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DETERMINATION OF THE FRACTION OF GIBBSITE AND BOEHMITE FORMS OF ALUMINUM IN TANK 51H SLUDGE

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Waste Processing Technology Section
Savannah River National Laboratory
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Prepared for the U.S. Department of Energy Under Contract Number DEAC09-08SR22470

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DETERMINATION OF THE FRACTION OF GIBBSITE AND BOEHMITE FORMS OF ALUMINUM IN TANK 51H SLUDGE

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SRS	Savannah River Site
LWO	Liquid Waste Organization
LTAD	Low Temperature Aluminum Dissolution
DWPF	Defense Waste Processing Facility
SRNL	Savannah River National Laboratory
AD	Analytical Development
ICP-ES	Inductively Couple Plasma-Emission Spectroscopy

SUMMARY

The Savannah River National Laboratory (SRNL) was tasked with developing a test to determine the fraction of the gibbsite and boehmite forms of aluminum in the sludge solids. Knowledge of the fractions of gibbsite and boehmite in the sludge contained in various waste tanks would facilitate better sludge mass reduction estimates and allow better planning/scheduling for sludge batch preparation.

The composite sludge sample prepared for use in the test from several small samples remaining from the original 3-L sample appears to be representative of the original sample based on the characterization data.

A Gibbsite/Boehmite Test was developed that uses 8 M NaOH and a temperature of 65 °C to dissolve aluminum. The soluble aluminum concentration data collected during the test indicates that, for the three standards containing gibbsite, all of the gibbsite dissolved in approximately 2 hours. Under the test conditions boehmite dissolved at more than an order of magnitude more slowly than gibbsite. An estimate based on the soluble aluminum concentration from the sludge sample at two hours into the test indicates the sludge solids contain a form of aluminum that dissolves at a rate similar to the 100% Boehmite standard. Combined with the XRD data from the original 3-L sample[2], these results provide substantial evidence that the boehmite form of aluminum predominates in the sludge. A calculation from the results of the Gibbsite/Boehmite test indicates the sludge contains ~3% gibbsite and ~97% boehmite.

The sludge waste in Tank 51H was recently treated under Low Temperature Aluminum Dissolution (LTAD) conditions and a substantial fraction of aluminum (i.e., sludge mass) was removed, avoiding production of over 100 glass canisters in Defense Waste Processing Facility (DWPF). Results of the Gibbsite/Boehmite test indicate that the aluminum in this sludge was in the form of the more difficult to dissolve boehmite form of aluminum. Since boehmite may be the dominant form of aluminum in Savannah River Site (SRS) waste tank sludge, this result suggests that the conditions of the LTAD process can be used to dissolve both the gibbsite and boehmite forms of aluminum in tank sludge and costly tank infrastructure upgrades required for the higher temperature baseline process can be avoided. However, this conclusion should be confirmed by testing additional waste tank samples.

1.0 INTRODUCTION

The SRS Liquid Waste Organization (LWO) recently used caustic to dissolve aluminum in a Low Temperature Aluminum Dissolution (LTAD) process[1]. The LTAD process reduces the total mass of sludge solids being fed to the DWPF. Of the three predominant forms of aluminum believed to be present in the sludge solids, aluminum trihydrate (gibbsite), aluminum monohydrate (boehmite), and aluminosilicates, only the gibbsite form was expected to dissolve under the conditions of the LTAD process. Historical documents at SRS dealing with sludge processing generally assume that the gibbsite form of aluminum accounts for ~75% of the aluminum in sludge. The recent treatment of sludge waste in Tank 51H under LTAD conditions dissolved a substantial fraction of aluminum avoiding production of over 100 glass canisters in DWPF. These dissolution conditions of the LTAD process were compatible with existing waste tank infrastructure, and tank upgrades to accommodate the higher temperature conditions of the baseline process[2] were avoided.

In Tank 51H (Sludge Batch 5), the LTAD process contacted the sludge with ~4 M NaOH for a period of 46 days with the temperature maintained between 55 °C and 65 °C. Results show that 57-64% of the aluminum in the sludge solids dissolved.[1] A demonstration of the LTAD process in the SRNL with a 3-L sample of the Tank 51H sludge found ~40% of the aluminum dissolved using ~4 M NaOH at 55 °C for 21 days.[3] Both results indicate a much slower dissolution rate than expected if the gibbsite form of aluminum predominates in the sludge. The slow dissolution rate and x-ray diffraction (XRD) data from the characterization of the 3-L sample suggest that boehmite could be the predominant form of aluminum in the sludge solids for this sludge batch.

