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# Analysis of Tank 4 (FTF-4-15-22, 23) Surface and Subsurface Supernatant Samples in Support of Enrichment Control, Corrosion Control and Evaporator Feed Qualification Programs

L. N. Oji

September 2015

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## EXECUTIVE SUMMARY

This report provides the results of analyses on Savannah River Site Tank 4 surface and subsurface supernatant liquid samples in support of the Enrichment Control Program (ECP), the Corrosion Control Program (CCP) and the Evaporator Feed Qualification (EFQ) Program. The purpose of the ECP sample taken from Tank 4 in August 2015 was to determine if the supernatant liquid would be “acceptable feed” to the 2H and 3H evaporator systems.

The U-235 mass divided by the total uranium mass averaged  $5.48\text{E-}03 \pm 1.19\text{E-}04$  ( $5.48\text{E-}01 \pm 1.19\text{E-}02$  % uranium enrichment) for the Tank 4 surface sample and averaged  $5.57\text{E-}03 \pm 2.18\text{E-}04$  ( $5.57\text{E-}01 \pm 2.18\text{E-}02$  % uranium enrichment) for the Tank 4 subsurface sample. The U-235 concentration in the Tank 4 surface and subsurface samples averaged, respectively,  $1.17\text{E-}02 \pm 3.01\text{E-}04$  mg/L and  $1.18\text{E-}02 \pm 4.11\text{E-}04$  mg/L, while the U-238 concentrations in the Tank 4 surface and subsurface samples averaged  $2.11\text{E+}00 \pm 1.05\text{E-}02$  mg/L and  $2.11\text{E+}00 \pm 1.43\text{E-}02$  mg/L, respectively. The total uranium concentrations in the Tank 4 surface and subsurface samples averaged  $2.13\text{E+}00 \pm 1.08\text{E-}02$  mg/L and  $2.12\text{E+}00 \pm 1.41\text{E-}02$ , respectively.

Hence, the U-235/total uranium ratios in Tank 4 surface and subsurface samples are in line with the prior 2H evaporator ECP samples, and the calculated U-235 equivalents are  $\leq 0.83$  and  $\leq 0.84$  wt% for the surface and subsurface samples, respectively. These U-235 equivalents also meet the Evaporator system feed requirements.

The measured sodium and silicon concentrations for the Tank 4 surface sample averaged, respectively, 2.38 M and 3.80 mg/L. The Tank 4 subsurface sodium and silicon concentrations were, respectively, 2.37 M and 5.53 mg/L. The measured aluminum and free-OH concentrations in the Tank 4 surface sample averaged 0.10 M and 0.71 M, respectively, while these values were 0.10 M and 0.71, respectively, for the subsurface samples.

## TABLE OF CONTENTS

LIST OF FIGURES .....	vi
LIST OF ABBREVIATIONS .....	vii
1.0 Introduction .....	1
2.0 Experimental .....	1
3.0 Analytical Results .....	3
4.0 Conclusions .....	5
5.0 Quality Assurance .....	6
6.0 References .....	6
Appendix A. Tank 4 Surface samples (FTF-4-15-22) .....	A-1
Appendix B. Tank 4 Sub-Surface samples (FTF-4-15-23) .....	B-1

## LIST OF TABLES

Table 1 Tank 4 Sample Description and Analysis Suite .....	2
Table 2 Tank 4 Sample Volume, Mass and Appearance .....	2
Table 3 ECP, CCP and EFQ Average Analytical Data for Tank 4 Supernatant Samples. ....	5
Table 4 Tank 4 Surface Sample FTF-4-15-22: ECP Results .....	A-1
Table 5 Tank 4 Surface Sample FTF-4-15-22: CCP/EFQ Results .....	A-1
Table 6 Tank 4 Surface Sample FTF-4-15-22: Other Results from ECP/CCP/EFQ .....	A-1
Table 7 Tank 4 Surface Sample (FTF-4-15-22): Selected Elemental Analysis Results .....	A-2
Table 8 Tank 4 Sub-Surface Sample FTF-4-15-23: ECP Results .....	B-1
Table 9 Tank 4 Sub-Surface Sample FTF-4-15-23: CCP/EFQ Results .....	B-1
Table 10 Tank 4 Sub-Surface Sample (FTF-4-15-23): Select Elemental Analysis Results .....	B-1
Table 11 Tank 4 Sub-Surface Sample FTF-4-15-23: Other Results from ECP/CCP/EFQ .....	B-2

## LIST OF FIGURES

Figure 1 Tank 4 Supernate Surface (FTF-4-15-22, left) and Subsurface (FTF-4-15-23 right). Samples. ...	3
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## **LIST OF ABBREVIATIONS**

AD	Analytical development
CCP	Corrosion Control Program
CV	Cold vapor
DWPF	Defense Waste processing Facility
ECP	Enrichment Control Program
EFQ	Evaporator Feed Qualification
HTF	H Tank Farm
IC	Ion Chromatography
ICP-MS	Inductively Coupled Plasma-Mass Spectrometry
ICP-ES	Inductively Coupled Plasma – Emission Spectrometry
SpG	Specific Gravity
SRNL	Savannah River National Laboratory
SRR	Savannah River Remediation
TIC	Total Inorganic Carbon
TTQAP	Task Technical and Quality Assurance Plan
WAC	Waste Acceptance Criteria



