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Characterization of the March 2017 Tank 10 Surface Sample (Combination of HTF-10-17-30 and HTF-10-17-31) and Variable Depth Sample (Combination of HTF-10-17-32 and HTF-10-17-33)

S. H. Reboul July 2017 SRNL-STI-2017-00392, Revision 0

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S. H. Reboul

July 2017



OPERATED BY SAVANNAH RIVER NUCLEAR SOLUTIONS

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EXECUTIVE SUMMARY

Two surface samples (HTF-10-17-30 and HTF-10-17-31) and two variable depth samples (HTF-10-17-32 and HTF-10-17-33) were collected from SRS Tank 10 during March 2017 and submitted to SRNL for characterization. At SRNL, the two surface samples were combined in one container, the two variable depth samples (VDSs) were combined in another container, and then the two composite samples were each characterized by a series of physical, ionic, radiological, and elemental analysis methods. The surface sample composite was characterized primarily for Tank Farm corrosion control purposes, while the VDS composite was characterized primarily for Tank Closure Cesium Removal (TCCR) purposes.

The characterization results for the surface sample composite indicated that:

- the supernatant density was approximately 1.16 g/mL
- the supernatant sodium concentration was approximately 3.6 M and the dominant measured anions were nitrate, free hydroxide, sulfate, nitrite, and aluminate, at concentrations of approximately 1.2, 0.93, 0.48, 0.15, and 0.061 M, respectively
- the supernatant Cs-137 and Ba-137m activity concentrations were approximately 9.1E+07 and 8.6E+07 dpm/mL, respectively
- the total elemental cesium concentration in the supernatant was $\sim 2.1-2.4$ mg/L
- Cs-137 comprised ~20-23 % of the total elemental cesium

The characterization results for the <u>VDS</u> composite indicated that:

- the slurry and supernatant densities were both approximately 1.17 g/mL
- the total solids and dissolved solids concentrations were both approximately 19 wt%
- the insoluble solids concentration was < 1.0 wt%
- the slurry had no Bingham Plastic yield stress, the slurry viscosity was < 2.5 cP, and the slurry was considered to be Newtonian
- the supernatant sodium and potassium concentrations were approximately 3.2 and 0.0033 M, respectively
- the supernatant ammonium concentration was less than the minimum detection limit of ~ 0.003 M
- the detectable supernatant anions included nitrate, carbonate, sulfate, free hydroxide, nitrite, aluminate, oxalate, and chloride, with concentrations of approximately 1.0, 0.51, 0.38, 0.18, 0.13, 0.063, 0.009, and 0.004 M, respectively
- the supernatant Cs-137 and Ba-137m activity concentrations were approximately 8.6E+07 and 8.2E+07 dpm/mL, respectively
- the total elemental cesium concentration in the supernatant was approximately 2.1 mg/L, with Cs-137 contributing ~21 % of the cesium
- on a mass basis, the supernatant concentrations of Tc-99, total uranium, Np-237, and Pu-239 were approximately 0.63, 0.69, 0.051, and 0.0066 mg/L, respectively
- U-235 comprised ~13% of the total uranium mass
- the supernatant concentrations of stable elements sulfur, chromium, mercury, arsenic, selenium, and silver were approximately 13000, 15, 3.6, 0.15, 0.077, and 0.056 mg/L, respectively
- the supernatant concentrations of butanol, isobutanol, isopropanol, phenol, tetraphenylborate, and tributylphosphate were all less than the minimum detection limits, which ranged from 0.25 to 5.0 mg/L
- the supernatant concentration of total organic carbon was approximately 300 mg/L, much of which was contributed by oxalate

Comparison of the characterization results for the surface sample and the VDS suggests that the compositions of both samples were similar – both samples contained minimal insoluble solids and both samples contained moderately low concentrations of dissolved sodium salts. Based on the available data, the only significant difference between the surface sample and the VDS was the supernatant free hydroxide concentration, which for the VDS was about one-fifth that of the surface sample.

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LIST OF ABBREVIATIONS

AA	Atomic absorption
AD	Analytical Development
CVAA	Cold vapor atomic absorption
DI	Deionized
GC/MS	Gas chromatography/mass spectroscopy
HPLC	High performance liquid chromatography
ICP-AES	Inductively coupled plasma – atomic emission spectroscopy
ICP-MS	Inductively coupled plasma – mass spectroscopy
MDL	Minimum detection limit
MW	Molecular weight
n	Number of determinations
RSD	Relative standard deviation
SRNL	Savannah River National Laboratory
SRR	Savannah River Remediation
SRS	Savannah River Site
SVOA	Semivolatile organic analysis
TCCR	Tank closure cesium removal
TIC	Total inorganic carbon
TOC	Total organic carbon
VOA	Volatile organic analysis
VDS	Variable depth sample

1.0 Introduction

Two 200 mL dissolved salt surface samples from SRS Tank 10 were received at SRNL on March 22, 2017. These samples were collected 84.5 inches from the bottom of the tank, with identification numbers of HTF-10-17-30 and HTF-10-17-31.¹

Subsequently, two 200 mL dissolved salt variable depth samples (VDSs) from SRS Tank 10 were received at SRNL on March 23, 2017. These samples were collected 73.1 inches from the bottom of the tank, with identification numbers of HTF-10-17-32 and HTF-10-17-33.¹

At SRNL, the two surface samples were combined and mixed in a 500 mL polybottle, and then the two VDSs were combined and mixed in a separate 500 mL polybottle. The composited material in each of these polybottles served as the base material for use in characterizations supporting Tank Farm corrosion control and operation of the Tank Closure Cesium Removal (TCCR) unit.

The applicable scope of work for this characterization task is defined in Technical Task Request X-TTR-H-00065, Rev. 1,² Task Technical and Quality Assurance Plan SRNL-RP-2016-00705, Rev. 1,³ and in email correspondence from SRR included in Appendix A.