SRNL was tasked with developing a test to determine the fraction of the gibbsite and boehmite forms of aluminum in the sludge solids. A wet assay method for measuring gibbsite and boehmite was communicated to SRNL from a consultant.[4] However; this method was not viable for radioactive samples at SRNL because of the requirement to heat the sample in a high pressure autoclave. A test requiring only mild temperatures and pressures would be more amenable to use in the Shielded Cells.

Knowledge of the fractions of gibbsite and boehmite in the sludge contained in various waste tanks would facilitate better sludge mass reduction estimates and allow better planning/scheduling for sludge batch preparation. A Technical Task Request[5] and a Task Technical and Quality Assurance Plan[6] were generated for the task.

2.0 DESCRIPTION OF MATERIALS AND TEST PROCEDURE

2.1 SLUDGE SAMPLE DESCRIPTION AND PREPARATION FOR TESTING

Several small samples of sludge remaining from a 3-L sludge sample from Tank 51H (ID No. = HTF-51-07-77) were combined to provide material for the test. The 3-L sludge sample was used in the recent aluminum dissolution demonstration in the Shielded Cells.[3] The composite sludge sample used in the Gibbsite/Boehmite Test had not been subjected to the aluminum dissolution process. The weight percent total solids and density of the composite sludge sample were measured prior to starting the test. Approximately 45 ml of the sludge sample was filtered through a 0.45 µm nylon filter. The sludge solids remaining on the filter were washed four times with an equal volume of 0.01 M NaOH to displace the interstitial supernate. A small portion of the damp sludge solids was immediately used in the Gibbsite/Boehmite Test (see Section 2.3). The remaining washed sludge solids were dried in an oven at 100 °C to determine the moisture content and provide material for sample characterization. The damp sludge solids used in the test were found to contain 44 wt% solids (56 wt% water).

2.2 PREPARATION OF STANDARDS FOR TEST

Four standards were prepared for use in the Gibbsite/Boehmite Test. The standards included a 100% Gibbsite standard, a 75% Gibbsite/25% Boehmite standard, a 25% Gibbsite/75% Boehmite standard and a 100% Boehmite standard. Table 2.1 shows actual weights of Gibbsite and Boehmite used in each standard. Each of the standards contains the approximately the same total mass of material. The 75% Gibbsite standard therefore contains ~75% as much gibbsite as the 100% Gibbsite standard. The gibbsite and boehmite were analyzed previously and found to be essentially pure.[7]

Table 2-1. Gibbsite/Boehmite Standards

Standard	Wt of Gibbsite[a] (g)	Wt of Boehmite[b] (g)
100% Gibbsite	0.900	-
75% Gibbsite/25% Boehmite	0.681	0.221
25% Gibbsite/75% Boehmite	0.220	0.681
100% Boehmite	-	0.900

a Gibbsite, Hydrated Alumina ATH C333, Almatis Inc
b Boehmite, HiQ-B180, BASF Catalysts LLC

2.3 GIBBSITE/BOEHMITE TEST CONDITIONS

Cold bench-top tests were conducted using pure gibbsite, pure boehmite and various mixtures of gibbsite and boehmite to determine conditions that dissolve gibbsite within 1-3 hours at moderate temperatures (<85 °C) with minimal dissolution of boehmite. After numerous trials, a ratio of ~0.3 g of gibbsite (~0.1 g Al) to 16 ml of 8 M NaOH at 65 °C was found to dissolve all of the gibbsite in ~2 hours. At these conditions the rate of boehmite dissolution was quite low.

In order to provide sufficient material for sampling during the test, the quantities established above were tripled to ~0.3 g of Al to 50 ml of 8 M NaOH. Each of the standards shown in Table 2.1 was weighed into a 125 ml poly bottle providing ~0.3 g of Al in each bottle. The damp washed sludge solids were assumed to be approximately 50% water (determined after the test to be 56% water). Based on the previous analysis[3], the washed sludge solids were expected to contain ~25 wt% Al. Therefore, 2.465 g of the damp sludge solids were weighed into a 125 ml poly bottle providing ~0.3 g of Al.

