PNNL-18176, Rev. 1
WTP-RPT-184, Rev. 1

U.S. DEPARTMENT OF
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under Contract DE-AC05-76RL01830

Development and Characterization of Boehmite Component Simulant

RL Russell
RA Peterson
HD Smith
DE Rinehart
PM Aker
EC Buck

June 2009

Pacific Northwest
NATIONAL LABORATORY

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Test specification: 24590-PTF-TSP-RT-06-006, Rev 0 and 24590-PTF-TSP-RT-07-004, Rev 0
Test plan: TP-RPP-WTP-469, Rev 0 and TP-RPP-WTP-509, Rev 0
Test exceptions: 24590-WTP-TEF-RT-07-00008, 24590-WTP-TEF-RT-07-00016, 24590-WTP-TEF-RT-08-00013, 24590-WTP-TEF-RT-08-00014, 24590-WTP-TEF-RT-08-00015
R&T focus area: Pretreatment
Test Scoping Statement: None

Pacific Northwest National Laboratory
Richland, Washington 99352

Completeness of Testing

This report describes the results of work and testing specified by Test Specification, 24590-PTF-TSP-RT-06-006, Rev 0 and Test Plan TP-RPP-WTP-469, Rev 0 and TP-RPP-WTP-509, Rev. 0. The work followed the quality assurance requirements outlined in the Test Specification and Test Plan. The descriptions provided in this report are an accurate account of both the conduct of the work and the data collected. Test plan results are reported. Also reported are any unusual or anomalous occurrences that are different from expected results. The test results and this report has been reviewed and verified.

Approved:

6/2/09

Gordon H. Beeman, Manager
Date
WTP R&T Support Project

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Abbreviations and Acronyms

AFA	anti-foaming agent
AV	axial velocity
BET	Brunauer, Emmet, and Teller surface area measurement method
BNI	Bechtel National, Incorporated
DIW	de-ionized water
DOE	U.S. Department of Energy
DST	double-shell tank
FIZ	Fachinformationszentrum
HLW	high-level waste
IC	ion chromatography
ICDD	International Centre for Diffraction Data
ICP-AES	inductively coupled plasma-atomic emission spectroscopy
ICP-MS	inductively coupled plasma-mass spectroscopy
ICSD	Inorganic Crystal Structure Database
LANL	Los Alamos National Laboratory
LAW	low-activity waste
ORNL	Oak Ridge National Laboratory
PEP	Pretreatment Engineering Platform
PNNL	Pacific Northwest National Laboratory
PSD	particle-size distribution
PTF	Pretreatment Facility
QAM	Quality Assurance Manual
QAP	quality assurance program
QARD	Quality Assurance Requirements and Description (document)

RPM	revolutions per minute
RPP	River Protection Project
R&T	research and technology
SARM	Standard Analytical Reference Material
SBMS	Standards-Based Management System
SEM	scanning electron microscopy
SST	single-shell tank
SWRI	Southwest Research Institute
TMP	transmembrane pressure
TRU	transuranic elements
UDS	undissolved solids
WTP	Hanford Tank Waste Treatment and Immobilization Plant
XRD	X-ray diffraction

Testing Summary

According to Bechtel National Inc.'s (BNI's) Test Specification 24590-PTF-TSP-RT-06-006, Rev 0, "Simulant Development to Support the Development and Demonstration of Leaching and Ultrafiltration Pretreatment Processes," simulants for boehmite, gibbsite, and filtration are to be developed that can be used in subsequent bench and integrated testing of the leaching/filtration processes. These simulants will then be used to demonstrate the leaching process and to help refine processing conditions that may impact safety basis considerations (Smith 2006). This report documents the results of the boehmite simulant development and blended simulant crossflow ultrafiltration leaching completed in accordance with the test plan TP-RPP-WTP-469 Rev 0 (a) (WTP Doc. No. 24590- 101-TSA-W000-0004-182-00001 Rev 00A) prepared and approved in response to the cited test specification.

This report also includes the results of the aluminate and anion effect on boehmite dissolution performed in accordance with the test plan TP-RPP-WTP-509, Rev 0(b) (WTP Doc. No. 24590-101-TSA-W000-0004-72-00019 Rev 00A) prepared and approved in response to the Test Specification 24590-WTP-TSP-RT-07-004, Rev 0 (Sundar 2007).

Objective

The test objectives for the work addressed in TP-RPP-WTP-469, Rev 0(a) are summarized in Table S.1 along with a discussion of how the objectives were met. The overall objective of the work described in this report was to develop a boehmite (orthorhombic AlOOH) simulant that appropriately mimics the performance of the actual waste boehmite for use in subsequent testing and to provide a technical performance basis for the use of this simulant.

The test objectives for the work addressed in TP-RPP-WTP-509, Rev 0(b) are summarized in Table S.2 along with a discussion of how the objectives were met. The overall objective of the work from the test plan described in this report was to determine the effect of aluminate and other substantial anions on the solubility of boehmite in caustic solution.

(a) RL Russell, and HD Smith. 2007. "Test Plan for the Development and Demonstration of Leaching and Ultrafiltration Simulants." TP-RPP-WTP-469, Rev. 0, Pacific Northwest National Laboratory, Richland, Washington.

(b) RC Daniel, and RW Shimskey. 2007. "Test Plan for Simulant Testing in Support of Phase I Demonstration of the Ultrafiltration and Leaching Processes in the Integrated Test Facility." TP-RPP-WTP-509, Rev. 0. Pacific Northwest National Laboratory, Richland, Washington.

Table S.1. Test Objectives from TP-RPP-WTP-469, Rev. 0

Test Objective	Objective Met? (Y/N)	Discussion
1) Develop and characterize a gibbsite component simulant for testing of aluminum leaching and provide a basis for the selected simulant.	NA	This objective was addressed in report WTP-RPT-176, Rev. 0.
2) Develop and characterize a boehmite component simulant for testing of aluminum leaching and provide a basis for the selected simulant.	Y	A boehmite was chosen (APYRAL AOH20) based primarily on its similarity in the caustic leaching behavior to the actual waste from Tanks S-110 and S-101. The boehmite simulant leaching results were compared to the actual waste boehmite leaching results and found to match acceptably.
3) Develop and characterize inert ultrafiltration component simulant(s) for testing of the ultrafiltration system and provide a basis for the selected simulant(s), which may include high and low viscosity simulants. Also, methods to adjust the filtration behavior will be tested in an attempt to develop simulants with various compositions to accurately show the variation in filter flux.	NA	This objective was addressed in report WTP-RPT-183, Rev. 0.
4) Provide a blended simulant procedure for use in Phase 1 integrated demonstration unit testing and demonstrate that the behavior of the blended components is equivalent (or correlated) with the behavior of the individual components. The Phase 1 integrated demonstration waste simulant will possess characteristic chemical components and physical properties of a representative waste that would be processed in the WTP as identified from the EFRT issue M4 resolution team.	NA	This objective was addressed in report WTP-RPT-183, Rev. 0.

Table S.1 (Contd)

Test Objective	Objective Met? (Y/N)	Discussion
5) Evaluate available data to identify other potential candidate simulants that may be necessary to be developed during Stage 2 of the simulant development work for full evaluation of the leaching/ultrafiltration performance. This would be due to the identification of an additional physical or chemical property that was found to be fundamental to the process.	N	This activity has not yet been performed.

Table S.2. Test Objectives from TP-RPP-WTP-509, Rev. 0

Test Objective	Objective Met? (Y/N)	Discussion
1) Determine the effect of initial aluminate ion concentration on the rate of boehmite leaching in caustic solutions and in the presence of soluble anions in a waste. The anions to be considered are those that are typically present in the Hanford Tank Farm wastes in significant amounts. This includes carbonate, free-hydroxide, nitrate, nitrite, oxalate, phosphate, and sulfate.	Y	Boehmite was leached in the presence of aluminate and other principal anions present in the Hanford tank waste. These were found to slow the dissolution of boehmite with the aluminate having a greater effect than the other principal anions. These results are discussed in Section 5.0.
2) Determine the sensitivity of the rate of dissolution of boehmite to soluble anions through a limited number of laboratory tests. The anions to be considered are those that are typically present in the Hanford Tank Farm wastes in significant amounts. This includes carbonate, free hydroxide, nitrate, nitrite, oxalate, phosphate, and sulfate.	NA	As of March 20, 2008, PNNL has been released from this objective by Test Exception, 24590-WTP-TEF-RT-07-00016.
3) Determine the effect of scaling the length of the ultrafilter element from 2ft to 8ft on the filtrate flux over the expected operating range of the ultrafilter using the Cell Unit Filter (CUF).	NA	This objective was addressed in report WTP-RPT-168, Rev. 0.

Table S.2 (Contd)

Test Objective	Objective Met? (Y/N)	Discussion
4) Use an 8-ft-long filter element in the CUF unit to determine the effect of temperature on the filtration of a waste simulant over the range of temperature conditions for the leaching processes.	NA	This objective was addressed in report WTP-RPT-168, Rev. 0.
5) Use a 2-ft-long filter element in the CUF unit to evaluate the effect of the fine particle fraction in the ultrafiltration simulant on fouling of the filter element over the range of concentrations of operating solids. The fine particle fraction is defined as those particles with diameters smaller than the 10th percentile (i.e., the dp10) of the particle-size number distribution.	NA	This objective was addressed in report WTP-RPT-183, Rev. 0.
6) Perform various simulant aging tests to understand the changes that may occur to the simulant in storage and to ensure the adequacy of the simulant for use in the Pretreatment Engineering Platform (PEP).	NA	These results will be presented in a letter report that is yet to be released.
7) Perform Cr-simulant leaching tests to establish that the Cr-simulant from the larger batch exhibits similar or better leaching behavior than the initial trial batch during caustic and oxidative leaching operations.	NA	These results will be presented in a report that is yet to be released.
8) Perform leaching tests to determine the mass loss, and aluminum and chromium dissolution rates during caustic leaching under varying temperature processing conditions without aeration in both UFP-1A/B and UFP-2A/B vessels as well as to measure the effect of aeration on chromium leaching in UFP-2A/B.	NA	These results will be presented in a report that is yet to be released.
9) Perform leaching tests to develop an accurate model for the dissolution of boehmite.	NA	These results will be presented in a report that is yet to be released.

Table S.2 (Contd)

Test Objective	Objective Met? (Y/N)	Discussion
10) Perform leaching tests to verify the effect of aluminate ions on the performance of the boehmite component B3 during caustic leach at temperatures lower than 100°C and to determine the effect of temperature on the dissolution rate of boehmite component B7.	NA	These results will be presented in a report that is yet to be released.
11) Perform leaching tests to determine the extent of boehmite conversion one would expect under leaching conditions during the planned testing in PEP.	NA	These results will be presented in a report that is yet to be released.

Success Criteria

This work meets the third of the Success Criteria described in TP-RPP-WTP-469, Rev 0 (Russell 2007). How this success criterion was met is listed in Table S.3. This work also meets the first two success criteria described in TP-RPP-WTP-509, Rev. 0 (Daniel 2007) which is listed in Table S.4.

Table S.3. Results and Performance against Success Criteria of TP-RPP-WTP-469, Rev. 0

List Success Criteria	Explain How the Tests Did or Did Not Meet the Success Criteria
1. Development of proceduralized recipes that document each simulant (boehmite, gibbsite, and ultrafiltration) per "Guideline for R&T Simulant Development, Approval, Validation, and Documentation."	A boehmite simulant was chosen based on 1) crystallography for crystal size, 2) XRD to identify boehmite specie, and 3) comparison to actual waste boehmite dissolution rate for leaching behavior. This information is presented in Section 2.0. APYRAL AOH20 from Nabaltec was chosen, based primarily on its similarity in dissolution rate for leaching behavior to the actual waste from Tanks S-110 and S-101. The crystallography and XRD identified that it was pure boehmite with uniform crystal size. The other simulants are addressed in reports WTP-RPT-176, Rev. 0 (gibbsite), WTP-RPT-183, Rev. 0 (filtration and blended simulants), and WTP-RPT-164, Rev. 0 (chromium).
2. Development of a gibbsite simulant that has physical properties—in particular crystal size and habit—similar to that observed in prior actual waste samples and the development of a correlation that predicts gibbsite simulant dissolution rate as a function of gibbsite properties such as crystal size	This criterion is addressed in report WTP-RPT-176, Rev. 0.

Table S.3 (Contd)

List Success Criteria	Explain How the Tests Did or Did Not Meet the Success Criteria
and habit, as well as other physical properties.	
3. Development of a boehmite simulant that has a dissolution rate—in particular at 100ºC—similar to that observed in prior actual waste samples and the development of a correlation that predicts simulant boehmite dissolution rate as a function of system properties of crystal size, crystal habit, operating temperature, hydroxide concentration, mixing conditions.	Several boehmite sources were characterized by particle size distribution (PSD), X-ray diffraction (XRD), scanning electron microscopy (SEM), surface-area analysis, and caustic leaching kinetics. These results were then compared against available data from caustic leaching of boehmite in actual waste. It was found that B3 and B5 matched the closest to the actual waste, but due to the commercial availability of B3 and its more uniform particle size, it was chosen over B5. More details of this are given in Sections 3.0 and 8.0. A boehmite dissolution rate model is being developed for hydroxide and temperature. This model will be reported in a later report. Insufficient data exist to address crystal size, habit, or mixing conditions on the boehmite rate model.
4. Provide a blended simulant procedure for use in Phase 1 integrated demonstration unit testing and demonstrate that the behavior of the blended components is equivalent (or correlated) with the behavior of the individual components. The Phase 1 integrated demonstration waste simulant will possess characteristic chemical components and physical properties of a representative waste that would be processed in the WTP as identified from the EFRT issue M4 resolution team.	This criterion is addressed in report WTP-RPT-183, Rev. 0.
5. Evaluate available data to identify other potential candidate simulants that may be necessary to be developed during Stage 2 of the simulant development work for full evaluation of the leaching/ultrafiltration performance. This would be due to the identification of an additional physical or chemical property that was found to be fundamental to the process.	This criterion is not addressed in this report.

Table S.4. Results and Performance against Success Criteria of TP-RPP-WTP-509, Rev. 0

List Success Criteria	Explain How the Tests Did or Did Not Meet the Success Criteria
1. Development of empirical information that allows determination of the effect of initial aluminate ion concentration on the kinetics of boehmite leaching in a waste simulant.	It was determined that initial aluminate ion concentration slows the kinetics of the boehmite dissolution in the waste simulant. More details are discussed in Section 6.0.
2. Determination of the sensitivity of boehmite leaching to carbonate, free-hydroxide, nitrate, nitrite, oxalate, phosphate, and sulfate anions in a waste-simulant solution.	As of March 20, 2008, PNNL has been released from this objective by Test Exception, 24590-WTP-TEF-RT-07-00016.
3. Determination of the effect of scaling the length of the ultrafilter element from 2 ft to 8 ft on the performance of the filter over the expected process operating range in transmembrane pressure, axial velocity, and ultrafiltration temperature.	This criterion is addressed in report WTP-RPT-168, Rev. 0.
4. Determination of the effect of temperature on the filtration flux for the waste simulant over the range of solid concentrations and temperature conditions for the leaching processes.	This criterion is addressed in report WTP-RPT-168, Rev. 0.
5. Determination of the effect of fine-particle concentration on the propensity of the waste simulant to foul the ultrafilter element over the range of concentrations of operating solids in the waste simulant.	This criterion is addressed in report WTP-RPT-183, Rev. 0.

Test Exceptions

Test exception number 24590-WTP-TEF-RT-07-00008 was received from Bechtel National, Inc. (BNI) on November 12, 2007. The test plan (TP-RPP-WTP-469, Rev 0) only allowed for a single test to validate the performance of a blended simulant composition for use in the Pretreatment Engineering Platform (PEP) for process demonstration. This test was carried out at a filtration temperature of 45°C, as was the oxidative leaching operation. However, the filtration temperature in the PEP during process demonstration remained undecided between 45°C and 25°C. Filtration at 45°C could potentially lead to post-precipitation of phosphates and other soluble salts if the solution is cooled to 25°C before being transferred to the permeate collection vessels UFP-62A/B/C and the subsequent ion-exchange feed vessel CXP-01. Therefore, a second test to validate the blended simulant composition for the PEP process demonstration was performed at 25°C with the results of these tests discussed in report WTP-RPT-183.

Four test exceptions were issued for Test Plan TP-RPP-WTP-509. These test exceptions are summarized in Table S.5 along with a brief description of how each exception impacted existing objectives and test plan scope.

Table S.5. Test Exceptions to Test Plan TP-RPP-WTP-509

Test Exception Number	Description of Test Exception
24590-WTP-TEF-RT-07-00016, Rev. 0	This test exception released PNNL from test objective 2 (see Table S.2).
24590-WTP-TEF-RT-08-00013, Rev. 0	This test exception did not affect any existing test plan objectives. It added test objectives concerned with 1) aging of the PEP simulants during storage and 2) leaching of the chromium simulant. These are objectives 6 and 7 in Table S.2, and the results will be released at a later date.
24590-WTP-TEF-RT-08-00014, Rev. 0	This test exception both affected existing test objectives and added new test objectives. Tests associated with objective 4 were modified slightly in response to this test exception with the temperatures examined being changed slightly. The results of this testing is reported in WTP-RPT-168. In addition, a new objective concerned with the influence of temperature and aeration on caustic leaching processes was added. This is objective 8 in Table S.2, and the results will be released at a later date.
24590-WTP-TEF-RT-08-00015, Rev. 0	This test exception did not affect any existing objectives in the test plan. It added three new test objectives concerned with 1) in-depth assessment of the leaching kinetics with respect to dissolved aluminate concentration and 2) the extent of leaching under planned PEP operating conditions. The added objectives are 9, 10, and 11 in Table S.2, and the results will be released at a later date.

Quality Requirements

Pacific Northwest National Laboratory (PNNL) is operated for the U.S. Department of Energy (DOE) by Battelle under Contract DE-AC05-76RL01830. PNNL implements a Quality Assurance Program that is based upon the requirements as defined in DOE Order 414.1C, “Quality Assurance,” and 10 CFR 830, “Energy/Nuclear Safety Management,” Subpart A—“Quality Assurance Requirements.” PNNL has chosen to implement the requirements of DOE Order 414.1C and 10 CFR 830, Subpart A by integrating them into the laboratory’s management systems and daily operating processes. The procedures necessary to implement the requirements are documented through the laboratory’s Standards-Based Management System (SBMS).

PNNL implemented the RPP-WTP quality requirements by performing work in accordance with the River Protection Project – Waste Treatment Plant Support Program (RPP-WTP) Quality Assurance Plan (RPP-WTP-QA-001, QAP). Work was performed to the quality requirements of NQA-1-1989 Part I, *Basic and Supplementary Requirements*, NQA-2a-1990, Part 2.7, and DOE/RW-0333P, Rev 13, *Quality Assurance Requirements and Descriptions (QARD).* These quality requirements were implemented through the River Protection Project–Waste Treatment Plant Support Program (RPP-WTP) Quality Assurance Manual (RPP-WTP-QA-003, QAM). The analytical requirements are implemented through RPP-WTP's Statement of Work (WTPSP-SOW-005 and RPP-WTP-QA-005, respectively) with Southwest Research Institute (SWRI). The requirements of DOE/RW-0333P, Rev 13, *Quality Assurance Requirements and Descriptions (QARD),* were not required for this work.

A matrix that cross-references the NQA-1 and NQA-2a requirements with RPP-WTP's procedures for this work is given in TP-RPP-WTP-469. It includes justification for those requirements not implemented.

Experiments that were not method-specific were performed in accordance with RPP-WTP's procedures QA-RPP-WTP-1101 "Scientific Investigations" and QA-RPP-WTP-1201 "Calibration and Control of Measuring and Testing Equipment" so that sufficient data were taken with properly calibrated measuring and test equipment to obtain quality results.

RPP-WTP addressed internal verification and validation activities by conducting an Independent Technical Review of the final data report in accordance with PNNL's procedure QA-RPP-WTP-604. This review verifies that the reported results were traceable, inferences and conclusions were soundly based, and the reported work satisfied the Test Plan objectives. This review procedure is part of PNNL's RPP-WTP QAM.

R&T Test Conditions

The research and technology (R&T) test conditions, as defined in the Test Specifications 24590-PTF-TSP-RT-06-006, Rev. 0(a) and 24590-WTP-TSP-RT-07-004, Rev. 0(b) are summarized in Table S.6 and S.7, respectively.

(a) GL Smith. Nov. 2006. *Simulant Development to Support the Development and Demonstration of Leaching and Ultrafiltration Pretreatment Processes*. 24590-PTF-TSP-RT-06-006, Rev. 0.

(b) PS Sundar. April 2007. *Simulant Testing in Support of Phase I Demonstration of the Ultrafiltration and Leaching Processes in the Integrated Test Facility*. 24590-WTP-TSP-RT-07-004, Rev. 0.

Table S.6. R&T Test Conditions from 24590-PTF-TSP-RT-06-006, Rev. 0

List R&T Test Conditions	Were Test Conditions Followed?
1) Gibbsite Simulant Development— • Review the available literature and actual waste testing and characterization data. • Based on this review, target parameters for the proposed simulant that will be developed. Note that the development of these criteria must also consider the requirements for scaled testing. It will likely be necessary to develop simulants with a range of parameters that can be adjusted to provide a scaled simulant for use in the integrated test platform. • Search available vendors and preparation methods to identify available sources of boehmite materials. This review will include identifying available characterization data associated with each source material and will recommend which boehmite source materials should be obtained and tested. This recommendation should consider the diversity of both particle size and morphology in identifying candidate samples for additional testing. • Prepare and test the identified boehmite source materials. It is anticipated that testing will involve multiple samples over a range of material properties, including particle size and morphology. At a minimum, the following properties will be measured for each gibbsite source material: • Particle size • Surface area • Crystal pattern by XRD • Dissolution rate under a variety of fixed testing conditions measured under multiple conditions. Note that sufficient data must be obtained for selected samples to provide an adequate description of the reaction-rate equation. Further note that the temperature range should be sufficient to provide a range of behaviors. In addition, sufficient information, including density and water content, should be obtained to provide meaningful correlation to actual waste samples. • Equilibrium solubility under various test	Not applicable to this report. Results discussed in WTP-RPT-176.

Table S.6 (Contd)

List R&T Test Conditions	Were Test Conditions Followed?
conditions. • A correlation will be developed to predict the dissolution rate as a function of other physical characteristics. In addition, a boehmite source—or blend of boehmite sources—will be selected to best meet the criteria defined in 1). These results will be compared against the simulant basis criteria, and an appropriate method to correlate simulant performance to actual waste performance will be documented.	
2) Boehmite Simulant Development— • Review the available literature and actual waste testing and characterization data. • Based on this review, target parameters for the proposed simulant that will be developed. Note that the development of these criteria must also consider the requirements for scaled testing. It will likely be necessary to develop simulants with a range of parameters that can be adjusted to provide a scaled simulant for use in the integrated test platform. • Search available vendors and preparation methods to identify available sources of boehmite materials. This review will include identifying available characterization data associated with each source material and will recommend which boehmite source materials should be obtained and tested. This recommendation should consider the diversity of both particle size and morphology in identifying candidate samples for additional testing. • Prepare and test the identified boehmite source materials. It is anticipated that testing will involve multiple samples over a range of material properties, including particle size and morphology. At a minimum, the following properties will be measured for each boehmite source material: • Particle size • Surface area • Crystal pattern by XRD • Dissolution rate under a variety of fixed testing conditions measured under multiple conditions. Note that sufficient data must be obtained for selected samples to provide an adequate description of the reaction-rate equation. Further note that the temperature range	Boehmite sources were researched, and different boehmites were studied to see which matched the caustic leaching behavior from the past actual waste testing. Based on this, it was determined which vendors to approach and which sources of boehmite to obtain for testing. Five samples of boehmite were obtained from two sources, Nabaltec (APYRAL) and prepared by PNNL using gibbsite and pseudo-boehmite. The d_{50} particle sizes varied from ~8 µm to ~0.8 µm. XRD showed very strong, crystalline boehmite peaks with no amorphous material present. These results are discussed in Section 2. Each boehmite source obtained was fully characterized by XRD, PSD, the Brunauer, Emmet, and Teller surface area measurement method (BET), SEM, and caustic dissolution before testing as shown and discussed in Section 2 of this report. A correlation was developed to predict the dissolution rate as a function of other physical characteristics as discussed in Section 2.5 and Section 4 of this report. A boehmite source was selected (APYRAL AOH20) for further testing and discussed in Section 3 of this report. A boehmite dissolution rate model is being developed for hydroxide and temperature. This model will be reported in a later report. Insufficient data exist to address crystal size, habit, or mixing conditions on the boehmite rate model.

Table S.6 (Contd)

List R&T Test Conditions	Were Test Conditions Followed?
should be sufficient to provide a range of behaviors. In addition, sufficient information, including density and water content, should be obtained to provide meaningful correlation to actual waste samples. • Equilibrium solubility under various test conditions. • A correlation will be developed to predict the dissolution rate as a function of other physical characteristics. In addition, a boehmite source—or blend of boehmite sources—will be selected to best meet the criteria defined in 1). These results will be compared against the simulant basis criteria, and an appropriate method to correlate simulant performance to actual waste performance will be documented.	
3) Filtration Simulant Development— • Review the available literature and actual waste testing and characterization data. Based on this review, target parameters for the proposed simulant will be developed. • Search available preparation methods to identify available sources of filtration simulant materials. This review includes identification of available characterization data associated with each source material and will provide recommendations for which filtration source materials should be obtained and tested. This task also evaluates whether existing preparation methods should be modified to meet the target parameters and whether selected components from various vendors/preparation methods should be blended for evaluation. • Prepare and test the identified filtration simulant source materials. It is anticipated that testing will involve multiple samples over a range of material properties.	Not applicable to this report. Results discussed in WTP-RPT-183.

Table S.7. R&T Test Conditions from 24590-WTP-TSP-RT-07-004, Rev. 0

List R&T Test Conditions	Were Test Conditions Followed?
1) Boehmite Dissolution Tests – examine the impact of aluminate, hydroxide, and other principal anions on boehmite dissolution kinetics.	Tests were performed using different amounts of aluminate, hydroxide, and other principal anions with boehmite and sampled at various times throughout the test to determine the effect these things had on the boehmite dissolution kinetics. It was found that increasing the hydroxide concentration increases the boehmite dissolution rate. It was also found that the presence of aluminate does affect the dissolution of boehmite significantly while the presence of the other anions did not have any significant effect on the boehmite dissolution.
2) Boehmite Dissolution Tests – verify the effect of temperature on the dissolution of boehmite component B7 and verify the effect of aluminate ion on the performance of the boehmite component B3 during caustic leach at temperatures lower than 100°C.	Not applicable to this report. It will be addressed in a future report.
3) Boehmite Dissolution Tests – provide greater discrimination on anion impact by performing tests under a greater range of anion concentrations.	Not applicable to current testing. PNNL was released from this requirement by Test Exception 24590-WTP-TEF-RT-07-00016.
4) Filtration Tests – test a base simulant under identical process conditions with 2-ft and 8-ft filter elements.	Not applicable to this report. Results discussed in WTP-RPT-168.
5) Filtration Tests - increase the fines loading in filtration test base simulant to evaluate the impact of fouling on filtration performance.	Not applicable to this report. Results discussed in WTP-RPT-183.
6) Filtration Tests – use an 8-ft filter element to measure the filtration rate as a function of temperature up to 45°C for the base filtration simulant.	Not applicable to this report. Results discussed in WTP-RPT-168.
7) Aging Tests – will be performed in the 250-gal tote and a 1-gal container in the laboratory, a container in a heat-cycled oven, and a baffled 1-gal container that is mixed in the laboratory. Samples will be taken throughout the tests and characterized by particle size distribution, settling, rheology, and centrifuged solids content to evaluate the effect of aging on the behavior of the simulant.	Not applicable to this report. It will be addressed in a future report.
8) Chromium Simulant Leaching Tests – will be performed with both a caustic leach and an oxidative leach to evaluate the leaching performance of the various vendor batches of Cr-simulant.	Not applicable to this report. It will be addressed in a future report.
9) PEP Leaching Support Tests – are to be carried out with the vendor-produced 250-gal batch of the PEP simulant and the vendor-produced CrOOH Test Batch 1 simulant slurry. The tests are directed to determine the mass loss and aluminum and chromium dissolution rates during	Not applicable to this report. It will be addressed in a future report.

Table S.7 (Contd)

List R&T Test Conditions	Were Test Conditions Followed?
caustic leaching under varying temperature processing conditions without aeration in both UFP-1A/B and UFP-2A/B vessels as well as to measure the effect of aeration on chromium leaching in UFP-2A/B.	
10) PEP Leaching Support Tests – will be performed using a vendor-produced 250-gal batch of the PEP simulant. The tests are directed to measure the extent of boehmite conversion expected under leaching conditions during the planned testing in the PEP.	Not applicable to this report. It will be addressed in a future report.

Simulant Use

It is not possible to use actual Hanford tank waste in the Pretreatment Engineering Platform (PEP) because of safety, cost, and volume. To address the need for demonstrating separation and leaching processes at PEP, PNNL developed a waste simulant that mimics the chemical, leaching, and ultrafiltration behaviors of actual tank waste according to Test Plan TP-RPP-WTP-469. The development of the boehmite portion of this simulant is described in this report. The reasoning behind the simulant composition, a comparison of it to the actual tank waste, and a description of it are described in this report.

Discrepancies and Follow-on Tests

None.

1.0 Background

At the River Protection Project-Waste Treatment and Immobilization Plant (RPP-WTP), the high level waste (HLW) is separated from the low-activity waste (LAW) liquid stream by ultrafiltration in the Pretreatment Facility (PTF). The concentrated HLW will undergo caustic and oxidative leaching processes to dissolve and wash out materials (aluminum, chromium, phosphates, and sulfates) that would otherwise limit HLW loading in the immobilized waste glass. The current design calls for the leaching processes to be carried out in the ultrafiltration process vessels (UFP-1a, UPF-1b, UFP-2a and UFP-2b). The concentrated HLW solids are sequentially caustic leached, washed, and oxidatively leached and washed once more during pretreatment. While the caustic leaching dissolves the aluminum in the HLW solids, the oxidative leaching is carried out to oxidize the chromium using a sodium permanganate ($NaMnO_4$) solution and dissolve it in a mild caustic solution. The HLW solids are concentrated after each leach and wash operation using the cross-flow ultrafiltration system to perform crossflow filtration.

Caustic-leaching experiments were first performed on actual Hanford tank sludge samples in FY 1993. The original caustic-leaching experiments were performed as a prelude to acid dissolution of the sludge solids with the intent that the acid-dissolved fraction would be processed through solvent extraction to separate the very small mass fraction of the radioactive elements (the transuranics [TRUs], ^{90}Sr, and ^{137}Cs) from the bulk mass of non-radioactive components (Lumetta et al. 1996). In this respect, caustic leaching was meant to remove the large amount of aluminum from the waste, thus reducing the nitric acid demand and simplifying the solvent extraction feed. Subsequently, caustic leaching was chosen as the baseline method for Hanford tank sludge pretreatment; this process was sometimes referred to as "Enhanced Sludge Washing" (Lumetta et al. 1997). Following this decision, caustic-leaching tests were performed under a standard set of conditions at the Pacific Northwest National Laboratory (PNNL) and Los Alamos National Laboratory (LANL); these tests were conducted from FY 1995 through FY 1997. In subsequent years, a limited number of parametric caustic-leaching experiments were performed at PNNL and also at Oak Ridge National Laboratory (ORNL). After the WTP project was established, a limited number of laboratory-scale caustic-leaching experiments were performed using a standard testing protocol, but these were generally focused on processing double-shell tank (DST) wastes rather than the single-shell tanks (SST) where the bulk of the sludge is stored.

Caustic-leaching data are needed on the various types of wastes to be processed through the WTP to support the plant design. The data needed include 1) removal of key HLW sludge components (e.g., Al, Cr, P, and S) as a function of caustic concentration, temperature, and time, 2) the behavior of radionuclides during the leaching process, 3) particle-size distribution, and 4) identification of the chemical and mineral forms of important sludge components (e.g., Al, Cr, and P) in the sludge solids. These new data will support the development of various waste simulants for scaled process demonstrations.

Aluminum in the wastes is believed to be present in the two most common mineralogical phases: gibbsite (monoclinic $Al(OH)_3$) and boehmite (orthorhombic AlOOH). Other phases present include bayorite, dawsonite, alumina silicates, and amorphous aluminum hydroxide. The dissolution rates of the two primary mineralogical phases are considerably different. Therefore, the leaching kinetics will depend on the relative amounts of these phases in the waste as well as particle size, crystal habit (i.e., particle size and shape), operating temperature, hydroxide activity, aluminum solubility limits, particle Reynolds number associated with the mixing system, etc. While there may be other phases of aluminum compounds in the waste solids, they are present in relatively smaller amounts and therefore are considered less significant to the caustic leaching for removing aluminum from the HLW.

The simulant development strategy is based on a two-phased approach. The first phase includes simulant development and the validation of initial single-component chemical and physical simulants for boehmite, gibbsite, chromium, and filtration. The data obtained from the actual waste testing (Sundar 2006) are to serve as benchmarks for defining the simulant characteristics and behaviors to develop chemical, rheological, and physical simulants to more closely simulate actual waste and as a basis for revising the parameters used in evaluating WTP process performance using the appropriate process models.

The second phase of the simulant development work involves refinement of the first four component simulants based on additional tank waste characterization data. In addition, the other component simulants (such as phosphate) will be developed during this stage. This report only addresses the first phase of the simulant development work relating to the boehmite simulant development.

This report is broken into four tasks. The first task screened a set of available boehmite simulants and down-selected one of these simulants for additional testing, based on existing actual waste data. This task is described in Section 2 of this report. The second task was to use the chosen boehmite simulant to assess the impact of process variables on boehmite leaching performance. This task is described in Sections 3 and 4. The next task was to assess the performance of the boehmite simulant when blended with the remainder of the simulant components under prototypic processing conditions. This task is described in Sections 5 and 6. The final task was to use more recent actual waste boehmite leaching data and to re-evaluate the selection of the boehmite component. This task also evaluated some additional boehmite sources that had been identified. This task is described in Section 7.

2.0 Initial Boehmite Source Characterization

As described in Section 1, the first task associated with the boehmite simulant development was to identify available boehmite sources and evaluate them against existing actual waste data. Three APRYAL samples of boehmite were obtained from Nabaltec (see Appendix F for manufacturer's product information) and evaluated based on the particle sizes that most closely matched the actual waste boehmite particle sizes. An additional sample was obtained from BASF (BASF G250). However, upon receipt, this sample was determined to be pseudoboehmite based on the X-ray diffraction (XRD) evaluation. No other commercially available source of boehmite was identified. Therefore, two samples were converted from non-boehmite material into boehmite through autoclaving gibbsite (or pseudoboehmite) in water at 300°C for 4 hours stirred at 600 rpm (Panias 2001). These samples were the pseudoboehmite from BASF (G250) and a gibbsite material from Almatis (C31C). These samples were labeled as shown in Table 2.1. These boehmite samples were then characterized as described in the following subsections.

Table 2.1. Boehmite Samples and Sources

Sample ID	Boehmite Sources
B1	Nabaltec APYRAL AOH60
B2	Nabaltec APYRAL AOH20Y
B3	Nabaltec APYRAL AOH20
B4	PNNL Autoclaved BASF G250 (pseudoboehmite)
B5	PNNL Autoclaved Almatis C31C (gibbsite)

2.1 X-Ray Diffraction (XRD)

The XRD instrument used for this analysis was a Scintag PAD V X-ray Diffractometer. Data were taken from 2 to 65 degrees 2-theta with a step size of 0.02 degrees 2-theta and counting time between 2 and 19 seconds per step. Copper Kα X-rays were used. Tube conditions were 45 kV and 40 mA.

Phase identification was done with the JADE search match routines (Version 8.0, Materials Data Inc.) with comparison to the International Centre for Diffraction Data (ICDD) database PDF-2 release 2006, Version 2.0602, which includes the Inorganic Crystal Structure Database (ICSD) maintained by Fachinformationszentrum (FIZ), Karlsruhe, Germany. The first identification pass was done with no chemistry restrictions and usually identified TiO_2, the internal standard. The next pass was done with the identified chemistry restrictions of Al, O, and H with probable phases boehmite, gibbsite, and "pseudoboehmite" required. Consequently, the aluminum-containing phases were identified.

All of the boehmite samples analyzed showed an excellent match to the boehmite standard XRD card. No amorphous material or contamination was detected in these samples except for the internal standard of rutile. These XRD patterns are shown in Figure 2.1 through Figure 2.5.

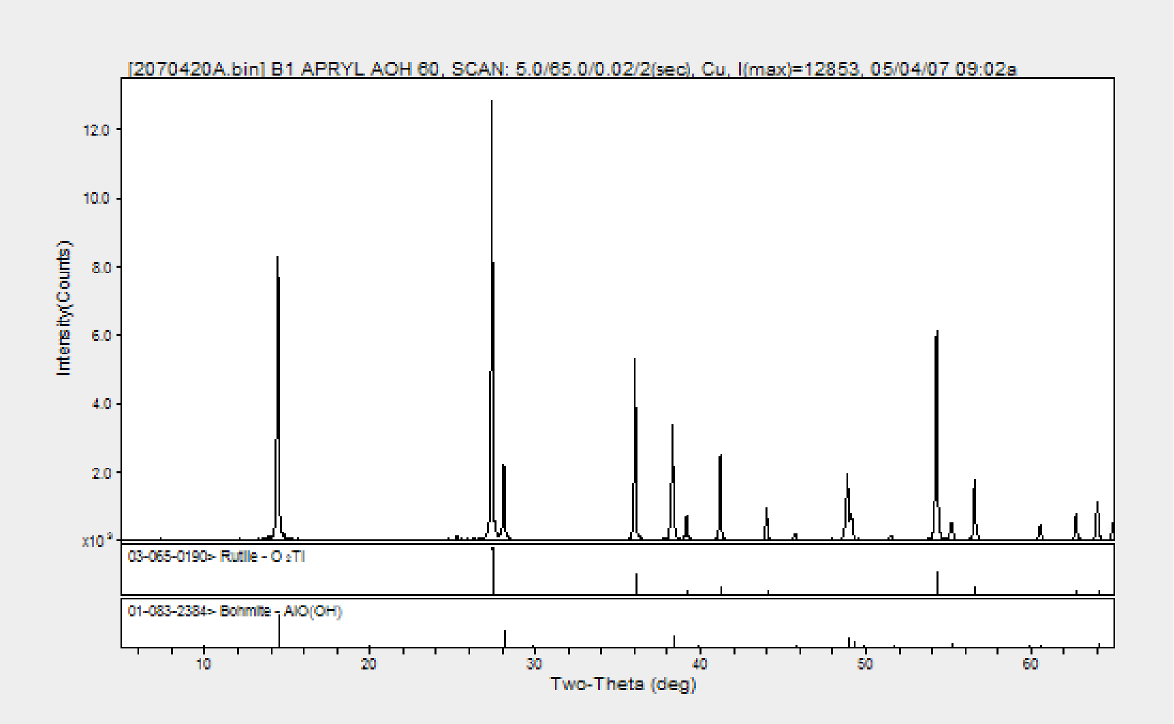


Figure 2.1. XRD Pattern of Boehmite Sample B1

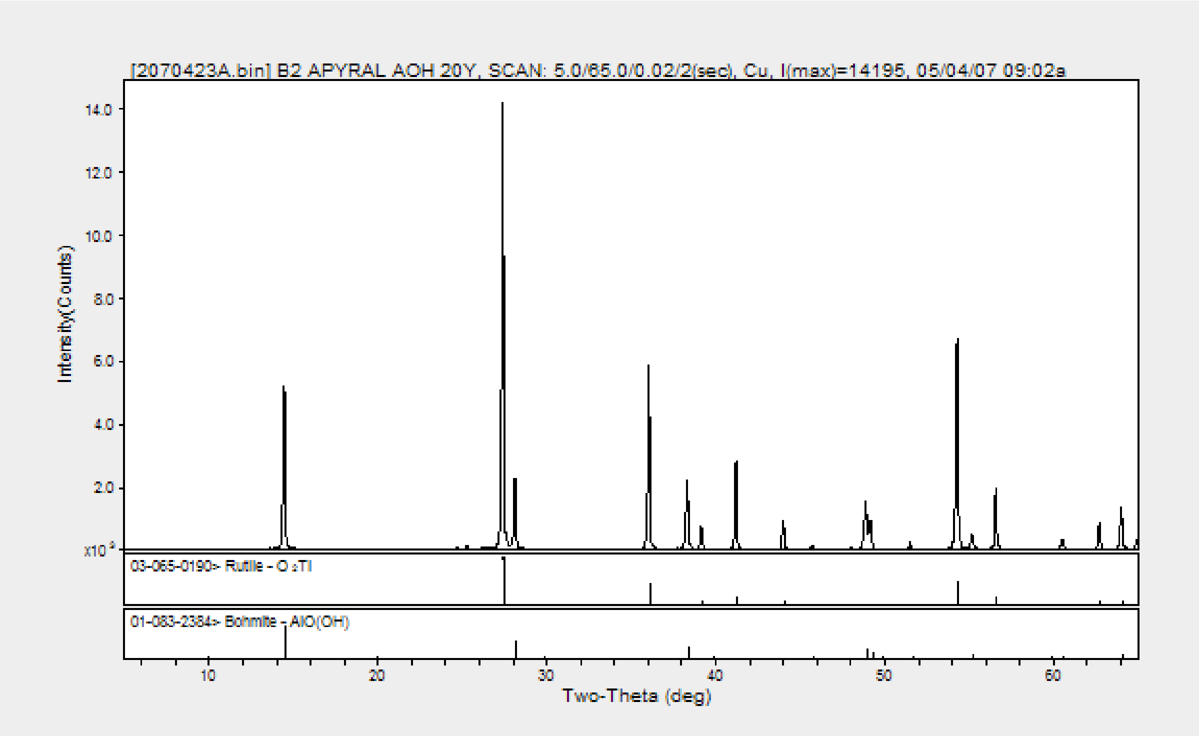


Figure 2.2. XRD Pattern of Boehmite Sample B2

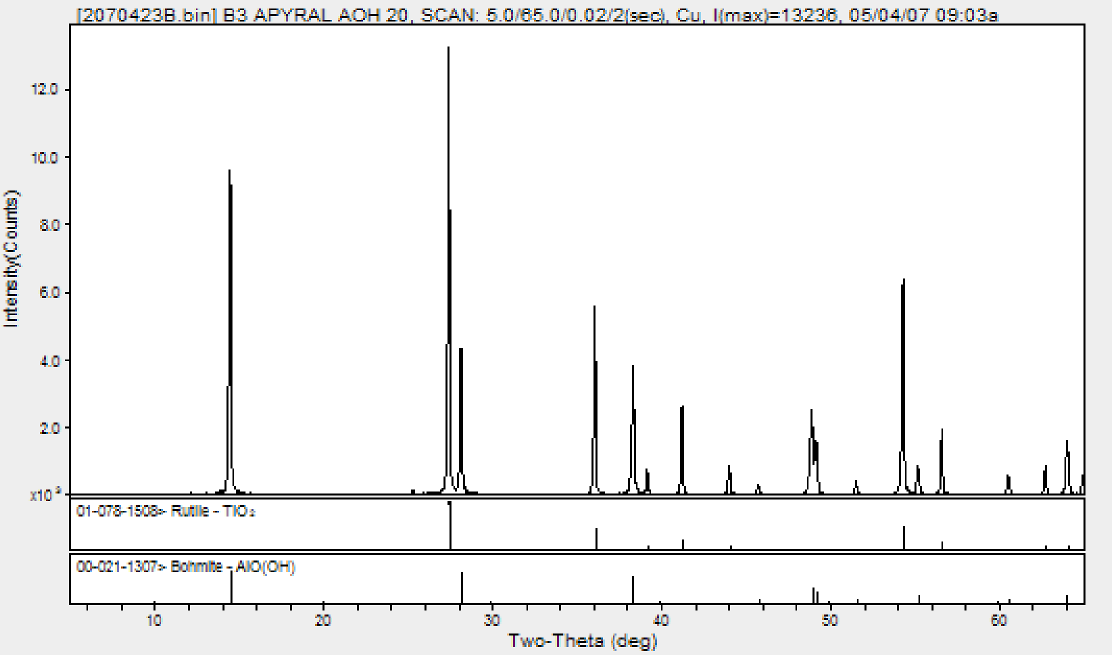


Figure 2.3. XRD Pattern of Boehmite Sample B3

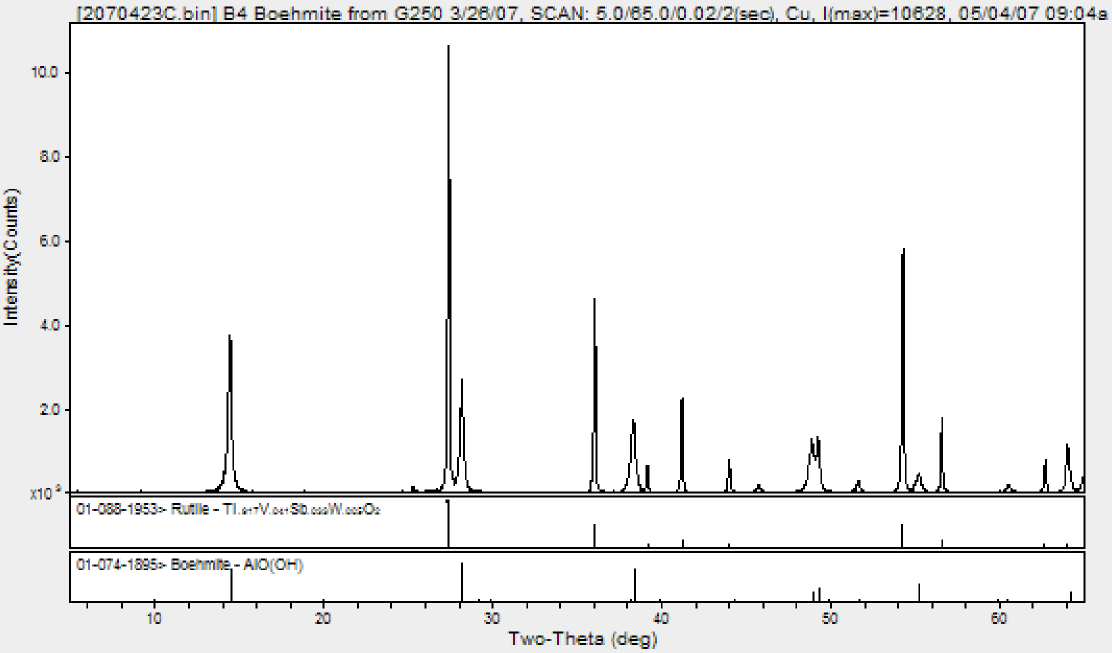


Figure 2.4. XRD Pattern of Boehmite Sample B4

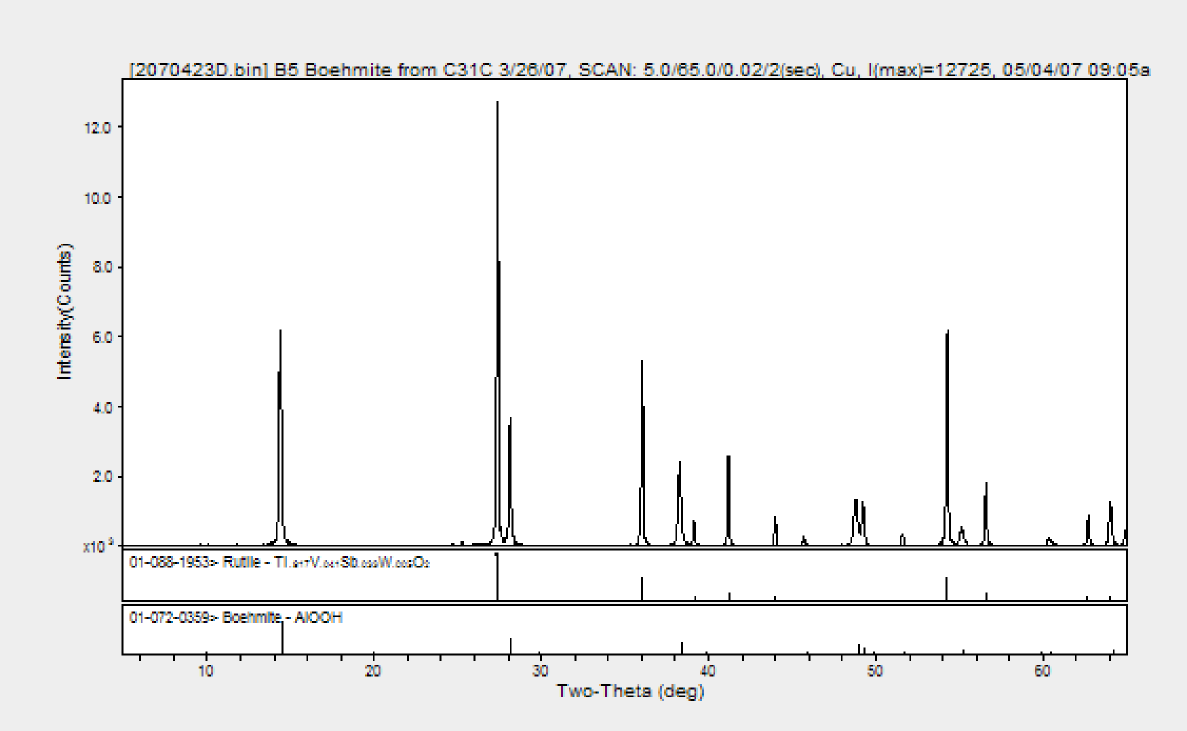


Figure 2.5. XRD Pattern of Boehmite Sample B5

2.2 Particle-Size Distribution (PSD)

The boehmite samples were analyzed with a S3000 Microtrac Analyzer according to procedure TPR-RPP-WTP-222, Rev. 3. Nominally 0.2 grams of each sample (run in duplicate) was placed into approximately 10 grams of de-ionized water (DIW), and 2 to 3 drops of Darvan® 821A Dispersing Agent were added. The resultant slurry was de-agglomerated using an ultrasonic horn (MICROGON Ultrasonic Cell Disrupter) intermittently for 15 to 20 seconds. A transfer pipette was used to mix the slurry and transfer the required amount to the analyzer. The amount of the slurried sample aliquot required for analysis varied with the actual mass of the sample and the actual volume of DIW in the sonicated sample. Neither the amount of sample nor the amount of water was critical to the analytical process since the internal system software visually indicated to the analyst the amount needed for analysis. These results are shown in Table 2.2 and Table 2.3 and Figure 2.6 and Figure 2.7.

