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Characterization of Infrequent Samples from the Concentration, Storage, and Transfer Facility: Leak Detection Box (LDB) Drain Cell Sample LIMS# 20195

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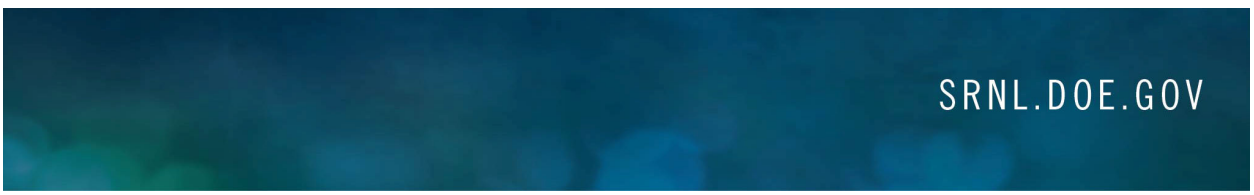
November 2020

SRNL-STI-2020-00534, Rev. 0



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EXECUTIVE SUMMARY

Savannah River Remediation Engineering (SRR-E) requested that the Savannah River National Laboratory (SRNL) analyze the Concentration, Storage, and Transfer Facility (CSTF) samples from the following Tank Farm areas: the sump encasement, catch tank, drain cell and waste tank annulus. In general, these CSTF samples will be analyzed on an infrequent basis and analyses will include detection for total beta/gamma, total alpha activities and pH measurements.

This report presents characterization results for the Leak Detection Box Drain Cell (LDB-DC) sample. The results are measurements for total gamma, total alpha, total beta, pH, density, anions, elementals and total mercury for the LDB-DC sample.

Most of these analyses were performed in triplicate. A summary of the average analytical results based on one sigma analytical uncertainty for the LDB-DC sample includes the following.

The measured density for the LDB-DC "as-received" CSTF sample was 0.98 (0.22 %RSD) and the directly measured pH was 7.66. The total alpha activity for the LDB-DC sample was reported as a less than value (upper limits) either because of possible spectral interferences or because there is not much alpha activity in the sample. Thus, total alpha activities averaged <1.74E+02 dpm/mL.

The total beta in the LDB-DC sample was above the instrument detection limits and averaged 3.08E+04 (3.25E-01 %RSD) dpm/mL.

The measured cesium-137 average activity (total gamma) in the LDB-DC sample was 2.52E+04 dpm/mL (1.95%RSD). The corresponding $Ba\text{-}137^{m}$ activities, calculated as 94.7% of the Cs-137 values, is 2.39E+04 dpm/mL (1.95 %RSD). Cesium 134, at an average of < 5.35 dpm/mL, was below the instrument detection limit.

The total activity of the beta and gamma emitting radionuclides measured within the uncertainty of the analyses equals 5.47E+04 dpm/ml, which is still about an order of magnitude less than 8.69E+05 dpm/mL, which is the procedural limit for the LDB-DC sample.

Sulfate (2.89 ± 0.01 mg/L), nitrate (2.64 ± 0.35 mg/L), phosphate (1.59 ± 0.04 mg/L) and chloride (1.18 ± 0.02 mg/L) were the four predominant anions in the LDB-DC sample. All other anions were below the instrument detection limits and total mercury concentration in the sample was below the instrument detection limit (<0.01 mg/L).

The predominant cation concentrations in the LDB-DC sample are Na [26.35 ± 1.34 (5.10 %RSD)] mg/L, K [23.5 ± 0.49 (2.13 %RSD)] mg/L, Ca [3.57 ± 0.01 (0.40 %RSD)] mg/L and Mg [≤0.40 mg/L]. The concentrations of other cations were below instrument detection limits.

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LIST OF ABBREVIATIONS

ARD	Analytical Research and Development
CSTF	Concentration, Storage and Transfer Facility
DMA	Direct Mercury Analysis
EMES	Environmental, Materials and Energy Sciences
HAD	High Activity Drain
IC	ion chromatography
ICP-AES	Inductively Coupled Plasma Atomic Emission Spectroscopy
LDB-DC	Leak Detection Box-Drain Cell
LSC	Liquid Scintillation Counting
MDA	minimum detection activity
MDL	minimum detection limit
n/a	not applicable
RSD	relative standard deviation
SRNL	Savannah River National Laboratory
SRR	Savannah River Remediation
St.Dev.	standard deviation
TTQAP	Technical Task and Quality Assurance Plan
TTR	Technical Task Request
UL	upper limit