## 1.0 Introduction

Compositional feed limits have been established to ensure that a nuclear criticality event for the 2H and 3H Evaporators<sup>i</sup> is not possible. The limits are established by the Enrichment and Corrosion Control Programs. The Enrichment Control Program (ECP) requires feed sampling to determine the equivalent enriched uranium content prior to transfer of waste other than recycle transfers.<sup>ii</sup> The Corrosion Control Program (CCP) establishes concentration and temperature limits for key constituents and periodic sampling and analysis to confirm that waste supernate is within these limits.<sup>iii</sup>

In August 2015, Savannah River Remediation (SRR) retrieved tank samples from two locations within the Savannah Rivers Site (SRS) Tank 4, which is a current planned feed source tank for the evaporators. These two supernatant samples were delivered to the Savannah River National Laboratory (SRNL) on August 19, 2015 for analyses to support the ECP, CCP and Evaporator Feed Qualification (EFQ) Programs. As summarized in Table 1, the two Tank 4 samples were identified as FTF-04-15-22 (surface sample) and FTF-4-15-23 (subsurface sample), respectively. The surface sample was collected from a height of 136.1 inches from the tank bottom and the variable depth sample or subsurface sample was collected 42 inches from the tank bottom.

This work is governed by the Technical Task Request and the detailed experimental plan is presented in the Task Technical and Quality Assurance Plan.<sup>iv,v,vi</sup> Requirements for performing reviews of technical reports and the extent of review are established in Manual E7 Procedure 2.60. SRNL documents the extent and type of review using the SRNL Technical Report Design Checklist contained in WSRC-IM-2002-00011, Rev. 2.

## 2.0 Experimental

As shown in Figure 1, the two Tank 4 supernatant samples were essentially free of any visible settled insoluble solids and the solutions were relatively clear and transparent with a slightly cloudy appearance. In general, the visual appearance of the samples was consistent with supernatant liquid containing <1 wt. % insoluble solids based on experience with these sample types. Measured masses, approximate volumes and sample descriptions are provided in Table 2.

The ECP, CCP and EFQ analysis requirements for the Tank 4 slurry supernatant samples are summarized in Table 1. The ECP analysis includes inductively-coupled plasma-mass spectroscopy (ICP-MS) for uranium isotopic analysis and radiochemical separation and counting methods for Pu-238, Pu-239/240, and Pu-241. Sample preparation for ECP analyses involved dilution with 2M nitric acid. The CCP analysis included ion chromatography (IC) for anions (nitrate and nitrite), acid titration for free hydroxide, and gamma scan for detectable gamma-emitting isotopes. The sample preparation for IC, titration and gamma analysis involved dilution with de-ionized water. The density of each “as-received” sample was measured by determining the weight of 1.0 mL sample portions in triplicate and the specific gravity (SpG) was calculated from these density measurements relative to the density of water.

Samples used for I-129 and Tc-99 analysis (2 mL portions) were transferred from the SRNL shielded cells in green shielded bottles for analysis by the SRNL Analytical Development (AD) group without in-cell dilution of the original samples.

For the I-129 analysis, aliquots of these tank supernatant samples were diluted in acid and spiked with a potassium iodide (KI) carrier. The samples were decontaminated with strikes of actinide and ammonium molybdenum phosphate (AMP) resin to facilitate removal of interfering isotopes. Sodium sulfite was added to the material to reduce the iodine. Silver nitrate was added to the solution to precipitate the iodine as silver iodide (AgI), which was separated by filtration. The filtrate was analyzed for I-129

activity using low energy photon/x-ray, thin-windowed, semi-planar, high purity germanium detectors. 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.

For the Tc-99 analysis, aliquots of these tank supernatant samples were oxidized with nitric acid. Two matrix blanks, spiked with a Tc-99 standard and Tc-99m, were also prepared. The 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 sodium iodide (NaI)-well gamma spectrometer, and were used to correct the Tc-99 analyses for any technetium losses from the radiochemical separations. The percent recovery of one of the Tc-99 spiked matrix blanks was applied to the entire set of samples to calibrate the Liquid Scintillation Counting (LSC) analysis for Tc-99.

Analysis for mercury, although not requested by the customer, is required for residual waste disposal purposes. Aliquots of ten-fold diluted, unfiltered original samples were submitted to AD for mercury analysis by a cold vapor (CV) Hg digestion method. This digested CV mercury method is an extension of the standard SRNL mercury analysis method. This modified method ensures that any organo-mercuric compounds, if present in the sample matrix, are converted to elemental mercury vapor which the instrument can detect in a flameless atomic absorption technique at 253.7 nm. This modified analysis method for mercury involves addition of various organo-mercuric digestion reagents including concentrated sulfuric acid, concentrated nitric acid and potassium permanganate to the original samples before CVHg analysis (See SRNL AD procedure for mercury analysis).

All of the analyses were performed and reported in triplicate as shown in Appendices A and B and the averages and standard deviations for the analytical results are presented in Table 3.

