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Tank 21H Supernate Sample Cross-check Analysis - 2007

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Summary

The intent of this analysis was to perform cross-checks against routine F/H Lab analyses (corrosion, enrichment, soluble uranium, plutonium and feed qualification program). This Tank 21H supernate sample was analyzed mainly for the following contaminants and radionuclides: free OH⁻, NO₂⁻, NO₃⁻, SO₄⁻², CO₃⁻², Al, Si, Na and Pu-isotopes, total gamma and U- isotopes.

Results of this analysis indicate the following:

- The density of the 'as-received' Tank 21H supernate is 1.01 (st.dev. = 0.01) g/mL.
- Total beta/gamma and total alpha for the Tank 21H 'as-received' supernate are, respectively, 1.49E+06 (st.dev. = 6.17E+04) and <1.93E+04 dpm/mL.
- Plutonium isotopic (Pu-238, Pu-239/240, Pu-241 and Pu-242) concentrations in the supernate were, respectively, <1.22E+02, 1.07E+02 (st.dev.= 2.04E+01), < 2.25E+03 and < 5.87E+05 dpm/mL.
- Cesium-137 concentration in the Tank 21H 'as-received' supernate sample was 5.68E+05 (st.dev. = 1.94E+04)dpm/mL.
- Uranium-235 and uranium-238 concentrations in the supernate sample were, respectively, 1.77E-01 (st.dev.=1.79E-02) and 2.93E+01 (st.dev.=9.12E-01) mg/L.
- Other uranium isotopic species (U-233, U-234, and U-236) and Np-237 concentrations in the supernate were all below the method detection limit of < 4.16E-02 mg/L.
- The concentrations for aluminum, silicon and sodium were, respectively, 1.44E+01 (st.dev.=1.64E+00), 3.27E+01 (st.dev.=6.50E+00) and 9.98E+03 (st.dev. =1.61E+02) mg/L.
- The concentrations for nitrate, nitrite, and sulfate were, respectively, 3.53E+03 (st.dev.= 2.19E+01), 6.20E+03 (st.dev.= 3.27E+01) and 3.11E+01 (st.dev.= 1.74E+00) mg/L.
- The concentrations for free hydroxide (OH⁻) and carbonate were, respectively, 8.37E-02 (st.dev. =3.70E-02) and 7.99E-05 (st.dev.=1.43E-05) molar.

1.0 Introduction

Liquid Waste Processing Engineering personnel requested a crosscheck analysis of F/H Laboratory sample results for Tank 21H samples (CSTF sample ID HTF-21-06-111). The intent of this analysis was to perform cross-checks against routine F/H Laboratory analyses (corrosion, enrichment, soluble uranium, plutonium and feed qualification program). The Tank 21H sample analyzed was pulled and delivered to Savannah River National Laboratory (SRNL) on October 10, 2006. This sample was analyzed in accordance with Technical Task Request number HLE-TTR-2006-002. This work complies with the work scope and documents results of the chemical analysis of the “as received” Tank 21H supernate sample as defined in the following plan: L. N. Oji, “Task Technical Quality Assurance and Characterization Plan for Tank 21H Supernate Sample Cross-check Analysis-2006,” WSRC-RP-2005-01947, Rev. 0.

2.0 Results of Analyses of Tank 21H Supernate Sample

This Tank 21H supernate sample was analyzed for the following chemicals and radionuclides: free OH^- , NO_2^- , NO_3^- , SO_4^{2-} , CO_3^{2-} , Al, Si, Na and Pu-isotopes, total gamma and U- isotopes. The analysis of this Tank 21H sample for other chemicals, such as mercury, was performed to meet disposal requirements for analytical residues. The “as received” supernate sample was a clear solution with no visible suspended particles or debris. The sample preparation, which involved an eleven-fold dilution of the original “as received” Tank 21H supernate sample with either 5.0 M nitric acid or de-ionized water, was accomplished in the SRNL shielded cell and submitted to SRNL Analytical Development (AD) for the required analyses. Preparation for Silicon analysis required the use of the warm-acid-strike protocol. Results of duplicate analysis are given for each species along with an average and standard deviation (st.dev.). In cases where one or both of the results are below the limit of detection, no standard deviation is given. For species where the concentration fell below the lower limit of detection, the lower limit of detection is reported, preceded by “<”.

