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Disclaimer:

Analysis of Tank 38H (HTF-38-21-24, -25) and Tank 43H (HTF-43-21-26, -27) Samples for Support of the Enrichment Control and Corrosion Control Programs

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Analysis of Tank 38H (HTF-38-21-24, -25) and Tank 43H (HTF-43-21-26, -27) Samples for Support of the Enrichment Control and Corrosion Control Programs

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Prepared for the U.S. Department of Energy under contract number DE-AC09-08SR22470.

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EXECUTIVE SUMMARY

SRNL analyzed samples from Tank 38H and Tank 43H to support ECP and CCP. The results indicate the concentrations of most species in the Tank 38H surface sample decreased from the previous surface sample. The sub-surface sample from Tank 38H contained dark solids suspended in the solution. The Tank 38H sub-surface sample shows only minor changes in concentration for soluble species in the solution (e.g., Na, Al, Cs-137) relative to the previous sample, but a large increase in concentrations for species typically associated with sludge solids (e.g., U, Pu, Fe, Si) likely because of an increase in sludge solids from the previous sample. The large differences in the concentrations of major components between the Tank 38H surface and sub-surface samples indicate significant stratification of solution species within the tank.

The Tank 43H surface sample is more concentrated than the previous samples while the Tank 43H sub-surface sample is fairly similar to the previous sample. The solution concentrations measured in the Tank 43H surface and sub-surface samples indicate some stratification within the tank.

The Tank 38H surface sample shows a slight increase in uranium concentrations and a large decrease in plutonium concentrations compared to the previous sample. The Tank 38H sub-surface sample shows a large increase in uranium and plutonium concentrations compared to the previous sample likely because of an increase in sludge solids. The total uranium and plutonium concentrations in the two Tank 43H samples both increased from previous sample results.

The sum of the major cations versus the sum of the major anions shows a difference of <10% for the two Tank 38H samples and the two Tank 43H samples providing an indication of good data quality for the non-radioactive analytes in the samples.

The silicon concentrations measured in the Tank 38H surface sample decreased compared with the previous sample results while the silicon increased significantly in the sub-surface sample likely due to the presence of sludge solids. The Tank 43H surface sample silicon concentration decreased compared to the previous surface sample results. The Tank 43H sub-surface sample silicon concentration increased relative to the previous surface sample. The samples analyzed show silicon concentrations ranging from 40.8 to 275 mg/L.

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LIST OF ABBREVIATIONS

ARD	Analytical Research and Development
DI	de-ionized
CCP	Corrosion Control Program
ECP	Enrichment Control Program
IC	ion chromatography
ICP-ES	inductively coupled plasma emission spectroscopy
ICP-MS	inductively coupled plasma mass spectrometry
%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

1.0 Introduction

Feed limits have been established for the 2H-Evaporator system to ensure nuclear criticality is not possible and corrosion is minimized.[1] These limits are protected by the Enrichment Control Program (ECP) and the Corrosion Control Program (CCP) that require periodic sampling and analysis to confirm that the waste supernate composition stays within the limits.[2,3]

Savannah River Remediation (SRR) obtained samples from two different heights within each of the two waste tanks supporting the 2H-Evaporator operations on March 26, 2021. The Tank 38H (evaporator drop tank) and Tank 43H (evaporator feed tank) samples were received by the Savannah River National Laboratory (SRNL) Shielded Cells on March 26, 2021. Analysis of these samples provides information necessary for determining compliance with the ECP and CCP. The sample characterization was requested via a Technical Task Request[4] (TTR) and conducted based on a Task Technical and Quality Assurance Plan (TTQAP).[5]

2.0 Experimental Procedure

The samples from Tank 38H and 43H 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 sampling height and mass of each sample. Figure 2-1 shows a photograph of the samples in the clear beakers. Both the surface sample and the sub-surface sample from Tank 43H were mostly clear and showed no visible undissolved solids when poured into the plastic beakers. The surface sample from Tank 38H was mostly clear with no visible solids also. However, the sub-surface sample from Tank 38H contained dark solids suspended in the solution. After settling overnight, the Tank 38H sub-surface sample bottle contained a clear solution with a thin layer of dark solids sitting on the bottom. Based on experience with past samples, the solids in the Tank 38H subsurface sample represent less than 1 wt% insoluble solids.

