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Analytical Results from Salt Solution Feed Tank (SSFT) Samples HTF-16-6 and HTF-16-40

T. B. Peters

September 2016

SRNL-STI-2016-00407, Revision 0



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EXECUTIVE SUMMARY

Two samples from the Salt Solution Feed Tank (SSFT) were analyzed by SRNL, HTF-16-6 and HTF-16-40. Multiple analyses of these samples indicate a general composition almost identical to that of the Salt Batch 8-B feed and the Tank 21H sample results.

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LIST OF ABBREVIATIONS

AD	Analytical Development
ARP	Actinide Removal Process
ICPES	Inductively Coupled Plasma Emission Spectroscopy
MCU	Modular Caustic-Side Solvent Extraction Unit
SRNL	Savannah River National Laboratory
SSFT	Salt Solution Feed Tank
SSRT	Salt Solution Receipt Tank

1.0 Introduction

Tank 49H is the source of the material that is processed in each Salt Batch. Material from Tank 49H is transferred to the Actinide Removal Process (ARP), where at this time, it is simply filtered through 512-S. This filtrate is sent to the Salt Solution Receipt Tank (SSRT), which in turn is transferred to the Salt Solution Feed Tank (SSFT). Material from the SSFT is then processed at the Modular Caustic-Side Solvent Extraction Unit (MCU) to remove the cesium.

Recently the question was posed as to whether or not the contents of Tank 49H had been adequately mixed when the Salt Batch 8-B material from Tank 21H was introduced,^Δ which would have led to stratification in Tank 49H.

SRNL was requested to analyze two samples removed from the SSFT. One sample (HTF-16-6) was pulled on 1/8/16, and the second (HTF-16-40) on 4/26/16.

2.0 Experimental Procedure

Both of the samples arrived in 82 mL steel dip bottles. Samples were diluted ~5-10 fold with deionized water for dose reasons and delivered to Analytical Development (AD) for multiple analyses; gammascan, PuTTa, ⁹⁰Sr, Inductively Coupled Plasma Emission Spectroscopy (ICPES), and Ion Chromatography Anions (IC-Anions). Sample dilutions are already accounted for in the reported results. The samples were not otherwise altered.

Results from the analyses were compared to the Salt Batch 8-B blend feed estimates.ⁱ As well as the Tank 21H sample results for the Salt Batch 8 characterization sample.ⁱⁱ

2.1 Quality Assurance

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.

3.0 Results and Discussion

3.1 Sample HTF-16-6 (January 2016)

The results of the analyses from this sample were compared to the Salt Batch 8-B blend estimate and the Tank 21H results and are given as ratios of the Salt Batch 8-B or Tank 21H values divided by the HTF-16-6 sample result. Relevant results are reported in Table 1. Values in parentheses are the 1-sigma analytical uncertainty. The analytical uncertainty for the cations (ICPES) and anions (IC-A) is 10%.

^Δ When material is introduced into Tank 49H, the only mixing is through the addition of the new material.

Table 1. First Results from Sample HTF-16-6.

Analyte	Result	Tank 21H/Result	SB 8-B/Result
¹³⁷ Cs	1.26E+08 dpm/mL (5.0%)	3.74	3.44
²³⁸ Pu	3.52E+03 dpm/mL (18%)	16.0	32.7
⁹⁰ Sr	1.01E+04 dpm/mL (24%)	48.7	102
Al	1.50E+03 mg/L	3.61	3.51
B	2.05E+01 mg/L	3.46	3.13
Cr	2.01E+01 mg/L	3.60	3.43
K	1.49E+02 mg/L	4.33	4.07
Na	3.84E+04 mg/L	3.70	3.78
P	6.49E+02 mg/L	3.56	2.70
Cl	1.29E+02 mg/L	2.56	2.99
nitrite	1.09E+04 mg/L	3.52	3.40
nitrate	3.21E+04 mg/L	3.81	3.87
phosphate	1.40E+02 mg/L	3.88	3.82
sulfate	1.57E+03 mg/L	3.36	3.53

If one excludes the Pu and Sr results, which are subject to residual monosodium titanate (MST) in the ARP filter system, then a pattern emerges. On average, the Salt Batch 8-B results are $3.47 \pm 0.38 \times$ (%RSD = 11%) the results from the HTF-16-6 sample. On average, the Tank 21H results are $3.51 \pm 0.56 \times$ (%RSD = 16%) the results from the HTF-16-6 sample.

