

PNNL-18755 WTP-RPT-191, Rev 0

Prepared for the U.S. Department of Energy under Contract DE-AC05-76RL01830

PEP Run Report for Integrated Test A; Caustic Leaching in UFP-VSL-T01A, Oxidative Leaching in UFP-VSL-T02A

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PEP Run Report for Integrated Test A; Caustic Leaching in UFP-VSL-T01A, Oxidative Leaching in UFP-VSL-T02A

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Test Specification: 24590-PTF-TSP-RT-07-001 Rev 2 Work Authorization: WA# 2007-024 Test Plan: TP-RPP-WTP-506, Rev. 0.4; TP-WTP-PEP-044, Rev 0.2 Test Exceptions: 24590-PTF-TEF-RT-08-00002 24590-WTP-TEF-RT-09-00003 24590-PTF-TEF-RT-09-00001 24590-WTP-TEF-RT-09-00002 Rev 0 24590-WTP-TEF-RT-09-00001 Rev 1 R&T focus area: Pretreatment

Test Scoping Statement: N/A

Prepared for the U.S. Department of Energy Under Contract DE-AC05-76RL01830

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-07-001 Rev 2 "Pretreatment Engineering Platform (PEP) Testing (Phase 1)" and Test Plans TP-RPP-WTP-506 Rev 0.4 "Pretreatment Engineering Platform (PEP) Testing (Phase 1) and TP-WTP-PEP-044 Rev 0.2" Test Plan for the PEP Parallel Laboratory Testing." The work and any associated testing followed the quality assurance requirements outlined in the Test Specification/Plan. The descriptions provided in this test 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 have been reviewed and verified.

Approved:

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

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Acknowledgments

In addition to the authors, this report was supported by many dedicated staff involved in laboratory experiments, sampling, data acquisition, and technical reviews.

Individuals on the operations and sampling crew are especially recognized for supporting the Pretreatment Engineering Platform Project by working unpredictable, long, and off-hour shifts for the ten months of PEP operations. The tests and reports could not have gotten this far without their extraordinary effort.

The authors would also like to thank Wayne Cosby for valuable editorial support, Dave MacPherson and Kirsten Meier for Quality Assurance support throughout the project, Chrissy Charron, Andrea Boehler, and Mona Champion for their administrative support through the months of PEP testing, and Wesley Lawrence, James Huckaby, Steve Barnes, Donald Bachand, Robert Burke, and Gordon Beeman for their leadership and technical guidance during PEP operations. We are also grateful to Brady Hanson and Michael Minette for the management and coordination of data analysis activities.

Abbreviations and Acronyms

Testing Summary

Pacific Northwest National Laboratory (PNNL) was tasked by Bechtel National Inc. (BNI) on the River Protection Project-Hanford Tank Waste Treatment and Immobilization Plant (RPP-WTP) project to perform research and development activities to resolve technical issues identified for the Pretreatment Facility (PTF). The Pretreatment Engineering Platform (PEP) was designed, constructed, and operated as part of a plan to respond to issue M12, "Undemonstrated Leaching Processes." The PEP, located in the Process Development Laboratory-West (PDL-W) in Richland, Washington, is a $\frac{1}{4}$.5-scale test platform^(a) designed to simulate the WTP pretreatment caustic leaching, oxidative leaching, ultrafiltration solids concentration, and slurry washing processes. The PEP replicates the WTP leaching processes using prototypic equipment and control strategies. The PEP also includes nonprototypic ancillary equipment to support the core processing.

The PEP testing program was conducted with a waste simulant that was developed in response to Task 5 from the M12 External Flowsheet Review Team (EFRT) issue response plan.^(b) The testing included the following tests with simulated Hanford tank waste:

- Shakedown/Functional testing: tested process operations (e.g., slurry transfers, steam heating of the vessels and the accumulation of condensate, filter backpulsing and flushing), process controls (e.g., transmembrane pressure [TMP] and axial flow velocity in the filter loop), and certain test functions (e.g., in-line slurry sampling accuracy and precision).
- Integrated Test A: demonstrated integrated processing when caustic leaching $(98^{\circ}C)$ is performed in UFP-VSL-00001A/B with the Cr simulant component added after the post-caustic-leach washing step.
- Integrated Test B: demonstrated integrated processing when the caustic leaching (98°C) is performed in UFP-VSL-00002A with the Cr simulant component added after the post-caustic-leach washing step.
- Integrated Test D: demonstrated integrated processing when the caustic leaching is performed at a lower temperature (85°C) in UFP-VSL-00002A and with the Cr simulant component added to the initial batch of simulant.

Integrated Test C was deleted from the scope of the testing as per ICN-TP-RPP-WTP-506_R0.2.^(c)

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⁽a) The scale of $\frac{1}{4}$. was chosen because this scale enables the ultrafiltration loop to be configured to meet two important criteria: 1) using one filter bundle, the ratio of solids in the feed tank to filter surface area will be the same as in the plant, and 2) using five filter bundles, the type and extent of mixing in the feed vessel will be approximately prototypic during the solids washing processes.

⁽b) SM Barnes, and R Voke. 2006. *Issue Response Plan for Implementation of External Flowsheet Review Team (EFRT) Recommendations - M12: Undemonstrated Leaching Process*. 24590-WTP-PL-ENG-06-0024 Rev. 0, Bechtel National, Inc., Richland, Washington.

⁽c) GB Josephson, OP Bredt, JK Young, and DE Kurath. 2009. *Test Plan for Pretreatment Engineering Platform (PEP) Testing (Phase I)*. TP-RPP-WTP-506, Rev 0.2, Pacific Northwest National Laboratory, Richland, Washington.

Two process flowsheets are currently being evaluated for the ultrafiltration process (UFP) and leaching operations. The baseline flowsheet has caustic leaching conducted in the UFP-1 ultrafiltration feed preparation vessels (i.e., vessels UFP-VSL-T01A and B in the PEP; vessels UFP-VSL-00001A and B [UFP-1] in the WTP PTF). The alternative scenario has caustic leaching performed in the UFP-2 ultrafiltration feed vessels (i.e., vessel UFP-VSL-T02A^(a) in the PEP and vessels UFP-VSL-00002A and B [UFP-2] in the WTP PTF).

With both flowsheets, 19-M sodium hydroxide solution (NaOH, caustic) is added to the waste slurry to leach solid aluminum compounds (e.g., gibbsite, boehmite). Caustic addition is followed by a heating step that uses direct injection of steam to accelerate the leaching process. Following the caustic leach, the vessel contents are cooled using vessel cooling jackets and/or external heat exchangers. The main difference between the two scenarios is that for leaching in UFP-1, the 19-M NaOH is added to unconcentrated waste slurry (3 to 8-wt% solids), while for leaching in UFP-2, the slurry is concentrated to nominally 20-wt% solids using cross-flow ultrafiltration before adding caustic. After cooling, the leached slurry is concentrated and washed with an aqueous solution of 0.01-M NaOH (referred to as inhibited water) to remove soluble salts. If the resulting waste solids remain high in chromium, sodium permanganate reagent is added, and the slurry is circulated to oxidize and dissolve the chromium solids. Following the oxidative leaching of chromium-containing solids, the slurry is washed to remove the dissolved chromium and concentrated.

The work described in this report includes a narrative of the conduct of Integrated Test A and the data obtained during the test. This report summarizes test operations and difficulties encountered during Integrated Test A. For test summary operations for the other tests, refer to WTP-RPT-190 for Shakedown/Functional Testing, WTP-RPT-192 for Integrated Test B, and WTP-RPT-193 for Integrated Test D.

In addition, reports have been written that specifically address the following:

- 1. Filtration Scale-Up (WTP-RPT-185)
- 2. Caustic Leaching Scale-Up (WTP-RPT-186)
- 3. Solids Washing (WTP-RPT-187)
- 4. Oxidative Leaching Scale-Up (WTP-RPT-188).

This report only includes very limited data analysis. Supporting data files have been supplied on transportable hard drives because of the large amount of data.

Objectives

1

Table S.1 summarizes the objectives for the entire PEP testing program along with a discussion of contributions made by the results of Integrated Test A to meeting these objectives.

⁽ a) In this report, the UFP vessels are generally denoted as Tank T01A/B and Tank T02A. In some cases alternative designations are used to maintain continuity with previous documentation (e.g., test specification/plan).

	Objective	
Test Objective	Met?	Discussion
Caustic leach process: Compare	Yes	Integrated Test A data were analyzed and compared with
engineering- and laboratory-scale		laboratory-scale data with results presented in reports WTP-RPT-186
results to determine impact of		and WTP-RPT-197.
scale-up.		
Oxidative leach process: Compare	Yes	Integrated Test A data were analyzed and compared with
engineering- and laboratory-scale		laboratory-scale data with results presented in reports WTP-RPT-188
results to determine impact of		and WTP-RPT-197.
scale-up.		
Cross-flow ultrafiltration: Monitor	Yes	Integrated Test A data were analyzed and compared with
cross-flow filter performance at		laboratory-scale data with results presented in reports WTP-RPT-185
engineering- and laboratory-scale		and WTP-RPT-197.
to determine scale-up.		
Slurry wash process: Determine	Yes	Integrated Test A data were analyzed with results presented in reports
the post-caustic and		WTP-RPT-187 and WTP-RPT-197.
oxidative-leaching slurry wash		
efficiencies.		
Process integration: Evaluate the	Yes	Supporting data from Integrated Test A are presented, and results to
chemical addition, filter operation		meet this objective are discussed in WTP-RPT-197.
cycle performance, and pressure		
pot operations. Also perform mass		
balances for aluminum, chromium,		
manganese, sodium, hydroxide,		
oxalate, phosphate, sulfate, and		
water and monitor permeates for		
post-filtration precipitation.		
Monitor the performance of the	Yes	The data required to meet this objective were provided on compact
recirculation system pumps, filters,		discs transmitted in the following reference: Letter from GH Beeman
and heat exchanger to support		to H Hazen, "Subcontract No. 24590-QL-HC9-WA49-00001, Project
engineering fabrication decisions		No. 53569 (WA-024) Engineering Ties Data Transmittal: The
for these components.		Electronic File Enclosed With This Letter Has Been Reviewed for
		Technical Accuracy Per the QA Program,
		WTP/RPP-MOA-PNNL-00392, dated 4/10/09.

Table S.1. Summary of Test Objectives and Results

Test Exceptions

A summary description of the Test Exceptions applied to these tests is shown in Table S.2.

Test Exceptions	Description of Test Exceptions
1) 24590-PTF-TEF-RT-08-	This test exception:
00002 incorporated into ICN-1	Added a stage during the filter conditioning section of the 1.
to Test Plan TP-RPP-WTP-506.	Shakedown/Functional Test where the simulant slurry is concentrated from
	approximately 5-wt% solids to 20-wt% solids in one operation. This is in
	addition to the previously specified low-solids filter and high-solids filter
	testing.
	Documented the Joint Test Group (JTG) decision regarding the number of 2.
	replicate samples to be collected at various processing times.
	Revised the terminology specifying the Coriolis densitometer (CD) sample 3.
	locations that were changed to be consistent with PEP operating procedures.
	Renamed the "center" array to "inner."
	The sampling specified in the low-solids filtration test over-specifies the 4.
	sample collection timing required. The technical requirement is to get 30
	unique samples. The sampling schedule specified is not required to achieve
	this test objective.
2) 24590-PTF-TEF-RT-09-	In several steps, the sampling location was changed from the filer-loop 1.
00001 incorporated into ICN-2	in-line location to a middle-low CD sample loop location in the
and ICN-3 to Test Plan	UFP-VSL-T02A vessel. This change impacted sampling in the
TP-RPP-WTP-506.	Shakedown/Functional and all of the Integrated tests (ref CCN 187749).
	Added a step to the Shakedown/Functional Test (step A.1.31) to add sodium 2.
	permanganate to UFP-VSL-T02A to assess possible foaming issue (ref CCN
	187749).
	Changed location of second sample for parallel laboratory-scale Cells Unit 3.
	Filter (CUF) testing from the in-line filter loop to the middle-low CD port in
	the UFP-VSL-T02A (Step A.1.10; Shakedown/Functional Test) (ref CCN
	187749).
	Collected samples for parallel laboratory leaching test before and after caustic 4.
	addition in UFP-VSL-T01A (A.1.20; Shakedown/Functional Test) and
	UFP-VSL-T02A (Step A.1.15; Shakedown/Functional Test), and in the
	Integrated Test steps (B.1.2; Integrated Test A; B.2.6; Integrated Tests B/D)
	(ref CCN 192734).
	Deleted reconfiguration of the filter loop to bypass UFP-VSL-T02A and 5.
	circulate flush water with UFP-PMP-T42A and/or UFP-PMP-T43A to allow
	a representative in-line sample to be collected. This step (Step A.1.17;
	Shakedown/Functional Test) could not be done under the operating
	restrictions in place on the operation of the filter loop (ref CCN 192734.)
	Eliminated Step A.1.25 (filter-loop bypass test with tracer) from the 6.
	Shakedown/Functional Test. This test was conducted after Integrated Test B
	was completed (ref CCN 187753).
	Modified Step A.1.29 (Shakedown/Functional Test) to eliminate the removal 7.
	of solids from UFP-VSL-T02A before the high-solids filter test. This step
	was not needed as the amount of solids is less than anticipated (ref CCN
	187752).
	Modified Step A.1.30 (Shakedown/Functional Test) to include five filter 8.
	backpulses before starting the high-solids filter test (ref CCN 187752).
	Modify Step B.1.8 (Integrated Test A) to allow 80% of caustic to be added 9.
	during in-line simulant transfers to UFP-VSL-T01B and 20% to be added
	directly to UFP-VSL-T01B (ref CCN 187748).

Table S.2. Test Exceptions

As documented in the PEP Test Plan, the deviations from the Test Specification are provided in Table S.3.

Test Specification Reference		Exception Taken
Section 6.4.4 "Analytical measurements will be		Three method exceptions are required under this Test Plan:
made in conformance to the Guidelines for		1. Caustic leach and oxidative leach samples taken during this
Performing Chemical Physical, and Rheological		testing must be separated more quickly than the standard
<i>Properties Measurements</i> ^(a) as applicable."		method using syringes. This testing will use a modified
		method with a shorter centrifuge time and will apply higher
		g forces (e.g., 4000 g vs. 1000 g).
		Impact on results: If the standard method were used, the
		longer time could very well lead to greater dissolution and
		inaccurate results. Laboratory testing will be conducted
		with simulants to confirm that this method of sample
		handling is adequate.
	2.	Densities of samples smaller than 10-mL can only be
		established within two significant figures of accuracy.
		Density measurements for this Test Plan require greater
		accuracy. Therefore, a more accurate method employing a
		pycnometer will be used.
		Impact on results: The change to a pycnometer will
		generate more precise results than the standard method.
		The main impact is expected to be on analysis time. The
		pycnometer method will be slower.
	3.	The process for determining the wt% undissolved solids
		(UDS) content of the slurries will in some cases be
		determined with the use of a moisture analyzer. In
		addition, the method of drying samples will be modified to
		allow glass fiber filters to be used in drying the samples.
		Impact on results: Both modifications are intended to
		decrease the time required to obtain results.

Table S.3. Deviations from Test Specification

Results and Performance Against Success Criteria

The PEP system tests were designed to generate the data necessary to:

- Provide engineering-scale system performance data. This information is used to support the projections of the WTP computer process models for the waste-processing campaign.
- Confirm the operability and functionality of UFP system components.

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The WTP Research and Technology (R&T) success criteria for achieving these objectives are discussed in Table S.4. The success criteria for the entire PEP testing program are provided with discussion limited to the success criteria covered by the scope of this report. The success criteria not addressed in this report are shaded in gray.

⁽a) GL Smith and K Prindiville. 2002. *Guidelines for Performing Chemical, Physical, and Rheological Properties Measurements*. 24590-WTP-GPG-RTD-001 Rev. 0, Bechtel National, Inc., Richland Washington.

Success Criteria	How Testing Did or Did Not Meet Success Criteria
UFP System Process Performance	
Measure the aluminum leaching performance of the PEP and laboratory systems as a function of time under WTP UFP-1 and UFP-2 projected leaching conditions at bounding high and low process temperatures (nominally 100°C and 80° C).	Aluminum leaching at 98°C was measured as a function of time under WTP UFP-1 projected leaching conditions during Integrated Test A. Results are presented in this report and in reports WTP-RPT-186 and WTP-RPT-197.
Compare aluminum leach performance in UFP-1 where all of the NaOH is added in-line to the case where a fraction of the	Results to meet this success criterion are discussed in report WTP-RPT-197.
total NaOH is added directly to the tank.	
Measure chromium leaching performance in the PEP and laboratory systems as a function of time at the WTP projected conditions in UFP-2 for both the UFP-1 and UFP-2 aluminum leaching flowsheets.	Chromium leaching was measured as a function of time under WTP projected leaching conditions during Integrated Test A. Results to meet this success criterion are discussed in reports WTP-RPT-188 and WTP-RPT-197.
Evaluate the process control strategy for specification of required reagent additions, including NaOH, NaMnO ₄ , and wash solutions provided in the PEP Phase 1 Testing Process Description.	The process control strategy for specifying the amount of reagent was to analyze a sample of the simulant feed for aluminum and chromium content and specify the amount of NaOH and NaMnO ₄ based on the result. The amount of reagent added was correct within the analytical error of the analysis method. A comparison of targeted and delivered reagent additions is provided in report WTP-RPT-188 for Integrated Tests A and B. Additional discussion and results for Integrated Test D are provided in report WTP-RPT-197.
Measure the filter system performance at the nominal flow velocity and TMPs for the solids concentration and washing stages for the UFP-1 and UFP-2 aluminum leaching flowsheets. Evaluate the control strategy for make-up additions from UFP-VSL-00001A/B to	Filter performance was measured at a nominal axial flow velocity of 15 ft/sec and a TMP of 40 psid during solids concentration and washing during Integrated Test A. Actual permeate flux and filtration conditions are provided in this report. A summary of filter system performance is provided in WTP-RPT-197. The control strategy for make-up additions between
UFP-VSL-00002A/B during initial dewatering process.	UFP-VSL-T01A/B and UFP-VSL-T02A was demonstrated during post-caustic-leach concentration in Integrated Test A. The control strategy was determined to be adequate, although maintaining a high operating level in UFP-VSL-T02A until the end of processing should reduce the possibility of entraining air into the recirculation pumps. Results to meet this success criterion are discussed in report WTP-RPT-197.
Measure the wash water volumes required to remove or reduce the free hydroxide following the aluminum leaching stage and dissolved chromium after the oxidative leaching process to the specified concentrations.	The volume of wash water required to reduce free hydroxide following the aluminum leach was evaluated for Integrated Test A, and results to meet this success criterion are discussed in reports WTP-RPT-187 and WTP-RPT-197.

Table S.4. Success Criteria

Success Criteria	How Testing Did or Did Not Meet Success Criteria
Confirm whether the WTP process control	Results to meet this success criterion are discussed in report
strategies for ultrafilter system filling,	WTP-RPT-197.
operating, backpulsing, draining, flushing,	
and cleaning are adequate for stable operation. Provide to WTP data to	
determine whether backpulsing is a	
required and effective means of restoring	
the filter permeate rates to make certain	
that production throughput is maintained	
and determine whether operation of the	
backpulse system induces any process or	
equipment operations issues.	
Use only the process information and data	This success criterion was met by developing a run sheet of all the
available to the WTP PTF operating staff	operating parameters (e.g., transfer volumes, reagent addition
during WTP operations (e.g., caustic and	volumes, control levels) based on prototypic characterization data
permanganate addition volumes, permeate mass balances for solids concentration) to	before the start of Integrated Test A. Changes to the run sheet made during the test itself were based only on data that would be available
operate the PEP.	to the plant, and were not, for example, based on information from
	nonprototypic samples. This success criterion is fully discussed in
	WTP-RPT-197.
Confirm whether the elevated temperature	Results to meet this success criterion are discussed in report
pulse-jet mixer (PJM) operating strategy is	WTP-RPT-197.
adequate for stable PEP and WTP	
operation.	
Measure the heat-up rate and	The heat up, thermal control at 98°C, and cool-down performance of
controllability of the PEP UFP-VSL-00001 and UFP-VSL-00002 vessels and the	UFP-VSL-T01A/B was measured during Integrated Test A. Thermal profiles are provided in this report. This information for the other
cooling performance for UFP vessels.	tests is discussed in the other run reports and summarized in
	WTP-RPT-197.
Measure the performance of the in-line	The blending of process chemicals added in-line is presented in
addition of process chemicals into the	reports WTP-RPT-190 and WTP-RPT-197.
simulated wastes and determine the extent	
of blending in the process vessels.	
Monitor ultrafilter performance (to include	Ultrafilter visual inspection results are presented in reports
visual inspection of the filter tubes, tube	WTP-RPT-193 and WTP-RPT-197.
sheets, and heads from an ultrafilter for any evidence of flow mal-distribution	
and/or solids buildup at least once during	
Phase 1).	
Measure, record, and control ultrafiltration	Slurry flow rate, temperature, and TMP were recorded and controlled
temperature, TMP, and slurry flow during	during Integrated Test A. Results are provided in this report.
filter-loop operations.	
Record any solids accumulations observed	Results to meet this success criterion are discussed in report
during any operating stage or maintenance	WTP-RPT-197.
evolution.	