A graduated cylinder was used to add 50 ml of 8 M NaOH to each of the five bottles (4 standards and 1 sludge sample). The bottles were shaken to thoroughly mix the solids and sodium hydroxide solution. A 2 ml aliquot was removed from each bottle and placed in a 10 ml syringe fitted with a 0.45 µ nylon filter cartridge. Each solution was filtered through a syringe filter into a bottle containing 13 ml of 3 M nitric acid. These samples represent the time zero samples for the test. The five bottles were placed in a shaker oven set 65 °C. The start times of each bottle were staggered at five-minute intervals to allow time for sample collection and minimize time outside of the oven. At half hour intervals, each bottle was removed from the oven and a 2 ml aliquot filtered into a bottle containing 13 ml of 3 M nitric acid. Visual observations showed the bottle containing 100% Gibbsite was essentially clear at 1.5 hours. The test was continued for 4 hours collecting samples from each bottle every 30 minutes.

After collecting the final samples at four hours after starting the test, the sludge sample was filtered through a 0.45 µ nylon filter and the solids washed four times with an equal volume of 0.01 M NaOH to displace the interstitial liquid. The washed sludge solids were dried to determine the wt% solids and provide material for characterization.

2.4 PREPARATION OF SAMPLES FOR ANALYSIS

The nitric acid solutions of samples collected during the test were submitted to Analytical Development (AD) for determination of the aluminum concentration by Inductively Coupled Plasma-Emission Spectroscopy (ICP-ES).

The dried sludge solids from the initial sludge solids used in the test and from the solids remaining after the Gibbsite/Boehmite Test were prepared for analysis by dissolution using the sodium peroxide fusion method. The dissolved material was sent to AD for determination of the aluminum concentration by ICP-ES.

3.0 RESULTS AND DISSCUSSION

3.1 CHARACTERIZATION OF THE INITIAL SLUDGE SAMPLE

Table 3-1 shows the results from the measurement of the weight percent solids and density of the initial composite sludge sample. The results from the composite sample used for the Gibbsite/Boehmite test show reasonable agreement with the measurements from the original 3-L sludge slurry sample.[3] The composite sample appears to be slightly more concentrated than the original 3-L sample based on the weight percent solids measurements.

Table 3-1. Weight Percent Solids and Density of the Initial Tank 51H Composite Sludge Slurry Sample and the Original 3-L Sludge Slurry Sample

Analyte (Method)	Units	Composite Sample Average	Original 3-L Sample Average[d]
Weight Percent Total Solids	Wt%	16.1%[a]	15.5%
Weight Percent Dissolved Solids	Wt%	7.16%[b]	6.75%
Weight Percent Soluble Solids	Wt%	6.47%[c]	6.12%
Weight Percent Insoluble Solids	Wt%	9.64%[c]	9.39%
Density of Slurry	g/ml	1.07 [a]	1.08
Density of Supernate	g/ml	1.04 [b]	1.06

a Value shows the result of a single measurement.
b Value represents the average of two or more determinations.
c Values for the weight percent soluble solids and weight percent insoluble solids were calculated from the measured weight percent total solids and weight percent dissolved solids.
d Data taken from Reference 3

After filtration and washing, the remaining solids from the composite sludge sample not used in the Gibbsite/Boehmite test were dried and dissolved for analysis. The damp sludge solids used in the test were found to contain 44 wt% solids (56 wt% water). Table 3-2 shows the result of the analysis of the washed sludge solids from the composite sample. A direct comparison to the composition of the original 3-L sample cannot be made since the solids from the composite sample were washed. However, after normalizing the main components of the sludge solids to iron, a comparison of the composite sample and the original 3-L sample indicates a similar composition for the two samples (see Table 3-3). In contrast, the Al/Fe ratio drops to ~1.7 in the sludge solids generated from the 3-L LTAD demonstration.[3] Although the composite sample was prepared from several small samples remaining from the original 3-L sample; the composite sample appears to be representative of the original sample based on the characterization data.

Table 3-2. Composition of the Sodium Peroxide Fusion Dissolutions of the Initial Washed Sludge Solids from the Composite Sample