All four samples received the analyses required by the ECP that includes sample dissolution (discussed later) and determination of uranium isotopes by inductively coupled plasma-mass spectrometry (ICP-MS) and determination of plutonium isotopes by radiochemical separation and counting methods. All four samples were also submitted for gamma spectroscopy to determine the Cs-137 concentration and inductively coupled plasma-emission spectroscopy (ICP-ES) to determine Na, Al, Si, and other metals. All four samples from both Tank 38H and Tank 43H 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 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 (22 °C).

For the samples receiving the CCP analysis suite, de-ionized (DI) water dilutions were made in triplicate from a well-mixed (unfiltered) sample and submitted to Analytical Research and Development (ARD) 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 ECP analysis suite were prepared for analysis using the warm acid strike method.[6] 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 ARD for analysis by ICP-ES, ICP-MS for uranium isotopics,

plutonium isotopics, and gamma spectroscopy. Additionally, in an effort to reach lower detection limits for U-233, the warm acid strike samples were analyzed using the U-233/234/235/236 method that combines uranium separation with ICP-MS.[7]

Quality Assurance

Requirements for performing reviews of technical reports and the extent of review are established in Manual E7, Procedure 2.60.[8] SRNL documents the extent and type of review using the SRNL Technical Report Design Checklist.[9] This review meets the acceptable criteria to comply with the TTR[4] requesting this work with a functional classification of Safety Class and per guidance in the TTQAP.[5] Data are recorded in the electronic laboratory notebook system as notebook/experiment number Y7081-00081-44.[10]

Table 2-1. Sampling Height and Sample Mass of the Tank 38H and 43H Samples

Sample ID	Sample Type	Sampling Height (inches from bottom)	Sample Mass (g)
HTF-38-21-24	Surface	surface	85.6
HTF-38-21-25	Sub-surface	224”	106.6
HTF-43-21-26	Surface	surface	98.4
HTF-43-21-27	Sub-surface	137”	103.9