Table S.4. Success Criteria

Success Criteria	How Testing Did or Did Not Meet Success Criteria
Monitor the permeate production rate of	The permeate production rate of each ultrafilter was recorded during
each ultrafilter assembly in operation.	Integrated Test A. Results are provided in this report. The permeate
	production rates for each test are presented in the relevant run report.
Record operating time of each ultrafilter	The operating time of each ultrafilter assembly was recorded during
assembly.	Integrated Test A. Results are provided in this report. The operating
	time of each filter assembly for each test is discussed in the relevant
	run report and summarized in report WTP-RPT-197.
Record each ultrafilter assembly cleaning	Results to meet this success criterion are discussed in reports
event (backpulse, flush, chemical cleaning,	WTP-RPT-190 and WTP-RPT-193 and are summarized in
$etc.$).	WTP-RPT-197.
Evaluate the pulse pot operation and	Backpulsing was conducted to increase the declining permeate rate
backpulse operation strategies contained in	through the course of post-caustic-leach concentration. Backpulse
Pretreatment Engineering Platform (PEP)	operations are evaluated in report WTP-RPT-197.
Phase 1 Testing Process Description.	
Evaluate permeate and permeate blends for	Permeate samples were collected during Integrated Test A for
precipitation of solids, particularly	precipitation studies. Results to meet this success criterion are
aluminum and oxalate solids.	discussed in reports WTP-RPT-197, WTP-RPT-200, and
	WTP-RPT-205.

Table S.4. Success Criteria

Quality Requirements

1

The PNNL Quality Assurance (QA) Program is based upon the requirements as defined in the U.S. Department of Energy (DOE) Order 414.1C, *Quality Assurance* and 10 CFR 830, *Energy/Nuclear Safety Management*, Subpart A—*Quality Assurance Requirements* (a.k.a. the Quality Rule). PNNL has chosen to implement the following consensus standards in a graded approach:

- ASME NQA-1-2000: Quality Assurance Requirements for Nuclear Facility Applications, Part 1, *Requirements for Quality Assurance Programs for Nuclear Facilities*
- ASME NQA-1-2000, Part II, Subpart 2.7: Quality Assurance Requirements for Computer Software for Nuclear Facility Applications
- ASME NQA-1-2000, Part IV, Subpart 4.2: Graded Approach Application of Quality Assurance Requirements for Research and Development.

The procedures necessary to implement the requirements are documented in PNNL's "How Do I…?" (HDI) .^(a)

The RPP-WTP quality requirements are implemented by performing work in accordance with the *River Protection Project—Hanford Tank Waste Treatment and Immobilization 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),* as applicable. These quality requirements are implemented through the *River Protection Project—Waste Treatment*

⁽a) The system for managing the delivery of laboratory-level policies, requirements, and procedures.

Plant Support Program (RPP-WTP) Quality Assurance Manual (RPP-WTP-QA-003, QAM). The requirements of DOE/RW-0333P Rev 13, *Quality Assurance Requirements and Descriptions (QARD)* and 10 CFR 830 Subpart A were not required for this work.

The RPP-WTP addresses internal verification and validation activities by conducting an independent technical review of the final data report in accordance with RPP-WTP's procedure QA-RPP-WTP-604. This review procedure is part of PNNL's *RPP-WTP Quality Assurance Manual* (RPP-WTP-QA-003). Following this procedure, a technical review would verify that the reported results are traceable, that inferences and conclusions are soundly based, and the reported work satisfies the objectives.

R&T Test Conditions

The R&T test conditions as defined in the Test Specification are summarized in Table S.5. The R&T test conditions for the entire PEP testing program are provided with discussion limited to the R&T test conditions covered by the scope of this report. R&T test conditions not addressed in this report are shaded in gray.

List R&T Test Conditions	Were Test Conditions Followed?
General Requirements	
Perform mass balances for selected constituents,	Necessary samples were taken to conduct a mass
including aluminum, chromium, manganese, sodium,	balance for Integrated Test A. Mass balance results are
hydroxide, oxalate, phosphate, sulfate, and water, to	provided in report WTP-RPT-197.
evaluate leaching and washing process performance.	
Evaluate ultrafilter performance (to include visual	This R&T test condition is discussed in reports
inspection of the filter tubes, tube sheets, and heads from	WTP-RPT-193 and WTP-RPT-197.
an ultrafilter for any evidence of flow mal-distribution	
and/or solids buildup or evidence of potential failure).	
Assess the blending achieved during in-line additions of	In-line addition of wash water during Integrated
leaching and washing solutions.	Tests A and B is discussed in reports WTP-RPT-187
	and WTP-RPT-197.
Record any solids accumulations observed during any	This R&T test condition is discussed in report
operating stage or maintenance evolution (e.g.,	WTP-RPT-197.
photography, particle-size distribution).	
Leaching Operations	
Maintain caustic leaching temperature at the required	The temperature during caustic leaching was
setpoint and record steam usage to remain in the	maintained at setpoint with steam usage measured
temperature range.	during Integrated Test A with the exceptions provided
	in this report. Additional discussion of this condition is
	provided in reports WTP-RPT-186 and WTP-RPT-197.
Maintain oxidative leaching temperature at the required	The temperature during oxidative leaching was
setpoint.	maintained at setpoint during Integrated Test A.
	Additional discussion of this condition is provided in
	reports WTP-RPT-188 and WTP-RPT-197.
Obtain periodic samples during the leaching operations to	This R&T condition was met for the caustic-leaching
monitor the amount of aluminum or chromium that has	tests discussed in this report. Additional discussion of
dissolved and concentrations of the reactants and	this R&T condition is provided in reports
products in the liquid fraction in the vessel.	WTP-RPT-186, WTP-RPT-188, and WTP-RPT-197.

Table S.5. R&T Test Conditions

List R&T Test Conditions	Were Test Conditions Followed?
Provide data to demonstrate the WTP process control	This R&T test condition is discussed in report
strategy for the caustic and permanganate addition.	WTP-RPT-197.
Measure the rheology of the slurry simulant and the shear	Slurry rheology was measured with samples taken
strength of the settled solids before and following each	before and after each leaching unit operation. Results
leaching unit operation.	are provided in this report. Rheology results for the
Concentration Operations	other tests are included in the relevant run reports.
Monitor the permeate production rate of each ultrafilter	Permeate production for each ultrafilter assembly was
assembly in operation.	monitored during testing, and results are provided in
	this report. Permeate production rates for the other
	tests are included in the relevant run reports.
Record operating time of each ultrafilter assembly.	The operating time of each ultrafilter was recorded
	during Integrated Test A, with results discussed in
	report WTP-RPT-197.
Record each ultrafilter assembly "cleaning" event	Ultrafilter cleaning events were not part of Integrated
(backpulse, flush, chemical cleaning, etc.).	Test A efforts; therefore, they are not included in this
	report. However, backpulse events are summarized in
	this report. A summary of the filter cleaning events is
	provided in report WTP-RPT-197, and details of the
	final prototypic filter cleaning are presented in report WTP-RPT-193. Additional filter cleaning events are
	also discussed in reports WTP-RPT-190 and
	WTP-RPT-192.
Confirm pulse pot operation and backpulse operation	This R&T test condition is discussed in report
strategies.	WTP-RPT-197.
Control ultrafiltration temperature, TMP, and slurry flow	Ultrafilter temperature, TMP, and slurry flow rate were
as specified in test-specific run sheets.	controlled. Deviations from values specified in the
	Integrated Test A run sheet are noted in this report.
	R&T conditions for the other tests are included in the
	relevant run reports.
Collect and retain permeate samples for extended	Samples were collected and retained for extended
precipitation studies (including permeate/simulated	precipitation studies. The results of the precipitation
supernatant blended cases) from each concentration	studies are discussed in reports WTP-RPT-197, WTP-RPT-200, and WTP-RPT-205.
cycle. Demonstrate WTP ultrafiltration system control scheme	This R&T test condition is discussed in report
in normal operating modes (e.g., fill and startup,	WTP-RPT-197.
operation, backpulsing, flush and drain, cleaning and	
return to service).	
Washing Operations	
Wash slurries using a washing protocol to be specified in	Slurries were washed as specified in the Integrated
test specific run sheets.	Test A run sheet. Washing results are discussed in
	reports WTP-RPT-187 and WTP-RPT-197.
Sample permeate immediately before each wash solution	Permeate was sampled and monitored during washing
addition to monitor washing performance/efficiency.	with results provided in reports WTP-RPT-187 and
	WTP-RPT-197.

Table S.5. R&T Test Conditions

List R&T Test Conditions	Were Test Conditions Followed?
Measure rheology of the washed solids.	The rheology of washed solids was measured with
	results provided in this report. Rheology results for the
	other tests are included in the relevant run reports with
	selected results also discussed in reports WTP-RPT-187
	and WTP-RPT-197.

Table S.5. R&T Test Conditions

Simulant Use

PEP process testing was performed with a nonradioactive aqueous slurry of simulant waste chemicals and solids. The simulant composition and make-up recipe were provided by WTP as documented in Simulant Recommendation for Phase 1 Testing in the Pretreatment Engineering Platform.^(a) Aqueous chemical concentrations were within the ranges expected for waste feeds to the PTF. The hydroxide concentration was marginally one standard deviation lower than the average concentration expected in the feeds to the plant. The oxalate and phosphate components were at the lower end of the expected ranges, but the oxalate component was at the solubility limit, and the phosphate component was at or near the solubility limit. The solids components and blend were selected to obtain targeted solids mass loss (aluminum and chromium leaching and oxalate washing) and treatment time. The simulant was not selected to represent any particular Hanford tank waste type.

The simulant was blended from the components listed below. The basis for selecting the individual components and the comparison to actual waste behavior is provided where applicable in the indicated references:

- Boehmite (for Al) (Russell et al. 2009a)
- Gibbsite (for Al) (Russell et al. 2009b)
- Chromium oxyhydroxide (CrOOH) slurry (Rapko et al. 2007)
- Sodium oxalate
- Filtration simulant (Russell et al. 2009c)
- Supernate.

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A separate chromium solids slurry simulant was prepared and added to the PEP process after post-caustic-leach washing (a nonprototypic addition) during the Shakedown/Functional Tests and Integrated Tests A and B. This approach was taken because laboratory-scale tests had shown that the high-temperature caustic leaching step dissolved significant amounts of the CrOOH solids (Russell et al. 2009a). In Integrated Test D, the chromium solids component of the simulant was added during the simulant make-up process to demonstrate the PTF permanganate addition strategy.

⁽a) P Sundar. 2008. *Simulant Recommendation for Phase 1 Testing in the Pretreatment Engineering Platform*. 24590-PTF-RPT-RT-08-006, Rev 0, Bechtel National, Inc., Richland, WA.

Simulant was procured from NOAH Technologies Corporation (San Antonio, TX). Samples of each simulant batch were characterized to make certain that chemical and physical properties requirements were met. Batches of the simulant were procured as follows:

- A 15-gallon trial batch of the blended simulant for laboratory testing to demonstrate the efficacy of the simulant fabrication procedure.
- A 250-gallon scale-up batch of the blended simulant to demonstrate scale-up of the simulant fabrication procedure to an intermediate scale.
- Batches 0, 1, and 2, each nominally 3500 gallons, of blended simulant for the Shakedown/Functional Tests and Integrated Tests A and B. These batches did not contain the CrOOH component.
- The CrOOH solids slurry for the Shakedown/Functional Test and Integrated Tests A and B was obtained in two separate batches, containing nominally 18 and 36 kg of Cr as CrOOH.

Discrepancies and Follow-on Tests

No discrepancies or follow-on tests were identified.
1.0 Introduction

This report summarizes test activities for Integrated Test A. Integrated Test A is the first of three tests aimed to address concerns raised by the External Flowsheet Review Team (EFRT). In October 2005, the EFRT reviewed the design of the Hanford Tank Waste Treatment and Immobilization Plant (WTP) and identified several issues that could impact operation of the plant if not addressed. In response to the EFRT issues, an engineering-scale integrated platform—named the Pretreatment Engineering Platform (PEP)—was built to demonstrate the WTP system design and treatment process flowsheet.

The PEP is a $\frac{1}{4.5}$ linear scale test platform located in the Process Development Engineering Laboratory-West (PDL-W) at Pacific Northwest National Laboratory (PNNL). Area dimensions are $\frac{1}{4.5^2}$ scale, and volume dimensions are $\frac{1}{4.5^3}$ scale. The PEP is designed to simulate the WTP pretreatment caustic leaching, oxidative leaching, ultrafiltration solids concentration, and slurry washing processes. In addition, PEP operations collect data needed for design input and to improve prediction of full-scale plant performance and demonstrate component operation and operating characteristics. PEP equipment for conducting these core processes has been designed to be prototypic of the plant; however, it also includes nonprototypic ancillary equipment.

This is one in a series of reports that summarize Phase 1 testing results from PEP. Separate run reports have been prepared for each Phase 1 test (i.e., Shakedown/Functional Testing [Josephson et al. 2009], Integrated Test B [Geeting et al. 2009], and Integrated Test D [Sevigny et al. 2009]). In addition, reports have been written that specifically address the following:

- 1. Filtration Scale-Up (Daniel et al. 2009)
- 2. Caustic Leaching Scale-Up (Mahoney et al. 2009)
- 3. Solids Washing (Baldwin et al. 2009)
- 4. Oxidative Leaching Scale-Up (Rapko et al. 2009).

Integrated Test A was conducted per TI-WTP-PEP-065, written in accordance with Test Plan TP-RPP-WTP-506,^(a) which was written in response to Test Specification 24590-PTF-TSP-RT-07-001, Rev 2.^(b) Integrated Test A demonstrated the integrated processes of caustic leaching in UFP-VSL-T01A/B at 98ºC and oxidative leaching in UFP-VSL-T02A.

1.1 Purpose

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The purpose of this testing program is to provide WTP with data to "...confirm the ultrafiltration process (UFP) system design and sludge treatment process flowsheet."^(c) The system design and

⁽a) G Josephson, O Bredt, J Young, and D Kurath. 2008. *Test Plan: Pretreatment Engineering Platform (PEP) Testing (Phase I).* TP-RPP-WTP-506, Rev 0, Pacific Northwest National Laboratory, Richland, WA, 99352.

⁽b) JL Huckaby and JR Markillie. 2008. *Pretreatment Engineering Platform (PEP) Testing (Phase I).* 24590-PTF-TSP-RT-07-001, Rev 2, Bechtel National, Inc., Richland, Washington.

⁽c) SM Barnes and R Voke. 2006. *Issue Response Plan for Implementation of External Flowsheet Review Team (EFRT) Recommendations—M12, Undemonstrated Leaching Processes*. 24590-WTP-PL-ENG-06-0024 Rev. 0, p. 24; Bechtel National, Inc., Richland, Washington.

treatment process flowsheet will be confirmed by evaluating the following operations on an engineering-scale integrated platform called the PEP:

- Solids concentration
- Aluminum leaching
- Washing
- Chromium leaching.

Tests were used to collect data needed for design input and to improve prediction of full-scale plant performance:

- Demonstration of component operation and determination of operating characteristics
- Integrated simulations of leaching scenarios.

The PEP data will be used by WTP along with laboratory-scale data and appropriate application of scaling to improve the WTP plant models used to predict plant operating performance.

1.2 Background

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In October 2005, an EFRT was assembled to challenge and provide a critical review of the WTP design. The review identified a number of issues and potential issues that if not addressed could impact the operation of the plant.^(a) One of the issues that must be addressed in the Pretreatment Facility (PTF) is "Undemonstrated Leaching Processes." The EFRT reported that neither the caustic leaching nor the oxidative leaching process has been demonstrated at greater than bench scale. Without a scale-up study, the EFRT indicated that the ability to predict the effectiveness of these processes is limited.

The WTP project under BNI and the U.S. Department of Energy (DOE) developed an issue response plan for implementation of EFRT recommendations: M12, Undemonstrated Leaching Processes to resolve the "Undemonstrated Leaching Processes" issue.^(b) The plan addresses related topics that are not specifically in response to EFRT concerns. These include caustic addition and leaching concerns that were better understood after the EFRT report was issued, information to support revision of the contract design basis for the PTF, including system capacities, and earlier initiatives on enhancing plant throughput capacity. The solution for closing this issue includes conducting engineering-scale testing of all leaching (caustic and oxidative), washing, and filtration processes. $^{(a)}$

The flowsheet and equipment design demonstration will be performed in the following two phases:

Phase 1—Confirm the UFP system design and sludge treatment process flowsheet.^(a)

⁽a) JP Henschel. March 17, 2006. Contract No. DE-AC27-01RV14136 - *Report of External Flowsheet Review Team for the Hanford Tank Waste Treatment and Immobilization Plant: "Comprehensive Review of the Waste Treatment Plant Flowsheet and Throughput."* Letter to RJ Schepens. CCN 132846, Bechtel National, Inc., Richland, Washington.

⁽b) S Barnes and R Voke. 2006. *Issue Response Plan for Implementation of External Flowsheet Review Team (EFRT) Recommendations—M12, Undemonstrated Leaching Processes*. 24590-WTP-PL-ENG-06-0024, Rev 0, p. 5; Bechtel National, Inc., Richland, Washington.

Phase 2—Confirm the UFP system performance over a range of anticipated plant process conditions including the principal types of WTP feeds.

The data provided by the Phase 1 engineering-scale testing will be used to confirm the performance of the selected process flowsheet design and equipment based on post-Phase 1 test modeling.

On October 23 and 24, 2006, a WTP Project ultrafiltration leaching process technical review meeting was held to review the Issue Response Plan for M12.^(a) Participants included members of DOE, EFRT, the WTP project, and PNNL. At the meeting, PNNL made recommendations on the methodology and scale for the leaching processes. (a)

Members of the EFRT also presented their assessment (*Integrated Test Platform, M12—Test Review*) with specific recommendations.^(a) The general system requirements recommended by EFRT and PNNL staff members included the following:

- Principal processing equipment elements of the UFP system need to be included in the demonstration (including UFP-VSL-00001A/B, UFP-VSL-00002, the ultrafiltration loop recirculation pumps, and the ultrafilters with supporting equipment).
- The demonstration system needs to be flexible to accommodate testing of the baseline processes and process options for a range of operating conditions and processing times.
- Geometric similarity (prototypic) is needed in the key pieces of processing equipment (including UFP-VSL-00001A/B and UFP-VSL-00002).
- Ultrafilter elements must be a prototypic length and diameter to obtain expected filter performance data. The test equipment should be scaled down by using fewer filter elements in each assembly.

The following system parameters were developed and approved by the EFRT consultants:

- The engineering-scale test facility should be a factor of 4.5 smaller than the full-scale PTF.
- Each ultrafilter assembly should contain 12 full-scale elements.

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Five ultrafilter assemblies should be provided (based on M-13 recommendations).

Additional bases for the engineering-scale test system factor of 4.5 are provided in *Scaling Relationships for the Pretreatment Engineering Platform* (Kuhn et al. 2008). Some of the scaling factors are listed below.

- Selected processes within the PEP will be operated at both scale-time (4.5 times shorter than plant time) and plant time in separate test runs to address scaling issues.
- Prototypic tanks will be dimensionally scaled with a radius $\frac{1}{4}$. times their full-scale radius and operating height ¹/4.5 that of full-scale. The tank volume will be ¹/(4.5)³ or \sim ¹/90th of the plant.
- The filtration design is scaled by filter area. The total filtration area is $\frac{1}{(4.5)^2}$ or $\sim \frac{1}{20^{th}}$ that of the plant.