2.0 Objectives

The specific objectives were to characterize:

- a) the surface sample supernatant for
- density
- corrosion control anions
- elemental constituents including aluminum, potassium, and sodium
- cesium isotopes and Ba-137m
- total elemental cesium
- other incidental constituents
- b) the VDS slurry for
- density
- solids distribution
- rheology

c) the VDS supernatant for

- all principal anions and the ammonium cation
- elemental constituents including aluminum, arsenic, mercury, potassium, selenium, and sodium
- Tc-99, cesium isotopes, Ba-137m, uranium isotopes, Np-237, and Pu-239
- total elemental cesium
- butanol, isobutanol, isopropanol, phenol, tetraphenylborate, tributylphosphate, total organic carbon
- other incidental constituents

3.0 Methodology

3.1 Laboratory Methods

<u>Densities of the Surface Sample Supernatant and the VDS Slurry</u>: Densities were measured using weightcalibrated balances and 8-9 mL volume-calibrated plastic test tubes. Three individual sample aliquots were utilized in each set of density measurements (one set for the surface sample supernatant, one set for the VDS slurry, and one set for the VDS supernatant). Supernatant was generated as a filtrate by passing each slurry through a 0.45 μ m filtration membrane. The density of a deionized (DI) water standard was determined along with each set of density determinations, to demonstrate measurement accuracy. Density measurements for the surface sample supernatant were conducted at a temperature of ~24 °C, while those for the VDS slurry and supernatant were conducted at a temperature of ~26 °C. These temperatures were governed by the Shielded Cells conditions at the time of the measurements.

<u>Solids Distribution of the VDS</u>: Total solids and dissolved solids determinations of the VDS were performed by driving water from slurry and supernatant aliquots (respectively) at a nominal temperature of ~110 °C. Supernatant was generated as a filtrate by passing the slurry through a 0.45 μ m filtration membrane. Three individual slurry aliquots and three individual supernatant aliquots were utilized in the solids distribution measurements. The mass of each aliquot was ~3.0 g. Insoluble and soluble solids concentrations were calculated based on the total solids and dissolved solids measurements. The dissolved solids content of a 15 weight percent sodium chloride solution was determined alongside the sample determinations, to demonstrate measurement accuracy.

<u>Rheological Properties of the VDS</u>: Flow curves identifying VDS slurry shear stress as a function of shear rate were generated using the Haake RV-30 roto-viscometer fitted with an NV sensor and associated cup, at a temperature of 25° C. The shear rate was increased linearly from 0-2700/s over a seven minute period, held at 2700/s for one-half minute, and then reduced linearly from 2700-0/s over a seven minute period. The flow curves were analyzed under the assumption that the slurry was either a Newtonian fluid or a Bingham Plastic fluid. Duplicate rheology measurements were performed on a single 9.0 mL VDS slurry aliquot. Duplicate rheology measurements on a DI water aliquot were also performed, for quality assurance purposes. The flows curves for the slurry measurements are given in Appendix B, whereas the yield stress and viscosity results are reported in Section 4.0.

<u>Elemental Analysis of the Surface Sample Supernatant</u>: In preparation for the elemental analyses (prior to submittal), three supernatant aliquots were each diluted by an average factor of ~29 (on a volume basis), using 0.5 M HNO₃. Supernatant was generated as a filtrate by passing the surface sample slurry through a 0.45 μ m filtration membrane. Use of the 0.5 M HNO₃ diluent resulted in a final solution pH of ~1, which was considered beneficial for minimizing loss of constituents through sorption to the walls of the sample submittal vessels and through potential precipitation reactions. An applicable "acid blank" and a multi-element standard were submitted and analyzed alongside the acidified/diluted supernatant, for quality assurance purposes. Inductively coupled plasma atomic emission spectroscopy (ICP-AES) was performed on the acidified/diluted supernatant aliquots, to quantify elemental aluminum, potassium, sodium, and other incidental elemental constituents. Dilution-correction of the results was performed prior to reporting.

Anion Analysis of Surface Sample Supernatant: In preparation for the anion analyses (prior to submittal), three supernatant aliquots were each diluted by an average factor of ~28 (on a volume basis), using deionized water. Supernatant was generated as a filtrate by passing the surface sample slurry through a 0.45 μ m filtration membrane. Anion chromatography was performed on the diluted supernatant aliquots to quantify bromide, chloride, fluoride, formate, nitrate, nitrite, oxalate, phosphate, and sulfate. Base titration analyses were performed to quantify free hydroxide. Aluminate was quantified based on the ICP-AES supernatant aluminum concentration results, assuming 100% of the aluminum was present as aluminate. Applicable "blanks" were submitted and analyzed alongside the supernatant aliquots, for quality assurance purposes. Dilution-correction of the results was performed prior to reporting.

Elemental Analysis of the VDS Supernatant: Three undiluted aliquots of the VDS supernatant were submitted for ICP-AES, standard atomic absorption (AA), cold vapor atomic absorption (CVAA), and inductively coupled plasma mass spectroscopy (ICP-MS) analyses. The VDS supernatant was acquired by decanting clear solution off the top of the VDS, following an insoluble solids settling period of approximately four and two-thirds days. The ICP-AES analyses provided quantification of aluminum, potassium, sodium and other incidental elemental constituents. The standard AA analyses provided quantification of arsenic and selenium. The CVAA analyses provided quantification of mercury. Note that permanganate-sulfate digestions were performed on the supernatant aliquots prior to the CVAA analyses. The ICP-MS analyses provided quantification of elemental silver, thorium, and uranium, based on the applicable isotope distributions. For silver, the concentrations of isotopes 107 and 109 were summed, and the sum was adjusted for the Pd-107 contribution, as calculated from the Pd-105 concentration and the relative fission yield. The elemental thorium concentration was calculated based on the Th-232 concentration, assuming Th-232 was the primary contributor of thorium mass. For uranium, the concentrations of isotopes 233-236 and 238 were summed to identify the total elemental mass concentration. Applicable "blanks" were submitted and analyzed alongside the supernatant aliquots, for quality assurance purposes. In the case of the ICP-AES analyses, a multi-element standard was also submitted for quality assurance purposes.

<u>Anion Analysis of the VDS Supernatant</u>: Three undiluted aliquots of the VDS supernatant were submitted for anion chromatography, base titration, and total inorganic carbon (TIC) analyses. The VDS supernatant was acquired by decanting clear solution off the top of the VDS, following an insoluble solids settling period of approximately four and two-thirds days. Applicable "blanks" were submitted and analyzed alongside the supernatant aliquots, for quality assurance purposes. Anion chromatography was performed to quantify bromide, chloride, fluoride, formate, nitrate, nitrite, oxalate, phosphate, and sulfate. Base titration analyses were performed to quantify free hydroxide. TIC analyses were performed to quantify carbonate. Aluminate was quantified based on the ICP-AES supernatant aluminum concentration results, assuming 100% of the aluminum was present as aluminate.