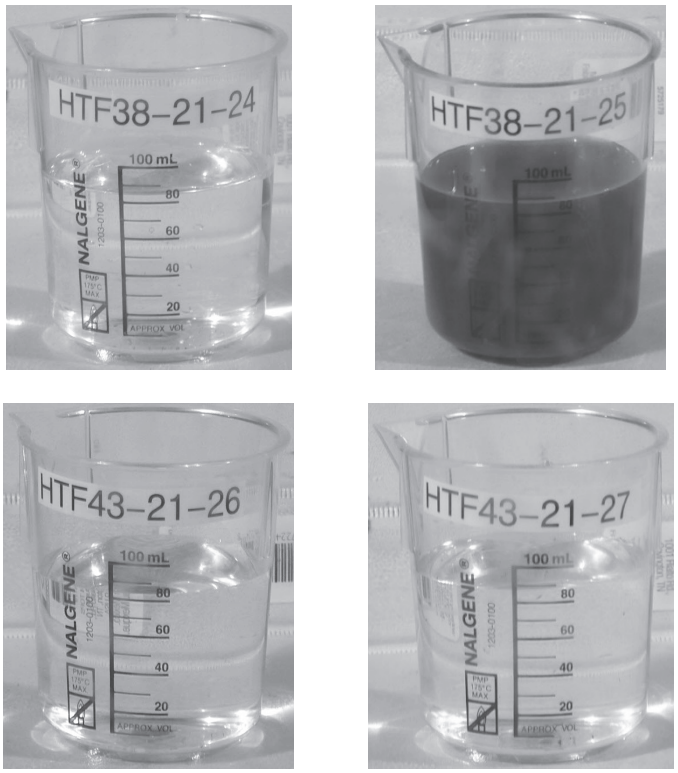

Figure 2-1. Samples from Tank 38H and 43H

3.0 Results and Discussion

Tables 3-1 and 3-2 contain the results from the analysis of the 2H-Evaporator samples. The tables show the average concentrations and the percent relative standard deviation (%RSD) for the triplicate sample preparations unless otherwise noted. Results preceded by "<" indicate the analyte was below the limits of quantification for all three replicate aliquots of the sample. In this case, the less than value in the table results from averaging the three less than values for the replicates. 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 less than or equal to value in the table results from averaging all values for all three replicates. The %RSD presented in the tables 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 ARD. 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.[11,12,13]

The results in Table 3-1 indicate the concentrations of most species in the Tank 38H surface sample decreased from the previous surface sample with the sodium concentration dropping from 2.83 M in the previous sample to 1.68 M in the current sample.[14] The Tank 38H sub-surface sample shows only minor changes in concentration for soluble species in the solution (e.g., Na, Al, Cs-137) relative to the previous sub-surface sample, but a large increase in concentrations for species typically associated with sludge solids (e.g., U, Pu, Fe, Si). The current Tank 38H sub-surface sample contains more sludge solids than the previous sample. The sodium concentration in the Tank 38H sub-surface sample of 7.31 M is similar to the value of 7.24 M observed in the previous sample.[14] The weight fraction of U-235 to U-total in the Tank 38H surface sample increased from 0.82 in the previous sample to 1.02 in the current sample. The U-235 to U-total weight fraction rose from 0.61 to 0.67 in the sub-surface sample.[14] Table 3-2 provides results from the two methods used to measure uranium isotopes on these samples. The Separation/ICP-MS method provides lower detection limits for the less abundant uranium isotopes. The large differences in the sodium and other major component concentrations between the Tank 38H surface and sub-surface samples indicate significant stratification of solution species remains within the tank.

The Tank 43H surface sample is more concentrated than the previous surface sample as indicated by the increase in the sodium concentration from 3.60 M to 5.10 M.[14] The Tank 43H sub-surface sample is fairly similar to the previous sub-surface sample for most species analyzed. The sodium concentration in the Tank 43H sub-surface sample decreased from 6.80 M in the previous sample to 6.27 M in the current sample. The uranium concentration in both Tank 43H samples increased while the U-235 weight fraction decreased slightly from the previous analysis.[14] The Pu-238 and Pu-239/240 concentrations in both Tank 43H samples increased compared to the previous samples.[14] The Tank 43H surface sample is less concentrated than the sub-surface sample indicating some stratification within the tank. Note: One replicate from the Tank 43H surface sample and one replicate from the Tank 43H sub-surface sample were switched during the warm acid strike sample preparations. The composition of the Tank 43H surface and sub-surface samples are sufficiently different that the switch was obvious from the data and consistent over all of the analytical methods conducted on the samples. The data in the tables reflects the correct three replicates for each of the samples.

The results for most species in the table, along with the density measurement results, generally show small %RSD values for the replicate analyses. The sum of the major cations versus the sum

of the major anions shows a difference of <10% for the Tank 38H samples and the Tank 43H samples providing an indication of good data quality for the non-radioactive analytes in the samples.

The Cs-137 results for the Tank 38H surface sample decreased from the previous sample while the sub-surface sample shows a similar concentration to the previous analysis.[14] The Tank 43H surface sample shows a higher Cs-137 concentration while the sub-surface sample remained similar to the previous analysis.[14]

The silicon concentrations measured in the Tank 38H surface sample decreased compared with the previous sample results while the silicon increased significantly in the sub-surface sample likely due to the presence of sludge solids.[14] The Tank 43H surface sample silicon concentration decreased compared to the previous surface sample results.[14] The Tank 43H sub-surface sample silicon concentration increased relative to the previous surface sample.[14] The standards used for the silicon analysis (50 mg/L silicon in the solution prepared by warm acid strike diluted to final concentrations of 0.5, 1.0, and 2.0 mg/L) were close to the target concentrations with differences from the targeted concentrations of <6%. The silicon concentration was below detectable levels in the process blank. The samples analyzed show silicon concentrations ranging from 40.8 to 275 mg/L.