Table 2.2. Volume Distribution Particle-Size Results

Percentile	B1 (µm)	B2 (µm)	B3 (µm)	B4 (µm)	B5 (µm)
5	0.423	0.632	1.078	0.144	0.507
20	0.559	1.989	3.910	0.301	0.829
30	0.627	4.285	5.417	0.446	1.058
40	0.698	6.157	6.725	0.579	1.310
50	0.776	7.738	7.969	0.780	1.595
60	0.874	9.256	9.254	1.199	1.939
70	1.007	10.90	10.71	2.052	2.382
80	1.225	12.95	12.59	3.564	3.034
90	1.734	16.22	15.64	6.807	4.299
95	2.531	19.53	18.76	11.36	5.807

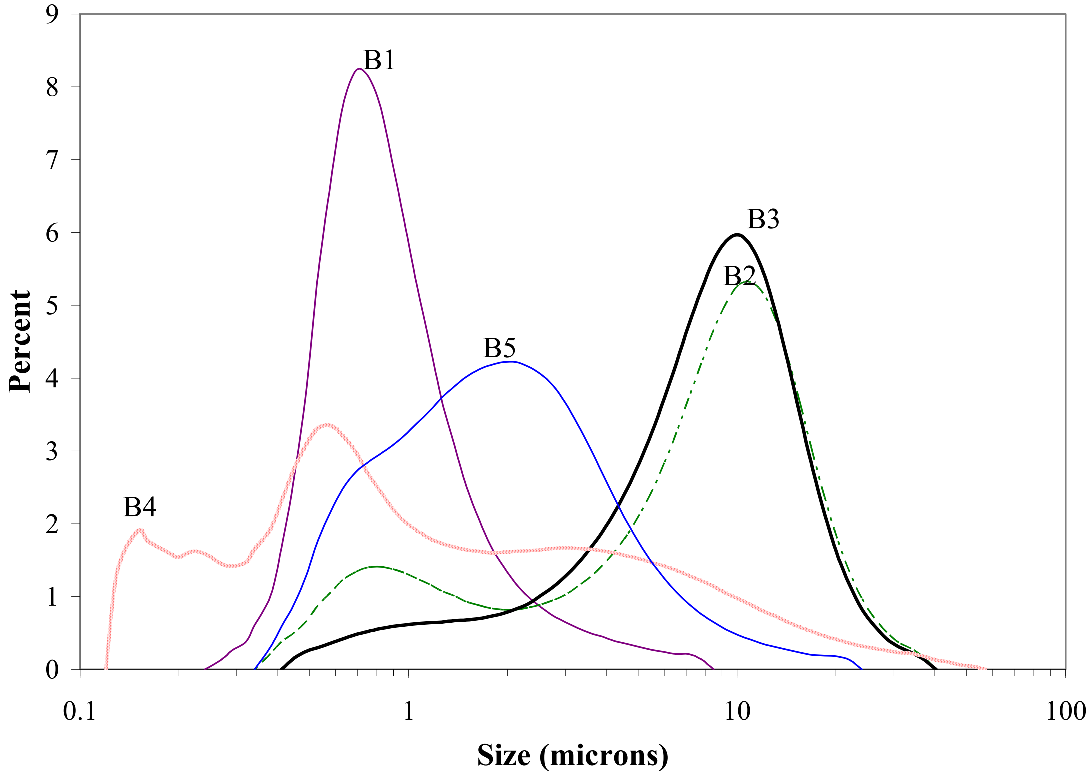


Figure 2.6. Boehmite Volume Distribution Particle-Size Plot

Table 2.3. Number Distribution Particle-Size Results

Percentile	B1 (µm)	B2 (µm)	B3 (µm)	B4 (µm)	B5 (µm)
5	0.286	0.366	0.430	0.125	0.365
20	0.378	0.416	0.477	0.134	0.412
30	0.424	0.451	0.512	0.139	0.447
40	0.465	0.489	0.554	0.146	0.487
50	0.506	0.531	0.603	0.154	0.533
60	0.549	0.579	0.664	0.166	0.590
70	0.601	0.639	0.748	0.183	0.665
80	0.669	0.725	0.880	0.212	0.782
90	0.785	0.886	1.166	0.285	1.016
95	0.908	1.078	1.612	0.395	1.290

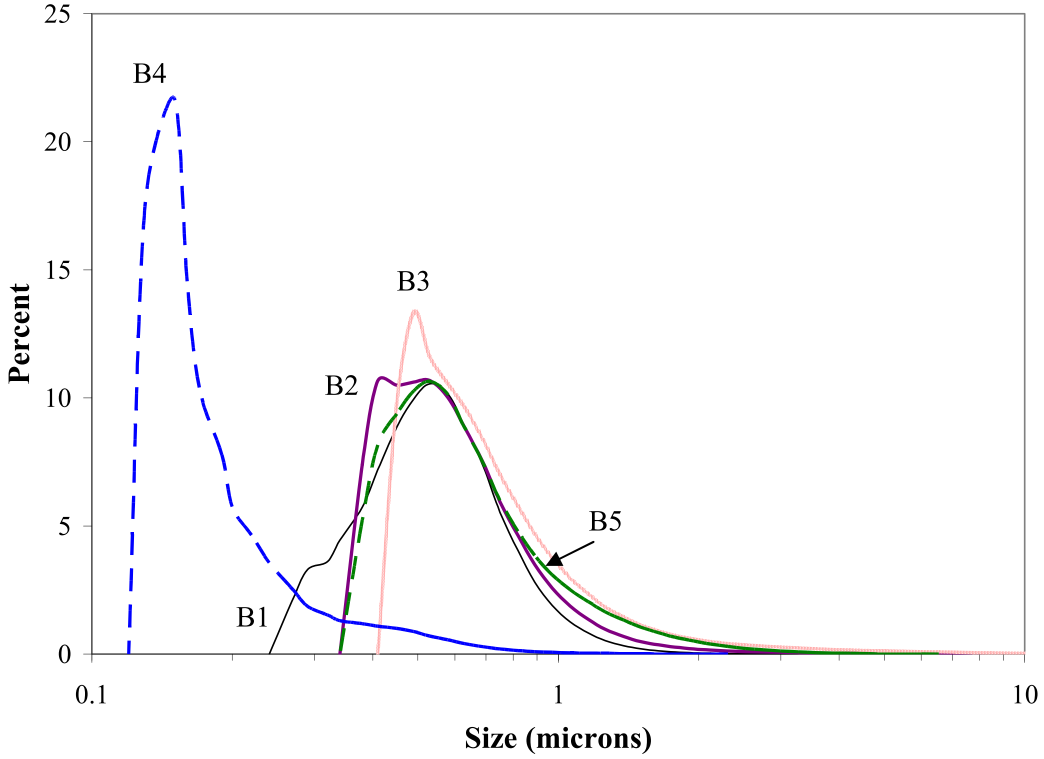
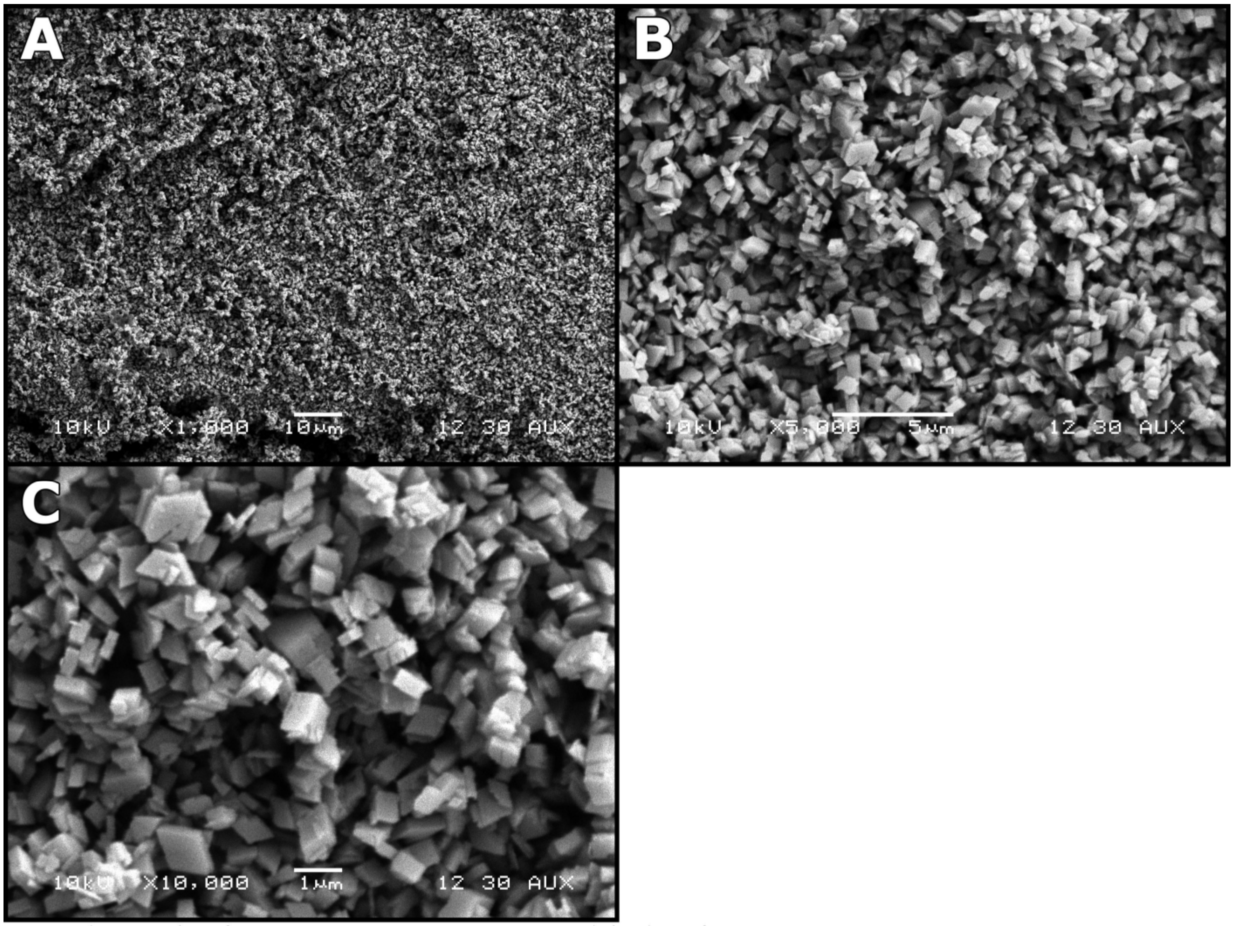


Figure 2.7. Boehmite Number Distribution Particle-Size Plot

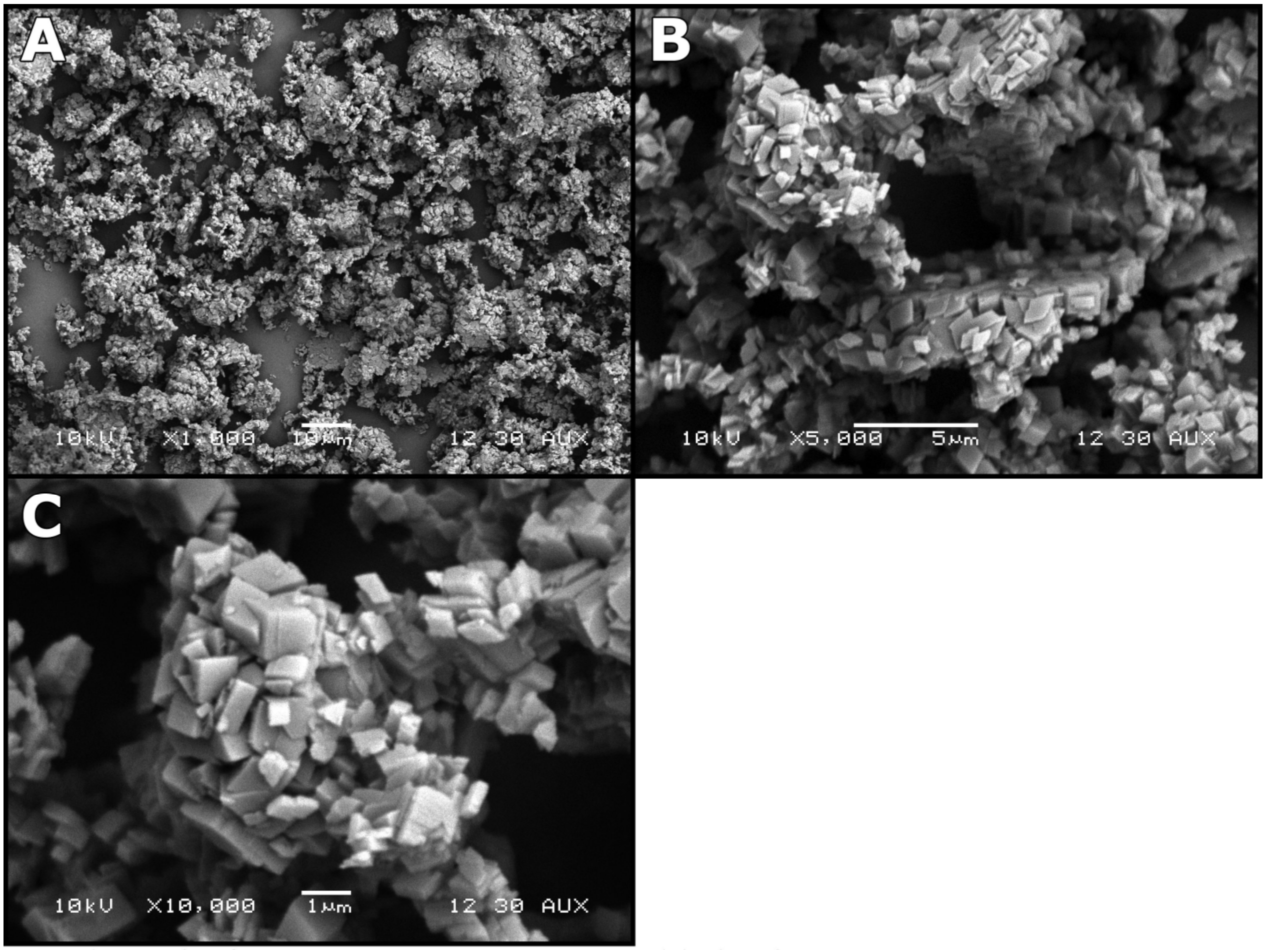
2.3 Scanning Electron Microscope (SEM)

The SEM images were obtained with a JEOL JSM-5900 SEM with the sample on a glass slide and coated with carbon. The micrographs are secondary electron images. They all showed similar crystal structure with the exception of B4, which showed larger agglomerated particles as shown in Figure 2.8 through Figure 2.12.



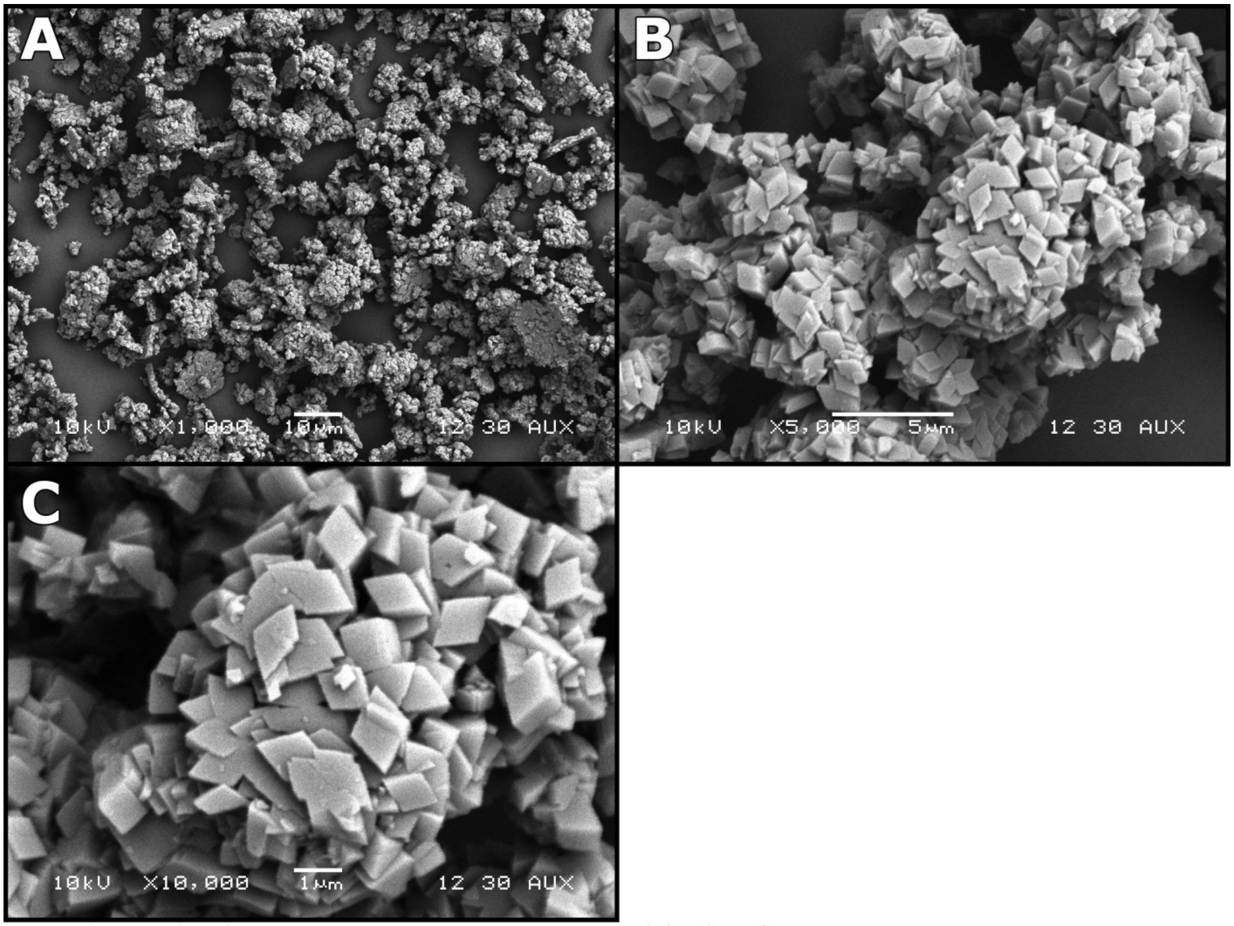
SEM micrographs of APYRAL AOH 60. Average particle size of 0.69 µm.

Figure 2.8. SEM Picture of Boehmite Sample B1 at 1,000×, 5,000×, and 10,000×



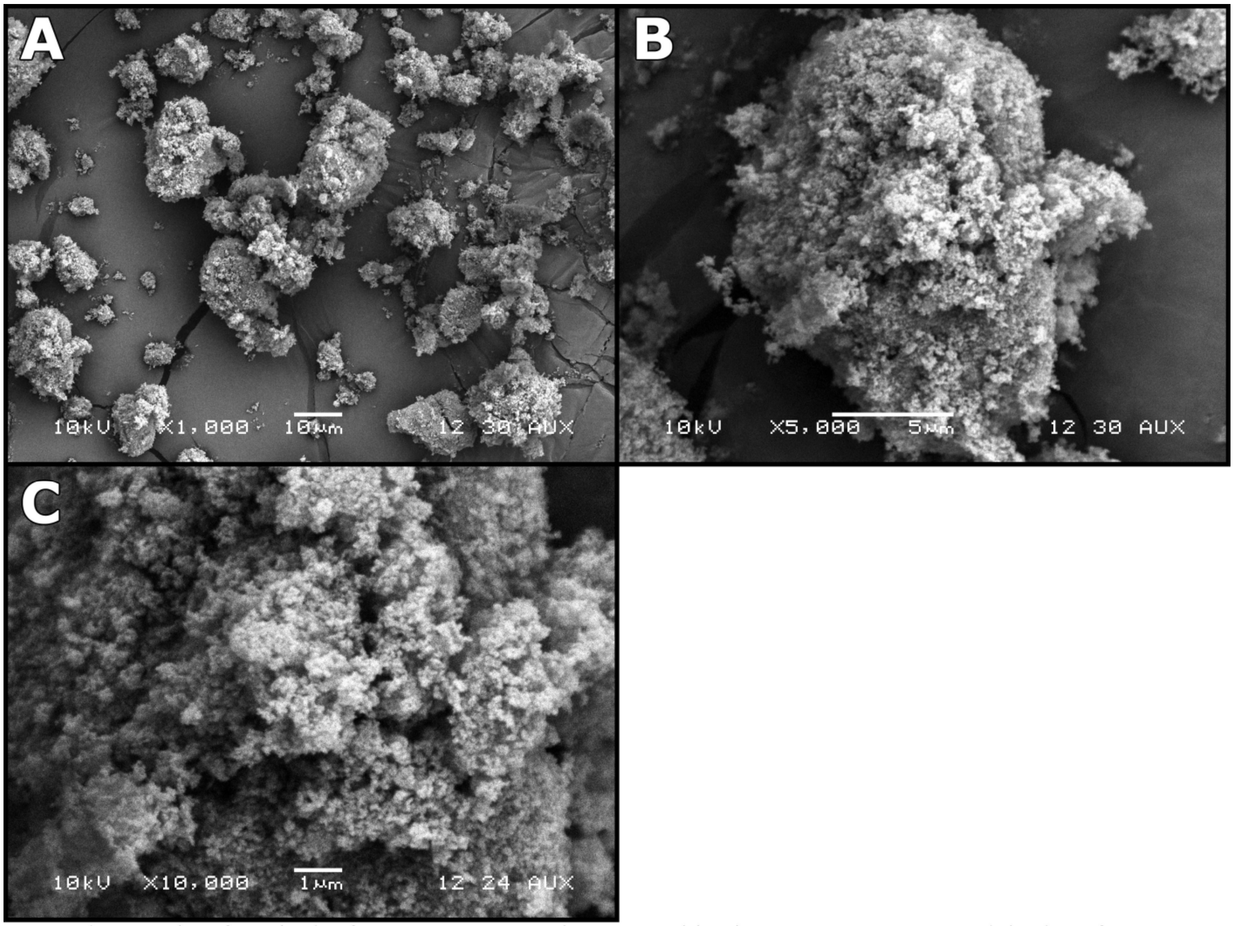
SEM micrographs of B2 APYRAL AOH 20Y. Average particle size of 0.72 µm.

Figure 2.9. SEM Picture of Boehmite Sample B2 at 1,000×, 5,000×, and 10,000×



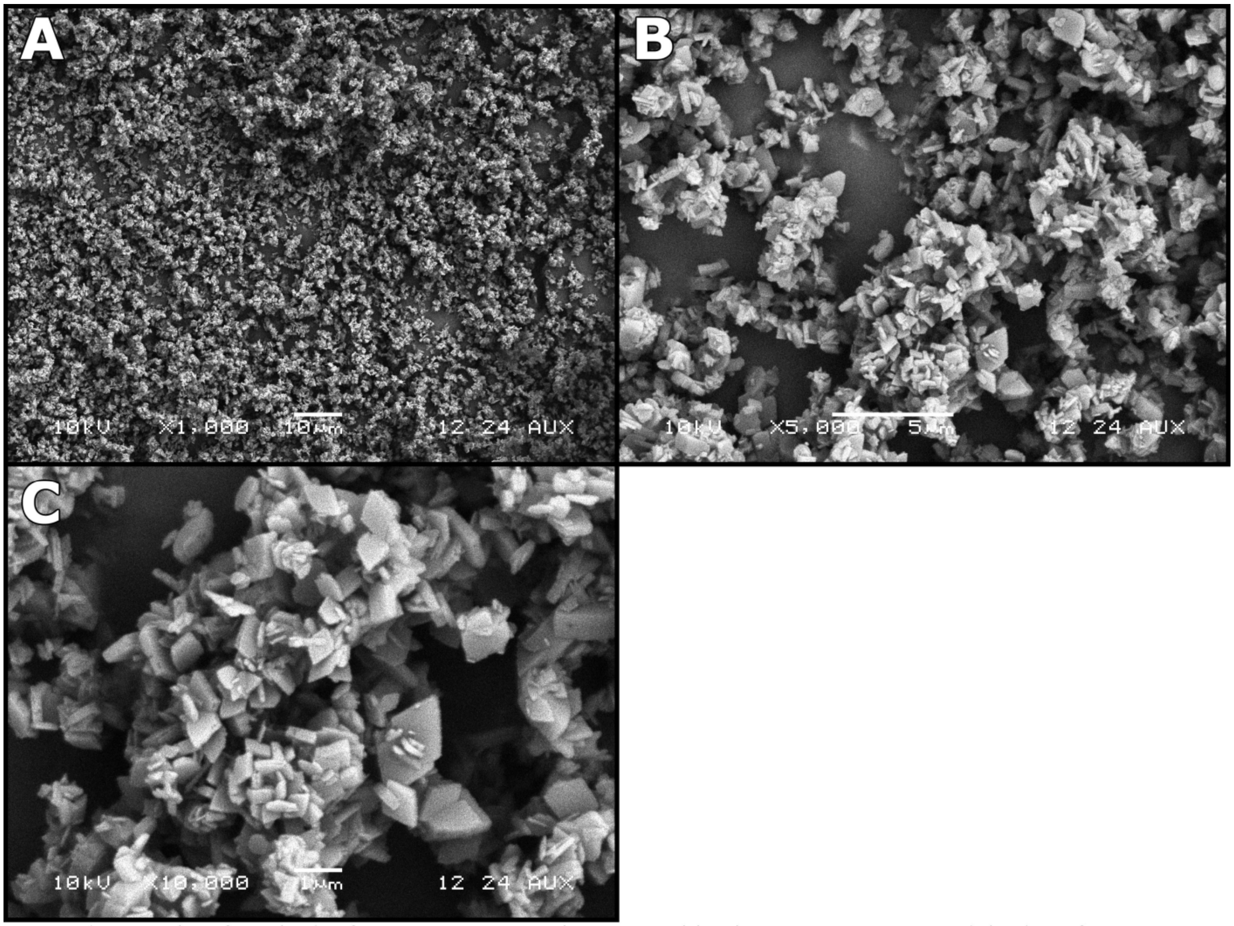
SEM micrographs of B3 APYRAL AOH 20 . Average Particle Size of 0.83 μm.

Figure 2.10. SEM Picture of Boehmite Sample B3 at 1,000×, 5,000×, and 10,000×



SEM micrographs of Boehmite from g-250 300°C 4hr 3-26-07 blend 4,7,8,12. Average particle size of 8.3 µm.

Figure 2.11. SEM Picture of Boehmite Sample B4 at 1,000×, 5,000×, and 10,000×



SEM micrographs of Boehmite from C31C 300°C 4hr 3-26-07 blend 2,9,10. Average particle size of 0.67 µm.

Figure 2.12. SEM Picture of Boehmite Sample B5 at 1,000×, 5,000×, and 10,000×

2.4 Surface Area

Surface-area measurements were acquired with a Quantachrome Instruments Monosorb Surface Area Analyzer. The Monosorb is a direct-reading dynamic-flow surface-area analyzer that uses a modified Brunauer, Emmett, and Teller (BET) equation for single-point determinations of surface area. Measurements were taken in accordance with PNL-RW-05-BET and OCRWM-BET-01.

Standard surface-area reference material (SARM) was used to calibrate the instrument over the anticipated range of interest. Each sample was weighed and dried for no less than 30 minutes before analysis. The Monosorb measures the quantity of a gas adsorbed on a solid surface by sensing the change in thermal conductivity of a flowing mixture of an adsorbate and an inert carrier gas, nitrogen and helium, respectively. The specific surface area is calculated by dividing the average of the surface-area measurements obtained by the BET method by the weight of the sample. Based on the small sample used for B1 and the fact that the data are not consistent with the PSD and the leaching results, these data may not be accurate and should be used as information only. This measurement also did not agree with the manufacturer's reported value, which matches the PSD and leaching results better. The source of this error is most likely due to the small sample size (for which the reason is unknown) and the small amount of signal that would have been received from it. Also, because the surface area was so far from 2.84 m^2 (the single point of calibration), there may not have been a linear response. These results are shown in Table 2.4.

Table 2.4. Surface Area of Boehmite Samples

Sample ID	Mass (g)	Surface Area (m^2)	Specific Surface Area (m^2/g)	Std Dev	Manufacturer Surface Area (m^2/g)
B1	0.0076	0.0832	10.95	1.98	2.4
B2	0.1871	0.5906	3.173	0.058	3.0
B3	0.1727	1.765	10.22	1.11	6.0
B4	0.1209	1.482	12.25	3.84	NA
B5	0.2402	1.164	4.846	0.011	NA

2.5 Screening Leach Test

A screening-test approach to provide dissolution-rate data for available boehmite materials was used that could be compared to the data for actual waste boehmite dissolution rates.

These caustic leaching tests were set up as shown in Figure 2.13 using 1-wt% boehmite solids in 800 grams of 3-M NaOH solution that was heated to a temperature of 100°C while stirring in a 1-liter PMP reaction vessel. The reaction vessel is a straight-side wide-mouth jar with an inside height of 116 mm and an inside diameter of 110 mm. It has three PMP baffles of which each are 92 mm long, 5 mm thick, and 19 mm wide evenly spaced around the vessel. A stainless steel stir shaft 8 mm in diameter and 305 mm long with a 95-mm-wide blade welded on the bottom is used to stir the vessel. The blade is 13 mm tall, and each blade is bent 45° from vertical. A heating jacket is wrapped around the vessel to maintain the test mixture at a constant temperature throughout the test. In contrast, Lumetta used ~ 100-mL samples in bottles on a shaker table. The temperature was measured with a calibrated thermocouple and controlled with a calibrated temperature controller. Boehmite was added as a powder to the reaction vessel through the sample port while stirring after the NaOH solution had reached 100°C, which started the clock for the test. The test solution was sampled while stirring at 1, 2, 4, 8, and 24 hours. Each sample consisted of 5-mL supernatant, which was filtered through a 0.45-μm syringe filter after being drawn from the reaction vessel and then analyzed for aluminum and sodium by inductively coupled plasma-atomic emission spectrometry (ICP-AES). The actual amounts of constituents used in each test are shown in Appendix B, and the analytical results are shown in Appendix A.

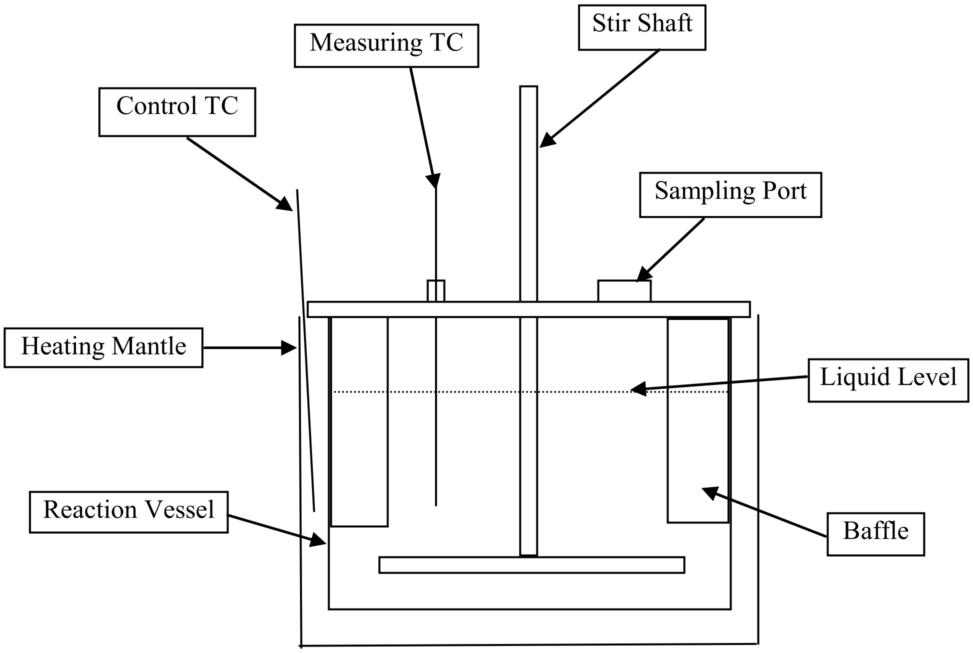


Figure 2.13. Schematic Drawing of the Caustic Leaching Test Setup

The results from the boehmite leaching tests as well as results from actual waste leaching are given in Table 2.5. Lumetta et al. (2001) and Lumetta and Hallen (2007) reported boehmite dissolution in S-110 waste and S-101 waste under the same testing conditions of temperature and caustic concentration. Note that the tank waste tests used washed solids, and as such had minor concentrations of other anions present. However, these anion levels were very low and are not expected to impact the dissolution rates. When Lumetta's data are plotted with the boehmite leaching data from these tests, as shown in Figure 2.14, B3 and B5 match the closest.

Table 2.5. Screening Boehmite Leaching Tests Compared to Actual Waste Leaching Results

Time - h	Fraction of Boehmite Dissolved						
	B1	B2	B3	B4	B5	S-110 (a)	S-101 (b)
0	0	0	0	0	0	0	0
1	0.111	0.106	0.106	0.431	0.075	--	--
2	0.188	0.176	0.175	0.625	0.126	--	--
4	0.310	0.288	0.290	0.790	0.220	0.245	0.321
8	0.502	0.435	0.445	0.919	0.347	0.420	--
24	0.958	0.853	0.806	1.024	0.712	0.731	0.660
F (~28)	0.968	0.845	0.778	1.004	0.698	--	--

(a) Lumetta et al. (2001).
(b) Lumetta and Hallen (2007).

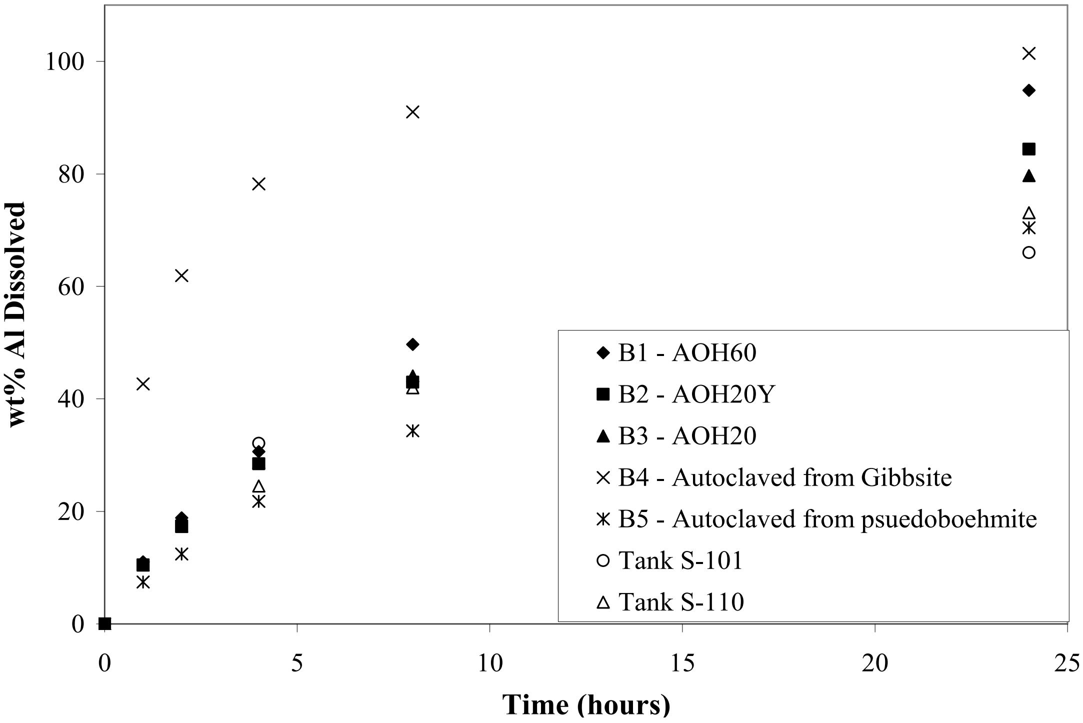


Figure 2.14. Screening Boehmite Leaching Test Results Compared to Actual Waste Results

When the boehmite simulant results are plotted with the data from Lumetta (as shown in Table 2.5 and Figure 2.14), B3 and B5 match the results from tank waste S-101 and S-110 (high boehmite containing tank waste) the best. Based on the SEM pictures (shown in Figure 2.10 and Figure 2.12, respectively) the B3 sample has more uniform crystals whereas the B5 sample appears to have a more dispersed range of crystal sizes. The primary driver in selecting B3 over B5 was the commercial availability of B3. Note that the tank waste tests used washed solids, and as such had minor concentrations of other anions present. However, these anion levels were very low and are not expected to impact the dissolution rates.

Therefore, the B3 boehmite is recommended because of the crystalline structure and particle size as well as its availability in commercial quantities at $1.95 per lb while B5 is a laboratory-produced batch with little experience to say how easily reproducible the material is. Also, it would most likely be expensive to produce compared to the commercial material.

3.0 Boehmite Simulant Testing Matrix Results

As indicated in Section 1, the second task as part of boehmite simulant development was to assess the impact of process conditions on the simulant leaching behavior. This section deals with assessing the impact on the dissolution rate for boehmite from temperature, hydroxide concentration, approach to solubility, and stirring rate. These variables were tested to determine the effect of each on the dissolution rate of the B3 (APYRAL AOH20) boehmite, as shown in Table 3.1. This test matrix is based on a statistical design with the temperature, sodium hydroxide concentration, and the expected boehmite percent solubility of the sample calculated with the equation of Panias et al. (2001) being the primary variables and the degree of mixing (stirring rate) being a secondary variable. The time to achieve a specific percent boehmite dissolution is the dependent variable, and the temperature, stirring rate, and hydroxide concentration are the independent variables in this experimental design. The actual amounts used in the tests are shown in Appendix B. Boehmite solubility is directly related to the strength of the hydroxide concentration. Therefore, to keep the amount of boehmite added at the correct percentage of its solubility limit, the amount of boehmite added had to be adjusted for each test based on the hydroxide concentration being used. If a constant amount of boehmite had been added, then the percent of the boehmite solubility limit would have changed as the concentration of hydroxide changed.

