

12 January 2017

SRNL-L3100-2016-00229, Rev. 0

To: E. J. Freed

From: C. L. Crawford *CLC*

Results for the Fourth Quarter Calendar Year 2016 Tank 50H Salt Solution Sample

Approved by:

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SUMMARY

In this memorandum, the chemical and radionuclide contaminant results from the Fourth Quarter Calendar Year 2016 (CY16) sample of Tank 50H salt solution are presented in tabulated form. The Fourth Quarter CY16 Tank 50H samples (a 200 mL sample obtained 6" below the surface (HTF-5-16-90) and a 1 L sample obtained 66" from the tank bottom (HTF-50-16-91) were obtained on October 4, 2016 and received at Savannah River National Laboratory (SRNL) on the same day.¹ Prior to obtaining the samples from Tank 50H, a single pump was run at least 4.4 hours and the samples were pulled immediately after pump shut down.¹ The information from this characterization will be used by Defense Waste Processing Facility (DWPF) & Saltstone Facility Engineering for the transfer of aqueous waste from Tank 50H to the Saltstone Production Facility, where the waste will be treated and disposed of in the Saltstone Disposal Facility. This memorandum compares results, where applicable, to Saltstone Waste Acceptance Criteria (WAC) limits and targets.² The chemical and radionuclide contaminant results from the characterization of the Fourth Quarter CY16 sampling of Tank 50H were requested by Savannah River Remediation (SRR) personnel³ and details of the testing are presented in the SRNL Task Technical and Quality Assurance Plan (TTQAP).⁴ This memorandum is part of Deliverable 2 from SRR request.³ Data pertaining to the regulatory limits for Resource Conservation and Recovery Act (RCRA) metals will be documented at a later time per the TTQAP for the Tank 50H saltstone task.⁴

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The following facts pertaining to the WAC are drawn from the analytical results provided in this memorandum:

- WAC targets or limits were met for all analyzed chemical and radioactive contaminants for which the detection limits are below the WAC targets or limits.
- Norpar 13 and Isopar L have higher detection limits⁵ compared with the Saltstone WAC. The data provided in this memorandum is based upon the Norpar 13 and Isopar L concentrations from the sample obtained 6” below the surface. Due to the limited solubility of these materials in aqueous solution and the limited mixing of the tank by a single pump for 4.4 hours before sampling, Norpar 13 and Isopar L concentrations may not represent the overall concentrations of the analytes in Tank 50H.
- Minimum detection limits are reported for ⁹⁴Nb, ²⁴⁷Cm, ²⁴⁹Cf, and ²⁵¹Cf as determined from the minimum detectable activity associated with the radiochemical methods used for these radionuclides. The reported detection limits are above the requested SRR target minimum detection limit concentrations.⁶ However, the reported minimum detection limits reported for the Fourth Quarter CY16 Tank 50H sample for these four radionuclides are all lower than the estimated detection limits initially established by SRNL in 2009.⁷ Thus per guidance from SRR,⁶ SRNL continues to achieve as low as practical detection limits for these radionuclides.

TABLES CONTAINING RESULTS

Unless otherwise stated, all of the concentrations presented in the tables (except upper limits) are averages based on triplicate analyses of the Fourth Quarter CY16 Tank 50H samples. A memorandum reporting the average Cs-137 value has been previously issued.⁸ The standard deviation of each average is also presented. Several of the contaminants were either not detected in the slurry samples or detected at values below the method reporting limit (MRL). For contaminants not detected or detected below the MRL, the result is preceded by a “<”, which indicates the result is an upper limit based on the sensitivity of the method used to analyze the individual analyte. If only one value out of the triplicate analysis is above the detection limit, then that single value is reported and noted in the tables. Also, if only two values out of the triplicate analyses are above the detection limit, then the average of those two values is reported and noted in the tables. Data reported for atomic absorption (AA), cold-vapor atomic absorption (CVAA), inductively coupled plasma atomic emission spectroscopy (ICP-AES) and inductively coupled plasma mass spectroscopy (ICP-MS) are derived from the digested Tank 50H supernate by the aqua regia method. All analytical methods shown by the acronyms in the tables for this memorandum have been previously defined in the TTQAP.⁴

