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Kw: Leaching, sludge, calorimetry
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Aluminum Leaching of "Archived" Sludge from Tanks 8F, 11H, and 12H

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Summary

This study investigated the leaching of aluminum compounds from archived sludge samples from Tanks 8F, 11H, and 12H. The conclusions from this study follows.

- This study found boehmite present as the predominant aluminum compound in sludge from Tank 11H and 12H. We did not identify an aluminum compound in Tank 8F sludge. We did not detect any amorphous aluminum hydroxide in the samples. The amount of goethite (FeOOH) measured 4.2 wt% while hematite (Fe_2O_3) measured 3.7 wt% in Tank 11H sludge.
- The recommended recipe for removing gibbsite in sludge proved inefficient for digesting boehmite, removing less than 50% of the compound within 48 hour. The recipe did remove boehmite when the test ran for 10 days (i.e., 7 more days that the recommended (baseline) leaching period).
- Additions of fluoride and phosphate to Tank 12H archived sludge did not improve the aluminum leaching efficiency of the baseline recipe.

Introduction

Aluminum can promote formation or dissolution of networks in hydroxide solid solutions.[1] When present in large amounts it will act as a network former increasing both the viscosity and the surface tension of melts. This translates into poor free flow properties that affect pour rate of glass production in the Defense Waste Processing Facility (DWPF). To mitigate this situation, DWPF operations limit the amount of aluminum contained in sludge.

The baseline sludge processing flowsheet at the Savannah River Site (SRS) previously included: sludge retrieval with inhibited water (0.01 M NaOH/0.01 M $NaNO_2$) and transferring to an ESP (Extended Sludge Process) processing Tank (Tank 40H and 51H), leaching sludge containing high concentrations of aluminum with hot caustic solution (3 M NaOH), adding additional sludge to blend with the treated sludge to lower the aluminum content of sludge, and washing the blended sludge solids with inhibited water to remove salts.[2] The aluminum dissolution step of the ESP converts aluminum oxide and oxide-hydroxide into soluble hydroxides.[3] However, not all the aluminum phases dissolve at the same rate. Boehmite, an oxide hydroxide of aluminum, does not readily dissolve with current baseline leaching procedure. Although the current baseline operation no longer includes caustic leaching of high aluminum sludge, this study provides guidance for the Al leaching behavior of Tank 11H, 12H and 8F archived sludge. The leached and blended sludge solids will contain a large fraction of the transuranic elements and insoluble fission products. The retrieval, leachate, and wash solutions require processing in the Salt Waste Processing Facility (SWPF) with other soluble waste to remove ^{137}Cs as well as ^{90}Sr and soluble actinides. Operations will subsequently combine the waste stream containing ^{137}Cs from the SWPF with the sludge solids in the DWPF process. The combined sludge and Cs-containing stream gets immobilized in a glass matrix for deep geologic disposal. Decontaminated soluble salt solution from the SWPF transfers to Z Area for treatment and disposal as Saltstone. High Level Waste Engineering requested a study of the fate of aluminum in these various process steps.[4] This report describes the experiments to examine the distribution and speciation of aluminum in the various High Level Waste facilities prior to feeding the DWPF and the SWPF.

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[1] J. F. Stebbins, P. F. McMillan and D. B. Dingwell, "Structure, Dynamics and Properties of Silicate Melts," Reviews in Mineralogy, Mineralogical Society of America, Vol. 32, 1994
[2] R. F. Bradley and A. J. Hill Jr., "Chemical Dissolving of Sludge From a High Level Waste Tank at the Savannah River Plant," DP-1471, November 1977.
[3] B. M. Rapko et al., "The Chemistry of Sludge Washing and Caustic Leaching Processes for Selected Hanford Tank Waste," PNNL 11089, March 1996.
[4] J. Pike, "Aluminum Dissolution," HLW-SDT-TTR-99-51.1, October 1999.

Experimental Section

Characterization of Sludge Samples

Before sludge was removed from the satellite area, personnel agitated the sludge with a spatula and then removed sufficient quantities. The sludge was pulverized with a mortar and pestle. Personnel then digested portions of the sludge (about 0.250 grams) with aqua regia in a sealed bomb and heated in a muffle furnace. We submitted the digested liquid (diluted up to 250 mL of water) from the sludge for inductively-coupled plasma emission spectroscopy (ICP-ES) and cold-vapor absorption spectroscopy (CV-Hg, Na and K).

Thermal analysis of the sludge was conducted on an ISI (Instrument Specialist Instruments) DSC 7 (Differential Scanning Calorimeter). The thermal program included heating rates of 10 °C/Min and temperatures up to 600 °C. Sample mass ranged from 30 to 40 mg.

Sludge Washing Conditions

The initial sodium content in the sludge equals about 10.2 wt % for the Tank 8F sludge, 9.37 wt % for Tank 11H sludge, and 1.57 wt % for Tank 12H sludge. Due to interference in the calorimetric analysis and sample homogeneity, the researcher decided to lower the sodium content of the sludge to about 1 wt %. Operations also plan to wash sludge before leaching. Operations will conduct washing with inhibited water. Since washing will be conducted before leaching, we did not investigate the effect of washing on aluminum speciation. Personnel added 10 mL of distilled-deionized water to 3 grams and shook the slurry for 5 minutes. After shaking, the slurry centrifuged for 5 minutes at 2000 rpm. The liquid portion was decanted and the compacted sludge was disturbed (mix) with a spatula. Additional water was then added and the washing was repeated twice more.

Sludge Leaching Conditions

Personnel located samples from “archived” sludge of Tanks 8F, 11H and 12H. The aluminum concentration in “archived” sludge from Tanks 8F, 11H and 12H measured 3.17 ± 0.05, 11.4 ± 4 and 32 ± 0.9 wt %, respectively. The target amount of aluminum in sludge for DWPF equals 8.8 wt % assuming all of the aluminum present is in the boehmite form, or 11.4 wt % assuming aluminum is present as gibbsite. The sludge receives additional dilution by combining with the frit stream, and will receive waste from salt processing much later (projected for 2009), to an Al value of 4.4 wt % in the DWPF stream. This amount avoids phase immiscibility in glass.[5]

[5] C. M. Jantzen, J. B. Pickett, K. G. Brown, T. B. Edwards, and D. C. Beam, “Process Models fro the DWPF Facility: Part I. Predicting Glass Durability from Composition Using a Thermodynamic Hydration Energy Reaction Model (Thermo) U,” WSRC-TR-93-672, Rev. 1, September, 18, 1993

The required amount of sodium hydroxide to completely remove the aluminum in sludge as indicated in the baseline flowsheet equals 3 to 3.5 times the amount of aluminum (in moles) in sludge. Personnel weighted about 3 grams of sludge and placed it in a polypropylene centrifuge tube. To this sludge, personnel added caustic 3 to 3.5 times the amount of aluminum in moles. In the case of archived sludge from Tank 12H, 8 mL of 50 wt % caustic solution (0.02 moles) and 5 mL of inhibited water (i.e., 0.01 M NaOH and 0.001 M $NaNO_2$ in water). The method for determination of the required amount of 50 wt % NaOH solution and inhibited water added follows (Table 1 and 2). For the case of 3 gram of "archived" Tank 12H sludge, processing requires about 0.11 to 0.13 moles of caustic. In addition, a criteria exists that the final solution should have a free [OH]> 3 M. Personnel also prepared a leaching solution of similar composition containing 0.03 and 0.01 M sodium fluoride (NaF) and sodium phosphate (Na_2PO_4). Leaching test temperatures range from 85 to 87 °C.

Table 1. The amount of caustic, inhibited water, NaF and Na_3PO_4 added to the Archived sludge samples.

Tank	50 wt% NaOH (mL)	Inhibited H_2O (mL)	Concentration of NaF and Na_3PO_4 (Molar)
12H	8	5	0
12H*	8	5	0.03 and 0.01
11H	7	6	0
8F	3	10	0

* sample was analyzed for Al (ICP-ES) only. Data is shown in Figure 6.

Table 2. The amount of Al and caustic used for leaching archived sludge from 8F, 11H and 12H.

Tank	Mass of Sludge (g)	Moles of Al	Total Moles of NaOH*	NaOH/Al moles ratio
8F	3.1	0.004	0.054	13.5
11H	3.3	0.012	0.082	6.8
12H	2.9	0.035	0.162	4.6

*about 0.04 moles of NaOH are required for $[OH]_{free} > 3M$

Experimental Results and Discussion

The Calorimetry Spectra of Iron and Aluminum Hydroxide Compounds

A key process in the baseline sludge pretreatment flowsheet involves aluminum leaching. A description of the process chemistry follows.

1. $Al(OH)_3$ (gibbsite) + (3M) NaOH $\rightarrow$ $NaAlO_{2\ (soluble)}$ + $2H_2O$

2. AlO•OH (boehmite) + (3M) NaOH $\rightarrow$ $NaAlO_{2\ (soluble)}$ + H_2O

Both reactions have negative free energies, indicating dissolution will occur. However, the rate of boehmite dissolution is about an order of magnitude slower than gibbsite dissolution at the same conditions.[2] Therefore, sludge with a large fraction of boehmite phase requires more rigorous conditions (e.g., higher OH^- and temperature) and longer leaching times to yield total aluminum removal comparable to sludge loaded with gibbsite. The current baseline sludge leaching procedure assumes a large fraction (~75%) of the aluminum in sludge exists as gibbsite. Therefore, personnel require increased understanding of the conditions influencing the formation of boehmite and gibbsite. Boehmite can form directly from precipitation or through aging of gibbsite.[6] Some of the SRS Tanks containing high-aluminum sludge (e.g., 15H) have dried and reach temperatures over 80 °C for long times. Under these conditions, most of the gibbsite may have transformed into a boehmite phase negatively affecting the baseline-leaching recipe.

Personnel at the Savannah River Technology Center (SRTC) previously tested the efficiency of the leaching process (aluminum leaching) in a large-scale experiment.[7] In that test, personnel removed more than 78 wt % of the aluminum in Tank 15H sludge within one week. The test follows the recommended "baseline" leaching recipe for this operation.[8] Pre-analysis of the sludge indicated most of the aluminum existed in the form of gibbsite.

Recent studies on the solubility of digested sludge in glass revealed the required weight percent of aluminum hydroxide in sludge should range from 8 to 13 (depending if supernatant is added to the glass).[5] If the sludge contains large amounts of boehmite compound the digested sludge will contain unacceptable amounts of aluminum.

This study focused on the leaching characteristics of archived sludge from Tanks 8F, 11H and 12H. The high radioactivity of the "archived" sludge from these

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[6] K. Wefers and C. Misra, "Oxides and Hydroxides of Aluminum," Alcoa Technical Paper # 19, Alcoa Laboratories 1987.
[7] B. A. Hamm, "Demonstration of In-Tank Sludge Processing Part I. Aluminum Dissolution, Sludge Washing and Settling Results," DPST-83-668, July 12, 1983.
[8] G. W. Wilds, "Technical Data Summary; In-Tank Sludge Processing," DPSTD-84-100-TL, April 24, 1984.

Tanks limits the amount of sludge that personnel can analyze outside the Shielded Cells facilities. The maximum amount of sludge that can be withdrawn within the radiation control limits falls well below the minimum amount of samples required for solid characterization and quantification by techniques such as X-ray Diffraction and EDS (Energy Dispersed Spectroscopy). In addition, analyzing such a small quantity of sludge (<1 mg) by SEM (Scanning Electron Microscopy) and TEM (Transmission Electron Microscopy) yields information more representative of sample in-homogeneity rather than of the whole sample population. We needed a technology that evaluated a larger amount of sample and without the need for sample removal from the Shielded Cells.