Known quantities of boehmite (see Appendix B) were exposed with stirring in 800 grams of caustic-leach solution under the conditions listed in Table 4.1 in a reaction vessel. Note that tests 1 and 9 are duplicates and tests 8 and 10 are duplicates. In addition, tests 11 through 13 are triplicate experiments. These replicates were performed to provide an understanding of the reproducibility of the tests. Also, these tests were performed as a statistical design to obtain the maximum amount of information in a limited data set.

These tests were performed in exactly the same manner as those described in Section 2.5. Samples of supernatant were taken at 1, 2, 4, 8, and 24 hours and analyzed to determine how much boehmite had dissolved by measuring aluminum and sodium concentration with ICP-AES.

The temperature range for the test matrix was chosen to provide an upper and lower bound on the expected plant operating condition for boehmite leaching. A heating jacket around the vessel maintained the test mixture at a constant temperature throughout the test.

The percent boehmite solubility primary variable is an indicator of the boehmite sample size relative to the leach solution for each test condition. It is defined as the ratio of the aluminum concentration in the liquid phase to the maximum possible concentration (solubility limit). Thus, when the target value is 50% solubility, the test contains sufficient boehmite to reach 50% of the solubility limit, assuming total dissolution of boehmite. The boehmite solubility was calculated with the equation of Panias et al. (2001), which is an empirical model based on historical solubility data. The percent solubility range was chosen to provide insight into the impact of the approach to solubility on the dissolution rate.

$$C_{Al2O3} = A_1 \times 10^{-6} T^3 + A_2 \times 10^{-3} T^2 + A_3 \times 10^{-2} T + A_4 \qquad (3.1)$$

where C_{Al2O3} is the solubility of boehmite in grams per liter of Al_2O_3; T is the temperature in degrees Celsius; and A_1, A_2, A_3 and A_4 are parameters dependent on the initial concentration of sodium hydroxide in the solution according to the following equations:

$$A_1 = -0.0618925C_{Na2O} + 1.36953 \tag{3.2}$$

$$A2 = 0.02301\ C_{Na2O} + 0.1707 \tag{3.3}$$

$$A3 = 2.498x10\text{-}6\ C_{3Na2O} - 3.106x10\text{-}4\ C_{2Na2O} + 5.483x10\text{-}2\ C_{Na2O} - 1.332 \tag{3.4}$$

$$A4 = 3.236x10\text{-}6\ C_{3Na2O} - 7.887x10\text{-}4\ C_{2Na2O} + 1.584x10\text{-}1\ C_{Na2O} - 2.518 \tag{3.5}$$

where C_{Na2O} is the initial concentration of sodium hydroxide in grams per liter of Na_2O.

The leach-solution hydroxide concentration was defined as the concentration before the start of testing, and the concentrations tested were chosen to bound the expected range of conditions in the WTP.

The stirring rates were chosen to provide different levels of solids suspension in the supernatant, based on the degree of agitation of the surface of a water sample in the test vessel. The minimal speed was just slow enough to leave the water surface undisturbed when viewed with reflected light. The full stir speed resulted in a well-agitated surface, and the intermediate mixing rate resulted in minor agitation of the surface. The minimal stir rate (40 rpm) was intended to be just sufficient to keep the solids from completely settling on the bottom of the container to maintain contact with the bulk liquid. The full-speed stir rate was intended to give a completely homogeneous suspension of the solids, and the ½-speed stir rate was intended to result in an intermediate solids suspension. The digital rate controllers for the stirring motors were set to specific revolutions per minute (rpm), and the rates at different settings were verified by direct observation and manual timing. The variation in the stirring rates is expected to be less than 1%.

Stirring rates of 40, 60, and 120 rpm were employed for these tests. In general, it was observed that at 40 rpm, most of the boehmite settled to the bottom of the reaction vessel, and the supernatant remained visibly clear. At 60 rpm, the settled layer was thinner, and some material appeared to be suspended in the supernatant. At 120 rpm, there was no settled layer, and the mixture appeared to be a uniform milky white. It can be surmised that the access of the hydroxide solution to the aluminum phase was much better at the highest rate of stirring than at the lowest.

Table 3.1. Boehmite Testing Matrix

Test ID	% Solubility	Temperature (°C)	Mixing (rpm)	Leach Soln Conc. at Beginning
MTB-1	100	100	Full (120)	5 M NaOH
MTB-2	50	100	Minimal (40)	5 M NaOH
MTB-3	100	100	Minimal (40)	1 M NaOH
MTB-4	50	100	Full (120)	1 M NaOH
MTB-5	100	60	Minimal (40)	5 M NaOH
MTB-6	50	60	Full (120)	5 M NaOH
MTB-7	100	60	Full (120)	1 M NaOH
MTB-8	50	60	Minimal (40)	1 M NaOH
MTB-9	100	100	Full (120)	5 M NaOH
MTB-10	50	60	Minimal (40)	1 M NaOH
MTB-11	75	80	½ Speed (80)	3 M NaOH
MTB-12	75	80	½ Speed (80)	3 M NaOH
MTB-13	75	80	½ Speed (80)	3 M NaOH

Figure 3.1 shows the effect of hydroxide concentration on boehmite dissolution with the 5-M NaOH solution dissolving more than the 1-M NaOH solution (MTB-6 and MTB-7) with the percent solubility difference taken into account. This indicates that hydroxide concentration has an effect on the boehmite dissolution, which becomes greater with time. Complete results of these tests are shown in Appendix A.

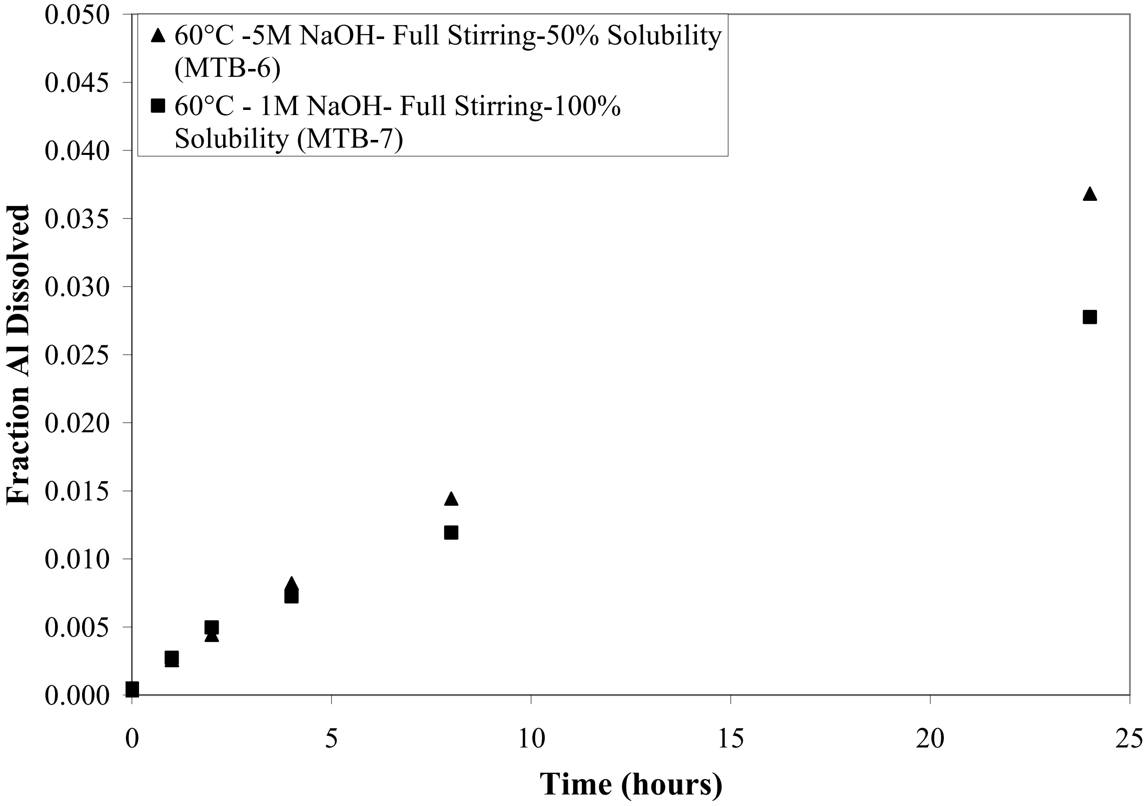


Figure 3.1. Effect of Hydroxide Concentration on Boehmite Dissolution.

Figure 3.2 compares the dissolution data for boehmite leached at 100°C and 60°C in 5 M NaOH at full stir speed (120 rpm) (MTB-1 and MTB-6). The temperature over the range tested has a significant effect on the dissolution rate for the boehmite tested (B3).

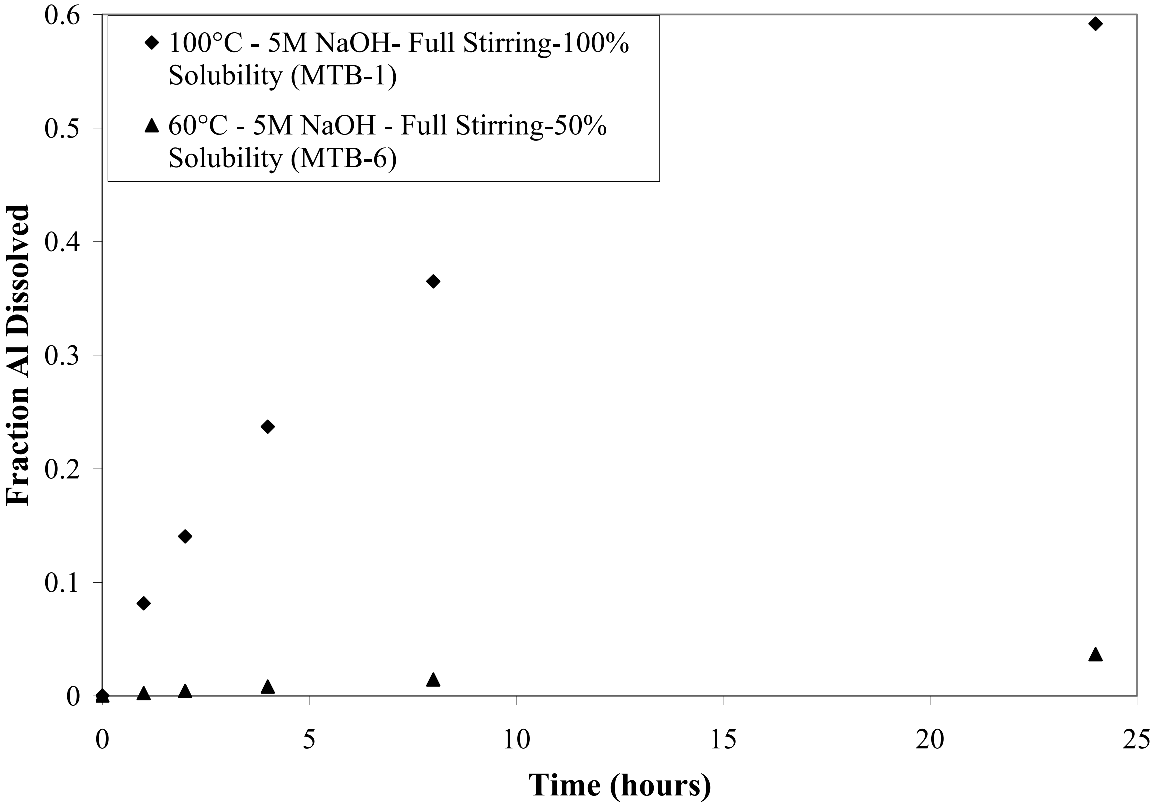


Figure 3.2. Effect of Temperature on Boehmite.

Figure 3.3 shows that the stir rate at 60°C for both 1 M and 5 M NaOH had a significant effect on the boehmite dissolution (MTB-5 through MTB-8). The data also indicate that the hydroxide concentration has a significant effect. In all cases, an increase in dissolution was observed with increased stirring rate (mixing).

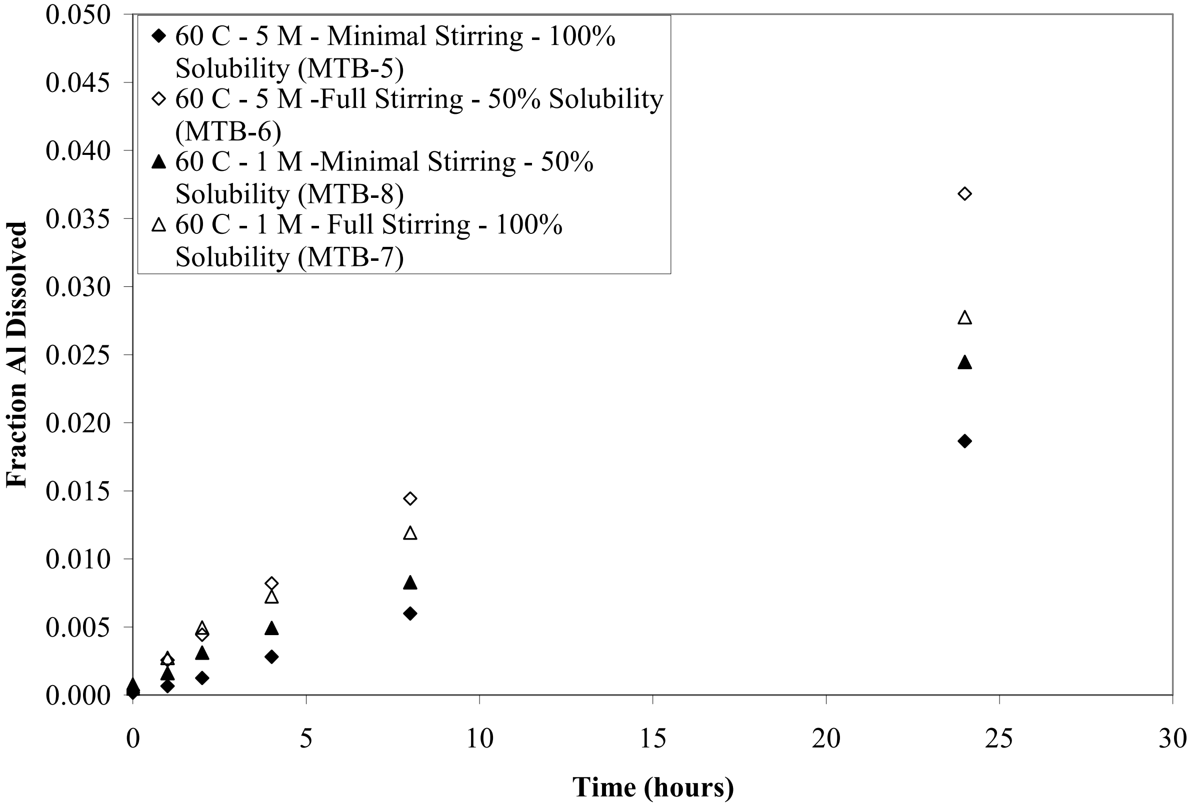


Figure 3.3. Effect of Mixing, Hydroxide Concentration, and Solubility Approach on Boehmite Dissolution in 1 M (MTB-7 and MTB-8) and 5 M NaOH (MTB-5 and MTB-6) at 60°C

4.0 Aluminate and Anion Effects on Boehmite Dissolution Rate

This section continues the task of assessing the impact of process conditions on leaching performance. In particular, this section assesses the effect of anions on the kinetics of boehmite dissolution and addresses objective 1 in Table S.2. This work was carried out under a separate test plan (TP-RPP-WTP-509, Rev. 0). Two sets of tests were completed: 1) one that examined the effect of the aluminate anion on the rate of boehmite dissolution and 2) another that determined if the presence of secondary anions typical of Hanford tank wastes (carbonate, free-hydroxide, nitrate, nitrite, oxalate, phosphate and sulfate) affect boehmite dissolution rate. Both sets of tests provided insight into how composition variations in the suspending phase impact the effectiveness of dissolution processes. In addition, the aluminate anion studies provided information on the consequences of gibbsite in the waste due to the expected fast dissolution of gibbsite relative to boehmite.

These boehmite dissolution tests were performed at 100°C in 800 grams of varying concentrations of NaOH solutions with 1-wt% boehmite solids (8 g boehmite per 800 g NaOH solution). These experiments were performed the same as the previous ones described in Section 2.5. They were fully mixed at 120 rpm, and samples were taken at 1, 2, 4, 8, and 24 hours and analyzed for Al, Na, free OH^-, and anions using ICP-AES, potentiometric titration, and ion chromatography (IC).

Table 4.1 outlines the test matrix used to determine the aluminate and anion concentration effect on the boehmite dissolution kinetics. These tests were performed in a full factorial design with triplicate points at each of the two corner points to provide an estimate of the experimental error. The variables in this matrix were the initial aluminate ion concentration (compared to boehmite solubility at 100°C), the caustic concentration, and the presence or absence of added anions. The added anions were added in bulk as sodium salts to the base solution in the ratios shown in Table 4.2 to examine the effects of the anion concentrations on the boehmite dissolution rate for Tests 5 to 8, 11, and 12 listed in Table 4.1. These salts were added to bring the total sodium concentration from the salts to 2 M. These anion ratios were selected to resemble supernate from a blend of REDOX sludge wastes. Table 4.3 shows the actual amounts of the components used in these tests.

Table 4.1. Test Matrix for Aluminate Ion Tests

Test No.	Aluminate Ion Concentration (% of boehmite solubility at 100°C – defined by Panias [2001]) (% of solubility)	(M)	Caustic Concentration (M)	Added Anions
1	75	0.784	5	No
2	50	0.522	5	No
3,9,10	75	0.149	1	No
4	50	0.100	1	No
5	75	0.784	5	Yes
6,11,12	50	0.522	5	Yes
7	75	0.149	1	Yes
8	50	0.100	1	Yes

Table 4.2. Molar Concentration of Sodium Salts Used for Anion Tests

Component	Chemical Formula	Concentration (M)	Sodium Contribution (M)
Sodium phosphate	Na_3PO_4-$12H_2O$	0.008	0.024
Sodium sulfate	Na_2SO_4	0.005	0.010
Sodium carbonate	Na_2CO_3	0.36	0.72
Sodium oxalate	$Na_2C_2O_4$	0.01	0.02
Sodium nitrite	$NaNO_2$	0.33	0.33
Sodium nitrate	$NaNO_3$	0.90	0.90
Total	----	----	2.00

Table 4.3. Aluminate and Anion Test Matrix Components Used

Test ID	Boehmite (g)	NaOH (g)	$NaAlO_2$ (g)	$Na_2C_2O_4$ (g)	Na_3PO_4 (g)	Na_2SO_4 (g)	Na_2CO_3 (g)	$NaNO_3$ (g)	$NaNO_2$ (g)	OH^- Conc. Added M
ATM-1	9.62	919.25	48.20	0	0	0	0	0	0	5
ATM-2	9.46	919.25	32.09	0	0	0	0	0	0	5
ATM-3	7.91	787.25	9.16	0	0	0	0	0	0	1
ATM-4	7.88	787.25	6.15	0	0	0	0	0	0	1
ATM-5	10.69	919.25	48.20	1.01	2.28	0.53	28.62	57.37	17.08	5
ATM-6	10.53	919.25	32.09	1.01	2.28	0.53	28.62	57.37	17.08	5
ATM-7	8.98	787.25	9.16	1.01	2.28	0.53	28.62	57.37	17.08	1
ATM-8	8.95	787.25	6.15	1.01	2.28	0.53	28.62	57.37	17.08	1
ATM-9	7.91	787.25	9.16	0	0	0	0	0	0	1
ATM-10	7.91	787.25	9.16	0	0	0	0	0	0	1
ATM-11	10.53	919.25	32.09	1.01	2.28	0.53	28.62	57.37	17.08	5
ATM-12	10.53	919.25	32.09	1.01	2.28	0.53	28.62	57.37	17.08	5

4.1 Aluminate Effect on Boehmite Dissolution

These aluminate anion studies provided information on the effect of gibbsite in the waste due to the expected fast dissolution of gibbsite relative to boehmite. The aluminate simulated dissolved gibbsite in the supernate. Aluminate was found to have a significant impact on the boehmite dissolution in 5 M NaOH and reduced the dissolution rate for boehmite by a factor of about 4.3 as shown in Figure 4.1. Test conditions for the baseline test (BSM-6) are given in Table 5.2 and only contained boehmite and sodium oxalate in 5 M NaOH with no initial aluminate. An increase of aluminate concentration from 50% to 75% boehmite solubility caused a decrease of 25 ± 7 mg/kg hr in the average rate of boehmite dissolution. Table 4.4 summarizes the aluminum dissolution rate with added aluminate ions during the different tests (ATM-1, 2, 3, 4, 9, 10). The actual concentrations versus time are given in Appendix A for each test. The boehmite dissolution rate is affected more by the hydroxide concentration than by the aluminate concentration as is shown by this table. However, a drop in the boehmite dissolution rate is also observed with increased aluminate in the 1 M NaOH.

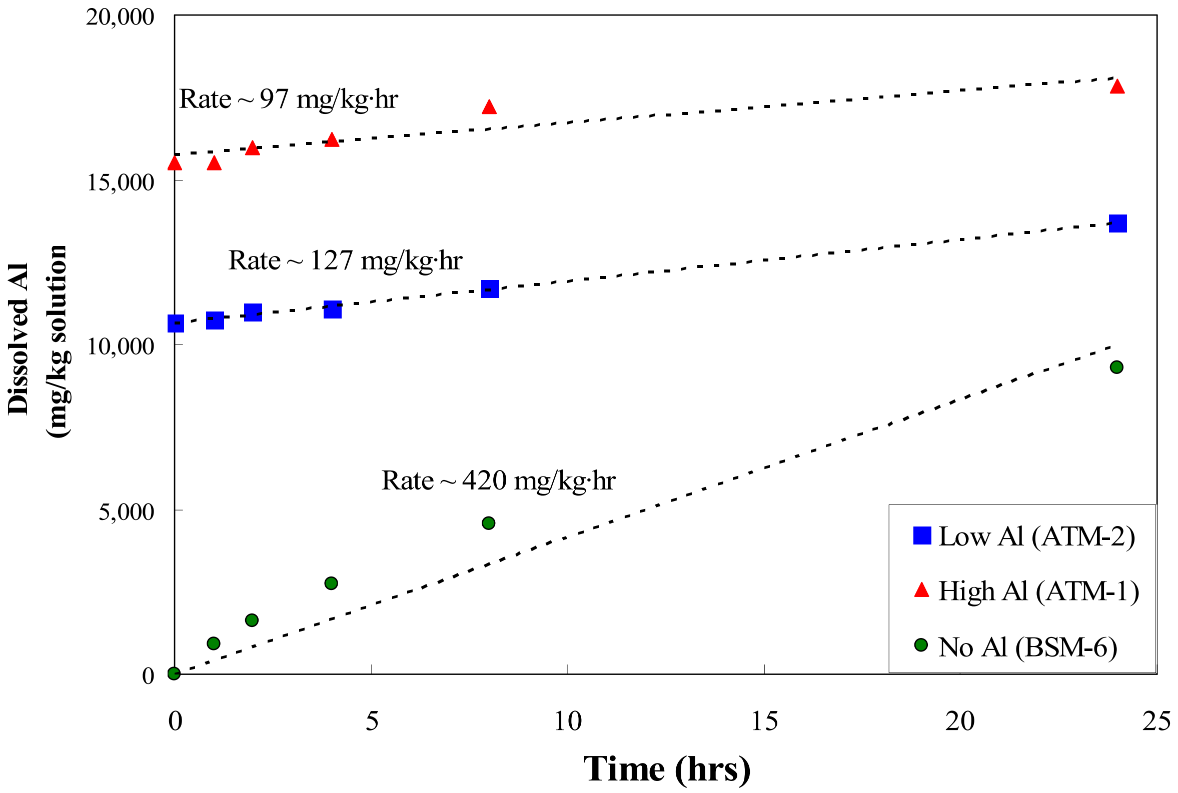


Figure 4.1. Aluminate Effect on Boehmite Dissolution (BSM-6, ATM-1 and ATM-2) in 5 M NaOH

Table 4.4. Aluminum Dissolution Rates with Aluminate Added

Test ID	Aluminate Level [(a)]	OH^- Concentration	Rate [mg/kg·hr]	Error [mg/kg·hr]
ATM-4	50%	1 M	64	11
ATM-3,9,10	75%	1 M	39	6
ATM-2	50%	5 M	127	11
ATM-1	75%	5 M	97	11

(a) Percent boehmite solubility

4.2 Anion Effect on Boehmite Dissolution

These tests were used to determine if the presence of secondary anions typical of Hanford tank wastes (carbonate, free-hydroxide, nitrate, nitrite, oxalate, phosphate, and sulfate) effect boehmite dissolution. This set of tests provided insight into how composition variations in the suspending phase impact the effectiveness of the dissolution process. Figure 4.2 shows the effect of the anions on the boehmite dissolution rate in 5 M NaOH. Anion addition slowed the rate of boehmite dissolution with the effect being approximately twice as great in the 5 M NaOH as in the 1 M NaOH as shown in Table 4.5. The actual concentrations versus time are shown in Appendix A. The boehmite dissolution rate is affected more by the hydroxide and aluminate concentrations than by the anion concentration as is shown by this table. However, a drop in boehmite dissolution rate is also observed with anion addition in the 1 M NaOH. Note that no significant impact of anion addition was observed in testing with actual waste with only nitrate as the added anion.

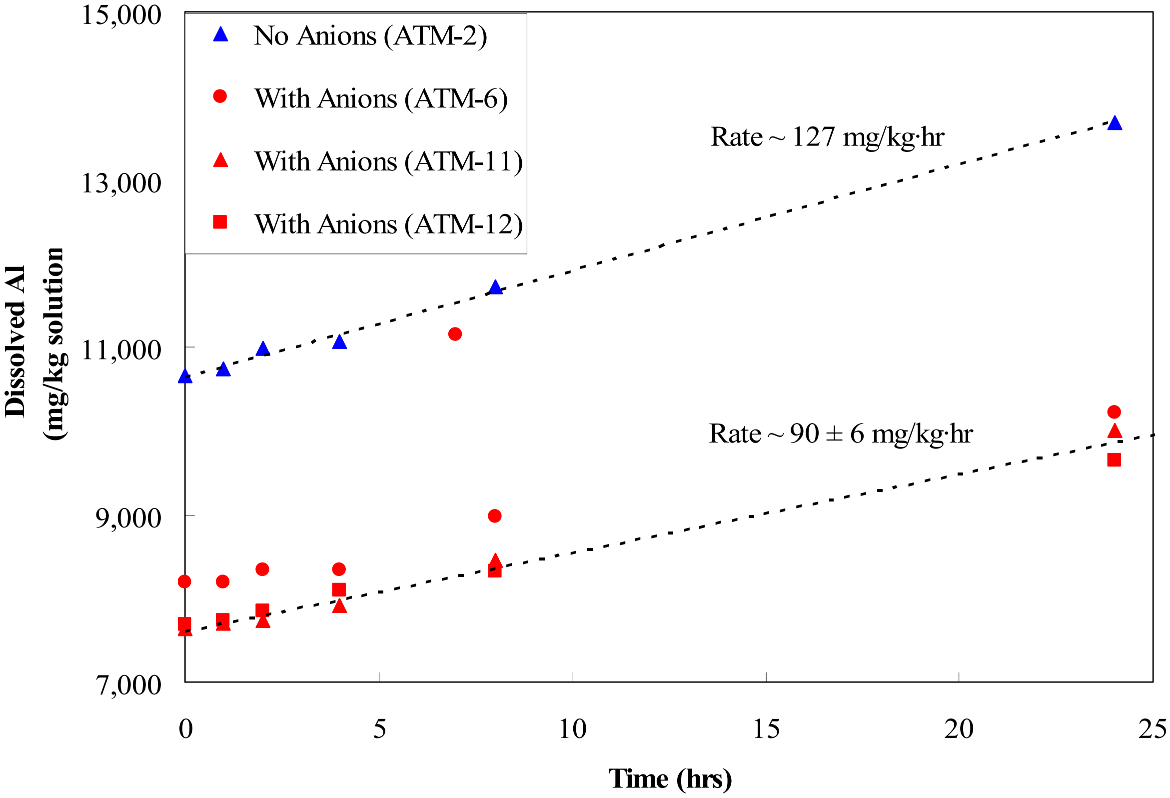


Figure 4.2. Anion Effect on Boehmite Dissolution (ATM-2 and ATM-6,11,12) in 5 M NaOH

Table 4.5. Aluminum Dissolution Rates with Anions Added

Test ID	Aluminate Level [(a)]	OH^- Concentration	Rate [mg/kg·hr]	Error [mg/kg·hr]
ATM-8	50%	1 M	48	11
ATM-7	75%	1 M	27	11
ATM-6,11,12	50%	5 M	90	6
ATM-5	75%	5 M	65	11
(a) Percent boehmite solubility				

5.0 Testing of Boehmite in a Blended Simulant

As indicated in Section 1, the third task for simulant development was to assess the impact of blending the boehmite simulant component with the balance of the simulant components. The B3 boehmite simulant that was chosen to be the "component" simulant was blended in a specified ratio with the other chosen "component" simulants for gibbsite and ultrafiltration. The blended simulant tests used a filtration simulant that contained a blend of solids that are characteristic of actual waste, including boehmite, gibbsite, oxalate, and iron-rich sludge. The blended simulant was then tested over a subset of the matrix of conditions in which the component simulants had been tested previously with different combinations of components present in each test. These results show how the other components interact with the boehmite and whether they hinder, help, or have no effect on its dissolution.

Table 5.1 provides the composition of the supernate that was used in these tests. This supernate composition was chosen to provide continuity in boehmite leaching experiments. It is based on a 3.2-M Na concentration, which resembles supernate from a blend of REDOX sludge wastes.(a)

Table 5.2 provides the sequence of tests performed in this test matrix. Tests 1 through 13 were performed at 100°C. Tests 1 through 6 along with tests 8 and 9 provide a statistical design to evaluate the effect of gibbsite and filtration components on the dissolution of boehmite. Tests 7, 10, 11, 12, and 13 examine the effect of mixing energy on the dissolution of alumina (both boehmite and gibbsite). Tests 14 through 19 were performed at 45°C and provide equilibrium measurements of boehmite solubility, which is important to evaluating boehmite dissolution kinetics. Tests 14 through 16 also examine the mixing energy effect, and Tests 17 through 19 examine the supernate effect on the dissolution of alumina. The tests examining only gibbsite dissolution (BSM-3 and BSM-4) are not discussed in this report as they are discussed in WTP-RPT-176.(b)

Table 5.1. Supernate Composition at 3.2 M Na

Component	Chemical Formula	Concentration (M)
Sodium phosphate	Na_3PO_4-$12H_2O$	0.013
Sodium sulfate	Na_2SO_4	0.008
Sodium carbonate	Na_2CO_3	0.576
Sodium oxalate	$Na_2C_2O_4$	0.016
Sodium nitrite	$NaNO_2$	0.528
Sodium nitrate	$NaNO_3$	1.44

(a) SK Fiskum et al. Feb. 2008. *Characterization and Leach Testing for REDOX Sludge and S-Saltcake Actual Waste Sample Composites*. WTP-RPT-157, Rev. A.

(b) RL Russell et al. Dec. 2008. *Development and Characterization of Gibbsite Component Simulant*. WTP-RPT-176, Rev. A.

Table 5.2. Blended Component Test Matrix

Test	Sodium Oxalate (g)	Gibbsite (g)	Boehmite (g)	Sludge Simulant (g)	19M NaOH (g)	Condensate (DIW) (g)	Supernate (g)	Mixing Speed (rpm)	[OH] Molarity	Na Molarity	Notes
BSM-1	6.50	22.73	22.73	12.99	200.27	274.96	259.82	140	5.8	7.1	Full factorial for Gibbsite/Boehmite/Sludge Solids—oxalate as secondary variable
BSM-8	6.50	22.73	22.73	12.99	200.27	274.96	259.82	140	5.8	7.1	Replicate of BSM-1
BSM-9	6.50	22.73	22.73	12.99	200.27	274.96	259.82	140	5.8	7.1	Replicate of BSM-1
BSM-2	0.00	22.73	22.73	0.00	200.27	274.96	259.82	140	5.8	7.1	Full factorial for Gibbsite/Boehmite/Sludge Solids—oxalate as secondary variable
BSM-3	0.00	22.73	0.00	12.99	200.27	274.96	259.82	140	5.8	7.1	Full factorial for Gibbsite/Boehmite/Sludge Solids—oxalate as secondary variable
BSM-4	6.50	22.73	0.00	0.00	200.27	274.96	259.82	140	5.8	7.1	Full factorial for Gibbsite/Boehmite/Sludge Solids—oxalate as secondary variable
BSM-5	0.00	0.00	22.73	12.99	200.27	274.96	259.82	140	5.8	7.1	Full factorial for Gibbsite/Boehmite/Sludge Solids—oxalate as secondary variable
BSM-6	6.50	0.00	22.73	0.00	200.27	274.96	259.82	140	5.8	7.1	Full factorial for Gibbsite/Boehmite/Sludge Solids—oxalate as secondary variable
BSM-7	6.50	22.73	22.73	12.99	200.27	274.96	259.82	280	5.8	7.1	Mixing Energy—Compare Tests 1, 10 and 11
BSM-10	6.50	22.73	22.73	12.99	200.27	274.96	259.82	90	5.8	7.1	Mixing Energy—Compare Tests 1 and 7
BSM-11	6.50	22.73	22.73	12.99	200.27	274.96	259.82	50	5.8	7.1	Mixing Energy—Compare Tests 6 and 12
BSM-12	6.50	0.00	22.73	0.00	200.27	274.96	259.82	90	5.8	7.1	Mixing Energy—Compare Tests 6 and 11
BSM-13	6.50	0.00	22.73	0.00	200.27	274.96	259.82	50	5.8	7.1	Test of 45C condition and mixing energy—Compare to Tests 1, 14, and 15
BSM-14	7.02	24.56	24.56	14.04	152.07	297.06	280.70	140	4.5	5.9	Test of 45C condition and mixing energy—Compare to Tests 1, 13, and 15
BSM-15	7.02	24.56	24.56	14.04	152.07	297.06	280.70	90	4.5	5.9	Test of 45C condition and mixing energy—Compare to Tests 1, 13, and 14
BSM-16	7.02	24.56	24.56	14.04	152.07	577.76	280.70	50	4.5	5.9	Test of 45C condition and supernate effect—compare with test 13
BSM-17	7.02	24.56	24.56	14.04	152.07	577.76	0.00	140	4.5	4.5	Test of 45C condition and supernate effect—compare with test 14
BSM-18	7.02	24.56	24.56	14.04	152.07	577.76	0.00	90	4.5	4.5	Test of 45C condition and supernate effect—compare with test 15
BSM-19	7.02	24.56	24.56	14.04	152.07	577.76	0.00	50	4.5	4.5	Test of 45C condition and supernate effect—compare with test 16

The impact of stirring speed on the boehmite dissolution is shown in Figure 5.1 with gibbsite present and in Figure 5.2 without gibbsite present. This indicates that as long as there is reasonable stirring in the slurry, boehmite dissolution will occur, and the amount of stirring is not a controlling variable. Note that 90 rpm consistently gave higher dissolution than at faster stirring rates. At this time no rationale for this behavior has been found.

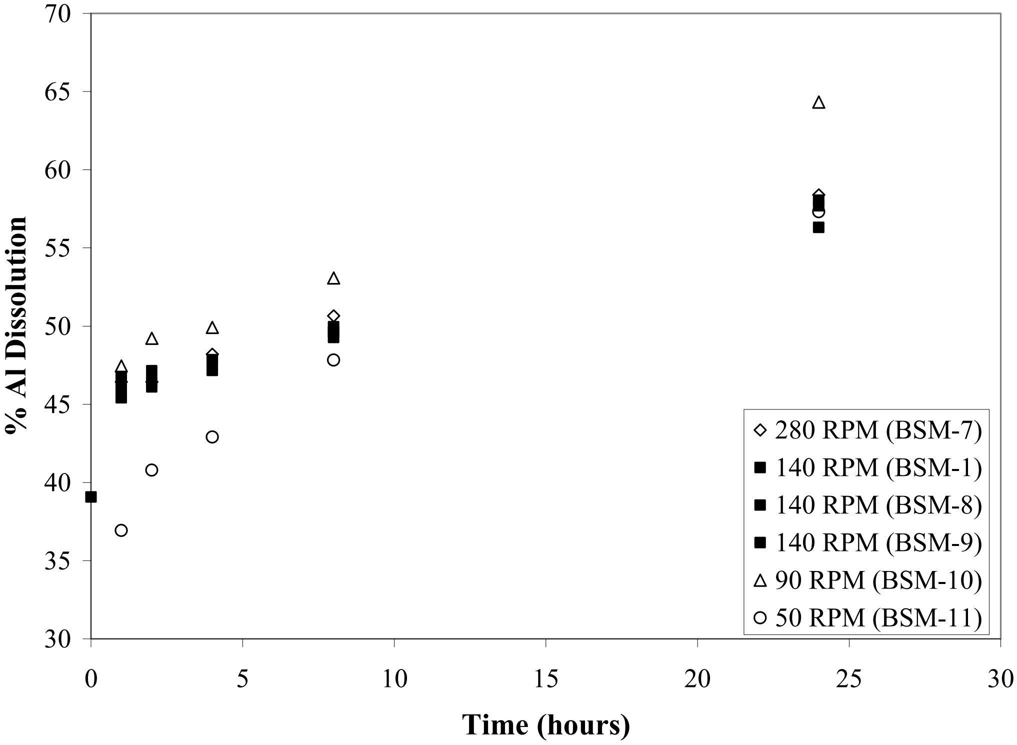


Figure 5.1. Impact of Stirring Speed on Boehmite Dissolution with Gibbsite Present

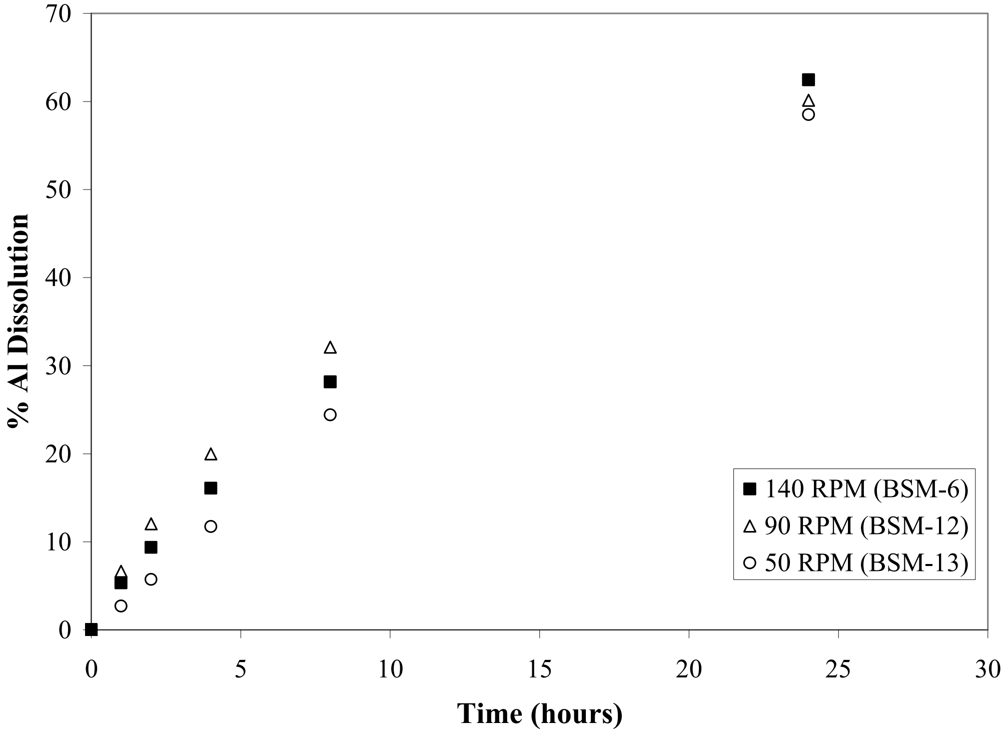


Figure 5.2. Impact of Stirring Speed on Boehmite Dissolution without Gibbsite Present

Figure 5.3 shows the effect of supernate on the boehmite dissolution equilibrium. Supernate appears to increase boehmite dissolution at any given time.

The effect of each simulant component on the boehmite dissolution is shown in Figure 5.4. Note that for the tests with gibbsite present, the gibbsite dissolution was subtracted based on the results from tests BSM-3 and BSM-4 (gibbsite only Al source). This boehmite in caustic without anything else added yields approximately 80% dissolution. These results indicate that gibbsite has a significant effect on the dissolution of boehmite. Sludge has a slight effect, and oxalate did not appear to have any significant effect on the boehmite dissolution, however.

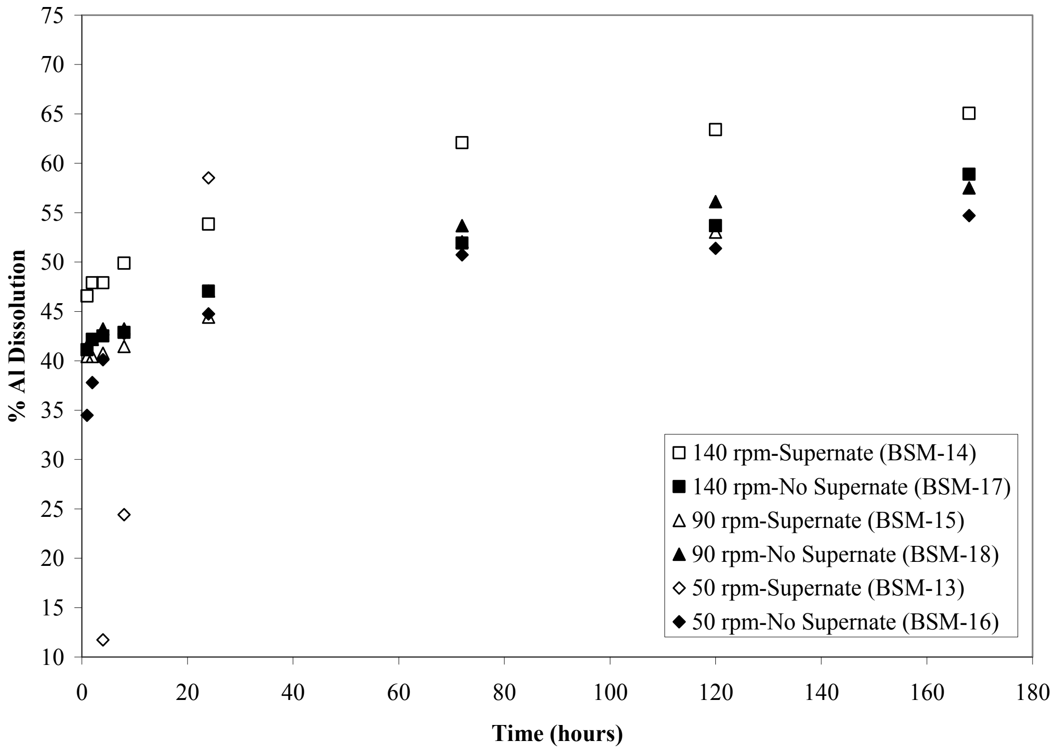


Figure 5.3. Effect of Supernate on the Boehmite Dissolution Equilibrium

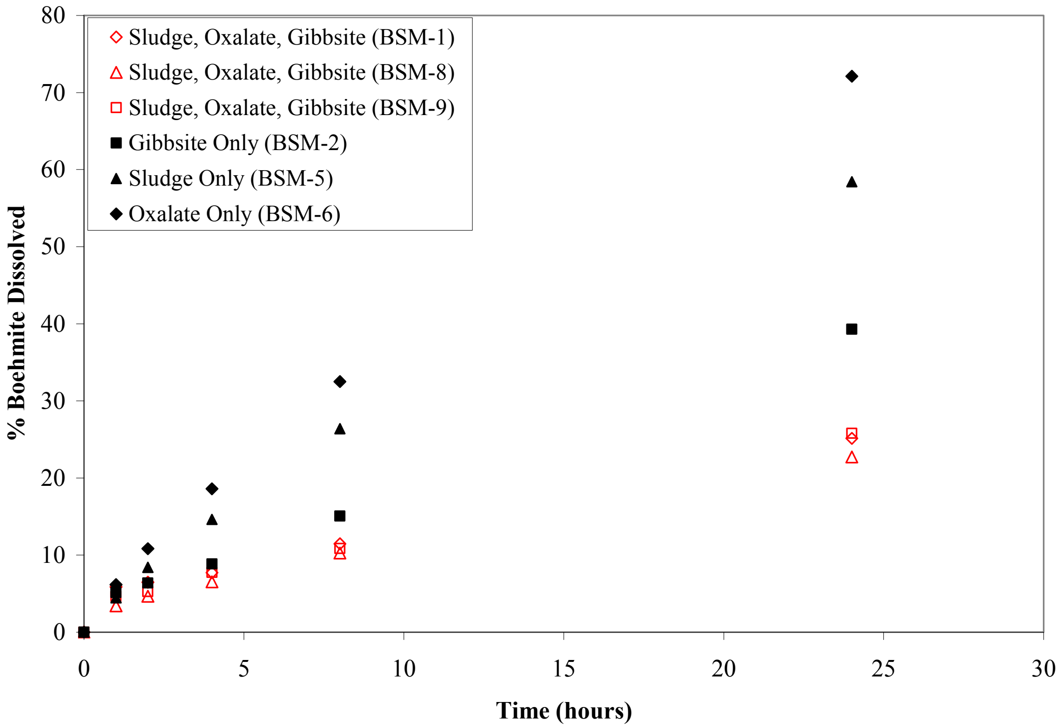


Figure 5.4. Effect of Each Simulant Component on Boehmite Dissolution

Figure 5.5 shows the approach to boehmite dissolution equilibrium. Samples indicate that the boehmite solubility limit is greater than 20,000 mg/L Al under the tested conditions. Also note that there appears to be some dissolution up to 172 hours.