<u>Ammonium Analysis of the VDS Supernatant</u>: Three undiluted aliquots of the VDS supernatant were submitted for cation chromatography to quantify ammonium. The VDS supernatant was acquired by decanting clear solution off the top of the VDS, following an insoluble solids settling period of approximately four and two-thirds days. An applicable "blank" was submitted and analyzed alongside the supernatant aliquots, for quality assurance purposes.

<u>Cesium Isotopes and Ba-137m in the Surface Sample and VDS Supernatants</u>: In the case of the surface sample, three supernatant aliquots were each diluted by an average factor of ~29 (on a volume basis), using 0.5 M HNO₃, and then submitted for analysis. The surface sample supernatant was generated as a filtrate by passing the surface sample slurry through a 0.45 µm filtration membrane. In contrast, in the case of the VDS, three undiluted supernatant aliquots were submitted for analysis, with the VDS supernatant acquired by decanting clear solution off the top of the VDS, following an insoluble solids settling period of approximately four and two-thirds days. Cs-133 was quantified by ICP-MS. Cs-134, Cs-137, and Ba-137m were quantified by gamma spectroscopy. Cs-135 was quantified by ICP-MS, following a chemical separation process to remove barium. Total elemental cesium was calculated by summing the mass concentrations of Cs-133, 134, 135, and 137. Applicable "blanks" were submitted and analyzed alongside the supernatant aliquots, for quality assurance purposes. Dilution-correction of the surface sample results was performed prior to reporting.

<u>Additional Radioisotopes in the VDS Supernatant</u>: Three undiluted aliquots of the VDS supernatant were submitted for ICP-MS analyses. The VDS supernatant was acquired by decanting clear solution off the top of the VDS, following an insoluble solids settling period of approximately four and two-thirds days. The specific radioisotopes quantified by ICP-MS included Tc-99, Th-232, U-233, U-234, U-235, U-236, U-238, Np-237, Pu-239, and Pu-240. An applicable "blank" was submitted and analyzed alongside the supernatant aliquots, for quality assurance purposes.

<u>Organic Constituents in the VDS Supernatant</u>: Three undiluted aliquots of the VDS supernatant were submitted for analysis. The VDS supernatant was acquired by decanting clear solution off the top of the VDS, following an insoluble solids settling period of approximately four and two-thirds days. Butanol, isobutanol, and isopropanol were quantified by gas chromatography/mass spectroscopy volatile organic analysis (GC/MS-VOA). Phenol and tributylphosphate were quantified by GC/MS semi-volatile organic analysis (GC/MS-SVOA). Tetraphenylborate was quantified by high performance liquid chromatography (HPLC). Total organic carbon (TOC) was quantified using a high temperature total carbon analyzer. An applicable "blank" was submitted and analyzed alongside the supernatant aliquots, for quality assurance purposes.

3.2 Format of the Reported Results

Mean results, based on the average of all applicable analytical determinations, are reported in this document, along with the percent relative standard deviation (%RSD) and the number of determinations (n) feeding each mean. %RSD provides an indication of the measurement variation between replicate determinations, but is typically not an indicator of analytical accuracy. In general, the one sigma analytical uncertainty as reported by Analytical Development was 10%, although it was sometimes lower or higher. Specifically, the one sigma analytical uncertainties reported by AD for concentrations exceeding the minimum detection limits (MDLs) were: a) ~10% for base titration, anion chromatography (excluding the oxalate analysis), ICP-AES, ICP-MS, and TIC/TOC analyses; b) ~20% for AA, CVAA, Cs-135, GC/MS, HPLC, and anion chromatography oxalate analyses; and c) ~5% for Cs-137/Ba-137m quantified by gamma spectroscopy. Given these uncertainties, only one to two of the leading digits reported for the AD analysis results should be considered significant.

3.3 Assessment of the Results

Multiple approaches were used to assess the validity of the analytical data being reported. The primary goal of this was to demonstrate that the reported results were both reasonable and consistent with expectations. Focus areas of the assessment included: a) densities, solids distribution, and rheology; b) charge balance of the ions in the supernatant; and c) dominant supernatant salts feeding the dissolved solids. Discussion of the assessment approaches and results is included in Section 4.0. Note that when characterization results were compared, percent differences were calculated as follows:

% Difference = $100 \times [(absolute value of the difference between results) \div (the average result)].$

The leading three digits of each numerical result were utilized when calculating % differences, regardless of the number of digits considered significant.

3.4 Quality Assurance

Standard laboratory quality assurance protocols were used to assure analytical data quality. This included use of blanks, standards, and replicate determinations.

Requirements for performing reviews of technical reports and the extent of review are established in manual E7 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. All R&D directions, analytical data, and pertinent supporting information for this task are captured in electronic notebook #L5578-00303-01.

4.0 Results and Discussion

4.1 Sample Weights and Appearances

Composite masses of the combined surface samples (HTF-10-17-30 and HTF-10-17-31) and the combined VDSs (HTF-10-17-32 and HTF-10-17-33) were 394 and 466 g, respectively.

For observation purposes, the two sample composites were transferred to separate clear graduated bottles and agitated to suspend the insoluble solids. Following agitation, each of the sample composites appeared to contain a minor quantity of insoluble solids, as evidenced by the medium brown color and the mild visible turbidity. The surface sample composite appeared slightly more turbid than the VDS composite, suggesting that the insoluble solids content of the surface sample composite might be slightly higher than that of the VDS composite.

Based on the graduations, the approximate total volumes of the surface and VDS composites were 335 and 400 mL, respectively. Since the targeted sample volume for each of the composites was 400 mL (2 x 200 mL), it appears that something hindered collection of one or both of the surface samples, while nothing hindered collection of the VDSs.

Photographs of the surface sample and VDS composites are given below in Figure 4-1. In the first photograph, both composites are shown immediately following agitation, with suspended insoluble solids causing the slurries to appear moderately brown and turbid. In the second and third photographs, the settled surface sample and VDS composites are shown, respectively, with relatively thin layers of insoluble solids accumulated on the bottoms of the bottles and reduced quantities of suspended solids, as

evidenced by the greater supernatant solution clarities. Note that the second and third photographs were taken after allowing the surface sample and VDS composites to sit undisturbed overnight.