Mercury (Hg) speciation data shown in Table 1, Table 2, and Table 5 are taken from previous work.⁹ These species include elemental mercury (Hg(0)), monomethyl mercury, ethyl mercury, and dimethyl mercury. Monomethyl, ethyl, and dimethyl mercury are organomercury species. The concentration values for the organomercury species are calculated from the Hg speciation data on a mg Hg/L basis.⁹

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As a sample calculation for monomethyl mercury, information from Table 1 of reference 9 shows that the reported monomethyl concentration on a mg Hg/L basis is 61.6 mg Hg/L. This value is then multiplied by the formula weight of monomethyl mercury from the WAC² (215.62 g monomethyl mercury/mol) divided by the molecular weight of Hg (200.6 g Hg/mol). Thus the calculated concentration of the species monomethyl mercury is $61.6 \text{ mg Hg/L} \times (215.62 \text{ g monomethyl mercury/mol} / 200.6 \text{ g Hg/mol}) = 66.2 \text{ mg monomethyl mercury/L}$.

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Table 1. Chemical Contaminants from Fourth Quarter CY16 Tank 50H Samples and Saltstone WAC, Revision 16, Attachment 8.1 Limits²

<u>Chemical Name (Formula)</u>	<u>Method</u>	<u>Average Concentration (mg/L)</u>	<u>Std. Dev.</u>	<u>WAC Limit (mg/L)</u>
Aluminate ($\text{Al}(\text{OH})_4^-$)	ICP-ES	1.59E+04 ^a	4.34E+01	4.08E+05
Ammonium (NH_4^+)	IC	< 1.00E+01	NA	2.12E+02
Carbonate (CO_3^{2-})	TIC	1.53E+04 ^b	8.65E+01	1.20E+05
Chloride (Cl^-)	IC	4.28E+02	2.08E+00	7.95E+03
Fluoride (F^-)	IC	< 1.00E+02	NA	4.07E+03
Free Hydroxide (OH^-)	Total Base	3.45E+04 ^b	9.00E+02	1.58E+05
Nitrate (NO_3^-)	IC	1.14E+05	1.15E+03	4.37E+05
Nitrite (NO_2^-)	IC	3.05E+04	3.21E+02	2.14E+05
Oxalate ($\text{C}_2\text{O}_4^{2-}$)	IC	4.51E+02	3.21E+00	2.72E+04
Phosphate (PO_4^{3-})	IC	4.27E+02	9.07E+00	2.94E+04
Sulfate (SO_4^{2-})	IC	5.17E+03	4.04E+01	5.69E+04
Arsenic (As)	AA	9.99E-02	2.22E-03	2.30E+01
Barium (Ba)	ICP-ES	< 1.56E-01	NA	6.19E+02
Cadmium (Cd)	ICP-ES	< 2.02E+00	NA	3.10E+02
Chromium (Cr)	ICP-ES	5.00E+01	5.37E-01	1.24E+03
Lead (Pb)	ICP-MS	4.07E-01	1.17E-02	6.19E+02
Total Mercury (Hg)	CVAA	7.04E+01	1.34E+01	3.25E+02
Elemental Mercury (Hg(0))	CVAFS	1.47E+00	1.06E-01	1.82E+01
Monomethyl Mercury (CH_3Hg)	CVAFS w/ Distillation	6.62E+01	6.05E+00	3.50E+02
Ethyl Mercury ($\text{C}_2\text{H}_5\text{Hg}$)	CVAFS w/ Distillation	< 1.99E-01	NA	3.73E+02
Selenium (Se)	AA	2.51E-01	1.97E-02	4.46E+02
Silver (Ag)	ICP-ES	< 2.06E+00	NA	6.19E+02
Aluminum (Al)	ICP-ES	4.51E+03	1.23E+01	1.16E+05
Potassium (K)	AA	3.95E+02	1.99E+01	3.03E+04
n-Butanol ($\text{C}_4\text{H}_9\text{OH}$)	VOA	< 5.00E-01 ^c	NA	7.73E+00
i-Butanol ($\text{C}_4\text{H}_9\text{OH}$)	VOA	< 5.00E-01 ^c	NA	7.73E+00
i-Propanol ($\text{C}_3\text{H}_7\text{OH}$)	VOA	< 2.50E-01 ^c	NA	1.88E+00
Phenol ($\text{C}_6\text{H}_5\text{OH}$)	SVOA	< 1.00E+01 ^c	NA	7.50E+02
Isopar L (----)	SVOA	< 2.66E+01 ppm ^{c,d}	NA	1.10E+01 ppm
Total Organic Carbon (----)	TOC	2.47E+02 ^b	1.00E+00	5.00E+03
Tetraphenylborate [TPB] ($\text{B}(\text{C}_6\text{H}_5)_4^-$)	HPLC	< 5.00E+00	NA	5.00E+00