We decided to evaluate the sludge by thermal analysis techniques. We used a Differential Scanning Calorimeter unit, currently available in the Shielded Cells, for analyzing the sludge. This unit registers the heat given off or taken in by the sample during uniform heating. The amount of heat is proportional to the amount of compound transformed (reversibly or irreversibly) during the heating. In the case of sludge, the DSC can easily detect the hydroxide and oxihydroxide compound decomposition.

Aluminum, iron and manganese are the major SRS sludge components. The major manganese phase in sludge is Mn_2O_3 (a manganese oxide phase).[9] Therefore, we do not expect DSC data to contain information related to manganese compounds. For ease in the interpretation of the DSC data, further analysis of the thermal decomposition of aluminum and iron hydroxide compounds is needed.

The thermal decomposition of gibbsite ($Al(OH)_3$) and boehmite (AlOOH) occurs at different temperatures. Gibbsite thermally decomposes at 250 °C in an open environment and at 310 °C in a closed environment (see Figures 1 and 2).[6] Rapidly heating gibbsite – conditions depend on the gibbsite particle size – transform the particles to boehmite and subsequently to gamma alumina, a defect containing alumina. Slow heating transforms gibbsite into chi alumina, another defect containing alumina compound. The slow heating allows the water molecules inside the gibbsite particles to escape.

Boehmite thermally decomposes at 450 °C in an open environment and at 525 °C in a closed environment (see Fig. 2).[6] Also shown in Figure 2, is the thermal fingerprint of diaspore (an aluminum oxide hydroxide) and amorphous boehmite. Therefore the DSC allows the researcher to determine the presence of amorphous compounds. Both boehmite and diaspore decompose at the same temperature. Amorphous boehmite decomposes at 500 °C.

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[9] Lumetta, G. J.; Burgeson, I. E.; Wagner, M. J.;Liu,J.;Chen, Y. L. Washing and Caustic Leachingof Hanford Tank Sludge: Results of FY 1997 Studies, PNNL-11633 UC-721, August 1997.

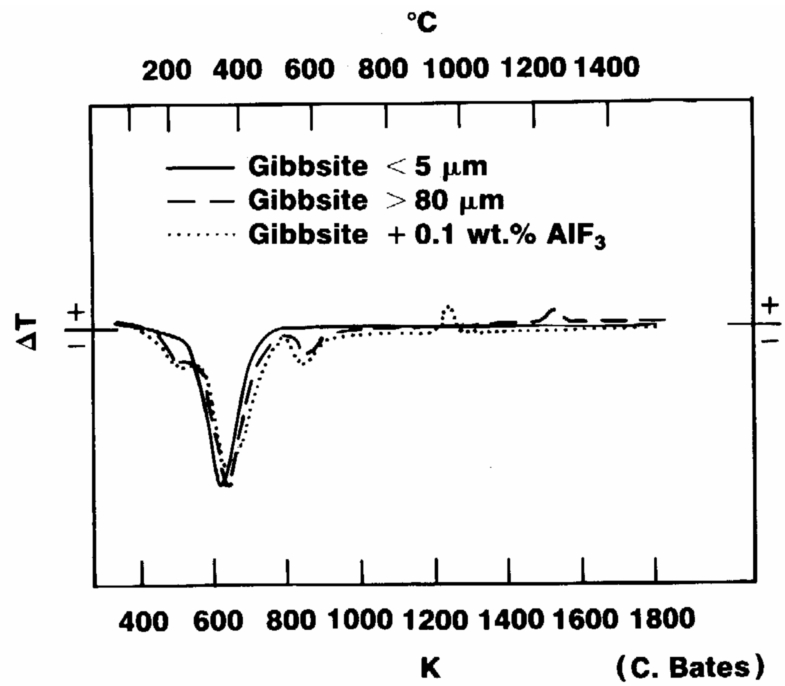

Figure 1. The calorimetry scan of the compound gibbsite.

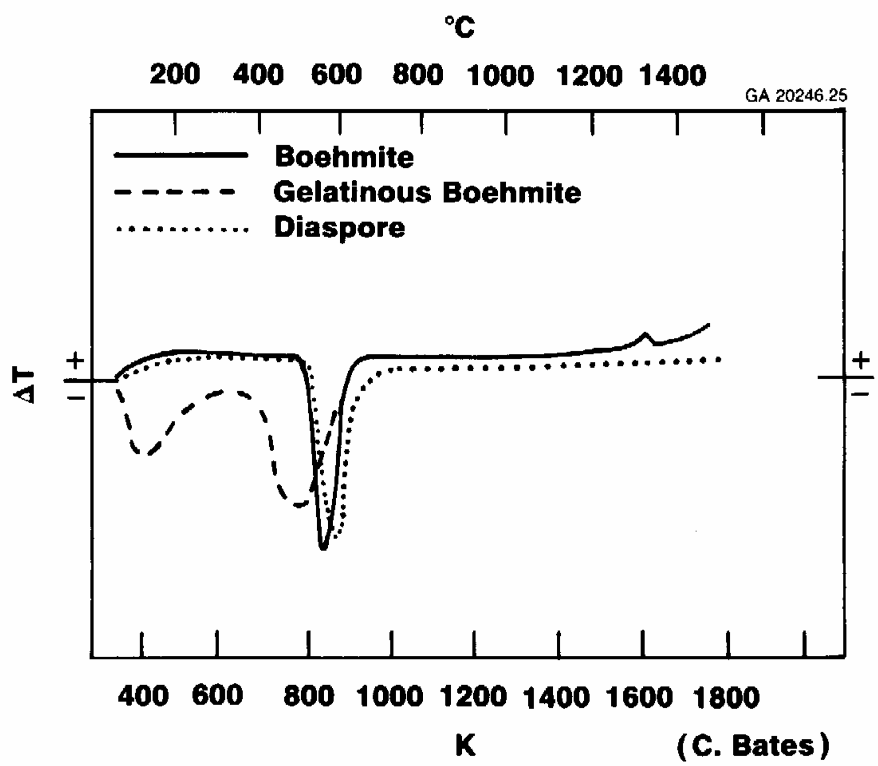

Figure 2. The calorimetric scan of boehmite and its amorphous form. Figures 1 and 2 taken from K. Wefers and C. Misra, "Oxides and Hydroxides of Aluminum," Alcoa Technical Paper # 19, Alcoa Laboratories 1987.

The thermal behavior of iron oxide compounds is presented in Table 3. Inspection of Table 3 reveals goethite decomposes at the same temperature as gibbsite (see Figure 1). Fortunately, SRS sludge is either high in aluminum (HM) or iron (Purex) but not both. Therefore, we should not expect simultaneously high concentrations of goethite and gibbsite in SRS waste (unless operations blend different sludge). The thermal fingerprint of sludge should not include strong decomposition peaks from both goethite and gibbsite. If the sludge contains large amounts of aluminum and iron, then selective aluminum leaching with strong caustic should not affect iron compounds. Therefore, we should expect identification of iron compounds with no interference. Figure 3 shows the thermal spectrum of Hematite, Goethite and Ferrihydrite. Inspection of Figure 3 reveals the decomposition temperature of each compound does not overlap. There is no thermal transition or decomposition of hematite from room temperature to 600 °C.[10]

Table 3. Diagnostic criteria of the iron oxide compounds with the DSC*

Compound	Thermal Event ºC under nitrogen
Hematite (• -Fe_2O_3)	None
Goethite (FeOOH)	280 – 400 (Endotherm)
Lepidocrite (FeOOH)	300-350 (Endotherm), 370-500 (Exotherm)
Ferrihydrite ($Fe(OH)_3$)	150 (Endotherm)
Feroxyhite ($Fe(OH)_3$)	250 (Endotherm)
Maghemite (•-Fe_2 O_3)	600-800 (Exotherm)
Magnetite (Fe_3O_4)	Converts directly to hematite

* “Iron Oxides in the Laboratory: Preparation and Characterization,” U. Schwertmann and R. M. Cornell, VCH publisher, NY, NY 1991.

Figure 4 shows the DSC spectrum of Tank 15H sludge simulant, Tank 15H sludge stimulant with 5, 10 and 20 wt. % goethite and finally, hematite. Looking at Figure 4, the decomposition peak from 280 to 330 °C is due to dehydroxylation of goethite to hematite. The Figure also includes the weight percent of goethite as determined from the area under this peak. The measured quantities agreed with the weighted goethite component except at low goethite concentrations (< 5wt%). The DSC measured above the weighted goethite amount. The data shown in Figures 3 and 4 are consistent with the information shown in Table 2.

[10] U. Schwertmann and R. M. Cornell “Iron Oxides in the Laboratory: Preparation and Characterization,”, VCH publisher, NY, NY 1991.

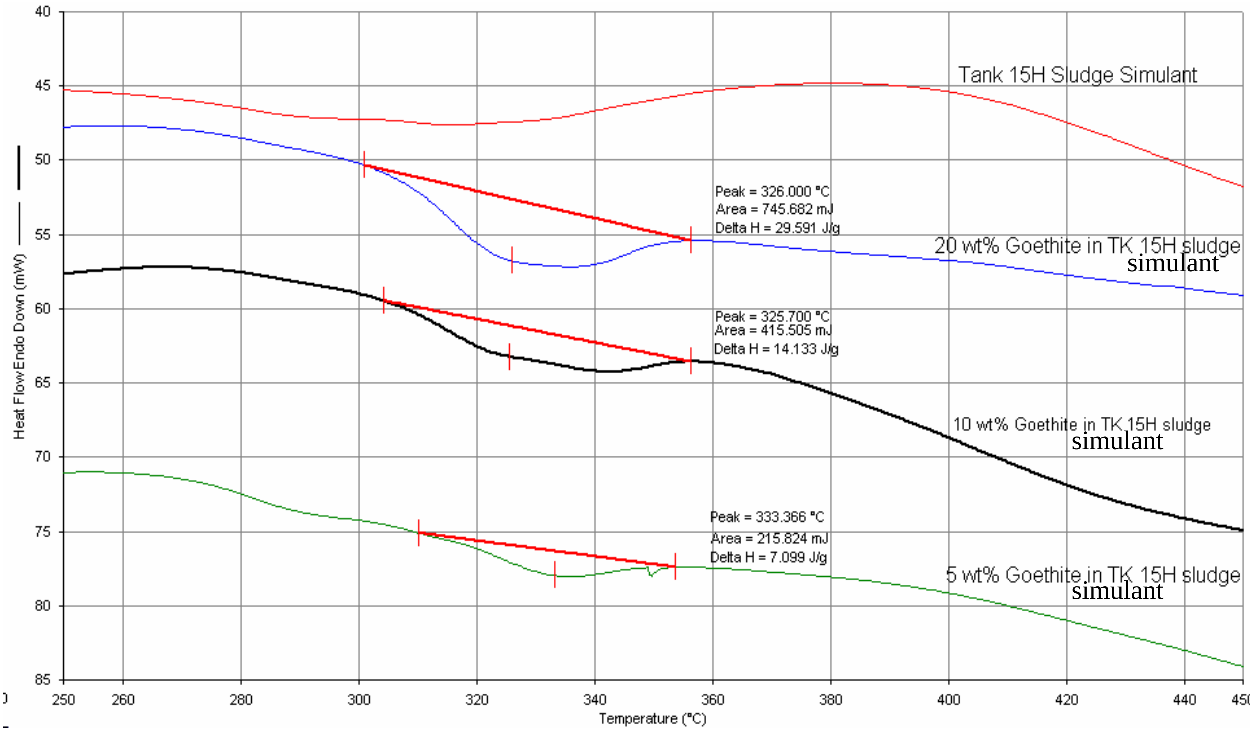

Figure 4. The thermal spectrum of Tank 15H simulant sludge containing 5, 10 and 20 wt% goethite.