**Table 1** Tank 4 Sample Description and Analysis Suite.

Sample	Sample ID	Sample location	SRNL Receipt Date	Date Sample was transferred into Shielded Cells
Tank 4 surface	FTF-4-15-22	Collected 136.1 inches from the tank bottom.	8/19/2015	8/24/2015
Tank 4 subsurface	FTF-4-15-23	Collected 42 inches from the tank bottom.		
Sample location		Analysis Suite summary		
Tank 4 Surface sample		ECP, CCP and EFQ Warm acid strike/ICPES for Si		
Tank 4 Subsurface sample (variable depth sample)		ECP and EFQ Warm acid strike/ICPES for Si		

**Table 2** Tank 4 Sample Volume, Mass and Appearance

Tank Sample ID	Sample location	Approx. Volume, mL	Mass, g	Clarity of supernate
FTF-4-15-22	surface sample	60	67.969	clear supernate, no visible solids
FTF-4-15-23	subsurface sample	80	87.871	clear supernate, no visible settled solids

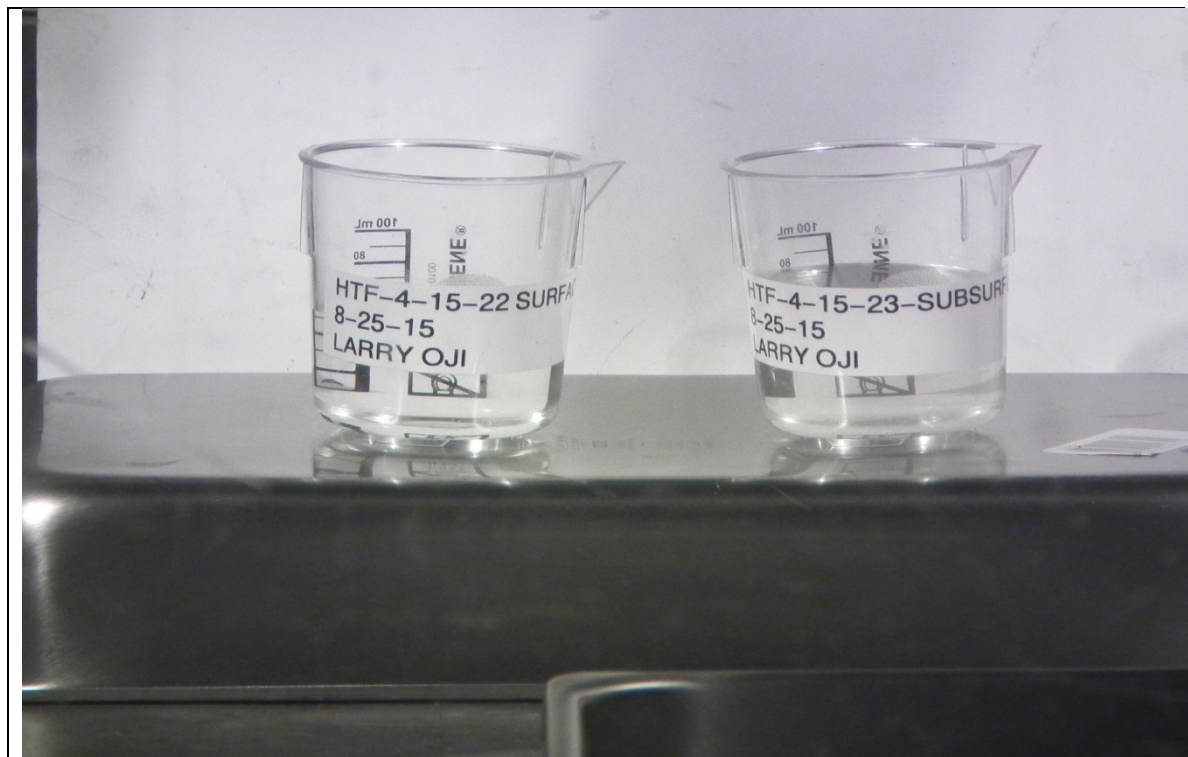


Figure 1 Tank 4 Supernate Surface (FTF-4-15-22, left) and Subsurface (FTF-4-15-23 right). Samples.

### 3.0 Analytical Results

Table 2 contains a description of the sampling location and the quantity of material received for the “as-received” Tank 4 surface and subsurface samples. As shown in Figure 1, these samples were essentially free of any visible settled insoluble solids. These Tank 4 samples (FTF-4-15-22, 23) were relatively clear with a slightly hazy and cloudy appearance. In general, the visual appearance of these samples was consistent with supernatant liquid containing <1 wt. % insoluble solids.

Table 3 contains a summary of the ECP/CCP/EFQ analytical results for the Tank 4 surface and subsurface samples. This summary table includes only the average values for the analytes and the standard deviations for each triplicate analysis. Analyses for selected cations in the Tank 4 supernatant samples, which were not requested by the customer, are also reported (performed to support cation/anion balance calculations and residual sample disposal requirements)

Results for the analytes that were below the limits of quantification are preceded by “<” and values preceded by “≤” (less than or equal to sign) indicate that at least one of the analytical replicates was above the instrument detection limit and at least one of the analytical results was below the detection limit or was an upper limit. Thus, where replicate analyses gave values 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 and provided only for values that were all above the detection limits.

The Pu-239 value reported in mg/L for the ECP analysis assumes that all of the activity measured as Pu-239/240 is from Pu-239. This assumption results in a high bias to the Pu-239 result and thus the

assumption is conservative with respect to the concentration of this fissile isotope. All measurements reported for U-233, U-234 and U-236 for Tank 4 samples are below the ICP-MS detection limit. The uranium enrichment calculations are based on U-total; where U-total includes only the masses of uranium isotopes U-235 and U-238.