1.0 Introduction

Savannah River Remediation Engineering (SRR-E) requested that the Savannah River National Laboratory (SRNL) analyze the Concentration, Storage, and Transfer Facility (CSTF) samples from the following areas: the sump encasement, catch tank, drain cell and waste tank annulus. In general, analyses on these CSTF samples will include detection for total beta/gamma, total alpha activities and pH measurements. CSTF operation personnel will request specific analyses when waste tank annulus samples are delivered to SRNL as part of this characterization scheme. These CSTF samples will be analyzed on an infrequent basis and analysis requests can be changed with a simple e-mail from the customer (SRR-E) to SRNL.

On October 09, 2020, Savannah River Remediation Engineering (SRR-E) delivered a Leak Detection Box Drain Cell (LDB-DC) sample identified as sample LIMS# 20195 to SRNL for characterization.

The Technical Task and Quality Assurance Plan (TTQAP) [1] outlines the planned analyses associated with this CSTF sample characterization scope. The tasks requested to be performed by SRNL are summarized in the Technical Task Request (TTR) [2].

2.0 Objectives

The primary objectives of this CSTF characterization effort (LDB-DC LIMS# 20195) were to analyze the LDB-DC sample, in triplicate, for the following: density, pH, total alpha, total beta and total gamma.

Other analyses performed include anions, total mercury and elementals. These secondary analysis results will be used to support residual sample disposal requirements in SRNL drains.

3.0 Experimental Setups/Sample Description and Preparations/Methodology

3.1 Leak Detection Box Drain Cell (LDB-DC) Sample Characterization.

The LDB-DC "as-received" CSTF sample was transferred from the original glass container into a separate labeled polymethyl pentene bottle. The total sample volume was about 80 mL. This sample was a clear liquid without any visible solids.

The LDB-DC "as-received" CSTF sample did not show any significant external measurable dosimetry readings. Therefore, aliquots of the sample were prepared in a SRNL radioactive hood, put directly into green Shielded Cell bottles and sent to the Analytical Research and Development (ARD) group for characterizations without any acid or water dilutions.

3.2 Format of the Reported Results

The mean results, based on the average of the applicable analytical determinations, are reported in this document, along with the percent relative standard deviation (%RSD). The %RSD provides an indication of the measurement variation between duplicate measurements but is typically not an indicator of analytical accuracy. In general, the one sigma analytical uncertainty as reported by ARD was 10%, although it was sometimes lower or higher. Specifically, the one sigma analytical uncertainties reported were: a) ~10% for base titration, IC, pH and ICP-AES; b) ~20% for Direct Mercury Analyses (DMA); and c) ~5% for Cs-137 determined by gamma spectroscopy and total alpha, and beta by Liquid Scintillation Counting (LSC). As such, only one to two of the leading digits reported for the analysis results should be considered significant.

A summary of the analytical methods used in these sample characterizations is presented in Appendix A. Appendix B contains the Laboratory Information Management System (LIMS) numbers for tracking the analytical data presented in this report. The sample analysis completion dates are tracked in LIMS.

In the LDB-DC sample characterization results presented below, values preceded by "<" (less than sign) indicate values were below minimum detection limits (MDLs). Values preceded by "≤" (less than or equal to sign) are indicated as having replicate results in which at least one of the analysis values was above the instrument detection limit or MDL and at least one of the analysis values was below the detection limit or was an upper limit. Thus, where replicate analyses were both above and below the detection limit, the average of all replicates above and below the detection limit is given and a "≤ "sign precedes the average value. The standard deviations were calculated only for values that were all above the detection limits. The minimum detectable activity (MDA) is defined as the value above which the 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 Inductively Coupled Plasma–Atomic Emission Spectroscopy (ICP-AES) analyses is equivalent to three times the standard deviation of the blank measurements.

4.0 Results and Discussion

Laboratory analyses were performed on the LDB-DC sample identified as sample LIMS# 20195 and most of the customer requested analyses were performed in triplicate.

The measured density for this LDB-DC sample was 0.98 (0.22 % RSD) and the pH was 7.66.

The measured cesium-137 average activity in the LDB-DC Sample, as shown in Table 1, was 2.52E+04 dpm/mL (1.95%RSD). The corresponding average Ba-137^{m} activity, calculated as 94.7% of the average Cs-137 values, is 2.39E+04 dpm/mL (1.95 %RSD). The average cesium-134 was reported to be <5.35 dpm/mL (below the instrument detection limit).