5.1 Summary

The important points learned from these tests are summarized below.

- As long as there is reasonable stirring in the slurry, boehmite dissolution will occur, and the amount of stirring is not a controlling variable.
- Supernate presence appears to increase boehmite dissolution at any given time.
- Gibbsite has a significant effect on the dissolution of boehmite.
- Sludge has a slight effect, and oxalate did not appear to have any significant effect on the boehmite dissolution.
- Samples indicate that the boehmite solubility limit is greater than 20,000 mg/L Al under the tested conditions and dissolution continues to occur up to 172 hours.

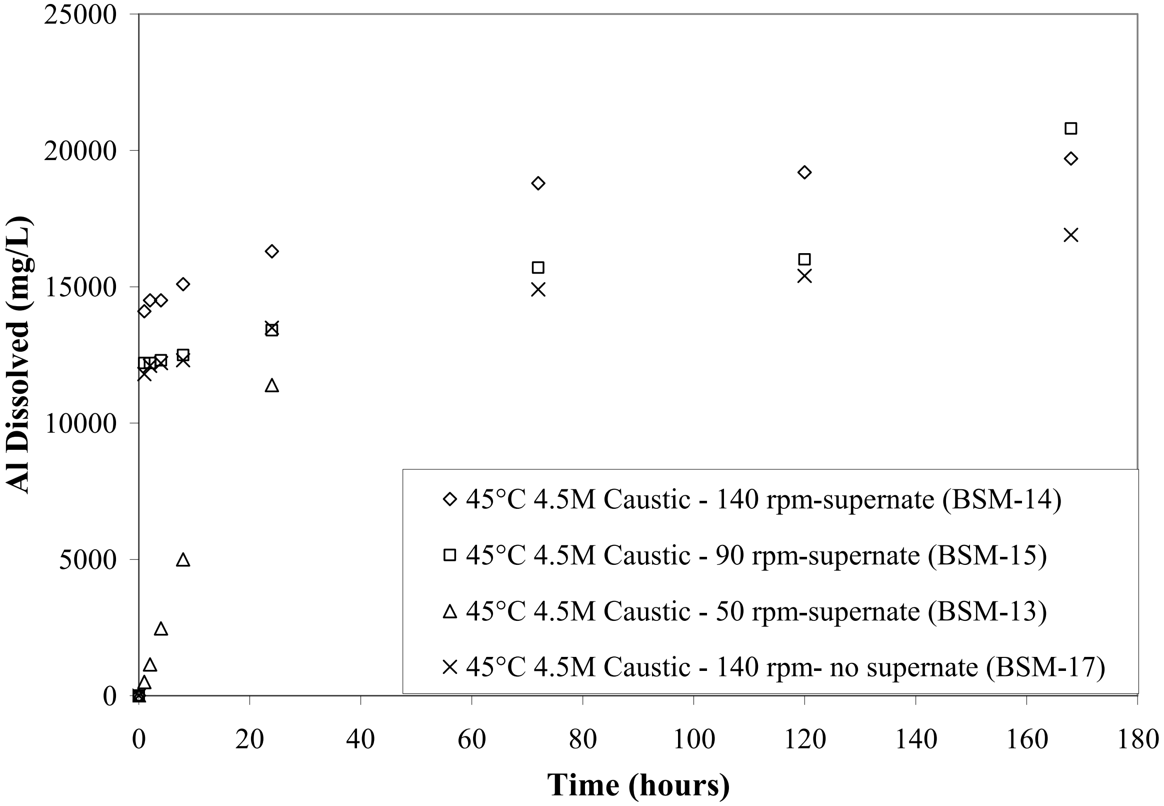


Figure 5.5. Approach to Boehmite Equilibrium at 45°C

6.0 Performance Testing of Blended Simulant

Section 5 assessed the impact of the blended simulant on leaching performance. The next step in the development of the complete simulant (not just the boehmite portion) was to assess the integrated performance of the blended simulant during leaching and filtration operations. This was carried out through a series of tests using the bench-scale crossflow ultrafiltration system. Note that all of the filtration performance data are presented in report WTP-RPT-183 while all of the leaching performance data are reported in this report. See Appendix E for a description of the crossflow ultrafiltration system.

Five crossflow ultrafiltration blended-matrix tests were performed with the compositions shown in Table 6.1. These tests were broken into two blocks of tests. The first block consisted of the first two tests under the initial planned leaching conditions. The first test was CBM-1 (original sample identification labeled as CBM) in which the slurry was dewatered and washed at 45°C. The next test (CBM-2—original sample identification labeled as CBM25) essentially repeated CBM-1 except the slurry was dewatered and washed after caustic leaching at 25°C instead of 45°C to determine the effect of temperature on the filter flux. As far as leaching, these tests were performed identically, and the process steps are compared for these tests in Table 6.2. The objective of these tests was to verify that the results obtained in test BSM-1 were replicated in an integrated crossflow ultrafiltration test. See Table 5.2 for BSM-1 conditions and Figure 5.4 for BSM-1 boehmite dissolution curve.

Table 6.1. Filtration Simulant UDS Composition in Grams per kg of UDS

Component	CBM-1 (g/kg UDS)	CBM-2 (g/kg UDS)	CBM-3 (g/kg UDS)	CBM-4 (g/kg UDS)	CBM-5 (g/kg UDS)
Fe-rich sludge	182	182	187	187	187
Gibbsite	345	345	355	355	355
Boehmite	345	345	355	355	355
Sodium oxalate	100	100	103	103	103
CrOOH slurry	27	27	*	*	*
AFA Added?	No	No	No	Yes	Yes
Fe-rich sludge source	PNNL [(a)]	PNNL [(a)]	Optima [(b)]	Noah [(c)]	Noah [(c)]

* The CrOOH slurry was added after the caustic leach in these simulants.
(a) Pacific Northwest National Laboratory, Richland, Washington.
(b) Optima Chemical, Douglas, Georgia.
(c) Noah Technologies Corporation, San Antonio, Texas.

Table 6.2. Process Steps for CBM-1 and CBM-2

Process Step	CBM-1	CBM-2
Initial Dewatering Temp. (°C)	45	25
Caustic Leaching Temp. (°C)	100	100
Caustic Leach Dewater Temp. (°C)	45	25
Incremental Caustic Leach Wash Steps	6	6
Caustic Washing Temperature (°C)	45	25
Oxidative Leaching Temp. (°C)	45	45
Incremental Oxidative Leach Wash Steps	4	4
Oxidative Washing Temperature (°C)	45	25

The next block of three tests (CBM-3, CBM-4, and CBM-5) were performed to determine the ability of the simulant-producing vendor to produce the simulant at various production quantities. CBM-3 was performed from a 25-L simulant batch, CBM-4 was performed from a 15-gallon simulant batch, and CBM-5 was performed from a 250-gallon simulant batch. The objective of these tests was to assess whether there were any significant differences in the leaching behavior from these three different scales of simulant production. These tests were performed similar to CBM-2 except a greater amount of caustic was used, and the chromium slurry was introduced after the caustic leach. Also, constant wash concentrations were used in these tests. Table 6.3 compares the conditions used for these three tests. This report will only present the chemical results from the leaching processes because the filtration results were presented in WTP-RPT-183.[(a)]

Table 6.3. Process Steps for CBM-3, CBM-4, and CBM-5

Process Step	CBM-3	CBM-4	CBM-5
Initial Dewatering Temp. (°C)	25	25	25
Caustic Leaching Temp. (°C)	100	100	100
Caustic Leach Dewater Temp. (°C)	25	25	25
Caustic Leach Wash Steps	16	16	16
Caustic Leach Wash Conc. (M)	0.01	0.01	0.01
Caustic Washing Temperature (°C)	25	25	25
CrOOH Slurry Addition Amount (kg)	1.73	1.73	1.73
CrOOH Slurry Wash (0.01M NaOH) (L)	15	15	15
Oxidative Leaching Temp. (°C)	25	25	25
Oxidative Leach Wash Steps	13	13	13
Oxidative Leach Wash Conc. (M)	0.01	0.01	0.01
Oxidative Washing Temperature (°C)	25	25	25

A 5-wt% undissolved solids (UDS) simulant slurry consisting of 34.6 wt% gibbsite, 34.6 wt% boehmite, 18.1 wt% filtration simulant slurry, and 10-wt% sodium oxalate was used for these tests. The slurry

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(a) Russell et al. Dec. 2008. *Development and Demonstration of Ultrafiltration Simulants*. WTP-RPT-183, Rev. A.

simulant was dewatered to the crossflow ultrafiltration system's minimum operating volume at a predicted concentration of ~20 wt% UDS. Next, the slurry simulant was removed from the crossflow ultrafiltration system to be caustic leached in the slurry reservoir tank. NaOH was blended with the slurry, and the volume of the addition was established to include the volume of water representing the leach solution volume increase that was predicted to occur from heating with steam injection in the UFP-2 vessel of the PEP. The leach solution was heated to 100°C over a 5.3-hour interval. Samples were taken at 0, 2, and 4 hours.

The slurry was then held at 100°C for 12 hours. Samples were taken every 2 hours during this time. Afterwards, the slurry was allowed to cool to room temperature over a 12-hour interval with samples taken every 2 hours again. At this point, the leached slurry in the slurry reservoir tank was allowed to enter the piping of the crossflow ultrafiltration system, and it was again dewatered to the crossflow ultrafiltration system's minimum operating volume. Then wash solutions (whose concentration and volume varied with test being performed) were added to the leached slurry and dewatered. Tests CBM-1 and CBM-2 used incremental wash solution concentrations as shown in Table 6.4. Tests CBM-3, CBM-4, and CBM-5 used constant concentration wash solutions of 0.01 M NaOH.

Table 6.4. Caustic Wash Concentrations for CBM-1 and CBM-2

	Wash Volume (L)	NaOH Conc. of Wash (M)
Wash 1	4.8	1.57
Wash 2	4.8	0.68
Wash 3	4.8	0.26
Wash 4	4.8	0.08
Wash 5	4.8	0.03
Wash 6	4.8	0.01

The slurry wash volume was prescribed by material balance. Once the prescribed quantity of wash water had been added and removed from the crossflow ultrafiltration system, the slurry was prepared for oxidative leaching in the slurry reservoir tank. At this point, a solution of 1 M sodium permanganate was added to the simulant slurry. The volume of sodium permanganate added was calculated to achieve a 1:1 molar ratio of Mn to the predicted quantity of Cr in the simulant solids. After the solution was added to the simulant slurry, it was mixed for 6 hours at room temperature (~22°C). Samples were taken at 0.5, 1, 2, 4, and 6 hours. After 6 hours, the oxidative leached slurry was dewatered and then washed with 0.01 M NaOH and dewatered again to achieve 90% removal of the solubilized Cr. CBM-1 and CBM-2 used 4 washes of 4.5 L 0.01 M NaOH. CBM-3, CBM-4, and CBM-5 used 13 washes of 700 mL 0.01 M NaOH.

Samples were collected throughout the tests to track the dissolution of the slurry components and were analyzed by ICP-AES, IC, and hydroxide titration.

6.1 Comparison of Tests CBM-1 and CBM-2 with BSM-1

With respect to leaching, the objectives of these two crossflow ultrafiltration tests (CBM-1 and CBM-2) were to

- assess the leaching process performance and reproducibility in the crossflow ultrafiltration system
- verify that the results obtained in test BSM-1 were replicated in an integrated crossflow ultrafiltration test that involved leaching and filtering on a larger scale.

6.1.1 Experimental Details

Nine liters of the 5-wt% UDS initial simulant slurry were placed in the crossflow ultrafiltration system simulant slurry reservoir tank. In the CBM-1 test, the simulant slurry was dewatered at 45°C to ~20 wt% UDS by adding 750 mL of simulant slurry to the tank for every 750 mL of supernate removed. The transmembrane pressure (TMP) and axial velocity (AV) were held constant at 40 psid (pounds per square inch) and 13 ft/s throughout the dewatering process. The caustic leach was performed at 100°C in the simulant slurry reservoir tank. After leaching, the low-solids slurry was dewatered at 45°C to ~17 wt% UDS at 40 psid TMP and 13 ft/s AV conditions. The leached slurry was washed with 4.8 L of varying NaOH concentration solution (see Table 6.4) at 45°C six times to remove the dissolved aluminum.

The simulant slurry was then oxidatively leached at 45°C for 6 hours with 1 M $KMnO_4$. The oxidatively leached simulant slurry was then washed at 45°C with four 4.8-L washes of 0.01 M NaOH to remove the dissolved chromium and dewatered to ~18 wt% UDS between each wash. The slurry was then dewatered to the target wt% UDS of ~20 wt%. The simulant slurry was then drained from the crossflow ultrafiltration system.

For CBM-2, the same test was performed except that the filtering operations and oxidative leaching were performed at 25°C instead of 45°C. Caustic leaching was performed at 100°C as in the previous test CBM-1. The oxidative leaching step in CBM-2 used $NaMnO_4$ (the preferred permanganate source) instead of $KMnO_4$ like CBM-1. No $NaMnO_4$ was available when it was needed during CBM-1, and therefore, $KMnO_4$ was used instead so that the test could proceed. The results indicated that there was no significant difference between them in the amount of chromium leached.

Slurry samples were analyzed at the Southwest Research Institute (SWRI)(a) with inductively coupled plasma (ICP)-mass spectroscopy (MS) and atomic emissions spectroscopy (AES) for cations. Anions measured with IC include nitrite (NO_2^-), nitrate (NO_3^{2-}), phosphate (PO_4^{3-}), sulfate (SO_4^{2-}), and oxalate ($C_2O_4^{2-}$). Hydroxide was measured by titration. Results of all analyses are listed in Appendix C. In general, the mass balance between elemental composition of the untreated simulant sludge and the elemental composition derived from the wash solutions agreed well.

The chemical leach factor was defined as the percentage difference in mass of a component in the waste after chemical leaching.

$$f_i = 1 - \frac{m_i^{final}}{m_i^{initial}} \tag{6.1}$$

where f_i was the leach factor for component *i*, $m_i^{initial}$ was the initial mass ratio of component *i* relative to Fe, and m_i^{final} was the final mass ratio of component *i* relative to Fe.

(a) Southwest Research Institute, 6220 Culebra Road, P.O. Drawer 28510. San Antonio, Texas 78228.

The boehmite leach factor is obtained by taking the Al leach factor and subtracting the gibbsite fraction and then dividing by the initial boehmite fraction.

$$f_b = \frac{f_{Al} - m_g}{m_b} \tag{6.2}$$

where f_b = boehmite leach factor
f_{Al} = Al leach factor
m_g = gibbsite mole fraction
m_b = boehmite mole fraction.

6.1.2 CBM-1 Results

Table G.1 of Appendix G provides a timeline for the CBM-1 test. All of the analytical results for this test are shown in Appendix C. This section will discuss primarily the Al, Cr, and Mn behavior during this leaching test. The aluminum concentration for test CBM-1 is shown in Figure 6.1 where there is a sharp initial increase in soluble Al, principally from the dissolution of gibbsite, and then a gradual increase due to the dissolution of boehmite during the caustic leaching. A decrease in Al content is observed during the washing of the leached simulant as the dissolved Al is being removed. No significant quantities of Al were dissolved after oxidative leaching and further washing.

In Figure 6.2, the chromium and manganese concentrations are shown. Following the permanganate addition (indicated by the increase in manganese concentration), there is a significant increase in the chromium concentration in the leachate due to its dissolution; however, some chromium was leached during the initial caustic leaching step also as shown by the increase of chromium concentration during the caustic leach.

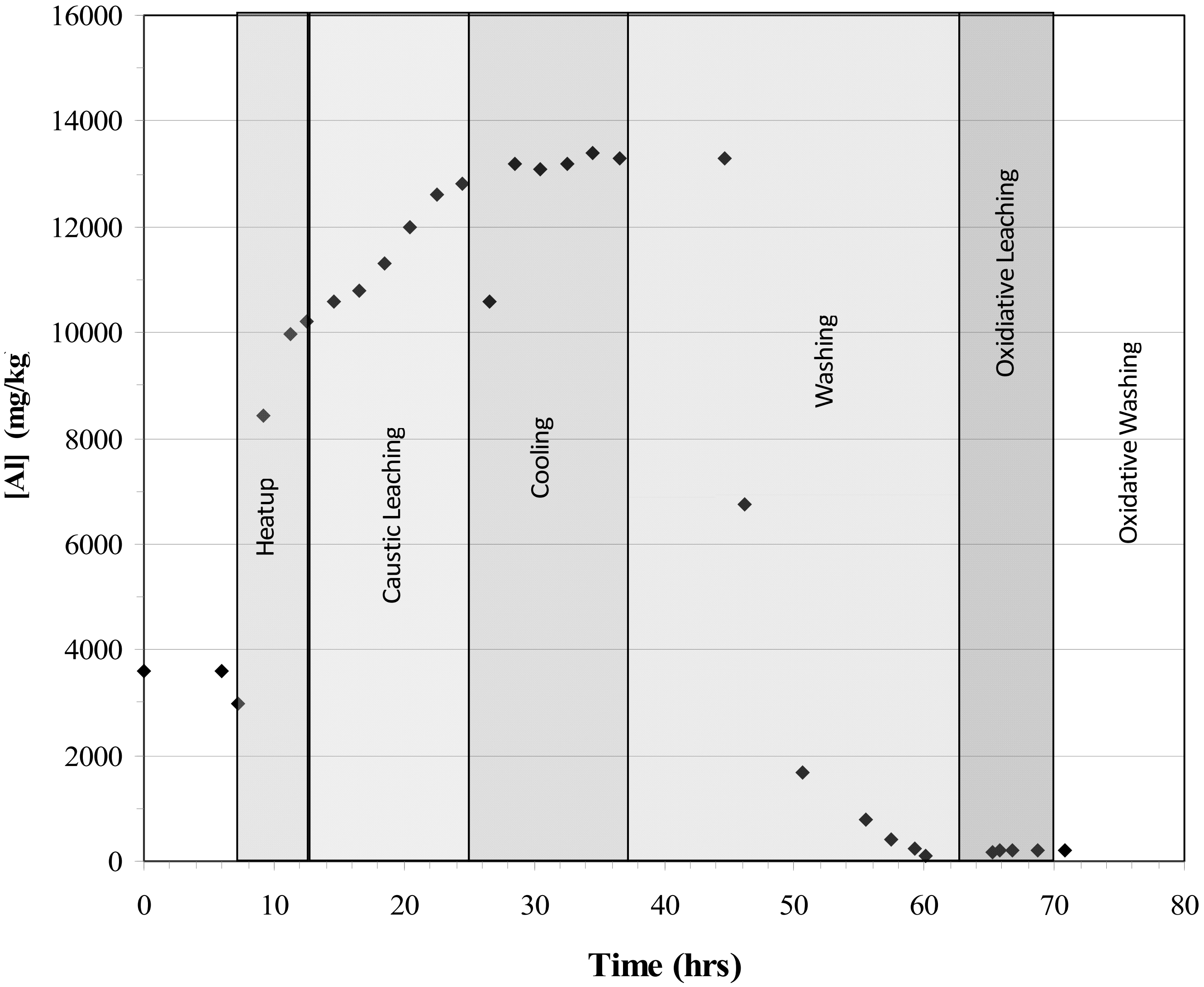

Figure 6.1. Concentration of Aluminum in Solution (mg/kg solution) in Test CBM-1

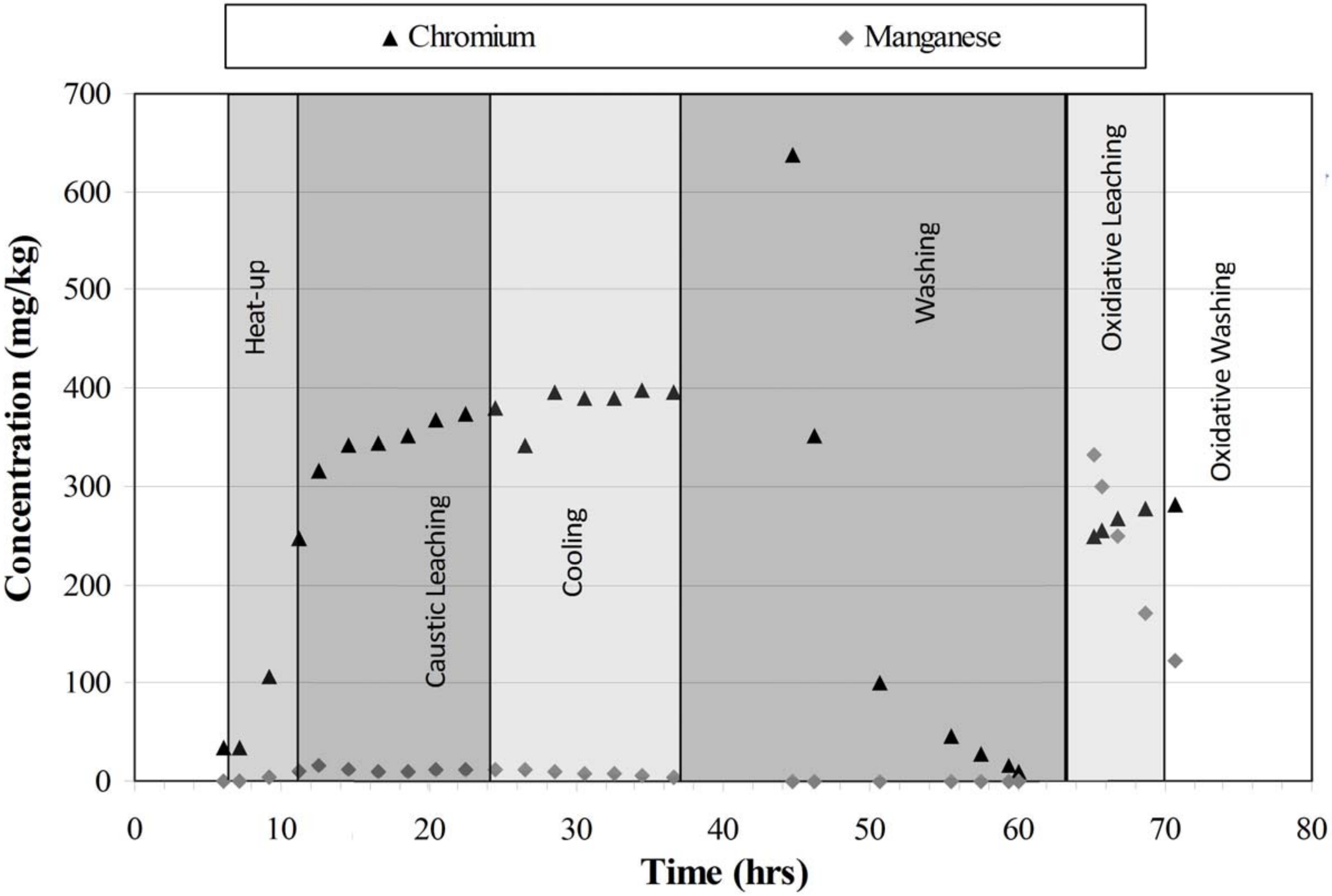

Figure 6.2. Dissolution of Chromium and Manganese in mg/kg solution in Test CBM-1

The slurry composition results are shown in Table 6.5. The levels of insoluble components such as calcium, iron, lead, manganese, neodymium, nickel, and zirconium were enriched significantly throughout the test.

Table 6.5. Analytical Slurry Analysis in CBM-1 (mg/kg solution)

	CBM-IN-SL	CBM-DW-SL	CBM-LC-SL	CBM-WS-SL	CBM-FSC-SL
Aluminum	13,700	53,800	67,500	42,500	85,400
Calcium	124	489	1,150	859	1,740
Chromium	365	1,420	1,240	503	504
Iron	3,630	15,000	36,600	27,400	55,100
Lead	176	722	1,580	1,190	2,380
Magnesium	36.9	150	353	269	545
Manganese	809	3,350	8,020	6,120	14,700
Neodymium	96.66	397	979	697	1,460
Nickel	120	485	1,200	905	1,800
Potassium	1,800	1,550	588	<135	478
Sodium	85,600	79,900	96,500	4,280	2,930
Zirconium	47.7	99	475	327	569
Nitrate	20,800	18,200	6,770	104	<109
Nitrite	6,490	5,840	1,970	<84.1	<109
Oxalate	3,460	13,700	2,700	1,040	138
Phosphate	291	220	<81.0	<84.1	<109
Sulfate	820	708	254	<84.1	<109

Notes: Values reported as “less than” are not quantifiable above background.
CBM-IN-SL = initial slurry composition
CBM-DW-SL = slurry composition following dewatering
CBM-LC-SL = slurry composition following the caustic leach
CBM-WS-SL= slurry composition following oxidative treatment
CBM-FSC-SL = final slurry composition.

The concentration ratios of various species in the solids are described in Table 6.6 for test CBM-1. The results indicate that chromium and aluminum are removed relative to iron and calcium.

Table 6.6. Ratio of Various Species in Solids from Test CBM-1

Ratio	Initial Conditions	At Start of Caustic Leaching Heat-up	After Caustic Leaching	Cool Down	End of Test
Al/Fe	2.8	3.4	1.5	1.5	1.5
Cr/Fe	0.09	0.09	0.02	0.02	0.01
Al/Ca	82	103	48	49	49
Cr/Ca	2.7	2.8	0.57	0.57	0.23

6.1.3 CBM-2 Results

Table G.2 of Appendix G provides a timeline for the process steps in test CBM-2. The concentration of aluminum in test CBM-2 is shown in Figure 6.3. See Appendix C for analytical data. There was a rapid increase in Al concentration during caustic leaching, principally from the dissolution of gibbsite, and then a gradual increase due to the dissolution of boehmite during the caustic leaching. Following washing, the Al content decreases, indicating that the dissolved Al was being removed, and then it essentially remains constant throughout the remaining steps of the test.

As shown in Figure 6.4, adding $NaMnO_4$ in the oxidative leaching step after 80 hours resulted in a peak in manganese concentration. Once the permanganate oxidant was added, chromium was solubilized through oxidation of Cr(III) to Cr(VI), and an immediate jump in the chromium concentration of the leachate was seen. The washing with progressively lower concentrations of caustic solution removed both aluminum and chromium from the leachate.

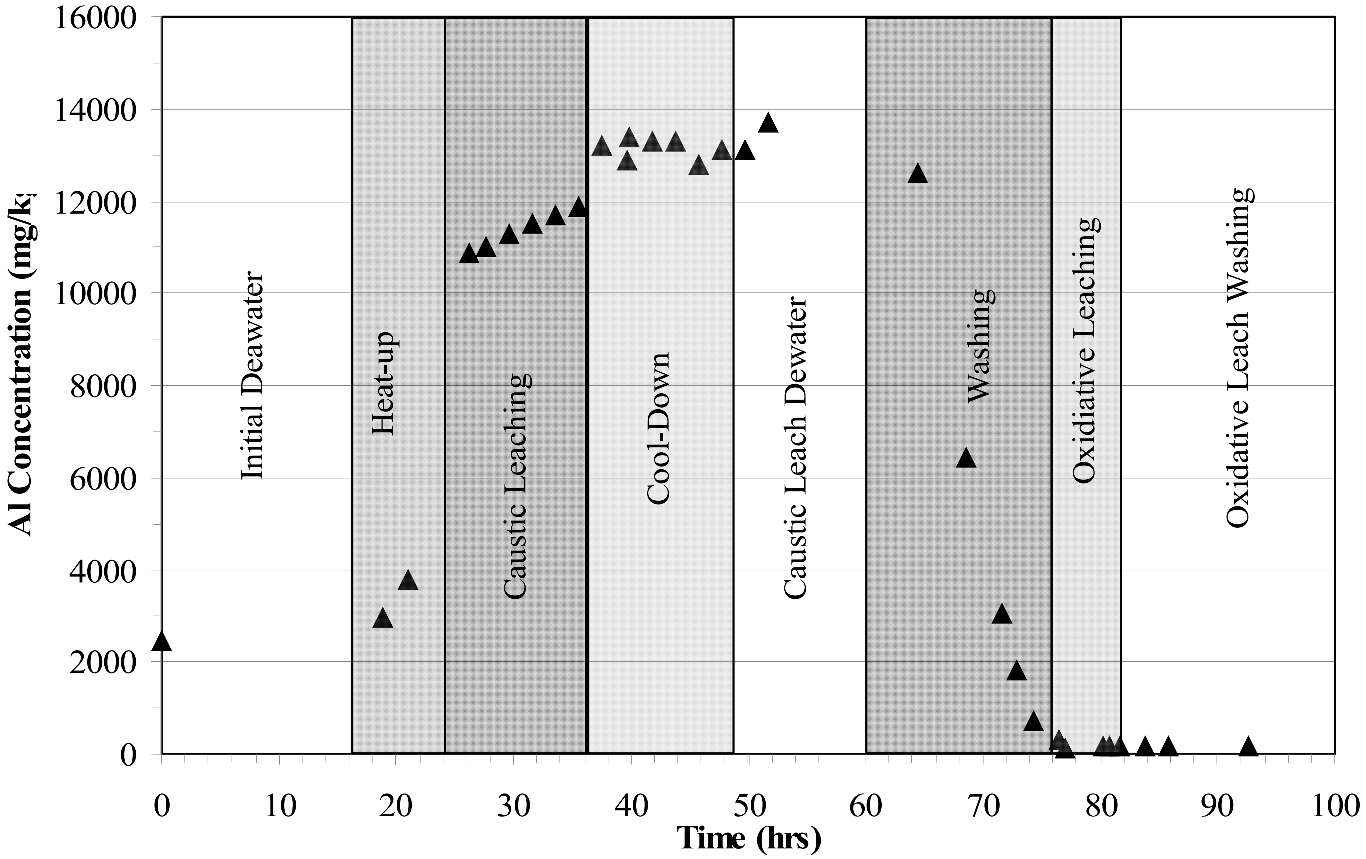

Figure 6.3. Dissolution of Aluminum in mg/kg Solution in Test CBM-2

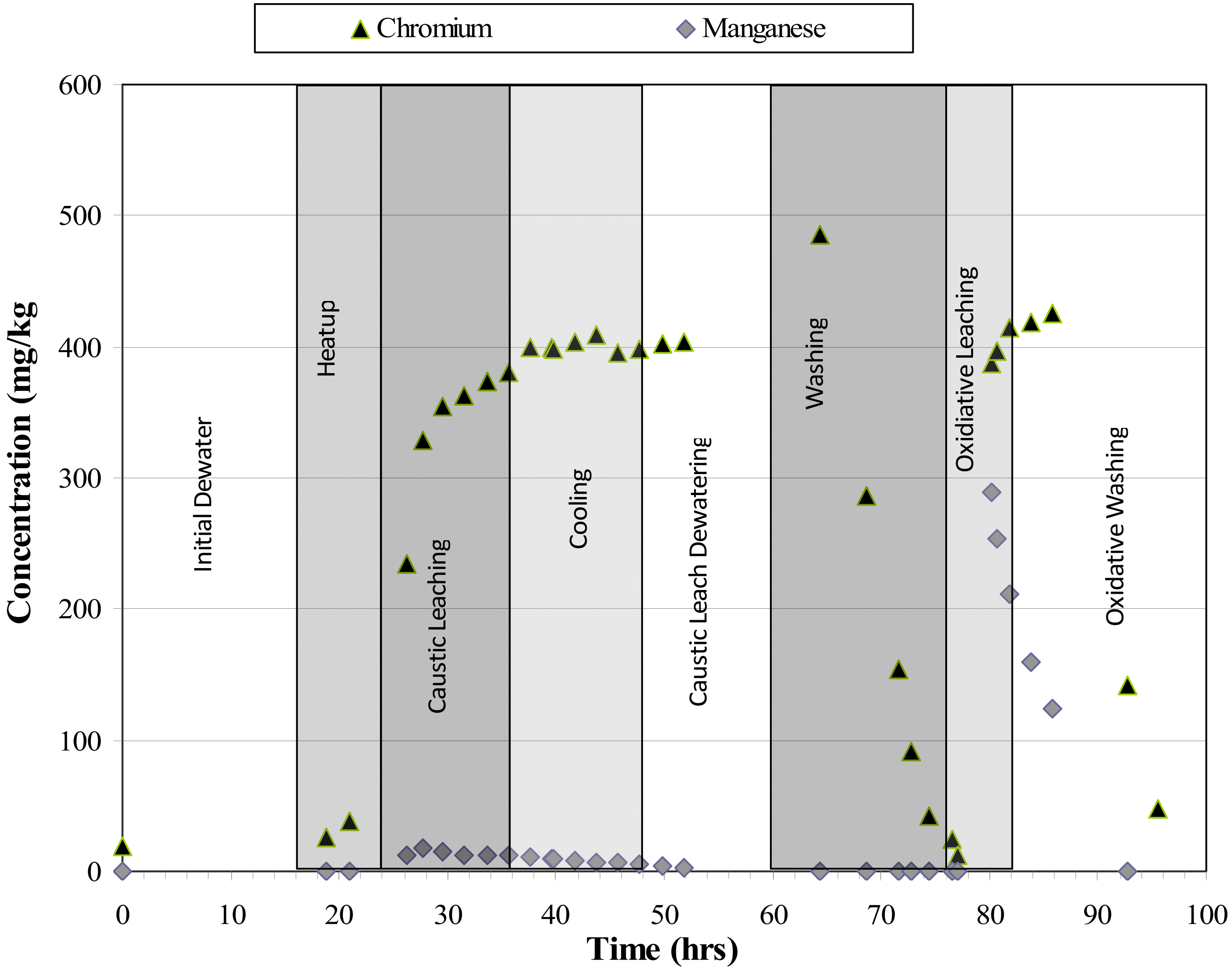

Figure 6.4. Chromium and Manganese Concentration in mg/kg solution in Test CBM-2

The slurry composition results are shown in Table 6.7 for test CBM-2. The levels of insoluble components such as calcium, iron, lead, manganese, neodymium, nickel, and zirconium were enriched significantly throughout the test because the aluminum and other soluble compoonents dissolved.

Table 6.7. Slurry Analysis (CBM-2) (mg/kg slurry)

	CBM25-IN-SL	CBM25-DW-SL	CBM25-LC-SL	CBM25-WS-SL	CBM25-FSC-SL
Aluminum	15,248	56,933	29,118	60,646	57,728
Calcium	119	472	486	1,103	1,088
Chromium	365	1,476	586	719	359
Iron	3,571	15,389	14,559	36,755	36,160
Lead	182	735	657	1,619	1,598
Magnesium	35.4	144	147	353	348
Manganese	757	3,266	3,098	7,744	9,033
Nickel	118	505	481	1,205	1,190
Potassium	1,585	1,284	225	<90	<98
Sodium	78,857	72,058	63,824	4,936	2,006
Zirconium	52	221	168	446	458
Nitrate	22,900	20,700	3,270	87	30.5
Nitrite	6,790	6,210	1,180	40.8	<22
Oxalate	3,600	15,200	12,800	<40	<44
Phosphate	356	272	<50	<40	<44
Sulfate	842	726	644	1,370	49.3

Notes: Values reported as “less than” are not quantifiable above background.
CBM25-IN-SL = initial slurry composition
CBM25-DW-SL = slurry composition following dewatering
CBM25-LC-SL = slurry composition following the caustic leach
CBM25-WS-SL= slurry composition following oxidative treatment
CBM25-FSC-SL = final slurry composition.

The concentration ratios of various species in the solids are described in Table 6.8 for test CBM-2. The results indicate that chromium and aluminum are removed relative to iron and calcium.

Table 6.8. Ratio of Various Species in Solids from Test CBM-2

	Initial Conditions	At Start of Caustic Leaching Heat-up	After Caustic Leaching	Cool Down	End of Test
Al/Fe	3.6	3.6	1.4	1.6	1.6
Cr/Fe	0.10	0.09	0.02	0.02	0.01
Al/Ca	108	116	43	55	53
Cr/Ca	2.9	3.1	0.55	0.64	0.19

6.1.4 Conclusions

The leach factors were calculated as defined in Equation 6.1 while the boehmite leach factors were calculated as defined in Equation 6.2. The overall leach factors for both the CBM-1 and CBM-2 tests are described in Table 6.9 and Table 6.10. The Al leach factors are essentially the same with errors of ±15% and overall, they are very low. When accounting for the distribution of gibbsite and boehmite in the initial simulant, the Al leach factor for boehmite is 0.12 and 0.23 for CBM-1 and CBM-2, respectively. The boehmite leach factor is estimated assuming 100% dissolution of gibbsite. These boehmite leach factors are consistent with those observed in tests BSM-1, 8, and 9 where approximately 10% of the boehmite leached in 8 hours under similar conditions. These results indicated that the results obtained from the integrated test are effectively the same as those observed in the bench-scale simulant tests.

Table 6.9. Al Leaching Ratios for Test CBM-1 and CBM-2

	CBM-1	CBM-2
Al leach factor based on Fe	0.50	0.56
Al leach factor based on Pb	0.45	0.52
Al leach factor based on Ca	0.52	0.55
Boehmite leach factor	0.08	0.15

Results indicate that the majority (80%) of the Cr dissolved during caustic leaching in CBM-1. Therefore, only an additional 10% of the Cr dissolved during oxidative leaching.

Table 6.10. Leaching Ratios of Various Species for Test CBM-1 and CBM-2

	CBM-1	CBM-2
Cr/Fe after ox leach	0.007	0.006
Cr/Fe after caustic leach	0.018	0.019
Cr/Nd after ox leach	0.27	---
Cr/Nd after caustic leach	0.70	---
Cr/Pb after ox leach	0.17	0.13
Cr/Pb after caustic leach	0.41	0.43
Cr/Ca after ox leach	0.23	0.19
Cr/Ca after caustic leach	0.57	0.64
Total Cr Leach Factor	0.92	0.93
Caustic Leach Factor	0.79	0.77

A higher temperature filtration resulted in significant post-filtration precipitation in both tests as observed in the samples.

6.1.5 Observations during initial Cold-CUF runs

Images of the leachates (supernatant solution) show some changes in the clarity of the solutions, which indicates that changes occurred during the caustic leaching steps after cooling to the filtration temperature. This coloration has been associated with the precipitation of iron, manganese, and oxalate as indicated by analytical analysis.

Images of the supernatant solution that were taken during the caustic leaching heat up at a rate of 0.236°C/min to 100°C in 5.3 hours are shown in Figure 6.5. These samples show an increase in coloration as the heating progresses at 0, 2, and 4 hours. This coloration appears to be indicative of post filtration precipitation. Close inspection indicates that a source of this coloration is very fine particulate material. Figure 6.6 shows the supernatant solution samples taken every 2 hours during the caustic-leaching period at 100°C, and Figure 6.7 shows the samples as they were cooled down to 25°C from 100°C over 12 hours. All of the photos shown are from CBM-2 but were common to CBM-1 also.

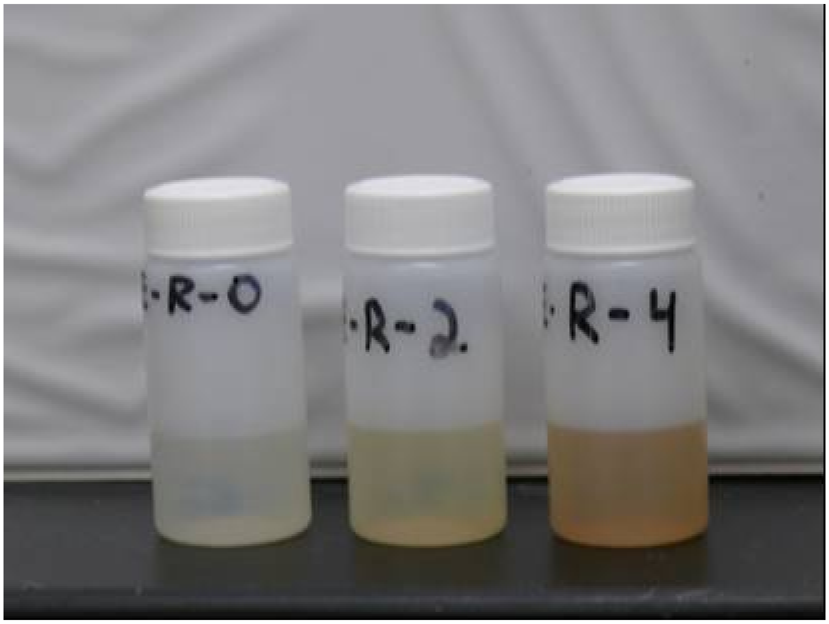

Figure 6.5. Caustic Leach Tank Heat Up Samples from CBM-2

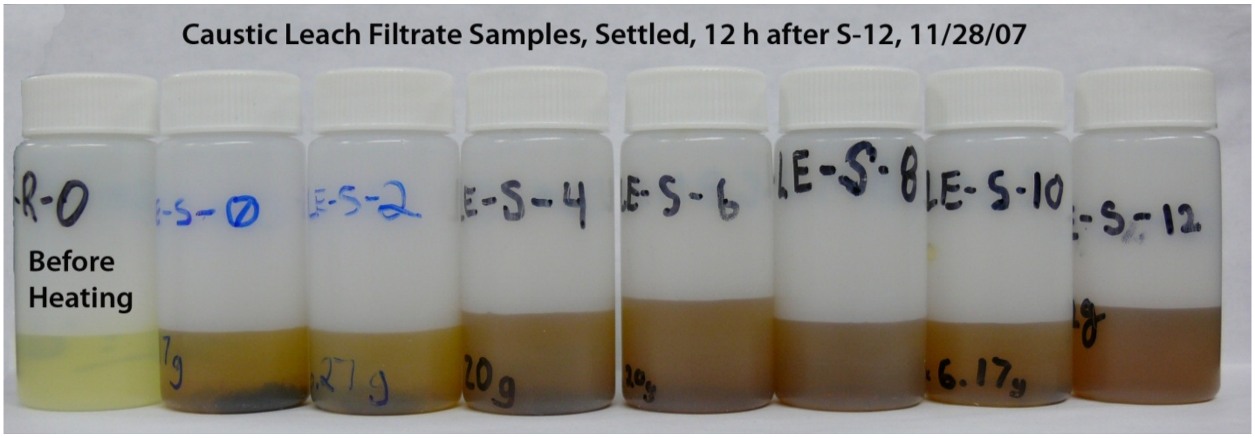

Figure 6.6. Photograph of 22-mL Sample Vials from the Caustic Leaching of Test CBM-2 (0, 2,4,6,8,10, and 12 hours from left to right)

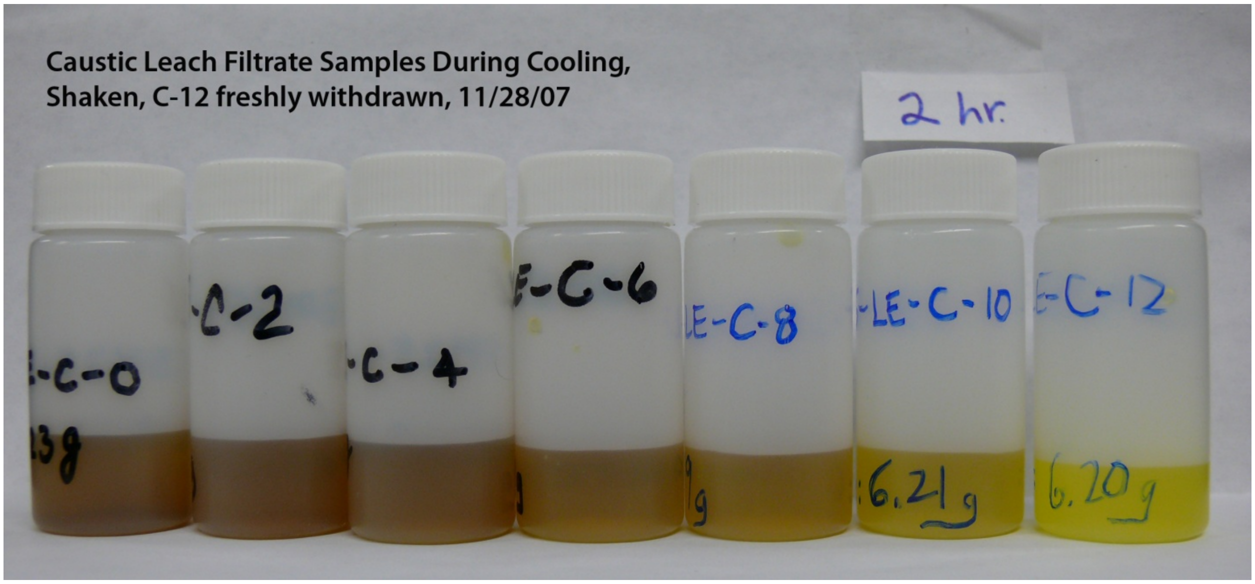

Figure 6.7. Photograph of 22-mL Sample Vials from the Caustic Leach Cooling of Test CBM-2 (0, 2,4,6,8,10, and 12 hours from left to right)

After the 12-hour leach sample, the tank was allowed to cool over 12 hours to ~ 40°C. Samples were taken every 2 hours during this process. These samples showed decreasing precipitation with time (see Figure 6.8). Finally, the tank was allowed to cool slowly from 40°C to ~25°C for another 12 hours. These samples show little evidence of continuing precipitation; however, close inspection of the bottom of the bottles does show the presence of a slight amount of precipitation in the samples.

