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Analysis of Tank 30H (HTF-30-19-91, -92, -93), Tank 32H (HTF-32-19-94, -95, -96), and Tank 37H (HTF-37-19-97, -98, -99) Samples for Support of the Evaporator Feed Qualification and Corrosion Control Programs for the 3H-Evaporator

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December 2019

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EXECUTIVE SUMMARY

SRNL analyzed samples from Tank 30H, Tank 32H, and Tank 37H to support the Evaporator Feed Qualification and Corrosion Control Programs for the 3H-Evaporator system. The samples from Tanks 30H, 32H, and 37H all contain highly concentrated salt solutions with sodium concentrations ranging from 13.7 M to 17.1 M except for the Tank 32H surface sample with a sodium concentration of 4.38 M. Hydroxide is the dominant anion in all of the samples followed by nitrate and nitrite. Only the two samples from Tank 32H indicate any significant stratification of solution composition in the tanks with the surface sample much more dilute than the VDS.

The surface sample and the VDS from Tank 30H are both generally more concentrated than the previous samples from the tank. The surface sample from Tank 32H is much more dilute than the previous surface sample while the Tank 32H VDS is slightly more concentrated than the previous VDS. The Tank 37H surface sample shows higher concentrations for most species than measured in the previous sample. The Tank 37H VDS has a similar sodium concentration to the previous sample with higher hydroxide, nitrite, and aluminate, and lower nitrate and carbonate concentrations.

The difference between the sum of the major cations versus the sum of the major anions is quite high for the surface samples from Tank 30H and Tank 37H (~ 18%) while the difference is $\leq$5% for the VDS from each of these tanks. The two Tank 32H samples show a difference between the sum of the major cations versus the sum of the major anions of 9-10% indicating reasonable data quality. The silicon concentrations measured in the VDS and surface samples from Tank 30H, Tank 32H, and Tank 37H were all below detection.

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LIST OF ABBREVIATIONS

AD	Analytical Development
DI	de-ionized
CCP	Corrosion Control Program
EFQ	Evaporator Feed Qualification
IC	ion chromatography
ICP-ES	Inductively Coupled Plasma Emission Spectroscopy
NAS	sodium aluminosilicate
%RSD	percent relative standard deviation
SRNL	Savannah River National Laboratory
SRR	Savannah River Remediation
TIC	total inorganic carbon
TTQAP	Task Technical and Quality Assurance Plan
TTR	Technical Task Request
VDS	variable depth sample

1.0 Introduction

Feed limits have been established for the 3H-Evaporator system to ensure nuclear criticality is not possible by preventing the accumulation of sodium aluminosilicate (NAS) solids in the evaporator and that corrosion is minimized. These limits are protected by the Evaporator Feed Qualification Program (EFQ) and the Corrosion Control Program (CCP) that require periodic sampling and analysis to confirm that the waste supernate composition stays within the limits.[1,2]

Savannah River Remediation (SRR) obtained samples from two different heights within each of the three waste tanks supporting the 3H-Evaporator operations on October 15, 2019. The Tank 30H (evaporator drop tank), Tank 32H (evaporator feed tank), and Tank 37H (alternate evaporator drop tank) samples were received by the Savannah River National Laboratory (SRNL) Shielded Cells on October 15-17, 2019. The analysis of these samples provides information necessary for determining compliance with the EFQ and CCP. The sample characterization was requested via a Technical Task Request (TTR) and accompanying Task Technical and Quality Assurance Plan (TTQAP).[3,4]

2.0 Experimental Procedure

The 3H-Evaporator samples from Tanks 30H, 32H, and 37H were opened in the SRNL Shielded Cells and poured into clear plastic beakers. The beakers were photographed, and the masses of the samples determined. Table 2-1 provides the measured mass, volume estimates, and a description of each of the six samples. Figures 2-1, 2-2, and 2-3 show photographs of each set of samples in clear beakers. Figure 2-4 shows all the tank samples together in poly bottles. (Note that the labels on the beakers in the photographs were mis-labeled with -10 instead of the correct -19 designating the year.) All of the samples except the Tank 32H surface sample (HTF-32-19-94) contained the white crystalline solids that were clearly precipitated salts based on observations during the transfer of the samples from the clear beakers to the poly storage bottles. None of the samples from each of the three tanks contained any dark sludge-like solids.