Analyte	Units	1st Replicate	2nd Replicate	Average	%RSD	Blank
Ag	mg/kg	<1.4E+02	<1.4E+02	-	-	<1.4E+02
Al	mg/kg	2.36E+05	2.39E+05	2.37E+05	1.0%	<5.9E+02
B	mg/kg	<1.2E+03	<1.2E+03	-	-	<1.2E+03
Ba	mg/kg	6.98E+02	6.93E+02	6.96E+02	0.5%	<1.2E+02
Be	mg/kg	<3.2E+01	<3.2E+01	-	-	<3.2E+01
Ca	mg/kg	2.01E+04	1.42E+04	1.72E+04	24%	5.57E+03
Cd	mg/kg	4.82E+02	4.93E+02	4.87E+02	1.6%	<9.1E+01
Ce	mg/kg	<1.2E+03	<1.2E+03	-	-	<1.2E+03
Cr	mg/kg	1.08E+03	8.51E+02	9.64E+02	17%	4.54E+02
Cu	mg/kg	4.98E+02	4.90E+02	4.94E+02	1.1%	1.06E+02
Fe	mg/kg	1.15E+05	1.16E+05	1.15E+05	0.7%	1.89E+03
Gd	mg/kg	<1.2E+02	<1.1E+02	-	-	<1.1E+02
K	mg/kg	<6.0E+03	<5.9E+03	-	-	<5.9E+03
La	mg/kg	4.74E+02	4.60E+02	4.67E+02	2.1%	<8.9E+01
Li	mg/kg	<1.9E+02	<1.9E+02	-	-	<1.9E+02
Mg	mg/kg	6.71E+03	5.26E+03	5.98E+03	17%	1.30E+03
Mn	mg/kg	2.79E+04	2.85E+04	2.82E+04	1.4%	<2.8E+01
Mo	mg/kg	<3.3E+02	<3.2E+02	-	-	<3.2E+02
Ni	mg/kg	1.11E+04	1.12E+04	1.11E+04	0.8%	<1.1E+03
P	mg/kg	3.43E+03	3.75E+03	3.59E+03	6.2%	3.86E+03
Pb	mg/kg	<5.5E+02	<5.4E+02	-	-	<5.4E+02
S	mg/kg	<1.6E+04	<1.6E+04	-	-	<1.6E+04
Sb	mg/kg	<1.1E+03	<1.1E+03	-	-	<1.1E+03
Si	mg/kg	4.58E+03	4.69E+03	4.64E+03	1.6%	<2.6E+02
Sn	mg/kg	<1.0E+04	<1.0E+04	-	-	<1.0E+04
Sr	mg/kg	<1.5E+03	<1.5E+03	-	-	<1.5E+03
Ti	mg/kg	2.65E+02	2.41E+02	2.53E+02	6.8%	2.28E+02
U	mg/kg	2.72E+04	2.80E+04	2.76E+04	2.0%	<3.6E+03
V	mg/kg	<1.1E+03	<1.1E+03	-	-	<1.1E+03
Zn	mg/kg	4.73E+02	4.65E+02	4.69E+02	1.1%	7.96E+02

Divide mg/kg values by 1E+04 to convert to wt % dried solids basis

Table 3-3. Comparison of the Normalized Composition of Washed Solids from the Composite Sample with the Original 3-L Sample

Analyte	Composite Sample	Original 3-L Sample
Fe	1.00	1.00
Al	2.06	2.14
Mn	0.24	0.24
U	0.24	0.22
Ni	0.10	0.09
Si	0.04	0.06

3.2 RESULTS OF THE GIBBSITE/BOEHMITE TEST

Based on the results of the sample analysis data the sludge solids used in the Gibbsite/Boehmite test contained 0.256 g of Al. This is slightly lower than the 0.3 g of Al targeted for the test but reasonably close given the fact that the dry weight and aluminum concentration in the solids was not known at the start of the test. Table 3-4 shows the total mass of Al and the mass of Al from gibbsite present in the sludge sample and the four standards used in the test. The last two standards shown in the table contain slightly more than 0.3 g of Al but less than 0.3 g of Al from gibbsite. The target proportions for the test of 0.3 g of Al in 50 ml of 8 M NaOH were based on conditions needed to dissolve all of the gibbsite in approximately two hours. The presence of additional aluminum in the standards from boehmite should not impact the rate of gibbsite dissolution. From the cold bench top tests conducted using pure gibbsite, pure boehmite, and various mixtures of gibbsite and boehmite, the rate of boehmite dissolution appears to be approximately linear and independent of the ratio of the mass of aluminum to the volume of 8 M NaOH at 65 °C over the short duration of the tests.