⁽a) S Barnes and R Voke. 2006. *Issue Response Plan for Implementation of External Flowsheet Review Team (EFRT) Recommendations—M12, Undemonstrated Leaching Processes*. 24590-WTP-PL-ENG-06-0024 Rev 0, Bechtel National, Inc., Richland, Washington.

- Fluid velocities in pipes are kept approximately the same (using commercially available pipe diameters) to mimic solids settling that may occur in plant piping.
- Chemical reaction times are not scaled.
- The pulse-jet mixer (PJM) nozzle velocity in the PEP will be scaled to provide the same mixing power per volume of tank contents. The plant targets a nozzle discharge velocity of 8 m/s in UFP-VSL-00001A/B and 12 m/s in UFP-VSL-00002A/B. For non-Newtonian fluids, the nozzle velocity will be the same in the PEP and WTP. For Newtonian fluids, the velocity is scaled by $1/(4.5)^{0.333}$. The total PJM cycle time will be scaled to maintain the fraction of the cycle in active drive mode. It is recognized that there is not a sharp distinction from Newtonian to non-Newtonian behavior. For the purpose of Phase 1 testing, we will scale the PJM velocities to maintain equal power per volume for the initial concentration and caustic leach. After the slurry is concentrated following the caustic leach, the PJMs will be adjusted to have the same PJM nozzle velocities as the plant (non-Newtonian scaling) and kept in that regime for the remainder of the process (post-caustic-leach wash, slurry wash, oxidative leaching, final slurry washing, and final concentration).

Section 6.4 of *Pretreatment Engineering Platform (PEP) Testing (Phase 1)* Test Specification $(24590-PTF-TSP-RT-07-001$ Rev 2) identifies the requirements for engineering-scale tests.^(a) The PEP data will be used by WTP along with laboratory-scale data and appropriate application of scaling to improve the WTP plant models used to predict plant operating performance.

1.3 Objective

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The objective of this report is to present the results of Integrated Test A with only very limited data analysis. This includes a test narrative, sample analyses, summaries of PEP monitoring data, evaluations or summaries of problems encountered, and deviations from the test protocols. Specific test objectives and summary results are provided in the summary section of this report.

⁽a) JL Huckaby and JR Markillie. 2008. *Pretreatment Engineering Platform (PEP) Testing (Phase I).* 24590-PTF-TSP-RT-07-001, Rev 2, Bechtel National, Inc., Richland, Washington.

2.0 Quality Assurance

The PNNL QA Program is based upon the requirements as defined in the U.S. Department of Energy (DOE) Order 414.1C, *Quality Assurance* and 10 CFR 830, *Energy/Nuclear Safety Management*, Subpart A—*Quality Assurance Requirements* (a.k.a. the Quality Rule). PNNL has chosen to implement the following consensus standards in a graded approach:

- ASME NQA-1-2000: Quality Assurance Requirements for Nuclear Facility Applications, Part 1, *Requirements for Quality Assurance Programs for Nuclear Facilities*
- ASME NQA-1-2000, Part II, Subpart 2.7: Quality Assurance Requirements for Computer Software for Nuclear Facility Applications
- ASME NQA-1-2000, Part IV, Subpart 4.2: Graded Approach Application of Quality Assurance Requirements for Research and Development.

The procedures necessary to implement the requirements are documented in PNNL's "How Do I…?" (HDI) ^(a)

The RPP-WTP quality requirements are implemented 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),* as applicable. These quality requirements are implemented through the *River Protection Project—Waste Treatment Plant Support Program (RPP-WTP) Quality Assurance Manual* (RPP-WTP-QA-003, QAM). The requirements of DOE/RW-0333P Rev 13, *Quality Assurance Requirements and Descriptions (QARD),* and 10 CFR 830 Subpart A were not required for this work.

The RPP-WTP addresses internal verification and validation activities by conducting an independent technical review of the final data report in accordance with RPP-WTP's procedure QA-RPP-WTP-604. This review procedure is part of PNNL's *RPP-WTP Quality Assurance Manual* (RPP-WTP-QA-003). Following this procedure, a technical review would verify that the reported results are traceable, that inferences and conclusions are soundly based, and the reported work satisfies the objectives.

Table 2.1 lists nonconformance reports (NCRs) active during Integrated Test A.

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⁽a) The system for managing the delivery of laboratory-level policies, requirements, and procedures.

Nonconformance			
Report Number	Description		
NCR 38767.1	Measurement: UFP-VSL-T02A spargers air flow rate.		
	Issue: Micro-Motion identifies flow rates below which the uncertainty is greater than 0.5% . In the case of the PEP, air flow rates below 0.090 kg/min have uncertainties greater than 0.5% . For the lowest flow rate reported (0.012 kg/min) on FT-1977), the estimated uncertainty is \sim 4%.		
	Affected instruments follow: FT-1973 (UFP-VSL-T01B steam ring purge), FT-1977 (UFP-VSL-T02A bottom sparger), FT-1981 (UFP-VSL-T01A steam ring purge), FT-1901 (UFP-VSL-T02A upper sparger), and FT-1995 (UFP-VSL-T02A steam ring purge).		
	Consequence: There is greater uncertainty associated with data for air flow rates < 0.090 kg/min.		
NCR 43398.1	Measurement: UFP-VSL-T01B temperature.		
	Issue and Consequence: TTK-0427 failed at installation. All data from TTK-0427 are invalid and unusable. However, there are 19 other temperature measurements available within vessel UFP-VSL-T01B, with TTK-0425 as the prototypic measurement.		
NCR 42402.1	Measurement: Slurry temperature in the filter loop.		
	Issues and Consequences: RTD thermowells in the filter loop do not extend into the process stream: TT-0513: UFP-HX-T02A; (cooling) slurry outlet temperature. No data are to be used for quality-affecting work. Data may be used for qualitative purposes only.		
	TT-0515: UFP-HX-T03A; (heating) slurry outlet temperature. No data are to be used for quality-affecting work. Data may be used for qualitative purposes only.		
	TT-0537: Filter UFP-FILT-T05A; outlet temperature (UFP-HX-T02A inlet). No data are to be used for quality-affecting work. Data may be used for qualitative purposes only.		
	TT-0791: Filter UFP-FILT-T01A inlet temperature. Data from this should not be used at all. Tank temperature data will be used for quality-affecting filter processing data.		
NCR 42317.1	Measurement: Filter-loop flow rate downstream from UFP-PMP-T43A.		
	Issue and Consequence: FE-0635 is reporting suspect flow values. Data from FE-0635 are unusable from 2/14/2009 through 2/27/2009.		
NCR 41589.1	Measurement: UFP-VSL-T01B Coriolis sample station on-line density.		
	Issue: DT-2101 failed.		
	Consequence: All readings after 12/23/2009 are not valid.		

Table 2.1. Description of NCRs During Integrated Test A

3.0 Experimental Methods and Analysis

The sections below describe PEP operations, testing overview, and the simulant and sample processing used for Integrated Test A.

3.1 Pretreatment Engineering Platform Description

The PEP test system is designed to perform engineering-scale demonstrations of most WTP pretreatment processes. These include (but are not limited to) vessel-to-vessel transfers, waste pumping, cross-flow filtration, filter cleaning, waste solids washing, chemical reagent addition and mixing, waste slurry heating and cooling, and waste chemical leaching. Refer to Sections 3.1.2 and 3.1.3 for a description of filter pulse pot and PJM operations, respectively.

The PEP is composed of prototypic and nonprototypic equipment as well as auxiliary bulk chemical and utility systems. Slurry is received at feed receipt vessels FRP-VSL-T01, FEP-VSL-T01, and HLP-VSL-T22. For Integrated Test A, only simulant in HLP-VSL-T22 was used. Simulant is then transferred to the upfront ultrafeed vessels UFP-VSL-T01A and B. For Integrated Test A, caustic leaching was done in the upfront vessels, and then it was transferred to UFP-VSL-T02A for post-caustic-leach concentration using the recirculation loop filter(s). The filters purchased for the PEP were obtained from the Mott Corporation (Farmington, CT) using the same specifications for the filters being purchased for the WTP-PTF. The filters are constructed of porous sintered 316 stainless steel with an effective filtration rating of $0.1 \mu m$. The PEP test system employs a combination of 8-ft-long and 10-ft-long filter elements (which were formed by welding either four or five 2-ft filter elements together). As such, the PEP elements have the same radial dimensions and filtration ratings as the 2-ft elements, but have a longer filtration length of either 96 in. or 120 in.

Cooling temperature control is done by cooling heat exchangers UFP-HX-T05A and B for UFP-VSL-T01A and B, respectively, and UFP-HX-T02A or the cooling jacket for UFP-VSL-T02A. For high-temperature processing, direct steam injection and heat exchangers UFP-HX-T04A and B are used for UFP-VSL-T01A and B, respectively, while direct steam injection or UFP-HX-T03A is used for UFP-VSL-T02A. Simulant can also be concentrated before it is caustic leached in UFP-VSL-T02A (Integrated Tests B and D). During concentration, permeate is sent to permeate vessels UFP-VSL-T62A and/or B. Once simulant is in UFP-VSL-T02A, further processing, such as oxidative leaching and washing (before and after oxidative leaching), followed by a final concentration, occurs. After processing is complete, the high-solids material is sent to waste storage vessel HLP-VSL-T27. For Integrated Test A, however, the high-solids material was not sent to HLP-VSL-T27. Refer to Figure 3.1 for an overview of the PEP processes and components.

Figure 3.1. PEP Simplified Process Overview

Equipment that was considered critical to evaluate the integrated system performance was scaled to be prototypic in the PEP. Vessels were scaled to be geometrically similar with a $\frac{1}{4.5}$ scale; the working height and diameter were scaled $\frac{1}{4}$.s. Pipe sizes are scaled to have approximately $\frac{1}{4}$.s the diameter, but the fluid velocity was approximately the same as the full-scale plant because solids settling does not scale. The pipe diameters in the scaled system were sometimes further reduced if the Reynolds number for fluid transfers in the scaled system was projected to drop below 2100. The PEP also contains equipment to provide feed, accept processed products, and provide services to support operations. The additional equipment was not designed to be prototypic of corresponding tanks, pumps, and services in the full-scale WTP. Refer to the Test Plan for the complete listing of prototypic equipment.^(a)

Two types of instrumentation were used on the PEP, instruments for monitoring and controlling the process and instruments for collecting quality-affecting data (Nuclear Quality Assurance [NQA]-1 instruments). The instrumentation and control for the test system were functionally prototypic to those specified for the plant with the exception that the PJMs and pulse pots had additional data capabilities to meet the PEP functional requirements.^(b) Refer to Appendix A for a summary of pertinent process instruments.

Process control strategies and control ranges were as specified in 24590-WTP-RPT-PET-07-002, Rev 1, *Pretreatment Engineering Platform (PEP) Phase I Testing Process Description*.^(c) The temperature bands for caustic leaching and oxidative leaching are given in the Test Specification, *Pretreatment Engineering Platform (PEP) Testing (Phase I draft)*, 24590-PTF-TSP-RT-07-001 Rev 2.

For detailed information on the PEP components and operations, refer to Pretreatment Engineering Platform (PEP) Phase I Testing Process Description, 24590-WTP-RPT-PET-07-002, Rev $1^(c)$ and Functional Requirements for the Pretreatment Engineering Platform (PEP), 24590-PTF-3YD-UFP-00002, Rev $1^{(b)}$

3.1.1 PEP Filtration System

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The PEP filtration system is composed of an ultrafiltration feed tank (UFP-VSL-T02A; henceforth also referred to as Tank T02A), a slurry circulation and filtration loop, a permeate metering and collection system, and a filter backpulse and cleaning system. The PEP filtration system is instrumented to measure the feed flow rate, temperature at four locations, and axial and transmembrane pressure (TMP) drop across each filter bundle. In addition, the system is configurable such that filter bundles 1 through 5 may be connected in series to the slurry circulation loop or bypassed such that flow is directed through filter bundle 1 or through filter bundles 2 through 5. A summary of process instrumentation is provided in Appendix A. In the following paragraphs, key process equipment for slurry filtration operations is identified and discussed. Interested readers are referred to the documents listed in Section 3.1 if more information is needed.

⁽a) G Josephson, O Bredt, J Young, and D Kurath. 2008. *Test Plan: Pretreatment Engineering Platform (PEP) Testing (Phase I).* TP-RPP-WTP-506, Rev. 0, Pacific Northwest National Laboratory, Richland, WA, 99352.

⁽b) B Stiver. 2007. *Functional Requirements for Pretreatment Engineering Platform (PEP)*, 24590-PTF-3YD-UFP-00002 Rev. 1, Bechtel National Incorporated, Richland, Washington.

⁽c) SD Lehrman SD. 2008. *Pretreatment Engineering Platform (PEP) Phase I Testing Process Description*. 24590-WTP-RPT-PET-07-002, Rev. 1, Bechtel National Incorporated, Richland, Washington.

3.1.1.1 Ultrafiltration Feed Tank

Tank T02A serves as a primary supply and mixing reservoir for slurry being circulated through the filtration loop. The contents of this tank are mixed with an array of six PJMs. Ancillary systems for Tank T02A include air spargers to limit flammable gas hold-up in the actual waste treatment system, bubblers to measure slurry density and level, laser-level sensors, and an array of resistance temperature detectors (RTDs) to measure the tank temperature profile. (Specific locations of the RTD arrays for tanks UFP-VSL-T02A and UFP-VSL-T01A are available in laboratory record book [LRB] 59944, pages 12-17.) Tank T02A is equipped with a water jacket supplied with chilled water to cool the contained slurry.

3.1.1.2 Slurry Filtration Circulation Loop

The filtration loop contains process equipment that is key to slurry dewatering and washing operations. It is composed of two slurry pumps, a series of five filter bundles, and two heat exchangers.

Two centrifugal slurry pumps, UFP-PMP-T42A and UFP-PMP-T43A (hereafter also referred to as Pumps T42A and T43A, respectively), are operated in series to provide the required slurry flow rate and pressure for the cross-flow filter bundles. The suction to Pump T42A is fed from Tank T02A. In addition, the feed to Pump T42A is connected to process inhibited water $(IW)^{(a)}$ supplies used for slurry washing and dilution operations. The discharge from Pump T42A feeds Pump T43A. Slurry discharge from Pump T43A can be fed through, or bypassed around, the cross-flow filter banks. Pumps T42A and T43A provide a combined filtration-loop flow rate and pressure of up to 150 gpm and 250 psig.

The cross-flow filter system is the core of slurry liquid-solid separations. It is composed of five filter bundles connected in series. These filter bundles are designated as UFP-FILT-T01A to -T05A (hereafter also referred to as Filters T01A through T05A). The filter loop is equipped with slurry bypass valves to allow slurry to flow through Filter Bundle 1 and/or Filters T02A through T05A.

The filters purchased for the PEP were obtained from the Mott Corporation (Farmington, CT) using the same specifications for the filters being purchased for the PTF. The filters are constructed of porous sintered 316 stainless steel with an effective filtration rating of 0.1 μ m. The PEP test system employs a combination of 8-ft-long and 10-ft-long filter elements (which were formed by welding either four or five 2-ft filter elements together). As such, the PEP elements have the same radial dimensions and filtration ratings as the 2-ft elements, but have a longer filtration length of either 96 in. or 120 in. A summary of the geometries of the five filter bundles is provided in Table 3.1.

The tubeside slurry flow rate and pressure are monitored by a series of flow meters and pressure transducers. Slurry flow to Pump T42A is measured by magnetic flow meter FT-0623. Slurry discharge flow from Pump T43A is measured by a second magnetic flow meter (FT-0635). Circulation loop pressure is monitored by a series of pressure transducers located at the entrance to each slurry pump, filter bundle, and heat exchanger.

The temperature in both Tank T02A and the slurry filtration loop is monitored with a combination of two in-line heat exchangers. The first heat exchanger, UFP-HX-T02A, is a spiral plate heat exchanger

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⁽a) Inhibited water typically refers to a 0.01-M solution of NaOH.

that uses chilled water to cool the circulating slurry. Heat exchanger UFP-HX-T02A controls the temperature of Tank T02A and the filtration loop. The second heat exchanger, UFP-HX-T03A, is a steam exchanger intended to heat the circulating slurry if needed. Both heat exchangers are equipped with a bypass loop so that they can be isolated from slurry flow. RTDs installed in thermowells monitor and control the performance of the heat exchanger.^(a) For the current testing, UFP-HX-T03A was not used and was bypassed. The final process element in the slurry circulation loop is a pressure control valve (SV-0609) that can be adjusted in combination with the slurry pumps to provide adequate backpressure for permeate production. After passing through SV-0609, the dewatered circulating slurry is recycled back into Tank T02A.

		Number of	Element Inside		Bundle
		Elements in	Diameter	Element	Surface Area
Filter $#$	Filter ID	Bundle	(inches)	Length (ft)	$(f t^2)$
	UFP-FILT-T01A	12	0.5	10	15.7
$\overline{2}$	UFP-FILT-T02A	12	0.5	10	15.7
3	UFP-FILT-T03A	12	0.5	10	15.7
$\overline{4}$	UFP-FILT-T04A	12	0.5	8	12.6
5	UFP-FILT-T05A	12	0.5	8	12.6
Total	--	--			72.3

Table 3.1. Specifications of the Five PEP Cross-Flow Filtration Bundles

3.1.1.3 Permeate Metering and Collection Systems/Filtration Backpulse Systems

The permeate metering and collection systems consist of Coriolis mass flow meters for monitoring permeate production rates and permeate collection tanks, and three pulse pots are connected to high-pressure air supplies for backpulsing the filter bundles.

Permeate (shell-side) mass production rates from Filters T01A through T05A are monitored by Coriolis flow meters. Permeate flow from each of the filter bundles is directed to three pulse pots (designated as UFP-PP-T01A to UFP-PP-T03A). Pulse pot UFP-PP-T03A serves filter bundle 1, pulse pot UFP-PP-T02A serves filter bundles 2 and 4, and pulse pot UFP-PP-T01A serves filter bundles 3 and 5. The pulse pots are filled with a sufficient volume of collected permeate to backpulse the filter bundles. Overflow from the pulse pots may be directed to 1) permeate or process slurry collection tanks (UFP-VSL-T62A and -T62B) during slurry dewatering operations, or 2) a return line to Tank T02A during continuous recycle filtration operations. Table 3.2 summarizes the permeate metering and pulse pot systems.

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⁽a) The RTD measuring the slurry outlet temperature (TT-0513) did not extend into the process stream, and data were to be used for qualitative purposes only. See discussion in Section 2 for a description of this nonconformance (NCR 42402.1).

	Permeate	Associated Pulse
Filter Bundle No./ID	Coriolis Meter	Pot
$1 - UFP-FILT-T01A$	FT-0720	UFP-PP-T03A
$2 - UFP-FILT-T02A$	FT-0755	UFP-PP-T02A
$3 - UFP-FILT-T03A$	FT-0765	$UFP-PP-T01A$
$4 - UFP-FILT-T04A$	FT-0775	$UFP-PP-T02A$
$5 - UFP-FILT-T05A$	FT-0785	$UFP-PP-T01A$

Table 3.2. Permeate Metering and Pulse Pot Configurations for PEP

During backpulsing, one of the pulse pots is isolated and charged with high-pressure air until the pulse pot pressure exceeds the filter bundle inlet pressure to be backpulsed by a given amount (typically 40 psid). After the target pulse pot pressure is reached, the valve isolating the pulse pot from the filter is opened, and the permeate collected in the pulse pot flows back through the filter element. The backflow of permeate forces any particles loose that are weakly entrained in the filter pores or that have caked on the filter surface. A description of the backpulsing process, as it was implemented in the PEP, is provided in the next section.

3.1.1.4 Backpulse Operations

The permeate metering and collection systems consist of Coriolis mass flow meters for monitoring permeate.

Backpulsing was employed to restore filter fluxes during solids concentration in the UFP-VSL-T02A recirculation loop. The backpulsing function of the filter loop can be operated only when actively filtering UFP-VSL-T02A contents. There are three variables that can be set by the operator: Level Drain Set Point, Backpulse Pressure Set Point, and Pressure Deadband for Completion.