An additional variable depth sample (VDS) from each tank was provided in case sludge solids were found requiring a weight percent insoluble solids analysis. Only the surface sample and the first VDS from each tank were analyzed for the CC/EFQ program. All six samples received the analyses required by the EFQ that includes determination of Cs-137 by gamma spectroscopy and inductively coupled plasma-emission spectroscopy (ICP-ES) to determine Na, Al, Si, and other metals. All six samples also received the analyses required by the CCP. The CCP analysis suite includes determination of free hydroxide and ion chromatography (IC). The total inorganic carbon (TIC) was also determined on the surface samples to provide a concentration for the carbonate anion present in the samples.

Density measurements were made on well-mixed (unfiltered) aliquots of the samples using calibrated volumetric tubes at ambient cell temperature (21 °C).

For the CCP analysis, de-ionized (DI) water dilutions were made in triplicate from a well-mixed (unfiltered) sample and submitted to Analytical Development (AD) for analysis. A blank of the DI water was also prepared along with the samples. The water dilutions were analyzed by ion chromatography, total inorganic carbon, and free hydroxide methods.

Triplicate aliquots of the well-mixed (unfiltered) sample from each sample receiving the EFQ analysis suite were prepared for analysis using the warm acid strike method.[5] A reagent blank and three silicon standard solutions were submitted for analysis with the samples. The samples prepared by warm acid strike were submitted to AD for analysis by ICP-ES and gamma spectroscopy.

Quality Assurance

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. This review meets the acceptable criteria to comply with the TTR requesting this work with a functional classification of Safety Class. Data are recorded in the electronic laboratory notebook system as notebook/experiment number Y7081-00081-34.

Table 2-1. Sampling Description and Mass of the Tank 30H, 32H, and 37H Samples

Tank	Sample ID	Sample Type	Sample Mass (g)	Est. Sample Vol. (mL)	Est. Solids Vol. (mL)	Description
Tank 30H	HTF-30-19-91	Surface	116.1	~75	~20	Dark tinted solution with white crystalline solids
Tank 30H	HTF-30-19-92	VDS	321.8	~205	~90	Dark tinted solution with white crystalline solids
Tank 30H	HTF-30-19-93	VDS	324.8	~205	~90	Dark tinted solution with white crystalline solids
Tank 32H	HTF-32-19-94	Surface	77.6	~65	~0	Yellow tinted solution with white crystalline solids
Tank 32H	HTF-32-19-95	VDS	312.4	~210	~30	Yellow tinted solution with white crystalline solids
Tank 32H	HTF-32-19-96	VDS	312.2	~210	~30	Yellow tinted solution with white crystalline solids
Tank 37H	HTF-37-19-97	Surface	112.4	~75	~25	Dark tinted solution with white crystalline solids
Tank 37H	HTF-37-19-98	VDS	325.0	~205	~90	Dark tinted solution with white crystalline solids
Tank 37H	HTF-37-19-99	VDS	324.0	~205	~90	Dark tinted solution with white crystalline solids