Table 3-4. Mass of Aluminum in the Sludge Solids and Standards

Analyte	Total Mass of Al (g)	Mass of Al (g) from Gibbsite
Washed Sludge Solids	0.256	-
100% Gibbsite Std	0.311	0.311
75% Gibbsite/25% Boehmite Std	0.335	0.235
25% Gibbsite/75% Boehmite Std	0.382	0.076
100% Boehmite Std	0.405	0.000

Table 3-5 shows the analytical results for the aluminum concentration in the solutions of each of the four standards and the sludge sample over the course of the four-hour test. Figure 3.1 shows a graphical representation of the same data. The data indicates that, for the three standards containing gibbsite, all of the gibbsite dissolved in approximately 2 hours at 65 °C in 8 M NaOH. The concentration of Al in solution at 2 hours for those three standards closely matches the expected concentration assuming complete gibbsite dissolution, based on the moles of gibbsite and volume of solution in each bottle. Under the test conditions boehmite dissolved at more than an order of magnitude more slowly than gibbsite. The rate of boehmite dissolution under the test conditions appears to be nearly linear at 1.3E-04 moles/hour. The graph in Figure 3.1 clearly shows the sludge sample contains aluminum that dissolves at a rate similar to the 100% Boehmite standard. Combined with the XRD data on the original 3-L sample[3], these results provide substantial evidence that the boehmite form of aluminum predominates in the sludge.

Table 3-5. Measured Aluminum Concentration (M) in Solution from Samples Obtained during the Gibbsite/Boehmite Test

Time from start of test (hr)	100% Gibbsite	75% Gibbsite 25% Boehmite	25% Gibbsite 75% Boehmite	100% Boehmite	Tank 51H Sludge Solids*
0	0.025	0.018	0.006	0.0001	0.0002
0.5	0.135	0.089	0.032	0.001	0.005
1	0.194	0.139	0.050	0.003	0.010
1.5	0.214	0.159	0.055	0.004	0.010
2	0.222	0.168	0.057	0.006	0.012
2.5	0.222	0.171	0.057	0.007	0.013
3	0.224	0.174	0.058	0.009	0.013
3.5	0.225	0.174	0.058	0.010	0.014
4	0.225	0.175	0.059	0.012	0.014

* The Tank 51H sludge solids were washed with 0.01 M NaOH to displace the interstitial supernate prior to the test.