Normally these results would indicate dilution at the source, but as a confirmation, the density of the HTF-16-6 sample was measured and found to be 1.271 g/mL, which is almost identical to that of either the Salt Batch 8-B material or the Tank 21H sample. This rules out dilution of the source material. The only other possible dilution source is that during laboratory processing in the cells, some of the sample that was supposed to be introduced into the sample bottle instead ended up on the side of the bottle. This is an occupational hazard due to the lack of depth perception and is difficult to avoid.

A second limited set of analyses on HTF-16-6 was performed and reported in Table 2. These samples were not diluted in any way. Values in parentheses are the 1-sigma analytical uncertainty. The analytical uncertainty for the cations (ICPES) is 10%. The analytical uncertainty for the ¹³⁷Cs is 5%.

Table 2. Second Results from Sample HTF-16-6.

Analyte	Result	Tank 21H/Result	SB 8-B/Result
¹³⁷ Cs	4.47E+08 dpm/mL	1.06	0.973
Al	5.12E+03 mg/L	1.06	1.03
B	7.18E+01 mg/L	0.987	0.892
Cr	7.00E+01 mg/L	1.03	0.986
K	5.35E+02 mg/L	1.20	1.13
Na	1.44E+05 mg/L	0.990	1.01
P	2.00E+02 mg/L	1.15	0.873

Again, when examining the ratio of the Salt Batch 8-B and Tank 21H results to the second set of HTF-16-6 results, a pattern emerges. On average, the Salt Batch 8-B results are 98.5% (%RSD = 8.8%) the results from the HTF-16-6 sample. On average, the Tank 21H results are 107% (%RSD = 7.5%) the results from the HTF-16-6 sample.

This comparison shows that the undiluted samples are essentially the same as either the Tank 49H feed (Salt Batch 8-B estimate) or the Tank 21H sample and reinforces the conclusion that the original sample was diluted during laboratory cells operations. Given the small difference in the comparisons between the Salt Batch 8-B and Tank 21H results, it is difficult to determine which the SSFT is more like.

3.2 Sample HTF-16-40 (April 2016)

Material from this sample was nominally diluted 10:1 with deionized water and sent for multiple analyses. The results of the analyses from this sample were compared to the Salt Batch 8-B blend estimate and the Tank 21H results and are given as ratios of the Salt Batch 8-B or Tank 21H values divided by the HTF-16-40 sample result. Relevant results are reported in Table 3. Values in parentheses are the 1-sigma analytical uncertainty. The analytical uncertainty for the cations (ICPES) and anions (IC-A) is 10%.

Table 3. Results from Sample HTF-16-40.

Analyte	Result	Tank 21H/Result	SB 8-B/Result
¹³⁷ Cs	4.75E+08 dpm/mL (5.0%)	0.996	0.916
²³⁸ Pu	8.11E+04 dpm/mL (5.6%)	0.695	1.42
⁹⁰ Sr	1.08E+06 dpm/mL (27%)	0.453	0.949
Al	5.42E+03 mg/L	0.997	0.970
B	6.97E+01 mg/L	1.02	0.918
Cr	7.01E+01 mg/L	1.03	0.985
Na	1.49E+05 mg/L	0.956	0.976
nitrite	4.14E+04 mg/L	0.927	0.895
nitrate	1.15E+05 mg/L	1.06	1.08
sulfate	7.28E+03 mg/L	0.723	0.759

If one excludes the Pu and Sr results^Σ, which are subject to residual MST in the ARP filter system, then a pattern emerges. On average, the Salt Batch 8-B results are 93.7% (%RSD = 9.7%) the results from the HTF-16-40 sample. On average, the Tank 21H results are 96.3% (%RSD = 11%) the results from the HTF-16-40 sample. This comparison shows that the HTF-16-40 sample is essentially the same as the Tank 49H feed (Salt Batch 8-B estimate) and the Tank 21H sample results. The similarity in the values makes it impossible to determine which sample the SSFT is more like.

4.0 Conclusions

Two samples from the Salt Solution Feed Tank (SSFT) were analyzed by SRNL, HTF-16-6 and HTF-16-40. Multiple analyses of these samples indicate a general composition almost identical to that of the Salt Batch 8-B feed and the Tank 21H sample results.

^Σ The sulfate results were also excluded from the comparison due to both results being lower than expected. The reason for the relatively high sulfate result in the SSFT is not known at this time.

5.0 References

ⁱ D. L. McWhorter, “Blend Evaluation for Tank 49H Feed for ISDP Salt Batch 8-B August 2015”, X-ESR-H-00769, August 4, 2015.

ⁱⁱ T. B. Peters, A. L. Washington II, “Sample Results from the Interim Salt Disposition Program Macrobatches 8 Tank 21H Qualification Samples”, SRNL-STI-2014-00561, January 2015.

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