Figure 4-1. Surface and VDS Composites Following Agitation and Settling

4.2 Characterization of the Surface Sample Composite

As shown in Table 4-1, the supernatant density of the surface sample supernatant was measured to be 1.16 g/mL, with an RSD of 0.6 %. This moderately low density reflects the moderately low sodium salt concentration, which as shown in Table 4-2, was ~3.6 M. Using an average correlation factor developed for nominal salt simulant solutions, the typical density for a 3.6 M salt solution is predicted to be ~1.17 g/mL.⁴ The small difference between the measured density value and the predicted density value gives confidence that the measured value is reasonable. The low RSD (0.6 %) of the mean indicates that the precision of the density measurements was good.

Mean Density, g/mL	%RSD (n=3)
1.16	0.6

Table 4-1. Density of the Surface Sample Supernatant

Ion	Mean Concentration, M	%RSD (n=3)
Na ⁺	3.59E+00	1.0
\mathbf{K}^+	< 1.1E-02	N/A
NO ₃ -	1.21E+00	0.6
Free OH ⁻	9.27E-01	3.9
SO_4^{2-}	4.80E-01	1.6
NO ₂ ⁻	1.46E-01	1.1
Al(OH) ₄ ⁻	6.09E-02	1.0
Br⁻	< 1.8E-02	N/A
$C_2O_4^{2-}$	< 1.6E-02	N/A
F ⁻	< 1.5E-02	N/A
Cl	< 8.0E-03	N/A
CHO ₂ ⁻	< 6.3E-03	N/A
PO_4^{3-}	< 3.0E-03	N/A
CO_{3}^{2-}	Not determined	N/A

 Table 4-2. Ionic Constituents in the Surface Sample Supernatant

Also shown in Table 4-2 are the measured concentrations of other ions, including potassium (the other key cation), as well as the principal anions impacting corrosion control, which includes nitrate, free hydroxide, sulfate, nitrite, and chloride. As shown in the table, the potassium concentration was minor, being lower than the MDL of 1.1E-02 M. The dominant anions included nitrate, free hydroxide, and sulfate, with concentrations of ~1.2, 0.9, and 0.5, respectively. (Note that carbonate was not measured, so its relative dominance was not accessed). Other detectable anions included nitrite and aluminate, with concentrations of ~0.15 and 0.06 M, respectively. The undetectable anions included bromide, oxalate, fluoride, chloride, formate, and phosphate, with concentrations below the MDLs of ~3E-03 to 2E-02 M. The RSDs for the ion determinations ranged from approximately 1-4 %, indicating good measurement precision.

Concentrations of the cesium isotopes and Ba-137m are given in Table 4-3, in terms of both activity and mass. As expected, Cs-137 and its short-lived progeny Ba-137m contributed the majority of the activity, at concentrations of \sim 9E+07 dpm/mL each. In contrast, the Cs-134 was undetectable (less than the MDL of \sim 3E+05 dpm/mL) and the Cs-135 was detected, but the Cs-135 concentration was reported as an upper

limit and known to be biased high, as indicated by the quality assurance protocols (the upper limit was \sim 800 dpm/mL). The low Cs-134 activity concentration was expected, due to the short half-life (\sim 2 years) coupled with the relatively high waste age (on the order of 40 years old). The RSDs for the Cs-133 and Cs-137/Ba-137m determinations ranged from approximately 1-2 %, indicating good measurement precision.

Isotope	Mean Activity Concentration, dpm/mL	Mean Mass Concentration, mg/L	%RSD (n=3)
Cs-133	N/A	1.58E+00	0.7
Cs-134	< 2.6E+05 (MDL)	< 8.9E-05 (MDL)	N/A
Cs-135	< 8.4E+02 (UL)	< 3.3E-01 (UL)	N/A
Cs-137	9.08E+07	4.70E-01	1.9
Ba-137m	8.59E+07	7.20E-08	1.9

 Table 4-3. Cesium and Barium Isotopes in the Surface Sample Supernatant

UL = upper limit, where the isotope was detected, but the result was biased high as indicated by the quality assurance protocols

On a mass basis, Cs-133 was the most dominant cesium isotope, at a concentration of \sim 1.6 mg/L. Cs-137 contributed the second most mass, at a concentration of \sim 0.5 mg/L, and Cs-135 contributed the third most mass, although its exact contribution is uncertain, due to knowledge that the analytical result was biased high. In contrast, the Cs-134 and Ba-137m contributed negligible masses at concentrations three or more orders of magnitude below those of the Cs-133, Cs-135, and Cs-137.

The total elemental cesium concentration and the isotopic distribution of the various cesium isotopes are given in Table 4-4. As shown in the table, the total cesium concentration (on a mass basis) was between approximately 2.1 and 2.4 mg/L. Based on this total, the Cs-133 contribution was 66-77 %, the Cs-137 contribution was 20-23 %, the Cs-135 contribution was ≤ 14 %, and the Cs-134 contribution was negligible (less than ~4E-03 %). Note that the total cesium concentration and the isotopic contributions should be considered estimates, as they do not take analytical uncertainties into account.

Isotope	Mean Concentration, mg/L	Isotopic Distribution, Mass % of Element
Cs-133	1.58	66 to 77
Cs-134	< 8.9E-05 (MDL)	< 4.3E-03
Cs-135	< 0.33 (UL)	≤ 14
Cs-137	0.47	20 to 23
Total Cs	2.05 to 2.38	

 Table 4-4. Distribution of Cesium Isotopes in the Surface Sample Supernatant

UL = upper limit, where the isotope was detected, but the result was biased high as indicated by the quality assurance protocols

Concentrations of elemental constituents are given in Table 4-5. As shown in table, aluminum and sodium were detected at concentrations of \sim 1600 and 83000 mg/L, respectively, with RSDs of 1.0 % indicating good measurement precision. Concentrations of all other elemental constituents determined by ICP-AES were less than the MDLs.