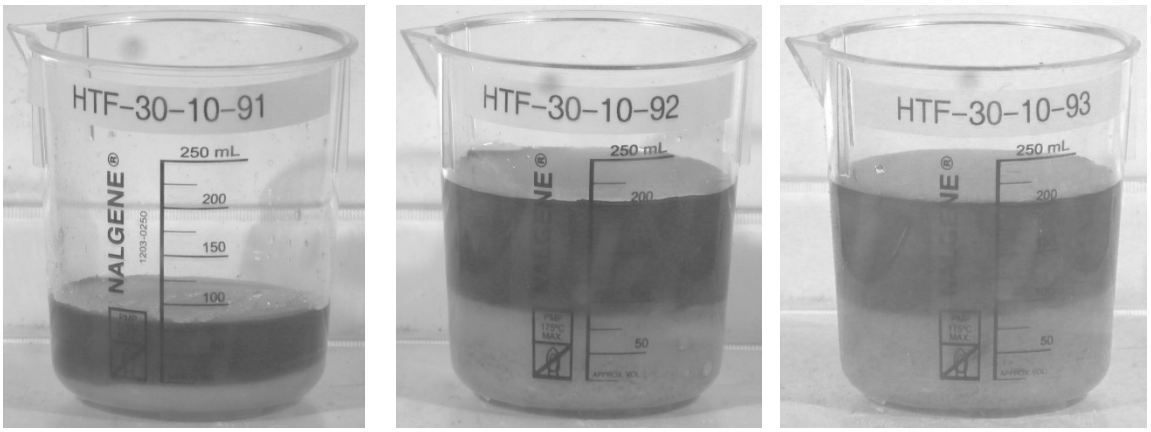

Figure 2-1. 3H-Evaporator Samples from Tanks 30H

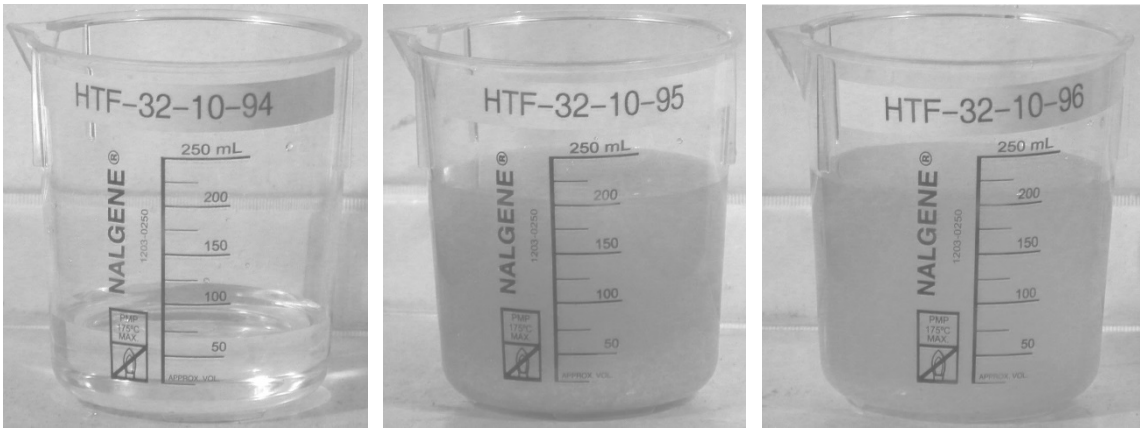

Figure 2-2. 3H-Evaporator Samples from Tanks 32H

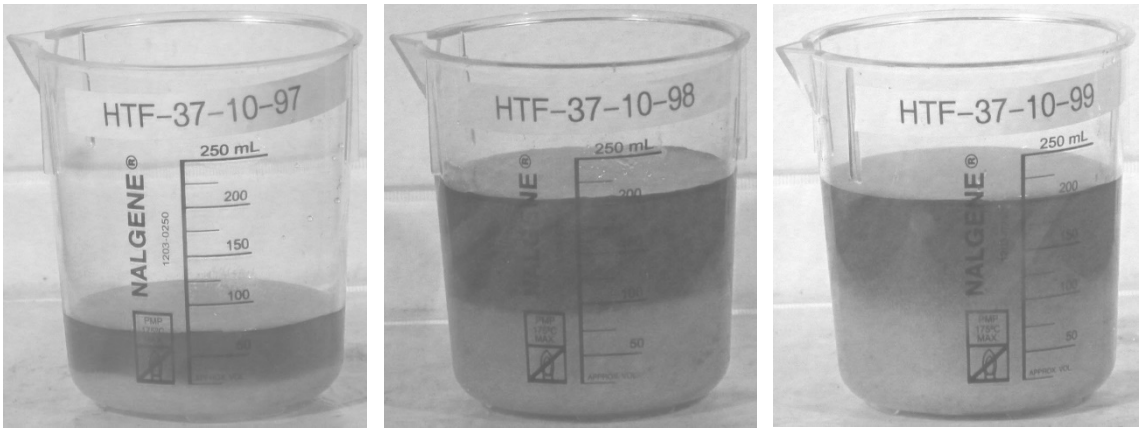

Figure 2-3. 3H-Evaporator Samples from Tanks 37H

Figure 2-4. View of the 3H-Evaporator Samples

3.0 Results and Discussion

The following tables contain the results from the analysis of the samples. The tables show the average concentrations and the percent relative standard deviations (RSD) for the triplicate sample preparations. Results preceded by "<" indicate the analyte was below the limits of quantification for all three replicate aliquots of the sample. Results preceded by "≤" indicate that at least one of the replicates for the sample was above the limits of quantification while one or more of the replicates analyzed were below detection. The percent RSD presented in the table only includes the uncertainty associated with sub-sampling/sample preparation in the Shielded Cells and the analytical method. The estimated one sigma percent uncertainty provides an indication of the uncertainty associated with the analytical method as reported by AD. Neither of these measures of uncertainty includes the uncertainty associated with sampling a large waste tank. Previous investigations indicate the uncertainty from taking a small sample from a large waste tank can be significant.[6,7,8]