The total alpha activities measured for the LDB-DC sub-sample were less than values (upper limits) because of possible spectral interferences or because there is not much alpha activity in the sample. Thus, total alpha activities averaged <1.74E+02 dpm/mL.

The total beta activities in the LDB-DC sub-sample were above the instrument detection limits and averaged 3.08E+04 (3.25E-05 %RSD) dpm/mL.

Taking into consideration the total beta activity measured in the sample (3.08E+04 dpm/mL) and the total activity of the Ba-137m gamma-emitting daughter in radiological equilibrium with Cs-137, as calculated from the gamma assay (2.39E+04 dpm/mL), the total activity of the beta and gamma emitting radionuclides measured within the uncertainty of the analyses equals 5.47E+04 dpm/mL (3.08E+04 + 2.39E+04 dpm/mL). This total is still about an order of magnitude less than 8.69E+05 dpm/mL, which is the procedural limit for the LDB-DC sample.

Table 1 Total alpha, Total beta and Total gamma Radiological Results for LDB-DC Sample LIMS# 20195

Analyte	Analysis-1 dpm/mL	Analysis-2 dpm/mL	Analysis-3 dpm/mL	Average dpm/mL	St.Dev.	%RSD, N =3*
Total alpha	<1.77E+02	<1.72E+02	<1.72E+02	**<1.74E+02**	n/a	n/a
Total beta	3.07E+04	3.09E+04	3.08E+04	**3.08E+04**	*1.00E+02*	3.25E-01
Cs-134	<5.54E+00	<5.10E+00	<5.40E+00	**<5.35E+00**	n/a	n/a
Cs-137	2.58E+04	2.49E+04	2.50E+04	**2.52E+04**	*4.93E+02*	1.95
@Ba-137	2.44E+04	2.36E+04	2.37E+04	**2.39E+04**	*4.67E+02*	1.95E+00

*Analysis performed in triplicate. n/a = not applicable. @ Ba-137 is calculated as 94.7 % of Cs-137 activity.

Auxiliary analyses performed on this LDB DC sample, used in support of residual sample disposal at SRNL High Activity Drains (HAD), included total mercury, anions (chloride) and elemental analysis. Results for these analyses are presented in Table 2 for elemental analysis and mercury and Table 3 for the anion results.

As presented in Table 3, sulfate (2.89 ± 0.01 mg/L), nitrate (2.64 ± 0.35 mg/L), phosphate (1.59 ± 0.04 mg/L) and chloride (1.18 ± 0.02 mg/L) were the four predominant anions in the LDB-DC sample. All other anions were below instrument detection limits. The total mercury concentration in the LDB-DC sample was below the instrument detection limit (<0.01 mg/L).

The predominant cation concentrations in the LDB-DC sample are Na [26.35 ± 1.34 mg/L (5.10 %RSD)], K [23.5 ±0.49 mg/L (2.13 %RSD)], Ca [3.57 ± 0.01 mg/L (0.40 %RSD)] and Mg [≤0.40 mg/L]. As shown in Table 2, the concentrations of other cations are below the instrument detection limits.

Table 2 Elemental Analyses of LDB-DC Sample LIMS# 20195

Element	Average, mg/L	St.Dev.	%RSD, N = 2*
Ag	**<0.17**	n/a	n/a
Al	**<0.24**	n/a	n/a
B	**<0.33**	n/a	n/a
Ba	**<0.09**	n/a	n/a
Be	**<0.01**	n/a	n/a
Ca	**3.57**	*0.01*	0.40
Cd	**<0.14**	n/a	n/a
Ce	**<0.72**	n/a	n/a
Co	**<0.18**	n/a	n/a
Cr	**<0.14**	n/a	n/a
Cu	**<0.40**	n/a	n/a
Fe	**<0.10**	n/a	n/a
Gd	**<0.12**	n/a	n/a
K	**23.25**	*0.49*	2.13
La	**<0.07**	n/a	n/a
Li	**0.19**	n/a	n/a
Mg	**≤0.40**	n/a	n/a
Mn	**<0.35**	n/a	n/a
Mo	**<0.57**	n/a	n/a
Na	**26.35**	*1.34*	5.10
Ni	**<0.88**	n/a	n/a
P	**<3.06**	n/a	n/a
Pb	**<1.21**	n/a	n/a
Sb	**<2.31**	n/a	n/a
Si	**<1.75**	n/a	n/a
Sn	**<1.06**	n/a	n/a
Sr	**<0.03**	n/a	n/a
Th	**<0.46**	n/a	n/a
Ti	**<0.04**	n/a	n/a
U	**<2.51**	n/a	n/a
V	**<0.10**	n/a	n/a
Zn	**<0.10**	n/a	n/a
Zr	**<0.06**	n/a	n/a
Hg	**<0.01**	n/a	n/a

*Analysis performed in duplicate. n/a = not applicable.