To check the results, a cation-anion normality balance was performed. The normal concentrations of cations (mainly  $\text{Na}^+$  and  $\text{K}^+$ ) were summed, as were the anions ( $\text{NO}_3^-$ ,  $\text{NO}_2^-$ ,  $\text{SO}_4^{2-}$ ,  $\text{Cl}^-$ ,  $\text{CO}_3^{2-}$ ,  $\text{PO}_4^{3-}$ ,  $\text{AlO}_2^-$ ,  $\text{C}_2\text{O}_4^{2-}$  and free  $\text{OH}^-$ ). The two sums were compared. For these comparisons, the primary contributing cations included  $\text{Na}^+$  and  $\text{K}^+$ , while the primary contributing anions included hydroxide, nitrite, nitrate, carbonate, formate, sulfate, phosphate, oxalate, chloride, and aluminate.

For the Tank 4 surface and subsurface samples the cations summed to 2.38 and 2.38 M, respectively, while the anions summed, respectively, to 2.25 and 2.26 M. Thus, the anions summed to about 94.5 % and 95.1 % of the cations, respectively. The difference between the cation and anion molarity values are within 6% of each other, which is relatively good when one takes into consideration the nominal uncertainties (1 sigma) for the analytical methods. The difference can be attributed to the analytical uncertainties and the cation/anion concentrations are considered to be the same based on the uncertainties.

Tables 4 through 11 in Appendices A and B contain all the analytical results for the characterization of Tank 4 surface and subsurface samples. These detailed analyses results are grouped by the required programs (ECP/CCP/EFQ) in separate sections of the tables.

The U-235 concentration in the Tank 4 surface and subsurface samples averaged  $1.17\text{E}-02 \pm 3.01\text{E}-04$  and  $1.18\text{E}-02 \pm 4.11\text{E}-04$  mg/L, respectively, while the U-238 concentrations in Tank 4 surface and subsurface samples averaged, respectively,  $2.11\text{E}+00 \pm 1.05\text{E}-02$  and  $2.11\text{E}+00 \pm 1.43\text{E}-02$  mg/L. The total uranium concentration in the Tank 4 surface and subsurface samples averaged  $2.13\text{E}+00 \pm 1.08\text{E}-02$  and  $2.12\text{E}+00 \pm 1.41\text{E}-02$  mg/L, respectively. Thus, the U-235/total uranium ratios ( $5.48\text{E}-03 \pm 1.19\text{E}-04$  and  $5.57\text{E}-03 \pm 2.18\text{E}-04$ ) in the Tank 4 surface and subsurface samples are in line with the prior 2H evaporator ECP samples<sup>a</sup>.

Based on the uranium, Pu-241 and Pu-239 concentrations, the calculated U-235 equivalent<sup>b</sup>  $\left[\frac{([U-235] + 1.4*[U-233] + 2.25*([Pu-241] + [Pu-239]))}{[\text{total uranium}]}*100\right]$  is  $\leq 0.83$  wt% for the Tank 4 surface sample and  $\leq 0.84$  wt% for the Tank 4 subsurface sample. It is worth noting that the evaporator system feed requirements, waste that has a U-235 equivalent enrichment of  $\leq 5.5$  wt% and a plutonium content of the fissionable elements of  $\leq 2$  wt%, are met.

Technetium was present at detectable levels in the Tank 4 surface and subsurface samples. The current Saltstone Tc-99 WAC limit is  $8.7\text{E}+04$  pCi/mL. In the next Saltstone WAC revision for Tc-99, this limit will be raised to  $2.11\text{E}+05$  pCi/mL.<sup>c</sup>

All I-129 analysis results for Tank 4 were present at detectable levels, with only the run two for the subsurface sample giving an upper limit value due to incomplete separation of I-129 from Cs-137. However, the analysis result for this run two (Tank 4 subsurface sample) was still less than the 63 pCi/mL customer desired Saltstone WAC limit. All other I-129 analysis for tank 4 samples meet the customer desired Saltstone Waste Acceptance Criteria (WAC) of 63 pCi/mL (140 dpm/mL) for I-129 in Tank 4.

<sup>a</sup> C. J. Martino, "Analysis of Tank 38H (HTF-38-13-156, 157) and Tank 43H (HTF-43-13-158, 159) Samples for Support of the Enrichment Control and Corrosion Control Programs," SRNL-TR-2013-00205, Rev. 0, October 2013.

<sup>b</sup> Section 1.1.2 of the Implementation Requirements and Actions of Section 4.3 of WSRC-TR-2003-00055, Rev. 9

<sup>c</sup> S. P. Hommel, "Recommended Tc-99 Concentration for Saltstone Waste Acceptance Criteria to Implement the FY2014 SDF SA," SRR-CWDA-2015-00007, January 12, 2015.