The Effect of Washing and Leaching on the Composition of the Sludge

Table 4 shows the composition of the sludge after washing and leaching for 48 hours. In general, washing increased the metal concentration except for sodium and, in the case of Tank 8F sludge, iron decreased. This observation agrees with the expected selective removal of salts from the sludge by the washing process. The aluminum concentration drastically decreased, except for "archived" sludge from Tank 8F, after leaching the sludge with 3.5 M NaOH solution. On the other hand, the iron, manganese and nickel concentrations of the sludge increased during leaching.

Table 4. The composition of a few elements of interest (in wt %) of Tank 8F, 11H and 12H as "received" sludge and after washing and leaching for 48 hours (see appendix 1 for the gravimetric data).

Sludge	Elements	"As Received"	After Washing	After Leaching
Tank 12H	Na	1.28 ± 0.28	1.11 ± 0.1	2.35 ± 0.35
	Al	34.2 ± 4.2	33 ± 3	11 ± 1.3
	Fe	4.3 ± 0.1	4.46 ± 0.03	7.3 ± 1.8
	Mn	2 ± 0.03	2.55 ± 0.01	5.44 ± 0.3
	Ni	1.2 ± 0.13	1.45 ± 0.11	1.54 ± 0.1
	Si	0.2 ± 0.02	0.73 ± 0.3	0.42 ± 0.08
Tank 11H	Na	2.5 ± 0.2	1.05 ± 0.07	1.8 ± 0.2
	Al	24.6 ± 2	24.5 ± 2	9.6 ± 2
	Fe	5.5 ± 0.2	5.7 ± 0.2	6.6 ± 0.3
	Mn	3.6 ± 0.4	3.9 ± 0.7	4.3 ± 0.3
	Ni	1.5 ± 0.2	1.71 ± 0.7	0.8 ± 1.3
	Si	0.3 ± 0.1	0.4 ± 0.1	0.2 ± 0.02
Tank 8F	Na	10.5 ± 1.5	1.2 ± 0.05	2.8 ± 0.2
	Al	$3.9 \pm .1$	4.4 ± 0.4	3.2 ± 0.4
	Fe	28 ± 1	26 ± 1.4	27 ± 1
	Mn	5.2 ± 0.2	6.6 ± 0.6	7.8 ± 2
	Ni	4.8 ± 0.2	5.4 ± 0.4	4 ± 0.15
	Si	$0.5 \pm .03$	0.8 ± 0.05	0.3 ± 0.2

We leached portions of wet sludge (after washing) from Tank 12H as a function of time and monitored the amount of aluminum in solution. Figure 5 displays the aluminum data. Examination of the data reveals an initial rapid increase in the aluminum concentration in solution. After 96 hours of leaching, the aluminum concentration slowly increased to a steady state value. Please note the aluminum leaching rate measured in this experiment can not correlate with the expected leaching rate in the Tank farm. Leaching rate depends on the ratio of liquid volume to solid, particle size, the type of solid compound, the mixing condition, temperature and chemistry of the solution.

The solubility of sodium aluminate (i.e., equilibrium between gibbsite and sodium aluminate) in 3M NaOH solution equals about 12,000 mg/L.[11] The concentration of aluminum seen in this study fell well below the solubility limit and we did not expect to see post-leaching precipitation of gibbsite. The steady-state aluminum concentration observed after leaching for long times could be low due to a decrease in the mass transfer rate (i.e., film or barrier formation) of aluminum from the sludge to the solution. The same observation can also result from a depletion of the aluminum content in the sludge (i.e., reactant depletion).

One may also observe from the data from Figure 5 the amount of time required for the aluminum concentration to reach steady state. More than a week proved necessary for reaching steady state. This amount of time proved similar to that observed in the leaching time of boehmite in 3M NaOH.[12] Under similar conditions, gibbsite completely dissolves in 3M NaOH in approximately 72 hours.[12] We also waited one week to leach samples from Tank 8F and 11H. Inspection of Figure 5 reveals the total amount of aluminum in solution (about 0.18 g in 13 mL of solution) is less than the amount of aluminum leached from Tank 12H archived sludge (from Table 4 the amount is 0.66 g). We attribute this difference to a suspension of aluminum enriched particles resulting from leaching. These particles did not settle during centrifuge. In addition, these particles were filtered out during liquid sampling.

The total amount of aluminum in 12H sludge before washing equals 34.2 wt %. After washing the amount of aluminum remaining in the sludge equaled 33 wt. % (i.e., salt removal concentrated the aluminum). Leaching the sludge reduced the amount of aluminum in sludge to 11 wt %. In this testing, we used 3 grams of wet sludge (87 wt % dried sludge) and it contained about 893 mg of aluminum.

Note from Table 4 that only a small amount of aluminum leached from Tank 8F sludge. This observation suggests the possibility of solid solution (co-precipitation) formation between iron and aluminum. In other words, aluminum substituted iron hydroxide. These co-precipitated aluminum solids prove more resistance to caustic leaching.

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[11] R. V. Lundquist and H. Leitch, "Solubility Characteristics of Sodium Aluminate," U. S. Department of the Interior, Bureau of Mines, Manuscript # 6504, 1964.
[12] E. J. Weber, "Aluminum Hydroxide Dissolution in Synthetic Sludge," DP-1617, March 1982.

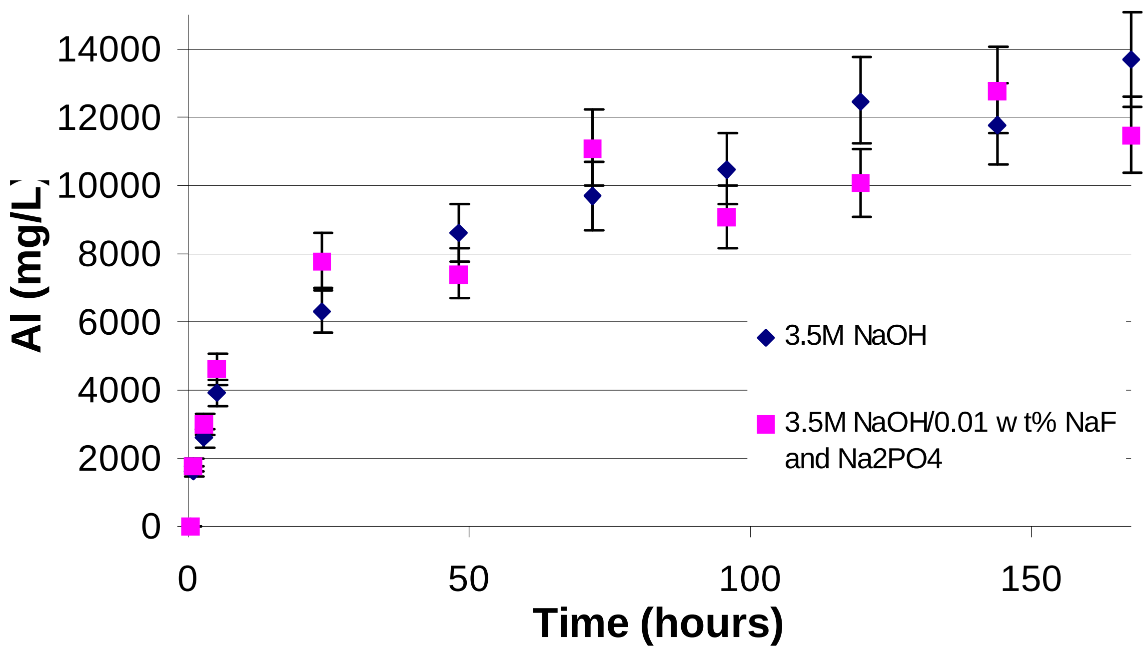

Fig. 5. The aluminum concentration in solution from "archived" Tank 12H sludge is shown as a function of time. Both data sets have 10 % uncertainty error.

Figure 5 also shows the aluminum leaching data from the "archived" sludge of Tank 12H with a "modified" leaching solution. The "modified" leaching solution contained 3.5 M NaOH as well as 0.03M NaF and 9 E-3 M Na_2PO_4. The researcher hoped this solution would improve – either increased the leaching rate or remove larger aluminum amounts – the aluminum leaching of the sludge since the baseline recipe for gibbsite removal is not as effective with boehmite. As seen from Figure 5, the data shows no appreciable difference between the modified and the current baseline-leaching recipe (3.5 M NaOH).

Figure 6 shows the percent of soluble solids from the leaching experiment of Tank 12H "archived" sludge. (For similar data from Tank 11H and 8F refer to Appendix 2.) We calculated the weight fraction of soluble and insoluble solids from the total and dissolved solids using the following formula.

w_{ds} = weight fraction of dissolved solids (wt dissolved solids/ wt of supernate)
w_{ts} = weight fraction of total solids (wt total solids/ wt of sludge slurry)
w_{is} = weight fraction of insoluble solids (wt insoluble solids/ wt of sludge slurry)
w_{ss} = weight fraction of soluble solids (wt dissolved solids/ wt of sludge slurry)

3. $w_{is} = (w_{ts} - w_{ds}) \div (1 - w_{ds})$

4. $w_{ss} = w_{ds} \times (1- w_{ts}) \div (1 - w_{ds})$

Looking at Figure 6, the computed percent soluble solids in the leaching solution increased from 10.3 wt % to 11.2 wt % in 6 days of leaching. Correspondingly, the computed insoluble solids decreased from 19 wt % to 18 wt %. The total solids remained at about ~29 wt % (10.3 + 19 or 11.2 + 18). This result shows conservation of mass during leaching.

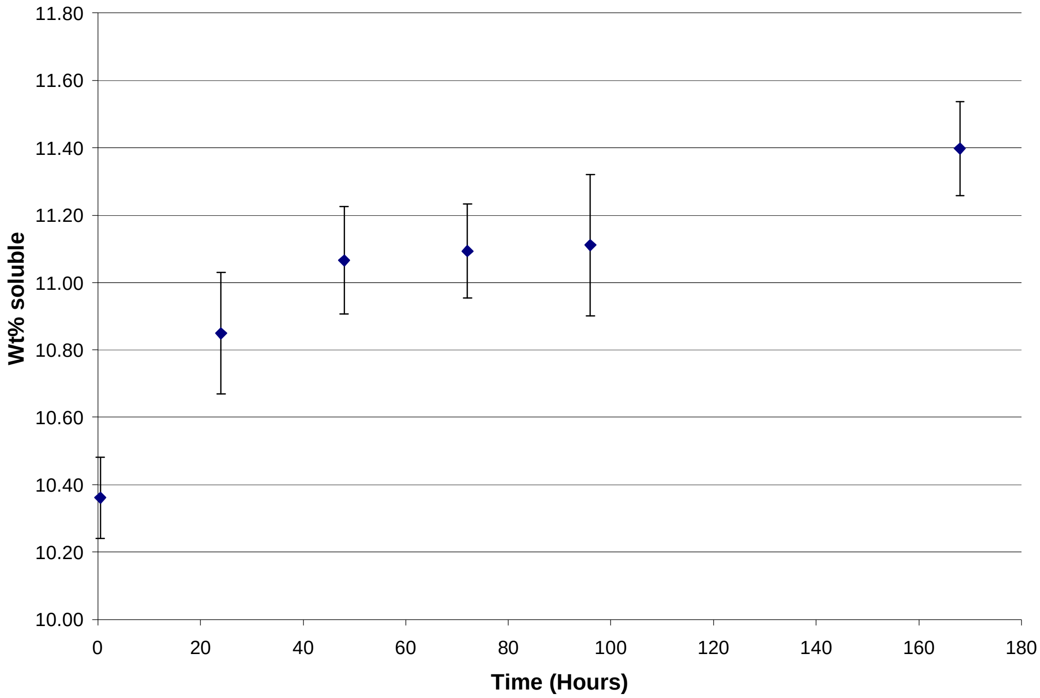

Fig. 6 The percent (by weight) of soluble solids after leaching Tank 12H sludge with 3.5 M NaOH solution for different length of times.