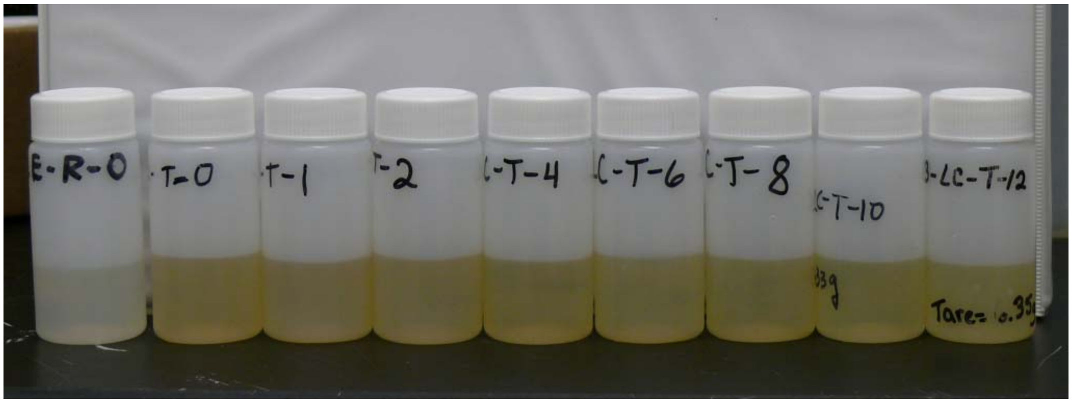

Figure 6.8. Samples After Cooling over 12 Hours of Test CBM-2 (R-0 is before heating, 0, 1, 2, 4, 6, 8, 10, 12 hours after cooling)

Figure 6.9 shows the difference in color and suspended particle content between the leached filtrate from the CBM-1 and CBM-2 tests. The CBM-1 sample was collected at 45°C and then cooled to room temperature, and the CBM-2 sample was collected at 25°C and allowed to equilibrate with the room temperature. This indicates that the higher filtration temperature of CBM-1 (45°C) appears to cause more precipitation when the filtrate is cooled than the lower filtration temperature of CBM-2 (25°C).

Figure 6.9. Photographs of the CBM-1 and CBM-2 Test Leach Filtrates at the End of Caustic Leaching

All the samples taken during the oxidative leaching process exhibited a strong purple color or dark color (see Figure 6.9). However, all of the wash samples were yellow, indicating the presence of chromium. The sample on the left in Figure 6.10 is the first wash solution. The bright yellow color is indicative of soluble Cr(VI). The sample on the right was one of the samples taken during oxidative leaching and shows the purple color indicative of permanganate (MnO_4^-).

This demonstrates that the reaction did not go to completion during the oxidative leaching timeframe or that there was excess permanganate present, but the reaction went to completion immediately after wash water was added.

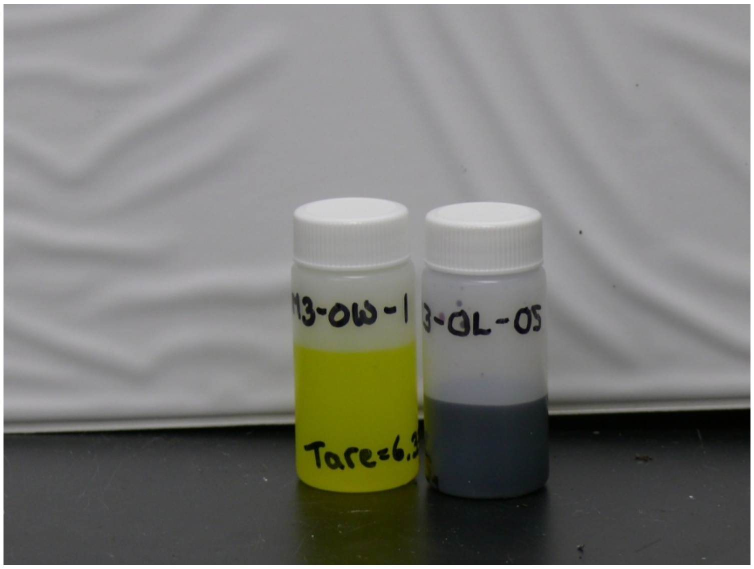

Figure 6.10. Oxidative Leach Samples
(Oxidative wash [OW] on the left and oxidative leach [OL] on the right)

6.2 Simulant Production Scale-Up Assessment Tests

6.2.1 Experimental Details

The next three tests (CBM-3, CBM-4, and CBM-5) determined the ability of the simulant-producing vendor to scale-up the simulant production process. The filtration simulants used in the CBM-3, CBM-4, and CBM-5 tests contained equal concentrations (in grams per kg of UDS) of boehmite, gibbsite, oxalate, and iron-rich sludge. Test CBM-3 (a 25-liter batch) was done using a sludge simulant prepared by Optima with the boehmite, gibbsite, and sodium oxalate added by PNNL. Tests CBM-4 (a 15-gallon batch) and CBM-5 (a 250-gallon batch) were run with a crossflow ultrafiltration simulant generated by Noah, which included all of the components (boehmite, gibbsite, sodium oxalate, and sludge solids with supernate). The results of these tests were compared with the results of test CBM-2, which was run with a simulant prepared by PNNL, and used a temperature of 25°C for the initial dewatering and all of the procedures after the caustic leach.

An anti-foaming agent (AFA) was added to the simulant at the start of tests CBM-4 and CBM-5. Test CBM-3 was run without any AFA added.

Samples of slurries, supernates, and filtrates were regularly collected during the test to monitor the progression of metal and anion dissolution. These samples were analyzed with ICP-MS and ICP-AES. All slurry samples and a few filtrate samples collected for the CBM-3 test were analyzed at PNNL. Southwest Research Institute analyzed the remainder of the CBM-3 filtrate samples and all solution and slurry samples collected for the CBM-4 and CBM-5 runs. Anions measured with IC include nitrite (NO_2^-), nitrate (NO_3^{2-}), phosphate (PO_4^{3-}), sulfate (SO_4^{2-}), and oxalate ($C_2O_4^{2-}$). In general, the mass balance between the elemental composition of the untreated simulant sludge and the elemental composition derived from the wash solutions agreed, although there were some outliers—that mostly came from samples analyzed by PNNL. Results of all analyses are listed in Appendix C.

All three tests started with a dewatering step of the initial slurry at 25°C to ~20 wt% UDS. After this step, a caustic leach was performed by adding 19 M NaOH of DIW to the slurries. The temperature was increased to 100°C over 5.3 hours and held for 12 hours, and then it was cooled to 25°C over 12 hours. Samples of the supernate were collected regularly every 2 hours during the leaching process every 2 hours for composition analysis and Al dissolution tracking.

After the caustic leaching, the slurries were dewatered to 17 wt% UDS. All systems subsequently underwent 16 incremental wash treatments (at 25°C), which each involved adding 700 mL of 0.01 M NaOH, vigorous mixing for approximately 30 minutes, and then dewatering by ~ 700 mL. Samples of the 16 wash supernates were collected during this time for analysis by ICP-AES for cations.

The next step involved performing an oxidative leach using $NaMnO_4$. This process was designed to remove chromium from the waste since this constituent inhibits glass formation. Since earlier tests (CBM-1 and CBM-2) had indicated that as much as 90 wt% of the CrOOH is dissolved during caustic leaching operation, the CrOOH slurry was added to the tests following post-caustic leach dewatering and wash operations so that quantitative results on the efficacy of the oxidative leaching process could be determined. CrOOH slurry and 0.01 M NaOH were added to the post-caustic leached, washed, and dewatered slurries, and the resulting mix was dewatered to 17% UDS. Then 1-M $NaMnO_4$ solution was added and allowed to leach for 6 hours at 25°C with continuous stirring during each test. Oxidative leach supernate samples were collected every 2 hours during these 6 hours to track Cr dissolution.

After the oxidative leach process, the slurry underwent 13 incremental washings, where each involved adding 700 mL of 0.01 M NaOH and then removing ~700 mL of supernatant solution through filtration. The filtrate solutions were collected for ICP analysis of the cations. The slurry remaining at the end of the incremental washing was then dewatered to ~20 wt%. A sample of the final slurry concentrate was collected for composition analysis. Process time-lines for each of these tests are provided in Table G.3 through Table G.5 in Appendix G.

6.2.2 Results

The physical properties of each simulant have been measured (data can be found in WTP-RPT-183,[(a)] Table 6.1) and have been found to be consistent. Given this, and the fact that they contain equal concentrations of key chemicals, it was expected for them to behave similarly to both caustic and oxidative leaching.

6.2.2.1 Aluminum Leaching Behavior

As shown in Table 6.11, the aluminum leach factors, calculated using the ICP measured slurry concentrations, are within 10% of each other, with the average being 0.61 except when compared to Pb. The average boehmite leach factor was determined to be 0.26 as shown in Table 6.12. The average Al and boehmite leach factors (0.61 and 0.26) that were determined using the data from CBM-3, CBM-4, and CBM-5 are slightly higher than those determined in the CBM-2 test (0.54 and 0.15). If only the iron and calcium are compared, the leach factors are within experimental error of ±15%, indicating that there is no real difference between simulants.

Table 6.11. Leaching Ratios of Aluminum for Test CBM-3, CBM-4, and CBM-5

	CBM-3	CBM-4	CBM-5
Al leach factor based on Fe	0.64	0.59	0.60
Al leach factor based on Pb	0.64	0.51	0.66
Al leach factor based on Ca	---	0.60	0.60

Table 6.12. Total Aluminum Leach Factors for the Different Filtration Simulants

Test	Al Leach Factor	Boehmite Leach Factor	Vendor
CBM-3	0.64	0.21	Optima/PNNL
CBM-4	0.57	0.28	Noah
CBM-5	0.62	0.29	Noah
Average	0.61	0.26	

(a) Russell et al. Dec. 2008. *Development and Demonstration of Ultrafiltration Simulants*. WTP-RPT-183, Rev. A.

Figure 6.11 plots the filtrate aluminum concentration (in mg/kg) as a function of time during the caustic-leach process. Post-leach dewatering was performed with 16 incremental washes. The filtrate analysis data show that the aluminum in the three feed simulants reacted essentially identically during the caustic-leaching process with each test following the same leaching trend.

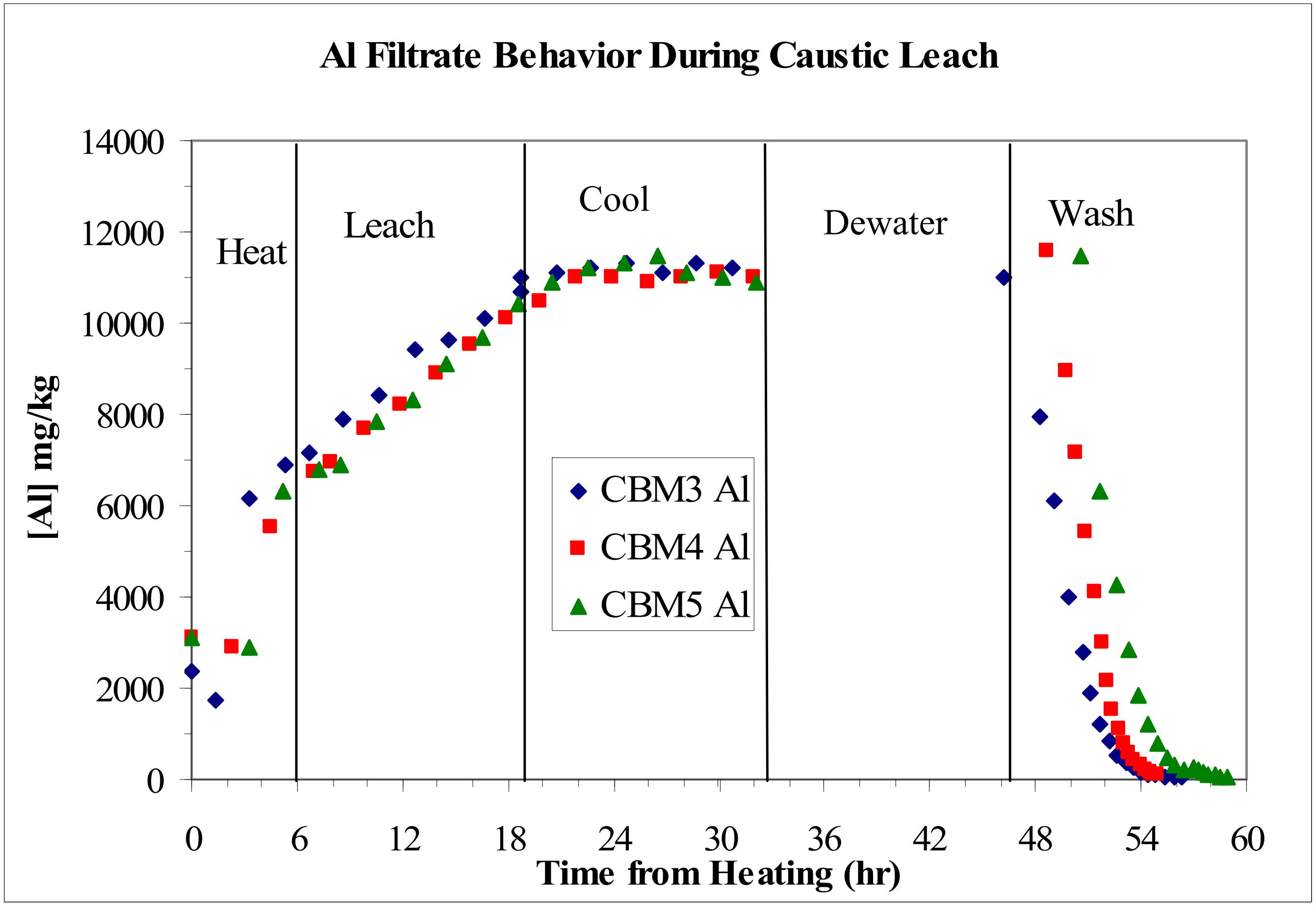

Figure 6.11. Dissolution of Aluminum in mg/kg Solution in Tests CBM-3, CBM-4, and CBM-5

6.2.2.2 Chromium and Manganese Leaching Behavior

The oxidative-leach process is effective at removing chromium from the waste slurry. As mentioned before, 1.73 kg of CrOOH was added to the slurry just before the start of the oxidative-leach process. This explains why the chromium concentration spikes just before the oxidative-leach process in the overall treatment process. However, as Figure 6.12 shows, this is rapidly dissolved when permanganate is added. Figure 6.12 shows that right after permanganate was added, the chromium concentration in the filtrate was very similar for all three PEP simulant systems. However, it does appear that after the initial dissolution, some of the chromium precipitates in the CBM-3 solid since the supernatant concentration decreases from ~5000 mg/kg to ~4000 mg/kg. CBM-4 and CBM-5 appear to be more effective at keeping chromium in solution since the filtrate chromium concentrations remain stable at around 5700 mg/kg throughout the oxidative-leach process.

The Cr(III) caustic-leach factors are 0.65, 0.58, and 0.69 for CBM-3, CBM-4, and CBM-5 tests, respectively. Analysis of the final slurry samples (see Appendix C) indicated that all three simulants react identically to the oxidative leach with effectively 100% of the chromium leached from the solids during this step.

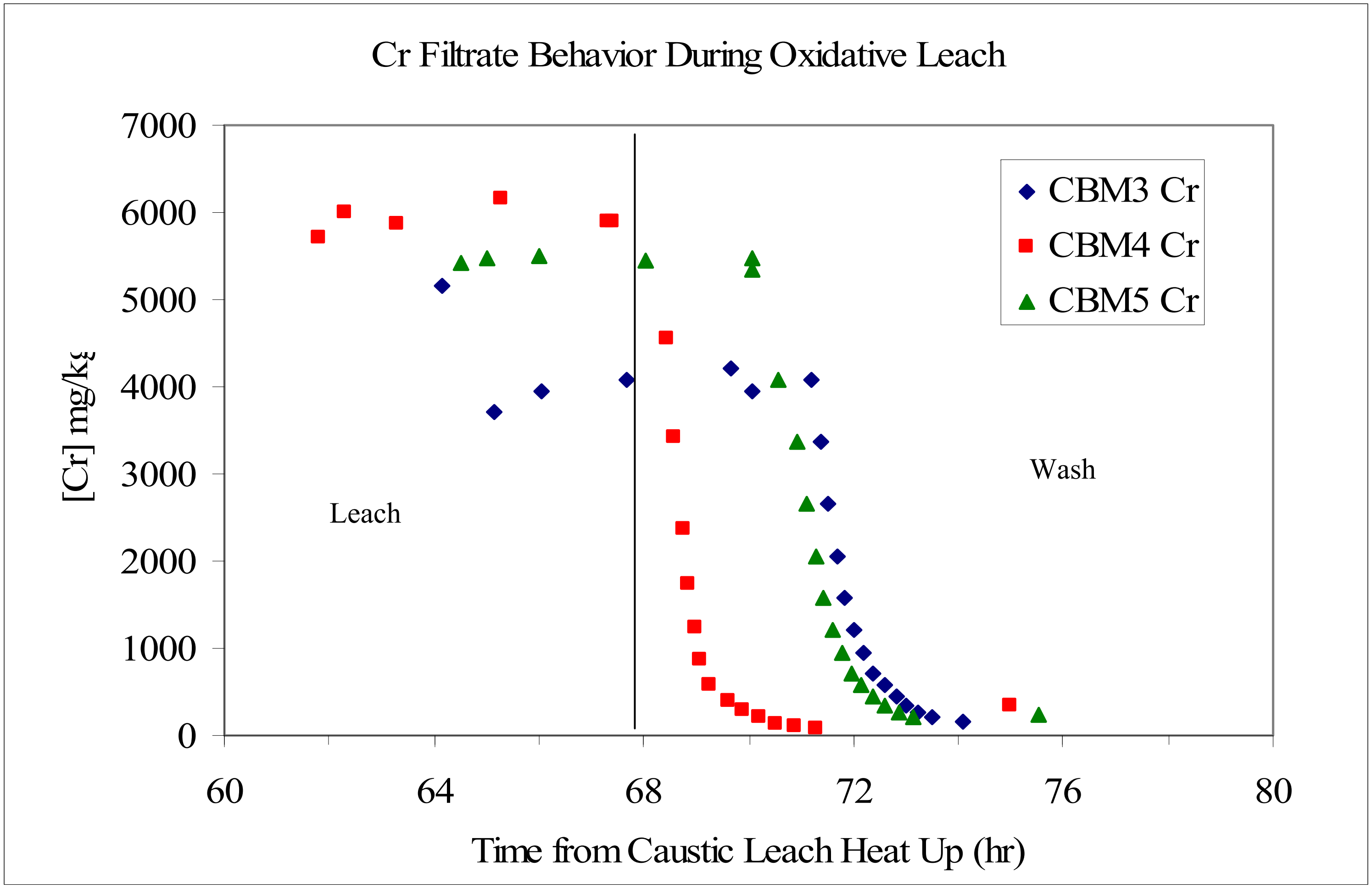

Figure 6.12. Chromium Concentration in mg/kg Solution During the Oxidative Leach Process

6.2.3 Summary

The purpose of these tests was to determine if sludge simulants, prepared by different suppliers in different batch quantities, had similar reaction properties when subjected to high-temperature caustic leach and room-temperature oxidative leach. The three simulants, labeled CBM-3, CBM-4, and CBM-5, showed similar reactive behavior in their chemical leaching properties.

The total aluminum leach factors determined for the three simulants were in agreement within 10%, with the average being 0.61.

Chromium in the three simulants reacted similarly. However, since all three reacted effectively to complete dissolution, it is difficult to determine any significant differences between the oxidative leaching behaviors in these three tests.

7.0 Boehmite Simulant Revision Testing

The initial selection of boehmite simulant was based on the crystallography and mineralogy. This boehmite simulant revision was required from Test Plan TP-RPP-WTP-469 to re-evaluate the selected boehmite simulant candidate and revise the selection, if required, to best simulate the boehmite dissolution behavior in the actual waste. Therefore, the objective of these tests was to test various boehmites under different leaching conditions and compare them to the actual waste testing results and then to determine the best boehmite simulant.

Two sets of tests were performed as described in Section 2.5. In the first set, B1, B3, B4, and B5 boehmite sources were used based on their initial leaching results and how they compared to the actual waste leaching results (Group 5) (Fiskum 2008). The second set of tests used new boehmite sources obtained from APYRAL that had particle sizes in the preferred range. These were AOH30 (B6) and AOH180E (B7). Table 7.1 shows the test matrix that was performed using 1-wt% boehmite solids for the first set of tests. Table 7.2 shows the test matrix that was performed using 1-wt% boehmite solids for the second set of tests. They were fully mixed at 120 rpm, and samples were taken at 1, 2, 4, 8, and 24 hours and analyzed for Al and Na using ICP-AES.

Table 7.1. Boehmite Revision Test Matrix for the First Set of Tests

Test ID	Boehmite Source	Temperature (°C)	Leach Soln. Conc. (M NaOH)
BCT2-1	B1	100	3
BCT2-2	B4	100	3
BCT2-3	B1	100	5
BCT2-4	B4	100	5
BCT2-5	B1	100	7
BCT2-6	B4	100	7
BCT2-7	B3	100	5
BCT2-8	B5	100	3
BCT2-9	B5	100	5
BCT2-10	B5	100	7

Table 7.2. Boehmite Revision Test Matrix for the Second Set of Tests

Test ID	Boehmite Source	Temperature (°C)	Leach Soln. Conc. (M NaOH)
BCT3-1	B6	100	3
BCT3-2	B7	100	3
BCT3-3	B6	100	5
BCT3-4	B7	100	5
BCT3-5	B6	100	7
BCT3-6	B7	100	7

The results of the boehmite simulant leaching in 5 M NaOH are shown in Figure 7.1, and the results of leaching in 3 M NaOH are shown in Figure 7.2. The data from Group 5 actual waste (REDOX sludge) are also shown for comparison to the boehmite simulants because it is a high-boehmite-containing tank waste (Fiskum 2008). B4 provides the same dissolution rate within the experimental uncertainty as B7, which was selected for additional testing, while B5 is measurably slower than the rest of the boehmite samples.

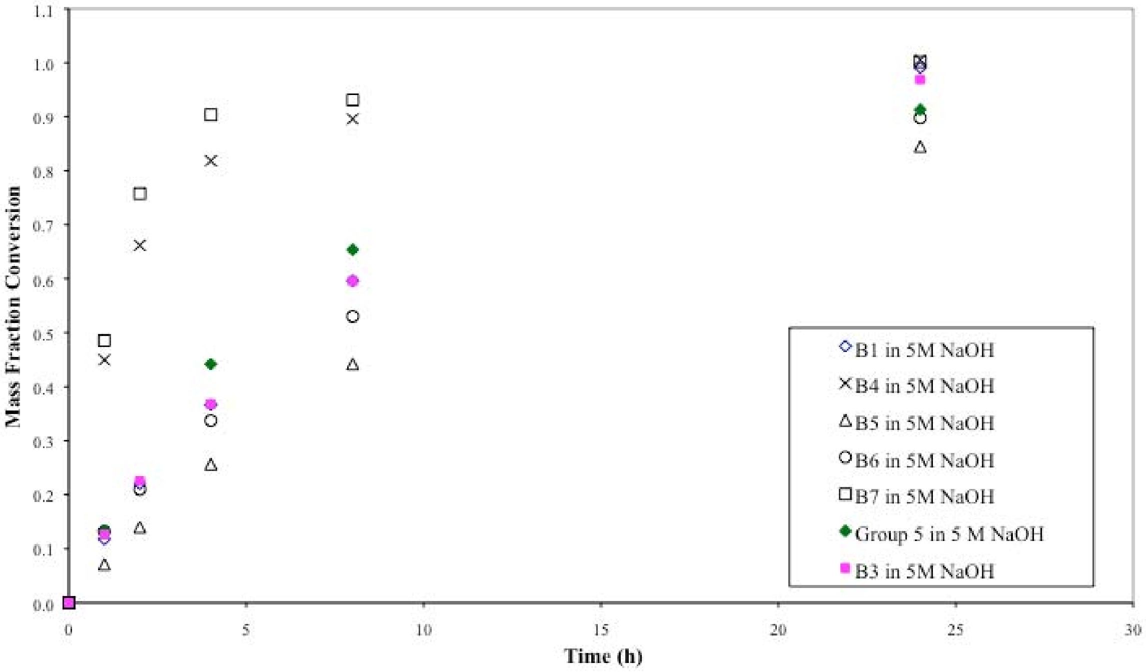

Figure 7.1. Boehmite Sources Compared with Actual Waste Group 5 (REDOX Sludge) in 5 M NaOH

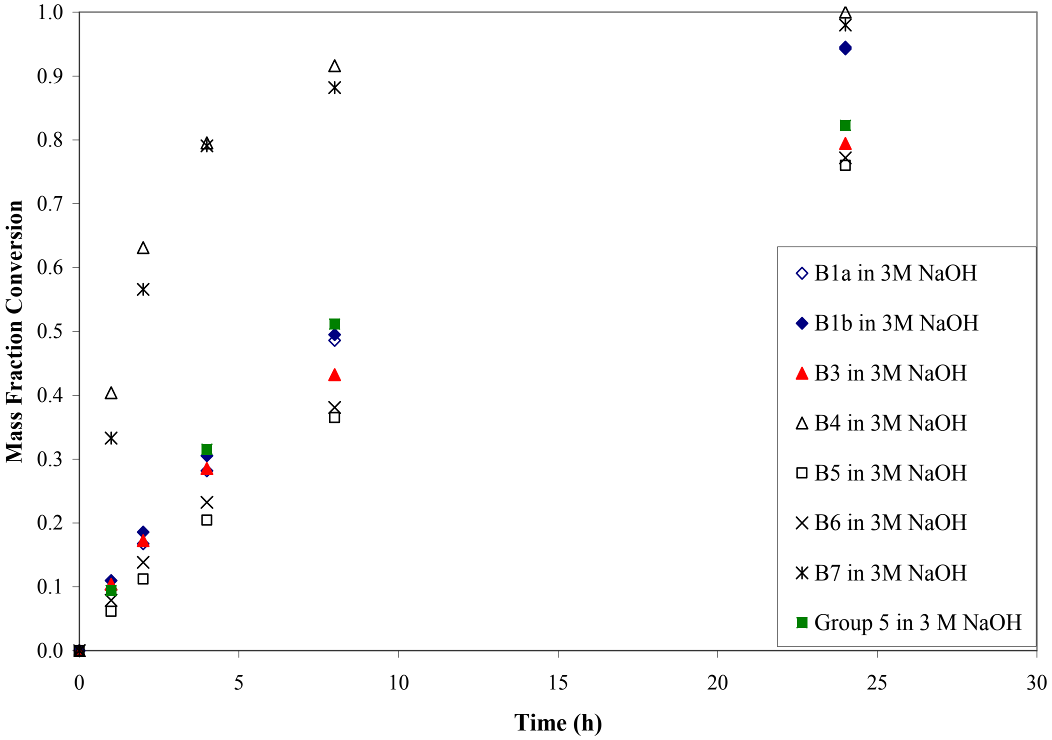

Figure 7.2. Boehmite Sources Compared with Actual Waste Group 5 (REDOX Sludge) in 3 M NaOH

8.0 Summary and Conclusions

8.1 Boehmite Selection

When the boehmite simulant results are plotted with the data from Lumetta, B3 and B5 match the results from tank waste S-101 and S-110 the best. B3 was a commercially available product and was therefore selected for further testing.

Therefore, the B3 (APYRAL AOH20) boehmite is recommended because of the crystalline structure and particle size as well as its availability in commercial quantities.

8.2 Impact of Reaction Conditions

Temperature, hydroxide concentration, approach to solubility, and stirring rate were tested to determine the effect of each of these on the dissolution rate of the B3 (APYRAL AOH20) boehmite. Increasing the hydroxide concentration increases the boehmite dissolution rate. The temperature over the range tested has a significant effect on the dissolution rate. The stirring rate at 60°C for both 1 M and 5 M NaOH had a significant effect on the boehmite dissolution. The effect was greater, however, in the 5 M NaOH, indicating that hydroxide concentration has a greater effect than the mixing rate. In all cases, an increased stirring (mixing) rate was observed to increase the dissolution rate but not to the degree found for increasing the temperature or hydroxide concentration over the ranges tested.

The B3 boehmite simulant that was chosen to be the “component” simulant was then blended in a specified ratio with the other chosen “component” simulants for gibbsite, sodium oxalate, supernatant, condensate (de-ionized water), and ultrafiltration slurry. This blended simulant was then retested over a subset of the matrix conditions in which the component simulants had been tested previously.

Gibbsite has a significant effect on the dissolution of boehmite. Ultrafiltration slurry and sodium oxalate did not appear to have any significant effect on the boehmite dissolution, however. Samples indicate that the boehmite solubility limit is greater than 20,000 mg/L Al under the tested conditions. Also note that there appears to be some boehmite dissolution up to 172 hours.

With regard to boehmite dissolution behavior, the effect of anions on the kinetics of boehmite dissolution was examined. Two sets of tests were completed: 1) one that examined the effect of the aluminate anion on the rate of boehmite dissolution and 2) another that determined if the presence of secondary anions typical of Hanford tank wastes (carbonate, free-hydroxide, nitrate, nitrite, oxalate, phosphate, and sulfate) affect boehmite dissolution.

The aluminate anion studies provided information on the consequences of gibbsite in the waste due to the expected fast dissolution of gibbsite relative to boehmite. The aluminate anion simulated dissolved gibbsite in the supernate. Aluminate was found to have a significant impact on the boehmite dissolution in 5 M NaOH and reduced the dissolution rate for boehmite by 4.3 times. An increase of aluminate concentration from 50% to 75% boehmite solubility caused a decrease of 25 ± 7 mg/kg-hr in the average rate of boehmite dissolution.

Anion addition slowed the rate of boehmite dissolution with the effect being approximately twice as great in 5 M NaOH than in 1 M NaOH. A drop in boehmite dissolution is also observed with anion addition in 1 M NaOH. Boehmite dissolution is affected more by the hydroxide and aluminate concentrations than by the anion concentration.

The objective of the boehmite simulant revision tests was to determine the effect of hydroxide concentration on the dissolution kinetics of different boehmites and compare these results to the actual waste testing to verify that the boehmite chosen matched the behavior of the actual waste as close as possible under different leaching conditions. B4 appeared to be about the same as B7, which was selected for additional testing, while B5 appeared to be slower than the rest of the boehmite samples.

8.3 Integrated Testing

The objective of these tests was to assess the integrated performance of leaching and filtration simultaneously. These tests used the blended filtration simulant that was planned to be used in the PEP tests. These tests were broken into two blocks of tests. The first block consisted of two tests under the initial planned leaching conditions. In the first test (CBM-1), the slurry was dewatered and washed at 45°C. The next test (CBM-2) was performed essentially to repeat CBM-1 except the slurry was dewatered and washed after caustic leaching at 25°C instead of 45°C to determine the effect of temperature on the filter flux.

The Al leach factors of these tests were essentially the same and overall, they were very low. When accounting for the distribution of gibbsite and boehmite in the initial simulant, the leach factor for boehmite is 0.08 and 0.15 for CBM-1 and CBM-2, respectively.

The leaching factor of chromium in CBM-2 was higher than in CBM-1. In CBM-2, more chromium was leached during the oxidative step than in CBM-1where the Cr/Fe ratio is much higher in CBM-2 than CBM-1. Results indicate that the majority (80%) of the Cr dissolved during caustic leaching in CBM-1. Therefore, only an additional 10% of the Cr dissolved during oxidative leaching.

The next block of tests (CBM-3, CBM-4, and CBM-5) determined the ability of the simulant-producing vendor to produce the simulant at various production quantities. They show similar reactive behavior with their measured chemical and physical properties being comparable.

The total aluminum leach factors determined for the three simulants were in agreement within 10%, with the average being 0.61. The CBM-3 simulant had a slightly higher (~ 20%) boehmite leach factor when compared with the other two tests, but this might be a result of the way the slurry samples collected throughout this test were treated for ICP-AES analysis.

Analysis of aluminum in the various supernates shows that, with respect to heating and caustic leaching, all three simulants react identically. The systems did not show any common reaction behavior to oxidative leach, but did respond in unison to the final wash process.

9.0 References

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Appendix A

Actual Analytical Data

Appendix A: Actual Analytical Data

Table A.1. Boehmite Screening Test Leaching Results (PNNL ASR# 7902)

Time (hrs)	CT-B1		CT-B2		CT-B3		CT-B4		CT-B5	
	Al (µg/mL)	Na (µg/mL)	Al (µg/mL)	Na (µg/mL)	Al (µg/mL)	Na (µg/mL)	Al (µg/mL)	Na (µg/mL)	Al (µg/mL)	Na (µg/mL)
0	--	68,000	--	67,800	--	66,300	--	66,200	--	67,100
1	561	68,500	535	68,200	532	67,200	2,170	67,500	379	68,100
2	950	67,900	887	67,500	881	68,300	3,150	68,400	634	67,000
4	1,560	68,800	1,450	69,100	1,460	68,200	3,980	67,100	1,110	69,000
8	2,530	70,100	2,190	68,300	2,210	68,400	4,630	68,400	1,750	69,700
24	4,830	74,000	4,300	74,500	4,060	74,200	5,160	73,400	3,590	75,000
Final	4,880	74,700	4,260	73,800	3,920	69,800	5,060	73,300	3,520	73,600

Table A.2. Al Boehmite Matrix Test Leaching Results (SWRI SSR# 30803)

Time (hrs)	MTB-1 (mg/L)	MTB-2 (mg/L)	MTB-3 (mg/L)	MTB-4 (mg/L)	MTB-5 (mg/L)	MTB-6 (mg/L)	MTB-7 (mg/L)	MTB-8 (mg/L)	MTB-9 (mg/L)	MTB-10 (mg/L)	MTB-11 (mg/L)	MTB-12 (mg/L)	MTB-13 (mg/L)
0	3.86	<3.00	1.11	1.20	<3.00	<3.00	1.26	<1.00	<3.00	<1.00	<2.00	<2.00	<2.00
1	2,300	468	103	163	11.2	21.9	7.06	2.07	2,370	2.07	128	133	118
2	3,960	909	211	285	21.3	37.8	12.8	4.02	4,050	3.62	228	245	221
4	6,690	1,780	452	490	48.0	69.9	18.7	6.37	6,620	6.34	404	423	404
8	10,300	3,540	905	830	102	123	30.8	10.7	9,470	11.6	714	740	724
24	16,700	9,870	2,260	1,750	318	314	71.7	31.6	17,600	31.6	1,710	1,720	1,710
Final	17,300	9,830	2,280	1,740	337	319	72.1	32.9	17,500	31.9	1,700	1,740	1,710

Table A.3. Na Boehmite Matrix Test Leaching Results (SWRI SSR# 30803)

Time (hrs)	MTB-1 (mg/L)	MTB-2 (mg/L)	MTB-3 (mg/L)	MTB-4 (mg/L)	MTB-5 (mg/L)	MTB-6 (mg/L)	MTB-7 (mg/L)	MTB-8 (mg/L)	MTB-9 (mg/L)	MTB-10 (mg/L)	MTB-11 (mg/L)	MTB-12 (mg/L)	MTB-13 (mg/L)
0	110,000	108,000	22,000	22,000	106,000	109,000	21,800	21,800	110,000	21,700	65,900	67,200	65,200
1	110,000	109,000	22,400	22,500	107,000	109,000	22,000	21,600	109,000	22,000	65,500	65,000	66,800
2	110,000	109,000	22,600	22,600	102,000	111,000	21,900	21.800	111,000	22,100	66,400	66,700	67,300
4	109,000	110,000	22,800	22,600	109,000	110,000	22,000	21,900	110,000	22,300	67,200	66,200	66,600
8	112,000	112,000	23,600	22,800	109,000	111,000	22,000	22,100	112,000	22,200	68,600	67,800	67,900
24	114,000	118,000	25,000	24,400	110,000	112,000	22,000	22,400	119,000	23,500	68,900	68,500	70,300
Final	117,000	119,000	25,200	24,100	110,000	112,000	22,100	22,200	118,000	23,300	69,200	69,100	67,600

Table A.4. Blended Matrix Al Boehmite Leaching Test Results (SWRI SSR# 31241, 31334, and 31084)

Time (hrs)	BSM-1 (mg/L)	BSM-2 (mg/L)	BSM-2a (mg/L)	BSM-5 (mg/L)	BSM-6 (mg/L)	BSM-6a (mg/L)	BSM-7 (mg/L)	BSM-8 (mg/L)	BSM-9 (mg/L)	BSM-10 (mg/L)	BSM-11 (mg/L)	BSM-12 (mg/L)	BSM-12a (mg/L)	BSM-13 (mg/L)	BSM-13a (mg/L)
0	7.81	5.89	N/A	8.51	8.64	<3.46	9.73	11,100	7.93	5.91	8.95	4.37	4.14	7.57	<3.45
1	13,300	13,200	10,800	716	992	1,080	13,200	12,900	13,100	13,500	10,500	1,270	1,070	506	437
2	13,400	13,400	10,600	1,350	1,740	1,930	13,200	13,100	13,200	14,000	11,600	2,220	1,940	1,150	921
4	13,600	13,800	10,800	2,350	2,990	3,230	13,700	13,400	13,600	14,200	12,200	3,730	3,220	2,470	1,890
8	14,200	14,800	11,500	4,240	5,230	5,150	14,400	14,000	14,100	15,100	13,600	5,930	5,170	5,000	3,930
24	16,400	18,700	13,000	9,390	11,600	9,520	16,600	16,000	16,500	18,300	16,300	11,900	9,680	11,400	9,420
Final	16,400	18,700	13,400	9,620	11,500	9,450	17,200	16,500	16,600	17,500	16,500	11,800	9,730	11,400	9,400

Table A.4.a. Blended Matrix Al Boehmite Leaching Test Results (SWRI SSR# 31241, 31334, and 31084)

Time (hrs)	BSM-14 (mg/L)	BSM-15a (mg/L)	BSM-16a (mg/L)	BSM-17a (mg/L)	BSM-18a (mg/L)	BSM-19a (mg/L)
0	5.99	7.58	4.89	4.48	<3.85	6.99
1	14,100	12,200	10,400	11,800	11,900	9,100
2	14,500	12,200	11,400	12,100	12,100	10,300
4	14,500	12,300	12,100	12,200	12,200	11,200
8	15,100	12,500	12,900	12,300	12,300	12,200
24	16,300	13,400	13,500	13,500	13,500	14,300
72	18,800	15,700	15,300	14,900	15,400	15,400
120	19,200	16,000	15,500	15,400	16,100	17,800
168	19,700	20,800	16,500	16,900	16,500	17,400
Final	20,000	17,200	17,800	17,900	16,900	18,100

Table A.5. Blended Matrix Na Boehmite Leaching Test Results (SWRI SSR# 31241, 31334, and 31084)

Time (hrs)	BSM-1 (mg/L)	BSM-2 (mg/L)	BSM-2a (mg/L)	BSM-5 (mg/L)	BSM-6 (mg/L)	BSM-6a (mg/L)	BSM-7 (mg/L)	BSM-8 (mg/L)	BSM-9 (mg/L)	BSM-10 (mg/L)	BSM-11 (mg/L)	BSM-12 (mg/L)	BSM-12a (mg/L)	BSM-13 (mg/L)	BSM-13a (mg/L)
0	120,000	122,000	N/A	120,000	118,000	96,500	119,000	119,000	121,000	118,000	119,000	119,000	104,000	120,000	97,500
1	119,000	117,000	93,300	122,000	120,000	98,300	118,000	117,000	118,000	116,000	115,000	120,000	98,300	119,000	103,000
2	117,000	120,000	93,800	122,000	119,000	98,100	118,000	118,000	118,000	116,000	118,000	121,000	100,000	121,000	99,100
4	119,000	125,000	93,300	122,000	124,000	98,700	120,000	118,000	121,000	117,000	115,000	121,000	98,000	121,000	100,000
8	120,000	123,000	94,600	124,000	126,000	98,900	120,000	118,000	120,000	118,000	115,000	123,000	98,400	123,000	106,000
24	118,000	130,000	93,900	125,000	138,000	101,000	128,000	119,000	118,000	120,000	117,000	129,000	99,500	129,000	108,000
Final	121,000	126,000	N/A	126,000	138,000	99,700	126,000	119,000	120,000	116,000	116,000	129,000	99,900	127,000	107,000

Table A.5.a. Blended Matrix Na Boehmite Leaching Test Results (SWRI SSR# 31241, 31334, and 31084)

Time (hrs)	BSM-14 (mg/L)	BSM-15a (mg/L)	BSM-16a (mg/L)	BSM-17a (mg/L)	BSM-18a (mg/L)	BSM-19a (mg/L)
0	98,800	80,900	76,700	54,200	49,900	53,300
1	95,300	79,100	69,500	50,500	52,500	51,500
2	96,400	80,600	73,800	56,200	52,400	56,100
4	96,600	80,600	75,100	56,600	49,700	57,000
8	97,400	77,800	81,000	57,400	54,800	56,300
24	98,800	78,400	72,900	59,400	56,400	63,600
72	103,000	80,900	77,600	57,100	53,100	57,800
120	98,200	79,600	76,400	57,200	53,100	69,800
168	98,200	101,000	80,500	59,500	46,400	61,200
Final	96,600	81,600	83,300	62,800	59,700	62,100

Table A.6. Aluminate Ion Effect on Boehmite Leaching Test Results (SWRI SSR# 31347, 31421, and 31382)