- a. Result is calculated from the measured Al concentration assuming all the Al is present as the OH compound.
b. Measurement performed on filtered supernate samples.
c. Measurement performed on duplicate samples rather than triplicate samples.
d. Result is calculated from the reported concentration of < 33 mg/L and the density of the slurry sample listed in Table 8.

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Table 2. Chemical Contaminants from Fourth Quarter CY16 Tank 50H Samples and Saltstone WAC, Revision 16, Attachment 8.2 Targets²

<u>Chemical Name (Formula)</u>	<u>Method</u>	<u>Average Concentration (mg/L)</u>	<u>Std. Dev.</u>	<u>WAC Target (mg/L)</u>
Boron (B)	ICP-ES	5.22E+01	1.77E+00	7.43E+02
Cobalt (Co)	ICP-MS	< 1.68E-02	NA	1.75E+02
Copper (Cu)	ICP-ES	< 7.36E+00	NA	7.43E+02
Iron (Fe)	ICP-ES	2.01E+01	1.94E+00	4.95E+03
Lithium (Li)	ICP-ES	1.18E+01	1.42E-01	7.43E+02
Manganese (Mn)	ICP-ES	< 2.82E-01	NA	7.43E+02
Molybdenum (Mo)	ICP-ES	1.65E+01	7.11E-02	7.43E+02
Nickel (Ni)	ICP-ES	< 8.13E+00	NA	7.43E+02
Silicon (Si)	ICP-ES	< 2.69E+01	NA	1.07E+04
Strontium (Sr)	ICP-ES	< 1.43E-01	NA	7.43E+02
Zinc (Zn)	ICP-ES	7.58E+00	1.61E-01	8.03E+02
Benzene (C₆H₆)	VOA	< 1.50E-01 ^a	NA	3.10E+02
Methanol (CH₃OH)	VOA	b	NA	1.88E+00
Dibutylphosphate [DBP] (C₈H₁₉O₄P)	IC	< 2.50E+02	NA	3.47E+02
Tributylphosphate [TBP] ((C₄H₉O)₃PO)	SVOA	< 7.50E-01 ^a	NA	7.50E+00
Toluene (C₆H₅CH₃)	VOA	< 1.50E-01 ^a	NA	3.10E+02
EDTA (C₁₀H₁₂N₂O₈⁴⁻)	HPLC	< 1.00E+02	NA	3.10E+02
NORPAR 13 (C_nH_{2·n})	SVOA	< 7.50E-01 ^a	NA	7.50E-01
Dimethyl Mercury ((CH₃)₂Hg)	CVAFS	1.06E-01	7.22E-04	1.00E+00

a. Measurement performed on duplicate samples rather than triplicate samples.

b. Currently, a routine method for detecting this species does not exist in Analytical Development (AD).