Another question of interest is the molecular form of aluminum in the leaching solution. For example, the aluminum can exist as aluminate ($NaAlO_2$) or hydroxide ($NaAl(OH)_4$) in the leaching solution. The reactions leading to these species follow.

5- $Al(OH)_3$ (in sludge) + NaOH = $NaAlO_2$ + $2H_2O$

6- $Al(OH)_3$ (in sludge) + NaOH = $NaAl(OH)_4$

The first reaction yields aluminate and water. One may expect an increase in percent soluble solids from the first reaction to reach a value of about 14.5 wt %, smaller than the gain one may expects from the second reaction (to reach a value of about 16.2 wt %). However, the error associated with this type of measurement is larger than the difference predicted by these two reactions. The measured result (about 14 wt %) in this study proved insufficient for differentiating which reaction dominated. Molecular spectroscopy is another method for differentiating aluminate from aluminum hydroxide.[13] The covalent bonds between hydrogen, oxygen and aluminum (Al-O-H) in aluminum hydroxide give rise to molecular adsorption due to the bending of Al-O-H bond. Figure 7 shows the infrared spectrum of aluminum in 3.5 M NaOH. Figure 7 clearly show an infrared peak near 950 cm^{-1} due to the bending of the Al-O-H bonds in aluminum hydroxide. On the other hand, aluminate does not have an Al-O-H bond and expect no infrared bands in the 900 to 1000 cm^{-1} region.

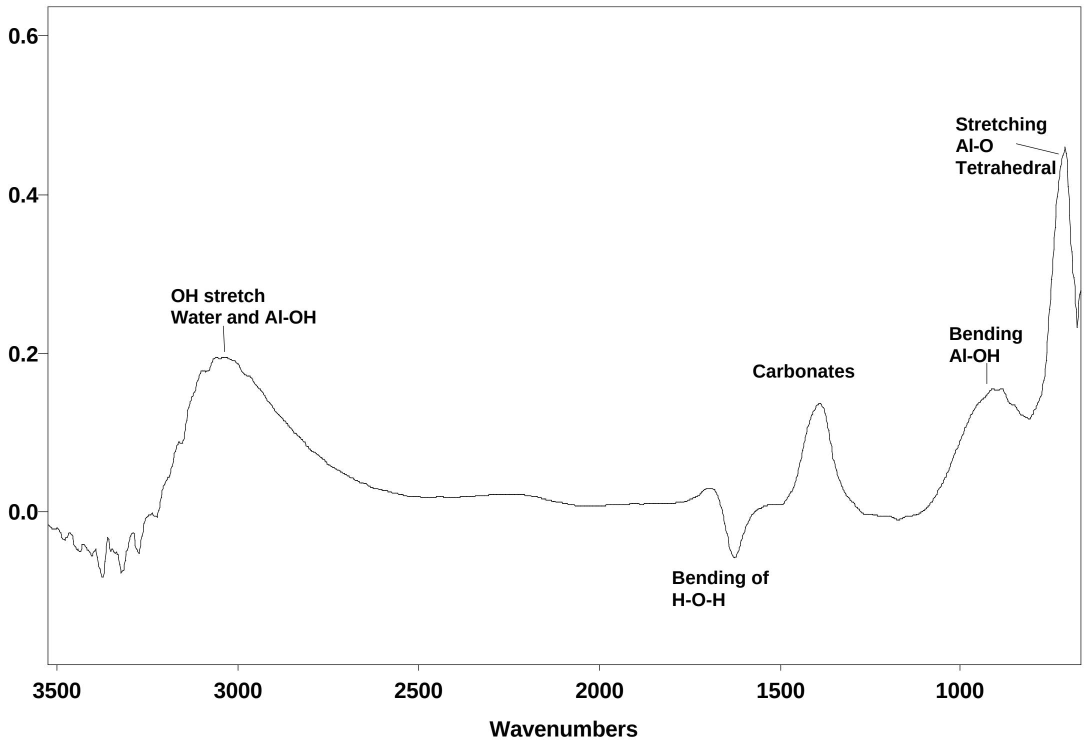

Fig. 7. The infra-red spectrum of aluminum ions in 3.5 M NaOH solution. The spectrum clearly shows Al ions exist as $Al(OH)_4^-$ (as noted by the presence of the peak at 940 cm^{-1}) and not as AlO_2^-. In the case of AlO_2^-, no Al-OH bending will be seen in the infrared.

[13] Johnston et al. Raman Study of Aluminum Speciation in Simulated Alkaline Nuclear Waste. Environ. Sci. Technol. **2002**, 36, 2451-2458

Calorimetry Results of Leached Sludge

Personnel examined various mixtures of boehmite and Purex sludge by calorimetry to determine the linear response of the equipment (DSC) in the Shielded Cells. Figure 8 shows this calibration as the average of two measurements for each point. The thermal response proved linear. The limit of detection (LOD) equaled 3.5 wt % (2 x intercept / slope). Researchers used this calibration curve to determine the amount of boehmite remaining in the sludge.

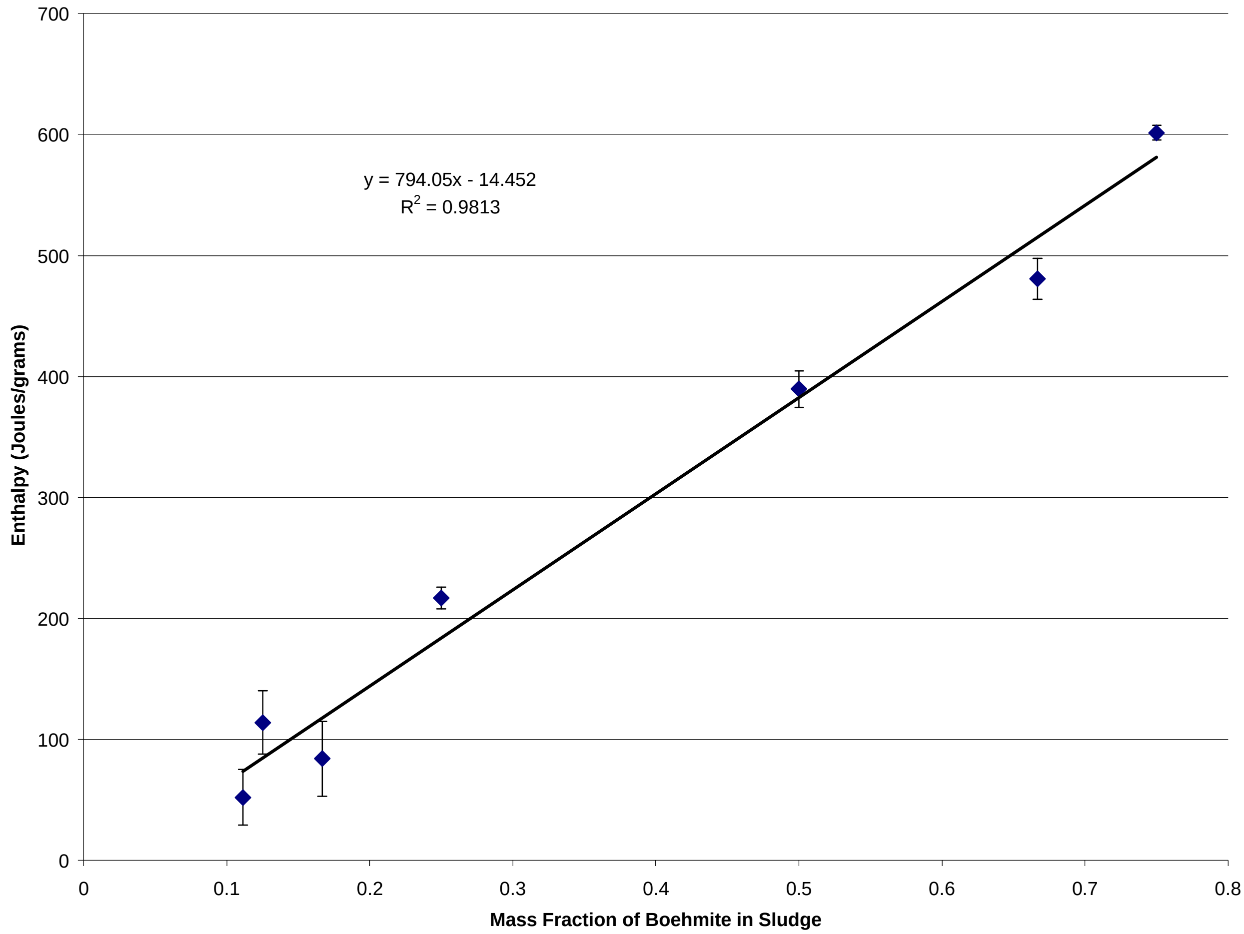

Figure 8. The thermal response of the differential scanning calorimeter located in the shielded cells. Datum comes from the decomposition of mixtures of boehmite in Purex sludge.

The researchers measured the remaining amount of boehmite in sludge after aluminum leaching. Figure 9 provides the calorimetry results from the leached Tank 12H sludge. We did not observe any evidence of amorphous aluminum hydroxide or gibbsite by the calorimeter. The washing procedure removed the gibbsite. Glancing at Figure 9 reveals that some of the aluminum oxide hydroxide compound (boehmite) remained after digesting for 168 hour. We measured the area under the peak for each curve in Figure 9. Calculations included measured area to yield specific heat of decomposition (Joules/gram of material). The ratio of the specific heat of decomposition of a material to pure boehmite yields the wt% boehmite in that material. The final amount estimated by calorimetry equals 9.2 wt % boehmite while the "As Received" amount equaled 20.8 wt % (see Table 4). The 11.6 wt % loss from boehmite would yield 161 mg of the aluminum in solution while chemical analyses detected 224 mg. This implies that the remaining aluminum must come from aluminum residing in iron hydroxide (solid solution). Please recall that about 11 wt % aluminum remained in sludge after leaching. At the start of the leaching test, the researchers assumed the aluminum in Tank 12H sludge existed as gibbsite. Therefore, we used the recipe of 3.5 molar caustic to aluminum ratio and caustic strength of the leachate of 3 molar for leaching. This explains the remaining amount of boehmite in the sludge after leaching for 48 hours. Leaching the sludge for 10 days proved sufficient to nearly remove all of the boehmite (see Fig. 5). This finding indicates that the baseline recipe for gibbsite removal is mass-transfer limited (mixing conditions were not ideal) in removing boehmite.