Time (hrs)	ATM-1		ATM-2		ATM-3		ATM-4		ATM-5	
	Al (mg/L)	Na (mg/L)	Al (mg/L)	Na (mg/L)	Al (mg/L)	Na (mg/L)	Al (mg/L)	Na (mg/L)	Al (mg/L)	Na (mg/L)
0	19,100	123,000	13,000	122,000	3,320	25,200	2,090	24,400	16,100	157,000
1	19,100	125,000	13,100	122,000	3,290	25,200	2,590	24,800	15,200	147,000
2	19,700	127,000	13,400	121,000	3,770	25,500	2,670	24,400	15,600	150,000
4	20,000	127,000	13,500	121,000	3,660	25,700	2,590	24,900	15,700	151,000
8	21,200	127,000	14,300	124,000	4,060	25,700	2,720	24,900	16,000	150,000
24	22,000	129,000	16,700	125,000	4,640	26,400	3,970	25,900	17,600	153,000
Final	22,400	128,000	15,300	126,000	4,610	26,600	3,820	25,900	20,200	175,000

Table A.6.a. Aluminate Ion Effect on Boehmite Leaching Test Results
(SWRI SSR# 31347, 31421, and 31382)

Time (hrs)	ATM-6		ATM-7		ATM-8		ATM-9		ATM-10	
	Al (mg/L)	Na (mg/L)	Al (mg/L)	Na (mg/L)	Al (mg/L)	Na (mg/L)	Al (mg/L)	Na (mg/L)	Al (mg/L)	Na (mg/L)
0	10,500	145,000	3,180	63,900	2,110	61,100	3,790	25,100	3,370	24,900
1	10,500	143,000	3,200	63,400	2,180	63,000	3,390	25,500	3,430	25,200
2	10,700	146,000	3,260	68,400	2,260	63,600	3,470	25,700	3,480	25,500
4	10,700	142,000	3,330	57,100	2,310	62,600	3,510	25,700	3,530	25,300
8	11,500	147,000	3,430	65,200	2,530	63,700	3,630	25,800	3,640	25,400
24	13,100	145,000	3,920	65,800	3,440	65,500	4,330	27,200	4,240	26,700
Final	14,300	161,000	3,890	64,700	3,460	66,300	4,240	27,000	4,250	26,600

Table A.6.b. Aluminate Ion Effect on Boehmite Leaching Test Results
(SWRI SSR# 31347, 31421, and 31382)

Time (hrs)	ATM-11		ATM-12	
	Al (mg/L)	Na (mg/L)	Al (mg/L)	Na (mg/L)
0	9,840	130,000	9,880	133,000
1	9,940	132,000	9,950	133,000
2	9,970	132,000	10,100	135,000
4	10,200	132,000	10,400	134,000
8	10,900	134,000	10,700	134,000
24	12,900	140,000	12,400	135,000
Final	13,000	143,000	12,400	135,000

Table A.7. Al Boehmite Simulant Revision Testing Results (First Set)
(SWRI SRR# 32072, 32104, and 31989)

Time (hrs)	BCT2-1 (mg/L)	BCT2-2 (mg/L)	BCT2-3 (mg/L)	BCT2-4 (mg/L)	BCT2-5 (mg/L)	BCT2-6 (mg/L)	BCT2-7 (mg/L)	BCT2-8 (mg/L)	BCT2-9 (mg/L)	BCT2-10 (mg/L)
0	<2.50	3.60	<2.50	<2.50	<2.50	4.68	<2.50	5.93	9.32	3.47
1	482	2,170	636	2,500	903	3,950	729	311	380	551
2	844	3,390	1,190	3,680	1,560	5,150	1,300	566	748	1,070
4	1,420	4,270	1,980	4,550	2,650	5,590	2,120	1,030	1,370	1,920
8	2,450	4,920	3,220	4,980	4,480	5,840	3,430	1,840	2,360	3,240
24	4,750	5,370	5,350	5,590	6,130	6,560	5,580	3,830	4,510	6,090
Final	4,760	5,370	5,400	5,560	6,420	6,510	5,760	3,830	4,530	6,020

Table A.8. Na Boehmite Simulant Revision Testing Results (First Set)
(SWRI SRR# 32072, 32104, and 31989)

Time (hrs)	BCT2-1 (mg/L)	BCT2-2 (mg/L)	BCT2-3 (mg/L)	BCT2-4 (mg/L)	BCT2-5 (mg/L)	BCT2-6 (mg/L)	BCT2-7 (mg/L)	BCT2-8 (mg/L)	BCT2-9 (mg/L)	BCT2-10 (mg/L)
0	65,200	67,200	109,000	110,000	153,000	155,000	110,000	68,000	113,000	148,000
1	64,400	66,200	109,000	110,000	157,000	154,000	110,000	67,800	112,000	155,000
2	64,200	68,400	110,000	112,000	151,000	155,000	111,000	67,200	113,000	157,000
4	64,200	67,100	106,000	111,000	150,000	156,000	109,000	68,900	113,000	158,000
8	67,000	68,400	111,000	112,000	158,000	158,000	110,000	71,500	114,000	159,000
24	70,500	72,700	112,000	120,000	163,000	169,000	117,000	80,100	121,000	171,000
Final	69,600	72,800	111,000	120,000	168,000	168,000	119,000	79,200	120,000	168,000

Table A.9. Al Boehmite Simulant Revision Testing Results (Second Set)
(SWRI SRR# 32040, 32104)

Time (hrs)	BCT3-1 (mg/L)	BCT3-2 (mg/L)	BCT3-3 (mg/L)	BCT3-4 (mg/L)	BCT3-5 (mg/L)	BCT3-6 (mg/L)
0	<2.50	<2.50	<2.50	<2.50	<2.50	2.84
1	397	1,970	701	2,820	804	4,620
2	698	3,350	1,120	4,400	1,500	5,710
4	1,170	4,680	1,800	5,250	2,640	5,960
8	1,920	5,220	2,830	5,410	4,180	5,970
24	3,890	5,800	4,790	5,820	6,110	6,170
Final	3,880	5,920	4,830	5,810	6,030	6,120

Table A.10. Na Boehmite Simulant Revision Testing Results (Second Set)
(SWRI SRR# 32040, 32104)

Time (hrs)	BCT3-1 (mg/L)	BCT3-2 (mg/L)	BCT3-3 (mg/L)	BCT3-4 (mg/L)	BCT3-5 (mg/L)	BCT3-6 (mg/L)
0	64,800	64,900	108,000	109,000	152,000	155,000
1	65,400	65,300	110,000	106,000	150,000	153,000
2	65,400	65,800	109,000	108,000	154,000	153,000
4	64,500	66,500	107,000	110,000	154,000	155,000
8	66,000	68,000	109,000	112,000	156,000	156,000
24	71,600	74,700	110,000	120,000	168,000	160,000
Final	69,900	75,100	111,000	120,000	164,000	159,000

Appendix B

Leaching Testing Conditions

Appendix B: Testing Conditions

Table B.1. Boehmite Screening Test Leaching Conditions

	CT-B1	**CT-B2**	**CT-B3**	**CT-B4**	**CT-B5**
OH^- conc. (M)	3	3	3	3	3
Leach Temp (°C)	98.1	98.6	98.6	98.3	98.8
Density (g/mL)	1.12	1.12	1.12	1.12	1.12
Boehmite added (g)	8.00	8.00	8.00	8.00	8.01
Caustic soln added (g)	802.09	801.97	802.10	802.02	802.01

Table B.2.a. Boehmite Matrix Test Leaching Conditions

	MTB-1	**MTB-2**	**MTB-3**	**MTB-4**	**MTB-5**	**MTB-6**	**MTB-7**
OH^- conc. (M)	5	5	1	1	5	5	1
Leach Temp (°C)	99.3	98.8	98.4	98.7	60.0	59.3	60.3
Density (g/mL)	1.19	1.19	1.04	1.04	1.19	1.19	1.04
Boehmite added (g)	42.16	21.08	9.53	4.77	25.49	12.74	4.58
Caustic soln added (g)	806.01	806.00	806.01	806.01	806.01	806.02	806.02

Table B.2.b. Boehmite Matrix Test Leaching Conditions

	MTB-8	**MTB-9**	**MTB-10**	**MTB-11**	**MTB-12**	**MTB-13**
OH^- conc. (M)	1	5	1	3	3	3
Leach Temp (°C)	60.2	98.7	60.4	79.9	79.4	78.8
Density (g/mL)	1.04	1.19	1.04	1.12	1.12	1.12
Boehmite added (g)	2.29	42.16	2.29	14.50	14.50	14.50
Caustic soln added (g)	806.01	806.04	806.03	806.03	806.01	806.01

Table B.3. Blended Matrix Boehmite Leaching Test Conditions

	BSM-1	BSM-2	BSM-2a	BSM-5	BSM-6
OH^- conc. (M)	19	19	19	19	19
Leach Temp (°C)	101.5	101.6	98.1	102.0	100.9
Boehmite added (g)	22.73	22.73	22.73	22.73	22.73
Gibbsite added (g)	22.73	22.73	22.74	---	---
Caustic soln added (19M) (g)	200.31	200.29	200.28	200.29	200.27
Condensate added (g)	198.28	274.97	274.97	198.28	274.98
Supernate added (g)	259.91	259.93	259.83	259.84	259.83
Sludge slurry added (g)	89.65	---	---	89.63	---
Sodium oxalate added (g)	6.49	---	---	---	6.50

Table B.3.a. Blended Matrix Boehmite Leaching Test Conditions

	BSM-6a	BSM-7	BSM-8	BSM-9	BSM-10
OH^- conc. (M)	19	19	19	19	19
Leach Temp (°C)	100.7	101.8	103.9	101.3	100.4
Boehmite added (g)	22.73	22.73	22.73	22.73	22.75
Gibbsite added (g)	---	22.73	22.73	22.73	22.74
Caustic soln added (19M) (g)	200.27	200.29	200.28	200.27	200.28
Condensate added (g)	274.98	198.36	198.71	198.32	198.32
Supernate added (g)	259.82	259.84	259.84	259.96	259.83
Sludge slurry added (g)	---	89.66	89.66	89.81	89.67
Sodium oxalate added (g)	6.50	6.50	6.51	6.51	6.50

Table B.3.b. Blended Matrix Boehmite Leaching Test Conditions

	BSM-11	BSM-12	BSM-12a	BSM-13	BSM-13a
OH^- conc. (M)	19	19	19	19	19
Leach Temp (°C)	99.0	99.9	100.9	99.4	101.0
Boehmite added (g)	22.74	22.73	22.74	22.73	22.74
Gibbsite added (g)	22.74	---	---	---	---
Caustic soln added (19M) (g)	200.26	200.28	200.28	200.26	200.27
Condensate added (g)	198.42	274.98	274.93	275.21	274.96
Supernate added (g)	259.81	259.81	259.84	259.84	259.84
Sludge slurry added (g)	89.66	---	---	---	---
Sodium oxalate added (g)	6.50	6.50	6.50	6.50	6.49

Table B.3.c. Blended Matrix Boehmite Leaching Test Conditions

	BSM-14	BSM-15	BSM-15a	BSM-16	BSM-16a
OH^- conc. (M)	19	19	19	19	19
Leach Temp (°C)	96.8	104.5	98.8	100.6	98.6
Boehmite added (g)	24.55	24.57	24.56	24.55	24.56
Gibbsite added (g)	24.55	24.56	24.56	24.56	24.56
Caustic soln added (19M) (g)	152.04	152.06	152.06	152.07	152.07
Condensate added (g)	214.22	214.67	234.67	214.68	234.68
Supernate added (g)	280.85	281.57	280.70	282.39	280.71
Sludge slurry added (g)	96.90	96.43	76.43	96.44	76.45
Sodium oxalate added (g)	7.02	7.01	7.02	7.01	7.02

Table B.3.d. Blended Matrix Boehmite Leaching Test Conditions

	BSM-17	BSM-17a	BSM-18	BSM-18a	BSM-19	BSM-19a
OH^- conc. (M)	19	19	19	19	19	19
Leach Temp (°C)	100.5	99.6	103.2	100.2	102.3	100.0
Boehmite added (g)	24.56	24.56	24.55	24.56	24.55	24.56
Gibbsite added (g)	24.56	24.56	24.56	24.56	24.56	24.56
Caustic soln added (19M) (g)	152.05	152.07	152.05	152.08	152.05	152.07
Condensate added (g)	495.37	515.39	495.38	515.38	495.72	515.38
Supernate added (g)	---	---	---	---	---	---
Sludge slurry added (g)	96.44	76.44	96.42	76.43	96.42	76.44
Sodium oxalate added (g)	7.02	7.02	7.02	7.02	7.02	7.02

Table B.4. Aluminate and Anion Effect on Boehmite Leaching Test Conditions

	ATM-1	ATM-2	ATM-3	ATM-4
OH^- conc. (M)	5	5	1	1
Leach Temp (°C)	101.0	100.8	100.8	100.7
Density (g/mL)	1.19	1.19	1.04	1.04
Boehmite added (g)	9.62	9.46	7.91	7.88
Sodium aluminate added (g)	48.20	---	9.16	6.15
Caustic soln added (g)	919.26	919.24	787.26	787.25
	ATM-5	**ATM-6**	**ATM-7**	**ATM-8**
OH^- conc. (M)	5	5	1	1
Leach Temp (°C)	98.8	100.8	101.8	101.9
Density (g/mL)	1.19	1.19	1.04	1.04
Boehmite added (g)	10.69	10.53	8.98	8.95
Sodium aluminate added (g)	48.20	32.09	9.15	6.16
Caustic soln added (g)	919.27	919.27	787.26	787.25
$Na_2C_2O_4$ added (g)	1.00	1.01	1.01	1.01
Na_3PO_4 added (g)	2.28	2.28	2.28	2.28
Na_2SO_4 added (g)	0.53	0.53	0.53	0.53
Na_2CO_3 added (g)	28.62	28.62	28.62	28.62
$NaNO_3$ added (g)	57.37	57.37	57.37	57.37
$NaNO_2$ (g)	17.08	17.08	17.08	17.08
	ATM-9	**ATM-10**	**ATM-11**	**ATM-12**
OH^- conc. (M)	1	1	5	5
Leach Temp (°C)	93.5	100.5	97.6	100.3
Density (g/mL)	1.04	1.04	1.19	1.19
Boehmite added (g)	7.91	7.91	10.53	10.53
Sodium aluminate added (g)	9.16	9.16	32.09	32.09
Caustic soln added (g)	787.24	787.25	919.25	919.27
$Na_2C_2O_4$ added (g)	---	---	1.00	1.01
Na_3PO_4 added (g)	---	---	2.28	2.28
Na_2SO_4 added (g)	---	---	0.53	0.53
Na_2CO_3 added (g)	---	---	28.62	28.62
$NaNO_3$ added (g)	---	---	57.37	57.37
$NaNO_2$ (g)	---	---	17.08	17.08

Table B.5. Boehmite Simulant Revision Testing Conditions (Set 1)

	BCT2-1	**BCT2-2**	**BCT2-3**	**BCT2-4**	**BCT2-5**	**BCT2-6**	**BCT2-7**	**BCT2-8**	**BCT2-9**	**BCT2-10**
OH^- conc. (M)	3	3	5	5	7	7	5	3	5	7
Leach Temp (°C)	98.3	98.7	98.8	98.7	99.8	101.4	98.4	98.1	97.4	98.3
Density (g/mL)	1.12	1.12	1.19	1.19	1.30	1.30	1.19	1.12	1.19	1.30
Type of Boehmite Used	AOH-60	PNNL G-250	AOH-60	PNNL G-250	AOH-60	PNNL G-250	AOH-20	PNNL C31C	PNNL C31C	PNNL C31C
Boehmite added (g)	8.01	8.00	8.00	8.00	8.01	8.00	8.01	8.00	8.00	8.00
Caustic soln added (g)	802.01	802.01	802.02	802.00	802.02	802.00	802.00	802.02	802.01	802.01

Table B.6. Boehmite Simulant Revision Testing Conditions (Set 2)

	BCT3-1	**BCT3-2**	**BCT3-3**	**BCT3-4**	**BCT3-5**	**BCT3-6**
OH^- conc. (M)	3	3	5	5	7	7
Leach Temp (°C)	98.2	99.1	99.1	99.1	97.8	99.8
Density (g/mL)	1.12	1.12	1.19	1.19	1.30	1.30
Type of Boehmite Used	AOH-30	AOH-180E	AOH-30	AOH-180E	AOH-30	AOH-180E
Boehmite added (g)	8.00	8.00	8.00	8.00	8.00	8.00
Caustic soln added (g)	802.02	802.01	802.00	802.00	802.01	802.03

Appendix C

Data from Solutions (CBM-1 through CBM-5)

Appendix C: Data from Solutions (CBM-1 through CBM-5)

Table C.1. Data from Solutions (CBM-1)

Sample ID	CBM-DW-FIL (mg/kg soln)	CBM-DW-FIL (mg/kg soln)	CBM-LE-R-0 (mg/kg soln)	CBM-LE-R-2 (mg/kg soln)	CBM-LE-R-4 (mg/kg soln)	CBM-LE-S-0 (mg/kg soln)	CBM-LE-S-2 (mg/kg soln)	CBM-LE-S-4 (mg/kg soln)	CBM-LE-S-6 (mg/kg soln)	CBM-LE-S-8 (mg/kg soln)	CBM-LE-S-10 (mg/kg soln)	CBM-LE-S-12 (mg/kg soln)
Time	**0.00**	**6.00**	**7.20**	**9.18**	**11.20**	**12.55**	**14.55**	**16.55**	**18.55**	**20.45**	**22.53**	**24.53**
Aluminum	3,600	3,600	2,990	8,420	9,960	10,200	10,600	10,800	11,300	12,000	12,600	12,800
Calcium	<1.65	<1.65	<2.73	<1.99	<2.02	<2.47	<2.59	<2.25	<2.06	<2.03	<2.05	<2.13
Chromium	35.1	35.1	33.4	107	248	316	342	343	352	369	375	381
Iron	<3.30	<3.30	13.7	16	14.6	15.1	17	10.7	12.3	15.1	14.1	16.1
Lead	1.3	1.3	9.84	16.2	27.2	36.9	36.9	34.1	36.4	38.3	37.3	37.2
Magnesium	<1.65	<1.65	<2.73	<1.99	<2.02	<2.47	<2.59	<2.25	<2.06	<2.03	<2.05	<2.13
Manganese	<0.165	<0.165	0.295	4.64	10.6	15.8	12.6	9.14	11	12	12.4	12.9
Neodymium	<0.165	<0.165	<0.273	<0.199	<0.202	<0.247	<0.259	<0.225	<0.206	<0.203	<0.205	<0.213
Nickel	<0.165	<0.165	<0.273	<0.199	<0.202	<0.247	<0.259	<0.225	<0.206	<0.203	<0.205	<0.213
Potassium	1,690	1,690	739	738	741	745	730	722	744	772	740	743
Sodium	87,300	87,300	108,000	106,000	107,000	107,000	107,000	105,000	105,000	108,000	105,000	106,000
Zirconium	0.802	0.802	5.99	8.46	8.2	7.66	6.44	5.97	5.64	5.9	5.47	5.46
Nitrate	22,800	22,800	8,120	8,270	8,240	8,200	8,340	8,240	8,350	8,620	8,190	8,180
Nitrite	6,920	6,920	2,390	2,400	2,430	2,400	2,440	2,420	2,450	2,510	2,420	2,390
Oxalate	991	991	403	526	702	854	785	808	837	839	846	840
Phosphate	339	339	<110	84.5	92.3	<100	<105	<94.9	<86.5	<85.9	<82.9	<85.9
Sulfate	882	882	306	295	287	289	311	305	338	316	310	303
< is below quantifiable limits.												

Table C.1 (Contd)

Sample ID	**CBM-LE-C-2 (mg/kg soln)**	**CBM-LE-C-4 (mg/kg soln)**	**CBM-LE-C-6 (mg/kg soln)**	**CBM-LE-C-8 (mg/kg soln)**	**CBM-LE-C-10 (mg/kg soln)**	**CBM-LE-C-12 (mg/kg soln)**	**CBM-LC-FIL (mg/kg soln)**
Time (hrs)	**26.53**	**28.53**	**30.53**	**32.53**	**34.53**	**36.63**	**44.67**
Aluminum	10,600	13,200	13,100	13,200	13,400	13,300	13,300
Calcium	<2.59	<2.10	<2.00	<1.89	<1.91	2.93	<1.39
Chromium	342	396	391	391	399	397	638
Iron	17	12.6	10.6	8.84	9.09	6.84	9.08
Lead	36.9	29.1	24.9	21.7	18.9	16	13.4
Magnesium	<2.59	<2.10	<2.00	<1.89	<1.91	<2.12	<1.39
Manganese	12.6	10.6	8.78	7.21	5.81	4.16	0.796
Neodymium	<0.259	<0.210	<0.200	<0.189	<0.191	<0.212	<0.139
Nickel	<0.259	<0.210	<0.200	<0.189	<0.191	<0.212	<0.139
Potassium	730	736	733	739	747	732	791
Sodium	107,000	107,000	105,000	106,000	107,000	107,000	106,000
Zirconium	6.44	4.63	4.28	3.89	3.7	3.39	3.88
Nitrate	8,270	8,240	8,240	8,230	8,330	8,250	8,290
Nitrite	2,440	2,430	2,400	2,420	2,440	2,410	2,740
Oxalate	727	731	591	578	459	465	376
Phosphate	<84	<85.2	83.8	<77.2	<76.9	<86.8	102
Sulfate	306	310	499	302	302	303	329
< is below quantifiable limits.							

Table C.1 (Contd)

Sample ID	CBM-W1-FIL (mg/kg soln)	CBM-W2-FIL (mg/kg soln)	CBM-W3-FIL (mg/kg soln)	CBM-W4-FIL (mg/kg soln)	CBM-W5-FIL (mg/kg soln)	CBM-W6-FIL (mg/kg soln)	CBM-OL-05 (mg/kg soln)	CBM-OL-1 (mg/kg soln)	CBM-OL-2 (mg/kg soln)	CBM-OL-4 (mg/kg soln)	CBM-OL-6 (mg/kg soln)
Time (hrs)	**46.22**	**50.58**	**55.55**	**57.45**	**59.33**	**60.13**	**65.25**	**65.75**	**66.75**	**68.75**	**70.75**
Aluminum	6,760	1,690	779	423	225	117	187	190	197	204	214
Calcium	<1.14	<1.21	<1.27	<1.39	<1.38	<1.35	<2.81	<2.40	<2.65	<2.96	<2.30
Chromium	352	99.8	46.7	27.2	16.7	10.7	250	255	268	277	281
Iron	4.04	<2.43	<2.54	<2.78	<2.75	<2.69	<5.62	<4.81	<5.29	<5.93	<4.61
Lead	3.33	0.541	0.138	<0.139	<0.138	<0.135	0.363	0.446	0.348	0.319	0.242
Magnesium	<1.14	<1.21	<1.27	<1.39	<1.38	<1.35	<2.81	<2.40	<2.65	<2.96	<2.30
Manganese	0.158	<0.121	<0.127	<0.139	<0.138	<0.135	332	299	249	171	123
Neodymium	<0.114	<0.121	<0.127	<0.139	<0.138	<0.135	<0.281	<0.240	<0.265	<0.296	<0.230
Nickel	<0.114	<0.121	<0.127	<0.139	<0.138	<0.135	<0.281	<0.240	<0.265	<0.296	<0.230
Potassium	404	111	52.6	26.2	14.2	<8.08	715	706	711	706	704
Sodium	67,900	32,000	20,900	11,600	5,940	2,930	4,180	3,780	3,710	3,800	5,420
Zirconium	1.47	0.291	<0.127	<0.139	<0.138	<0.135	<0.281	<0.240	<0.265	<0.296	<0.230
Nitrate	4320	862	382	200	118	67.3	131	115	123	129	114
Nitrite	1250	274	134	81.2	56.9	<53.9	<114	<96.2	<106	<119	<92.2
Oxalate	1,140	4,100	8,000	4,750	2,360	1,020	1,150	1,100	1,110	1,160	1,150
Phosphate	<48.3	<49.9	<51.7	<56.3	<55.3	<53.9	<114	<96.2	<106	<119	<92.2
Sulfate	159	<49.9	<51.7	<56.3	<55.3	<53.9	<114	<96.2	<106	<119	<92.2
< is below quantifiable limits.											

Table C.2. Data from Solutions (CBM-2)

Sample ID	**CBM25-DW-FIL (mg/kg soln)**	**CBM25-DW-FIL (mg/kg soln)**	**CBM25-LE-R-0 (mg/kg soln)**	**CBM25-LE-R-2 (mg/kg soln)**	**CBM25-LE-R-4 (mg/kg soln)**	**CBM25-LE-S-0 (mg/kg soln)**	**CBM25-LE-S-2 (mg/kg soln)**	**CBM25-LE-S-4 (mg/kg soln)**	**CBM25-LE-S-6 (mg/kg soln)**	**CBM25-LE-S-8 (mg/kg soln)**	**CBM25-LE-S-10 (mg/kg soln)**	**CBM25-LE-S-12 (mg/kg soln)**
Time (hrs)	**0.00**	**0.00**	**18.77**	**21.02**	**26.27**	**27.67**	**29.60**	**31.60**	**33.60**	**35.60**	**37.60**	**39.60**
Aluminum	2,470	2,470	2,990	3,780	10,900	11,000	11,300	11,500	11,700	11,900	13,200	12,900
Calcium	<1.26	<1.26	<1.77	<1.73	<1.96	2.17	2.5	2.15	2.19	1.97	2.13	1.8
Chromium	19	19	26.1	37.8	235	328	354	363	373	380	399	399
Iron	<1.26	<1.26	13	11.7	14.6	18	16.9	14.7	13.8	14.1	13.9	11.4
Lead	0.38	0.38	7.74	7.66	26.1	41	46.2	41.7	36.4	36.3	38.8	32.1
Magnesium	<1.26	<1.26	<1.77	<1.73	<1.96	<1.71	<1.76	<1.52	<1.38	<1.65	<1.71	<1.49
Manganese	<0.126	<0.126	<0.177	0.214	12.5	18	15.3	12.8	12.1	11.7	11.5	9.96
Nickel	<0.126	<0.126	<0.177	<0.173	<0.196	<0.171	<0.176	<0.152	<0.138	<0.165	<0.171	<0.149
Potassium	1,710	1,710	634	621	634	630	678	629	635	668	654	623
Sodium	82,300	82,300	103,000	103,000	103,000	106,000	102,000	104,000	102,000	103,000	105,000	104,000
Zirconium	0.477	0.477	6.33	6.4	10.2	9.63	8.44	7.41	6.83	6.47	6.55	5.93
Nitrate	25,700	25,700	7,750	7,790	7,750	7,710	7,700	7,840	7,790	7,750	7,790	7,900
Nitrite	7,580	7,580	2,460	2,470	2,460	2,440	2,460	2,440	2,410	2,400	2,450	2,460
Oxalate	718	718	292	291	633	836	928	814	697	688	777	627
Phosphate	412	412	134	129	129	127	126	128	128	116	114	118
Sulfate	932	932	297	305	303	299	299	320	303	301	314	313

< is below quantifiable limits.

Table C.2 (Contd)

Sample ID	CBM25-LE-C-0 (mg/kg soln)	CBM25-LE-C-2 (mg/kg soln)	CBM25-LE-C-4 (mg/kg soln)	CBM25-LE-C-6 (mg/kg soln)	CBM25-LE-C-8 (mg/kg soln)	CBM25-LE-C-10 (mg/kg soln)	CBM25-LE-C12 (mg/kg soln)	CBM25-LC-FIL (mg/kg soln)
Time (hrs)	**39.77**	**41.77**	**43.77**	**45.77**	**47.77**	**49.82**	**51.77**	**64.37**
Aluminum	13,400	13,300	13,300	12,800	13,100	13,100	13,700	12,600
Calcium	1.7	1.66	<1.62	<1.69	<1.76	<1.79	<2.01	<1.28
Chromium	398	403	409	395	398	402	404	486
Iron	10.7	10.9	10.2	8.03	8.48	7.42	7.15	5.45
Lead	32.5	33.5	24.2	23.5	21	17.7	15.4	5.22
Magnesium	<1.58	<1.61	<1.62	<1.69	<1.76	<1.79	<2.01	<1.28
Manganese	9.8	8.09	7.28	6.22	5.71	4.21	3.13	0.224
Nickel	<0.158	<0.161	<0.162	<0.169	<0.176	<0.179	<0.201	<0.128
Potassium	631	648	631	597	593	612	608	585
Sodium	105,000	102,000	102,000	96,700	97,600	98,500	102,000	97,600
Zirconium	5.93	5.67	4.94	4.43	4.21	3.86	3.69	2.68
Nitrate	7,780	7,790	7,730	7,540	7,400	7,340	7,320	7,650
Nitrite	2,470	2,460	2,410	2,400	2,360	2,320	2,330	2,370
Oxalate	655	613	510	514	511	472	407	295
Phosphate	112	121	110	107	101	102	100	115
Sulfate	299	313	300	299	290	294	286	295

< is below quantifiable limits.

Table C.2 (Contd)

Sample ID	**CBM25-W1-FIL (mg/kg soln)**	**CBM25-W2-FIL (mg/kg soln)**	**CBM25-W3-FIL (mg/kg soln)**	**CBM-W4-FIL (mg/kg soln)**	**CBM-W5-FIL (mg/kg soln)**	**CBM-W6-FIL (mg/kg soln)**	**CBM-OL-05 (mg/kg soln)**	**CBM-OL-1 (mg/kg soln)**	**CBM-OL-2 (mg/kg soln)**	**CBM-OL-4 (mg/kg soln)**	**CBM-OL-6 (mg/kg soln)**	**CBM25-W3-OL (CBM25-WS-SUP) (mg/kg soln)**	**CBM-FSC-FIL (mg/kg soln)**
Time (hrs)	**68.58**	**71.60**	**72.85**	**74.35**	**76.57**	**77.08**	**80.27**	**80.77**	**81.77**	**83.77**	**85.77**	**92.68**	**95.57**
Aluminum	6,430	3,060	1,810	746	329	150	172	171	176	176	180	191	29.5
Calcium	<1.21	<1.19	<1.30	<1.56	<1.58	<1.53	<2.11	<2.09	<2.15	<2.15	<1.88	<1.39	<1.32
Chromium	287	154	91.5	42.9	23.9	12	387	397	415	419	425	142	47.1
Iron	2.88	1.2	<1.30	<1.56	<1.58	<1.53	<2.11	<2.09	<2.15	<2.15	<1.88	1.86	8.73
Lead	1.48	0.372	0.137	<0.156	<0.158	<0.153	0.503	0.619	0.508	0.461	0.457	<0.139	0.395
Magnesium	<1.21	<1.19	<1.30	<1.56	<1.58	<1.53	<2.11	<2.09	<2.15	<2.15	<1.88	<1.39	<1.32
Manganese	<0.121	<0.119	<0.130	<0.156	<0.158	<0.153	289	253	212	159	124	0.359	1.8
Nickel	<0.121	<0.119	<0.130	<0.156	<0.158	<0.153	<0.211	<0.209	<0.215	<0.215	<0.188	<0.139	0.279
Potassium	326	148	88.6	47.4	19.5	14.9	23	30.7	18.9	22	22.5	14.3	<7.94
Sodium	67,100	39,300	25,600	15,400	7,350	3,510	4,760	4,750	4,820	4,720	4,730	3,910	612
Zirconium	1.27	0.456	0.304	<0.156	<0.158	<0.153	<0.211	<0.209	<0.215	<0.215	<0.188	<0.139	<0.132
Nitrate	87	1,740	917	369	171	84.8	100	102	101	98.2	97.7	89.8	20.6
Nitrite	40.8	514	278	122	63.7	36.9	39.4	40.2	42.1	44.2	45.4	38.5	16.8
Oxalate	<40.0	3,010	5,710	9,490	4,360	<30.7	1,900	1,920	1,920	1,880	1,900	2,050	<27.6
Phosphate	<40.0	<24.7	<26.6	<31.3	<31.9	<30.7	<42.3	<43.0	<43.0	<44.3	<42.5	<28.0	<27.6
Sulfate	1,370	68.2	37	<31.3	<31.9	1,480	<42.3	<43.0	<43.0	<44.3	<42.5	<28.0	60.3

< is below quantifiable limits.

Table C.3. Data from Solutions (CBM-3)

Sample ID	CBM3-DW-FIL (mg/L soln)	CBM3-LE-R-0 (mg/kg soln)	CBM3-LE-R-2 (mg/kg soln)	CBM3-LE-R-4 (mg/kg soln)	CBM3-LE-S-0 (mg/kg soln)	CBM3-LE-S-2 (mg/kg soln)	CBM3-LE-S-4 (mg/kg soln)	CBM3-LE-S-6 (mg/kg soln)	CBM3-LE-S-8 (mg/kg soln)	CBM3-LE-S-10 (mg/kg soln)	CBM3-LE-S-12 (mg/kg soln)	CBM3-LE-C-0 (mg/kg soln)
Time	**0.00**	**1.33**	**3.33**	**5.33**	**6.67**	**8.67**	**10.67**	**12.67**	**14.67**	**16.67**	**18.67**	**18.75**
Aluminum	2,920	1,729	6,170	6,880	7,170	7,890	8,400	9,420	9,610	10,100	10,700	11,000
Calcium	[4.4]	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	5.43
Chromium	7.62	3.50	8.13	13.20	16.20	18.10	18.80	19.80	20.00	20.30	21.00	20.50
Iron	[1.2]	25.0	34.2	37.1	43.5	39.8	64.4	28.8	29.6	35.5	34.4	33.4
Lead	[7.9]	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	63.5
Magnesium	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	<1.23
Manganese	[0.082]	<0.14	0.52	2.13	4.69	4.99	10.8	3.23	3.36	4.58	4.11	4.23
Nickel	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	<0.123
Potassium	1,970	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	577
Sodium	107,000	144,000	142,000	144,000	145,000	143,000	139,000	142,000	141,000	143,000	142,000	125,000
Zirconium	[0.30]	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	10.8
Nitrate	24,100	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
Nitrite	7,560	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
Oxalate	795	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
Phosphate	439	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
Sulfate	889	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA

[ ] is below quantifiable limits but above method detection limits.
NA = not analyzed

Table C.3 (Contd)

Sample ID	CBM3-LE-C-2 (mg/kg soln)	CBM3-LE-C-4 (mg/kg soln)	CBM3-LE-C-6 (mg/kg soln)	CBM3-LE-C-8 (mg/kg soln)	CBM3-LE-C-10 (mg/kg soln)	CBM3-LE-C-12 (mg/kg soln)	CBM3-LC-FIL (mg/L soln)	CBM3-W-1 (mg/kg soln)	CBM3-W-2 (mg/kg soln)	CBM3-W-3 (mg/kg soln)	CBM3-W-4 (mg/kg soln)	CBM3-W-5 (mg/kg soln)
Time	**20.75**	**22.75**	**24.75**	**26.75**	**28.75**	**30.75**	**58.25**	**60.27**	**61.10**	**61.88**	**62.67**	**63.15**
Aluminum	11,100	11,200	11,300	11,100	11,300	11,200	14,100	7,950	6,110	4,000	2,790	1,900
Calcium	4.93	4.28	3.88	3.24	2.92	2.93	[4.3]	<1.03	<2.61	<1.34	<1.62	<1.15
Chromium	20.9	20.9	21.1	21.0	21.1	20.8	26.3	NA	NA	NA	NA	NA
Iron	29.2	25.0	23.6	20.8	18.5	16.8	14.8	5.82	2.93	2.14	<1.62	<1.15
Lead	62.1	54.9	52.8	45.5	44.4	41.3	[53]	9.19	3.67	1.18	0.54	0.16
Magnesium	<1.21	<1.21	<1.16	<1.22	<1.18	<1.16	NA	<1.03	<2.61	<1.34	<1.62	<1.15
Manganese	3.69	2.75	2.69	2.03	1.83	1.60	[0.49]	<0.103	<0.261	<0.134	<0.162	<0.115
Nickel	<0.121	<0.121	<0.116	<0.122	<0.118	<0.116	NA	<0.103	<0.261	<0.134	<0.162	<0.115
Potassium	586	579	568	570	593	581	691	504	338	227	152	105
Sodium	126,000	124,000	127,000	126,000	126,000	126,000	160,000	89,800	69,500	45.400	31,900	24,400
Zirconium	10.80	9.80	9.30	8.58	8.28	7.96	4.78	3.29	1.76	0.81	0.36	0.21
Nitrate	NA	NA	NA	NA	NA	NA	4,540	3,720	2,790	1,950	1,300	895
Nitrite	NA	NA	NA	NA	NA	NA	1,570	1,270	837	598	392	268
Oxalate	NA	NA	NA	NA	NA	NA	136	448	982	2,010	3,290	6,060
Phosphate	NA	NA	NA	NA	NA	NA	102	79.4	54.8	36.6	25.0	16.5
Sulfate	NA	NA	NA	NA	NA	NA	185	153	101	72.4	51.6	32.4

[ ] is below quantifiable limits but above method detection limits.
NA= not analyzed.

Table C.3 (Cont)

Sample ID	**CBM3-W-6 (mg/kg soln)**	**CBM3-W-7 (mg/kg soln)**	**CBM3-W-8 (mg/kg soln)**	**CBM3-W-9 (mg/kg soln)**	**CBM3-W-10 (mg/kg soln)**	**CBM3-W-11 (mg/kg soln)**	**CBM3-W-12 (mg/kg soln)**	**CBM3-W-13 (mg/kg soln)**	**CBM3-W-14 (mg/kg soln)**	**CBM3-W-15 (mg/kg soln)**	**CBM3-W-16 (mg/kg soln)**	**CBM3-OL-05 (mg/kg soln)**
Time	**63.65**	**64.15**	**64.63**	**65.12**	**65.57**	**66.00**	**66.40**	**66.87**	**67.30**	**67.87**	**68.33**	**76.17**
Aluminum	1,220	823	541	371	253	177	122	87	61	45	33	44
Calcium	<1.88	<1.72	<1.51	<1.55	<1.38	<1.52	<1.54	<1.59	<1.62	<1.5	<1.21	1.87
Chromium	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
Iron	<1.88	2.05	<1.51	<1.55	<1.38	<1.52	<1.54	<1.59	<1.62	<1.5	<1.21	1.87
Lead	0.19	<0.172	<0.151	<0.155	<0.138	<0.152	<0.154	<0.159	<0.162	<0.15	<0.121	0.20
Magnesium	<1.88	<1.72	<1.51	<1.55	<1.38	<1.52	<1.54	<1.59	<1.62	<1.5	<1.21	2.04
Manganese	<0.188	<0.172	<0.151	<0.155	<0.138	<0.152	<0.154	<0.159	<0.162	<0.15	<0.121	3,470
Nickel	<0.188	<0.172	<0.151	<0.155	<0.138	<0.152	<0.154	<0.159	<0.162	<0.15	<0.121	0.20
Potassium	63.4	40.6	30.7	20.4	14.6	<9.1	<9.21	<9.56	<9.72	<8.97	<7.28	49
Sodium	17,600	14,800	12,600	10,200	7,020	5,250	3,630	2,520	1,800	1,340	1,020	10,000
Zirconium	<0.188	<0.172	<0.151	<0.155	<0.138	<0.152	<0.154	<0.159	<0.162	<0.15	<0.121	0.20
Nitrate	530	351	225	157	104	73.1	52.7	39.5	30.9	24.3	17.7	679
Nitrite	167	114	76.3	54.7	38.1	29.5	23.3	19.1	16.8	<15	<12.1	<11.4
Oxalate	8,640	11,900	14,800	13,100	8,520	5,980	3,930	2,560	1,660	1,110	720	151
Phosphate	<19	<17.4	<15.2	<15.7	<13.9	<15.2	<15.4	<16	<16.2	<15	<12.1	<11.4
Sulfate	23.5	<17.4	<15.2	<15.7	<13.9	<15.2	<15.4	<16	<16.2	<15	<12.1	97.7

< is below quantifiable limits.
NA= not analyzed.

Table C.3 (Contd)

Sample ID	**CBM3-OL-1 (mg/kg soln)**	**CBM3-OL-2 (mg/kg soln)**	**CBM3-OL-4 (mg/kg soln)**	**CBM3-OL-6 (mg/kg soln)**	**CBM3-OL-FIL (mg/kg soln)**	**CBM3-OW-1 (mg/kg soln)**	**CBM3-OW-2 (mg/kg soln)**	**CBM3-OW-3 (mg/kg soln)**	**CBM3-OW-4 (mg/kg soln)**	**CBM3-OW-5 (mg/kg soln)**	**CBM3-OW-6 (mg/kg soln)**	**CBM3-OW-7 (mg/kg soln)**
Time	**77.13**	**78.05**	**79.67**	**81.67**	**82.05**	**83.2**	**83.37**	**83.53**	**83.68**	**83.83**	**84.00**	**84.18**
Aluminum	24	30	44	39	77	46.3	36.6	29.3	23.7	19.7	16.4	15.4
Calcium	3.77	3.08	<1.87	6.80	2.75	2.64	2.36	2.50	2.04	1.88	2.43	2.12
Chromium	3,950	4,090	5,150	4,210	3,940	4,110	3,220	2,480	1,900	1,480	1,140	855
Iron	2.00	<1.99	4.88	4.20	4.12	2.31	<1.59	1.88	<1.59	<1.17	<1.88	<1.73
Lead	<0.189	<0.199	<0.204	<0.223	<0.127	<0.116	<0.159	<0.153	<0.159	<0.117	<0.188	<0.173
Magnesium	<1.89	<1.99	<2.04	<2.23	<1.27	1.86	1.59	<1.53	<1.59	<1.17	<1.88	<1.73
Manganese	3,410	3,090	3,470	2,720	2,650	<0.116	<0.159	<0.153	<0.159	<0.117	<0.188	<0.173
Nickel	<0.189	<0.199	<0.204	<0.223	<0.127	<0.116	<0.159	<0.153	<0.159	<0.117	<0.188	<0.173
Potassium	55.7	42.8	49.0	35.7	33.6	15.3	10.4	<9.16	<9.55	<7.01	<11.3	<10.4
Sodium	8,060	8,130	10,000	8,170	7,860	5,780	4,670	3,700	2,940	2,340	1,840	1,480
Zirconium	<0.189	<0.199	<0.204	<0.204	0.20	<0.116	<0.144	<0.143	<0.159	<0.117	<0.178	<0.173
Nitrate	699	697	884	711	668	558	413	321	246	189	149	115
Nitrite	<11.4	<12	<12.4	<13.5	<13	13.4	<16	<15.4	<16	<11.7	<18.9	<17.3
Oxalate	148	148	193	165	137	96.8	78.6	63.1	50.8	38.4	33.5	27.1
Phosphate	<11.4	<12	<12.4	<13.5	<13	<11.7	<16	<15.4	<16	<11.7	<18.9	<17.3
Sulfate	84.4	80.7	94.6	76.3	75.4	43.5	32.8	26.5	21.1	16.7	<18.9	<17.3

< is below quantifiable limits.

Table C.3 (Contd)

Sample ID	CBM3-OW-8 (mg/kg soln)	CBM3-OW-9 (mg/kg soln)	CBM3-OW-10 (mg/kg soln)	CBM3-OW-11 (mg/kg soln)	CBM3-OW-12 (mg/kg soln)	CBM3-OW-13 (mg/kg soln)	CBM-FSC-FIL (mg/kg soln)
Time	**84.38**	**84.60**	**84.80**	**85.02**	**85.25**	**85.52**	**86.10**
Aluminum	10.9	10.5	9.03	7.34	9.31	6.26	8.0
Calcium	<1.6	1.65	<1.87	<1.49	<1.67	<1.26	<1.54
Chromium	719	503	375	287	222	170	170
Iron	<1.6	<1.26	<1.87	<1.49	<1.67	<1.26	<1.54
Lead	<0.16	<0.126	<0.187	<0.149	<0.167	<0.126	<0.154
Magnesium	<1.6	<1.26	<1.87	<1.49	<1.67	<1.26	<1.54
Manganese	<0.16	<0.126	<0.187	<0.149	<0.167	<0.126	<0.154
Nickel	<0.16	<0.126	<0.187	<0.149	<0.167	<0.126	<0.154
Potassium	<9.57	<7.57	<11.2	<8.95	<10	<7.53	<9.23
Sodium	1,250	1,030	849	724	634	560	534
Zirconium	<0.16	<0.126	<0.187	<0.149	<0.167	<0.126	<0.154
Nitrate	92.1	68.9	59.0	44.7	39.2	29.5	30.1
Nitrite	<16	<12.6	<18.7	<15	<16.7	<12.6	<15
Oxalate	23.0	<12.6	<18.7	<15	<16.7	<12.6	<15
Phosphate	<16	<12.6	<18.7	<15	<16.7	<12.6	<15
Sulfate	<16	<12.6	<18.7	<15	<16.7	<12.6	<15

< is below quantifiable limits.