**Table 3 ECP, CCP and EFQ Average Analytical Data for Tank 4 Supernatant Samples.**

Analytes	Tank 4 Surface HTF-4-15-22		Tank 4 Subsurface HTF-4-15-23		Methods	Units
	Average	Stdev.	Average	Stdev.		
U-233	<2.24E-03	-	<2.22E-03	-	ICP-MS	mg/L
U-234	<2.24E-03	-	<2.22E-03	-	ICP-MS	mg/L
U-235	1.17E-02	3.01E-04	1.18E-02	4.11E-04	ICP-MS	mg/L
U-236	<2.24E-03	-	<2.22E-03	-	ICP-MS	mg/L
U-238	2.11E+00	1.05E-02	2.11E+00	1.43E-02	ICP-MS	mg/L
Total U	2.13E+00	1.08E-02	2.12E+00	1.41E-02	ICP-MS	mg/L
U-235/U-total	5.48E-03	1.19E-04	5.57E-03	2.18E-04	Calc.	-
Pu-238	8.25E-06	3.19E-07	7.64E-06	2.97E-07	PuTTA	mg/L
Pu-239**	1.12E-03	1.23E-04	1.15E-03	1.11E-04	PuTTA	mg/L
Pu-239/240	1.54E+02	1.70E+01	1.58E+02	1.53E+01	PuTTA	dpm/mL
Pu-241	<1.65E-06	-	<1.46E-06	-	Pu-238/241	mg/L
Cs-137	3.31E+08	7.80E+06	3.23E+08	1.61E+07	gamma scan	dpm/mL
Ba-137m	3.12E+08	6.48E+06	3.05E+08	1.52E+07	gamma scan	dpm/mL
Tc-99	3.56E+04	2.35E+03	3.74E+04	1.15E+03	Tc-99	pCi/mL
I-129	1.26E+01	3.06E+00	≤1.09E+01	-	I-129	pCi/mL
OH <sup>-</sup>	0.709	0.02	0.713	0.03	Titration	M
NO <sub>2</sub> <sup>-</sup>	3.828E-01	1.630E-03	3.884E-01	6.692E-03	IC	M
NO <sub>3</sub> <sup>-</sup>	3.016E-01	4.752E-04	3.013E-01	5.793E-03	IC	M
F <sup>-</sup>	<0.006	-	<0.006	-	IC	M
CHO <sub>2</sub> <sup>-</sup>	<0.003	-	<0.002	-	IC	M
Cl <sup>-</sup>	0.006	0.000	0.006	0.000	IC	M
PO <sub>4</sub> <sup>3-</sup>	<0.001	-	<0.001	-	IC	M
SO <sub>4</sub> <sup>2-</sup>	0.087	0.002	0.085	0.002	IC	M
C <sub>2</sub> O <sub>4</sub> <sup>2-</sup>	0.031	0.001	0.031	0.001	IC	M
Br <sup>-</sup>	<0.007	-	0.007	-	IC	M
CO <sub>3</sub> <sup>2-</sup>	0.247	0.002	0.250	0.005	TIC	M
Al	2687.7	6.35	2741.7	0.00	ICP-ES	mg/L
B	11.14	0.15	10.90	0.17	ICP-ES	mg/L
Ca	2.49	1.44	1.31	1.06	ICP-ES	mg/L
Cr	71.17	0.44	70.82	0.11	ICP-ES	mg/L
Fe	3.55	0.72	10.53	0.53	ICP-ES	mg/L
K	272.43	10.22	278.98	1.70	ICP-ES	mg/L
La	0.52	0.07	0.56	0.05	ICP-ES	mg/L
Mo	13.27	1.04	11.88	0.84	ICP-ES	mg/L
Na	54376.7	276.83	54538	256.34	ICP-ES	mg/L
P	50.71	3.58	52.73	0.78	ICP-ES	mg/L
S	2779.3	165.12	3041.0	167.97	ICP-ES	mg/L
Si	3.80	0.80	5.53	0.41	ICP-ES	mg/L
V	0.75	0.10	0.83	0.08	ICP-ES	mg/L
Hg	6.12E+01	5.59E-01	4.82E+01	2.20E-01	CVAA-Hg	mg/L
Na	2.375E+00	1.209E-02	2.372E+00	1.115E-02	ICP-ES	M
Total cation	2.381	-	2.379	-	Calc.	M
Total anion	2.249	-	2.262	-	Calc.	M
SpG 25 °C	1.10	0.002	1.11	0.01	Calc.	-

\*\* The Pu-239 mass concentrations were calculated from the Pu-239/240 results, based on the assumption that all activity was due to Pu-239 (as opposed to Pu-240). Note that the ICP-MS results for Pu-239 were all below the minimum detection limits.

#### 4.0 Conclusions

The U-235 mass divided by the total uranium mass averaged  $5.48\text{E-}03 \pm 1.19\text{E-}04$  ( $5.48\text{E-}01 \pm 1.19\text{E-}02$  % uranium enrichment) for the Tank 4 surface sample and averaged  $5.57\text{E-}03 \pm 2.18\text{E-}04$  ( $5.57\text{E-}01 \pm 2.18\text{E-}02$  % uranium enrichment) for the Tank 4 subsurface sample. The U-235 concentration in the Tank 4 surface and subsurface samples averaged, respectively,  $1.17\text{E-}02 \pm 3.01\text{E-}04$  mg/L and  $1.18\text{E-}02 \pm 4.11\text{E-}04$  mg/L, while the U-238 concentrations in the Tank 4 surface and subsurface samples averaged

$2.11\text{E}+00 \pm 1.05\text{E}-02$  mg/L and  $2.11\text{E}+00 \pm 1.43\text{E}-02$  mg/L, respectively. The total uranium concentrations in the Tank 4 surface and subsurface samples averaged  $2.13\text{E}+00 \pm 1.08\text{E}-02$  mg/L and  $2.12\text{E}+00 \pm 1.41\text{E}-02$ , respectively.