Table 3 Chromatography Analyses Results for LDB-DC Sample LIMS# 20195

Analyte	Analysis-1, mg/L	Analysis-2, mg/L	Average, mg/L	St.Dev.	%RSD, N = 2*
Fluoride, F^-	<1	<1	<1	n/a	n/a
Formate, HCO_2^{1-}	<1	<1	<1	n/a	n/a
Chloride, Cl^-	1.16	1.19	**1.18**	*0.02*	1.81
Nitrite, NO_2^-	<1	<1	<1	n/a	n/a
Nitrate, NO_3^-	2.89	2.39	**2.64**	*0.35*	13.39
Phosphate, PO_4^{3-}	1.62	1.56	**1.59**	*0.04*	2.67
Sulfate, SO_4^{2-}	2.9	2.88	**2.89**	*0.01*	0.49
Oxalate, $C_2O_4^{2-}$	<1	<1	<1	n/a	n/a
Bromide, Br^{1-}	<1	<1	<1	n/a	n/a
pH	7.66	7.66	**7.66**	n/a	n/a

*Analysis performed in duplicate. n/a = not applicable.

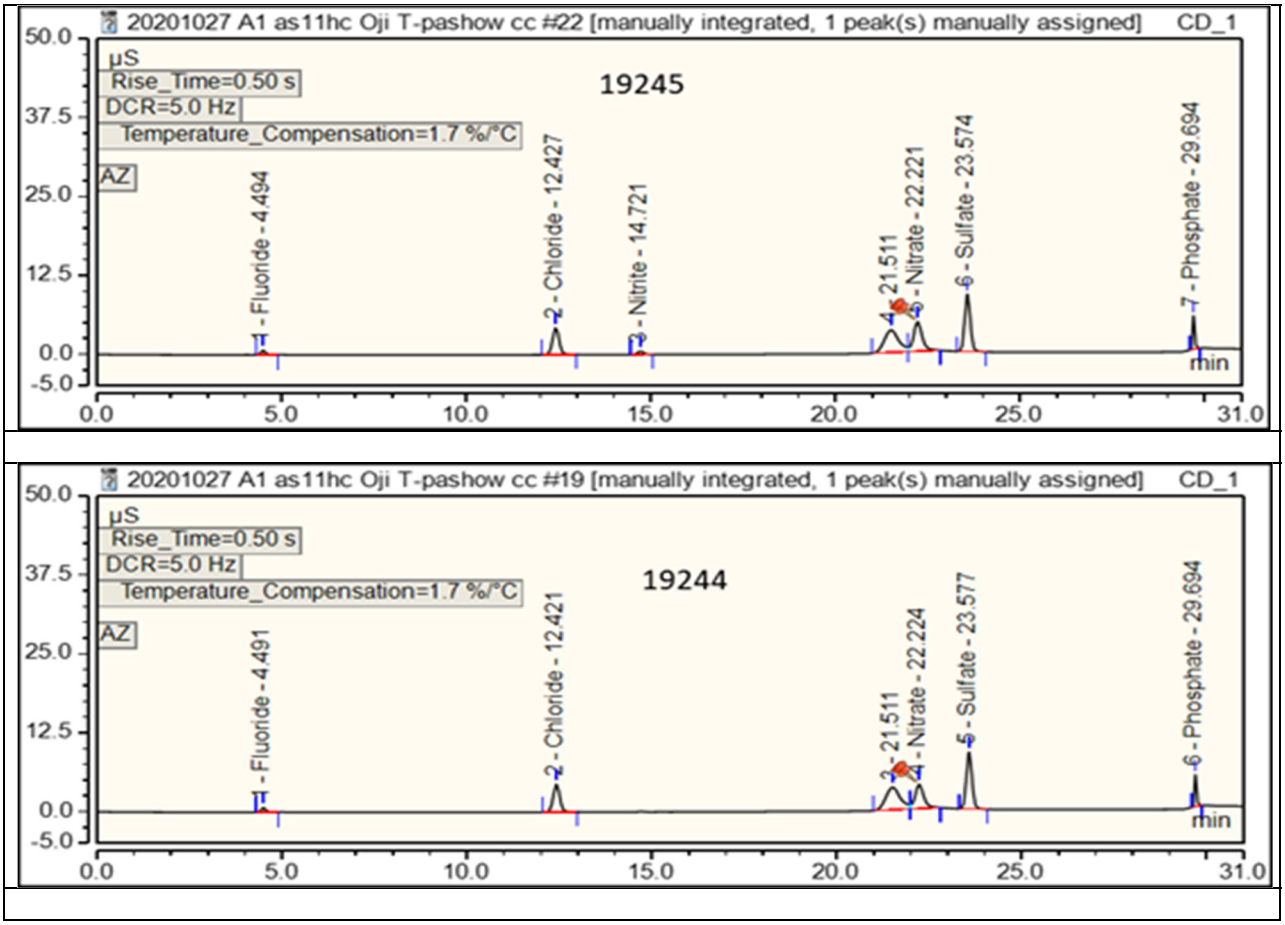

Figure 1. **LDB-DC Sample Characterization-IC chromatograms for samples LW19244 and LW19245.**

5.0 Conclusions

On October 09, 2020, SRR-E delivered a LDB-DC sample identified as sample LIMS# 20195 to the SRNL for characterization.

Most of the LDB-DC sample analyses were performed in triplicate with the %RSD, a measure of the variability between each duplicate data set expressed as a percentage. This %RSD values were used to estimate the analytical result quality for each analyte. A summary of the average analytical results based on one sigma analytical uncertainty for the LDB sample includes the following.

The measured density of the LDB-DC "as-received" CSTF sample is 0.98 (0.22 %RSD) and the directly measured pH was 7.66.

The total alpha activity for the LDB-DC sample was less than value (upper limits) because of possible spectral interferences or because there is not much alpha activity in the sample. Thus, total alpha activities averaged <1.74E+02 dpm/mL.

The total beta in the LDB-DC sample was above the instrument detection limits and averaged 3.08E+04 (3.25E-01 %RSD) dpm/mL.