Constituent	Mean Concentration, mg/L	%RSD (n=3)
Ag	< 2.2E+01	N/A
Al	1.64E+03	1.0
В	< 2.8E+01	N/A
Ba	< 1.7E+00	N/A
Be	< 7.0E-01	N/A
Са	< 1.8E+01	N/A
Cd	< 2.3E+01	N/A
Ce	< 5.8E+01	N/A
Со	< 2.3E+01	N/A
Cr	< 2.3E+01	N/A
Cu	< 7.9E+01	N/A
Fe	< 2.9E+01	N/A
Gd	< 1.7E+01	N/A
Κ	< 4.2E+02	N/A
La	< 1.3E+01	N/A
Li	< 2.2E+01	N/A
Mg	< 3.1E+00	N/A
Mn	< 3.0E+00	N/A
Мо	< 6.7E+01	N/A
Na	8.26E+04	1.0
Ni	< 3.7E+01	N/A
Р	< 3.0E+02	N/A
Pb	< 3.0E+02	N/A
S	< 1.9E+04	N/A
Sb	< 3.1E+02	N/A
Si	< 2.9E+02	N/A
Sn	< 1.9E+02	N/A
Sr	< 6.1E-01	N/A
Th	< 8.1E+01	N/A
Ti	< 6.7E+01	N/A
U	< 3.1E+02	N/A
V	< 8.8E+00	N/A
Zn	< 3.2E+01	N/A
Zr	< 1 0E+01	N/A

 Table 4-5. Elemental Constituents in the Surface Sample Supernatant

4.3 Characterization of the VDS Composite

Density, solids distribution, and rheology results for the VDS composite are given in Table 4-6. Together these results suggest that: a) the VDS slurry contained minimal insoluble solids; and b) the supernatant phase contained a moderately low concentration of dissolved salts. Specifically, three characteristics support the presence of minimal insoluble solids – first, that the slurry density and supernatant density are the same (both are 1.17 g/mL); second, that the total solids concentration and the dissolved solids concentration are essentially the same (both are \sim 19 wt%), which results in a very low calculated insoluble solids content (<1.0 wt%, when taking the anticipated uncertainties into account); and third, that the Bingham Plastic yield stress of the slurry is zero (see Appendix B for the flow curves illustrating zero yield stress). Similarly, three characteristics support the presence of a moderately low concentration

of dissolved salts – first, that the supernatant density is moderately low (1.17 g/mL); second, that the measured dissolved solids concentration is moderately low (~19 wt%); and third, that the viscosity is moderately low (2.5 cP). As shown in the table, the RSDs for the density, solids distribution, and rheology measurements were all approximately one percent or less, indicating good measurement precision.

Property	Mean Result	%RSD (n)
Slurry density, g/mL	1.17	0.1 (3)
Supernatant density, g/mL	1.17	1.1 (3)
Total solids, wt% of slurry	18.8	0.8 (3)
Dissolved solids, wt% of supernatant	18.9	1.0 (3)
Insoluble solids (calculated value), wt% of slurry	< 1.0	N/A
Soluble solids (calculated value), wt% of slurry	17.8 to 18.8	N/A
Bingham Plastic yield stress of the slurry, Pa	0.0	N/A
Viscosity of the slurry, cP	2.5	< 0.1 (2)

Table 4-6. Densities, Solids Distribution, and Rheology of the VDS

Regarding the viscosity measurement, it should be noted that the 2.5 cP result is presumed to be biased high, as suggested by: a) the water standard viscosity measurements, which were approximately 60% higher than the standard value for water; and b) OLI modeling projections, based on the sodium salt concentrations identified in Table 4-8, which estimate the viscosity of the VDS supernatant to be ~1.5 cP (see Appendix C for the OLI input and output). Given this situation, the 2.5 cP viscosity result is viewed as an upper bound, with the actual viscosity of the slurry being < 2.5 cP. The relatively high uncertainty (and associated positive bias) of the viscosity measurement is thought to be due to the degraded condition of the NV sensor and associated cup that has been stored in the Shielded Cells unused for several years, without maintenance or replacement. Note that the NV sensor and cup are different from the MV1 sensor and cup routinely used for rheology measurements of concentrated sludge slurries - in contrast to the NV sensor and cup, the MV1 sensor and cup are in good condition, as evidenced by accurate viscosity quantification of the associated oil standard. If needs for future rheology measurements of low insoluble solids dissolved salt solutions are anticipated, replacement of the existing NV sensor and cup should be pursued.

Ion concentrations in the VDS supernatant and an assessment of the associated charge balance are given in Table 4-7. As shown in the table, the sodium concentration was \sim 3.2 M, and the dominant anions were nitrate, carbonate, and sulfate, at concentrations of \sim 1.0, 0.5, and 0.4 M, respectively. The potassium concentration was three orders of magnitude below the sodium concentration, but still detectable at a concentration of \sim 3E-03 M. In contrast, the ammonium concentration was below the MDL of \sim 3E-03 M. Detectable but less dominant anions included free hydroxide, nitrite, aluminate, oxalate, and chloride, at concentrations of \sim 0.2, 0.1, 0.06, 0.009, and 0.004 M, respectively. The other anions (fluoride, formate, bromide, and phosphate) were undetectable, at concentrations below the MDLs, which ranged from approximately 1E-03 to 5E-03 M. RSDs varied from 0.3 to 8.9%, which is considered normal for these types of analyses.

For the charge balance comparison, the molar ion concentrations were converted to equivalence concentrations based upon the applicable ionic charges – one for the measurable monovalent ions including sodium, potassium, free hydroxide, nitrite, aluminate, and chloride – and two for the measurable divalent ions including carbonate, sulfate, and oxalate. Note that the ions with concentrations

below the MDLs were not included in the charge balance assessment, as they were assumed to have an insignificant impact on the total charge balance.