The Level Drain Set Point is the height of the fluid in the pulse pot used for the backpulse. The Backpulse Pressure Set Point is the amount above the filter inlet pressure that the pulse pot should be charged to (i.e., if the inlet pressure is 100 psig and the Backpulse Pressure Set Point is set to 40 psig, the control system will charge the pulse pot to 140 psig).

The Pressure Deadband for Completion is the amount above the filter inlet pressure that will cause the backpulse to be marked as finished (i.e., if the inlet pressure is 100 psig and the Deadband is set to 10 psig, the backpulse will complete when the pulse pot pressure indicator gets down to 110 psig).

During filtering, the operator presses the "Start Backpulse" button in the human-machine interface (HMI), which initiates the backpulse cycle. The first step is to close all valves entering and leaving the pulse pot. Next, the high-pressure air line is opened, and the pulse pot is pressurized to 50 psig. The high-pressure air valve is closed, and the drain valve to UFP-VSL-T62A/B is opened. The pulse pot fluid level falls until reaching the Level Drain Set Point when the drain valve is closed. The high-pressure air valve is opened again and pressurizes the pulse pot to the sum of the filter inlet pressure plus the Backpulse Pressure Set Point (100 psig + 40 psig = 140 psig in the above example). The air valve is closed, and the backpulse cycle pauses for 15 seconds. The fast-acting valve then opens, and the pressure in the pulse pot pushes fluid back through the filter until the pressure in the pulse pot is equal to the filter inlet pressure plus the Deadband (100 psig + 10 psig = 110 psig in the above example). The final step is

to return to filtering conditions. The fast-acting valve closes, and the filter outlet valve and pulse pot outlet valve to UFP-VSL-T62A/B (not the drain valve) are opened.

3.1.2 Data Acquisition System

The Data Acquisition System (DAS) for PEP is composed of four data acquisition servers and one graphical user interface (GUI) personal computer. They are networked together, along with the rest of the PEP equipment as depicted in Figure 3.2.

Figure 3.2. PEP Equipment

The GUI personal computer resides in the control room and is used to start and stop the data acquisition servers and set the recording rate. Each server that is located out in the high bay is independent of the others while collecting data. A global positioning system (GPS) receiver is used to provide a time stamp to each DAS server.

3.1.2.1 Background

The PEP data acquisition and control system was designed to be very robust and flexible in operation. While the same sensors are used for controlling the PEP and recording data, the sensor outputs are split into two loops to make certain that the DAS is completely separate from the control system. This

requirement was put into the system to allow changes to the control system of the PEP to be performed without the rigorous verification and validation process that is required of any quality-affecting software program. This allowed many changes to the control system to be made during operation of the PEP system without affecting the quality of the data. The DAS and all related software did undergo PNNL software control procedures to make sure that the data meet quality standards.

From each signal splitter, one loop went to the control system, and the other went to the DAS. Four- to 20-ma outputs from all sensors were selected to make sure that the system was reliable and prevent long cable runs from introducing error in the signals. The data acquisition servers are manufactured by Microstar Laboratories, located in Bellevue, WA. Each server is capable of storing a large number of channels at a high sampling rate. For PEP, they have been set to a maximum sampling rate of 100 Hz. Each server stores its information in a file local to that server. After data were collected, they were transferred to the raw data server and to the database server for analysis.

3.1.2.2 Data Retrieval

Over a terabyte of data were recorded by the four data acquisition servers during operations of the PEP during shakedown and Phase 1 testing. Those data are stored in binary files that are not human readable. Software was developed to provide analysts with an easy tool to retrieve the data they required by specifying the date/time, instruments, and sampling rate. This tool is composed of three main components: the data ingest server, the database itself, and the data retrieval tool. The data ingest server watches a specified directory on the database server, and when new files are put there, it uploads the data from that location into the database and transfers those data files into a directory where processed files are stored. The database itself uses Microsoft's Structured Query Language (SQL) server as a basis for the large database that stores all data and sensor information for the PEP. Finally, the data retrieval tool is a web-based application that allows staff to recover the data they need by specifying the time/date, instruments, and sampling rate necessary. The software produces a text file that can easily be imported into Excel or any other analysis package. This method provides a data file that meets quality standards for the PEP project.

Another method for retrieving data was commonly referred to the DAS widget. This tool is a simple application that allows users with detailed knowledge of the system to retrieve data if they do not have access to the web-based tool or if they need access to the data before the data have been imported into the database. This tool did not go through the software control procedure and does not provide data that meets quality standards for PEP. It will read a file from one server and produce a file with all the raw (ma) values from every instrument on that server. With that data and the information in the measurement and test equipment (M&TE) list, staff can retrieve the data from all sensors in engineering units. It is more time consuming to retrieve data in this manner, but it allows data to be read in near real-time. This method was commonly used to provide near real-time data for PJM tuning.

3.1.2.3 Data Storage

All data recorded during operation of the PEP were first copied to the raw data server in PDL-W and then copied to the database server in the Laboratory Support Building (LSB). This procedure is detailed in the data-management plan and provides redundant storage of all data to confirm that data are not lost.

3.1.3 Pulse-Jet Mixing Operations

The leaching vessels UFP-VSL-T02A and UFP-VSL-T01A and B were equipped with PJMs and several different operating programs to allow plant prototypic mixing, low-level operation, and additional data collection.

PJM mixing technology involves a pulse tube coupled with a jet nozzle. The nozzle end of the tube is immersed in the tank while periodic pressure, vacuum, and venting are supplied to the opposite end. Changing the applied pressure produces four operating phases for the pulse tube: 1) the drive mode, when pressure is applied, and the PJM tube discharges its contents at high velocity through the nozzle; 2) the vent mode, when the pressure is vented to the atmosphere, and the level inside the pulse tube and tank approach the same fill level; 3) the refill mode, when vacuum is applied to refill the pulse tube; and 4) the equilibration mode, when the pressure is vented to the atmosphere, and the pulse tube and tank approach the same fill level. The PJM system uses these operating phases in sequence to mix contents in the vessel.

PJM operations at PEP consisted of one of several control modes: *Standard Mode* (Regular), *Standard Mode Short Cycle Mode, Standard Mode* (*Star Pattern*—UFP-VSL-T02A only), and *Simple Mode*. However, PJM controls at PEP were not prototypic. Integrated Test A PJM operations used the control modes *Standard Mode* (Regular) and *Simple Mode*. PJM operating control modes used in PEP had the same basic cycle: *Drive Phase*, *Vent Phase*, and *Vacuum Phase*, followed by a *Hold* (or *Vent*) *Phase*. A timer set to the overall cycle time started at the beginning of each *Drive Phase*. A new *Drive Phase* began when either the cycle timer ran out, or the cycle entered the final *Hold*/*Vent Phase*, whichever was later. This condition existed to make sure the *Vacuum Phase* had completed before moving on to the next cycle.

The main difference between the *Standard Mode* and *Simple Mode* was the way each phase was handled. In *Standard Mode* operation, the *Drive* and *Vent* phases were time based while the *Vacuum Phase* was level based. The final *Hold*/*Vent Phase* filled the rest of the Cycle Time. In *Simple Mode*, the *Vacuum Phase* was also time-based.

Standard Mode (Regular) Description

Standard Mode operations in PEP controlled each PJM individually based on that PJM level instrument within one overall cycle time. Each cycle was controlled by five main variables: Cycle Time, Drive Time Set Point, Vent Time Set Point, L1 Level, and L2 Level. Additionally, each PJM could be enabled or disabled individually.

The entered Cycle Time controlled the peak-to-peak distance between cycles. However, if the value of the entered cycle time set point was shorter than the combined entered times for the PJM phases, the *actual* PJM cycle time would be the time it took the PJMs to complete the first three phases (*Drive*, *Vent*, and *Vacuum*) and the fourth phase (*Hold*) would be skipped.

The *Drive Phase* was controlled by the time entered as the Drive Time Set Point (corrected for temperature if enabled). Each PJM began the *Drive Phase* at the same instant but could have different lengths of time specified for driving each PJM. If any PJM did not finish within 30 seconds of the PJM with the shortest drive time, that PJM would be disabled (until it was re-enabled by the operator), and the cycle would continue to the next phase.

The *Vent Phase* was controlled by the Vent Time Set Point, which had no correction factors. Each PJM began the *Vent Phase* at the same instant, but could have different lengths of time specified for each PJM. If any PJM did not finish within 30 seconds of the PJM with the shortest vent time, that PJM would be disabled (until it was re-enabled by the operator), and the cycle would continue to the next phase. Generally, each Vent Time Set Point was set to a small number such as 500 milliseconds to smooth out the transition between the *Drive* and *Vacuum Phases*.

The *Vacuum Phase* was controlled by the L1 Level, which was set at the top of the desired PJM stroke. All PJMs began with the *Vacuum Phase* at the same instant, but each PJM L1 Level was set separately. If any PJM did not reach the L1 Level within 30 seconds of the first PJM to reach its L1 Level, that PJM was disabled (until it was re-enabled by the operator), and the cycle continued to the next phase. When tank temperatures were greater than 60° C, and the tank level was above the PJM head, the *Vacuum Phase* was disabled, and each PJM vented to atmosphere until it reached the L1 Level set point. If the tank level was below the L1 Level, then the PJMs entered a control *Short Cycle Mode*. The *Standard Mode* in PEP also contained a *Short Cycle Mode* used during gravity refill operation when the tank level did not exceed the PJM head and during extremely low tank levels (to avoid overblowing). A Short Cycle in PEP consisted of a *Drive Phase* in which each enabled PJM drives for 20% of the Drive Time Set Point followed by a 10-second *Vent Phase*.

The final phase in the control *Standard Mode* was a *Hold Phase*. All valves to the PJM were closed, causing the fluid level in each PJM to remain constant at the L1 Level.

Standard Mode operation in PEP also contained a temperature linearization variable that reduced the drive time to prevent overblows as the temperature increased—causing fluid viscosity changes. The variable was expressed as a slope, the percent of drive time decrease per degrees Celsius increase from 0° C. This function was largely untested, but may have been enabled at some nominal slope during operations.

To prevent reaching the pump heel level in tank UFP-VSL-T02A in PEP, while still operating PJMs prototypically, UFP-VSL-T02A additionally could operate in *Standard Mode* (*Star Pattern*). The *Star Pattern* option followed the same rules as the regular PJM *Standard Mode*. The difference was that only two PJMs were enabled at a given time. When the operator set the *Star Pattern* button, the HMI, the center PJM, and one other PJM would follow through a set number of cycles. Then the outer PJM would be disabled, and a different outer PJM would be enabled for a set number of cycles. The pattern of enabling and disabling the five outer PJMs formed a five-point star.

Simple Mode Description:

As mentioned above, *Simple Mode* operated substantially the same as *Standard Mode*. Each cycle consisted of *Drive*, *Vent*, *Vacuum*, and *Hold*/*Vent Phases*. There were two main differences. The first difference was that all PJMs operated together as one unit. There were no individual levels used or individually enabling or disabling PJMs. The second difference was that each phase was controlled by a time. An operator set the Cycle Time, Drive Time Set Point (uncorrected), Vent Time Set Point, and Vacuum Time Set Point (uncorrected). Additionally, the operator could choose whether the last phase

holds the PJM level or *vents* (returning the PJM level to the same as the tank level). There was an "uncorrected" target phase time entered by the operator, followed by a "corrected" time HMI display at the completion of a phase, indicating the actual time it took to complete the phase.

The Drive time had additional correction factors for temperature (same as in *Standard Mode*) and tank level. The tank level correction (if enabled) reduced the drive time according to the user-entered slope (% drive time/inch) for every inch below the top of the PJM head (46 inches by default). For example, if the Drive Time Set Point was 10,000 milliseconds, the Drive Slope was 1%/inch, and the tank level was at 36 inches, then the corrected drive time would be:

(10,000 milliseconds) * {1.00 – [0.01/inch * (46 - 36 inches)]} = 9000 milliseconds

The tank level used in the calculation was determined by the operator using either the tank bubbler, laser, or Drexelbrook probe.

The Vacuum time also contained a correction factor for tank level, but not for temperature. As the tank level decreased, the PJM fill level would decrease during the *Vacuum Phase;* therefore, a vacuum correction set point was implemented. The vacuum correction (if enabled) increased the vacuum time according to a user-entered vacuum slope (% vacuum time/inch) for every inch the tank level was below 46 inches. For example, if the Vacuum Time Set Point was 10,000 milliseconds, the vacuum slope was 1%/inch, and the tank level was at 36 inches, then the corrected drive time would be:

 $(10,000 \text{ milliseconds})$ * $\{1.00 + [0.01/inch * (46 - 36 inches)]\}$ = 11,000 milliseconds

The operator selectable *Hold Phase* was created to give the option to more closely resemble PJM *Standard Mode* operation. The *Hold Phase* prevented the PJM level from dropping after completing the *Vacuum Phase*. However, since PJM phase set points were time based and there was no level checking in the PJMs in *Simple Mode*, the PJM fill level could increase over successive cycles when using the *Hold Phase*—whether or not the drive or the vacuum time set points were decreased or increased, respectively. The successive increase in PJM level over time could result in fluid traveling all the way up into the hoses on top of the tank. If the *Hold Phase* was disabled, each PJM just vented to the tank level during the remainder of the PJM cycle.

3.2 Testing Overview

The following is a summary level overview of testing reported herein. Refer to the Test Instruction TI-WTP-PEP-065, hereafter referred to as TI, and the LRBs (60230, 60235, 60269, 60108, 60229, 60271, and 60279) for additional details.

- 1. Transferred simulant from HLP-VSL-T22 to UFP-VSL-T01A (Batch 1) and added NaOH in-line.
- 2. Performed UFP-VSL-T01A leaching at 98°C for 16 hours and cooling using UFP-HX-T05A.
- 3. Completed initial transfer to UFP-VSL-T02A from UFP-VSL-T01A through UFP-HX-T05A.
- 4. Performed post-caustic-leach concentration from UFP-VSL-T01A contents (Batch 1) using UFP-FILT-T01A in UFP-VSL-T02A recirculation loop.
	- a. Filters were drained before solids concentration.
- 5. Transferred simulant from HLP-VSL-T22 to UFP-VSL-T01B (Batch 2) and added NaOH in-line (80%) and to the top of UFP-VSL-T01B (20%) .
- 6. Completed UFP-VSL-T01B leaching at 98C for 16 hours and cooling using UFP-HX-T05B.
- 7. Performed post-caustic-leach concentration from UFP-VSL-T01B contents (Batch 2) using UFP-FILT-T01A in UFP-VSL-T02A recirculation loop.
- 8. Repeated HLP-VSL-T22 transfers to UFP-VSL-T01A/B, leaching and cooling in UFP-VSL-T01A/B, and post-caustic-leach concentration in UFP-VSL-T02A for a combined total of six batches (three UFP-VSL-T01A and three UFP-VSL-T01B batches).
	- a. Conducted backpulsing of UFP-FILT-T01A to enhance the filtration rate.
- 9. Evaluated bypass of the filter loop using a CsBr tracer.
- 10. Performed post-caustic-leach slurry wash using all five filters, UFP-FILT-T01A through -T05A.
- 11. Completed oxidative leaching in UFP-VSL-T02A at 25°C for 6 hours.
- 12. Washed and reconcentrated slurry in UFP-VSL-T02A using all five filters, UFP-FILT-T01A through -T05A.
- 13. Integrated Test A was put on "hold" due to UFP-VSL-T02A slurry air entrainment issues.
- 14. Demonstrated final solids concentration in UFP-VSL-T02A using all five filters, UFP-FILT-T01A through -T05A.
- 15. Completed the High-Solids Filter Test after completing Integrated Test B.
- 16. Repeated the CsBr tracer test after completing the High-Solids Filter Test.

3.2.1 Test Preparation

Several maintenance activities and changes were made to instrumentation and PEP systems before Integrated Test A. These modifications are summarized below. NOTE: The items below are not necessarily in chronological order:

• Pressure-relief modification work on the UFP-VSL-T02A recirculation loop was completed and approved by WTP on 1/29/09. The modification work routed the vents from heat-exchanger valves PSV-0515 (UFP-HX-T02A) and PSV-0519 (UFP-HX-T03A) to vessel HLP-VSL-T27. The filter-loop modification also installed a new rupture disk at the discharge of the UFP-PMP-T43A pump, which also relieves to vessel HLP-VSL-T27. Two configuration control locks were added to HLP-VSL-T27 (from IW and UFP-VSL-T02A drain) to make sure that adequate capacity was available in the HLP-VSL-T27 vessel if the new rupture disk downstream of pump UFP-PMP-T43A failed. A new hardware interlock was also added to shut down the UFP-PMP-T42A and UFP-PMP-T43A pumps if the level in the HLP-VSL-T27 vessel increases above the new level probe. This hardware interlock made certain that HLP-VSL-T27 would not overfill if the rupture disk failed. Vessel HLP-VSL-T27 will no longer receive solids from UFP-VSL-T02A and will not be used for receipt of any material.

A lock remained on V05161, the isolation valve between UFP-HX-T03A and the low-point drain, until the PEP Hazards Analysis was revised—the initial Hazards Analysis did not evaluate operation of UFP-PMP-T42A and UFP-PMP-T43A pumps with the filter loop isolated from UFP-VSL-T02A.

o Software changes associated with pressure-relief filter-loop modification included adding the LS 1090 interlock for filter-loop overflow to vessel HLP-VSL-T27. If the HLP-VSL-T27 tank is full, pumps UFP-PMP-T42A/T43A would not start, and an alarm would show on the Alarm Server.

- Following the pressure relief modification described above, the UFP-VSL-T02A recirculation filter-loop in-service leak check was performed.
- The UFP-VSL-T02A recirculation loop return nozzle was repositioned because it was positioned in a nonprototypic location, resulting in the returning slurry impinging on the steam ring. Modifications to the return nozzle involved bending the nozzle in two places to redirect flow to the side of the steam ring (Operations Manager email, 07/20/09, 09:29).
- The laser in UFP-VSL-T02A was repositioned since it gave erroneous readings in previous testing efforts, and an air purge was installed.
- Level instruments in all the PJMs, Drexelbrooks, were replaced. The new Drexelbrooks had a Teflon coating, which is resistant to caustic, and included an inactive zone on the admittance probes. The modification was supposed to provide better control of PJM operation, but the probes still did not function reliably during leach operations. After installation, the Drexelbrooks were respanned.
	- o Software changes associated with this installment involved modifying the span of the level instruments in the three UFP vessels.
- An additional tank level instrument (Drexelbrook) was installed in UFP-VSL-T01A to provide additional tank level detection in the event that the bubblers plug and/or the laser performance becomes unreliable.
	- o Software and HMI changes associated with this installation include adding LT_0311 for the Drexelbrook and modifying HS-0323 to allow switching between laser, bubbler, and Drexelbrook for UFP-VSL-T01A level controls.
- Coriolis densitometer (CD) sampler stations in UFP-VSL-T01B and UFP-VSL-T02A vessels were repaired, and then in-service leak checks were performed on all CD sampler stations in the three UFP vessels.
- The DAS graphical user interface software was modified with an alarm to alert operators when the DAS is not responding properly.
- Indicators to all pump start/stop screens showing which interlocks are tripped (red indicates an interlock is engaged) were added to the HMI, making it easier for the HMI operator to see when a pump turns off unexpectedly.
- The simple mode PJM code was updated to include a vacuum time setpoint, current vacuum time indicator, vacuum slope input field for tank level compensation for tank levels below 46 inches (this field is inactive when tank levels are above 46 inches), and replacing the vacuum phase with the vent phase when tank temperatures exceed 60° C.

The filters in the UFP-VSL-T02A recirculation loop were not cleaned before Integrated Test A.

3.2.2 General Operations

General operations through the course of Integrated Test A are listed as follows:

• Operation instructions, per Operations Manager, between 01/31/09 and 2/2/09 were to use the lasers in the UFP vessels for level and process controlling. Whenever a level measurement was questionable, such as the second or third in the case of UFP-VSL-T01A, another level instrument was used for comparison, and the most credible (knowing transfer volume or permeate mass removed coupled with the vessel volume vs. level correlation) was used.

