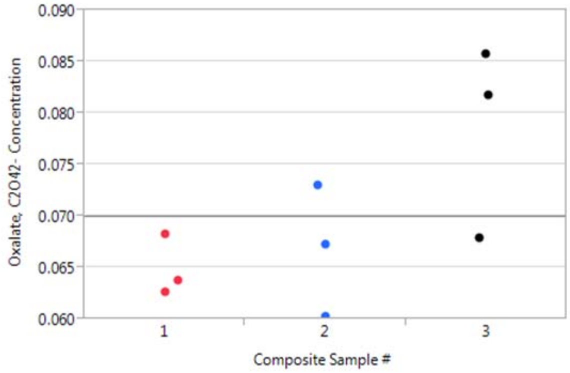
Diagnostics for Concentrations

Wilk-Shapiro	0.925 > 0.829 crit. value->SNS
Dixon High Outlier	Run 1 of Sample 1 -> SNS
Dixon Low Outlier	Run 2 of Sample 2 -> SNS

Normal Statistics

Component	Estimate
Mean (MLE)	6.1044444
Std.Dev. (Bias-corr. MLE)	0.2574933
Student's t UCL95	6.2640515

Oxalate, C2 O 4 2− (*wt %*)
All Results (circles) above MDCs



Means and Standard Deviations on Original Scale

Sample	Number	Mean	Std Dev	Std Err Mean
1	3	0.06483	0.00297	0.00171
2	3	0.06687	0.00636	0.00367
3	3	0.07843	0.00934	0.00539
Pooled	9	0.07004	0.00675	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	0.8903	2	6	0.4586
Brown-Forsythe	0.5380	2	6	0.6097
Levene	1.9672	2	6	0.2203
Bartlett	0.9072	2		0.4036

Statistically non-significant measurement variance heterogeneity

Analysis of Variance

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	0.00032288	0.000161	3.5487	0.0961
Error	6	0.00027296	0.000045		
C. Total	8	0.00059584			

Statistically non-significant sampling variance.

Diagnostics for Concentrations

Wilk-Shapiro	0.894 > 0.829 crit. value->SNS
Dixon High Outlier	Run 2 of Sample 3 -> SNS
Dixon Low Outlier	Run 3 of Sample 2 -> SNS

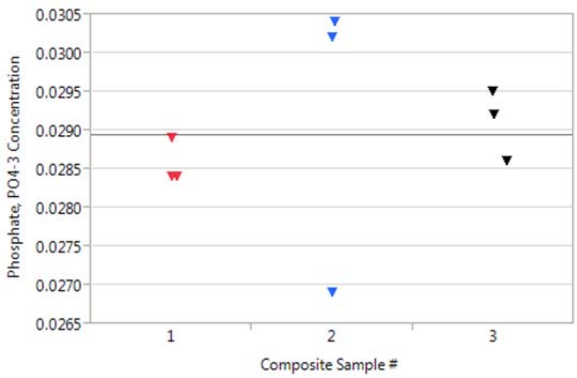
Normal Statistics

Component	Estimate
Mean (MLE)	7.0044E-2
Std.Dev. (Bias-corr. MLE)	8.6302E-3
Student's t UCL95	7.5394E-2

Appendix F: Statistical Analysis of the Tank 16-H Annulus Samples

Table F26. Supporting Results for Anions

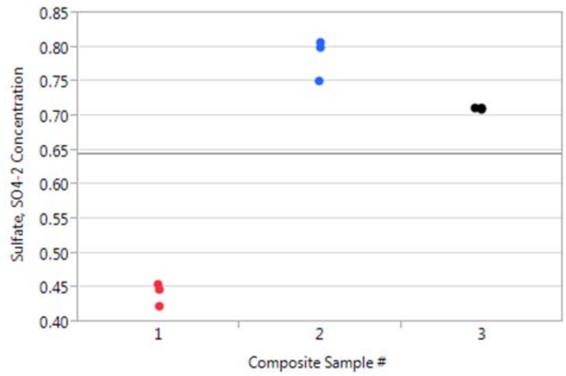
Phosphate, PO 4 -3 (*wt %*)
All Results (triangles) below MDCs



Minimum Detection Concentrations (MDCs)