Table 3-1. ECP, CCP, and other Analytical Data for Tank 38H and Tank 43H Samples. (Averages and %RSD values are of triplicate measurements)

analyte	method	units	est. 1σ	HTF-38-21-24		HTF-38-21-25		HTF-43-21-26		HTF-43-21-27	
				average	%RSD	average	%RSD	average	%RSD	average	%RSD
Sample Type	--	--	--	Surface		Sub-surface		Surface		Sub-surface	
density @ 22°C	grav.	g/mL	5%	1.07	1.1%	1.29	0.6%	1.22	0.1%	1.27	1.1%
U-233	ICP-MS	mg/L	20%	<1.01E-02	--	1.44E-02	2.6%	<1.01E-02	--	<1.01E-02	--
U-234	ICP-MS	mg/L	20%	<1.01E-02	--	1.97E-01	3.4%	<1.01E-02	--	<1.01E-02	--
U-235	ICP-MS	mg/L	20%	1.83E-01	0.8%	1.53E+00	2.8%	2.01E-01	0.7%	1.26E-01	1.4%
U-236	ICP-MS	mg/L	20%	1.07E-02	5.5%	1.30E-01	4.5%	1.34E-02	3.9%	<1.01E-02	--
U-238	ICP-MS	mg/L	20%	1.78E+01	0.5%	2.43E+02	3.2%	2.79E+01	0.9%	1.98E+01	0.2%
Total U	calc.	mg/L	--	1.79E+01	0.5%	2.45E+02	3.2%	2.81E+01	0.9%	1.99E+01	0.3%
U-235 / U	calc.	%	--	1.02	0.9%	0.63	0.5%	0.72	0.3%	0.63	1.1%
Pu-238	PuTTA	mg/L	10%	7.11E-05	7.2%	3.03E-02	10%	2.83E-04	7.5%	6.67E-04	28%
		dpm/mL		2.70E+03		1.15E+06		1.08E+04		2.54E+04	
Pu-239 [a]	PuTTA	mg/L	20%	2.29E-03[b]	32%	3.03E-01	12%	5.17E-03[b]	10%	1.19E-02	20%
Pu-239/240	PuTTA	dpm/mL		3.16E+02[b]		4.19E+04		7.13E+02[b]		1.65E+03	
Pu-241	Pu238/41	mg/L	20%	<3.33E-06	--	1.05E-03	9.6%	≤3.11E-06	--	<3.70E-06	--
		dpm/mL		<7.61E+02		2.40E+05		≤7.13E+02		<8.46E+02	
Cs-137	gamma scan	dpm/mL	5%	9.35E+07	3.0%	2.33E+08	9.4%	2.04E+08	2.6%	2.10E+08	1.3%
Ba-137m				8.85E+07		2.21E+08		1.93E+08		1.99E+08	
OH^-_{free}	titration	M	10%	6.37E-01	1.1%	2.05E+00	4.1%	1.66E+00	0.2%	2.12E+00	2.5%
F^-	IC	M	10%	<1.08E-02	--	<1.05E-02	--	<1.09E-02	--	<1.08E-02	--
CHO_2^-	IC	M	10%	5.70E-03	1.2%	1.74E-02	0.9%	1.87E-02	0.5%	2.57E-02	0.7%