- Stable level measurements were taken typically before and after transfers, concentrations, and washing to verify that the target tank level was met, or permeate mass was removed. Stable level measurements had recirculation pumps, spargers, and PJMs off and were then returned to the original configuration.
	- o The specific gravity was recorded any time a bubbler-level measurement was made to confirm level credibility since the bubblers plugged. When bubblers plugged, operators blew the lines with high pressure air $(\sim 20 \text{ psig})$ and/or used small amounts of IW $(\leq 200 \text{ -mL})$ followed by high-pressure air to clear the lines.
- FRP-VSL-T01 and FEP-VSL-T01 vessel bubblers were turned off and were only turned on temporarily for completing shift rounds.
- Agitators were kept on for vessels containing simulant, the receipt vessels (FRP-VSL-T01, HLP-VSL-T22, and FEP-VSL-T01), and permeate vessels UFP-VSL-T62A and/or B as determined by the Operations Manager.
- The temperature setpoint on UFP-HX-T02A was adjusted as necessary to maintain the target run sheet temperature in the filter loop and then set to automatic mode.
- CD sampler stations were kept ON only when sampling was taking place, and then were turned off when not in use to minimize wear-and-tear on the sample pumps.
	- o Not all CD sample leg elevations were submerged for CD monitoring; therefore, affected sample legs were not completed on the CD reading datasheets.
	- o UFP-VSL-T01B CD sampler station monitoring (recording of temperature and density on datasheet) was not completed because of a nonconformance record (NCR) on the Coriolis flow meter. The two readings that were taken gave erroneous values; therefore, no more readings were taken thereafter.
- In-line caustic additions sent to the HLP-PMP-T21 discharge while transferring simulant from HLP-VSL-T22 to UFP-VSL-T01A/B were done per current operating procedures and not per the TI. The run sheet listed simulant and caustic volumes and addition rates separately; however, the flow meter downstream from the pump, FE-0119, only measures the combined flow rate (LRB 60108, pg. 109). A transfer rate for the simulant and caustic was computed and used as a target for each batch.
- On 2/1/09 (12:11, Operations Manager), an expedited change was approved on having the discharge valves for heat exchanger UFP-HX-T04A/B and UFP-HX-T05A/B open whether or not the heat exchangers are aligned or bypassed (LRB 60229, pg. 29).
- PJM drive times were adjusted after tuning parameters were reached to maintain steady pump operations in UFP-VSL-T02A.
	- o The PJM stroke was adjusted as necessary to avoid overblows (when the pulse tube discharges, all its contents but pressurized air are still applied, causing the air to come out of the bottom of the PJM) and/or to maintain filter-loop flow-rate targets per the run sheet. These adjustments gave stroke lengths outside the tolerance of the run sheet target.
	- o The PJM drive pressures had to be adjusted and tuned by the Lead Test Engineer between ambient and 98°C vessel temperatures.

o (2/16/09 Test Director email). After post-oxidative-leach washing, the PJMs were tuned under standard mode, and standard mode was used unless the PJMs had unstable performance. Standard and simple mode PJM operation was used interchangeably as necessary to achieve stable PJM operations.

3.3 Simulant

PEP process testing was performed with a nonradioactive aqueous slurry of simulant waste chemicals and solids. The simulant composition and make-up recipe were provided by WTP as documented in Simulant Recommendation for Phase 1 Testing in the Pretreatment Engineering Platform.^(a) Aqueous chemical concentrations were within the ranges expected for waste feeds to the PTF. The hydroxide concentration was marginally one standard deviation lower than the average concentration expected in the feeds to the plant. The oxalate and phosphate components were at the lower end of the expected ranges, but the oxalate component was at the solubility limit, and the phosphate component was at or near the solubility limit. The solids components and blend were selected to obtain targeted solids mass loss (aluminum and chromium leaching and oxalate washing) and treatment time. The simulant was not selected to represent any particular Hanford tank waste type.

The simulant was blended from the components listed below. The basis for selecting the individual components and the comparison to actual waste behavior are provided where applicable in the indicated references:

- Boehmite (for Al) (Russell et al. 2009a)
- Gibbsite (for Al) (Russell et al. 2009b)
- Chromium oxyhydroxide (CrOOH) slurry (Rapko et al. 2007)
- Sodium oxalate
- Filtration simulant (Russell et al. 2009c)
- Supernate.

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A separate chromium solids slurry simulant was prepared and added to the PEP process after post-caustic-leach washing (a nonprototypic addition) during the Shakedown/Functional Tests and Integrated Tests A and B. This approach was taken because laboratory-scale tests had shown that the high-temperature caustic-leaching step dissolved significant amounts of the CrOOH solids (Russell et al. 2009a). In Integrated Test D, the chromium solids component of the simulant was added during the simulant make-up process to demonstrate the PTF permanganate addition strategy.

Simulant was procured from NOAH Technologies Corporation (San Antonio, TX). Samples of each simulant batch were characterized to make sure that chemical and physical property requirements were met. Batches of the simulant were procured as follows:

 A 15-gallon trial batch of the blended simulant for laboratory testing to demonstrate the efficacy of the simulant fabrication procedure.

⁽a) P Sundar. 2008. *Simulant Recommendation for Phase 1 Testing in the Pretreatment Engineering Platform.* 24590-PTF-RT-08-006, Rev 0, Bechtel National, Inc., Richland, Washington.

- A 250-gallon scale-up batch of the blended simulant to demonstrate scale-up of the simulant fabrication procedure to an intermediate scale.
- Batches 0, 1, and 2, each nominally 3500 gal, of blended simulant for the Shakedown/Functional Tests and Integrated Tests A and B. These batches did not contain the CrOOH component.
- Batch 3, nominally 1200 gal, for Integrated Test D. This batch contained the CrOOH solids component.
- The CrOOH solids slurry for the Shakedown/Functional Test and Integrated Tests A and B was obtained in two separate batches containing nominally 18 and 36 kg of Cr as CrOOH.

3.3.1 Sampling

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Four tests as described in the *Pretreatment Engineering Platform (PEP)* Testing (Phase I) Test Plan^(a) were conducted at the PEP over 5 months, which generated approximately 3300 samples, of which roughly 1400 were stored as archive samples. A significant number of the original intact samples and processed aliquots were analyzed for chemical composition, such as metals, anions, and hydroxide or physical properties, such as undissolved solids, density, and particle size. The Test Instruction for each test contained a sample collection and analysis table that summarized sampling events and the analysis to be performed on the collected samples. Appendix B provides a list of every sample collected in the Integrated Test A and the associated disposition. A list of samples taken and their purpose is provided in Appendix C. The actual time that samples were taken was recorded in the Test Instruction.

In general, slurry samples were collected using either the vessel's in-tank sampler or an in-line sampler. In-line samples were obtained from the slurry recirculation loop or transfer piping by drawing a side stream from the process flow as shown in Figure 3.3. To obtain a sample, the second valve was fully opened, and then the first valve was opened sufficiently to allow samples to be safely obtained. Samples were collected from the volume between the second and first valves while isolated from the process flow pipe. The vent valve and pressure gauge shown below, however, were not used for Integrated Test A sample collection. The sample line and valves were purged with at least three line volumes before each sampling event.

⁽a) GB Josephson, OP Bredt, JK Young, and DE Kurath. 2009. *Test Plan for Pretreatment Engineering Platform (PEP) Testing (Phase I)*. TP-RPP-WTP-506, Rev. 0.4, Pacific Northwest National Laboratory, Richland, Washington.

Figure 3.3. Simple In-Line Sample Valving

A schematic of the in-tank sampling system for UFP-VSL-T01A/B and UFP-VSL-T02A is shown in Figure 3.4. Samples were obtained with the sample loop in recirculation mode with slurry returned to the vessel. To obtain a sample, a valve was used to divert the entire flow to the sample bottle. The sampling valve and line were purged before each sample to minimize cross contamination with previous sampling events. Sample heights are provided in Appendix D.

Permeate (liquid) samples were taken from the permeate piping between the filter and the pulse pot. The sample line and valves were purged with at least three line volumes before each sampling event.

Figure 3.4. In-Tank Sampling, Showing the Three Radial Positions at Three Heights and Sampling Flow Loop

The organization responsible for the analysis is given in Table 3.3.

Table 3.3. Sample Analysis Organizations

The following sections provide a description of the

- Sample naming convention
- Sample processing during PEP testing
- Analytes of interest
- Analytical methods
- Physical properties.

3.3.2 Sample Naming Convention

Every sample was given a unique sample name. The sample names were a composite of either six or seven separate descriptors. The descriptors are defined in Table 3.4, and the acronyms for each descriptor are defined in Table 3.5. The spacings between descriptors were separated by either an underscore symbol (\angle) or a space (). Sample naming examples are provided after Table 3.5.

Descriptor	Identification	Explanation
Descriptor 1	Test Name	Refers to the Shakedown Test, Functional Test, Integrated Test A, Integrated Test B, or Integrated Test D.
Descriptor 2	Location	Identifies the location that the sample is being collected from based on Table 3.5. For example, a sample collected from a tank will include the acronym of the tank followed by the location within the tank.
Descriptor 3	Test Process Step	Refers to the process step as identified in the sample collection and analysis table in the governing Test Instruction. The test process step includes an identifier for processes that are repeated during testing.
Descriptor 4	Sequential Number	Unique number identifier that increments by 1 each time a sample was collected. All subsamples and separated samples generated from the <i>original sample</i> had the same unique number identifier.
Descriptor 5	Sample Routing	Refers to the sample destination, type of analysis, storage, or archive.
Descriptor 6	Store code	Final destination code.
Descriptor 7	Separated-sample identifier	Refers to the type of <i>subsample</i> ; e.g., decantate, solids, rinse solution. For a <i>separated-sample</i> , a descriptor of the matrix was added after the sample routing identifier.

Table 3.4. Sample Naming Nomenclature Definitions

The sample names are constructed in the following manner:

Descriptor1_ Descriptor2_ Descriptor3a_ Descriptor3b_ Descriptor4_ Descriptor5_ Descriptor6_ Descriptor7

The following description is an example of sample naming for samples collected from HLP-VSL-T22 during Functional testing.

- The sample name for the first Functional Test grab slurry sample collected from the top of HLP-VSL-T22 during process step 1 that was to be archived would have been: F_T22GT_001_XX_0001_ARC_1.
- For the same sample location and type described above, analyzed for PSD on a bulk solid that was not rinsed and ICP on the decantate, the original sample vial would have been labeled: F_T22GM_001_XX_0009_XSP_4_B for the solid.
- Since the solid phase always stayed in the original sample vial, and the liquid was decanted off into a new vial, then the new vial would have been labeled: F_T22GM_001_XX_0009_DEN_0_D for the decantate.
- The decantate from the above sample container could have been subsampled for ICP and Raman; the ICP container would have been labeled as follows: F_T22GM_001_XX_0009_ICP_2_D.
- The Raman container would have been labeled as follows: F_T22GM_001_XX_0009_RAM_3_D.

3.3.3 Sample Processing

The samples were collected in pre-labeled sample containers that were prepared and staged within PDL-W based upon the Sample Collection and Analysis table in the governing Test Instruction. The required analysis determined the sample volume and sample collection container. Sample handling flow diagrams are given in Figure 3.5 through Figure 3.7 for samples collected and processed in PDL-W. Figure 3.6 is a continuation of Figure 3.5 and follows the handling of the wet centrifuged solids. Figure 3.7 indicates the sample flow for samples requiring UDS measurement. The letters "D," "B," "R," and "S," which are shown in the figures, were included in the sample names for phase-separated processed samples. The letter "D" was included in the decanted supernatant sample names. The letter "B" was included in the wet centrifuged solids sample names. The letter "R" was included in the sample name of the combined rinse solution. The letter "S" was included in the sample names of the wet rinsed centrifuged solids. These figures represent two basic sample processing methods. One approach was used during non-leaching test steps, and another was used during leaching.

3.3.3.1 Non-Leaching Samples

Non-leaching samples were not always processed immediately after collection. Because samples might be collected at any time during testing, and testing was being performed 24 hours per day, non-leached samples collected after 0600 hours were typically processed and prepared for shipment by 0600 hours the morning after samples were collected. Non-leach samples used for the analyses listed below were shipped as collected in the container size specified and with the exception of the archive samples; tare and full sample weights were not required.

- Weight percent undissolved solids (UDS) (50-mL)
- Density (50-mL)
- Heat capacity (50-mL)
- Shear strength $(2 \times 1-L)$
- Shear stress vs. shear rate on original intact slurry (100-mL)
- Shear stress vs. shear rate on supernatant (100-mL)
- Total organic carbon on the slurry (50-mL)
- Inductively coupled plasma Silicon $(50\text{-}mL)$
- Archive (50-mL) .

During a sampling event, if an intact slurry sample was collected for density analysis and another intact slurry sample was collected for weight percent UDS, then density measurements were performed on the supernatant in addition to the intact slurry submitted for density analysis. These density measurements were higher accuracy than density measurements obtained following the weight percent UDS method described in Bechtel procedure, *Guidelines for Performing Chemical, Physical, and Rheological Properties Measurements* (24590-WTP-GPG-RTD-001 Rev. 0).(a)

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⁽a) GL Smith and K Prindiville. 2002. *Guidelines for Performing Chemical, Physical, and Rheological Properties Measurements*. 24590-WTP-GPG-RTD-001 Rev. 0, Bechtel National, Inc., Richland Washington.

Figure 3.5. Sample Processing for Decantate Handling

Figure 3.6. Sample Processing for Solids

Figure 3.7. Sample Processing for UDS

A single 50-mL sample was centrifuged and the supernatant decanted to allow multiple laboratories to perform several analyses simultaneously on the supernatant. To perform this phase separation, the original sample was centrifuged at ~4500 G with a swinging bucket rotor in PDL-W. The centrifuging time was initially set for 10 minutes. This centrifuging time was sufficient to cause phase separation during shakedown, Integrated Test A, and oxidative leaching. The actual centrifuging time is recorded on the sample bench sheets. After centrifuging, the supernatant was decanted and submitted for some or all the analyses listed below:

- \bullet ICP
- total dissolved solids
- density
- IC
- TOC
- Raman
- free hydroxide.

Not all these analyses were necessarily performed on every decanted supernatant. The required analysis was identified in the Sample Collection and Analysis table in the governing Test Instruction. Density was determined on the decanted supernatant at PDL-W for all samples being analyzed by Raman spectroscopy before the samples were transported for analysis. In addition, at times, density was determined by Southwest Research Institute (SwRI) before ICP analysis. The wet centrifuged solids from this same container were either submitted for ICP analysis, if it was required, or stored in the original sample collection container as excess. A tare weight (before filling) and gross weight (after filling) of the sample container were documented on sample bench sheets.

For Dow Corning to quantify the anti-foam agent (AFA) compounds by Gel Permeation Chromatography (GPC) in the solid and supernatant fractions of the slurry, two 50-mL slurry samples were collected and centrifuged at 4500 G for 10 minutes, and the supernatant was decanted into two additional properly labeled 50-mL containers before shipping. Weighing empty and filled containers was not required.

For SwRI to quantify silicon by ICP in the AFA, a 50-mL slurry sample was collected and shipped as collected during non-leaching process steps. Once at SwRI, the slurry was centrifuged at 2200 G for an hour, the supernatant was separated from the wet centrifuged solids, and then each phase was analyzed separately. During leaching, the phase separation occurred in PDL-W following the standard practice of centrifuging at ~4500 G for 10 minutes.

A single 50-mL container was filled for particle-size distribution (PSD), X-ray diffraction (XRD), and scanning electron microscopy (SEM). Initially, these samples were centrifuged. The supernatant was decanted and stored while the wet solids were submitted for analysis. Intact slurry samples were submitted after shakedown and Integrated Test A and initial Integrated Test B simulant characterization. If a phase-separated sample was submitted, then descriptor 7 was included in the sample name. If descriptor 7 was not in the name, then an intact sample was submitted for analysis. Weighing empty and filled containers was not required.

For parallel CUF and bench-scale testing, varying amounts of either permeate or slurry samples were collected. A tare weight (before filling) and gross weight (after filling) of the sample container was recorded on the sample container. These samples were delivered to APEL by the next business day after sample collection.

3.3.3.2 Leaching Samples

The core critical analyses for leached samples were weight percent UDS, slurry density, inductively coupled plasma, ion chromatography, Raman, and free hydroxide analyses. Two 50-mL samples were collected for the UDS and density analyses. Another 50-mL sample was collected for the remaining core analyses.

- During caustic leaching, the original samples were immediately placed in a thermostatically controlled water bath at $20 \pm 2^{\circ}$ C to decrease the sample temperature to $25 \pm 5^{\circ}$ C after collection. Once the desired temperature was reached, which typically took less than 10 minutes for 50-mL containers, the samples were allowed to sit for 24 hours in PDL-W. The density sample was shipped as collected. The two other samples to be used for analysis were centrifuged following the sample process described for non-leached samples.
- During oxidative leaching, sample processing began immediately after sample collection. The oxidative leach density sample was shipped as collected while the other two samples were phase separated immediately after collection by centrifuging and decanting the supernatant as described above for non-leached samples. To allow for consistency in comparing analytical results, the samples for ICP analysis collected immediately before adding $NaMnO₄$ were phase separated immediately after collection. The oxidatively leached centrifuged solids from samples collected immediately before adding N_aMnO_4 and at the end of oxidative leaching for analysis were weighed and rinsed three times with 0.01-M NaOH solution. The actual quantity of rinse is recorded on sample bench sheets. The wet solids were vortexed three times to suspend and rinse the solids after each addition of NaOH solution. The rinsate was collected and weighed. The density of the rinsate was measured at PDL-W, and then it was split into subsamples for ICP and Raman analysis.

During leaching, additional 50-mL samples were collected for archiving. Caustic leached archived samples were also cooled and phase separated as described above before storage. The oxidatively leached archived samples were also immediately phase separated after collection as described above. The centrifuged oxidatively leached solids that went to storage were not rinsed.

Undissolved Solids

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The sample processing for UDS is given in Figure 3.7*.* The UDS was measured at PNNL only when results were needed the same day the sample was collected. A halogen moisture analyzer (HG63, Mettler Toledo) was used to determine the wt% UDS under procedure TPR-RPP-WTP-648. If results for multiple samples were required, then a modified version of the oven method outlined in *Guidelines for Performing Chemical, Physical, and Rheological Properties Measurements* $(24590-WTP-GPG-RTD-001, Rev. 0)$ was used.^(a)

⁽a) GL Smith and K Prindiville. 2002. *Guidelines for Performing Chemical, Physical, and Rheological Properties Measurements*. 24590-WTP-GPG-RTD-001 Rev. 0, Bechtel National, Inc., Richland Washington.

Supernatant Density

Density measurements were performed at PDL-W on samples that were analyzed by Raman. Density was measured in accordance with TPR-WTP-PEP-054, *Determination of Density using Pycnometer or Graduated Cylinder*. All other density values were analyzed externally by SwRI.

3.3.4 Analytes of Interest

The analytes of interest, analytical techniques, and required estimated quantitative limits were set for the analytical laboratories and are provided in Table 3.6. The limits included processing factors associated with preparation and analyses. For techniques where multiple analytes were measured, all analytes were requested to be reported. The preparative and analytical quality control (QC) requirements specified in Hanford Analytical Services Quality Assurance Requirements Documents (HASQARD), Rev. 2 were required to be applied to only the analytes of interest or target analytes. All other analytes were considered opportunistic, and QC failures for these analytes did not require corrective action as described in HASQARD, Rev. 2. For example, opportunistic analytes were expected to include (but are not limited to) As, B, Sn, and Zn. No additional analytes were introduced into the list of target analytes.
Analyte	Solids	Liquid solution	Analytical Technique
	μ g/g	μ g/mL	
AI	$4.0E + 01$	$5.0E + 00$	
Ca	$2.0E + 01$	$5.0E + 00$	
Ce	$8.0E + 00$	$7.5E + 01$	
Cr	$5.0E + 00$	$1.0E + 00$	
Fe	$3.0E + 02$	7.5E+01	
K	$3.0E + 01$	$1.5E + 01$	
La	$6.0E + 00$	$7.5E + 01$	
Mg	$7.0E + 00$	$5.0 E + 00$	
Mn	$1.0E + 01$	$5.0E + 00$	ICP-AES
Na	$3.0E + 02$	7.5E+01	
Nd	$1.0E + 01$	$5.0E + 00$	
Ni	$2.0E + 01$	$3.0E + 01$	
P	$5.0E + 01$	$1.0E + 01$	
Pb	$3.0 E + 01$	$7.5E + 1$	
Si	$3.0E + 03$	$7.5E + 01$	
Sr	$2.0E + 01$	$5.0E + 00$	
Zr	$9.0E + 01$	7.5E+01	
Cs	N/A	4 pg/mL	ICP-MS
Chloride	N/A	$1.2E + 02$	
Nitrite	N/A	$1.2E + 02$	
Nitrate	N/A	$4.0E + 01$	Ion Chromatography
Phosphate	N/A	$1.2E + 02$	(water-soluble species)
Sulfate	N/A	$6.0E + 01$	
Oxalate	N/A	$2.5E + 02$	
Hydroxide	N/A	5E-03 M	Titration
Total inorganic carbon	N/A	$2.0E+02$ (as C)	Coulometer
Density	0.9 to 1.7 gm/mL	0.9 to 1.7 gm/mL	Gravimetric
TDS	N/A	N/A	Gravimetric
UDS	N/A	N/A	Gravimetric

Table 3.6. Estimated Quantitative Limits for Solids and Supernatants in Sample Loop Testing, PEP Operation, and Simulant Acceptance Samples

3.3.5 Analytical Methods

This section describes the analytical methods used to determine the chemical composition of the PEP simulant samples collected during testing. Analytical results are provided in Section 5.