The measured cesium-137 average activity (total gamma) in the LDB-DC sample was 2.52E+04 dpm/mL (1.95%RSD). The corresponding average $Ba\text{-}137^{m}$ activity, calculated as 94.7% of the Cs-137 values, is 2.39E+04 dpm/mL (1.95 %RSD). Cesium 134, at an average activity of < 5.35 dpm/mL, was below the instrument detection limit. Therefore, the total activity of the beta and gamma emitting radionuclides (3.08E+04 + 2.39E+04 dpm/mL) measured within the uncertainty of the analyses equals 5.47E+04 dpm/mL, which is still about an order of magnitude less than 8.69E+05 dpm/mL, which is the procedural limit for the LDB-DC sample.

Sulfate (2.89 ± 0.01 mg/L), nitrate (2.64 ± 0.35 mg/L), phosphate (1.59 ± 0.04 mg/L) and chloride (1.18 ± 0.02 mg/L) were the four predominant anions in the LDB-DC sample. All other anions were below the instrument detection limits and the total mercury concentration in the sample was below the instrument detection limit (<0.01 mg/L).

The predominant cation concentrations in the LDB-DC sample are Na [26.35 ± 1.34 mg/L (5.10 % RSD)], K [23.5 ± 0.49 mg/L (2.13 % RSD)], Ca [3.57 ± 0.01 mg/L (0.40%RSD)] and Mg [≤0.40 mg/L]. The concentrations of other cations were below the instrument detection limits.

6.0 Quality Assurance

The TTQAP details the planned activities and associated quality assurance implementing procedures for the characterization of the LDB-DC sample (TTQAP, SRNL-RP-2020-00565, Rev. 0, October 12, 2020) [1]. The documents referenced in the TTQAP include the following: L. N. Oji: ELN: L5575-00080-14 (Electronic Notebook (Production); SRNL, Aiken, SC 29808 (2014) and in various ARD notebooks contain the analytical data. Other relevant QA documents include the TTR (X-TTR-H-00101, Rev 0, August. 05, 2020)[3].

Requirements for performing reviews of technical reports and the extent of review are established in manual E7 2.60. This document, including all calculations was reviewed by Design Verification by Document Review [4,5]. SRNL documents the extent and type of review using the SRNL Technical Report Design Checklist contained in WSRC-IM-2002-00011, Rev. 2.