Analytical methods used included atomic absorption (AA) for Na analysis and inductively coupled plasma-emission spectroscopy (ICP-ES) for Si, Na, Al, and SO_4^{2-} . AD used Ion chromatography (IC) to measure the anions (NO_2^- , NO_3^-), and a titration method for CO_3^{2-} and free OH^- . Radionuclide contaminants were determined using counting techniques for gamma and inductively coupled plasma-mass spectroscopy (ICP-MS) and plutonium-thenoyltrifluoroacetone (PUTTA) for the actinides. Where interferences exist, the counting was preceded by a chemical separation.

The results of the chemical analyses are given in Tables 1 and 2.

Table 1. Tank 21H Sample Results for Selected Anions, Cations and General Parameters.

Analyte	Sample 1	Sample 2	Average	St. Deviation	Units
Nitrate (NO ₃ ⁻)	3.55E+03	3.52E+03	3.53E+03	2.19E+01	mg/L
Nitrite (NO ₂ ⁻)	6.22E+03	6.18E+03	6.20E+03	3.27E+01	mg/L
Free Hydroxide (OH ⁻)	1.10E-01	5.76E-02	8.37E-02	3.70E-02	molar
Carbonate (CO ₃ ⁻²)	9.00E-05	6.98E-05	7.99E-05	1.43E-05	molar
Sulfate (SO ₄ ⁻²)	3.23E+01	2.99E+01	3.11E+01	1.74E+00	mg/L
Formate (CHO ₂ ⁻)	3.34E+02	2.30E+02	2.82E+02	7.38E+01	mg/L
Phosphate (PO ₄ ⁻³)	< 1.23E+01	< 1.22E+01	< 1.23E+01	-	mg/L
Mercury (Hg)	1.31E+01	1.35E+01	1.33E+01	2.32E-01	mg/L
Aluminum (Al)	1.55E+01	1.32E+01	1.44E+01	1.64E+00	mg/L
Sodium (Na) from AA	8.11E+03	8.27E+03	8.19E+03	1.13E+02	mg/L
Sodium (Na) from ICP-ES **	9.87E+03	1.01E+04	9.98E+03	1.61E+02	mg/L
Silicon (Si)	3.73E+01	2.81E+01	3.27E+01	6.50E+00	mg/L
Density	1.00	1.01	1.01	0.01	g/mL

** Recommended for use in cross-check analysis

Table 2. Tank 21H Sample Results for Selected Radionuclides

Analyte	Sample 1	Sample 2	Average	St. Deviation	Units
Uranium-233 (²³³ U)	< 4.23E-02	< 4.08E-02	< 4.16E-02	-	mg/L
Uranium-234 (²³⁴ U)	< 4.23E-02	< 4.08E-02	< 4.16E-02	-	mg/L
Uranium-235 (²³⁵ U)	1.65E-01	1.90E-01	1.77E-01	1.79E-02	mg/L
Uranium-236 (²³⁶ U)	< 4.23E-02	< 4.08E-02	< 4.16E-02	-	mg/L
Uranium-238 (²³⁸ U)	2.95E+01	2.89E+01	2.92E+01	4.74E-01	mg/L
Neptunium-237 (²³⁷ Np)	< 4.23E-02	< 4.08E-02	< 4.16E-02	-	mg/L
Plutonium-238 (²³⁸ Pu)	< 1.71E+02	< 7.38E+01	< 1.22E+02	-	dpm/mL
Plutonium-239/240	9.30E+01	1.22E+02	1.07E+02	2.04E+01	dpm/mL
Plutonium-241 (²⁴¹ Pu)	< 1.11E+03	< 3.39E+03	< 2.25E+03	-	dpm/mL
Plutonium-242 (²⁴² Pu)	< 5.98E+02	< 5.76E+02	< 5.87E+02	-	dpm/mL
Cesium-137 (¹³⁷ Cs)	5.54E+05	5.82E+05	5.68E+05	1.94E+04	dpm/mL
Total Alpha	< 1.89E+04	< 1.96E+04	< 1.93E+04	-	dpm/mL
Total Beta/Gamma	1.45E+06	1.54E+06	1.49E+06	6.17E+04	dpm/mL

Distribution:

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