As shown in Table 4-7, the sum of the pertinent cations was 3.22 eq/L, while the sum of the pertinent anions was 3.21 eq/L. The difference between these values is ~0.3 %, a value which indicates very high data consistency, as it is significantly below the total anticipated sampling and analysis uncertainty. Note that such high data agreement is atypical and should not interpreted to mean that the accuracy of the data is higher than expected – it simply means that the random uncertainties balanced themselves out fortuitously in a manner that favored high data consistency. An illustration of the typical 10% one sigma uncertainty still being applicable is given in the difference observed between the measured sodium concentration (~3.2 M) and the sodium concentration predicted based on the 1.17 g/mL supernatant density (~3.6 M).⁴

Ion	Mean	%RSD	Correspor	nding eq/L	Difformance
1011	Concentration, M	(n=3)	Cationic	Anionic	Difference
Na ⁺	3.22E+00	0.5	3.22	N/A	
K^+	3.32E-03	8.9	0.00332	N/A	
NH4 ⁺	< 2.8E-03	N/A	N/A	N/A	
NO ₃ ⁻	1.04E+00	5.0	N/A	1.04	
CO_{3}^{2}	5.06E-01	0.4	N/A	1.01	
SO_4^{2-}	3.81E-01	5.4	N/A	0.762	
Free OH ⁻	1.84E-01	1.9	N/A	0.184	
NO ₂ ⁻	1.28E-01	2.3	N/A	0.128	
Al(OH) ₄	6.31E-02	0.3	N/A	0.0631	
$C_2O_4^{2-}$	9.20E-03	2.3	N/A	0.0184	
Cl	4.06E-03	2.5	N/A	0.00406	
F ⁻	< 5.3E-03	N/A	N/A	N/A	
CHO ₂	< 2.2E-03	N/A	N/A	N/A	
Br	< 1.3E-03	N/A	N/A	N/A	
PO_4^{3-}	< 1.1E-03	N/A	N/A	N/A	
			$\Sigma = 3.22$	$\Sigma = 3.21$	0.3%

Table 4-7. Ionic Constituents in the VDS Supernatant and the Associated Charge Balance

A separate assessment utilizing the measured ion concentrations was performed to gauge consistency between the primary constituent concentrations and the measured dissolved solids content. Projected wt% values were calculated based on the molar concentrations of the known sodium salts, the molecular weights (MWs) of the sodium salts, and the measured density of the supernatant phase (1.17 g/mL). The results of these calculations are presented in Table 4-8, which shows that the projected dissolved solids content that was measured in the Shielded Cells. As mentioned above, such excellent agreement is atypical and should not be interpreted to mean that the accuracy of the data is higher than expected - it simply means that the random uncertainties balanced themselves out fortuitously in a manner that favored high data consistency. Nonetheless, the excellent agreement provides tangible support that the analytical results are reasonable.

Constituent	Molarity	MW, g	Projected wt%	Measured wt%	Difference
NaNO ₃	1.04	85.0	7.56		
Na ₂ CO ₃	0.506	106	4.58		
Na_2SO_4	0.381	142	4.62		
NaOH	0.184	40.0	0.63		
NaNO ₂	0.128	69.0	0.75		
NaAl(OH) ₄	0.0631	118	0.64		
$Na_2C_2O_4$	0.00920	134	0.11		
NaCl	0.00406	58.4	0.02		
			$\Sigma = 18.9$	18.9	0.0%

Table 4-8.	Projected	Dissolved	Solids (Content	Based or	n the Primar	y Constituents
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Concentrations of select isotopes in the VDS supernatant are given in Table 4-9, in terms of both activity and mass, where applicable. On an activity basis, the relative dominance of the detected radioisotopes was:

Cs-137/Ba-137m >> Tc-99 >> Pu-239 > Cs-135 > U-233 > Np-237 > U-234 > U-236 > U-235 ≈ U-238,

with activity concentrations ranging over eight orders of magnitude, from \sim 9E+07 dpm/mL down to \sim 0.4 dpm/mL. In contrast, on a mass basis, the relative dominance of the detected isotopes was:

Cs-133 > Tc-99 > U-238 > Cs-137 > Cs-135 > U-235 > Np-237 > U-236 > U-233 > Pu-239 > U-234 >> Ba-137m,

with mass concentrations ranging over seven orders of magnitude, from ~ 1.5 mg/L down to $\sim 7E-08$ mg/L. The RSDs of the isotope measurements were ~ 3 % or less, indicating good measurement precision.

Radionuclide	Mean Activity Concentration, dpm/mL	Mean Mass Concentration, mg/L	%RSD
Tc-99	2.36E+04	6.27E-01	2.2
Cs-133	N/A	1.50E+00	1.3
Cs-134	< 1.6E+05	< 5.4E-05	N/A
Cs-135	3.68E+02	1.44E-01	1.1
Cs-137	8.64E+07	4.47E-01	0.8
Ba-137m	8.17E+07	6.84E-08	0.8
Th-232	< 4.9E-04	< 2.0E-03	N/A
U-233	2.53E+02	1.18E-02	0.8
U-234	5.32E+01	3.83E-03	2.1
U-235	4.25E-01	8.86E-02	0.6
U-236	3.65E+00	2.54E-02	2.3
U-238	4.17E-01	5.58E-01	0.6
Np-237	8.01E+01	5.12E-02	0.3
Pu-239	9.12E+02	6.61E-03	3.2
Pu-240	< 1.0E+03	< 2.0E-03	N/A

 Table 4-9.
 Select Isotopes in the VDS Supernatant

As shown in Table 4-10, the total elemental cesium concentration was ~ 2.1 mg/L, with Cs-133 comprising ~ 72 % of the mass, Cs-134 comprising less than $\sim 3E-03$ % of the mass, Cs-135 comprising

 \sim 7 % of the mass, and Cs-137 comprising \sim 21 % of the mass. Note that these isotopic percentages should be considered estimates, as they do not take analytical uncertainties into account.

Isotope	Mean Concentration, mg/L	Isotopic Distribution, Mass % of Total Cs
Cs-133	1.50	71.7
Cs-134	< 5.4E-05	< 2.6E-03
Cs-135	0.144	6.9
Cs-137	0.447	21.4
Total Cs	2.09	

 Table 4-10. Distribution of Cesium Isotopes in the VDS Supernatant

As shown in Table 4-11, the total elemental uranium concentration was ~0.7 mg/L, with U-233 comprising ~2 % of the mass, U-234 comprising ~0.6 % of the mass, U-235 comprising ~13% of the mass, U-236 comprising ~4 % of the mass, and U-238 comprising ~81 % of the mass. As mentioned above, these isotopic percentages should be considered estimates, as they do not take analytical uncertainties into account.