3.3.5.1 Preparation for ICP or ICP-MS

For the solutions, aliquots were diluted with hydrochloric acid and then analyzed. Dilutions were made with a representative aliquot of approximately 2 grams combined with 5-mL of 50% hydrochloric acid and then diluted to a final volume of 50-mL with DI water.

Two preparative techniques were used for solids. The first preparative technique was lithium metaborate/tetraborate fusion, referred to as prep method "80/20 Fusion." Aluminum, barium, cerium, chromium, iron, lanthanum, manganese, neodymium, silicon, and strontium were reported from the fusion. The second preparative method used concentrated nitric, perchloric, hydrofluoric, and hydrochloric acids in an open vessel and was referred to as the "Teflon" prep method. If residue remained from the "Teflon" method, then the residue was separated, dried, and fused using the lithium metaborate/tetraborate fusion technique. Both the "Teflon" and residue preparations were analyzed. The remaining metals were reported from either the "Teflon" digestion only or the combination of the two. If an analyte was detected in both the Teflon digestate and the residue fusions, then the reported result was the sum of the results obtained from the two preparations. These results were identified as "combined" on Sample Analysis Data Sheets while analytes reported from only the fusion were identified as "80/20 Fusion," and analytes reported solely from the acid digestions were identified as "Teflon" on the Sample Analysis Data Sheets. The preparative QC samples included a sample duplicate, preparation blank (PB), solid laboratory control samples (LCS) consisting of obsidian rock and basalt rock, and an aqueous laboratory control sample (LCS) and a matrix spike (MS) for "Teflon." The results are reported on an as-received or wet weight basis. The percent solids were determined at 105°C and reported on the Sample Analysis Data Sheets for all solids.

3.3.5.2 Metals Analysis by ICP-AES

All metals were determined by ICP-AES using SwRI procedure TAP01-0406-130 with the exception of cerium, cesium, lanthanum, and neodymium. The preparative QC samples (duplicate, PB, LCS, MS) were processed along with analytical workstation QC (initial and continuing calibration verifications, initial and continuing blanks, interference check samples, and post-digestion spikes).

3.3.5.3 Metals Analysis by ICP-MS

Cerium, cesium, lanthanum, and neodymium were determined by ICP-MS in accordance with SwRI procedure TAP01-0406-046. The preparative QC samples (duplicate, PB, LCS, MS) were processed along with analytical workstation QC (initial and continuing calibration verifications, initial and continuing blanks, interference check samples, and post-digestion spikes).

3.3.5.4 Anions (IC)

Decanted supernatant and rinsants were analyzed by IC for chloride, nitrate, nitrite phosphate, sulfate, and oxalate at SwRI in accordance with procedure SwRI TAP 01-0406-042. Approximately 0.25 g of the sample was diluted to 50-mL using DIW. Since the dilution was performed by weight, the sample results are reported on a weight basis. The standard reporting by the laboratory is nitrate as N, nitrite as N, and phosphate as P. Shortly after PEP testing began, PNNL requested that results be reported as nitrate, nitrite, and phosphate and not as nitrogen or phosphorus. The IC analytical report narrative identifies conversion factors used to report as anions. QC samples generated at the analytical workstation included a sample replicate determination, preparation blank, LCS, and matrix spike (MS).

3.3.5.5 Total Inorganic Carbon/Total Organic Carbon (TIC/TOC)

The samples were analyzed for total carbon (TC) using a Dohrman DC-80 Carbon Analyzer in accordance with procedure SwRI TAP 01-0406-001. The liquids were directly injected, and the slurry was analyzed using the soil/sludge sampler. Another aliquot of the sample was acidified with sulfuric

acid and sparged to remove inorganic carbon and then analyzed for TOC. The TIC is calculated from the difference in the TC and TOC results. All samples were analyzed in duplicate, and average results were reported when the relative percent differences (RPDs) were less than 20%. If the RPD was greater than 20%, then the sample was analyzed twice more, and the average of the quadruplicate analysis was reported. The liquids were corrected for density, and all sample results were reported on a weight basis. QC samples were generated at the analytical workstation and included a sample replicate determination, preparation blank, LCS, and MS.

3.3.5.6 Hydroxide

The free-hydroxide concentration was determined by potentiometric titration with standardized HCl according to procedure RPG-CMC-228, "Determination of Hydroxyl (OH-) and Alkalinity of Aqueous Solutions, Leachates, and Supernates and Operation of Brinkman 636 Auto-Titrator." The free hydroxide was defined as the first inflection point on the titration curve. QC samples were generated at the analytical workstation and included a sample replicate determination, preparation blank, and blank spike (BS).

3.3.5.7 Raman

Raman was used to quantify aluminate, carbonate, chromate, hydroxide, nitrate, nitrite, oxalate, phosphate, and sulfate following procedure RPG-CMC-240. If precipitate formed in the solution samples submitted for Raman before the analysis, then the samples were centrifuged, and aliquots of the liquid were pipetted and analyzed. Two sets of Raman results were reported for shakedown, Integrated Test A, and some of the Integrated Test B samples. The first set of results was generated using calibrations that were periodically adjusted to optimize performance of QC check samples. The second set of results was recalculated based on the original calibration parameters. The generation of these two sets of results and the discovery of the calibration adjustments are documented as RPP-WTP CAR, number 42708.1. Only Raman results from the recalculation are provided. As a result of using the original calibration parameters, an occasional QC sample falls outside of established performance limits. QC samples were generated at the analytical workstation and included a sample replicate determination, preparation blank, BS, and MS.

3.3.5.8 Preparation for Gel Permeation Chromatography (AFA components)

Duplicate samples were collected to support this analysis. Both samples were centrifuged and phase separated at PDL-W before shipping. Once at the analytical laboratory, one of the two liquid fractions was mixed with toluene while the other was mixed with tetrahydrofuran (THF). Both were shaken for an hour and allowed to settle overnight. The upper organic layer was removed and allowed to concentrate. The toluene extract was dried at room temperature to completeness and brought to 3.0-mL toluene. The THF extracts were concentrated to approximately 2-mL at room temperature and then placed in an 80°C oven to dry to completeness and brought up to 3.0-mL THF. One of the two solids fractions was extracted with 10-mL of toluene while the other was extracted with 10.0-mL of THF. In both cases, the solvent was added directly into the receipt vessel, and the initial sample and solvent were shaken for 2 hours. All solutions were filtered through 0.45-µm PTFE syringe filters. The calibration was verified before samples were analyzed and after every 15 samples.

3.3.5.9 Gel Permeation Chromatography (AFA components)

The toluene extract allowed polydimethylsiloxane (PDMS) to be analyzed by gel permeation chromatography (GPC) while the THF extract allowed polypropylene glycol (PPG) to be analyzed by GPC. The toluene extract was analyzed by a Waters 717 autosampler and a Waters 2410 differential refractometer. Two columns, a PLgel 5-µm guard column and a PLgel 5-µm Mixed-C column, were used for separation, and an HPLC grade toluene at a flow of 1.0-mL/minute was used as the eluent. Both the columns and detector were heated to 45°C. The THF extract was analyzed by a Waters 2695 Separation Module equipped with a vacuum degasser and a Waters 2410 differential refractometer. Two columns, a PLgel 5-µm guard column and a PLgel 5-µm Mixed-C column, were used for separation, and a certified grade THF at a flow of 1.0-mL/minute was used as the eluent. Both the columns and detector were heated to 35^oC. The quantity of PDMS and PPG was used to determine the amount of AFA, O2-3183A Antifoam, in the samples. However, SwRI had a back-up approach for tracing the fate of AFA by quantifying the Si in the liquid and solid fractions. SwRI received the intact slurry, separated the solid from the liquid, and analyzed each fraction for Si by ICP. Since the AFA is a proprietary composition belonging to Dow Corning, they performed the analytical work. The calibration was verified before samples were analyzed and after every 15 samples.

3.3.6 Physical Properties

This section describes the methods used to determine the physical properties of the PEP simulant samples, the crystal form and solids environment, density, wt% UDS, rheology, particle-size attributes, and heat capacity. A more detailed outline of the methods used in this section is given, where applicable, in the appropriate test data package supplied with the sample results for each characterization.

3.3.6.1 Percent UDS and Density

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Weight percent UDS, wt% dissolved solids, bulk slurry density, and supernatant density were determined following 24590-WTP-GPG-RTD-001, Rev 0. Some steps in 24590-WTP-GPG-RTD-001, Rev 0_s ^(a) were not performed because the results generated from these steps were not needed, or steps were slightly modified to reduce analysis time. These modifications are not believed to impact the final results.

Slurry sample UDS analysis was performed with the following modifications:

- Steps 4, 6, and 7 were omitted because the settling data were not required.
- Steps 8 and 9. Centrifuge the cones at \sim 1000 G for 1 hour. Record the volume of the total sample and the volume of centrifuged solids on the physical properties data sheet. After this, the WTP procedures required that the supernatant be decanted into a pre-weighed graduated cylinder to obtain the supernatant mass and volume. Rather than use a graduated cylinder, the volume of supernatant was calculated as the difference between the volume of the total sample and the volume of centrifuged solids.

⁽a) GL Smith and K Prindiville. 2002. *Guidelines for Performing Chemical, Physical, and Rheological Properties Measurements*. 24590-WTP-GPG-RTD-001 Rev. 0, Bechtel National, Inc., Richland Washington.

- Step 11 requires decanting the centrifuged supernatant liquid to a pre-weighed graduated cylinder. Because of the high concentration of NaOH in some of the samples, some of the liquid was left in the cylinder. Rather than decant, the centrifuged supernatant liquid was pipetted to a preweighed glass Petri dish or vial, and the mass of the supernatant liquid was recorded.
- Step 12 was omitted because air-drying was not necessary.

Separated slurry samples UDS analysis was performed with the following modifications:

- Step 8 requires centrifuging at \sim 1000 G for 1 hour. Samples were centrifuged at \sim 4500 G for either 10 minutes or 1 hour. The centrifuge time is documented on the sample handling benchsheets in *TDP-WTP-349.*
- Wet solids (designated as "B") and decanted supernatants (designated as "D") were sent to SwRI along with empty vial and separated sample weights. These weights are also documented on the sample handing benchsheets in *TDP-WTP-349.*
- The wet solids were transferred to a tared container using DIW to completely remove the solids. The solids and DIW rinses were then oven dried to constant weight.
- The total weight of the supernatant was calculated from the sample-handling benchsheets. Since it was not feasible for the entire volume of supernatant to be oven dried, approximately 5-mL was transferred to a Petri dish for drying.
- Density was determined on the supernatant liquid using a 5-mL volumetric flask.

3.3.6.2 Slurry and Supernatant Density

Three preweighed 5-mL Class A volumetric flasks were filled to the specified line with the liquid sample and reweighed. The density was calculated as the mass of the sample divided by the certified volume of the volumetric flask.

Slurry samples requiring a higher accuracy supernatant density than provided by the UDS analysis were subsampled and centrifuged for 1 hour at 1000 G, and the phase-separated supernatant was analyzed as described in the paragraph above.

3.3.6.3 Rheological Measurements

Rheological testing was conducted in accordance with RPL-COLLIOD-02, *Measurement of Physical and Rheological Properties of Solutions, Slurries and Sludges*. For the current study, two regions of tank waste flow behavior were considered: 1) incipient motion in settled tank waste solids (shear strength), and 2) non-elastic flow of tank waste slurries and supernates (flow curve).

Shear-Strength Testing

For settled tank waste slurry solids, a finite stress must be applied before the material will begin to flow. The stress required to transition the material from elastic deformation to viscous flow is referred to as the shear strength, and its origin can be attributed to static and kinetic friction between individual particles and/or aggregates, the strength of the matrix supporting the coarse fraction (i.e., the interstitial fluid), and sludge cohesion arising from interparticle adhesive forces such as van der Waals forces.

The shear strength was measured using the vane method. For the vane technique, the stress required to begin motion is determined by slowly rotating a vane immersed in the test sample's settled solids while continuously monitoring the resisting torque as a function of time. A material's static shear strength is then associated with the maximum torque measured during the transition from initial to steady-state vane rotation.

The maximum torque required for incipient motion is dependent on vane geometry. To account for vane-geometry effects, the shear strength is expressed in terms of the uniform and isotropic stress acting over the surface area of the cylinder of rotation swept out by the vane. The shear strength is related to the maximal torque during incipient motion according to Equation 3.1 (Barnes and Dzuy 2001):

$$
\tau_{SS} = \frac{M_{max}}{4\pi R^3 \left(\frac{H}{2R} + \frac{1}{3}\right)}
$$
\n(3.1)

Here, τ_{ss} is the shear strength (N/m²), M_{max} is the maximum torque (N·m), and *R* and *H* are the radius and height of the cylinder of rotation swept out by the vane (m). Because the shear band observed upon slow rotation of the vane does not extend appreciably beyond the vane paddles, *R* and *H* are taken to be the dimensions of the vane itself.

The proximity of the vane to the sample container inner surfaces as well as the free surface of the settled solids can impact shear-strength results. As such, certain geometric constraints must be satisfied for the test to be considered independent of container geometry (Table 3.7).

Table 3.7. Vane Immersion Depth and Container Geometry Constraints for Shear-Strength Tests Using the Vane Technique

Constraint	Criterion	For 8×16 mm (R \times H) Vane
Vane height to radius	H < 7R	$H < 56$ mm (Satisfied)
Container radius to vane radius	$R_{\text{cont}} > 2R$	R_{cont} > 16 mm
Immersion depth to vane height	h > H	$h > 16$ mm
Separation between bottom of vane and container floor (h_{floor})	$h_{floor} > 0.5H$	$h_{floor} > 8$ mm
$R =$ radius		
$H = height$		

Flow-Curve Testing

The non-elastic flow of tank waste slurries and supernates is characterized with rotational viscometry. The typical result of such testing is a set of flow-curve data, which show the stress response of a material to a range of applied rates-of-deformation. Specifically, flow-curve testing allows characterization of a material's shear stress, τ , which is the response as a function of applied shear rate, $\dot{\gamma}$. Once measured, the flow-curve data can be interpreted with several constitutive equations for the viscous stress/rate-of-strain relationship. Such analysis allows the flow behavior over a broad range of conditions

to be described with just a few rheological descriptors, such as viscosity, yield stress, consistency, and flow index.

A concentric cylinder rotational viscometer operated in controlled-rate mode was used for flow-curve testing of tank waste slurries and supernates. Rotational viscometers operate by placing a given volume of test sample into a measurement cup of known geometry. A cylindrical rotor attached to a torque sensor is then lowered into the sample until the slurry is even with, but does not cover, the top of the rotor. A single-point determination of a fluid's flow properties is made by spinning a rotor at a known rotational speed, Ω , and measuring the resisting torque, *M*, acting on the rotor. The torque acting on the rotor can be directly related to the shear stress at the rotor using the equation,

$$
\tau = \frac{M}{2\pi HR_i^2} \tag{3.2}
$$

The shear stress has units of force per area $(N/m²)$. The rotational rate is related to the shear rate. However, calculating the fluid shear rate at the rotor is complicated by the fact that the shear rate depends on both the measurement-system geometry and the fluid rheological properties. For the simplest fluids (i.e., Newtonian fluids), the shear rate of the fluid at the rotor can be calculated given the geometry of the cup rotor shear by using the equation,

$$
\dot{\gamma} = \left(\frac{2R_o^2}{R_o^2 - R_I^2}\right)\Omega\tag{3.3}
$$

Here, the shear rate has units of inverse seconds (s^{-1}) . Calculating the shear rate for materials showing more complex shear-stress versus shear-rate behavior (i.e., non-Newtonian fluids) requires estimates of yield stress and a degree of shear-thinning or shear-thickening. As the goal of rheological testing is to determine and quantify such behavior, these values are typically not known. This requirement can be circumvented by using a cup-and-rotor system with a small gap $(\sim 1 \text{ mm})$ for fluid shear. For fluid flow in small-gap cup and rotor systems, shear-rate effects introduced by fluid properties are minimized such that Equation 3.3 provides an accurate determination of shear rate for non-Newtonian materials.

The resistance of a fluid to flow is often described in terms of the fluid's apparent viscosity, η_{app} , which is defined as the ratio of the shear stress to the shear rate:

$$
\eta_{app} = \frac{\tau}{\dot{\gamma}}
$$
\n(3.4)

For Newtonian fluids, the apparent viscosity is independent of the shear rate. For non-Newtonian fluids, the apparent viscosity will vary as a function of the shear rate. The unit of apparent viscosity is Pa·s, although it is typically reported in the unit of centipoise (cP; where $1 \text{ cP} = 1 \text{ mPa·s}$).

Flow-curve data are usually combined plots of τ and η_{app} as a function of $\dot{\gamma}$. As stated above, flow-curve data can be interpreted with several constitutive equations (i.e., flow curves), allowing

characterization of those data with just a few rheological descriptors. The behavior of tank waste sludges, slurries, and supernates can be described by four common flow-curve equations:

Newtonian—Newtonian fluids flow as a result of any applied stress and show constant viscosity over all shear conditions. The flow curve for Newtonian fluids is

$$
\tau = \eta \dot{\gamma} \tag{3.5}
$$

where η is the Newtonian viscosity.

Ostwald (Power Law)—Power-law fluids flow as a result of any applied stress and have viscosities that either increase or decrease with increasing shear rate. They are described by,

$$
\tau = m\dot{\gamma}^n \tag{3.6}
$$

where *m* is the power-law consistency index, and *n* is the power-law index. Power-law fluids with $n < 1$ are referred to as pseudoplastic (shear-thinning), whereas power-law fluids with $n > 1$ are referred to as dilatant (shear-thickening).

Bingham Plastic—Bingham plastics are fluids that show finite yield points. A finite stress (i.e., the yield stress), must be exceeded before these types of materials flow. Once flow is initiated, the stress response of the material is Newtonian over the rest of the shear-rate range. Bingham plastics are described by

$$
\tau = \tau_o^B + k_B \dot{\gamma} \tag{3.7}
$$

where τ_o^B is the Bingham yield index, and k_B is the Bingham consistency index.

Casson—Fluids that behave in accordance with a Casson model show a finite yield followed by pseudoplastic behavior. They are described by,

$$
(\tau)^{0.5} = (\tau_o^c)^{0.5} + (k_c \dot{\gamma})^{0.5}
$$
 (3.8)

where τ_o^C is the Casson yield index, and k_c is the Casson consistency index. Although more limited in the types of flow behavior that it can describe relative to the Herschel-Bulkley equation, the Casson model is popular because it is capable of accurately describing many shear-thinning fluids and because units on the parameters are more physically meaningful (e.g., the consistency is in Pa·s versus $Pa·sⁿ$ for the Herschel-Bulkley model).

Power-law fluids, Bingham plastics, and Casson fluids are referred to as non-Newtonian fluids. Generally, liquids without internal and/or interconnected structures (such as tank waste supernatants) are Newtonian. Sludges and slurries are typically non-Newtonian, but their exact behavior depends on the concentration of solids and suspending phase chemistry. Sufficiently dilute slurries may show Newtonian behavior.