Cl^-	IC	M	10%	<5.79E-03	--	6.77E-03	1.0%	5.10E-03	0.1%	6.14E-03	0.6%
NO_2^-	IC	M	10%	5.98E-01	1.0%	1.87E+00	1.1%	1.59E+00	2.3%	1.89E+00	0.8%
NO_3^-	IC	M	10%	2.32E-01	2.3%	1.73E+00	3.1%	7.05E-01	2.0%	9.23E-01	2.1%
PO_4^{3-}	IC	M	10%	1.02E-03	0.4%	1.17E-02	1.0%	3.14E-03	5.1%	4.68E-03	0.8%
SO_4^{2-}	IC	M	10%	1.12E-02	0.0%	6.53E-02	1.0%	3.98E-02	0.9%	5.04E-02	0.6%
$C_2O_4^{2-}$	IC	M	10%	2.17E-03	0.5%	1.93E-02	4.6%	9.57E-03	0.4%	5.06E-03	1.0%
Br^-	IC	M	10%	<1.29E-02	--	<1.25E-02	--	<1.30E-02	--	<1.29E-02	--
CO_3^{2-}	TIC	M	10%	1.62E-01	1.4%	6.75E-01	0.3%	4.74E-01	2.7%	5.41E-01	0.5%
Al	ICP-ES	mg/L	10%	3.69E+02	1.1%	3.40E+03	1.6%	1.37E+03	1.0%	1.84E+03	0.5%
B	ICP-ES	mg/L	10%	4.74E+01	0.4%	1.34E+02	2.7%	1.13E+02	0.3%	1.32E+02	1.3%
Ca	ICP-ES	mg/L	10%	<5.63E+00	--	3.38E+01	2.0%	<5.66E+00	--	<5.62E+00	--
Cr	ICP-ES	mg/L	10%	1.17E+01	1.0%	1.17E+02	2.6%	5.20E+01	0.6%	8.24E+01	0.3%
Fe	ICP-ES	mg/L	10%	<1.39E+00	--	1.51E+02	3.5%	<1.40E+00	--	<1.39E+00	--
K	ICP-ES	mg/L	10%	<6.30E+01	--	5.09E+02	3.0%	2.60E+02	1.2%	3.37E+02	0.5%
Li	ICP-ES	mg/L	10%	<1.12E+01	--	<1.07E+01	--	<1.12E+01	--	<1.12E+01	--
Na	ICP-ES	mg/L	10%	3.87E+04	0.5%	1.68E+05	2.6%	1.17E+05	0.7%	1.44E+05	0.8%
		M		1.68E+00		7.31E+00		5.10E+00		6.27E+00	
P	ICP-ES	mg/L	10%	2.70E+01	0.2%	3.40E+02	3.1%	9.88E+01	1.7%	1.45E+02	0.5%
Si	ICP-ES	mg/L	10%	5.50E+01	0.7%	2.75E+02	4.7%	7.10E+01	0.3%	4.08E+01	1.9%
Zn	ICP-ES	mg/L	10%	<6.59E+00	--	<6.31E+00	--	<6.63E+00	--	<6.57E+00	--
TOC	TOC	mg C/L	10%	5.14E+02	1.6%	1.15E+03	2.4%	8.47E+02	1.7%	8.49E+02	0.7%
Anions	sum	M	--	1.84E+00	--	7.35E+00	--	5.09E+00	--	6.24E+00	--
Cations	sum	M	--	1.68E+00	--	7.32E+00	--	5.10E+00	--	6.28E+00	--