Table C.4. Data from Solutions (CBM-4)

Sample ID	CBM4-DW-FIL (mg/kg soln)	CBM4-LE-R-0 (mg/kg soln)	CBM4-LE-R-2 (mg/kg soln)	CBM4-LE-R-4 (mg/kg soln)	CBM4-LE-S-0 (mg/kg soln)	CBM4-LE-S-2 (mg/kg soln)	CBM4-LE-S-4 (mg/kg soln)	CBM4-LE-S-6 (mg/kg soln)	CBM4-LE-S-8 (mg/kg soln)	CBM4-LE-S-10 (mg/kg soln)	CBM4-LE-S-12 (mg/kg soln)	CBM4-LE-C-0 (mg/kg soln)
Time	**0.00**	**14.38**	**16.47**	**18.97**	**19.87**	**21.87**	**23.87**	**25.90**	**27.87**	**29.87**	**31.87**	**31.87**
Aluminum	3,100	2,920	5,540	6,760	6,930	7,690	8,220	8,870	9,550	10,100	10,500	10,500
Calcium	<2.14	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
Chromium	0.966	0.60	0.85	1.54	1.63	1.92	1.96	2.06	2.06	2.25	2.24	2.24
Iron	2.14	23.6	26.6	35.8	33.3	33.1	31.2	27.1	30.3	30.9	28.4	28.4
Lead	1.50	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
Magnesium	<2.14	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
Manganese	<0.107	<0.13	<0.129	<0.131	<0.139	0.28	0.57	0.497	0.593	0.48	0.352	0.352
Nickel	<0.107	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
Potassium	375	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
Sodium	96,900	145,000	142,000	143,000	141,000	144,000	142,000	142,000	142,000	144,000	142,000	142,000
Zirconium	0.00	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
Nitrate	16,500	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
Nitrite	4,820	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
Oxalate	3,270	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
Phosphate	1,590	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
Sulfate	13,000	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA

< is below quantifiable limits.
NA= not analyzed.

Table C.4 (Contd)

Sample ID	CBM4-LE-C-2 (mg/kg soln)	CBM4-LE-C-4 (mg/kg soln)	CBM4-LE-C-6 (mg/kg soln)	CBM4-LE-C-8 (mg/kg soln)	CBM4-LE-C-10 (mg/kg soln)	CBM4-LE-C-12 (mg/kg soln)	CBM4-LC-FIL (mg/kg soln)	CBM4-W-1 (mg/kg soln)	CBM4-W-2 (mg/kg soln)	CBM4-W-3 (mg/kg soln)	CBM4-W-4 (mg/kg soln)	CBM4-W-5 (mg/kg soln)
Time	**33.93**	**35.93**	**37.93**	**39.93**	**41.93**	**43.97**	**60.72**	**61.70**	**62.32**	**62.85**	**63.37**	**63.75**
Aluminum	11,000	11,000	10,900	11,000	11,100	11,000	11,600	8,940	7,140	5,410	4,130	2,980
Calcium	1.78	1.97	1.79	1.91	1.32	1.40	<2.3	<1.12	M1.49	<1.42	<1.41	<1.63
Chromium	2.33	2.32	2.36	2.39	2.32	2.31	2.50	1.94	1.45	1.11	0.913	0.691
Iron	24.8	24.1	21.4	20.4	16.8	15.2	12.8	5.02	7.93	<1.42	<1.41	2.40
Lead	55.6	52.4	48.0	48.2	43.4	40.0	24.7	11.7	4.22	1.95	0.874	0.358
Magnesium	<1.19	<1.27	<1.25	<1.16	<1.17	<1.14	<2.3	<1.12	<1.49	<1.42	<1.41	<1.63
Manganese	0.269	0.248	0.194	0.157	<0.117	0.131	<0.115	<0.112	<0.149	<0.142	<0.141	<0.163
Nickel	<0.119	<0.127	<0.125	<0.116	<0.117	<0.114	<0.115	<0.112	<0.149	<0.142	<0.141	<0.163
Potassium	189	178	183	186	171	180	233	169	133	96.5	74.7	50.1
Sodium	126,000	124,000	124,000	126,000	126,000	126,000	142,000	95,100	73,300	55,500	42,700	33,300
Zirconium	11.5	10.8	10.1	9.83	9.36	8.95	NA	5.93	3.63	1.95	0.978	0.578
Nitrate	NA	NA	NA	NA	NA	NA	4,070	3,660	2,610	1,990	1,470	1,040
Nitrite	NA	NA	NA	NA	NA	NA	1,450	1,310	829	621	453	321
Oxalate	NA	NA	NA	NA	NA	NA	122	338	755	1,420	2,460	3,870
Phosphate	NA	NA	NA	NA	NA	NA	385	358	233	176	130	90.9
Sulfate	NA	NA	NA	NA	NA	NA	3,350	3,160	2,030	1,540	1,140	836

< is below quantifiable limits.
NA= not analyzed.

Table C.4 (Cont)

Sample ID	CBM4-W-6 (mg/kg soln)	CBM4-W-7 (mg/kg soln)	CBM4-W-8 (mg/kg soln)	CBM4-W-9 (mg/kg soln)	CBM4-W-10 (mg/kg soln)	CBM4-W-11 (mg/kg soln)	CBM4-W-12 (mg/kg soln)	CBM4-W-13 (mg/kg soln)	CBM4-W-14 (mg/kg soln)	CBM4-W-15 (mg/kg soln)	CBM4-W-16 (mg/kg soln)	CBM4-OL-05 (mg/kg soln)
Time	**64.08**	**64.40**	**64.72**	**64.98**	**65.30**	**65.62**	**65.95**	**66.22**	**66.47**	**66.70**	**67.00**	**73.80**
Aluminum	2,160	1,530	1,130	809	577	411	299	220	158	116	85.7	24.7
Calcium	<2.06	<1.5	<1.77	1.82	<1.86	<1.66	<1.75	<1.72	<1.77	<1.49	<1.8	3.77
Chromium	0.432	0.418	0.329	0.238	<0.186	<0.166	<0.175	<0.172	<0.177	<0.149	<0.18	5,710
Iron	<2.06	<1.5	<1.77	<1.57	3.82	<1.66	<1.75	<1.72	<1.77	<1.49	<1.8	4.59
Lead	<0.206	<0.15	<0.177	<0.157	<0.186	<0.166	<0.175	<0.172	<0.177	<0.149	<0.18	<0.269
Magnesium	<2.06	<1.5	<1.77	<1.57	<1.86	<1.66	<1.75	<1.72	<1.77	<1.49	<1.8	<2.69
Manganese	<0.206	<0.15	<0.177	<0.157	<0.186	<0.166	<0.175	<0.172	<0.177	<0.149	<0.18	1,610
Nickel	<0.206	<0.15	<0.177	<0.157	<0.186	<0.166	<0.175	<0.172	<0.177	<0.149	<0.18	<0.269
Potassium	36.8	29.4	19.0	14.3	14.2	<9.97	<10.5	<10.3	<10.6	<8.92	<10.8	49.9
Sodium	25,300	19,900	16,700	14,700	13,200	10,300	8,680	6,210	4,490	3,410	2,520	7,690
Zirconium	0.268	<0.15	<0.177	0.184	<0.187	<0.167	<0.176	<0.17	<0.178	<0.142	<0.168	<0.269
Nitrate	735	528	378	264	196	138	109	82.3	61.8	46.9	39.9	564
Nitrite	233	169	126	89.6	69.7	50.7	41.7	33.7	27.4	21.2	20.6	<16.2
Oxalate	6,040	8,380	10,800	13,600	15,800	12,600	9,670	6,680	5,060	3,270	2,480	376
Phosphate	66.1	49.5	36.7	25.6	19.8	<16.8	17.6	<17.3	<17.8	<14.9	<18.1	<16.2
Sulfate	607	430	317	218	160	112	87.1	64.4	46.8	34.5	25.1	126
< is below quantifiable limits.												

Table C.4 (Contd)

Sample ID	CBM4-OL-1 (mg/kg soln)	CBM4-OL-2 (mg/kg soln)	CBM4-OL-4 (mg/kg soln)	CBM4-OL-6 (mg/kg soln)	CBM4-OL-FIL (mg/kg soln)	CBM4-OW-1 (mg/kg soln)	CBM4-OW-2 (mg/kg soln)	CBM4-OW-3 (mg/kg soln)	CBM4-OW-4 (mg/kg soln)	CBM4-OW-5 (mg/kg soln)	CBM4-OW-6 (mg/kg soln)	CBM4-OW-7 (mg/kg soln)
Time	**74.30**	**75.30**	**77.30**	**79.30**	**79.30**	**80.43**	**80.57**	**80.75**	**80.87**	**80.97**	**81.08**	**81.25**
Aluminum	54.6	41.2	32.4	12.1	12.1	40.3	37.4	39.5	34.0	31.3	29.4	30.9
Calcium	9.28	7.30	8.36	4.18	4.18	7.55	6.05	4.26	4.06	2.73	2.09	2.21
Chromium	5,990	5,880	6,160	5,900	5,900	4,540	3,430	2,360	1,750	1,250	864	582
Iron	<4.53	4.14	4.81	5.08	5.08	2.1	1.86	<1.98	<1.81	<1.57	<1.56	<1.61
Lead	<0.453	<0.304	<0.373	<0.235	<0.235	<0.204	<0.165	<0.198	<0.181	<0.157	<0.156	<0.161
Magnesium	<4.53	<3.04	<3.73	<2.35	<2.35	<2.04	<1.65	<1.98	<1.81	<1.57	<1.56	<1.61
Manganese	1,710	1,740	1,320	607	607	<0.204	<0.165	<0.198	<0.181	<0.157	<0.156	<0.161
Nickel	<0.453	<0.304	<0.373	<0.235	<0.235	<0.204	<0.165	<0.198	<0.181	<0.157	<0.156	<0.161
Potassium	44.4	42.9	46.1	46.0	46.0	28.4	21.9	19.4	13.6	<9.43	<9.38	<9.69
Sodium	8,070	7,960	8,290	7,810	7,810	5,740	4,140	3,030	2,080	1,590	1,090	767
Zirconium	<0.453	<0.304	<0.373	<0.235	<0.235	<0.204	<0.165	<0.198	<0.181	<0.157	<0.156	<0.162
Nitrate	583	578	611	585	585	410	300	212	160	115	82.8	63.9
Nitrite	<18.2	<18.3	<15	<14.2	<14.2	26.4	20.4	20.6	<18.2	<15.7	<15.7	<16.2
Oxalate	349	386	524	676	676	228	180	123	95.4	74.6	57.9	44.7
Phosphate	<18.2	<18.3	<15	<14.2	<14.2	<20.5	<16.6	<19.9	<18.2	<15.7	<15.7	<16.2
Sulfate	137	131	136	129	129	85.4	63.9	46.9	34.9	21.6	18.6	<16.2

< is below quantifiable limits.

Table C.4 (Contd)

Sample ID	CBM4-OW-7 (mg/kg soln)	CBM4-OW-8 (mg/kg soln)	CBM4-OW-9 (mg/kg soln)	CBM4-OW-10 (mg/kg soln)	CBM4-OW-11 (mg/kg soln)	CBM4-OW-12 (mg/kg soln)	CBM4-OW-13 (mg/kg soln)	CBM4-FSC-FIL (mg/kg soln)
Time	**81.25**	**81.63**	**81.90**	**82.18**	**82.50**	**82.88**	**83.28**	**86.97**
Aluminum	30.9	21.0	16.2	15.9	10.3	9.13	7.71	12.4
Calcium	2.21	2.06	1.68	1.77	1.63	1.69	1.42	<3.92
Chromium	582	402	287	199	137	97.4	67.0	348
Iron	<1.61	<1.7	<1.42	<1.77	<1.63	<1.69	<1.42	<3.92
Lead	<0.161	<0.17	<0.142	<0.177	<0.163	<0.169	<0.142	<0.392
Magnesium	<1.61	<1.7	<1.42	<1.77	<1.63	<1.69	<1.42	<3.92
Manganese	<0.161	<0.17	<0.142	<0.177	<0.163	<0.169	<0.142	<0.392
Nickel	<0.161	<0.17	<0.142	<0.177	<0.163	<0.169	<0.142	<0.392
Potassium	<9.69	<10.2	<8.51	<10.6	<9.78	<10.1	9.18	<39.2
Sodium	767	647	715	578	477	408	356	780
Zirconium	<0.162	<0.171	<0.142	<0.177	<0.164	<0.17	<0.142	NA
Nitrate	63.9	47.8	36.6	31.7	25.7	22.9	18.3	48.1
Nitrite	<16.2	<17	<14.2	<17.7	<16.3	<16.9	<14.2	<19.6
Oxalate	44.7	33.9	26.0	24.5	<16.3	<16.9	<14.2	<31.6
Phosphate	<16.2	<17	<14.2	<17.7	<16.3	<16.9	<14.2	<19.6
Sulfate	<16.2	<17	<14.2	22.8	<16.3	<16.9	<14.2	<19.6

< is below quantifiable limits.
NA= not analyzed.

Table C.5 (Contd)

Sample ID	CBM5-DW-FIL (mg/kg soln)	CBM5-LE-R-0 (mg/kg soln)	CBM5-LE-R-2 (mg/kg soln)	CBM5-LE-R-4 (mg/kg soln)	CBM5-LE-S-0 (mg/kg soln)	CBM5-LE-S-2 (mg/kg soln)	CBM5-LE-S-4 (mg/kg soln)	CBM5-LE-S-6 (mg/kg soln)	CBM5-LE-S-8 (mg/kg soln)	CBM5-LE-S-10 (mg/kg soln)	CBM5-LE-S-12 (mg/kg soln)	CBM5-LE-C-0 (mg/kg soln)
Time	**0.00**	**3.28**	**5.23**	**7.27**	**8.53**	**10.53**	**12.53**	**14.53**	**16.53**	**18.53**	**20.53**	**20.53**
Aluminum	3,130	2,900	6,340	6,800	6,920	7,860	8,320	9,130	9,670	10,400	10,900	10,900
Calcium	<2.77	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
Chromium	1.03	0.47	0.71	1.07	1.30	1.51	1.57	1.65	1.66	1.78	1.83	1.83
Iron	<2.77	31.2	32.8	38.9	42.2	41.4	41.4	43.5	42.8	40.7	39.4	39.4
Lead	<0.139	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
Magnesium	<2.77	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
Manganese	<0.139	<0.138	<0.129	0.21	0.98	1.78	1.57	1.43	1.28	1.01	0.887	0.887
Nickel	<0.139	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
Potassium	758	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
Sodium	95,900	148,000	149,000	146,000	149,000	149,000	148,000	149,000	145,000	151,000	148,000	148,000
Zirconium	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
Nitrate	19,400	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
Nitrite	5,710	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
Oxalate	760	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
Phosphate	1,880	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
Sulfate	16,200	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA

< is below quantifiable limits.
NA= not analyzed,

Table C.5 (Contd)

Sample ID	**CBM5-LE-C-2 (mg/kg soln)**	**CBM5-LE-C-4 (mg/kg soln)**	**CBM5-LE-C-6 (mg/kg soln)**	**CBM5-LE-C-8 (mg/kg soln)**	**CBM5-LE-C-10 (mg/kg soln)**	**CBM5-LE-C-12 (mg/kg soln)**	**CBM5-LC-FIL (mg/kg soln)**	**CBM5-W-1 (mg/kg soln)**	**CBM5-W-2 (mg/kg soln)**	**CBM5-W-3 (mg/kg soln)**	**CBM5-W-4 (mg/kg soln)**	**CBM5-W-5 (mg/kg soln)**
Time	**22.53**	**24.53**	**26.53**	**28.18**	**30.18**	**32.18**	**50.52**	**51.72**	**52.58**	**53.28**	**53.85**	**54.42**
Aluminum	11,200	11,300	11,500	11,100	11,000	10,900	11,500	6,320	4,280	2,850	1,830	1,200
Calcium	3.38	3.13	2.48	2.21	2.02	1.75	<2.64	<1.23	<1.58	<1.55	<1.6	<1.41
Chromium	1.87	1.92	1.90	1.91	1.83	1.89	1.88	1.48	0.944	0.659	0.482	0.389
Iron	35.8	31.3	25.9	22.8	18.5	17.1	12.8	6.13	3.03	2.14	<1.6	<1.41
Lead	<0.124	<0.119	<0.12	<0.121	<0.117	<0.12	<0.132	<0.122	<0.158	<0.155	<0.16	<0.147
Magnesium	<1.24	<1.19	<1.2	<1.21	<1.17	<1.2	<1.32	<1.22	<1.58	<1.55	<1.6	<1.47
Manganese	0.752	0.537	0.353	0.234	0.143	<0.12	<0.132	<0.122	<0.158	<0.155	<0.16	<0.147
Nickel	<0.124	<0.119	<0.12	<0.121	<0.117	<0.12	<0.132	<0.122	<0.158	<0.155	<0.16	<0.147
Potassium	231	205	221	218	204	220	266	158	118	74	42.5	29.1
Sodium	129,000	130,000	131,000	127,000	126,000	126,000	142,000	87,200	57,400	38,300	26,800	20,000
Zirconium	16.3	15.1	14.2	13.0	12.0	11.6	NA	4.97	1.84	0.665	0.267	<0.141
Nitrate	NA	NA	NA	NA	NA	NA	7,650	3,030	1,780	1,110	678	439
Nitrite	NA	NA	NA	NA	NA	NA	2,370	1,040	524	329	207	135
Oxalate	NA	NA	NA	NA	NA	NA	295	570	1,620	3,220	5,980	8,800
Phosphate	NA	NA	NA	NA	NA	NA	115	306	159	95.7	58.9	37.7
Sulfate	NA	NA	NA	NA	NA	NA	295	2,580	1,390	893	569	379

< is below quantifiable limits.
NA= not analyzed.

Table C.5 (Contd)

Sample ID	**CBM5-W-6 (mg/kg soln)**	**CBM5-W-7 (mg/kg soln)**	**CBM5-W-8 (mg/kg soln)**	**CBM5-W-9 (mg/kg soln)**	**CBM5-W-10 (mg/kg soln)**	**CBM5-W-11 (mg/kg soln)**	**CBM5-W-12 (mg/kg soln)**	**CBM5-W-13 (mg/kg soln)**	**CBM5-W-14 (mg/kg soln)**	**CBM5-W-15 (mg/kg soln)**	**CBM5-W-16 (mg/kg soln)**	**CBM5-OL-05 (mg/kg soln)**
Time	**54.92**	**55.52**	**55.97**	**56.43**	**56.98**	**57.22**	**57.58**	**57.87**	**58.18**	**58.53**	**58.92**	**64.52**
Aluminum	780	499	321	216	260	214	181	127	87.1	63.4	47.4	65.6
Calcium	<1.58	<1.54	<1.81	<1.45	<2.01	<2.1	<1.69	<1.57	<1.96	<1.67	<1.77	4.12
Chromium	0.321	0.243	0.240	0.165	0.189	0.219	0.197	0.17	<0.213	<0.168	<0.177	5,410
Iron	<1.58	<1.54	<1.81	<1.45	<2.01	<2.1	<1.69	<1.57	<1.96	<1.67	<1.77	3.28
Lead	<0.157	<0.155	<0.181	<0.146	<0.202	<0.21	<0.178	<0.157	<0.213	<0.168	<0.177	<0.229
Magnesium	<1.57	<1.55	<1.81	<1.46	<2.02	<2.1	<1.78	<1.57	<1.96	<1.67	<1.77	<2.29
Manganese	<0.157	<0.155	<0.181	<0.146	<0.202	<0.21	<0.178	<0.157	<0.213	<0.168	<0.177	574
Nickel	<0.157	<0.155	<0.181	<0.146	<0.202	<0.21	<0.178	<0.157	<0.213	<0.168	<0.177	<0.229
Potassium	19.8	15.9	<10.9	<8.77	<12.1	<12.6	<10.7	<9.41	,12.8	<10.1	<10.6	46.6
Sodium	20,000	16,500	14,300	10,200	6,580	6,190	4,650	2,680	1,940	1,450	1,150	8,490
Zirconium	<0.158	<0.154	<0.181	<0.145	<0.201	<0.21	<0.169	<0.157	<0.196	<0.167	<0.177	<0.229
Nitrate	270	175	112	76.0	93.8	78.2	64.7	48.3	41.1	29.6	35.5	780
Nitrite	87.7	59.4	41.9	29.8	37.6	33.1	27.3	21.5	22.4	<16.8	19.2	22.9
Oxalate	12,400	15,800	11,800	7,430	5,360	3,550	2,610	1,800	1,170	767	1,080	319
Phosphate	24.4	16.8	18.2	<14.6	<20.1	<21	<17.8	<15.7	<21.4	<16.8	<17.7	14.7
Sulfate	238	151	92.5	64.5	77.8	62.7	51.3	35.3	26.5	18.4	24.1	260
< is below quantifiable limits.												

Table C.5 (Contd)

Sample ID	CBM5-OL-1 (mg/kg soln)	CBM5-OL-2 (mg/kg soln)	CBM5-OL-4 (mg/kg soln)	CBM5-OL-6 (mg/kg soln)	CBM5-OL-FIL (mg/kg soln)	CBM5-OW-1 (mg/kg soln)	CBM5-OW-2 (mg/kg soln)	CBM5-OW-3 (mg/kg soln)	CBM5-OW-4 (mg/kg soln)	CBM5-OW-5 (mg/kg soln)	CBM5-OW-6 (mg/kg soln)	CBM5-OW-7 (mg/kg soln)
Time	**65.02**	**66.02**	**68.02**	**70.07**	**70.07**	**70.55**	**70.92**	**71.10**	**71.30**	**71.43**	**71.60**	**71.77**
Aluminum	73.5	82.0	85.5	82.0	94.7	74.8	68.2	59.6	50.6	43.5	37.4	32.5
Calcium	<2.46	6.19	6.23	4.78	5.48	1.87	2.46	<2.14	<1.68	<1.57	<1.67	<1.97
Chromium	5,470	5,510	5,450	5,470	5,350	4,080	3,370	2,650	2,050	1,570	1,220	936
Iron	4.24	5.52	4.13	5.44	6.50	2.43	2.15	2.19	<1.68	<1.57	<1.67	<1.97
Lead	<0.246	<0.273	<0.322	<0.278	<0.215	<0.148	<0.204	<0.214	<0.168	<0.157	<0.167	<0.197
Magnesium	<2.46	<2.73	<3.22	<2.78	<2.15	<1.48	<2.04	<2.14	<1.68	<1.57	<1.67	<1.97
Manganese	647	693	819	666	0.285	<0.148	<0.204	<0.214	<0.168	<0.157	<0.167	<0.197
Nickel	<0.246	<0.273	<0.322	<0.278	<0.215	<0.148	<0.204	<0.214	<0.168	<0.157	<0.167	<0.197
Potassium	49.6	35.5	35.2	30.3	26.9	20.8	14.5	14.0	<10.1	<9.42	<10	<11.8
Sodium	8,770	8,870	8,700	8,810	8,930	6,540	5,420	4,290	3,420	2,670	2,190	1,760
Zirconium	<0.246	<0.272	<0.323	<0.28	0.26	<0.148	<0.204	<0.214	<0.168	<0.157	<0.167	<0.197
Nitrate	782	771	722	732	726	559	450	345	270	226	161	130
Nitrite	27.2	43.6	77.0	77.3	79.7	63.1	56.8	47.5	37.8	33.6	27.0	25.6
Oxalate	362	450	505	495	475	249	212	166	132	108	78.8	65.0
Phosphate	15.4	<16.4	<19.5	<16.8	31.6	<14.6	<20.3	<21.3	<16.8	<16.8	<16.7	<19.7
Sulfate	264	266	262	264	270	204	167	128	99.4	85.3	60.1	46.3

< is below quantifiable limits.

Table C.5 (Contd)

Sample ID	CBM5-OW-8 (mg/kg soln)	CBM5-OW-9 (mg/kg soln)	CBM5-OW-10 (mg/kg soln)	CBM5-OW-11 (mg/kg soln)	CBM5-OW-12 (mg/kg soln)	CBM5-OW-13 (mg/kg soln)	CBM5-FSC-FIL (mg/kg soln)
Time	**71.95**	**72.13**	**72.35**	**72.58**	**72.85**	**73.15**	**75.52**
Aluminum	28.8	25.6	22.6	20.3	19.2	17.6	24.6
Calcium	1.58	<1.77	<1.81	<1.74	<1.78	<1.63	<3.57
Chromium	723	581	449	347	274	218	240
Iron	<1.49	<1.77	<1.81	<1.74	<1.78	<1.63	<3.57
Lead	<0.149	<0.177	<0.181	<0.174	<0.178	<0.163	<0.178
Magnesium	<1.49	<1.77	<1.81	<1.74	<1.78	<1.63	<3.57
Manganese	<0.149	<0.177	<0.181	<0.174	<0.178	<0.163	<0.178
Nickel	<0.149	<0.177	<0.181	<0.174	<0.178	<0.163	<0.178
Potassium	<8.95	<10.6	<10.9	<10.4	<10.7	<9.79	<35.7
Sodium	1,440	1,210	1,020	857	746	667	859
Zirconium	<0.149	<0.177	<0.181	<0.174	<0.178	<0.163	<0.178
Nitrate	101	83.5	66.9	54.5	46.1	38.4	44.6
Nitrite	19.6	19.7	18.0	<17.1	<17.6	<16.2	<17.8
Oxalate	55.5	45.0	36.7	30.6	30.4	21.8	45.2
Phosphate	<19.7	<14.9	<17.6	<17.8	<17.6	<16.2	<17.8
Sulfate	36.9	31.1	21.5	<17.1	<17.6	<16.2	<17.8
< is below quantifiable limits.							

Appendix D

Physical Property Data for Cold-CUF Runs

Appendix D: Physical Property Data for Cold-CUF Runs

Table D.1. Initial Blended Component Slurries Physical-Property Measurements

	CBM-1	CBM-2	CBM-3	CBM-4	CBM-5
Slurry Density (g/mL)	1.26	1.26	1.26	1.26	1.26
Supernate Density (g/mL)	1.23	1.23	1.22	1.23	1.23
Total Solids (wt%)	32.2	31.6	31.6	32.3	32.4
Dissolved Solids (wt%)	27.9	27.9	26.9	28.0	27.9
Undissolved Solids (wt%)	4.3	3.7	4.7	4.3	4.5

Table D.2. Dewatered Blended Component Slurries Physical-Property Measurements

	CBM-1	CBM-2	CBM-3	CBM-4	CBM-5
Slurry Density (g/mL)	1.37	1.36	1.36	1.37	1.41
Supernate Density (g/mL)	1.23	1.23	1.22	1.23	1.23
Total Solids (wt%)	44.2	44.6	42.7	42.0	44.1
Dissolved Solids (wt%)	30.0	30.0	26.9	28.0	27.9
Undissolved Solids (wt%)	20.3	20.9	21.7	19.5	22.5

Table D.3. Post-Caustic Leaching Dewatered Blended Component Slurries Physical-Property Measurements

	CBM-1	CBM-2	CBM-3	CBM-4	CBM-5
Slurry Density (g/mL)	1.37	1.23	1.40	1.41	1.41
Supernate Density (g/mL)	1.25	1.24	1.28	1.28	1.28
Total Solids (wt%)	52.5	44.7	46.8	42.5	49.4
Dissolved Solids (wt%)	26.8	29.5	39.3	37.8	42.4
Undissolved Solids (wt%)	35.1	21.7	12.4	7.5	12.1

Table D.4. Post-Caustic Leach Washed Blended Component Slurries Physical-Property Measurements

	CBM-1	CBM-2	CBM-3	CBM-4	CBM-5
Slurry Density (g/mL)	1.14	1.18	---	1.10	1.17
Supernate Density (g/mL)	1.06	1.06	---	1.01	1.00
Total Solids (wt%)	18.9	23.8	28.1	19.8	22.2
Dissolved Solids (wt%)	2.19	9.83	0.40	0.93	0.29
Undissolved Solids (wt%)	17.1	15.5	27.8	19.0	22.0

Table D.5. Post-Oxidative Leached Component Slurries Physical-Property Measurements

	CBM-1	CBM-2	CBM-3	CBM-4	CBM-5
Slurry Density (g/mL)	1.11	1.17	1.09	1.10	1.11
Supernate Density (g/mL)	1.00	1.00	---	---	1.02
Total Solids (wt%)	16.6	22.9	11.03	15.05	14.49
Dissolved Solids (wt%)	0.83	1.02	2.84	2.56	2.90
Undissolved Solids (wt%)	15.9	22.1	8.4	12.8	11.9

Table D.6. Post-Oxidative Leached and Washed (Final) Blended Component Slurries Physical-Property Measurements

	CBM-1	CBM-2	CBM-3	CBM-4	CBM-5
Slurry Density (g/mL)	1.24	1.16	1.18	1.17	1.13
Supernate Density (g/mL)	1.00	1.00	0.998	0.999	0.999
Total Solids (wt%)	28.3	22.7	24.42	20.81	23.39
Dissolved Solids (wt%)	0.28	0.00	0.19	0.00	0.19
Undissolved Solids (wt%)	28.1	22.7	24.3	20.8	23.2

Table D.7. Chromium Added and Dewatered Blended Component Slurries Physical-Property Measurements

	CBM-3	CBM-4	CBM-5
Slurry Density (g/mL)	1.07	1.14	1.13
Supernate Density (g/mL)	--	1.01	1.01
Total Solids (wt%)	11.8	23.7	22.8
Dissolved Solids (wt%)	3.51	3.16	3.06
Undissolved Solids (wt%)	8.62	21.2	20.4

Appendix E

Description of Crossflow Ultrafiltration System

Appendix E: Description of Crossflow Ultrafiltration System

Filtration/Leaching Apparatus

The testing apparatus was a bench top system mounted on a skid that allowed up to 25 liters of a simulant waste solution to be circulated through a tubular filter that measures filter feed flow rates, filtrate flow rates, system pressures, and temperatures simultaneously. Note that the volume of this reservoir was chosen to maintain the same filter surface area to tank volume as is present in the full-scale facility. The testing apparatus used a heat exchanger on the main flow loop to cool the feed solution during filtration operations and had a heater on the main holding tank to perform leaching at elevated temperatures.

Crossflow Ultrafiltration System

The WTP Pre-Treatment Facility (PTF) plans to use crossflow ultrafiltration to separate the LAW liquid streams from the HLW slurry streams through the process. The filter elements are porous sintered metal tubes. The filter feed flows through the inside of the filter element axially while the feed permeate passes through the tube walls radially. Filtration occurs when the pressure differential between the inside and outside walls of the filter element (known as the transmembrane pressure) is high enough to drive the slurry permeate through the tubular walls. The axial flow across the filter walls minimizes solid buildup and allows filtration to occur continuously with minimal downtime for back-pulsing to remove the solids buildup.

The filters purchased for this testing were supplied by the Mott Corporation,(a) using the same specifications(b) for the filters being purchased for the WTP PTF. The filters were made with 316 stainless steel and have an effective filtration rating of 0.1 µm. The dimensions of the filter element used in these tests are shown in Figure E.1.

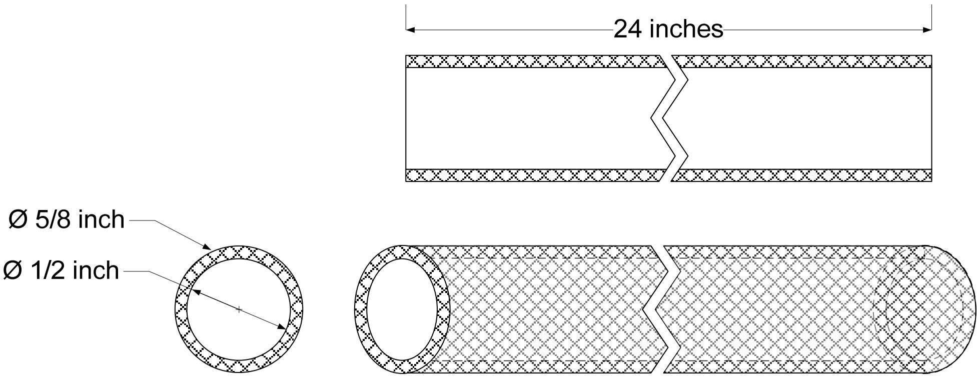


Figure E.1. Crossflow Ultrafiltration Element

(a) Mott Corporation, 84 Spring Lane, Farmington, CT 06032.

(b) BNI Specification WTP-070110.

The filter element for the laboratory simulant development task was received already installed in a tube-in-tube configuration with an outer tube surrounding the filter element to capture the permeate while the inlet and the outlet of the filter (which extend past the shell and provide access to the inside diameter of the filter) were welded to steel tubing of a matching outer and inner diameter. The shell side had two $^3/_8$-inch stainless steel tubes exiting from the filter assembly, one in the center to collect filtrate from the filter and the other near the inlet of the filter to function as a drain. Pressure ports (¼-inch stainless steel tubing) were installed on the inlet and outlet connections to the assembly to measure the pressure inside the filter. O-ring face seal fittings (Swagelok(a) VCO®) were also placed on the inlet and outlet filter feed tube connections for easy installation to the filtration/leaching skid. Figure E.2 and Figure E.3 show the crossflow ultrafiltration system.

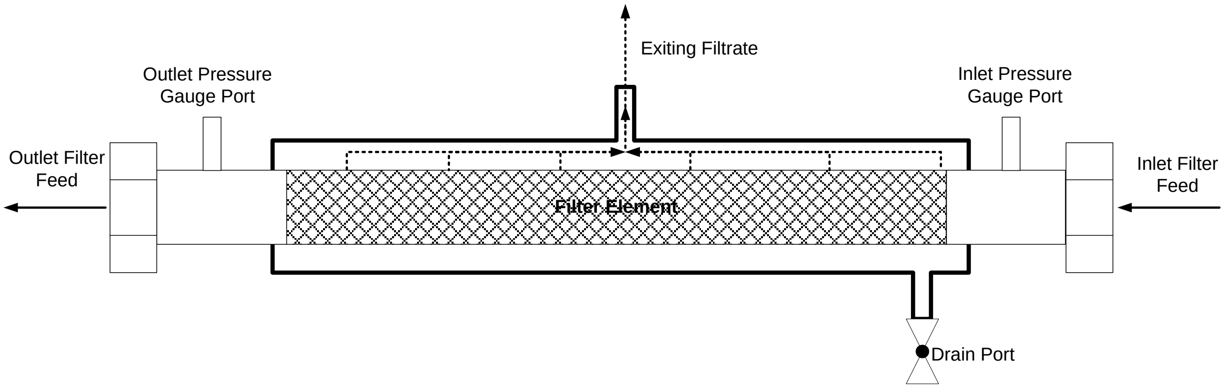


Figure E.2. Crossflow Ultrafiltration System Filter Assembly Sketch (*Not to Scale*)

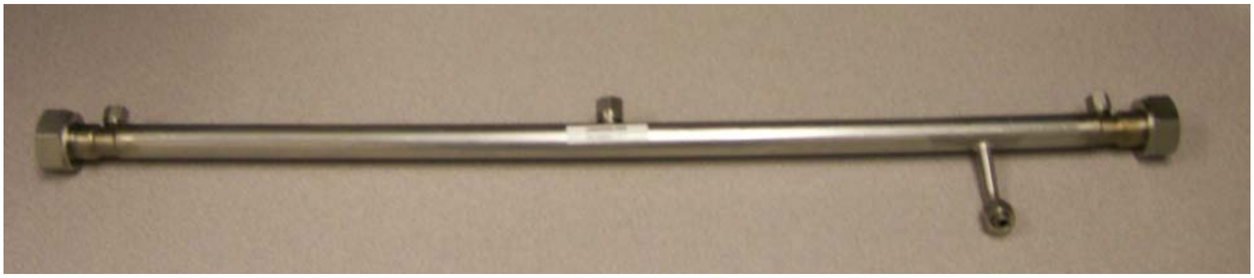


Figure E.3. Crossflow Ultrafiltration System Assembly

Crossflow Ultrafiltration System

The filter described in the section above was installed in a bench-top testing apparatus that circulates the test filtration simulants through the inside of the filter and diverts the filter permeate to a collection bottle or recycles it back into the slurry reservoir. Figure E.4 shows a piping diagram of the testing apparatus. Figure E.5 is a photograph of the assembled testing apparatus. The testing apparatus is commonly referred to as the crossflow ultrafiltration system.

(a) Swagelok Company, 31400 Aurora Road, Solon, Ohio, 44139.

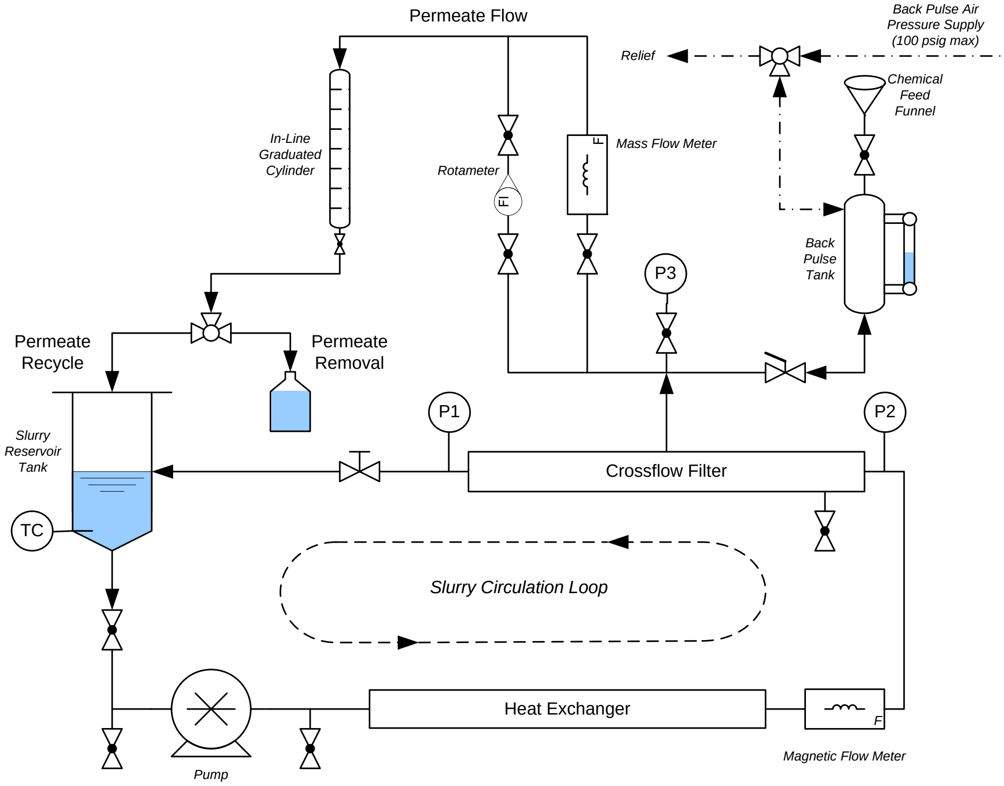


Figure E.4. Piping Diagram of Crossflow Ultrafiltration System (*Not to Scale*)

Figure E.5. Crossflow Ultrafiltration System

The crossflow ultrafiltration system has four main parts:

- Slurry reservoir tank
- Slurry recirculation loop
- Permeate flow loop
- Permeate back pulse chamber.

The slurry reservoir was a cylindrical 304-L stainless steel tank. There were two different tanks, one of 10-liter and the other of 25-liter capacity that could be used with the testing apparatus depending on the test that was to be performed. Note that the volume of this reservoir was chosen to maintain the same filter surface area to tank volume as is present in the full-scale facility. The larger reservoir, as shown in Figure E.5, permitted scaled leaching tests to be performed. The smaller reservoir allowed smaller volumes of simulant to be used. Agitation in the tank was provided with an overhead mixer using a 2-inch-diameter, three-blade marine propeller. To allow the system to be easily drained, the bottom of the tanks were sloped at a 15° angle. Baffles were installed on the tank wall to make sure that the slurry mixing was homogenous. Heat tape was installed around the walls of the tank for leaching at elevated temperatures. The heat tape was connected to a temperature controller that adjusted the electrical load to the heat tape based on a thermocouple input. A dual, Type-K thermocouple was installed inside the reservoir tank (extending just below the overhead mixing impeller) to measure the temperature of the slurry inside the reservoir. One of the thermocouple elements was connected to the heat tape's temperature controller and the other to a data-collection system.

The slurry recirculation loop directed slurry flow from the slurry reservoir, through the filter, and back into the slurry reservoir for filtration operations. The bottom of the slurry reservoir was connected to the suction side of the slurry recirculation pump—a progressive cavity rotary lobe pump. The discharge of the pump flowed through a single-pass shell-and-tube heat exchanger used to remove excess heat from the system caused by mechanical energy input from the mixer and pump as well as heat generated from frictional flow.

An exterior chiller circulated a water/antifreeze mixture through the exterior shell of the heat exchanger to remove heat away from the circulating slurry on the tube side of the heat exchanger. The chiller controlled the chilling-fluid temperature by monitoring the temperature of the slurry exiting the heat exchanger via a resistance temperature detector (RTD) installed in the discharge line.

The slurry then flowed through a magnetic flow sensor that monitored the volumetric flow of the slurry inside the slurry recirculation loop. The sensor's output was displayed on an external panel meter that generated an analog output signal monitored by a data-collection system. The data from this device were used to calculate the axial velocity (AV) inside the filter element.

The flowing slurry then entered the filter. Digital pressure gauges were installed on the inlet and outlet ports of the filter, which displayed the pressure at both locations in pounds per inch squared-gauge (psig) with an uncertainty of ± 1 psig. The gauges also transmit analog output signals to the data-collection system. The data from these devices were used to calculate the average pressure inside the filter and the axial pressure drop across the element.

A manual pinch valve was located at the filter's discharge. The valve was used to adjust the pressure inside the filter to drive permeate flow through the filter membrane wall. It was also connected to the slurry reservoir tank and was closed completely when the reservoir tank was isolated for leaching.

The permeate flow loop started at the center of the filter assembly where a polyethylene tube connected the filter to a manifold of ¼-inch stainless steel piping that directed the filter permeate through a series of measurement devices. A digital pressure gauge was installed at this point to measure the pressure on the permeate side of the filter in psig. Like the other two digital gauges, this instrument transmitted an analog output signal to the data-collection system. The transmembrane pressure (TMP) across the filter was then calculated by subtracting the pressure on the permeate side of the filter from the average pressure of the slurry inside the filter.

Flow from the filter was either diverted through a mass flow meter that could be calibrated up to 180 mL/min or to a user-calibrated rotameter that can measure flow up to 30 mL/s. The mass flow meter also measured the density of the permeate flow and transmitted two analog output signals to the data-collection system for the volumetric flow rate and the density. An in-line glass cylinder was installed on the discharge of both meters to take manual measurements of the permeate flow rate. Measurements were taken by closing a valve at the bottom of the cylinder and allowing the permeate to fill the glass cylinder. The liquid volume in the glass cylinder was measured by volume markings on the outside. The permeate flow rate was calculated from observed changes in permeate volume in the cylinder over a measured time interval.

Permeate exited through a 3-way valve connected to the slurry reservoir tank. This valve directed permeate either back to the slurry reservoir tank to be mixed back with the slurry or to a sampling hose used to collect permeate into sample containers.

The permeate back-pulse chamber was to the right of the permeate flow loop and connected to the filter at the same location as the permeate pressure gauge. The chamber was an approximately 500-mL steel vessel with a sight glass to track the permeate volume inside the chamber. The vessel had three entry ports:

- ¼-inch line with a two-way toggle valve on the bottom connecting the vessel to the permeate side of the filter
- ¼-inch line with a two-way valve connecting the top of the vessel to a funnel
- ¼-inch line with a three-way valve connecting the top of the vessel to a compressed air line and vent line connected to the top of the slurry reservoir tank.

