Characterization Plan and Dissolution Tests for Tank 16H Samples

by

P. L. Davis

Westinghouse Savannah River Company
Savannah River Site
Aiken, South Carolina 29808

M. S. Hay

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WESTINGHOUSE SAVANNAH RIVER COMPANY
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Distribution:
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C. A. Langton, 773-43A
V. G. Dickert, 703-H
TIMS (4)

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TO: W. E. STEVENS, 773-43A

FROM: M. S. HAY, 773-A MSH

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Summary

In support of the closure of Tank 16H, a sample of the solids residue on the bottom of the tank interior and three samples from the tank annulus will be sent to SRTC for analysis.[1] The results of the analysis of the samples from the tank interior and the annulus will define the source term inventory used for fate and transport modeling. In addition, the samples from the tank annulus will be used for dissolution tests to evaluate the effectiveness of various cleaning alternatives.

Characterization

A portion of each of the four samples from Tank 16H will be dried and digested by a minimum of two different methods. Each digestion will be done in triplicate. The digested solids samples will be diluted with deionized, distilled water prior to analysis. The diluted digestion sample will be analyzed for the species listed in the Table 1. Provided there is greater than 10 g of solids for each sample, a small portion of the dried solids of each sample will be contacted with a 0.01M NaOH solution (inhibited water) to analyze for the soluble species listed in Table 1. Other analyses will be conducted

as needed to determine the chemical composition of the samples. This work has been requested by a Technical Task Request.[2]

Salt Dissolution Tests

The samples from the tank annulus will be used to study methods for dissolving the material contained in the tank annulus. If the characterization of the annulus samples indicates the samples have essentially the same composition, the samples with similar compositions will be combined to make a composite sample. Samples with distinctly different compositions will be treated individually. Five variables will be controlled in the dissolution tests to simulate expected Tank 16H conditions and capabilities; the dissolution reagent, the volume of dissolution reagent, the temperature, the contact time between the reagent and the solids, and the amount of mixing. Mixing will be minimized by pouring the reagent onto the solids and allowing the mixture to sit undisturbed. Qualitative assessments of the amount of dissolution of the solids as a function of contact time will be made at periodic intervals up to 48 hours. Dissolution tests with each of the reagents will be conducted at ambient temperature and 60 °C. A minimal volume of the dissolution reagent, approximately 3X the volume of the solids, will be used in the tests. The following dissolution reagents will be used in the tests:

- 3 M NaOH at ambient temperature and 60 °C
- Inhibited Water (0.01 M NaOH) at ambient temperature and 60 °C
- Process Water at ambient temperature and 60 °C
- 4 wt % Oxalic Acid at ambient temperature and 60 °C.

The general procedure for the dissolution tests will be to place a weighed amount of the composite sample in a polysulfone (clear plastic) centrifuge tube. A minimal volume of the dissolution reagent (approximately 3X the volume of the solids) will be added to the centrifuge tube with minimal mixing. For tests run at 60 °C, the reagent will be heated to 60 °C and added to the centrifuge tube with no further heating of the tube to simulate plant capabilities. Estimates of the amount of dissolution will be made hourly by visual inspection. The tubes will be gently mixed after 48 hours if the dissolution with no mixing was minimal. The tubes will be centrifuged and the solution decanted from any remaining solids. Centrifuges tubes containing solids after the first contact with the reagent will be contacted with the reagent a second time for 48 hours. At the end of the dissolution tests, any remaining solids in the centrifuge tubes will be sealed in the tubes (after decanting) and stored in case further characterization of these solids is required. Dissolution tests in which all of the solids have dissolved may be terminated prior to 48 hours.

References

1. J. Newman, "Sampling Plan to Support HLW Tank 16", WSRC-TR-97-00350, October 30, 1997.

2. Technical Task Request HLE-TTR-98014.

Table 1. Characterization of Tank 16H Samples

Element	Method	Analysis of Total Dried Solids	Analysis of Inhibited Water Soluble Fraction
Cs^{137}	Gamma counting	√	
Sr^{90}	Liquid Scintillation	√	
Tc^{99}	Liquid Scintillation	√	
Se^{79}	Liquid Scintillation	√	
Pu^{238}	ICP-MS/Alpha PHA	√	
Pu^{239}	ICP-MS	√	
Pu^{241}	ICP-MS/ Gamma	√	
Am^{241}	Gamma counting	√	
Ag	ICP-ES/ICP-MS	√	
As	ICP-ES	√	
Se	ICP-ES	√	
Ca	ICP-ES	√	√
Cd	ICP-ES	√	√
Ce	ICP-ES	√	
Co	ICP-ES	√	√
Cu	ICP-ES	√	√
Mg	ICP-ES	√	√
Mn	ICP-ES	√	√
Pb	ICP-ES	√	√
Zn	ICP-ES	√	√
Zr	ICP-ES	√	√
Al	ICP-ES	√	√
Ba	ICP-ES	√	√

√ indicates the analyses that will be completed for each type of sample

Table 1. (Continued)

Element	Method	Analysis of Total Dried Solids	Analysis of Inhibited Water Soluble Fraction
Fe	ICP-ES	√	√
La	ICP-ES	√	√
Li	ICP-ES	√	√
Mo	ICP-ES	√	√
Na	ICP-ES	√	√
Ni	ICP-ES	√	√
Sn	ICP-ES	√	√
Sr	ICP-ES	√	√
Ti	ICP-ES	√	√
V	ICP-ES	√	√
B	ICP-ES	√	√
Cr	ICP-ES	√	√
P	ICP-ES	√	√
Si	ICP-ES	√	√
Hg	AA CV	√	
flouride	IC scan		√
chloride	IC scan		√
nitrate	IC scan		√
nitrite	IC scan		√
oxalate	IC scan		√
sulfate	IC scan		√
carbonate	titration		√

√ indicates the analyses that will be completed for each type of sample

Approvals

M. S. Hay, WPT, Task Leader — Date 2/9/98

W. E. Stevens, WPT Manager — Date 2/10/98

C. A. Langton, WPT, Design Check — Date 2/9/98