Icotono	Mean Concentration,	Isotopic Distribution,
Isotope	mg/L	Mass % of Total U
U-233	1.18E-02	1.7
U-234	3.83E-03	0.6
U-235	8.86E-02	12.9
U-236	2.54E-02	3.7
U-238	5.58E-01	81.2
Total U	6.88E-01	

Table 4-11. Distribution of Uranium Isotopes in the VDS Supernatant

The relative magnitudes of the uranium, neptunium, and plutonium concentrations identified seem to reflect the relative solubilities of these radioelements in the dissolved salt waste – namely, that uranium is most soluble (~700 ppb total uranium was identified), plutonium is least soluble (~7 ppb total plutonium was identified - this assumes Pu-239 drives the elemental plutonium mass), and the neptunium solubility is intermediate (~50 ppb total neptunium was identified - this assumes Np-237 drives the elemental neptunium mass).

Concentrations of elemental constituents in the VDS supernatant are given in Table 4-12, in units of mg/L. Detectable elements, in order of highest concentration to lowest concentration, included sodium (\sim 7E+04 mg/L), sulfur (\sim 1E+04 mg/L), aluminum (\sim 2000 mg/L), potassium (\sim 100 mg/L), chromium (\sim 15 mg/L), mercury (\sim 4 mg/L), uranium (\sim 0.7 mg/L), arsenic (\sim 0.15 mg/L), selenium (\sim 0.08 mg/L), and silver (\sim 0.06 mg/L). MDLs of the undetectable elements ranged from a high of 45 mg/L (for phosphorus) to a low of \sim 0.002 mg/L (for thorium). RSDs of the detected elements were all less than or equal to \sim 9 %, which is considered good for this type of determinations.

Constituent	Mean Concentration mg/L	% RSD (n)
A		70 (II)
Ag	5.55E-02	7.3(3)
Al	1.70E+03	0.3(3)
As	1.4/E-01	1.9 (2)
В	< 2.0E+00	N/A
Ba	< 1.2E-01	N/A
Be	< 4.8E-02	N/A
Ca	< 1.2E+00	N/A
Cd	< 1.5E+00	N/A
Ce	< 4.0E+00	N/A
Co	< 1.6E+00	N/A
Cr	1.46E+01	0.8 (3)
Cu	< 5.5E+00	N/A
Fe	< 2.0E+00	N/A
Gd	< 1.2E+00	N/A
Hg	3.55E+00	5.0 (3)
K	1.30E+02	8.9 (3)
La	< 9.0E-01	N/A
Li	< 1.6E+00	N/A
Mg	< 2.2E-01	N/A
Mn	< 2.1E-01	N/A
Мо	< 4.7E+00	N/A
Na	7.40E+04	0.5 (3)
Ni	< 2.6E+00	N/A
Р	< 4.5E+01	N/A
Pb	< 2.1E+01	N/A
S	1.26E+04	0.9 (3)
Sb	< 2.2E+01	N/A
Se	7.66E-02	5.0 (3)
Si	< 1.7E+01	N/A
Sn	<1.3E+01	N/A
Sr	< 4.2E-02	N/A
Th	< 2.0E-03	N/A
Ti	< 4 7E+00	N/A
U	6 88E-01	07(3)
V	< 7 0F-01	N/A
Zn	< 5 9F-01	N/A
Zr	< 7 1E-01	N/A
— 1	· / . 1 L/ V I	1 1/ 1 1

 Table 4-12.
 Elemental Constituents in the VDS Supernatant

Concentrations of organic constituents in the VDS supernatant are given in Table 4-13, in units of mg/L. Butanol, isobutanol, isopropanol, phenol, tetraphenylborate, and tributylphosphate were all undetectable, at concentrations less than the MDLs, which ranged from 0.25 to 5.0 mg/L. In contrast, the measured TOC concentration was ~300 mg/L, with most of the TOC coming from oxalate (~220 mg/L from oxalate, based on the oxalate concentration identified in Table 4-7). The RSD for the TOC measurements was ~2%, indicating high measurement precision.

Constituent	Mean Concentration, mg/L	%RSD (n)
Butanol	< 2.5E-01	N/A
Isobutanol	< 2.5E-01	N/A
Isopropanol	< 2.5E-01	N/A
Phenol	< 1.0E+00	N/A
Tetraphenylborate	< 5.0E+00	N/A
Tributylphosphate	< 1.0E+00	N/A
TOC	2.95E+02	1.6 (3)

 Table 4-13. Organic Constituents in the VDS Supernatant

4.4 Comparison of the Surface Sample and VDS Supernatants

Measurements performed on both the surface sample supernatant and the VDS supernatant are summarized in Table 4-14, for purposes of comparison. This includes analytical results for density, detectable concentrations of principal ions and of cesium isotopes, and the concentration of elemental cesium.

Measurement	Surface Sample Supernatant	VDS Supernatant	% Difference
Density, g/mL	1.16	1.17	0.9
Na^+ , M	3.59	3.22	11
NO_3 , M	1.21	1.04	15
Free OH ⁻ , M	0.927	0.184	130
SO_4^{2-}, M	0.480	0.381	23
NO_2 , M	0.146	0.128	13
Al(OH) ₄ , M	0.0609	0.0631	4
Cs-133, mg/L	1.58	1.50	5
Cs-135, mg/L	< 0.33 (UL)	0.144	N/A
Cs-137, mg/L	0.47	0.447	5
Total Cs, mg/L	2.05 to 2.38	2.09	N/A

Table 4-14. Comparison of Surface Sample and VDS Supernatant Measurements

UL = upper limit, where the isotope was detected, but the result was biased high as indicated by the quality assurance protocols

Comparison of the relative surface sample and VDS analytical results suggests that on the whole, the surface sample and the VDS were quite similar to one another. Specifically, the difference between the densities was minor (0.9%) and clearly within the bounds of the anticipated analytical uncertainties. The differences between the results for the constituent concentration measurements were higher, but still less than the typical two sigma analytical uncertainty of ~20% in most cases, suggesting that the differences would not be considered statistically different. This applies to the measured concentrations for sodium, nitrate, nitrite, aluminate, and the cesium isotopes, where the % differences ranged from 4-15%.

In contrast, the difference between the respective sulfate concentrations was 23 %, and the difference between the respective free hydroxide concentrations was 130%. A 23 % difference for sulfate is considered marginally higher than the expected analytical uncertainty, and as such, may or may not be a true statistically significant difference. On the other hand, the difference between the respective free hydroxide concentrations is sufficiently high (130 %) that there is high confidence that the difference is real, and that the VDS supernatant contains significantly less free hydroxide than the surface sample

supernatant. Without taking analytical uncertainties into account, the VDS supernatant appears to contain about one-fifth the free hydroxide of the surface sample supernatant. Although the surface sample was collected only about one foot higher than the VDS, the analytical results suggest that the free hydroxide concentration closer to the layer of saltcake was suppressed. Such reduction of hydroxide near the saltcake surface may be indicative of saltcake dissolution thermodynamics, local quiescence, and/or water addition dynamics.