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Table 3. Radionuclide Contaminants from Fourth Quarter CY16 Tank 50H Samples and Saltstone WAC, Revision 16, Attachment 8.3 Limits²

<u>Radionuclide</u>	<u>Method</u>	<u>Average Concentration</u> (pCi/mL)	<u>Std. Dev.</u>	<u>WAC Limit</u> (pCi/mL)
Tritium (³H)	Tritium counting	1.42E+03	1.53E+01	5.63E+05
Carbon-14 (¹⁴C)	C-14 Liquid scintillation	6.04E+02	6.99E+01	1.13E+05
Nickel-63 (⁶³Ni)	Ni-59/63	< 3.12E+00	NA	1.13E+05
Strontium-90 (⁹⁰Sr)	Sr-90 Liquid scintillation	9.74E+04	1.34E+04	3.15E+06
Technetium-99 (⁹⁹Tc)	Tc-99 Liquid scintillation	4.35E+04	7.87E+02	2.11E+05
Iodine-129 (¹²⁹I)	I-129 (w/ separation) Liquid scintillation	3.49E+01	2.73E+00	6.30E+01
Cesium-137 (¹³⁷Cs)	Gamma Scan	5.30E+05	2.03E+04	3.96E+06
Uranium-233 (²³³U)	ICP-MS	< 1.63E+02	NA	1.13E+04
Uranium-235 (²³⁵U)	ICP-MS	2.66E-01	4.55E-03	1.13E+02
Plutonium-241 (²⁴¹Pu)	Pu238/241 Liquid scintillation	1.00E+04	4.30E+02	8.38E+05
Total Alpha	Liquid Scintillation Counting	<1.92E+04	NA	2.13E+05

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Table 4. Radionuclide Contaminants from Fourth Quarter CY16 Tank 50H Samples and Saltstone WAC, Revision 16, Attachment 8.4 Targets²

<u>Radionuclide</u>	<u>Method</u>	<u>Average Concentration (pCi/mL)</u>	<u>Std. Dev.</u>	<u>WAC Target (pCi/mL)</u>
Aluminum-26 (²⁶Al)	Gamma scan (Cs removed)	1.25E-01 ^a	4.30E-02	2.88E+03
Cobalt-60 (⁶⁰Co)	Gamma scan (Cs removed)	3.55E-01 ^b	NA	9.75E+02
Potassium-40 (⁴⁰K)	Gamma scan (Cs removed)	3.63E+00 ^a	3.66E-01	1.00E+02
Nickel-59 (⁵⁹Ni)	Ni-59/63	< 2.58E+00	NA	1.13E+03
Selenium-79 (⁷⁹Se)	Se-79	2.24E+01	2.27E+00	1.90E+04
Yttrium-90 (⁹⁰Y)	Secular Equilibrium w/ Sr-90	9.74E+04	1.34E+04	3.15E+06
Zirconium-93 (⁹³Zr)	ICP-MS	< 8.46E+01	NA	1.00E+05
Niobium-94 (⁹⁴Nb)	Nb-94	< 5.99E-02	NA	1.53E+02
Rhodium-106 (¹⁰⁶Rh)	Secular Equilibrium w/ Ru-106	< 2.73E+00	NA	1.13E+06
Ruthenium-106 (¹⁰⁶Ru)	Gamma scan (Cs removed)	< 2.73E+00	NA	1.13E+06
Antimony-125 (¹²⁵Sb)	Gamma scan (Cs removed)	1.18E+01	3.78E-01	7.99E+03
Tellurium-125m (^{125m}Te)	Secular Equilibrium w/ Sb-125	1.18E+01	3.78E-01	1.83E+03
Tin-126 (¹²⁶Sn)	Gamma scan (Cs removed)	4.09E+02	2.37E+01	1.80E+04
Cesium-134 (¹³⁴Cs)	Gamma Scan	< 6.40E+01	NA	1.82E+04
Cesium-135 (¹³⁵Cs)	ICP-MS	< 1.94E+01	NA	2.50E+02
Barium-137m (^{137m}Ba)	Calculation (Secular Equilibrium w/ 94.6% of Cs-137)	5.01E+05	1.92E+04	3.75E+06
Cerium-144 (¹⁴⁴Ce)	Gamma scan (Cs removed)	< 5.63E+00	NA	1.13E+05
Promethium-147 (¹⁴⁷Pm)	Pm-147/Sm-151 Liquid scintillation	< 4.03E+01	NA	5.63E+06
Samarium-151 (¹⁵¹Sm)	Pm-147/Sm-151 Liquid scintillation	< 2.66E+01	NA	2.25E+04
Europium-154 (¹⁵⁴Eu)	Gamma scan (Cs removed)	< 6.40E-01	NA	1.62E+03
Europium-155 (¹⁵⁵Eu)	Gamma scan (Cs removed)	< 2.52E+00	NA	1.13E+04
Radium-226 (²²⁶Ra)	Ra-226	< 1.39E+00	NA	1.00E+03
Radium-228 (²²⁸Ra)	Gamma scan (Cs removed)	< 1.47E+00	NA	1.00E+04
Actinium-227 (²²⁷Ac)	Th-229/230	< 1.10E-02	NA	1.00E+04
Thorium-229 (²²⁹Th)	Th-229/230	3.32E-02 ^b	NA	1.63E+05
Thorium-230 (²³⁰Th)	Th-229/230	2.81E-02 ^a	5.06E-03	6.26E+03
Thorium-232 (²³²Th)	ICP-MS	< 1.85E-03	NA	2.88E+03
Protactinium-231 (²³¹Pa)	Pa-231	< 5.41E-01	NA	1.00E+03
Uranium-232 (²³²U)	U-232	4.17E+00	5.09E+00	9.06E+03
Uranium-234 (²³⁴U)	ICP-MS	< 1.05E+02	NA	1.13E+04
Uranium-236 (²³⁶U)	ICP-MS	1.21E+00	1.08E-02	1.13E+04
Uranium-238 (²³⁸U)	ICP-MS	5.27E+00	4.36E-02	1.13E+04