The results in Table 3-1 for the two samples from Tank 30H show both samples to be highly concentrated salt solutions with a sodium concentration of 14.1 M in the surface sample and 16.5 M in the VDS. Hydroxide is the main anionic species in both solutions. The other main anions in the two solutions are nitrite, nitrate, aluminate (aluminum), and carbonate. The RSD values of most components of the Tank 30H surface sample are somewhat higher than typical indicating some variability in the three replicate samples analyzed from each tank sample. This may result from sub-sampling difficulties due to the presence of crystallized salts in the samples. Inclusion of more or less of these solids during each sub-sampling event to obtain the three replicates could lead to the higher uncertainties. The composition of the surface sample and VDS from Tank 30H are generally quite similar with exception of the sodium concentration indicating little or no stratification in the tank. The sodium concentration measured in the surface sample (14.1 M) may be biased low based on a comparison to the sum of the anions (16.8 M). The sum of the major cations versus the sum of the major anions for the Tank 30H surface sample shows a difference of 18%. The sum of the major cations versus the sum of the major anions shows a difference of <5%

for the Tank 30H VDS providing an indication of good data quality. The surface sample and the VDS from Tank 30H are both generally more concentrated than the previous samples from the tank.[9]

The results for the two samples from Tank 32H in Table 3-1 indicate significant stratification throughout the tank depth profile. The sodium concentration measured in the Tank 32H VDS of 13.7 M is much higher than the 4.38 M sodium concentration measured in the surface sample. As with Tank 30H samples, the anionic constituent with the highest concentration is hydroxide followed by nitrite and nitrate. The anion results for the Tank 32H VDS show higher RSD values due to one replicate being >30% higher than the other two replicates. This may result from sub-sampling difficulties due to the presence of crystallized salts in the samples. Inclusion of more or less of these solids during each sub-sampling event to obtain the three replicates could lead to the higher uncertainties. The sum of the major cations versus the sum of the major anions indicates reasonable data quality with a difference of 8-10% for each of the two Tank 32H samples. The surface sample from Tank 32H is much more dilute than the previous surface sample that showed a sodium concentration of 13.8 M. The major anion concentrations of the current VDS are slightly more concentrated than the previous VDS although the nitrate concentration is lower. However, the sodium concentration of current VDS sample is slightly lower than the previous VDS sample.[9]

The results from the two samples from Tank 37H in Table 3-1, shows similar concentrations for most of the major components of the samples indicating minimal stratification in the tank. The anion results for both of the Tank 37H sample show higher RSD values due to one replicate being >30% higher than the other two replicates. This may result from sub-sampling difficulties due to the presence of crystallized salts in the samples. Inclusion of more or less of these solids during each sub-sampling event to obtain the three replicates could lead to the higher uncertainties. Sodium is the dominant cation with a concentration of 15.0 M in the Tank 37H surface sample and 17.1 M in the VDS. Hydroxide is the anion with the highest concentration is followed by nitrite and nitrate. The sum of the major cations versus the sum of the major anions shows a difference of ~18% for the Tank 37H surface sample likely due to the anion concentrations being biased high by the single more concentrated replicate. The sum of the major cations versus the sum of the major anions for the VDS indicates good data quality with a difference of ~5%. The Tank 37H surface sample shows higher concentrations for most species than measured in the previous sample. The Tank 37H VDS has a similar sodium concentration to the previous sample with higher hydroxide, nitrite, and aluminate, but lower nitrate and carbonate concentrations.[9]

All of the silicon concentrations measured in the VDS and surface samples from Tank 30H, Tank 32H and Tank 37H were below detection. The standards used for the silicon analysis (50 mg/L silicon in the solution prepared by warm acid strike to final concentrations of 0.5, 1.0, and 2.0 mg/L) were all close to the target concentrations with differences from the targeted concentrations of 0-10%. The silicon concentration was below detectable levels in the process blank.