Hence, the U-235/total uranium ratios in Tank 4 surface and subsurface samples are in line with the prior 2H evaporator ECP samples, and the calculated U-235 equivalents are, respectively  $\leq 0.83$  and  $\leq 0.84$  wt% for the Tank 4 surface and subsurface samples, which meets the evaporator system feed requirements.

The measured sodium and silicon concentrations for the Tank 4 surface sample averaged, respectively, 2.38 M and 3.80 mg/L. The Tank 4 subsurface sodium and silicon concentrations were, respectively, 2.37 M and 5.53 mg/L. The measured aluminum and free-OH concentrations in Tank 4 surface sample averaged 0.10 M and 0.71 M, respectively, while these values were 0.10 M and 0.71, respectively, for the subsurface samples.

## 5.0 Quality Assurance

Data are recorded in SRNL Electronic Notebook: L5575-00080 SRNL Electronic Notebook (Production); SRNL, Aiken, SC 29808 (2014) and various AD notebooks contain the analytical/experimental data.

## 6.0 References

- <sup>i</sup> D. A. Eghbali, "Nuclear Criticality Safety Evaluation: Operation of the 2H Evaporator System," N-NCS-H-00180, Rev. 0, September 2008.
- <sup>ii</sup> H. Bui, "CSTF Evaporator Feed Qualification Program," WSRC-TR-2003-00055, Rev. 9, November 19, 2014..
- <sup>iii</sup> K. B. Martin, "CSTF Corrosion Control Program," WSRC-TR-2002-00327, Rev. 8, July 22, 2014..
- <sup>iv</sup> H. Bui, "Tank 51 Enrichment Sample Analysis," X-TTR-H-00058, Rev. 0, June 02, 2015.
- <sup>v</sup> C. J. Martino, "Task Technical and Quality Assurance Plan for Analysis of Tank 38H and Tank 43H Enrichment Control Program and Corrosion Control Samples," SRNL-RP-2013-00522, Rev. 0, August 2013.
- <sup>vi</sup> J. R. Jacobs, "Tank 4 and 7 Corrosion Control/Enrichment Control and Evaporator feed Qualification Sample Analysis," X-TTR-F-00005, Revision 0, Dec. 4, 2014.

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## Appendix A. Tank 4 Surface samples (FTF-4-15-22)

**Table 4 Tank 4 Surface Sample FTF-4-15-22: ECP Results**

Analytes	Analysis-1	Analysis-2	Analysis-3	Average	St. Deviation	Units
U-233	<2.21E-03	<2.27E-03	<2.25E-03	<b>&lt;2.24E-03</b>	-	mg/L
U-234	<2.21E-03	<2.27E-03	<2.25E-03	<b>&lt;2.24E-03</b>	-	mg/L
U-235	1.17E-02	1.13E-02	1.19E-02	<b>1.17E-02</b>	3.01E-04	mg/L
U-236	<2.21E-03	<2.27E-03	<2.25E-03	<b>&lt;2.24E-03</b>	-	mg/L
U-238	2.11E+00	2.11E+00	2.13E+00	<b>2.11E+00</b>	1.05E-02	mg/L
U-Total	2.12E+00	2.12E+00	2.14E+00	<b>2.13E+00</b>	1.08E-02	mg/L
U-235/U-total	5.52E-03	5.35E-03	5.58E-03	<b>5.48E-03</b>	1.19E-04	-
Pu-239	9.76E-04	1.21E-03	1.17E-03	<b>1.12E-03</b>	1.23E-04	mg/L
Pu-241	<1.89E-06	<1.59E-06	<1.47E-06	<b>&lt;1.65E-06</b>	-	mg/L

**Table 5 Tank 4 Surface Sample FTF-4-15-22: CCP/EFQ Results**

Analytes	Analysis-1	Analysis-2	Analysis-3	Average	St. Deviation	Units
NO <sub>3</sub> <sup>-</sup>	3.01E-01	3.02E-01	3.02E-01	<b>3.02E-01</b>	4.75E-04	Mole/L
NO <sub>2</sub> <sup>-</sup>	3.82E-01	3.85E-01	3.82E-01	<b>3.83E-01</b>	1.63E-03	Mole/L
OH <sup>-</sup>	0.690	0.736	0.700	<b>0.709</b>	0.020	Molar
SpG @ 25 °C	1.10	1.10	1.10	<b>1.10</b>	0.00	-
Cs-137	3.22E+08	3.35E+08	3.36E+08	<b>3.31E+08</b>	7.80E+06	dpm/mL
Ba-137m	3.05E+08	3.17E+08	3.15E+08	<b>3.12E+08</b>	6.48E+06	dpm/mL