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Table 4. Radionuclide Contaminants from Fourth Quarter CY16 Tank 50H Samples and Saltstone WAC, Revision 16, Attachment 8.4 Targets², continued

<u>Radionuclide</u>	<u>Method</u>	<u>Average Concentration (pCi/mL)</u>	<u>Std. Dev.</u>	<u>WAC Target (pCi/mL)</u>
Neptunium-237 (²³⁷Np)	ICP-MS	< 1.19E+01	NA	1.00E+04
Plutonium-238 (²³⁸Pu)	Pu238/241 Pu alpha PHA	2.80E+04	1.13E+03	2.13E+05
Plutonium-239 (²³⁹Pu)	Pu238/241 Pu alpha PHA	7.13E+02	9.28E+01	2.13E+05
Plutonium-240 (²⁴⁰Pu)	Pu238/241 Pu alpha PHA	7.13E+02	9.28E+01	2.13E+05
Plutonium-242 (²⁴²Pu)	ICP-MS	< 6.43E+01	NA	2.13E+05
Plutonium-244 (²⁴⁴Pu)	ICP-MS	< 2.99E-01	NA	7.02E+04
Americium-241 (²⁴¹Am)	Am/Cm	6.25E+00	5.37E-01	2.13E+05
Americium-242m (^{242m}Am)	Am/Cm	< 1.36E-01	NA	4.50E+05
Americium-243 (²⁴³Am)	Am/Cm	< 3.41E-01	NA	2.13E+05
Curium-242 (²⁴²Cm)	Am/Cm	< 1.13E-01	NA	1.13E+04
Curium-244 (²⁴⁴Cm)	Am/Cm	2.88E+00	3.98E-01	2.13E+05
Curium-245 (²⁴⁵Cm)	Am/Cm	< 1.61E+00	NA	2.25E+05

- a. Measurement from average of two detectable values from the analyzed triplicate set.
b. Only one detectable value from the analyzed triplicate set.