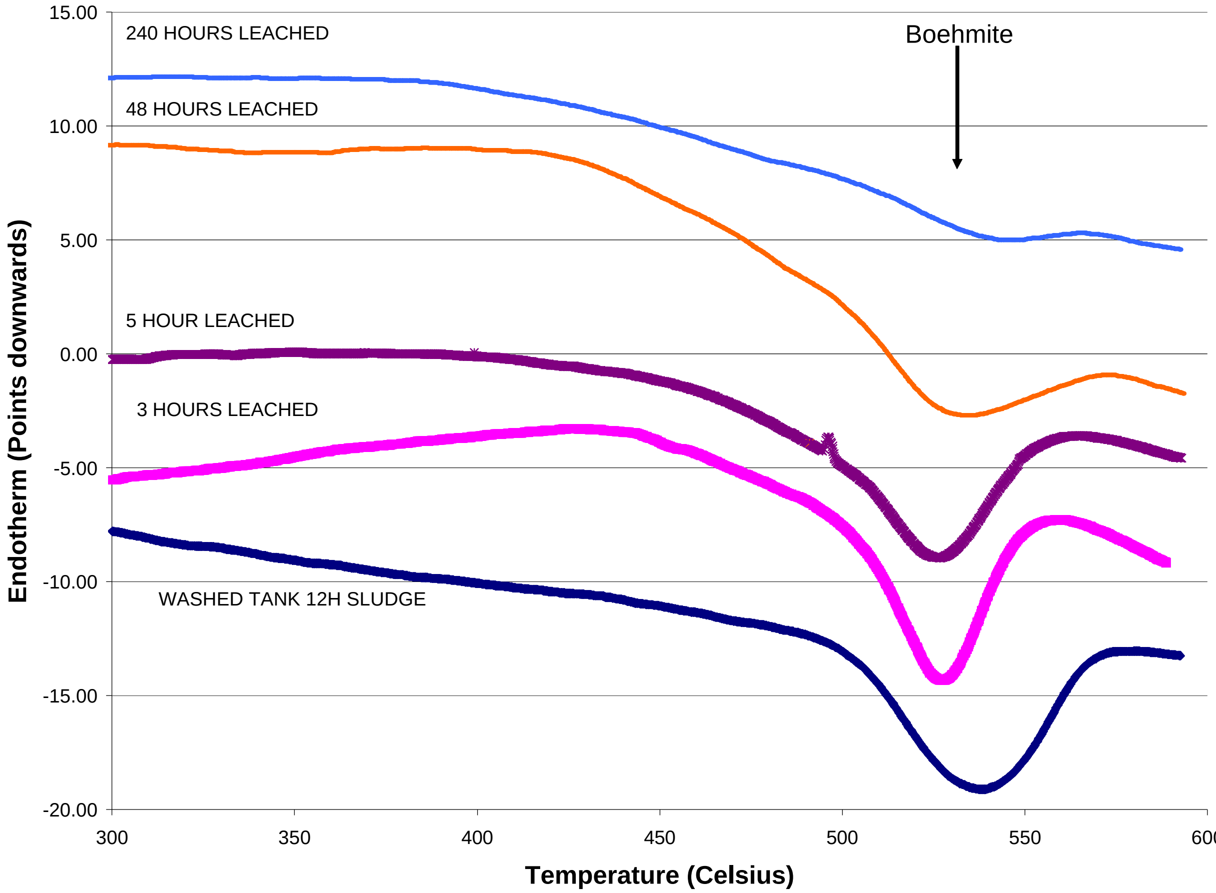

Figure 9. The calorimetry results of leached "Archived" Tank 12H sludge as a function of leaching time.

In the case of Tank 11H sludge, we obtained similar results (see Figure 10). Looking at Figure 10, we identified boehmite as the only crystalline aluminum hydroxide phase present.

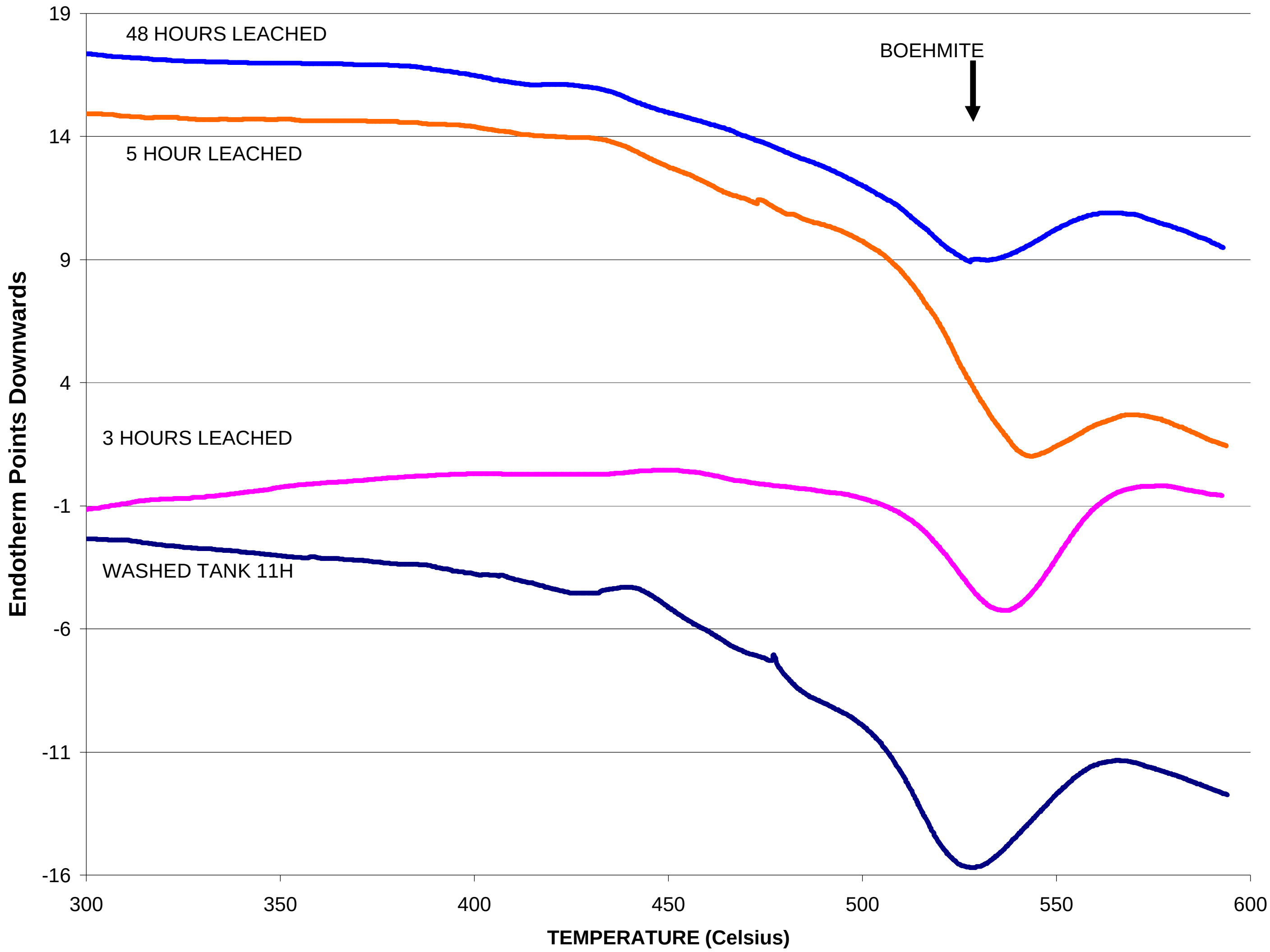

Figure 10. The calorimetry results from leached Tank 11H sludge as a function of leaching time.

Figure 11 shows the calorimetry results from the leaching of Tank 8F sludge. Glancing at Figure 11, one notes the fingerprints from goethite.[14] Goethite (FeOOH) is an iron hydroxide compound. After leaching for 5 hours at 90 °C, goethite converts to hematite (Fe_2O_3).[14] We did not detect any aluminum compound in this sludge by calorimetry. The sludge contains 4 wt% Al but the calorimetry technique did not detected any aluminum compound. We speculate the amount of aluminum is below the limit of detection of the technique or the aluminum resides in a solid solution with iron or manganese compound.

[14] Anand, R. R.; Gilkes, R. J.; "Variations in the Properties of Iron Oxides Within Individual Specimens of Lateritric Duricrust," Aust. J. Soil. Res. 25, PP. 287-302, 1987.

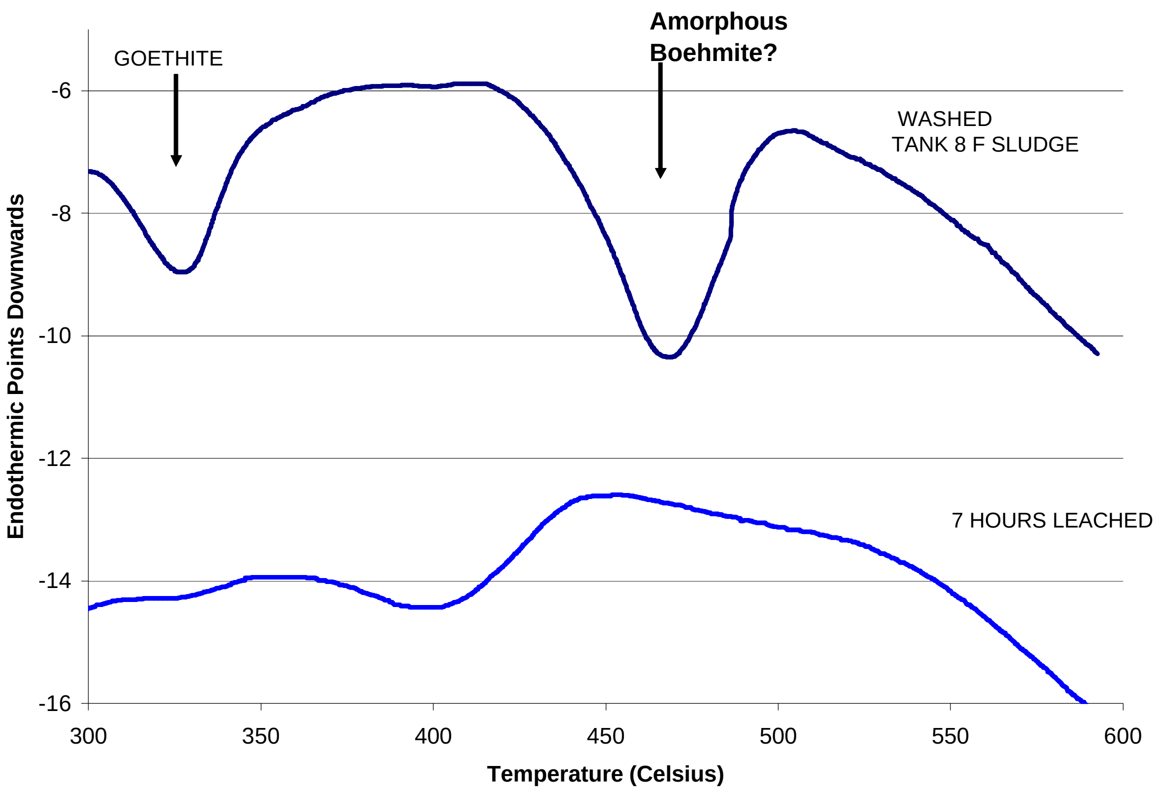

Figure 11. The calorimetry data of leached sludge from Tank 8F as a function of leaching time.

Thermal testing also characterized the iron hydroxide compounds such as goethite and Ferrihydrite compounds. Figure 12 shows the DSC spectrum of pure goethite (for comparison), pure ferrihydrite and "unwashed" Tank 11H archived sludge. Inspection of Figure 12 reveals "unwashed" Tank 11H archived sludge contains ferrihydrite, gibbsite and boehmite. This is revealed by the presence of endothermic peaks (pointing downward) at 185 °C, 310 °C and 550 °C. A careful look at Figure 12 reveals that goethite and gibbsite decompose at the same temperature. We assigned the decomposition peak at 310 °C to gibbsite decomposition since washing with inhibited water completely removed this peak (see absence of endothermic peak near 310 °C in Figure 10). Goethite is insoluble in inhibited water.

To determine the ferrihydrite to hematite ratio we computed the area under the ferrihydrite peak of the Tank 11H sludge spectrum. In dynamic scanning calorimetry, the product of the peak area with the heating rate (in this case was 10 °C per minute) yields the heat absorbed (in Joules) by the material during decomposition. The specific heat of decomposition is the ratio of the heat absorbed to the mass of the sample. The magnitude of the specific heat of decomposition is proportional to the mass of ferrihydrite in the sample. The relation between peak area and wt% composition of a compound follows.