SpG = Specific gravity

**Table 6 Tank 4 Surface Sample FTF-4-15-22: Other Results from ECP/CCP/EFQ**

Analytes	Analysis-1	Analysis-2	Analysis-3	Average	St. Deviation	Units
U-235/U-total *100	5.52E-01	5.35E-01	5.58E-01	<b>5.48E-01</b>	1.19E-02	%
Pu-238	8.11E-06	8.61E-06	8.02E-06	<b>8.25E-06</b>	3.19E-07	mg/L
Pu-239/240	1.35E+02	1.67E+02	1.61E+02	<b>1.54E+02</b>	1.70E+01	dpm/mL
I-129	1.45E+01	1.42E+01	9.10E+00	<b>1.26E+01</b>	3.06E+00	pCi/mL
Tc-99	3.36E+04	3.82E+04	3.49E+04	<b>3.56E+04</b>	2.35E+03	pCi/mL
SO <sub>4</sub> <sup>2-</sup>	0.087	0.086	0.089	<b>0.087</b>	0.002	Mole/L
CHO <sub>2</sub> <sup>-</sup>	<0.002	<0.003	<0.003	<b>&lt;0.003</b>	-	Mole/L
Cl <sup>-</sup>	0.006	0.006	0.006	<b>0.006</b>	0.000	Mole/L
F <sup>-</sup>	<0.006	<0.006	<0.006	<b>&lt;0.006</b>	-	Mole/L
PO <sub>4</sub> <sup>3-</sup>	<0.001	<0.001	<0.001	<b>&lt;0.001</b>	-	Mole/L
C <sub>2</sub> O <sub>4</sub> <sup>2-</sup>	0.032	0.031	0.031	<b>0.031</b>	0.001	Mole/L
Br <sup>-</sup>	<0.007	<0.007	<0.007	<b>&lt;0.007</b>	-	Mole/L
Inorganic carbon	2.97E+06	2.98E+06	2.94E+06	<b>2.96E+06</b>	1.97E+04	µgC/L
Organic carbon	8.92E+05	8.79E+05	8.56E+05	<b>8.76E+05</b>	1.84E+04	µgC/L
Total carbon	3.86E+06	3.86E+06	3.80E+06	<b>3.84E+06</b>	3.52E+04	µgC/L
CO <sub>3</sub> <sup>2-</sup>	0.247	0.248	0.245	<b>0.247</b>	0.002	M



**Table 7 Tank 4 Surface Sample (FTF-4-15-22): Selected Elemental Analysis Results**

<b>Analytes</b>	<b>Analysis-1</b>	<b>Analysis-2</b>	<b>Analysis-3</b>	<b>Average</b>	<b>St. Deviation</b>	<b>Units</b>
Al	2695.0	2684.0	2684.0	<b>2687.7</b>	<i>6.4</i>	mg/L
B	11.2	11.2	10.97	<b>11.1</b>	<i>0.2</i>	mg/L
Ca	4.0	2.4	1.1	<b>2.5</b>	<i>1.4</i>	mg/L
Cr	71.6	70.7	71.2	<b>71.2</b>	<i>0.4</i>	mg/L
Fe	4.2	3.7	2.8	<b>3.6</b>	<i>0.7</i>	mg/L
K	264.0	269.5	283.8	<b>272.4</b>	<i>10.2</i>	mg/L
La	0.5	0.5	0.6	<b>0.5</b>	<i>0.1</i>	mg/L
Mo	12.1	13.6	14.1	<b>13.3</b>	<i>1.0</i>	mg/L
Na	54670.0	54120.0	54340.0	<b>54376.7</b>	<i>276.8</i>	mg/L
P	54.8	48.1	49.3	<b>50.7</b>	<i>3.6</i>	mg/L
S	2684.0	2970.0	2684.0	<b>2779.3</b>	<i>165.1</i>	mg/L
Si	3.3	4.7	3.4	<b>3.8</b>	<i>0.8</i>	mg/L
V	0.9	0.7	0.7	<b>0.8</b>	<i>0.1</i>	mg/L
Hg	6.06E+01	6.16E+01	6.16E+01	<b>6.12E+01</b>	<i>5.59E-01</i>	mg/L

## Appendix B. Tank 4 Sub-Surface samples (FTF-4-15-23)

**Table 8 Tank 4 Sub-Surface Sample FTF-4-15-23: ECP Results**

Analytes	Analysis-1	Analysis-2	Analysis-3	Average	St. Deviation	Units
U-233	<2.25E-03	<2.22E-03	<2.17E-03	<b>&lt;2.22E-03</b>		mg/L
U-234	<2.25E-03	<2.22E-03	<2.17E-03	<b>&lt;2.22E-03</b>		mg/L
U-235	1.16E-02	1.16E-02	1.23E-02	<b>1.18E-02</b>	<i>4.11E-04</i>	mg/L
U-236	<2.25E-03	<2.22E-03	<2.17E-03	<b>&lt;2.22E-03</b>		mg/L
U-238	2.12E+00	2.10E+00	2.10E+00	<b>2.11E+00</b>	<i>1.43E-02</i>	mg/L
U-Total	2.14E+00	2.11E+00	2.11E+00	<b>2.12E+00</b>	<i>1.41E-02</i>	mg/L
(U-235/U-total)	5.42E-03	5.47E-03	5.82E-03	<b>5.57E-03</b>	<i>2.18E-04</i>	-
Pu-239	1.12E-03	1.27E-03	1.05E-03	<b>1.15E-03</b>	<i>1.11E-04</i>	mg/L
Pu-241	<1.23E-06	<1.64E-06	<1.50E-06	<b>&lt;1.46E-06</b>		mg/L