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Table 5. Chemical Contaminants Impacting Saltstone Disposal Unit (SDU) Flammability from Fourth Quarter CY16 Tank 50H Samples and Saltstone WAC, Revision 16, Table 2 Limits and Targets²

<u>Chemical Name (Formula)</u>	<u>Method</u>	<u>Average Concentration (mg/L)</u>	<u>Std. Dev.</u>	<u>WAC Limit/Target</u>
Isopar L (----)	SVOA	< 2.67E+01 ppm ^{a,b}	NA	1.10E+01 ppm (Limit)
Tetraphenylborate [TPB] (B(C ₆ H ₅) ₄)	HPLC	< 5.00E+00	NA	5.00E+00 mg/L (Limit)
Ammonium (NH ₄ ⁺)	IC	< 1.00E+01	NA	2.12E+02 mg/L (Limit)
Total Mercury (Hg)	CVAA	7.04E+01	1.34E+01	3.25E+02 mg/L (Limit)
Monomethyl Mercury (CH ₃ Hg)	CVAFS w/ Distillation	6.62E+01	6.05E+00	3.25E+02 mg/L (Limit)
Dimethyl Mercury ((CH ₃) ₂ Hg)	CVAFS	1.06E-01	9.18E-02	1.00E+00 mg/L (Target)

a. Measurement performed on duplicate samples rather than triplicate samples.

b. Result is calculated from the reported concentration of < 33 mg/L and the density of the slurry sample listed in Table 8.

Table 6. Other Organics Impacting SDU Flammability from Fourth Quarter CY16 Tank 50H Samples and Saltstone WAC, Revision 16, Table 3 Concentrations²

<u>Chemical Name (Formula)</u>	<u>Method</u>	<u>Average Concentration (mg/L)</u>	<u>Std. Dev.</u>	<u>WAC Concentrations (mg/L)</u>
Butanol (C ₄ H ₉ OH)	VOA	< 5.00E-01	NA	0.75
Tributylphosphate[TBP] ((C ₄ H ₉ O) ₃ PO)	SVOA	< 7.50E-01	NA	1.0
Isopropanol (C ₃ H ₇ OH)	VOA	< 2.50E-01	NA	0.25
Methanol (CH ₃ OH)	a	NA	NA	0.05
NORPAR 13 (C _n H _{2n})	SVOA	< 7.50E-01	NA	0.75

a. Currently, a routine method for detecting this species does not exist in AD.

Table 7. Processing Constituents from Fourth Quarter CY16 Tank 50H Samples and Saltstone WAC, Revision 16, Table 4 Limits²

<u>Processing Constituents</u>	<u>Method</u>	<u>Value</u>	<u>Std. Dev.</u>	<u>WAC Limit</u>
pH	Calculated	> 13	NA	> 10
Sodium Concentration	ICP-ES / AA	5.30 M	2.01E-01	2.5 M < [Na ⁺] < 7.0 M
Total Insoluble Solids	Calculated	~0 wt%	NA	< 15 wt%

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Table 8. Additional Measured Constituents⁴

<u>Constituent</u>	<u>Method</u>	<u>Average Value</u>	<u>Std. Dev.</u>
Density (slurry)	Measured (21.3°C)	1.2322 g/mL	0.0009
Specific Gravity	a	1.2347	0.0009
Total Solids	Measured	28.27 wt%	0.01
Total Beta	LSC	1.02E+06 pCi/mL	1.82E+04
Total Gamma	b	5.02E+05 pCi/mL	1.11E+04 ^c
Thorium-228 (²²⁸Th)	Gamma scan (Cs removed)	< 1.44E+01 pCi/mL	NA
Curium-247 (²⁴⁷Cm) ^d	Am/Cm	< 2.21E+00 pCi/mL	NA
Californium-249 (²⁴⁹Cf) ^d	Am/Cm	< 2.25E+00 pCi/mL	NA
Californium-251 (²⁵¹Cf) ^d	Am/Cm	< 1.92E+00 pCi/mL	NA
Beryllium (Be)	ICP-ES	< 6.51E-02 mg/L	NA

a. Calculated from the measured density of slurry and density of water at 21.1 °C.¹⁰

b. Calculated from the sum of gamma emitters (Sb-126, Sn-126, Sb-125, Am-241, Co-60 and Ba-137m).

c. Value is the “standard error of the mean” rather than the standard deviation of the measurements since its calculation involves multiple radionuclides.

d. Reported values are all below the estimated detection limits of 90.1 pCi/mL established by SRNL.⁷

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