7 Heat absorbed = Peak Area (Watts°C) ÷ Heating rate (°C per second)

8 Specific heat = Heat absorbed (Joules) ÷ mass of samples (g)

9 wt% of compound = specific heat sample ÷ specific heat of pure compound

The specific heat of decomposition of ferrihydrite in Tank 11H measured 70 J/g. The specific decomposition heat of “as made” ferrihydrite measured 873 J/g. The amount of ferrihydrite in Tank 11H sludge is 8 wt% (70÷873). Therefore, the corresponding amount of elemental iron residing in the ferrihydrite form is 4.2 wt%. For comparison, the initial amount of elemental iron in Tank 11H is 5.5 wt%. We assume that 1.3 wt% elemental Iron (the difference between 5.5 and 4.2 wt %) is in the form of hematite. Thus, the amount of hematite is 3.7 wt% in Tank 11H sludge. The ratio of goethite to hematite calculated to 1.1 in Tank 11H.

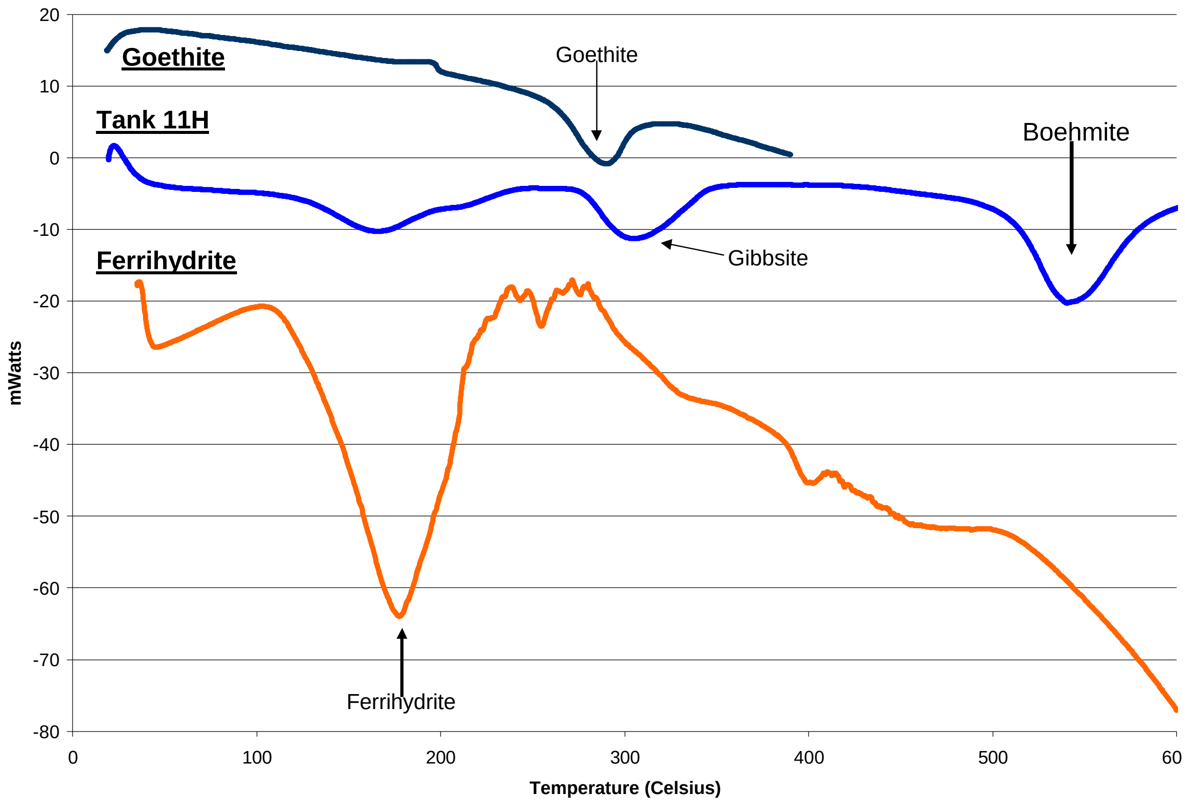

Figure 12. The DSC spectrum of “As received” Tank 11H archived sludge, goethite and Ferrihydrite.

Table 5 shows the effect of leaching on the amount of boehmite (as determined by calorimetry) and the amount of aluminum that we assume exists as solid solution with iron and manganese. The amount of boehmite was determined from the area under the decomposition peak assigned to boehmite as previously described in equations 7-9. The amount of elemental iron residing in solid solution was determined from the difference of the aluminum residing in boehmite (as determined by DSC) and total amount aluminum reported by ICP-ES. This calculation assumes aluminum exists either as an aluminum hydroxide compounds or resides as a “guest” in iron compounds as expressed in equation 10 (we already determined no amorphous Al compounds from the DSC data).

10 Total Al (ICP-AES) = Al in gibbsite (DSC) + boehmite (by DSC) + Al residing in the iron hydroxide compounds

Our estimation indicates lesser aluminum content in the sludge after leaching.

Table 5. The aluminum speciation in sludge in weight % relative to dried sludge before (“As Received”) and after leaching for 48 hours.

Tank	Boehmite (wt %) by Calorimetry		Solid Solution** (wt % elemental aluminum) from the difference of calorimetry and ICP-ES data	
	Before Leaching	After Leaching	Before Leaching***	After leaching
12H	20.8	9.2	15*	2
11H	17.3	8.6	7.2*	1.1
8F	0	0	4.4*	2.3

***We assume the aluminum resides (substitute in for Fe and Mn) with the iron and manganese compounds.**
** **The amount of aluminum in solid solution obtained by the difference between the elemental amount of aluminum in sludge (from ICP-ES) after complete digestion and the amount of aluminum residing in the boehmite from as determined by calorimetry.**
***** From complete aqua regia digestion (HCl-HNO_3)**

Conclusions

This study investigated the aluminum leaching behavior of archived sludge from Tanks 8F, 11H and 12H. The conclusions from this study follows.

- This study found boehmite present as the predominant aluminum compound in sludge from Tank 11H and 12H. We did not identify an aluminum compound in the Tank 8F sludge. We did not detect amorphous aluminum hydroxide in any of the samples. The amount of goethite (FeOOH) measured 4.2 wt% while hematite (Fe_3O_4) measured 3.7 wt% in Tank 11H sludge.
- The recommended recipe for removing gibbsite in sludge proved inefficient for digesting boehmite, removing less than 50% of the compound within 48 hours. The recipe removed nearly all the boehmite when the test ran for 10 days (i.e., 7 more days that the recommended (baseline) leaching period).
- Additions of fluoride and phosphate to Tank 12H archive sludge did not improve the aluminum leaching efficiency of the baseline recipe.

Appendix 1. The mass data of the element in Tank 8F, 11H and 12H archived sludge. The abbreviation “Tank12Lea” stands for leached Tank 12H sludge. The abbreviation “Tank12Was” and “Tank12AR” stands for washed and “As Received” Tank 12H sludge correspondingly.

ICPES Results from B-151 ARL 3580						
Analytical Development Section, SRTC, 5-5523				Calculated:	8/08/2001	
				Template:	Uni_Liq_Templt_Rev_1	
LIMS#, CUSTOMER:	165832 Fondeur			TASK TITLE:	HP_HLW01	
SAMPLE ID:	Tank12AR-1-AQ			Weight (g):	0.24	
ANALYSIS DATE/TIME:	8/08/2001	10:31		VOLUME (mL):	2500	
Data Source:	T56_081_01.dat			* Note: As of 5/12/97, As and Se willno longer be examined via ICP analysis.		
ANALYTE:	RESULTS:					
Ag		376	µg/g			
Al		383900	µg/g			
B		71.4	µg/g			
Ba		1330	µg/g			
Be		< 12	µg/g			
Ca		9397	µg/g			
Cd		169	µg/g			
Co		351	µg/g			
Cr		565	µg/g			
Cu		838	µg/g			
Fe		43000	µg/g			
La		211	µg/g			
Li		2640	µg/g			
Mg		3520	µg/g			
Mn		20076.4	µg/g			
Mo		1177.8	µg/g			
Na		15700	µg/g			
Ni		10730	µg/g			
P		2811	µg/g			
Pb		635	µg/g			
Sb		< 78	µg/g			
Si		2007	µg/g			
Sn		< 18	µg/g			
Sr		517	µg/g			
Ti		404	µg/g			
U		1650	µg/g			
V		177	µg/g			
Zn		1335	µg/g			
Zr		802	µg/g			
Note: Interelement correction applied for Uranium to all elements (except Uranium).						

ICPES Results from B-151 ARL 3580						
Analytical Development Section, SRTC, 5-5523				Calculated:	8/08/2001	
				Template:	Uni_Liq_Templt_Rev_1	
LIMS#, CUSTOMER:	165833 Fondeur			TASK TITLE:	HP_HLW01	
SAMPLE ID:	Tank12AR-2-AQ			Weight (g):	0.24	
ANALYSIS DATE/TIME:	8/08/2001	10:39		VOLUME (mL):	2500	
Data Source:	T56_081_01.dat			* Note: As of 5/12/97, As and Se willno longer be examined via ICP analysis.		
ANALYTE:	RESULTS:					
Ag		176	µg/g			
Al		300100	µg/g			
B		71.4	µg/g			
Ba		710	µg/g			
Be		< 23	µg/g			
Ca		8983	µg/g			
Cd		169	µg/g			
Co		351	µg/g			
Cr		565	µg/g			
Cu		913	µg/g			
Fe		42017	µg/g			
La		10	µg/g			
Li		2823	µg/g			
Mg		2847	µg/g			
Mn		19923.6	µg/g			
Mo		1062.2	µg/g			
Na		16300	µg/g			
Ni		13370	µg/g			
P		1009	µg/g			
Pb		439	µg/g			
Sb		< 164	µg/g			
Si		1890	µg/g			
Sn		< 294	µg/g			
Sr		739	µg/g			
Ti		218	µg/g			
U		17939	µg/g			
V		342	µg/g			
Zn		593	µg/g			
Zr		324	µg/g			
Note: Interelement correction applied for Uranium to all elements (except Uranium).						

ICPES Results from B-151 ARL 3580						
Analytical Development Section, SRTC, 5-5523				Calculated:	8/15/2001	
				Template:	Uni_Liq_Templt_Rev_1	
LIMS#, CUSTOMER:	165865 Fondeur			TASK TITLE:	HP_HLW01	
SAMPLE ID:	Tank12Was-1-AQ			Weight (g):	0.24	
ANALYSIS DATE/TIME:	8/15/2001	11:01		VOLUME (mL):	2500	
Data Source:	T56_081_02.dat			* Note: As of 5/12/97, As and Se willno longer be examined via ICP analysis.		
ANALYTE:	RESULTS:					
Ag		800	µg/g			
Al		359148	µg/g			
B		20	µg/g			
Ba		1460	µg/g			
Be		< 313	µg/g			
Ca		14200	µg/g			
Cd		324	µg/g			
Co		566	µg/g			
Cr		900	µg/g			
Cu		2000	µg/g			
Fe		44300	µg/g			
La		2600	µg/g			
Li		2820	µg/g			
Mg		4000	µg/g			
Mn		25043	µg/g			
Mo		1000	µg/g			
Na		12150	µg/g			
Ni		1340	µg/g			
P		2710	µg/g			
Pb		588	µg/g			
Sb		< 124	µg/g			
Si		4600	µg/g			
Sn		< 134	µg/g			
Sr		655	µg/g			
Ti		598	µg/g			
U		1740	µg/g			
V		220	µg/g			
Zn		1800	µg/g			
Zr		802	µg/g			
Note: Interelement correction applied for Uranium to all elements (except Uranium).						