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

[a] Pu-239 mass assumes entire Pu-239/240 activity is Pu-239 [b] Average of only two values since the third was below detection

Table 3-2. Comparison of Uranium Results for the Tank 38H and 43H Samples from Two Methods of Measurement

Standard ICP-MS Results

analyte	method	units	est. 1σ	HTF-38-21-24		HTF-38-21-25		HTF-43-21-26		HTF-43-21-27	
				average	%RSD	average	%RSD	average	%RSD	average	%RSD
Sample Type	--	--	--	Surface		Sub-surface		Surface		Sub-surface	
U-233	ICP-MS	mg/L	20%	<1.01E-02	--	1.44E-02	2.6%	<1.01E-02	--	<1.01E-02	--
U-234	ICP-MS	mg/L	20%	<1.01E-02	--	1.97E-01	3.4%	<1.01E-02	--	<1.01E-02	--
U-235	ICP-MS	mg/L	20%	1.83E-01	0.8%	1.53E+00	2.8%	2.01E-01	0.7%	1.26E-01	1.4%
U-236	ICP-MS	mg/L	20%	1.07E-02	5.5%	1.30E-01	4.5%	1.34E-02	3.9%	<1.01E-02	--
U-238	ICP-MS	mg/L	20%	1.78E+01	0.5%	2.43E+02	3.2%	2.79E+01	0.9%	1.98E+01	0.2%
Total U	calc.	mg/L	--	1.79E+01	0.5%	2.45E+02	3.2%	2.81E+01	0.9%	1.99E+01	0.3%
U-235 / U	calc.	%	--	1.02	0.9%	0.63	0.5%	0.72	0.3%	0.63	1.1%

Uranium Separation/ICP-MS Results

analyte	method	units	est. 1σ	HTF-38-21-24		HTF-38-21-25		HTF-43-21-26		HTF-43-21-27	
				average	%RSD	average	%RSD	average	%RSD	average	%RSD
Sample Type	--	--	--	Surface		Sub-surface		Surface		Sub-surface	
U-233	ICP-MS	mg/L	20%	<4.00E-03	--	1.40E-02	4.4%	<4.07E-03	--	<4.18E-03	--
U-234	ICP-MS	mg/L	20%	4.58E-03	0.5%	2.03E-01	7.3%	6.45E-03	2.1%	5.38E-03	1.3%
U-235	ICP-MS	mg/L	20%	1.84E-01	0.2%	1.46E+00	4.7%	2.01E-01	1.5%	1.25E-01	0.7%
U-236	ICP-MS	mg/L	20%	1.09E-02	0.8%	1.24E-01	5.4%	1.30E-02	1.3%	8.31E-03	1.1%
U-238	ICP-MS	mg/L	20%	1.75E+01	0.5%	2.36E+02	5.1%	2.76E+01	0.9%	1.96E+01	0.4%
Total U	calc.	mg/L	--	1.77E+01	0.5%	2.38E+02	5.1%	2.78E+01	0.9%	1.97E+01	0.4%
U-235 / U	calc.	%	--	1.04	0.3%	0.61	1.3%	0.72	0.6%	0.63	0.3%

4.0 Conclusions

The results indicate the concentrations of most species in the Tank 38H surface sample decreased from the previous surface sample. The Tank 38H sub-surface sample shows only minor changes in concentration for soluble species in the solution (e.g., Na, Al, Cs-137) relative to the previous sample, but a large increase in concentrations for species typically associated with sludge solids (e.g., U, Pu, Fe, Si) likely because of an increase in sludge solids from the previous sample. The large differences in the concentrations of major components between the Tank 38H surface and sub-surface samples indicate significant stratification of solution species within the tank.

The Tank 43H surface sample is more concentrated than the previous samples while the Tank 43H sub-surface sample is fairly similar to the previous sample. The solution concentrations measured in the Tank 43H surface and sub-surface samples indicate some stratification within the tank.

The Tank 38H surface sample shows a slight increase in uranium concentrations and a large decrease in plutonium concentrations compared to the previous sample. The Tank 38H sub-surface sample shows a large increase in uranium and plutonium concentrations compared to the previous sample likely because of an increase in sludge solids. The total uranium and plutonium concentrations in the two Tank 43H samples both increased from previous sample results.

The sum of the major cations versus the sum of the major anions shows a difference of <10% for the two Tank 38H samples and the two Tank 43H samples providing an indication of good data quality for the non-radioactive analytes in the samples.

The silicon concentrations measured in the Tank 38H surface sample decreased compared with the previous sample results while the silicon increased significantly in the sub-surface sample likely due to the presence of sludge solids. The Tank 43H surface sample silicon concentration decreased compared to the previous surface sample results. The Tank 43H sub-surface sample silicon concentration increased relative to the previous surface sample. The samples analyzed show silicon concentrations ranging from 40.8 to 275 mg/L.

5.0 Acknowledgements

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