The TTR requested functional classification “Safety Class” and the report, calculations and technical memoranda issued in this CSTF sample characterization effort have received technical review by design verification (E7 Manual, Procedure 2.60, section 5.3). The experimental work, the analyses, and peer check all comply with the customer quality assurance (QA) requirements.

7.0 References

1. L. N. Oji, S. Lucatero “Task Technical and Quality Assurance Plan for the Analysis of Infrequent Samples from the Concentration, Storage, and Transfer Facility” SRNL-RP-2020-00565, Rev. 0, October 12, 2020.
2. Azikiwe Hooker, “Infrequent CSTF samples” Technical Task Request X-TTR-H-00101, August 5, 2020.
3. L. N. Oji: ELN: L5575-00080-14 (Electronic Notebook (Production); SRNL, Aiken, SC 29808 (2014).

4 “Savannah River National Laboratory Technical Report Design Check Guidelines”, WSRC-IM-2002-00011, Revision 2, August 2004.

5 “Technical Reviews”, Manual E7, Procedure 2.60, Revision 18, December 2, 2019.

Appendix A: Summary of Analytical Methods

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

Samples are diluted as necessary to bring analytes within the instrument calibration 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 corrections were applied to the results.

Ion Chromatography for Anions (IC-Anions)

A three-point calibration curve is analyzed daily on the instrument with concentrations of 10, 25 and 50 µg/mL. For IC Anions, samples are diluted (LDB-DC sample aliquots are each diluted by a factor of ~21; on a volume basis) with a carbonate/bicarbonate diluent as necessary to bring analytes to within instrument calibration. IC was performed on the diluted supernatant aliquots, to quantify bromide, chloride, fluoride, formate, nitrate, nitrite, oxalate, phosphate, and sulfate.

Total mercury was analyzed by DMA.

The Direct Mercury Analysis (DMA) method is used for total mercury determinations where controlled heating in an oxygenated decomposition furnace is used to liberate mercury from solid and aqueous samples in the instrument. The sample is dried and then thermally and chemically decomposed inside the decomposition furnace. The decomposition products are carried by flowing oxygen to the catalytic section of the furnace. With the completion of oxidation, halogens and nitrogen/sulfur oxides are trapped. The remaining decomposition products are then carried to an amalgamator that selectively traps mercury. After the system is flushed with oxygen to remove any remaining gases or decomposition products, the amalgamator is rapidly heated, releasing mercury vapor. Flowing oxygen carries the mercury vapor through absorbance cells positioned in the light path of a single wavelength atomic absorption spectrophotometer. Absorbance (peak height or peak area) is measured at 253.7 nm as a function of mercury concentration.

The typical working range for this DMA method is 0.05 - 600 ng. The mercury vapor is first carried through a long pathlength absorbance cell and then a short pathlength absorbance cell. The lengths of the first cell and the second cell are in a ratio of 10:1 or another appropriate ratio. The same quantity of mercury is measured twice, using two different sensitivities, resulting in a dynamic range that spans at least four orders of magnitude. The instrument detection limit (IDL) for this method is 0.01 ng of total mercury.

Gross Alpha/Gross Beta

Aliquots of the LDB-DC sample were added to a 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.

Cs-137, Cs-134

Aliquots of the LDB-DC sample were analyzed by coaxial high purity germanium gamma-ray spectrophotometers to measure Cs-137 and Cs-134. Two LDB-DC sample aliquots were acidified and diluted using ~3.0 M nitric acid. (HNO_3) Gamma spectroscopy was performed on the acidified/diluted LDB-DC sample aliquots to quantify Cs-137 and Cs-134. Laboratory reagent blanks (~3.0 M HNO_3 acid) are analyzed as controls.

Densities:

Density measurements were conducted at a temperature of ~26 °C. Densities were measured using weight-calibrated balances and 2.0 mL volume-calibrated glass test tubes. Three individual LDB-DC sample aliquots were used to obtain triplicate measurements. The density of de-ionized water was also determined as a reference for comparison.

Appendix B: LDB-DC Sample Characterization; ARD Tracking Numbers*

Analytes	Method (s)	SRNL ARD Tracking Number (LIMS):
Anions	IC	LW19244 to LW19245
pH	pH	LW19235 to LW19236
Elemental	ICP-AES	LW19241 to LW19242
Hg	DMA	LW19238 to LW19239
Total Alpha	LSC	LW19228 to LW19230
Total Beta	LSC	LW19228 to LW19230
Total gamma	GAMMA SPEC	LW19228 to LW19230

*Project: IDs: LW-AD-PROJ-201015-2

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