5.0 Conclusions

1) The VDS slurry contained minimal insoluble solids (< 1 wt%) and its supernatant phase contained a moderately low concentration of sodium salts (\sim 3.2 M).

2) The viscosity of the VDS was measured to be 2.5 cP, but is presumed to be biased high, due to degradation of the infrequently-used NV measurement sensor and cup. The slurry is considered to be a Newtonian fluid.

3) Analytical results for the dissolved salts in the VDS supernatant demonstrated exemplary charge balance and exemplary agreement with the dissolved solids measurements.

4) Cs-137 comprised approximately 21 % of the total elemental cesium mass in the VDS supernatant.

5) Ion concentrations in the VDS supernatant were very similar to those of the surface sample, with exception of the free hydroxide concentration, which was significantly lower in the VDS supernatant.

6.0 Recommendation

Acquire a new NV rheology sensor and cup for use in the Shielded Cells, if routine viscosity measurements of low insoluble solids dissolved salt slurries or salt supernatants will be pursued in the future.

7.0 References

- ¹ Email message from C. B. Sudduth to S. H. Reboul, "*Identification of Tank 10 Samples*," Savannah River Remediation, March 29, 2017. (See Appendix D).
- ² Fairchild, P. A., "Sample Analysis for Tank 10H in Support of Tank Closure Cesium Removal Unit," Savannah River Remediation, X-TTR-H-00065, Rev. 1, April 12, 2017.
- ³ Oji, L. N., "Task Technical and Quality Assurance Plan for Tank 10H Characterization in Support of Tank Closure Cesium Removal," Savannah River National Laboratory, SRNL-RP-2016-00705, Rev. 1, July 2017.
- ⁴ Walker, D. D. and G. K. Georgeton, "*Viscosity and Density of Simulated Salt Solutions*," Westinghouse Savannah River Company, WSRC-RP-89-01088, October 19, 1989.

Appendix A. SRR Correspondence Addressing Analytical Requirements



Re: Fw: Tank 10 Surface and variable Depth Samples Terri Fellinger to: Boyd Wiedenman Cc: Scott Reboul, Mark Keefer, Peter02 Fairchild, Joshua Segura

03/30/2017 09:13 AM

Boyd/Scott

Understand. No worries.

Surface sample - Please filter per the Tank Farm direction. In addition to the sample analyses
specified in the TTR, please report the other anions that are obtained from the analyses (F, formate,
and oxalate - I believe this completes the list), ICP scan (report all elements observed), and total Cs
(as you noted below).



Boyd,

Thanks for the feedback. We will need to build up a layer of solids as well on the filter in the process. Until then, I would assume that some particles will pass through as well. What we are really interested in knowing if what the solution looks like that will be fed to the columns. I have discussed this with Dave Martin and here is what we would like to do:

Obtain a mixed aliquot of the VDS and allow it to settle into 2 distinct layers (we were not sure how
much sample is required for the analyses, but want to preserve as much of the original sample if
possible). In the field, we will stop recirculation, pull the sample and send to F/H Lab for analyses to
support the SAC. When the lab receives the sample, if there are solids, they typically would allow
those to settle and then aliquot or decant the clear solution prior to analysis. We anticipate this
process to take a minimum of 4 to 5 days.



 Re: Tank 10 Corrosion Data
 Image: Control Corrosion Data
 Image: Control C

History:

Scott,

As we discussed last Friday, the density of both samples (surface/VDS) are essentially the same. So if the Na (I am guessing the anions would provide insight) molarity comes back and it is low, we would like to delay the VDS special separations that would be required for comparing the results against the Saltstone WAC. Couple of concerns in doing the analysis for special separations would be... the large volume required for the sample analysis and the worry that the detection limit is to high to be of use (due to the dilute sample).







Appendix C. OLI Modeling Input and Output Applicable to the VDS Supernatant

SinglePoint Results 6/20/2017 12:48:17 PM

OLIAnalyzer

Calculation Summary

SinglePoint Calculation

Unit Set: Metric (moles)

Automatic Chemistry Model Aqueous (H+ ion) Databanks: Public

Isothermal Calculation 25.0000 °C 1.00000 atm

Stream Inflows Row Filter Applied: Only Non Zero Values

Species	mol
Water	55.5082
Sodium aluminate	0.0631000
Sodium carbonate	0.506000
Sodium chloride	4.06000e-3
Sodium hydroxide	0.184000
Sodium nitrate	1.04000
Sodium nitrite	0.128000
Sodium oxalate	9.20000e-3
Sodium sulfate	0.381000

Stream Parameters

Row Filter Applied: Only Non Zero Values column Filter Applied: Only Non Zero Values

Mixture Properties

StrAmt	57.8236	mol
Temp	25.0000	°C
Pres	1.00000	atm

Aqueous Properties

pH	13.2960	
Ionic Strength (x-based)	0.0543545	mol/mol
Ionic Strength (m-based)	3.29477	mol/kg
Osmotic Pressure	85.6309	atm
Elec Cond, specific	13.8867	mho/m
Elec Cond, molar	6.34396e-3	m2/ohm-mol
Viscosity, absolute	1.51778	cP
Viscosity, relative	1.70400	
Standard Liquid Volume	1.11482	L
Volume, Std. Conditions	1.05774	L

Appendix D. Email Message Identifying the March 2017 Tank 10 Sampling Locations

From: To:	Christie Sudduth/SRR/Srs Scott Reboul/SRNL/Srs@Srs
Date:	03/29/2017 06:47 AM
Subject:	Re: Identification of Tank 10 samples
Scott,	
HTE-10-1	7.30 was a surface sample (so was 84.5 inches from the tank bottom)

HTF-10-17-30 was a surface sample (so was 84.5 inches from the tank bottom) HTF-10-17-31 was a surface sample (so was 84.5 inches from the tank bottom) HTF-10-17-32 was a VDS sampled 73.12 inches from the tank bottom HTF-10-17-33 was a VDS sampled 73.12 inches from the tank bottom

Distribution:

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