Rheology Instrumentation

Rheological characterization was accomplished using an Anton Parr Rheometer (MCR 301) for shear-rate measurements, and shear strength was performed on a Rotovisco RV20 Measuring System M equipped with an M5 measuring head and RC20 controller sold by HAAKE Mess-Technik GmbH u Co. (now the Thermo Electron Corporation). The M5 measuring head is a "Searle" type viscometer capable of producing rotational speeds up to 500 rpm and measuring torques up to 0.049 N·m. The minimum rotational speed and torque resolution achievable by this measuring head are 0.05 rpm and 0.49 mN·m, respectively.

Calibration and analysis were performed according to TPR-WTP-PEP-049.

Specific measurement tools, such as cup-and-rotor assemblies and shear vanes, are attached to measure selected rheological properties. Shear-strength measurements employ an 8 mm \times 16 mm (R \times H) shear vane tool. Flow-curve measurements employed an MV1 stainless steel measuring cup and rotor. The dimensions of the MV1 and vane measuring systems are listed in Table 3.8.

	Vane/Rotor Radius	Vane/Rotor Height Container Radius		Gap Width
Measuring System	(mm)	(mm)	(mm)	(mm
Vane Tool			$>16^{(a)}$	
MV ₁	20 04	60		0.96

Table 3.8. Vane and Cup and Rotor Measuring System Dimensions

The temperature is controlled with a combination of the standard measuring system temperature jacket and a temperature-controlled recirculator. The jacket temperature is monitored using a Type-K thermocouple calibrated over 0° to 100°C and connected to a calibrated multichannel temperature display. The temperature control is employed only for flow-curve measurements. Shear-strength measurements are carried out at ambient cell temperature.

The rheometer was controlled, and data were acquired with a remote computer connection using the RheoWin Pro Job Manager Software, Version 2.96. During measurement, the software automatically collects and converts rotor torque readings into shear stresses based on Equation 3.1 (for vane testing) or Equation 3.2 (for flow-curve testing). Likewise, the software also automatically converts the rotational rate readings into shear rates based on Equation 3.3.

Rheology Materials and Methods

No sample treatment was performed before analysis with the exception of the mechanical agitation required to mix and subsample selected sample containers.

Shear-Strength Testing

Before testing, the simulant slurries that were provided for shear-strength testing were mixed thoroughly and subsequently allowed to settle for at least 48 to 72 hours. When possible, the shear strength was measured by immersing the $16-x16$ -mm ($D\times H$) vane tool to a depth of 15 mm into the settled solids. The vane was slowly rotated (0.03 1/sec) for 120 seconds. For the entire duration of rotation, the time, rotational rate, and vane torque were continuously monitored and recorded. At the end of the measurement, shear stress versus time data were parsed, and the maximum measured shear stress (i.e., the material's shear strength) was determined.

Flow-Curve Testing

Each flow curve was measured over an 11-minute period and split into three intervals. Over the first 5 minutes, the shear rate was smoothly increased from zero to 1000 s^{-1} . For the next minute, the shear rate was held constant at $1000 s⁻¹$. For the final 5 minutes, the shear rate was smoothly reduced back to zero. During this time, the resisting torque and rotational rate were continuously monitored and recorded.

Results are provided in the test data packages listed below, with summary results provided in Appendix H.

• Integrated Tests A and B TDP-WTP-359

3.3.6.4 Particle-Size Distribution

Particle sizes were characterized according to procedure RPL-COLLOID-01, Rev. 1, *Particle Size Analysis Using Malvern MS2000*. This procedure uses a Mastersizer 2000 (Malvern Instruments, Inc., Southborough, MA 01772 USA) with a Hydro S wet dispersion accessory. Malvern lists the Mastersizer particle-size measurement range as nominally 0.02 to 2000-um. The actual PSD measurement range is dependent on the accessory used as well as the properties of the solids being analyzed. The Malvern 2000 uses laser diffraction technology to define PSD.

The Hydro S wet-dispersion accessory consisted of a 150-mL dispersion unit coupled with a sample flow cell with a continuous variable and independent pump and stirrer and ultrasound. Flow, stirring rate, and sonication can be controlled and altered during measurement. PSD measurements can be made before, during, and after sonication, allowing the influence of each on the sample PSD to be determined. The primary measurement functions of the Malvern analyzer were controlled through Mastersizer 2000 software (Malvern Instruments, Ltd. Copyright 1998-2002). The properties applied to the test samples are summarized in Table 3.9.

The PSD measurements were conducted in either DIW or in a 0.01-M NaOH dispersion solution matrix, depending on the sample being analyzed. The sample dispersion was added drop-wise to the dispersion unit (while the pump and stirrer were active) until an obscuration in the range of 10 to 20% was reached.

Property	
Material selected for optical properties	Ferric Oxide Hydroxide
Refractive Index (RI)	2.94
Absorption	$\mathbf{0}$
Analysis mode	General purpose
Sensitivity	normal
Suspending Phase	Water/ 0.01 -M NaOH

Table 3.9. Properties Applied to Group 8 Test Materials

The size distributions of particles were measured under varying flow conditions before and after sonication. For each condition, multiple measurements of PSD were taken. The analyzer software then generated an average of these measurements. Both the individual measurement and average were saved to the analyzer data file.

3.3.6.5 X-Ray Diffraction

The sample mounts for XRD examination were prepared by first cleaning the solids. This procedure included centrifuging the solids into a pellet and decanting the solute. Fresh washing solution was added to the pellet, and it was resuspended. The pellets in solution were vortexed to reconstitute them into the solution, and the centrifuging procedure was repeated three times. Following the final centrifuging and decant, the remaining pellet was left to dry in a 105°C oven overnight. The pellet was then pulverized to a powder with a tungsten carbide milling chamber for 1 minute in the Angstrom shaker mill, mixed with an internal standard (rutile, TiO₂, or alumina, Al_2O_3), milled for another 2 minutes to make sure that the two powders were a homogenous mixture, and then mounted into an off-axis, zero background, quartz sample holder. The XRD examination was conducted according to procedure APEL-PAD-V, *Operation of Scintag Pad-V X-Ray Diffractometer*. The XRD instrument used for these samples was the PNNL Scintag PAD V XRD (property number WD33356), located in Laboratory 102 in the APEL building. The data range for the sample was 5° to 80° 2θ, with a step size of 0.02° 2 θ and count time of 2.0 seconds per step. Copper K α X-rays were used. The X-ray tube operating conditions were 45 kV and 40 mA. Phase identification was done by use of the JADE search match routines (Version 6.0, Materials Data Inc.) with comparison to the International Centre for Diffraction Data (ICDD) database PDF-2, release 1999, which includes the Inorganic Crystal Structure Database (ICSD) maintained by Fachinformationszentrum (FIZ), Karlsruhe, Germany. The chemistry provided for Group 7, in order of decreasing concentration, was Fe, Na, U, P, Ca, Al, Si, Bi, Sr, and Mg. Phase identifications were first done without chemistry restrictions. Searches were restricted to the PDF and ICSD inorganic sections.

The pattern was also examined using RIQAS (release 4.0.0.26, 6/10/2002, Materials Data Inc.) Rietveld analysis software. The phases identified above were input into the analysis along with a polynomial background and an amorphous hump at ~35° 2θ.

Details of the XRD analysis and results are provided in test data package, TDP-WTP-357, with summary results provided in Appendix H.

3.3.6.6 Scanning Electron Microscope

The final step just before the supernatant was decanted, the specimen was vortexed, and a small volume of slurry was drawn up using a pipette and placed on an aluminum stub within the same sample processing as above for the XRD sample preparation. The slurry was placed in an oven at 105°C to dry overnight. The sample was then coated with gold-palladium using a Polaron Range plasma sputter coater and analyzed with a JEOL SEM (property number WD30596) according to APEL-102-SEM, *Scanning Electron Microscope Examination*. Selected sample areas were evaluated by X-ray energy dispersive spectroscopy (EDS) for qualitative elemental composition.

Results are provided in the test data package, TDP-WTP-356, provided in Appendix H.

3.3.6.7 Heat Capacity

Approximately eight samples at key process steps were collected and analyzed for heat capacity during Integrated Tests A and B. Three nominal 30-mg subsamples were taken from each of these samples as they were vigorously stirred using a pipette with an enlarged tip to confirm that the slurry samples were representative. Each slurry sample was analyzed in triplicate, recognizing that obtaining a 30-mg representative sample is challenging.

A Perkin-Elmer DSC7 differential scanning calorimeter (DSC) was used to determine the heat capacity of each subsample at temperatures between 20°C and 95°C. The DSC7 was temperature and enthalpy calibrated using a gallium standard (NIST SRM 2234) (m.p. 29.8°C) and/or an indium standard (NIST SRM 2232) (m.p. 156.6°C).

The method used for heat-capacity measurement was as provided in the DSC7 operating manual. In this method, the heat capacity of the empty sample pan is measured to provide the "baseline"; the sample pan was a gold "volatile" sample pan designed to contain volatile samples such as the water in these PEP slurries. The heat capacity of a reference or standard material, such as sapphire or high-purity water, is then determined; the purity of these water standards was determined by measuring their electrical conductivity. Finally, the heat capacity of the sample is determined. The measured heat capacity of the sample is adjusted to remove the contribution of the empty pan. We further adjusted the reported heat capacity by applying a measured response factor for the high-purity water.

Optimally, the heat-capacity measurement approach would include analyzing a standard or reference material in the same container in which the sample was analyzed; however, because the analysis of these slurry samples required a hermetic seal, which cannot be broken and resealed, an equivalent gold volatile sample pan was used for the water. In addition, we characterized a sapphire standard to demonstrate instrument performance; the heat capacity of sapphire (0.8 J/g $^{\circ}$ C) is significantly lower than that of the PEP slurries (\sim 3.5 J/g) and does not provide an adequate calibration adjustment.

Results are provided in the heat capacity test data packages listed below and given in Appendix H.

- Integrated Tests A and B TDP-WTP-368
- Integrated Test B TDP-WTP-372

4.0 Test Narration

Caustic leaching in the ultrafiltration feed preparation vessels UFP-VSL-T01A and B was demonstrated in Integrated Test A using nonradioactive simulant. The subsequent sections summarize the process steps executed for Integrated Test A. Sampling events have been omitted from the Integrated Test A narrative for clarity, given the large number of sampling events and sample containers. A sample list is provided in Appendix B, while sampling events and analyses are included on the Integrated Test A Sampling Event Summary, Appendix C. For a more detailed time line of Integrated Test A, including sequential descriptions of testing problems, refer to Table E.1 in Appendix E, and/or the completed Test Instruction for Integrated Test A. Appendix J contains the Integrated Test A Operational Process Sheets that summarize the target parameters against the achieved test parameters. Values listed in Section 4 are generally not NQA-1 compliant and are for information only, as these values were taken off the HMI and were recorded in the Test Instruction and LRBs. NQA-1 data can be obtained from the DAS files provided separately on transportable hard drives, from the figures in this section, and the figures in Appendices F and G, which were plotted using NQA-1 compliant DAS data. Backpulsing, air entrainment issues, simulant degassing exercises, and level control will be discussed in Section 6.

4.1 Batch Preparation

The Integrated Test A started on 1/31/2009 at approximately 04:30, and most process steps were completed approximately 21 days later on 2/21/09 at 02:27. The High-Solids Filter Test and the repeat of the Filter-Loop Bypass Test were conducted 1 month later starting on 3/20/09 at 21:10 and ending on 3/21/09 at 17:14. The break was a result of troubleshooting problems with entrained air impacting the filter-loop flow rate. Figure 4.1 illustrates the start and completion times, duration, and schedule for Integrated Test A process steps.

PEP process testing was performed with a nonradioactive aqueous slurry of simulant waste solids and liquids that were stored in HLP-VSL-T22. Vessel HLP-VSL-T22 contained approximately 3000 gallons (76-in. tank elevation) of simulant at the start of the test. HLP-AGIT-T22 mixed the simulant at an agitator speed of 60 Hz before the transfers to UFP-VSL-T01A/B tanks. Before the first batch transfer, grab samples were collected from HLP-VSL-T22 from the middle and low elevations. However, the first attempt to collect the samples used an aluminum pipe, which partially dissolved (LRB 60108, pg. 110), yet analysis efforts revealed no impact to sampling results. Grab sampling was completed on 1/31/09 at 04:56.

Figure 4.1. Integrated Test A Schedule of Completion

Six UFP-VSL-T01 batches of simulant were leached, as prescribed by the run sheet in TI-WTP-PEP-065; three batches were processed in UFP-VSL-T01A, and three batches were processed in UFP-VSL-T01B. Simulant was transferred from HLP-VSL-T22 to UFP-VSL-T01A (three times; batches 1,3,5) and B (three times; batches 2,4,6), concurrent with in-line caustic addition; however, a fraction of the caustic (20%) was added to the top of the UFP-VSL-T01B batches. Caustic addition was split, in-line and to the top of the vessel, for UFP-VSL-T01B batches to demonstrate a proposed caustic control strategy that was based on slurry samples collected from UFP-VSL-T00001A/B. Integrated Test A caustic leaching operations demonstrated that if there was insufficient mixing in HLP-VSL-T22, causing an unknown aluminum solids inventory in UFP-VSL-T01B, operations could perform in-line caustic addition, then take samples from the UFP-VSL-T01B vessel to determine how much more caustic was needed for caustic leaching by adding it to the top of the vessel. Refer to Sections 9 and 14 in report WTP-RPT-197 for results on caustic additions made in-line versus on top of the vessel. Refer to Table 4.1 for transfer times for each of the six batches and Table 4.2 for the transfer volumes of simulant and 17.9-M caustic. Batches 1 and 2 transfers included \sim 1872 to \sim 1877 kg of simulant and \sim 637 kg of caustic, whereas the remaining batches had from \sim 1469 to \sim 1662 kg of simulant and \sim 621 to \sim 628 kg of caustic. AFA (1000-mL) was added after PJMs were turned on during the transfers. PJM parameters were set to previously tuned parameters, and adjustments were made to achieve the target run parameter nozzle velocity of 4.8 m/s and a PJM stroke length of 80%. Refer to Section 5.3 for actual PJM peak average nozzle velocities and stroke lengths.

			PJMs Started			
Batch ID	Time Start	Time End	(Inches) ^(b)	Transfer Time		
Batch 1: HLP-VSL-T22 to	1/31/09, 11:19	1/31/09, 11:59	$40+$	\sim 45 min		
UFP-VSL-T01A						
Batch 2: HLP-VSL-T22 to	1/31/09, 15:47	$1/31/09$, 17:48		\sim 115 min		
UFP-VSL-T01B						
Batch 3: HLP-VSL-T22 to	$2/2/09$, 19:49	$2/2/09$, $20:20$	30	\sim 30 min		
UFP-VSL-T01A						
Batch 4: HLP-VSL-T22 to	2/4/09, 14:41	$2/4/09$, 15:44	53	~ 60 min		
UFP-VSL-T01B						
Batch 5: HLP-VSL-T22 to	$2/7/09$, 10:56	2/7/09, 11:29	42	\sim 30 min		
UFP-VSL-T01A						
Batch 6: HLP-VSL-T22 to	$2/10/09$, 04:00	$2/10/09$, 06:00		\sim 115 min		
UFP-VSL-T01B						
(a) Data presented are based on HMI data.						
(b) Note: Target Level to Start PJMs was 25 inches.						

Table 4.1. Integrated Test A UFP Batch Transfer Times^(a)

Temperature change was noted when caustic was added to the top of UFP-VSL-T01B. For Batch 6, the UFP-VSL-T01B temperature after caustic addition to the top of the vessel reached ~65°C; therefore, with approval, the vessel contents were cooled using the heat exchanger to meet the initial heatup temperature target.

Batch					
No.	Vessel	Operation	Target Value	Units	Actual Value
$\mathbf{1}$	UFP-VSL-T01A	Total Transfer Volume	501	gal	501.3
$\mathbf{1}$	UFP-VSL-T01A	In-line $17.9-M$ Caustic Addition	639	kg	639
$\overline{2}$	UFP-VSL-T01B	Simulant Transfer Volume	479	Gal	--
$\overline{2}$	UFP-VSL-T01B	17.9-M Caustic Addition	511	kg	511
$\overline{2}$	UFP-VSL-T01B	17.9-M Caustic Addition to the top of the tank	128	kg	128.2
$\overline{3}$	UFP-VSL-T01A	Total Transfer Volume	426	gal	426
$\overline{3}$	UFP-VSL-T01A	In-line 17.9-M Caustic Addition	629	kg	629
$\overline{4}$	UFP-VSL-T01B	Simulant Transfer Volume	393	Gal	--
$\overline{4}$	UFP-VSL-T01B	17.9-M Caustic Addition	504	kg	--
$\overline{4}$	UFP-VSL-T01B	17.9-M Caustic Addition to the top of the tank	126	kg	126
5	UFP-VSL-T01A	Total Transfer Volume	415	gal	415
5	UFP-VSL-T01A	In-line $17.9-M$ Caustic Addition	625	kg	625
6	UFP-VSL-T01B	Simulant Transfer Volume	393	Gal	393
6	UFP-VSL-T01B	17.9-M Caustic Addition	499	kg	499
6	UFP-VSL-T01B (a) Data presented are based on HMI data.	17.9-M Caustic Addition to the top of the tank	125	kg	--

Table 4.2. Transfer Volumes Integrated Test A Batches^(a)

4.2 Batch Heatup and Leach

For each of the six batches, heating was conducted in two phases. Initial heating to a target temperature of 57ºC (within 3 hours) used heat exchangers UFP-HX-T04A and B; heat exchangers UFP-HX-T05A and B were by-passed. Final heating to the temperature of 98° C (the target heat-up duration was 3.8 hours) was reached with direct steam injection.

The PJM air supply lines had drain lines that diverted condensate formed in the air lines. PJMs switched from vacuum-fill to vent-fill upon reaching 60°C, and PJM drain valves on the drain lines were opened when tank temperatures exceeded 65° C to avoid adding too much condensate to the vessel. At 90C, the ring air purge was adjusted to meet high-temperature flow-rate settings. PJMs were tuned to run sheet nozzle velocity targets when the final leaching temperature of 98°C was reached. The target run parameter nozzle velocity was 4.8 m/s, and the PJM stroke length was 80% (refer to Section 5.3 for actual PJM peak average nozzle velocities and stroke lengths). Problems were noted with tuning the PJMs at this temperature (Batch 1). Increasing the drive pressure at the leach temperature would not produce higher PJM peak average nozzle velocities, as reported on the test instruction (TI). Generally, it was difficult to tune UFP-VSL-T01A/B PJMs at 98°C because level readings from the Drexelbrooks and lasers were not reliable at high temperatures.

The total leach-time target was 16 hours. During hours 1 through 15, Batches 1 through 6 had 2.9-L of IW added to the UFP-VSL-T01A/B vessels hourly to compensate for the low condensation accumulation rate. Table 4.3 summarizes heatup, leach times, and durations for each of the six batches.

				Caustic	Caustic	
	Heat Start	Heat End	Heat	Leach Start	Leach End	Duration
Batch $#$	Time	Time	Duration	Time	Time	(hr)
Batch 1	$1/31/09$,	$1/31/09$,		$1/31/09$,	$2/1/09$,	
UFP-VSL-T01A	14:40	19:40	5.65	19:40	11:40	16.55
Batch 2	$1/31/09$,	$2/1/09$,	5.22	$2/1/09$,	$2/1/09$,	16.35
UFP-VSL-T01B	22:07	03:20		03:20	19:59	
Batch 3	$2/2/09$,	$2/3/09$,	5.20	$2/3/09$,	$2/3/09$,	
UFP-VSL-T01A	21:33	02:45		02:45	18:45	16.00
Batch 4	$2/4/09$,	$2/4/09$,		$2/4/09$,	$2/5/09$,	
UFP-VSL-T01B	17:43	22:38	5.17	22:38	14:38	16.00
Batch 5	$2/7/09$,	$2/7/09$,		$2/7/09$,	$2/8/09$,	
UFP-VSL-T01A	12:41	17:28	4.78	17:28	09:29	16.02
Batch 6	$2/10/09$,	$2/10/09$,		$2/10/09$,	$2/11/09$,	
UFP-VSL-T01B	06:00	12:06	6.10	12:06	04:05	15.98
(a) Data presented are based on HMI data.						