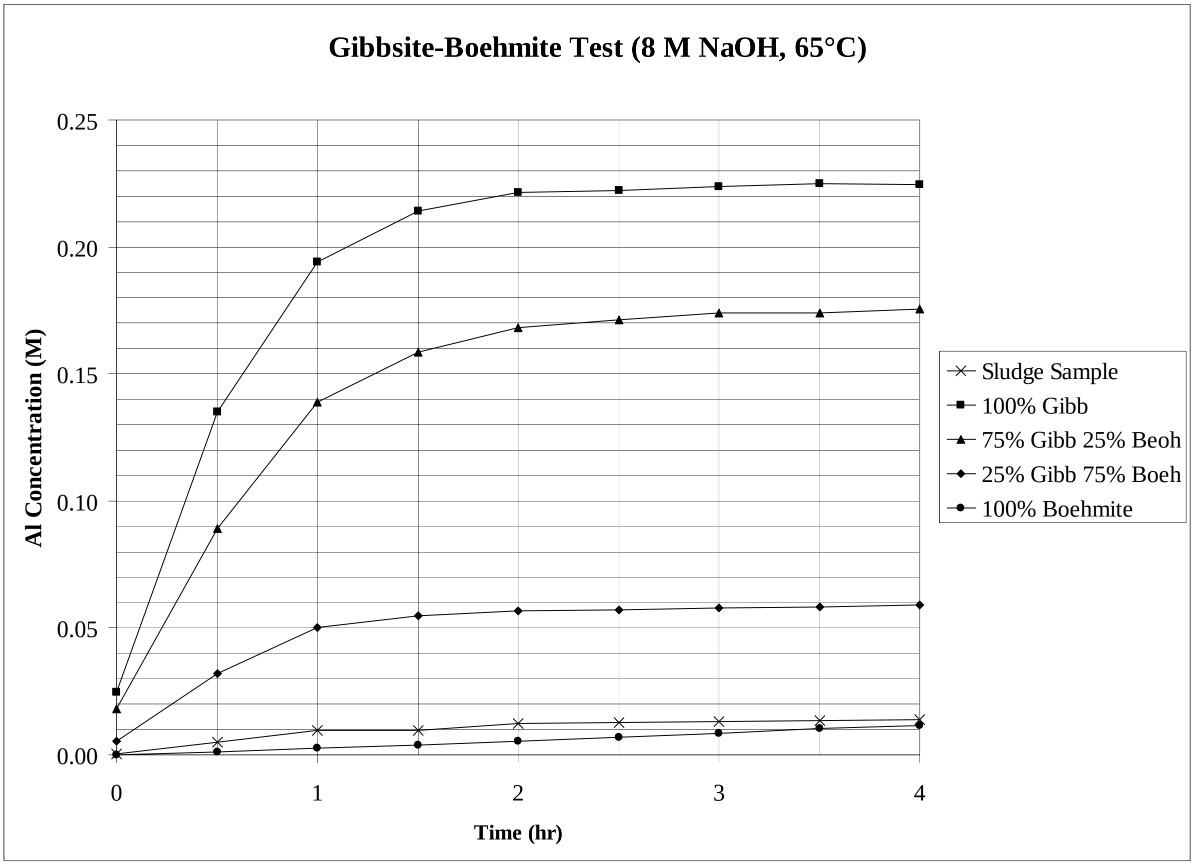

Figure 3.1 Aluminum Concentration as a Function of Time during the Gibbsite/Boehmite Test

The remaining sludge solids from the Gibbsite/Boehmite Test were washed, dissolved and analyzed. Since very little aluminum was dissolved from the sludge based on the samples obtained during the test, the composition remained essentially unchanged from the normalized composition shown in Table 3-3. The Al/Fe ratio dropped slightly from the original sample to 1.99. Table 3-6 shows the normalized composition of the washed sludge solids from the Gibbsite/Boehmite Test.

Table 3-6. The Normalized Composition of Washed Sludge Solids from the Gibbsite/Boehmite Test

Analyte	Final Sludge Solids
Fe	1.00
Al	1.99
Mn	0.26
U	0.24
Ni	0.10

3.3 ESTIMATION OF FRACTIONS OF GIBBSITE AND BOEHMITE IN THE TANK 51H SLUDGE SOLIDS

Since the test data indicates all of the aluminum in the form of gibbsite dissolves in two hours under the test conditions, the fraction of gibbsite in the sludge sample can be estimated from the amount of aluminum in solution measured in the 2-hour sample and the total mass of aluminum in the sludge sample. The total mass of aluminum in the washed sludge solids used in the test can be calculated as follows:

Mass of Al in sludge solids
= 2.465 g damp solids x 0.44 g dry solids/g damp solids x 0.236 g Al/g dry solids
= 0.256 g Al

The aluminum concentration in solution as measured in the 2-hour sample provides the mass of aluminum in solution resulting from gibbsite dissolution. The small amount of boehmite dissolved in the first two hours will be subtracted using the data from the 100% Boehmite standard.

Mass of Al in solution (total Al in solution minus the boehmite that dissolved)
= (0.012 mole/L – 0.006 mol/L) x 0.050 L x 26.98 g/mole
= 0.008 g Al

The estimated fraction of aluminum present as gibbsite in the sludge sample can then be calculated from the mass of aluminum in solution and the total mass of aluminum in the sludge sample.

Estimated fraction of Al present as gibbsite
= 0.008/0.256
= 0.031 (3.0%)

Subtracting the fraction of gibbsite from one yields the estimated fraction of aluminum present as boehmite in the sludge solids (0.97 or 97%).

3.4 CONCLUSIONS

The composite sludge sample prepared from several small samples remaining from the original 3-L sample appears to be representative of the original sample based on the characterization data.

A Gibbsite/Boehmite Test was developed that uses 8 M NaOH and a temperature of 65 °C to dissolve aluminum. The soluble aluminum concentration data collected during the test indicates that, for the three standards containing gibbsite, all of the gibbsite dissolved in approximately 2 hours. Under the test conditions boehmite dissolved at more than an order of magnitude more slowly than gibbsite. An estimate based on the soluble aluminum concentration from the sludge sample at two hours into the test indicates the sludge solids contain a form of aluminum that dissolves at a rate similar to the 100% Boehmite standard. Combined with the XRD data from the original 3-L sample[3], these results provide substantial evidence that the boehmite form of aluminum predominates in the sludge. A calculation from the results of the Gibbsite/Boehmite test indicates the sludge contains ~3% gibbsite and ~97% boehmite.

The sludge waste in Tank 51H was recently treated under LTAD conditions and a substantial fraction of aluminum (i.e., sludge mass) was removed, avoiding production of over 100 glass canisters in DWPF.[1] Results of the Gibbsite/Boehmite test indicate that the aluminum in this sludge was in the form of the more difficult to dissolve boehmite form of aluminum. Since boehmite may be the dominant form of aluminum in SRS waste tank sludge, this result suggests that the conditions of the LTAD process can be used to dissolve both the gibbsite and boehmite forms of aluminum in tank sludge and costly tank infrastructure upgrades required for the higher temperature baseline process[2] can be avoided. However, this conclusion should be confirmed by testing additional waste tank samples.

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