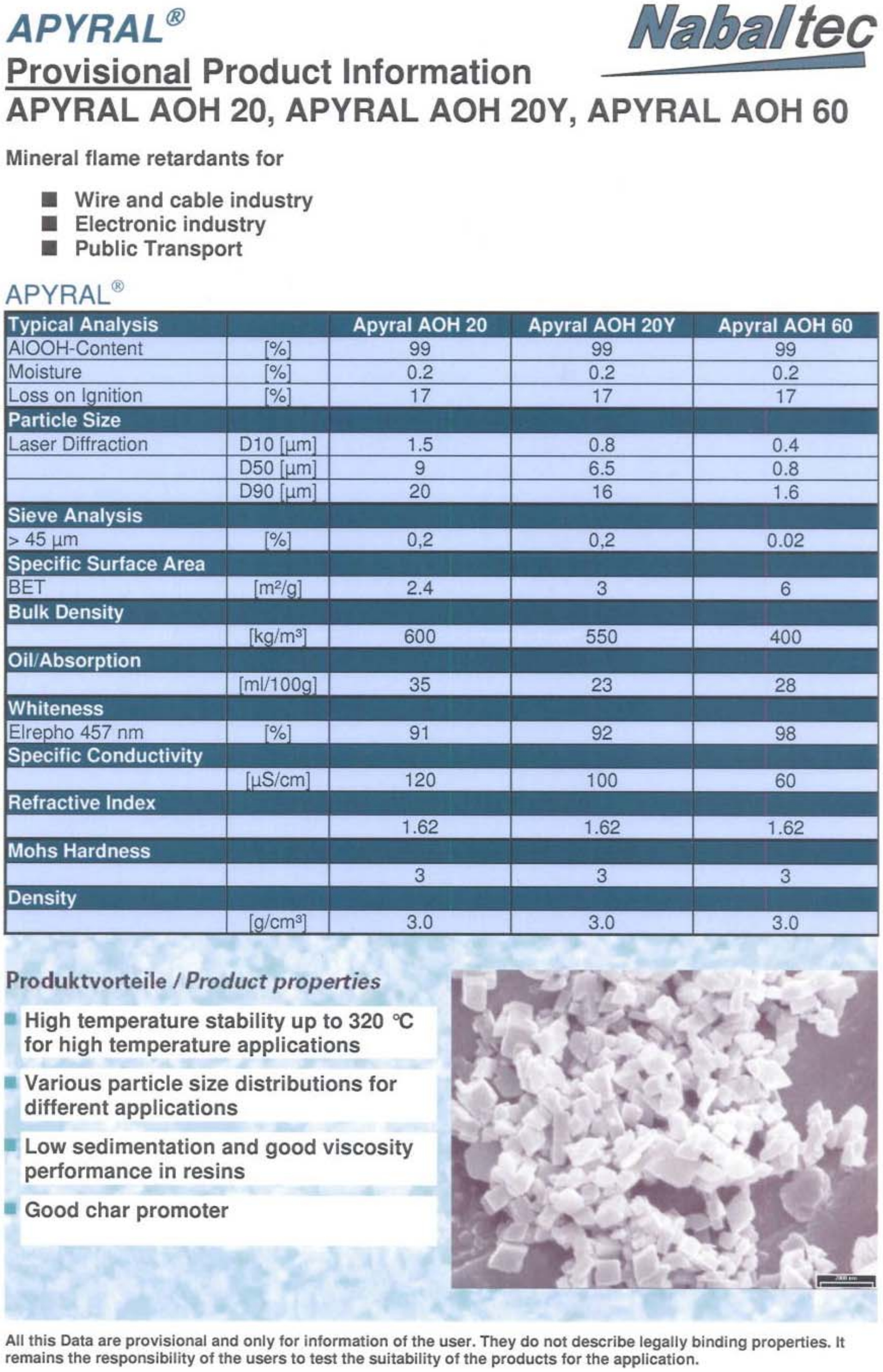
The bottom line was used to direct permeate flow between the chamber and the filter. The funnel on the top of the chamber was used to introduce cleaning and rinse solutions directly to the vessel. The compressed gas line was used to pressurize the fluid in the chamber and to vent the chamber to atmospheric pressure.

To back-pulse the filter, the vessel was first vented to atmospheric pressure. Next, permeate was allowed to fill the chamber by opening the toggle valve. Once the chamber was half full of permeate (as seen through the sight glass), the toggle valve was closed. The three-way valve was then positioned to allow compressed gas at 80 psig to fill the chamber and pressurize the fluid. The three-way valve was then positioned to isolate the now pressurized chamber. The slurry pressure inside the filter was then dropped below the pressure of the compressed gas line (< 20 psig). The toggle valve at the bottom of the tank was opened, allowing the pressurized permeate inside the chamber to flow backwards through the filter element. The toggle valve was closed when the permeate level was below the visible portion of the sight glass. After the back-pulse was completed, the three-way valve was positioned to vent the chamber back to atmospheric pressure.

Appendix F

APYRAL Boehmite Product Description

Appendix F: APYRAL Boehmite Product Description



APYRAL®

Nabaltec

Provisional Product Information

APYRAL AOH 20, APYRAL AOH 20Y, APYRAL AOH 60

Mineral flame retardants for

- Wire and cable industry
- Electronic industry
- Public Transport

APYRAL®

Typical Analysis		Apyral AOH 20	Apyral AOH 20Y	Apyral AOH 60
AlOOH-Content	[%]	99	99	99
Moisture	[%]	0.2	0.2	0.2
Loss on Ignition	[%]	17	17	17
Particle Size				
Laser Diffraction	D10 [µm]	1.5	0.8	0.4
	D50 [µm]	9	6.5	0.8
	D90 [µm]	20	16	1.6
Sieve Analysis				
> 45 µm	[%]	0,2	0,2	0.02
Specific Surface Area				
BET	[m²/g]	2.4	3	6
Bulk Density				
	[kg/m³]	600	550	400
Oil/Absorption				
	[ml/100g]	35	23	28
Whiteness				
Elrepho 457 nm	[%]	91	92	98
Specific Conductivity				
	[µS/cm]	120	100	60
Refractive Index				
		1.62	1.62	1.62
Mohs Hardness				
		3	3	3
Density				
	[g/cm³]	3.0	3.0	3.0

Produktvorteile / *Product properties*

- High temperature stability up to 320 °C for high temperature applications
- Various particle size distributions for different applications
- Low sedimentation and good viscosity performance in resins
- Good char promoter

All this Data are provisional and only for information of the user. They do not describe legally binding properties. It remains the responsibility of the users to test the suitability of the products for the application.

Appendix G

Operating Conditions for Crossflow Ultrafiltration Runs

Appendix G: Operating Conditions for Crossflow Ultrafiltration Runs

Table G.1. Timeline of the CBM-1 Test

Time (hrs)	Process Step	Action	Samples
0	Start of test	11.25 kg of 5 wt% UDS slurry	CBM-IN-SL
0-5.75	Initial dewatering	Removed 33.68 kg supernate and added 34.04 kg feed	CBM-DW-SL and CBM-DW-FIL
7	Preparation for caustic leaching	Added 6.94 kg of 19 M NaOH and 9.54 kg of DIW	
7.25-11.25	Caustic leaching	Heated to 100°C	CBM-LE-R-0,2,4
11.25-23.25	Caustic leaching	Leached at 100°C	CBM-LE-S-0,2,4,6,8,10,12
23.25-35.25	Caustic leaching	Cooled from 100°C to 45°C	CBM-LE-C-2,4,6,8,10,12
36.5-43.5	Caustic leach dewater	Removed 22.26 kg supernate	CBM-LC-SL and CBM-LC-FIL
44.5-48	Caustic leach washing	Added 5.01 kg of 1.57 M NaOH and removed 4.00 kg supernate	CBM-W1-FIL
48.75-53.5	Caustic leach washing	Added 4.89 kg of 0.68 M NaOH and removed 4.96 kg supernate	CBM-W2-FIL
54.5-55.5	Caustic leach washing	Added 4.84 kg of 0.26 M NaOH and removed 4.92 kg supernate	CBM-W3-FIL
56.25-56.75	Caustic leach washing	Added 4.82 kg of 0.08 M NaOH and removed 4.95 kg supernate	CBM-W4-FIL
58-58.5	Caustic leach washing	Added 4.81 kg of 0.03 M NaOH and removed 4.80 kg supernate	CBM-W5-FIL
59-59.4	Caustic leach washing	Added 4.80 kg of 0.01 M NaOH and removed 4.72 kg supernate	CBM-W6-FIL, CBM-WC-FIL, and CBM-WS-SL
62.5	NaOH concentration adjustment	Added 9.9 g of 19 M NaOH	CBM-POL-SUP
63.75-69.75	Oxidative leaching	Added 640 mL of 0.2 M $KMnO_4$	CBM-OL-05,1,2,4,6 and CBM-OL-SL
71.5-72.25	Oxidative leach washing	Added 4.70 kg of 0.01 M NaOH and removed 6.0 kg supernate	No sample taken.
73.3-74	Oxidative leach washing	Added 4.79 kg of 0.01 M NaOH and removed 2.53 kg supernate	No sample taken.
266-266.5	Oxidative leach washing	Added 2.33 kg of 0.01 M NaOH and removed 2.35 kg supernate.	CBM-W3-OL
267-267.75	Oxidative leach washing	Added 2.40 kg of 0.01 M NaOH and removed 2.48 kg supernate	CBM-FSC-SL

Table G.2. Timeline for Test CBM-2

Time (hrs)	Process Step	Action	Samples
0	Start of test	11.33 kg of 5 wt% UDS slurry	CBM25-IN-SL
0-14.75	Initial dewatering	Added 33.86 kg slurry and removed 34.67 kg supernate	CBM25-DW-SL and CBM25-DW-FIL
15.25	Preparation for caustic leaching	Added 6.94 kg of 19 M NaOH and 9.54 kg DIW	No sample taken.
16.25-24.4	Caustic leaching	Heated from 25°C to 100°C	CBM25-LE-R-0,2,4
24.4-36.4	Caustic leaching	Leached at 100°C	CBM25-LE-S-0,2,4,6,8,10,12
36.4-48.25	Caustic leaching	Cooled from 100°C to 45°C	CBM25-LE-C-0,2,4,6,8,10,12
49.25-60.4	Caustic leach dewater	Removed 20.30 kg supernate.	CBM25-LC-SL and CBM25-LC-FIL
60.6-64.6	Caustic leach washing	Added 5.01 kg of 1.57 M NaOH and removed 5.56 kg supernate	CBM25-W1-FIL
64.6-67.6	Caustic leach washing	Added 4.89 kg of 0.68 M NaOH and removed 5.10 kg supernate	CBM25-W2-FIL
68.6-70	Caustic leach washing	Added 4.88 kg of 0.26 M NaOH and removed 5.06 kg supernate	CBM25-W3-FIL
70.25-71.25	Caustic leach washing	Added 4.81 kg of 0.08 M NaOH and removed 4.89 kg supernate	CBM25-W4-FIL
71.8-72.8	Caustic leach washing	Added 4.80 kg of 0.03 M NaOH and removed 4.87 kg supernate	CBM25-W5-FIL
73-74	Caustic leach washing	Added 4.80 kg of 0.01 M NaOH and removed 4.81 kg supernate	CBM25-W6-FIL, CBM25-WC-FIL, CBM25-WS-SL, and CBM25-POL-SUP
76-82	Oxidative leaching	Added 128 mL of 1 M $NaMnO_4$	CBM25-OL-05,1,2,4,6 and CBM25-OL-SL
83.25-84	Oxidative leach washing	Added 4.81 kg of 0.01 M NaOH and removed 4.87 kg supernate	No sample taken.
84.25-86.25	Oxidative leach washing	Added 4.80 kg of 0.01 M NaOH and removed 4.83 kg supernate	No sample taken.
86.5-88.8	Oxidative leach washing	Added 4.80 kg of 0.01 M NaOH and removed 4.82 kg supernate	CBM25-W3-OL
89-91.5	Oxidative leach washing	Added 4.80 kg of 0.01 M NaOH and removed 4.81 kg supernate	CBM25-FSC-SL

Table G.3. Timeline of the CBM-3 Test

Time (hrs)	Process Step	Action	Samples
0	Start of test	Added 28.37 kg of 5 wt% UDS slurry	CBM3-IN-SL
0-5.5	Initial dewatering	Removed 21.68 kg supernate	CBM3-DW-SL and CBM3-DW-FIL
6.25-7	Preparation for caustic leaching	Added 8.57 kg of 19 M NaOH and 7.92 kg DIW	No sample taken.
7-12.3	Caustic leaching	Heated from 25°C to 100°C	CBM3-LE-R-0,2,4
12.3-24.3	Caustic leaching	Leached at 100°C	CBM3-LE-S-0,2,4,6,8,10,12
24.3-36.3	Caustic leaching	Cooled from 100°C to 25°C	CBM3-LE-C-0,2,4,6,8,10,12
49.3-63.5	Caustic leach dewater	Removed 19.27 kg supernate	CBM3-LC-SL and CBM3-LC-FIL
65-74	Caustic leach washing	Added 11.76 kg of 0.01 M NaOH and removed 11.76 kg supernate	CBM3-WC-FIL and CBM3-WS-SL
75.5	CrOOH slurry addition	Added 1.73 kg CrOOH slurry	No sample taken.
75.75	CrOOH slurry washing	Added 15 L of 0.01 M NaOH	No sample taken.
75.75-79.75	CrOOH slurry wash dewatering	Removed 16.34 kg of supernate	CBM3-CW-SL
81.3-87.3	Oxidative leaching	Added .326 L of 1 M $NaMnO_4$	CBM3-OL-05,1,2,4,6, CBM3-OL-SL, and CBM3-OL-FIL
88-92.2	Oxidative leach washing	Added ~9 kg of 0.01 M NaOH and removed 9.05 kg supernate	No sample taken.
92.3-92.75	Final dewatering	Removed 0.95 kg supernate	CBM3-FSC-SL

Table G.4. Timeline for test CBM-4

Time (hrs)	Process Step	Action	Samples
0	Start of test	Added 26.25 kg of 5 wt% UDS slurry	CBM4-IN-SL
0-8.7	Initial dewatering	Removed 19.64 kg supernate	CBM4-DW-SL and CBM4-DW-FIL
14	Preparation for caustic leaching	Added 8.66 kg of 19 M NaOH and 8.08 kg DIW	No sample taken.
14-19.5	Caustic leaching	Heated from 25°C to 100°C	CBM4-LE-R-0,2,4
19.5-31.5	Caustic leaching	Leached at 100°C	CBM4-LE-S-0,2,4,6,8,10,12
31.5-43.5	Caustic leaching	Cooled from 100°C to 25°C	CBM4-LE-C-0,2,4,6,8,10,12
45-60	Caustic leach dewater	Removed 19.17 kg supernate	CBM4-LC-SL and CBM4-LC-FIL
61-66.5	Caustic leach washing	Added 12.09 kg of 0.01 M NaOH and removed 11.34 kg supernate	CBM4-WC-FIL and CBM4-WS-SL
68	CrOOH slurry addition	Added 1.73 kg CrOOH slurry	No sample taken.
78.25	CrOOH slurry washing	Added 14.92 kg of 0.01 M NaOH	No sample taken.
78.5-80.7	CrOOH slurry wash dewatering	Removed 16.65 kg of supernate	CBM4-CW-SL
89.3-95	Oxidative leaching	Added .326 L of 1 M $NaMnO_4$	CBM4-OL-05,1,2,4,6, CBM4-OL-SL, and CBM4-OL-FIL
96.1-98.8	Oxidative leach washing	Added 9.42 kg of 0.01 M NaOH and removed 9.25 kg supernate	No sample taken.
99-101.5	Final dewatering	Removed 0.40 kg supernate	CBM4-FSC-SL

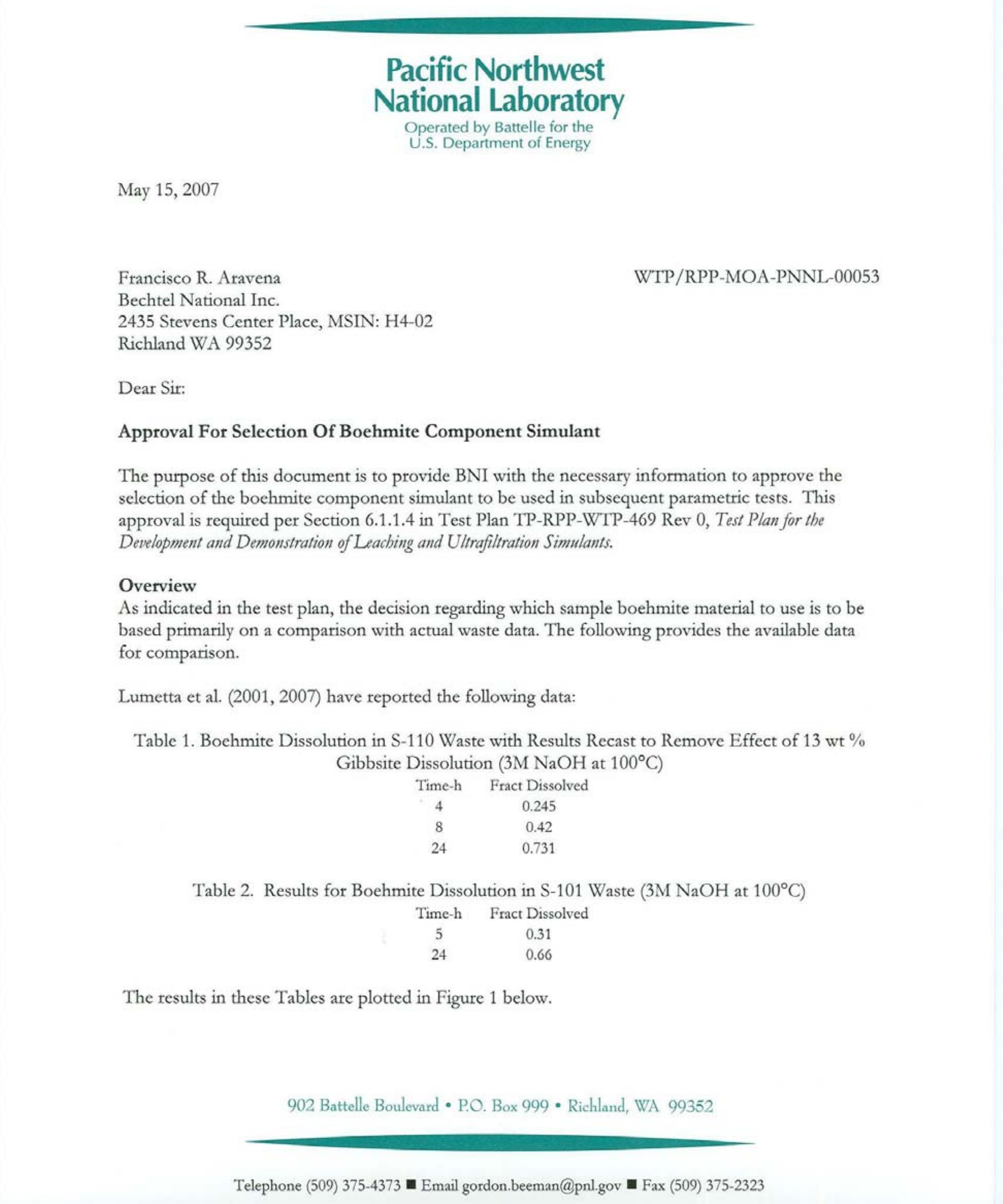
Table G.5. Timeline for test CBM-5

Time (hrs)	Process Step	Action	Samples
0	Start of test	Added 26.25 kg of 5 wt% UDS slurry	CBM5-IN-SL
0-9.75	Initial dewatering	Removed 20.66 kg supernate	CBM5-DW-SL and CBM5-DW-FIL
13	Preparation for caustic leaching	Added 8.57 kg of 19 M NaOH and 7.92 kg DIW	No sample taken.
13-18.3	Caustic leaching	Heated from 25°C to 100°C	CBM5-LE-R-0,2,4
18.3-30.3	Caustic leaching	Leached at 100°C	CBM5-LE-S-0,2,4,6,8,10,12
30.3-42.3	Caustic leaching	Cooled from 100°C to 25°C	CBM5-LE-C-0,2,4,6,8,10,12
43.1-60	Caustic leach dewater	Removed 19.19 kg supernate	CBM5-LC-SL and CBM5-LC-FIL
60.5-68.3	Caustic leach washing	Added 11.25 kg of 0.01 M NaOH and removed 11.41 kg supernate	CBM5-WC-FIL and CBM5-WS-SL
69	CrOOH slurry addition	Added 1.73 kg of CrOOH slurry	No sample taken.
69	CrOOH slurry washing	Added 14.36 kg of 0.01 M NaOH	No sample taken.
69.3-71.5	CrOOH slurry wash dewatering	Removed 15.48 kg of supernate	CBM5-CW-SL
73.75-79.75	Oxidative leaching	Added .326 L of 1 M $NaMnO_4$	CBM5-OL-05,1,2,4,6, CBM5-OL-SL, and CBM5-OL-FIL
80.3-83	Oxidative leach washing	Added 9.24 kg of 0.01 M NaOH and removed 9.27 kg supernate	No sample taken.
84-85.3	Final dewatering	Removed 1.21 kg of supernate	CBM5-FSC-SL and CBM5-FSC-FIL

Appendix H

Letters of Request for Approval for Test Plan Hold Points

Appendix H: Letters of Request for Approval for Test Plan Hold Points



Pacific Northwest
National Laboratory
Operated by Battelle for the
U.S. Department of Energy

May 15, 2007

Francisco R. Aravena
Bechtel National Inc.
2435 Stevens Center Place, MSIN: H4-02
Richland WA 99352

WTP/RPP-MOA-PNNL-00053

Dear Sir:

Approval For Selection Of Boehmite Component Simulant

The purpose of this document is to provide BNI with the necessary information to approve the selection of the boehmite component simulant to be used in subsequent parametric tests. This approval is required per Section 6.1.1.4 in Test Plan TP-RPP-WTP-469 Rev 0, *Test Plan for the Development and Demonstration of Leaching and Ultrafiltration Simulants.*

Overview

As indicated in the test plan, the decision regarding which sample boehmite material to use is to be based primarily on a comparison with actual waste data. The following provides the available data for comparison.

Lumetta et al. (2001, 2007) have reported the following data:

Table 1. Boehmite Dissolution in S-110 Waste with Results Recast to Remove Effect of 13 wt % Gibbsite Dissolution (3M NaOH at 100°C)

Time-h	Fract Dissolved
4	0.245
8	0.42
24	0.731

Table 2. Results for Boehmite Dissolution in S-101 Waste (3M NaOH at 100°C)

Time-h	Fract Dissolved
5	0.31
24	0.66

The results in these Tables are plotted in Figure 1 below.

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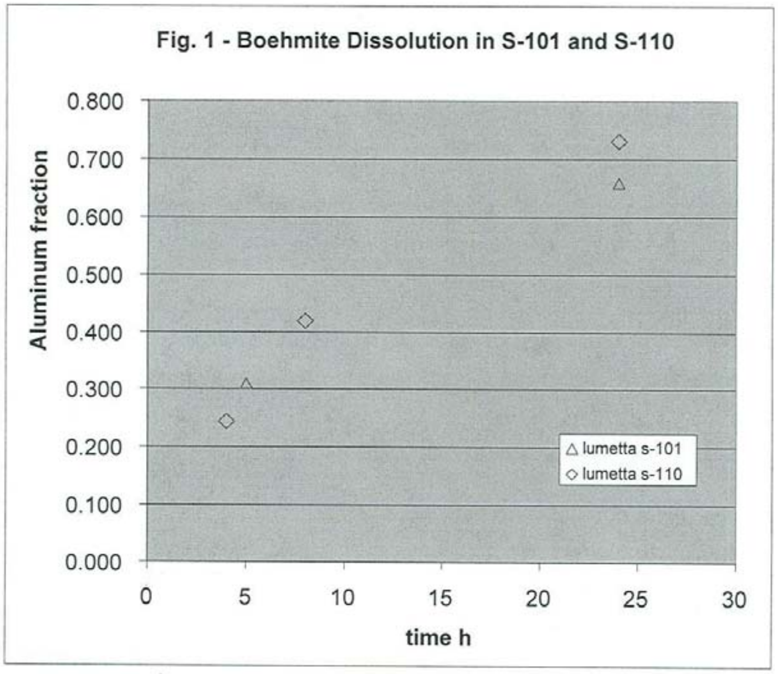


Simulants Obtained:

Five samples of Boehmite (as defined by the vendors) were obtained from two sources.

Sample ID	Boehmite samples
B1	APYRAL AOH60
B2	APYRAL AOH20Y
B3	APYRAL AOH20
B4	Autoclaved G250 (psuedoboehmite)
B5	Autoclaved C31C (gibbsite)

The 3 figures below are three of the XRD patterns – all are Boehmite.

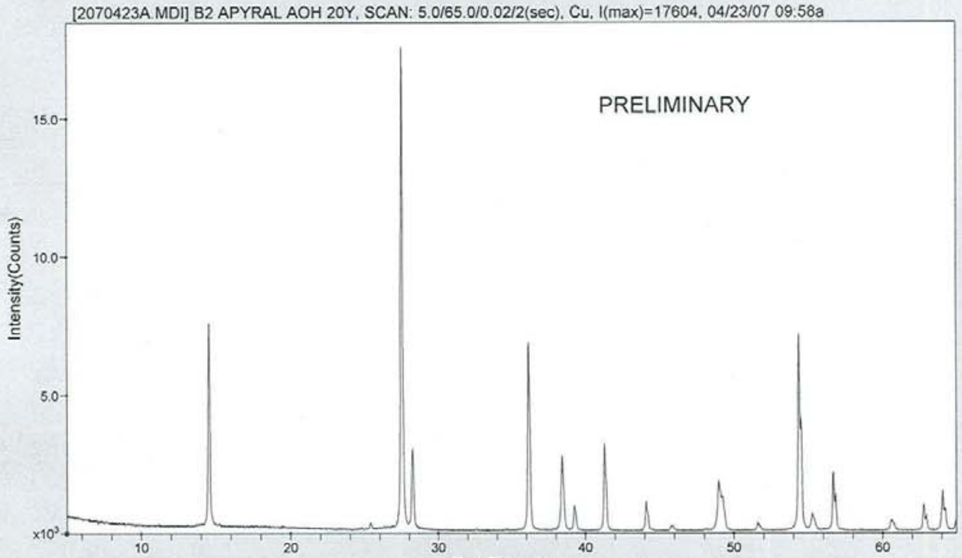


Figure 7.1. Raw data display, sample 2070423A B2 APYRAL AOH 20Y.

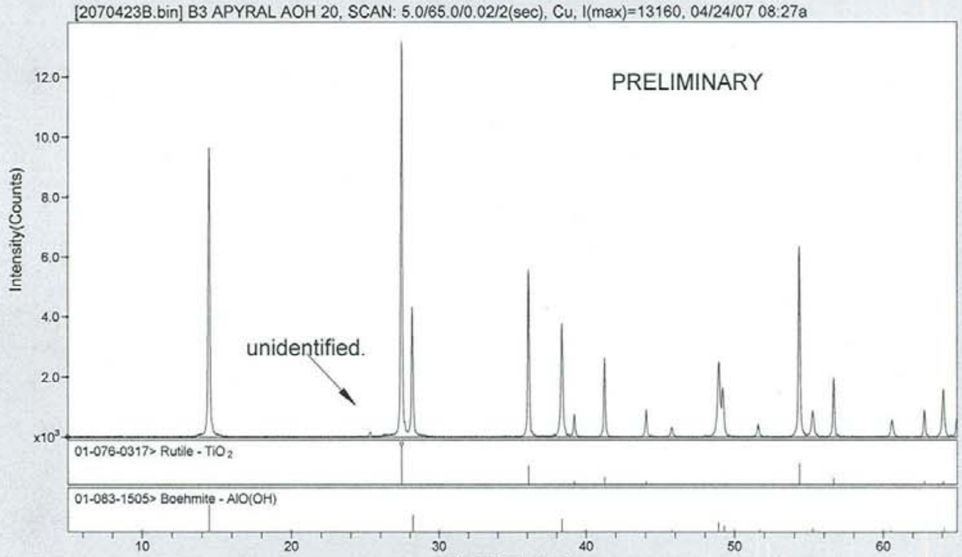


Figure 8.2. Phases identified, sample 2070423B APYRAL AOH 20.

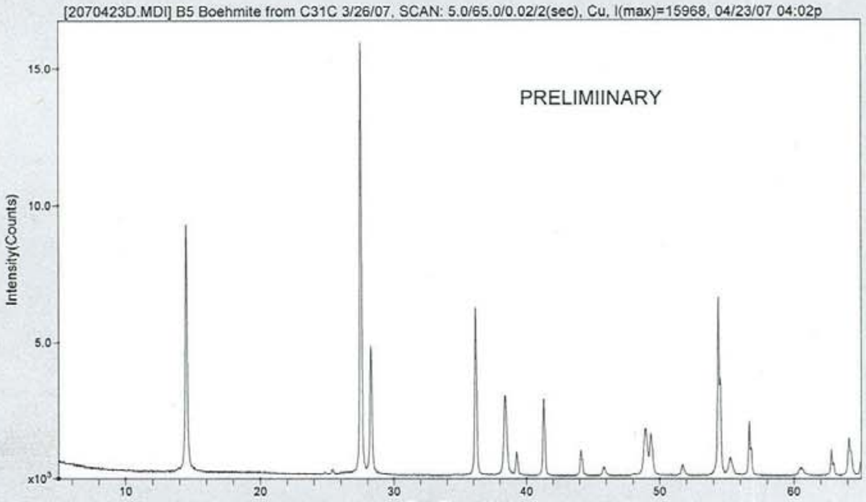


Figure 10.1. Raw data display, sample 2070423D, B5 Boehmite from C31C.

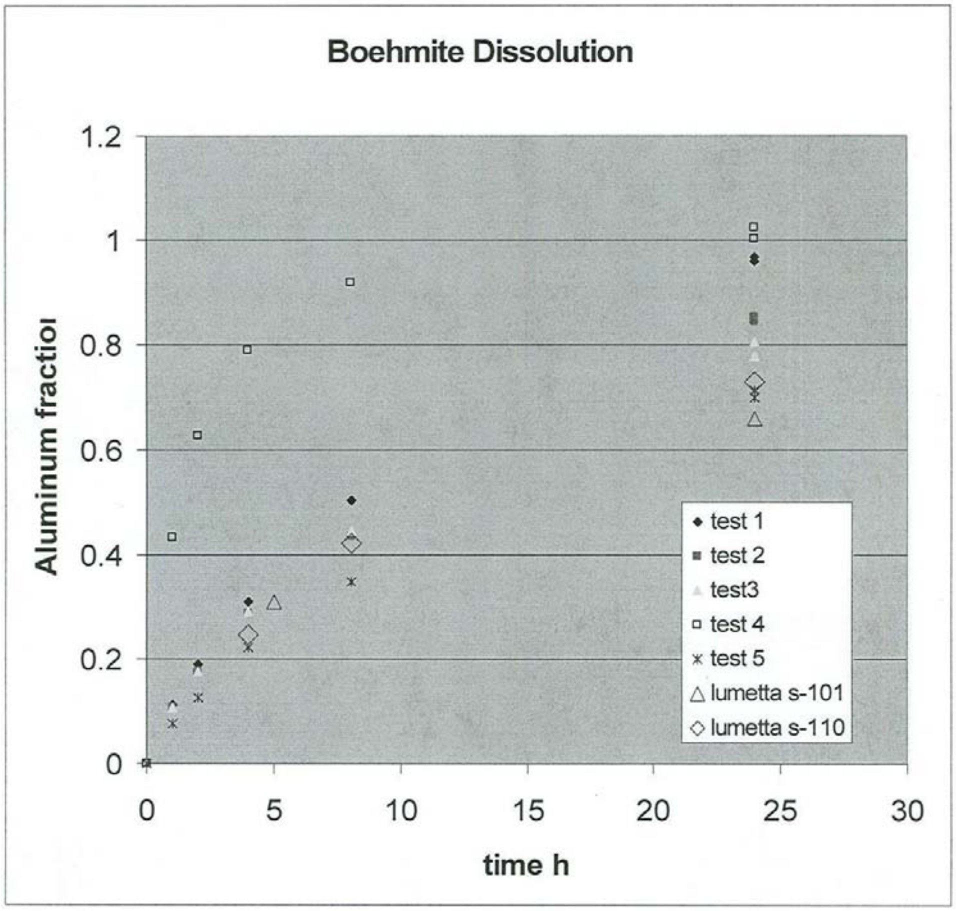
Boehmite Characterization Leaching Test

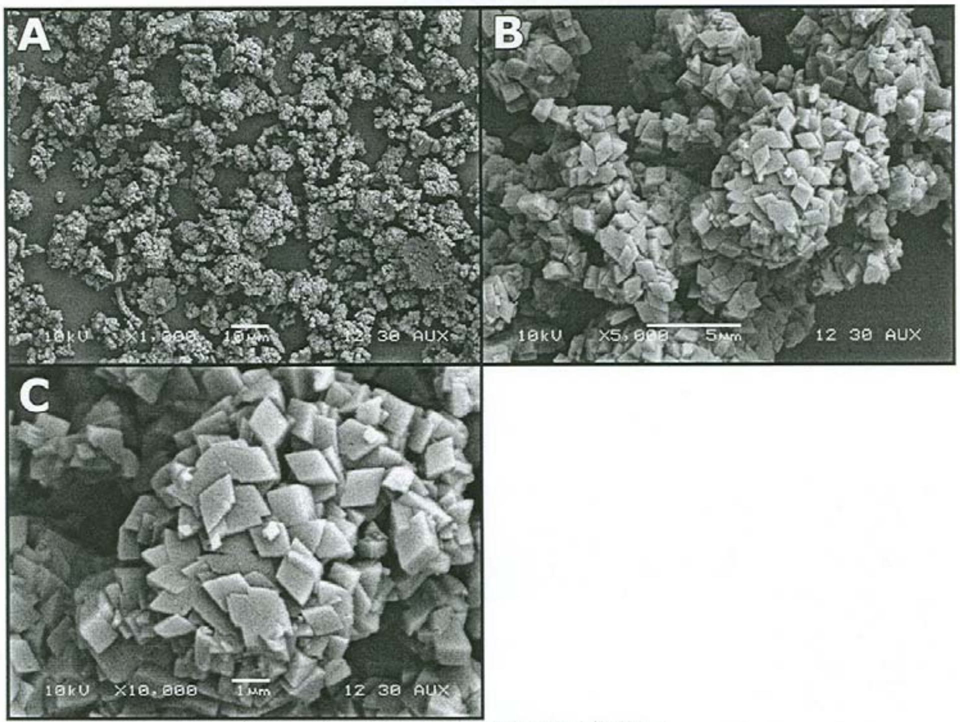
The results from the boehmite leaching tests are given in the table below.

	Fraction of Boehmite Dissolved				
Time - h	B1	B2	B3	B4	B5
0	0	0	0	0	0
1	0.11131	0.106151	0.105556	0.430556	0.075198
2	0.188492	0.175992	0.174802	0.625	0.125794
4	0.309524	0.287698	0.289683	0.789683	0.220238
8	0.501984	0.434524	0.445437	0.918651	0.347222
24	0.958333	0.853175	0.805556	1.02381	0.712302
F	0.968254	0.845238	0.777778	1.003968	0.698413

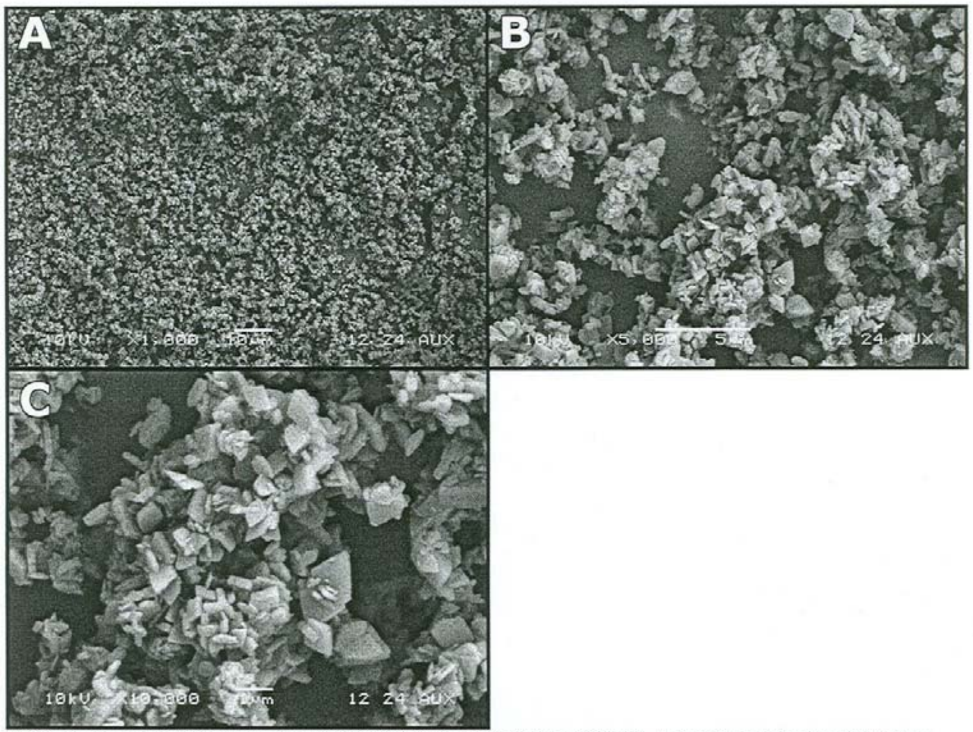
When plotted over the data from Lumetta, B3 and B5 match the results from S-101 and S-110 the best. Based on the SEM picture (shown below) the B3 is more crystalline whereas the B5 is more agglomerates with unsure particle sizes. B3 had a tight particle size of 8 µm whereas B5 has bimoc particle sizes from very small (~1 µm) to very large (~800 µm) with no real assurance of how accurate they were.

Therefore, we would recommend B3 because of the crystalline structure and particle size as well as is available in commercial quantities @ $1.95 per lb while B5 is a laboratory batch and we have littl experience to say how easily reproducible the material is. Also, it would be expensive to produce compared to the commercial material.





SEM micrographs of B3 APYRAL AOH 20Y. Average Particle Size of 0.83 µm.



SEM micrographs of Boehmite from C31C 300°C 4hr 3-26-07 blend 2,9,10. Average particle size of 0.67 µm.

If you have any questions, please contact Reid Peterson on 376-5340.

Sincerely,

Gordon H. Beeman

Gordon H. Beeman, Manager
RPP-WTP Support Program

GHB:c^2

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Pacific Northwest
National Laboratory
Operated by Battelle for the
U.S. Department of Energy

August 9, 2007

Mr. William L. Graves
Bechtel National Inc.
2435 Stevens Center Place, MSIN: H4-02
Richland WA 99352

WTP/RPP-MOA-PNNL-00087

Dear Mr. Graves:

**Subcontract NO. 24590-QL-HC9-WA49-00001 - Project 53019 (WA#2007-020)
Proposed Test Matrix for Blended Component Simulant Testing For Approval**

The purpose of this document is to provide BNI with the proposed test matrix for blend of component simulant testing for approval. This approval is required per Sections 6.1.1.6 and 6.1.2.6, in Test Plan TP-RPP-WTP-469 Rev 0, *Test Plan for the Development and Demonstration of Leaching and Ultrafiltration Simulants.*

Overview

Prior test work as part of this program has identified component simulants for gibbsite, boehmite and filtration behavior. Tests were performed using the gibbsite and boehmite simulant components only in simple sodium hydroxide test solution. The purpose of this set of tests is to measure the impact of the individual simulant components on the behavior of the other individual components. This documents deals only with the dissolution behavior of gibbsite and boehmite. The impact of the component simulants on filtration behavior will be dealt with in a later request. Also, the impact of various individual anions will be the subject of a separate test plan currently under development. Test Plan TP-RPP-WTP-469 Rev 0 calls for the test matrix to examine how the simulant components interact with each other and whether they hinder, help, or have no effect on the alumina dissolution.

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Proposed Test Matrix

Table 1 provides the sequence of tests proposed as part of this test matrix. Tests 1 through 6 provide a statistical design to evaluate the effect of gibbsite and filtration components on the dissolution of boehmite and the effect of filtration components on gibbsite dissolution. Tests 7 through 11 examine the effect of mixing energy on the dissolution of alumina. Tests 12 through 17 provide equilibrium measurements of boehmite solubility, which is important to evaluating boehmite dissolution kinetics. Tests 12 through 14 also examine the mixing energy effect and Tests 15 through 17 also examine the supernate effect on the dissolution of alumina.

Table 2 provides the composition of the supernate that will be used in the tests. It is based on a 3.2M Na concentration which matches the Group 5 waste supernate composition.

Table 1. Proposed Test Matrix.

Test	Oxalate (g)	Gibbsite (g)	Boehmite (g)	Sludge Simulant (g)	19M NaOH (g)	Condensate (g)	Supernate (g)	Mixing Speed (RPM)	[OH] Molarity	Na Molarity	Notes
1a	6.50	22.73	22.73	12.99	200.27	274.96	259.82	140	5.8	7.1	Full factorial for Gibbsite/Boehmite/Sludge Solids—oxalate as secondary variable
1b	6.50	22.73	22.73	12.99	200.27	274.96	259.82	140	5.8	7.1	Replicate of 1a
1c	6.50	22.73	22.73	12.99	200.27	274.96	259.82	140	5.8	7.1	Replicate of 1a
2	0.00	22.73	22.73	0.00	200.27	274.96	259.82	140	5.8	7.1	Full factorial for Gibbsite/Boehmite/Sludge Solids—oxalate as secondary variable
3	0.00	22.73	0.00	12.99	200.27	274.96	259.82	140	5.8	7.1	Full factorial for Gibbsite/Boehmite/Sludge Solids—oxalate as secondary variable
4	6.50	22.73	0.00	0.00	200.27	274.96	259.82	140	5.8	7.1	Full factorial for Gibbsite/Boehmite/Sludge Solids—oxalate as secondary variable
5	0.00	0.00	22.73	12.99	200.27	274.96	259.82	140	5.8	7.1	Full factorial for Gibbsite/Boehmite/Sludge Solids—oxalate as secondary variable
6	6.50	0.00	22.73	0.00	200.27	274.96	259.82	140	5.8	7.1	Full factorial for Gibbsite/Boehmite/Sludge Solids—oxalate as secondary variable
7	6.50	22.73	22.73	12.99	200.27	274.96	259.82	280	5.8	7.1	Mixing Energy—Compare Tests 1, 8 and 9
8	6.50	22.73	22.73	12.99	200.27	274.96	259.82	90	5.8	7.1	Mixing Energy—Compare Tests 1,7 and 9
9	6.50	22.73	22.73	12.99	200.27	274.96	259.82	50	5.8	7.1	Mixing Energy—Compare Tests 1, 7 and 8
10	6.50	0.00	22.73	0.00	200.27	274.96	259.82	90	5.8	7.1	Mixing Energy—Compare Tests 6 and 11
11	6.50	0.00	22.73	0.00	200.27	274.96	259.82	50	5.8	7.1	Mixing Energy—Compare Tests 6 and 10
12	7.02	24.56	24.56	14.04	152.07	297.06	280.70	140	4.5	5.9	Test of 45C condition and mixing energy—Compare to Tests 1, 13, and 14
13	7.02	24.56	24.56	14.04	152.07	297.06	280.70	90	4.5	5.9	Test of 45C condition and mixing energy—Compare to Tests 1, 12, and 14
14	7.02	24.56	24.56	14.04	152.07	297.06	280.70	50	4.5	5.9	Test of 45C condition and mixing energy—Compare to Tests 1, 12, and 14
15	7.02	24.56	24.56	14.04	152.07	577.76	0.00	140	4.5	4.5	Test of 45C condition and supernate effect—compare with test 12
16	7.02	24.56	24.56	14.04	152.07	577.76	0.00	90	4.5	4.5	Test of 45C condition and supernate effect—compare with test 13
17	7.02	24.56	24.56	14.04	152.07	577.76	0.00	50	4.5	4.5	Test of 45C condition and supernate effect—compare with test 14

Table 2. Supernate Composition at 3.2M Na

Component	Chemical Formula	Concentration (M)
Sodium phosphate	$Na_3PO_4\text{-}12H_2O$	0.013
Sodium sulfate	Na_2SO_4	0.008
Sodium carbonate	Na_2CO_3	0.576
Sodium oxalate	$Na_2C_2O_4$	0.016
Sodium nitrite	$NaNO_2$	0.528
Sodium nitrate	$NaNO_3$	1.44

Please contact Reid Peterson on 376-5340 if you have any questions.

Sincerely,

Gordon H. Beeman

Gordon H. Beeman, Manager
RPP-WTP Support Program

GHB:c^2

cc: RA Peterson (PNNL)
RL Russell (PNNL)
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