Table 3-1. ECP, CCP, and other Analytical Data for Tank 30H, 32H and 37H Samples. (Averages and %RSD values are of triplicate measurements)

analyte	method	units	est. 1σ	HTF-30-19-91		HTF-30-19-92		HTF-32-19-94		HTF-32-19-95	
				average	RSD	average	RSD	average	RSD	average	RSD
sample type	--	--	--	surface		vds		surface		vds	
density @ 21°C	grav.	g/mL	5%	1.57	0.9%	1.58	0.9%	1.20	0.2%	1.50	0.3%
Cs-137	gamma scan	dpm/mL	5%	3.93E+09	16%	4.19E+09	3.6%	4.14E+08	1.4%	2.68E+09	1.2%
Ba-137m				3.71E+09		3.97E+09		3.92E+08		2.54E+09	
OH^-	titration	M	10%	1.07E+01	9.3%	1.02E+01	4.3%	2.15E+00	2.7%	8.28E+00	18%
F^-	IC	M	10%	<2.82E-02	--	<2.84E-02	--	<2.08E-02	--	<2.76E-02	--
CHO_2^-	IC	M	10%	<1.19E-02	--	<1.20E-02	--	<8.79E-03	--	<1.17E-02	--
Cl^-	IC	M	10%	2.49E-02	14%	2.30E-02	5.7%	<1.12E-02	--	2.43E-02	18%
NO_2^-	IC	M	10%	2.87E+00	8.4%	2.68E+00	2.8%	5.20E-01	2.4%	2.69E+00	20%
NO_3^-	IC	M	10%	2.18E+00	15%	2.13E+00	7.4%	6.23E-01	2.1%	1.96E+00	25%
PO_4^{3-}	IC	M	10%	2.13E-02	16%	1.91E-02	5.5%	<4.17E-03	--	1.33E-02	26%
SO_4^{2-}	IC	M	10%	6.48E-03	14%	5.80E-03	5.4%	2.80E-02	2.1%	8.65E-02	26%
$C_2O_4^{2-}$	IC	M	10%	<6.09E-03	--	<6.13E-03	--	4.63E-03	1.8%	1.91E-02	32%
Br^-	IC	M	10%	<3.35E-02	--	<3.38E-02	--	<2.48E-02	--	<3.28E-02	--
CO_3^{2-}	TIC	M	10%	4.69E-02	11%	4.11E-02	4.1%	1.61E-01	2.3%	5.30E-01	40%
TOC	TOC	mg C/L	10%	<2.68E+02	--	<2.70E+02	--	<1.98E+02	--	3.23E+02	44%
Al	ICP-ES	mg/L	10%	2.19E+04	17%	2.45E+04	3.0%	8.77E+03	0.8%	2.24E+04	1.2%
		M		8.11E-01		9.10E-01		3.25E-01		8.28E-01	
B	ICP-ES	mg/L	10%	3.27E+02	16%	3.51E+02	3.4%	4.71E+01	0.5%	2.38E+02	1.3%
Ca	ICP-ES	mg/L	10%	≤1.83E+00	--	≤1.83E+00	--	≤1.52E+00	--	≤1.78E+00	--
Cr	ICP-ES	mg/L	10%	4.14E+02	17%	4.16E+02	3.9%	7.39E+01	1.2%	3.34E+02	1.8%
Fe	ICP-ES	mg/L	10%	2.94E+01	21%	3.07E+01	5.0%	5.92E+00	8.1%	2.32E+01	2.5%
K	ICP-ES	mg/L	10%	4.20E+03	17%	4.47E+03	2.7%	4.66E+02	2.2%	2.95E+03	0.6%
Li	ICP-ES	mg/L	10%	<8.24E+00	--	<8.27E+00	--	<6.32E+00	--	<7.82E+00	--
Na	ICP-ES	mg/L	10%	3.24E+05	17%	3.80E+05	0.9%	1.01E+05	0.8%	3.14E+05	0.7%
		M		1.41E+01		1.65E+01		4.38E+00		1.37E+01	
P	ICP-ES	mg/L	10%	6.54E+02	18%	5.88E+02	3.3%	<7.69E+01	--	3.82E+02	4.9%
Si	ICP-ES	mg/L	15%	<2.34E+01	--	<2.34E+01	--	<1.80E+01	--	<2.22E+01	--
Zn	ICP-ES	mg/L	10%	2.33E+01	19%	2.32E+01	3.8%	<2.93E+00	--	1.65E+01	1.8%
Anions	sum	M	--	1.68E+01	--	1.61E+01	--	4.01E+00	--	1.51E+01	--
Cations	sum	M	--	1.42E+01	--	1.66E+01	--	4.39E+00	--	1.38E+01	--

est.1σ = estimated one sigma percent uncertainty as reported by AD.