Sample	Number	Minimum MDC	Maximum MDC
1	3	2.84E-2	2.89E-2
2	3	2.69E-2	3.04E-2
3	3	2.86E-2	2.95E-2
Overall	9	2.69E-2	3.04E-2

Sulfate, SO 4 -2 (*wt %*)
All Results (circles) above MDCs



Means and Standard Deviations on Original Scale

Sample	Number	Mean	Std Dev	Std Err Mean
1	3	0.44167	1.665E-2	9.615E-3
2	3	0.78567	3.017E-2	1.742E-2
3	3	0.71067	1.528E-3	8.819E-4
Pooled	9	0.64600	1.992E-2	

Tests that the Measurement Variances are Equal

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	1.2767	2	6	0.3452
Brown-Forsythe	0.7853	2	6	0.4978
Levene	7.0431	2	6	0.0267
Bartlett	3.8151	2		0.0220

Statistically non-significant measurement variance heterogeneity

Analysis of Variance

Source	DF	SS	MS	F Ratio	Prob > F
Sample	2	1.9632E-1	9.816E-2	247.5	<.0001
Error	6	2.3800E-3	3.967E-4		
C. Total	8	1.9870E-1			

Statistically significant sampling variance.

Diagnostics for Sampling Model Residuals

Wilk-Shapiro	0.873 > 0.829 crit. value->SNS
Dixon High Outlier	Run 3 of Sample 2 -> SNS
Dixon Low Outlier	Run 2 of Sample 2 -> SNS

Variance Components

Component	Variance Comp	Std Dev
Composite Samples	3.258811E-02	0.18052
Measurements	3.966667E-04	0.01992
Total	3.298478E-02	0.18162
Mean Concentration	1.090678E-02	0.10444

Student's t UCL95

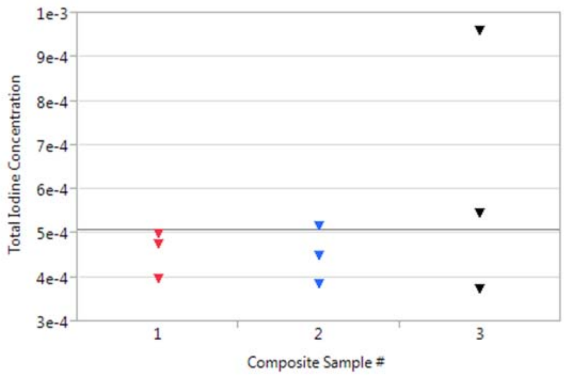
UCL95	0.95095

Appendix F: Statistical Analysis of the Tank 16-H Annulus Samples

Table F26. Supporting Results for Anions

Total Iodine (*wt %*)

All Results (triangles) below MDCs



Mixed Measurements (circles)and below MDC Results (triangles)

Minimum Detection Concentrations (MDCs)

Sample	Number	Minimum MDC	Maximum MDC
1	3	3.97E-4	4.99E-4
2	3	3.86E-4	5.16E-4
3	3	3.74E-4	9.60E-4
Overall	9	3.74E-4	9.60E-4

Distribution:

S. L. Marra, 773-A
T. B. Brown, 773-A
D. H. McGuire, 999-W
S. D. Fink, 773-A
C. C. Herman, 773-A
E. N. Hoffman, 999-W
F. M. Pennebaker, 773-42A
W. R. Wilmarth, 773-A
M. J. Mahoney, 705-1C
J. E. Occhipinti, 704-56H
A.W. Wiggins Jr., 241-168H
L. H. Connelly,773-B157
E.P Shine, 703-41A, Rm 233
D. P. Diprete, 773-41A-Rm 139
S. H. Reboul, 773-42A
J. P. Pavletich, 705-1C
R. H. Young. 703-41A-B152
M. S. Hay, 773-42A-Rm 148
KD Dixon, 705-1C
C. D. Walters, 241-162H
M. H. Layton, 705-1C
C. J. Coleman, 773-A-B113
T. B. Edwards, 999-W
M. K. Harris, 703-41A
M. B. Gorensak, 703-41A
G. C. Arthur, 241-284H
J. W. Rush, 241-284H
R. O. Voegtlen, 241-162H
J. R. Cantrell. 705-1C
G. M. Grim, 241-162H