**Table 9 Tank4 Sub-Surface Sample FTF-4-15-23: CCP/EFQ Results**

Analytes	Analysis-1	Analysis-2	Analysis-3	Average	St. Deviation	Units
NO <sub>3</sub> <sup>-</sup>	3.078E-01	2.968E-01	2.994E-01	<b>3.013E-01</b>	<i>5.793E-03</i>	Mole/L
NO <sub>2</sub> <sup>-</sup>	3.959E-01	3.829E-01	3.864E-01	<b>3.884E-01</b>	<i>6.693E-03</i>	Mole/L
OH <sup>-1</sup>	0.716	0.739	0.684	<b>0.713</b>	<i>0.03</i>	Molar
SpG @ 25 °C	1.10	1.12	1.11	<b>1.11</b>	<i>0.01</i>	--
Cs-137	3.30E+08	3.34E+08	3.04E+08	<b>3.23E+08</b>	<i>1.61E+07</i>	dpm/mL
Ba-137m	3.13E+08	3.16E+08	2.88E+08	<b>3.05E+08</b>	<i>1.52E+07</i>	dpm/mL

**Table 10 Tank 4 Sub-Surface Sample (FTF-4-15-23): Select Elemental Analysis Results**

Analytes	Analysis-1	Analysis-2	Analysis-3	Average	St. Deviation	Units
Al	2741.7	2741.7	2741.7	<b>2741.7</b>	<i>0.0</i>	mg/L
B	10.9	11.1	10.7	<b>10.9</b>	<i>0.2</i>	mg/L
Ca	2.5	0.8	0.6	<b>1.3</b>	<i>1.1</i>	mg/L
Cr	70.9	70.8	70.7	<b>70.8</b>	<i>0.1</i>	mg/L
Fe	10.9	9.9	10.8	<b>10.5</b>	<i>0.5</i>	mg/L
K	278.6	277.5	280.8	<b>279.0</b>	<i>1.7</i>	mg/L
La	0.6	0.5	0.6	<b>0.6</b>	<i>0.1</i>	mg/L
Mo	11.1	12.8	11.8	<b>11.9</b>	<i>0.8</i>	mg/L
Na	54390	54834	54390	<b>54538</b>	<i>256</i>	mg/L
P	52.4	53.6	52.2	<b>52.7</b>	<i>0.8</i>	mg/L
S	3096.9	3174.6	2852.7	<b>3041</b>	<i>168</i>	mg/L
Si	5.1	5.9	5.6	<b>5.5</b>	<i>0.4</i>	mg/L
V	0.7	0.8	0.9	<b>0.8</b>	<i>0.1</i>	mg/L
Hg	4.84E+01	4.79E+01	4.82E+01	<b>4.82E+01</b>	<i>2.20E-01</i>	mg/L

**Table 11 Tank 4 Sub-Surface Sample FTF-4-15-23: Other Results from ECP/CCP/EFQ**

Analytes	Analysis-1	Analysis-2	Analysis-3	Average	St. Deviation	Units
U-235/U-total*100	5.42E-01	5.47E-01	5.82E-01	<b>5.57E-01</b>	<i>2.18E-02</i>	%
Pu-238	7.57E-06	7.96E-06	7.38E-06	<b>7.64E-06</b>	<i>2.97E-07</i>	mg/L
Pu-239/240	1.55E+02	1.75E+02	1.45E+02	<b>1.58E+02</b>	<i>1.53E+01</i>	dpm/mL
I-129	8.11E+00	<1.59E+01	8.65E+00	<b>≤1.09E+01</b>	-	pCi/mL
Tc-99	3.76E+04	3.62E+04	3.85E+04	<b>3.74E+04</b>	<i>1.15E+03</i>	pCi/mL
SO <sub>4</sub> <sup>2-</sup>	0.087	0.084	0.084	<b>0.085</b>	<i>0.002</i>	Mole/L
CHO <sub>2</sub> <sup>-</sup>	<0.002	<0.002	<0.003	<b>&lt;0.002</b>	-	Mole/L
Cl <sup>-</sup>	0.006	0.006	0.006	<b>0.006</b>	<i>0.000</i>	Mole/L
F <sup>-</sup>	<0.006	<0.006	<0.006	<b>&lt;0.006</b>		Mole/L
PO <sub>4</sub> <sup>3-</sup>	<0.001	<0.001	<0.001	<b>&lt;0.001</b>	-	Mole/L
C <sub>2</sub> O <sub>4</sub> <sup>2-</sup>	0.032	0.031	0.031	<b>0.031</b>	<i>0.001</i>	Mole/L
Br <sup>-</sup>	<0.007	<0.007	<0.007	<b>&lt;0.007</b>	-	Mole/L
Inorganic carbon	2.97E+06	3.06E+06	2.96E+06	<b>3.00E+06</b>	<i>5.79E+04</i>	µgC/L
Organic carbon	8.49E+05	8.75E+05	8.48E+05	<b>8.57E+05</b>	<i>1.53E+04</i>	µgC/L
Total carbon	3.83E+06	3.94E+06	3.81E+06	<b>3.86E+06</b>	<i>7.24E+04</i>	µgC/L
CO <sub>3</sub> <sup>2-</sup>	0.248	0.255	0.246	<b>0.250</b>	<i>0.005</i>	M