Table 3-1. ECP, CCP, and other Analytical Data for Tank 30H, 32H and 37H Samples. (Averages and %RSD values are of triplicate measurements) Continued

analyte	method	units	est. 1σ	HTF-37-19-97		HTF-37-19-98	
				average	RSD	average	RSD
sample type	--	--	--	surface		vds	
density @ 21°C	grav.	g/mL	5%	1.53	0.8%	1.58	0.5%
Cs-137	gamma scan	dpm/mL	5%	3.47E+09	6.2%	3.76E+09	2.4%
Ba-137m				3.29E+09		3.55E+09	
OH^-	titration	M	10%	1.11E+01	26%	1.16E+01	13%
F^-	IC	M	10%	<2.79E-02	--	<2.85E-02	--
CHO_2^-	IC	M	10%	<1.18E-02	--	<1.20E-02	--
Cl^-	IC	M	10%	3.21E-02	26%	3.29E-02	15%
NO_2^-	IC	M	10%	3.20E+00	30%	3.15E+00	35%
NO_3^-	IC	M	10%	2.36E+00	20%	2.22E+00	38%
PO_4^{3-}	IC	M	10%	1.91E-02	36%	1.99E-02	21%
SO_4^{2-}	IC	M	10%	8.94E-03	31%	7.04E-03	26%
$C_2O_4^{2-}$	IC	M	10%	<6.01E-03	--	<6.16E-03	--
Br^-	IC	M	10%	<3.31E-02	--	<3.39E-02	--
CO_3^{2-}	TIC	M	10%	1.20E-01	19%	1.08E-01	31%
TOC	TOC	mg C/L	10%	<2.65E+02	--	<2.71E+02	--
Al	ICP-ES	mg/L	10%	2.10E+04	5.7%	2.20E+04	1.4%
		M		7.77E-01		8.14E-01	
B	ICP-ES	mg/L	10%	3.13E+02	6.6%	3.35E+02	0.7%
Ca	ICP-ES	mg/L	10%	<1.68E+00	--	2.39E+00[a]	6.6%
Cr	ICP-ES	mg/L	10%	4.28E+02	6.2%	4.33E+02	1.0%
Fe	ICP-ES	mg/L	10%	3.27E+01	7.3%	3.02E+01	4.9%
K	ICP-ES	mg/L	10%	3.82E+03	5.7%	4.23E+03	0.2%
Li	ICP-ES	mg/L	10%	<8.04E+00	--	<8.35E+00	--
Na	ICP-ES	mg/L	10%	3.45E+05	2.0%	3.92E+05	0.2%
		M		1.50E+01		1.71E+01	
P	ICP-ES	mg/L	10%	5.28E+02	1.0%	5.87E+02	0.9%
Si	ICP-ES	mg/L	15%	<2.28E+01	--	<2.37E+01	--
Zn	ICP-ES	mg/L	10%	2.40E+01	5.3%	2.29E+01	1.8%
Anions	sum	M	--	1.78E+01	--	1.81E+01	--
Cations	sum	M	--	1.51E+01	--	1.72E+01	--

est.1σ = estimated one sigma percent uncertainty as reported by AD.

a = only the two replicates above detection were used to calculate the average and RSD

4.0 Conclusions

The samples from Tanks 30H, 32H, and 37H all contain highly concentrated salt solutions with sodium concentrations ranging from 13.7 M to 17.1 M except for the Tank 32H surface sample with a sodium concentration of 4.38 M. Hydroxide is the dominant anion in all of the samples followed by nitrate and nitrite. Only the two samples from Tank 32H indicate any significant stratification of solution composition in the tank with the surface sample much more dilute than the VDS.

The surface sample and the VDS from Tank 30H are both generally more concentrated than the previous samples from the tank. The surface sample from Tank 32H is much more dilute than the previous surface sample while the Tank 32H VDS is slightly more concentrated than the previous VDS. The Tank 37H surface sample shows higher concentrations for most species than measured in the previous sample. The Tank 37H VDS has a similar sodium concentration to the previous sample with higher hydroxide, nitrite, and aluminate, and lower nitrate and carbonate concentrations.

The difference between the sum of the major cations versus the sum of the major anions is quite high for the surface samples from Tank 30H and Tank 37H (~ 18%) while the difference is $\leq$5% for VDS from each of these tanks. The two Tank 32H samples show a difference between the sum of the major cations versus the sum of the major anions of 9-10% indicating reasonable data quality. The silicon concentrations measured in the VDS and surface samples from Tank 30H, Tank 32H, and Tank 37H were all below detection.

5.0 Acknowledgements

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