ICPES Results from B-151 ARL 3580						
Analytical Development Section, SRTC, 5-5523				Calculated:	8/15/2001	
				Template:	Uni_Liq_Templt_Rev_1	
LIMS#, CUSTOMER:	165866 Fondeur			TASK TITLE:	HP_HLW01	
SAMPLE ID:	Tank12Was-2-AQ			Weight (g):	0.24	
ANALYSIS DATE/TIME:	8/15/2001	11:12		VOLUME (mL):	2500	
Data Source:	T56_081_01.dat			* Note: As of 5/12/97, As and Se willno longer be examined via ICP analysis.		
ANALYTE:	RESULTS:					
Ag		200	µg/g			
Al		309000	µg/g			
B		20	µg/g			
Ba		760	µg/g			
Be		< 283	µg/g			
Ca		13800	µg/g			
Cd		324	µg/g			
Co		566	µg/g			
Cr		946	µg/g			
Cu		2756	µg/g			
Fe		44345	µg/g			
La		453	µg/g			
Li		2820	µg/g			
Mg		4000	µg/g			
Mn		2607	µg/g			
Mo		400	µg/g			
Na		10107	µg/g			
Ni		15700	µg/g			
P		1110	µg/g			
Pb		588	µg/g			
Sb		< 172	µg/g			
Si		1004	µg/g			
Sn		< 182	µg/g			
Sr		630	µg/g			
Ti		100	µg/g			
U		174000	µg/g			
V		220	µg/g			
Zn		1200	µg/g			
Zr		802	µg/g			
Note: Interelement correction applied for Uranium to all elements (except Uranium).						

ICPES Results from B-151 ARL 3580						
Analytical Development Section, SRTC, 5-5523				Calculated:	4/02/2002	
				Template:	Uni_Liq_Templt_Rev_1	
LIMS#, CUSTOMER:	175501 Fondeur			TASK TITLE:	HP_HLW01	
SAMPLE ID:	Tank12Lea-1-AQ			Weight (g):	0.24	
ANALYSIS DATE/TIME:	4/02/2002	11:41		VOLUME (mL):	2500	
Data Source:	T62_092_02.dat			* Method Parameters +/- 10%		
ANALYTE:	RESULTS:					
Ag		1078	µg/g			
Al		123300	µg/g			
B		80	µg/g			
Ba		2350	µg/g			
Be		< 232	µg/g			
Ca		16233	µg/g			
Cd		525	µg/g			
Co		638	µg/g			
Cr		1700	µg/g			
Cu		3199	µg/g			
Fe		91018	µg/g			
La		3493	µg/g			
Li		4122	µg/g			
Mg		3824	µg/g			
Mn		57091	µg/g			
Mo		1043	µg/g			
Na		20339	µg/g			
Ni		18492	µg/g			
P		2189	µg/g			
Pb		2019	µg/g			
Sb		< 201	µg/g			
Si		5148	µg/g			
Sn		< 288	µg/g			
Sr		1148	µg/g			
Ti		659	µg/g			
U		163119	µg/g			
V		283	µg/g			
Zn		1329	µg/g			
Zr		402	µg/g			
Note: Interelement correction applied for Uranium to all elements (except Uranium).						

ICPES Results from B-151 ARL 3580						
Analytical Development Section, SRTC, 5-5523				Calculated:	4/02/2002	
				Template:	Uni_Liq_Templt_Rev_1	
LIMS#, CUSTOMER:	175502 Fondeur			TASK TITLE:	HP_HLW01	
SAMPLE ID:	Tank12Lea-2-AQ			Weight (g):	0.24	
ANALYSIS DATE/TIME:	4/02/2002	11:47		VOLUME (mL):	2500	
Data Source:	T62_092_02.dat			* Method Parameters +/- 10%		
ANALYTE:	RESULTS:					
Ag		400	µg/g			
Al		97300	µg/g			
B		80	µg/g			
Ba		1650	µg/g			
Be		< 314	µg/g			
Ca		14700	µg/g			
Cd		426	µg/g			
Co		800	µg/g			
Cr		1700	µg/g			
Cu		2900	µg/g			
Fe		55014	µg/g			
La		1300	µg/g			
Li		3600	µg/g			
Mg		3700	µg/g			
Mn		51841	µg/g			
Mo		500	µg/g			
Na		27307	µg/g			
Ni		15362	µg/g			
P		300	µg/g			
Pb		2000	µg/g			
Sb		< 64	µg/g			
Si		3408	µg/g			
Sn		< 48	µg/g			
Sr		500	µg/g			
Ti		400	µg/g			
U		163500	µg/g			
V		310	µg/g			
Zn		900	µg/g			
Zr		480	µg/g			
Note: Interelement correction applied for Uranium to all elements (except Uranium).						

ICPES Results from B-151 ARL 3580						
Analytical Development Section, SRTC, 5-5523				Calculated:	8/08/2001	
				Template:	Uni_Liq_Templt_Rev_1	
LIMS#, CUSTOMER:	165862 Fondeur			TASK TITLE:	HP_HLW01	
SAMPLE ID:	Tank11AR-1-AQ			Weight (g):	0.24	
ANALYSIS DATE/TIME:	8/08/2001	10:53		VOLUME (mL):	2500	
Data Source:	T56_081_01.dat			* Note: As of 5/12/97, As and Se willno longer be examined via ICP analysis.		
ANALYTE:	RESULTS:					
Ag		1989	µg/g			
Al		267012	µg/g			
B		215	µg/g			
Ba		11350	µg/g			
Be		< 344	µg/g			
Ca		1845	µg/g			
Cd		148	µg/g			
Co		328	µg/g			
Cr		460	µg/g			
Cu		539	µg/g			
Fe		53017	µg/g			
La		256	µg/g			
Li		786	µg/g			
Mg		2325	µg/g			
Mn		32026	µg/g			
Mo		275	µg/g			
Na		23204	µg/g			
Ni		16803	µg/g			
P		1428	µg/g			
Pb		420	µg/g			
Sb		< 142	µg/g			
Si		3928	µg/g			
Sn		< 185	µg/g			
Sr		894	µg/g			
Ti		298	µg/g			
U		3785	µg/g			
V		192	µg/g			
Zn		720	µg/g			
Zr		1093	µg/g			
Note: Interelement correction applied for Uranium to all elements (except Uranium).						

ICPES Results from B-151 ARL 3580						
Analytical Development Section, SRTC, 5-5523				Calculated:	8/08/2001	
				Template:	Uni_Liq_Templt_Rev_1	
LIMS#, CUSTOMER:	165863 Fondeur			TASK TITLE:	HP_HLW01	
SAMPLE ID:	Tan11AR-2-AQ			Weight (g):	0.24	
ANALYSIS DATE/TIME:	8/08/2001	10:49		VOLUME (mL):	2500	
Data Source:	T56_081_01.dat			* Note: As of 5/12/97, As and Se willno longer be examined via ICP analysis.		
ANALYTE:	RESULTS:					
Ag		1300	µg/g			
Al		225001	µg/g			
B		10	µg/g			
Ba		10650	µg/g			
Be		< 189	µg/g			
Ca		1654	µg/g			
Cd		56	µg/g			
Co		128	µg/g			
Cr		449	µg/g			
Cu		694	µg/g			
Fe		57044	µg/g			
La		500	µg/g			
Li		700	µg/g			
Mg		2300	µg/g			
Mn		40105	µg/g			
Mo		70	µg/g			
Na		28303	µg/g			
Ni		12803	µg/g			
P		10	µg/g			
Pb		480	µg/g			
Sb		< 278	µg/g			
Si		2107	µg/g			
Sn		< 494	µg/g			
Sr		553	µg/g			
Ti		107	µg/g			
U		3502	µg/g			
V		125	µg/g			
Zn		524	µg/g			
Zr		606	µg/g			
Note: Interelement correction applied for Uranium to all elements (except Uranium).						

ICPES Results from B-151 ARL 3580						
Analytical Development Section, SRTC, 5-5523				Calculated:	8/15/2001	
				Template:	Uni_Liq_Templt_Rev_1	
LIMS#, CUSTOMER:	165830 Fondeur			TASK TITLE:	HP_HLW01	
SAMPLE ID:	Tank11Was-1-AQ			Weight (g):	0.24	
ANALYSIS DATE/TIME:	8/15/2001	10:12		VOLUME (mL):	2500	
Data Source:	T56_081_01.dat			* Note: As of 5/12/97, As and Se willno longer be examined via ICP analysis.		
ANALYTE:	RESULTS:					
Ag		2700	µg/g			
Al		229014	µg/g			
B		370	µg/g			
Ba		9750	µg/g			
Be		< 375	µg/g			
Ca		2600	µg/g			
Cd		186	µg/g			
Co		90	µg/g			
Cr		360	µg/g			
Cu		900	µg/g			
Fe		54930	µg/g			
La		1100	µg/g			
Li		850	µg/g			
Mg		2800	µg/g			
Mn		38040	µg/g			
Mo		110	µg/g			
Na		10540	µg/g			
Ni		17870	µg/g			
P		1565	µg/g			
Pb		773	µg/g			
Sb		< 426	µg/g			
Si		4539	µg/g			
Sn		< 218	µg/g			
Sr		910	µg/g			
Ti		724	µg/g			
U		6050	µg/g			
V		230	µg/g			
Zn		855	µg/g			
Zr		610	µg/g			
Note: Interelement correction applied for Uranium to all elements (except Uranium).						

ICPES Results from B-151 ARL 3580						
Analytical Development Section, SRTC, 5-5523				Calculated:	8/15/2001	
				Template:	Uni_Liq_Templt_Rev_1	
LIMS#, CUSTOMER:	165831 Fondeur			TASK TITLE:	HP_HLW01	
SAMPLE ID:	Tank11Was-2-AQ			Weight (g):	0.24	
ANALYSIS DATE/TIME:	8/15/2001	10:22		VOLUME (mL):	2500	
Data Source:	T56_081_01.dat			* Note: As of 5/12/97, As and Se willno longer be examined via ICP analysis.		
ANALYTE:	RESULTS:					
Ag		1104	µg/g			
Al		264465	µg/g			
B		232	µg/g			
Ba		9050	µg/g			
Be		< 281	µg/g			
Ca		220	µg/g			
Cd		154	µg/g			
Co		70	µg/g			
Cr		308	µg/g			
Cu		511	µg/g			
Fe		60404	µg/g			
La		350	µg/g			
Li		853	µg/g			
Mg		2800	µg/g			
Mn		39530	µg/g			
Mo		15	µg/g			
Na		11310	µg/g			
Ni		16445	µg/g			
P		120	µg/g			
Pb		673	µg/g			
Sb		< 244	µg/g			
Si		2907	µg/g			
Sn		< 428	µg/g			
Sr		718	µg/g			
Ti		520	µg/g			
U		5150	µg/g			
V		210	µg/g			
Zn		650	µg/g			
Zr		350	µg/g			
Note: Interelement correction applied for Uranium to all elements (except Uranium).						

ICPES Results from B-151 ARL 3580						
Analytical Development Section, SRTC, 5-5523				Calculated:	4/02/2002	
				Template:	Uni_Liq_Templt_Rev_1	
LIMS#, CUSTOMER:	175499 Fondeur			TASK TITLE:	HP_HLW01	
SAMPLE ID:	Tank11Lea-1-AQ			Weight (g):	0.24	
ANALYSIS DATE/TIME:	4/02/2002	0.464		VOLUME (mL):	2500	
Data Source:	T62_092_02.dat			* Method Parameters +/- 10%		
ANALYTE:	RESULTS:					
Ag		1.04	µg/g			
Al		112017	µg/g			
B		133	µg/g			
Ba		8821	µg/g			
Be		< 334	µg/g			
Ca		3350	µg/g			
Cd		213	µg/g			
Co		109	µg/g			
Cr		426	µg/g			
Cu		1050	µg/g			
Fe		69700	µg/g			
La		1211	µg/g			
Li		1164	µg/g			
Mg		3570	µg/g			
Mn		46307	µg/g			
Mo		220	µg/g			
Na		20011	µg/g			
Ni		21502	µg/g			
P		1720	µg/g			
Pb		872	µg/g			
Sb		< 435	µg/g			
Si		2130	µg/g			
Sn		<163	µg/g			
Sr		9605	µg/g			
Ti		880	µg/g			
U		5940	µg/g			
V		420	µg/g			
Zn		1250	µg/g			
Zr		774	µg/g			
Note: Interelement correction applied for Uranium to all elements (except Uranium).						

ICPES Results from B-151 ARL 3580						
Analytical Development Section, SRTC, 5-5523				Calculated:	4/02/2002	
				Template:	Uni_Liq_Templt_Rev_1	
LIMS#, CUSTOMER:	175500 Fondeur			TASK TITLE:	HP_HLW01	
SAMPLE ID:	Tank11Lea-2-AQ			Weight (g):	0.24	
ANALYSIS DATE/TIME:	4/02/2002	11:35		VOLUME (mL):	2500	
Data Source:	T62_092_02.dat			* Method Parameters +/- 10%		
ANALYTE:	RESULTS:					
Ag		714	µg/g			
Al		80105	µg/g			
B		70	µg/g			
Ba		8779	µg/g			
Be		<	µg/g			
Ca		3050	µg/g			
Cd		173	µg/g			
Co		90	µg/g			
Cr		385	µg/g			
Cu		753	µg/g			
Fe		63300	µg/g			
La		458	µg/g			
Li		366	µg/g			
Mg		3030	µg/g			
Mn		39700	µg/g			
Mo		< 20	µg/g			
Na		16021	µg/g			
Ni		12506	µg/g			
P		80	µg/g			
Pb		795	µg/g			
Sb		<	µg/g			
Si		1670	µg/g			
Sn		<	µg/g			
Sr		9183	µg/g			
Ti		582	µg/g			
U		4060	µg/g			
V		380	µg/g			
Zn		456	µg/g			
Zr		371	µg/g			
Note: Interelement correction applied for Uranium to all elements (except Uranium).						

ICPES Results from B-151 ARL 3580						
Analytical Development Section, SRTC, 5-5523				Calculated:	4/02/2002	
				Template:	Uni_Liq_Templt_Rev_1	
LIMS#, CUSTOMER:	175497 Fondeur			TASK TITLE:	HP_HLW01	
SAMPLE ID:	Tank8Lea-1-AQ			Weight (g):	0.24	
ANALYSIS DATE/TIME:	4/2/2002	11:08 AM		VOLUME (mL):	2500	
Data Source:	T62_092_02.dat			*** Method Parameters +/- 10%**		
ANALYTE:	RESULTS:					
Ag		276	µg/g			
Al		38600	µg/g			
B		71.4	µg/g			
Ba		1020	µg/g			
Be		<10.4	µg/g			
Ca		9190	µg/g			
Cd		169	µg/g			
Co		351	µg/g			
Cr		565	µg/g			
Cu		838	µg/g			
Fe		272090	µg/g			
La		1060	µg/g			
Li		2640	µg/g			
Mg		3520	µg/g			
Mn		70490	µg/g			
Mo		57.8	µg/g			
Na		26900	µg/g			
Ni		46600	µg/g			
P		1910	µg/g			
Pb		588	µg/g			
Sb		<688	µg/g			
Si		4690	µg/g			
Sn		<167	µg/g			
Sr		517	µg/g			
Ti		274	µg/g			
U		165080	µg/g			
V		177	µg/g			
Zn		1060	µg/g			
Zr		802	µg/g			
Note: Interelement correction applied for Uranium to all elements (except Uranium).						

ICPES Results from B-151 ARL 3580						
Analytical Development Section, SRTC, 5-5523				Calculated:	4/02/2002	
				Template:	Uni_Liq_Templt_Rev_1	
LIMS#, CUSTOMER:	175498 Fondeur - 10x			TASK TITLE:	HP_HLW01	
SAMPLE ID:	Tank8Lea-2-AQ			Weight (g):	0.242	
ANALYSIS DATE/TIME:	4/2/2002	11:22 AM		VOLUME (mL):	2500	
Data Source:	T62_092_02.dat			*** Method Parameters +/- 10%**		
ANALYTE:	RESULTS:					
Ag		298	µg/g			
Al		41500	µg/g			
B		358	µg/g			
Ba		1090	µg/g			
Be		<10.3	µg/g			
Ca		9880	µg/g			
Cd		192	µg/g			
Co		403	µg/g			
Cr		812	µg/g			
Cu		901	µg/g			
Fe		290510	µg/g			
La		1170	µg/g			
Li		2830	µg/g			
Mg		3810	µg/g			
Mn		64300	µg/g			
Mo		73.7	µg/g			
Na		29200	µg/g			
Ni		50100	µg/g			
P		2050	µg/g			
Pb		794	µg/g			
Sb		<682	µg/g			
Si		4360	µg/g			
Sn		<165	µg/g			
Sr		552	µg/g			
Ti		287	µg/g			
U		177000	µg/g			
V		192	µg/g			
Zn		1140	µg/g			
Zr		904	µg/g			
Note: Interelement correction applied for Uranium to all elements (except Uranium).						

ICPES Results from B-151 ARL 3580					
Analytical Development Section, SRTC, 5-5523				Calculated:	8/08/2001
				Template:	
LIMS#, CUSTOMER:	165828 Fondeur			TASK TITLE:	HP_HLW01
SAMPLE ID:	Tank8AR-1-AQ			Weight (g):	0.24
ANALYSIS DATE/TIME:	8/08/2001	9:56:26		VOLUME (mL):	2500
Data Source:	T56_081_01.dat			* Note: As of 5/12/97, As and Se willno longer be examined via ICP analysis.	
ANALYTE:	RESULTS:				
Ag		0	µg/g		
Al		32182	µg/g		
B		0	µg/g		
Ba		736	µg/g		
Be		<	µg/g		
Ca		15191	µg/g		
Cd		0	µg/g		
Co		0	µg/g		
Cr		952	µg/g		
Cu		860	µg/g		
Fe		209989	µg/g		
La		0	µg/g		
Li		2098	µg/g		
Mg		3498	µg/g		
Mn		59441	µg/g		
Mo		0	µg/g		
Na		122169	µg/g		
Ni		40916	µg/g		
P		3042	µg/g		
Pb		0	µg/g		
Sb		<	µg/g		
Si		3151	µg/g		
Sn		<	µg/g		
Sr		403	µg/g		
Ti		312	µg/g		
U		356	µg/g		
V		0	µg/g		
Zn		2074	µg/g		
Zr		670	µg/g		
Note: Interelement correction applied for Uranium to all elements (except Uranium).					

ICPES Results from B-151 ARL 3580					
Analytical Development Section, SRTC, 5-5523				Calculated:	8/08/2001
				Template:	Uni_Liq_Templt_Rev_1
LIMS#, CUSTOMER:	165827 Fondeur			TASK TITLE:	HP_HLW01
SAMPLE ID:	Tank8AR-2-AQ			Weight (g):	0.24
ANALYSIS DATE/TIME:	8/08/2001	10:49		VOLUME (mL):	2500
Data Source:	T56_081_01.dat			* Note: As of 5/12/97, As and Se willno longer be examined via ICP analysis.	
ANALYTE:	RESULTS:				
Ag		0	µg/g		
Al		31262	µg/g		
B		0	µg/g		
Ba		710	µg/g		
Be		<	µg/g		
Ca		15191	µg/g		
Cd		0	µg/g		
Co		0	µg/g		
Cr		738	µg/g		
Cu		742	µg/g		
Fe		189389	µg/g		
La		0	µg/g		
Li		1975	µg/g		
Mg		3222	µg/g		
Mn		56901	µg/g		
Mo		0	µg/g		
Na		91289	µg/g		
Ni		37836	µg/g		
P		3000	µg/g		
Pb		0	µg/g		
Sb		<	µg/g		
Si		2701	µg/g		
Sn		<	µg/g		
Sr		364	µg/g		
Ti		308	µg/g		
U		107	µg/g		
V		0	µg/g		
Zn		1895	µg/g		
Zr		452	µg/g		
Note: Interelement correction applied for Uranium to all elements (except Uranium).					

ICPES Results from B-151 ARL 3580					
Analytical Development Section, SRTC, 5-5523				Calculated:	8/15/2001
				Template:	Uni_Liq_Templt_Rev_1
LIMS#, CUSTOMER:	165861 Fondeur			TASK TITLE:	HP_HLW01
SAMPLE ID:	Tank8Was-1-AQ			Weight (g):	0.24
ANALYSIS DATE/TIME:	8/15/2001	10:41		VOLUME (mL):	2500
Data Source:	T56_081_01.dat			* Note: As of 5/12/97, As and Se willno longer be examined via ICP analysis.	
ANALYTE:	RESULTS:				
Ag		0	µg/g		
Al		48300	µg/g		
B		0	µg/g		
Ba		763	µg/g		
Be		<	µg/g		
Ca		15191	µg/g		
Cd		0	µg/g		
Co		0	µg/g		
Cr		1055	µg/g		
Cu		891	µg/g		
Fe		253800	µg/g		
La		0	µg/g		
Li		2126	µg/g		
Mg		3440	µg/g		
Mn		70900	µg/g		
Mo		0	µg/g		
Na		12500	µg/g		
Ni		50400	µg/g		
P		3051	µg/g		
Pb		0	µg/g		
Sb		<	µg/g		
Si		8200	µg/g		
Sn		<	µg/g		
Sr		394	µg/g		
Ti		316	µg/g		
U		370	µg/g		
V		0	µg/g		
Zn		2084	µg/g		
Zr		731	µg/g		
Note: Interelement correction applied for Uranium to all elements (except Uranium).					

ICPES Results from B-151 ARL 3580					
Analytical Development Section, SRTC, 5-5523				Calculated:	8/15/2001
				Template:	Uni_Liq_Templt_Rev_1
LIMS#, CUSTOMER:	165829 Fondeur			TASK TITLE:	HP_HLW01
SAMPLE ID:	Tank8Was-2-AQ			Weight (g):	0.24
ANALYSIS DATE/TIME:	8/15/2001	10:03		VOLUME (mL):	2500
Data Source:	T56_081_01.dat			* Note: As of 5/12/97, As and Se willno longer be examined via ICP analysis.	
ANALYTE:	RESULTS:				
Ag		0	µg/g		
Al		39700	µg/g		
B		0	µg/g		
Ba		683	µg/g		
Be		<	µg/g		
Ca		15191	µg/g		
Cd		0	µg/g		
Co		0	µg/g		
Cr		635	µg/g		
Cu		711	µg/g		
Fe		262200	µg/g		
La		0	µg/g		
Li		1946	µg/g		
Mg		3280	µg/g		
Mn		61100	µg/g		
Mo		0	µg/g		
Na		11500	µg/g		
Ni		57600	µg/g		
P		2991	µg/g		
Pb		0	µg/g		
Sb		<	µg/g		
Si		7800	µg/g		
Sn		<	µg/g		
Sr		374	µg/g		
Ti		304	µg/g		
U		190	µg/g		
V		0	µg/g		
Zn		1884	µg/g		
Zr		391	µg/g		
Note: Interelement correction applied for Uranium to all elements (except Uranium).					