Table 4.3. Start and Stop Times to Heat, and Caustic Leach Integrated Test A Batches^(a)

For the most part, the six batches were heated according to the Test Plan. However, as noted previously, Batch 6 UFP-VSL-T01B exceeded the initial heat-up temperature because of the higher-than-expected caustic addition rate; therefore, with Test Director guidance, the vessel contents were cooled using the heat exchanger to meet the initial heatup temperature run sheet target. On 1/31/09, 47 minutes of steam injection was inadvertently used to do the initial heating for UFP-VSL-T01B Batch 2 instead of using heat exchanger UFP-HX-T04B. Approximately 0.3 inches (per laser) of condensate was added during this time to UFP-VSL-T01B based on stable level measurements. Problems were noted with the automatic temperature controller during caustic leaching. While in automatic temperature control, leach temperatures reached 101.5° C (13:30, 2/1/09, LRB 60229, pg. 32) for UFP-VSL-T01B Batch 2, 103.8°C (17:37, 2/7/09, LRB 60229, pg. 150) for UFP-VSL-T01A Batch 5, and 103°C (19:35,

2/10/09, LRB 60230, pg. 21) for UFP-VSL-T01B Batch 6. The high temperature during UFP-VSL-T01B Batch 2 leaching may be due to a high-high level alarm that occurred ~30 minutes earlier that turned off the steam supply in UFP-VSL-T01B. The steam supply was turned back on immediately. However, the UFP-VSL-T01B PJMs were off since 13:05, 2/1/09; PJMs in UFP-VSL-T01B were turned on at 13:31, 2/1/09 (LRB 60229, pg. 33). With the UFP-VSL-T01B PJMs off, the prototypic RTD, located in the bottom region of Tank T01B, did not contact the steam heated slurry in the upper region of Tank T01B. Consequently, steam supply continued based on the cool temperature reading from the UFP-VSL-T01B prototypic RTD. Operators realized the caustic leach temperature target had been exceeded only when the UFP-VSL-T01B PJMs were turned back on and the slurry was mixed. The high temperature during UFP-VSL-T01A Batch 5 leaching may be due to the UFP-VSL-T01A PJMs locking up; no mixing was taking place (17:36, 2/7/2009, LRB 60229, pg. 150). UFP-VSL-T01A PJMs were turned back on at 17:40, and tank temperature starting decreasing. The cause for the high temperature during Batch 6 leaching is unclear since UFP-VSL-T01B PJMs were on. In addition, the controller would occasionally switch from auto to manual without operator action, resulting in unexpected temperature drops as low as 94°C (Batch 2). An unexpected shutoff for steam injection for Batch 4 dropped the leach temperature to 95.6°C.

See Figure 4.2 through Figure 4.7 for batch heating and cooling temperature profiles.

4.2.1 Cooling

Six batches of simulant were leached and cooled between 02/01/09, 11:57, and 2/11/09, 07:10. Each of the batches was cooled to 60ºC by recirculating tank contents through heat exchangers UFP-HX-T05A and B (aligned and bypassed as necessary to reach cooling curve targets); heat exchangers UFP-HX-T04A and B were bypassed. The exception was Batch 2. With approval, post-caustic-leach cooling in UFP-VSL-T01B to filtration temperature was accomplished by slowing the speed of pump UFP-PMP-T41B to 1.2 GPM instead of aligning and by-passing UFP-HX-T05B. The pump UFP-PMP-T41B speed was increased periodically to flush settled solids out of the recirculation line.

PJMs switched from vent-fill to vacuum-fill upon dropping to 60° C, and the steam-ring air purge was adjusted to meet standard flow-rate settings. Refer to Table 4.4 for batch cool-down start and stop times.

Partially cool-down	Start Time	End Time	Duration (hr)		
Batch 1 UFP-VSL-T01A	$2/1/09$, 11:57	2/1/09, 14:59	3.03		
Batch 2 UFP-VSL-T01B	$2/1/09$, $20:15$	$2/1/09$, $22:56$	2.68		
Batch 3 UFP-VSL-T01A	$2/3/09$, 19:10	2/3/09, 21:55	3.00		
Batch 4 UFP-VSL-T01B	2/5/09, 14:45	2/5/09, 17:30	2.80		
Batch 5 UFP-VSL-T01A	2/8/09, 09:29	2/8/09, 12:33	3.07		
Batch 6 UFP-VSL-T01B	$2/11/09$, 04:25	2/11/09, 07:10	2.72		
(a) Data presented are based on HMI data.					

Table 4.4. Start/Stop Times to Partially Cool Integrated Test A Batches^(a)

Figure 4.2. UFP-VSL-T01A Caustic Leach Batch 1 Heating and Partial Cooling

Figure 4.3. UFP-VSL-T01B Caustic Leach Batch 2 Heating and Partial Cooling

Figure 4.4. UFP-VSL-T01A Caustic Leach Batch 3 Heating and Partial Cooling

Figure 4.5. UFP-VSL-T01B Caustic Leach Batch 4 Heating and Partial Cooling

Figure 4.6. UFP-VSL-T01A Caustic Leach Batch 5 Heating and Partial Cooling

Figure 4.7. UFP-VSL-T01B Caustic Leach Batch 6 Heating and Partial Cooling

CD sampling during leaching was interrupted with CD sampling station pump problems. Consequently, the CD sampling station vacuum system was used to collect samples, or affected samples were delayed and collected during the next sample collection time (refer to Appendix E for test deviations). Other problems during leaching noted were the UFP-VSL-T01A/B PJMs overblowing. The level probes did not operate properly at various times during the operation, but specifically during or after heating. PJM drive times and drive pressures were frequently adjusted to stop the PJM overblows.

The laser and bubbler levels were used to monitor UFP-VSL-T01A/B tank levels during heating and caustic leaching and to monitor the UFP-VSL-T02A tank level during the automatic transfers from the leach tanks. Drexelbrooks in both UFP-VSL-T01A and UFP-VSL-T02A vessels were not working reliably for much of the test. Refer to Section 6.3 for level control and monitoring issues.

4.3 Post-Caustic-Leach Solids Concentration

Six leached batches were concentrated between 2/2/09, 01:34, and 2/14/09, 14:06. During solids concentration, caustic-leached feed was supplied by UFP-VSL-T01A and UFP-VSL-T01B alternately until a prototypic batch at 17-wt% UDS was achieved in UFP-VSL-T02A. This is to say that while one upfront UFP vessel was leaching, the other upfront UFP vessel was transferring batches to UFP-VSL-T02A.

The filters were cleaned during Shakedown/Functional Testing efforts; refer to WTP-RPT-190 for a description of filter cleaning exercises. Before the first transfer, ultrafilters were drained just before the concentration of solids was started; the shell side was not drained. The filter-loop draining was completed at 20:40, 2/1/09. Then the filter loop was filled with simulant to bring the level down in

UFP-VSL-T02A to operate and tune the PJMs to target run sheet parameters. The target run parameter nozzle velocity was 7.3 m/s, and the PJM stroke length was 80%. Refer to Section 5.3 for actual PJM peak average nozzle velocity and stroke length.

Solids were concentrated in UFP-VSL-T02A using one filter bundle, UFP-FILT-T01A. The filter-loop configuration had heat exchanger UFP-HX-T02A aligned, and filters UFP-FILT-T02A through -T05A and heat exchanger UFP-HX-T03A were bypassed. When the contents of UFP-VSL-T02A reached 44.7 inches, one 11-gallon make-up addition from the feed preparation vessels (UFP-VSL-T01A/B) to the feed vessel (UFP-VSL-T02A) was automatically triggered by the program logic controller (PLC). Solids concentration was completed for each batch when the feed tank (UFP-VSL-T01A/B) had been emptied to the heel. The initial batch was transferred to UFP-VSL-T02A on 2/1/09, 16:34. The solids concentration to 17-wt% UDS was completed at 14:06 on 2/14/09. The concentration lasted 300 hours.

Slurry was transferred from UFP-VSL-T01A/B to UFP-VSL-T02A as needed to maintain a slurry level of 44.7 inches in UFP-VSL-T02A. The laser level was the preferred method for measuring level; however, the bubbler was also used when laser performance became unstable. Vessel recirculation valves V03125 and V04125, for UFP-VSL-T01A and B, respectively, were adjusted to obtain different flow rates for recirculation and batch transfers to UFP-VSL-T02A. During concentration, the temperature setpoint for heat exchanger UFP-HX-T02A was adjusted periodically to maintain the slurry at the target temperature of 25°C.

Permeate was routed to UFP-VSL-T62B during solids concentration. Permeate samples were collected between the UFP-FILT-T01A filter bundle and the pulse pot at three evenly spaced times and inspected for particle breakthrough. No observations of particle breakthrough were noted.

Post-caustic-leach concentration filtering rates continued to decrease over time with the Filter T01A axial pressure drop increasing gradually. At 18:20, 2/4/09, the Test Director emailed that backpulsing would be applied through the course of filtration to improve permeate rates. Permeate flow rate after backpulsing was the same for all backpulse variations. See the backpulse description in Section 6.1. Figure 4.8 through Figure 4.35 show the filtration periods for each of the batches – where the prototypic UFP-VSL-T02A temperature was taken from TTK-0619. Flow meters upstream from Pump T42A and downstream from Pump T43A measure in gallons per minute; however axial velocity is calculated from the volume flow rate - where 15 ft/s correlates to 109 gallons per minute. The equation for TMP (average of inlet and outlet filter bundle pressure minus the shell side pressure) uses the inlet and outlet pressures for each filter bundle, where the outlet pressure of one filter bundle serves as the inlet pressure of the subsequent filter bundle. Because of this arrangement, TMP values for UFP-FILT-T02A and UFP-FILT-T05A are shown on Figures 4.8 through 4.35 even though only UFP-FILT-T01A was aligned. Refer to Section 8, Filtration, of report WTP-RPT-197 for TMP and temperature-corrected filter flux versus wt% solids plots, along with permeate production rates.

Figure 4.8. Axial Velocity Based on Flow Measurements at the Suction to Pump T42A and the Discharge to Pump T43A for Post-Caustic-Leach Concentration, Batch #1; Valve SV-0609 Setting (for information only) Scale 0 to 2 Correlates to 0% and 100% Open, Respectively

Figure 4.9. TMP for Post-Caustic-Leach Concentration, Batch #1

Figure 4.10. Tank T02A (TTK-0619) Temperature for Post-Caustic-Leach Concentration, Batch #1

Figure 4.11. Permeate Production Rate for Post-Caustic-Leach Concentration, Batch #1

Figure 4.12. Axial Velocity Based on Flow Measurements at the Suction to Pump T42A and the Discharge to Pump T43A for Post-Caustic-Leach Concentration, Batch #2; Valve SV-0609 Setting (for information only) Scale 0 to 2 Correlates to 0% and 100% Open, Respectively

Figure 4.13. TMP for Post-Caustic-Leach Concentration, Batch #2

Figure 4.14. Tank T02A (TTK-0619) Temperature for Post-Caustic-Leach Concentration, Batch #2

Figure 4.15. Permeate Production Rate for Post-Caustic-Leach Concentration, Batch #2

Figure 4.16. Axial Velocity Based on Flow Measurements At the Suction to Pump T42A and the Discharge to Pump T43A for Post-Caustic-Leach Concentration, Batch #3; Valve SV-0609 Setting (for information only) Scale 0 to 2 Correlates to 0% and 100% Open, Respectively

Figure 4.17. TMP for Post-Caustic-Leach Concentration, Batch #3

Figure 4.18. Tank T02A (TTK-0619) Temperature for Post-Caustic-Leach Concentration, Batch #3

Figure 4.19. Permeate Production Rate for Post-Caustic-Leach Concentration, Batch #3

Figure 4.20 shows adjustments made to the filter-loop backpressure valve and pump speeds in response to pump vibration and permeate rate cycling from 10:00 to 21:00 on 2/8/09.

Figure 4.20. Axial Velocity Based on Flow Measurements at the Suction to Pump T42A and the Discharge to Pump T43A for Post-Caustic-Leach Concentration, Batch #4; Valve SV-0609 Setting (for information only) Scale 0 to 2 Correlates to 0% and 100% Open, Respectively

Figure 4.21. TMP for Post-Caustic-Leach Concentration, Batch #4

Figure 4.22. Tank T02A (TTK-0619) Temperature for Post-Caustic-Leach Concentration, Batch #4

Figure 4.23. Permeate Production Rate for Post-Caustic-Leach Concentration, Batch #4

Figure 4.24. Axial Velocity Based on Flow Measurements at the Suction to Pump T42A and the Discharge to Pump T43A for Post-Caustic-Leach Concentration, Batch #5; Valve SV-0609 Setting (for information only) Scale 0 to 2 Correlates to 0% and 100% Open, Respectively

Figure 4.25. TMP for Post-Caustic-Leach Concentration, Batch #5

Figure 4.26. Tank T02A (TTK-0619) Temperature for Post-Caustic-Leach Concentration, Batch #5

Figure 4.27. Permeate Production Rate for Post-Caustic-Leach Concentration, Batch #5

Figure 4.28. Axial Velocity Based on Flow Measurements at the Suction to Pump T42A and the Discharge to Pump T43A for Post-Caustic-Leach Concentration, Batch #6; Valve SV-0609 Setting (for information only) Scale 0 to 2 Correlates to 0% and 100% Open, Respectively

Figure 4.29. TMP for Post-Caustic-Leach Concentration, Batch #6

Figure 4.30. Tank T02A (TTK-0619) Temperature for Post-Caustic-Leach Concentration, Batch #6

Figure 4.31. Permeate Production Rate for Post-Caustic-Leach Concentration, Batch #6
Integrated Test A—Post-Caustic-Leach Concentration, All Batches

Figure 4.32. Axial Velocity Based on Flow Measurements at the Suction to Pump T42A and the Discharge to Pump T43A for Post-Caustic-Leach Concentration, All Batches; Valve SV-0609 Setting (for information only) Scale 0 to 2 Correlates to 0% and 100% Open, Respectively

Figure 4.33. TMP for Post-Caustic-Leach Concentration, All Batches

Figure 4.34. Tank T02A (TTK-0619) Temperature for Post-Caustic-Leach Concentration, All Batches

Figure 4.35. Permeate Production Rate for Post-Caustic-Leach Concentration, All Batches

Even though filters UFP-FILT-T02A through -05A were bypassed during post-caustic-leach concentration, a drip leak from filters UFP-FILT-T02A and -T04A permeate lines to pulse pot UFP-PP-T02A was observed on 2/4/09 (LRB 60229, pg. 94 at 08:15). Apparently, the pulse pot UFP-PP-T02A manual isolation (VV70164) and the automatic valves were not isolating the air pressure source. An additional manual isolation valve from the air line was closed (VV70162) (LRB 60230, pg. 43). Permeate line sample gauges were left aligned to monitor line pressure for the remainder of the test.

At 13:26, $2/8/09$ (LRB 60229, pg. 161), the permeate rate was cycling every \sim 25 minutes (LRB 60108, pg. 158). By 16:20, pump vibration was heard (LRB 60229, pg. 163) while UFP-VSL-T02A was at the trigger batch level of ~45 inches. Pump speed and backpressure (valve SV-0609) adjustments were made up until 21:00, 2/8/09, to alleviate pump vibration. By 21:02, the permeate rate was back to "normal" (LRB 60229, pg. 167). Figure 4.36 through Figure 4.38 show permeate, TMP, the filter-loop flow rate, and backpressure valve settings during the cycling. Refer to Section 6.2 for a discussion of filter-loop operation issues. Figure 4.38 shows the backpressure valve SV-0609 setting adjustments made during the permeate rate cycling in efforts to maintain the target filter-loop flow rate of 109 gpm.

Figure 4.36. Permeate Production Rate During a Select Time Period in Integrated Test A

Figure 4.37. Filter TMP During a Select Time Period in Integrated Test A

Figure 4.38. Slurry Flow Rate and Valve SV-0609 Setting During a Select Time Period in Integrated Test A; Valve SV-0609 Setting (for information only) Scale 0 to 2 Correlates to 0% and 100% Open, Respectively

Spargers maintained target flow rates during solids concentration with periodic cleaning. At 05:00, 2/14/09 (LRB 60230, pg. 45), brown fluid was seen in the three steam-ring air purge rotameters, mostly in the UFP-VSL-T02A rotameter. The brown substance could have been simulant drawn into the sparger lines. Refer to Appendix J for more sparger operation information.

Occasionally, there were PJM overblow observations in UFP-VSL-T02A during solids concentration. Potential overblowing in UFP-VSL-T02A may have caused the recirculation pump vibration and unstable filter-loop flow rates caused by entrained air and indicated by a faulty flow meter located downstream of recirculation pump UFP-PMP-T43A (NCR 42317.1). Refer to Section 6.2 for a discussion of slurry air entrainment.

The 300 hours of post-caustic-leach concentration produced an 18-wt% UDS—the target was 17-wt% UDS. Refer to Section 5 for sample concentration results through the course of post-caustic-leach concentration in UFP-VSL-T02A. For a discussion on the dissolution of aluminum solids for Integrated Test A caustic-leach batches, refer to report WTP-RPT-197.

4.4 Tracer Test

The test to measure the bypass of the filter-loop return nozzle jet in UFP-VSL-T02A was conducted with a chemical tracer when solids concentration steps were completed on $2/14/09$. After aligning the five filter bundles in preparation for the tracer test, the UFP-VSL-T02A level dropped to 47.1 inches, per the laser. With approval from the Test Director, 30 gallons of IW were added to compensate for the greater-than-expected tank level decrease. The PJMs were tuned to the target peak average velocity of 12 m/s and a stroke length of 80%. See Section 5.3 for actual PJM peak average nozzle velocity and stroke length.

On 2/14/09 at 21:26, the CsBr tracer was injected into the suction side of the recirculation pump UFP-PMP-T42A, and the tracer concentration in the loop and UFP-VSL-T02A was monitored via sample collections. The filter-loop flow rate was maintained at 90 ± 5 gpm, as opposed to the run sheet target of 109 ± 10 gpm to minimize unstable filter loop pump(s) performance.

Since the target filter-loop flow rate was not achieved during the tracer test above, the tracer test was repeated after the High Solids Filter Test on 3/21/09. The steam-ring air purge and upper and lower air spargers were kept off during the repeat tracer test. The tracer test results suggest that the slurry was nearly completely mixed between 2 and 4 minutes, and overall, there was no bypass of the filter-loop return nozzle. Tracer test results are reported in the washing report, WTP-RPT-187.

4.5 Post-Caustic-Leach Washing

The post-caustic-leach slurry wash, using IW (0.01-M NaOH), started on 2/15/09 at 01:13 and ended at 22:55. The purpose of the post-caustic-leach slurry wash was to remove soluble solids. There were 100 IW transfers with 41.6 kg per transfer. The target level in UFP-VSL-T02A to initiate wash water transfers was 44.5 inches. The wash was conducted using five ultrafilter bundles (all five filters aligned and heat exchanger UFP-HX-T02A aligned, but UFP-HX-T03A heat exchanger bypassed) with permeate routed to UFP-VSL-T62B. AFA was added after every 3rd batch transfer to maintain a targeted AFA

concentration of 350 ppm. However, filter-loop flow rate problems, caused by air entrainment (and consequently unstable UFP-PMP-T42A and T43A pump performance), were observed through the course of the post-caustic-leach concentration washing.

After the $12th$ CD sample event (wash #56), per approval, the lower air spargers were turned off temporarily, and the steam-ring air purge rate was set to 0.07 kg/min temporarily to improve the filter-loop flow rate. The steam ring air and lower and upper air spargers were turned off from 14:26 to 19:32 on 2/15/09 to minimize pump vibration. Refer to Figure 4.39 through Figure 4.42 for filtration activities during the post-caustic-leach concentration washing – where the prototypic UFP-VSL-T02A temperature was taken from TTK-0619. The number of IW batches required to reach the target of 0.25-M free hydroxide following the aluminum leaching stage was 64 batches for Integrated Test A, compared to the WTP projections to reach the target of 0.25-M free hydroxide of 64 batches. Post-caustic-leach washing efficiency is summarized in report WTP-RPT-187, however, an overall wash efficiency for Integrated Test A was 1.00. In addition, Section 8, Filtration, of report WTP-RPT-197 provides TMP and temperature-corrected filter flux versus wt% solids plots—along with permeate production rates.

Figure 4.39. Axial Velocity Based on Flow Measurements at the Suction to Pump T42A and the Discharge to Pump T43A for Post-Caustic-Leach Slurry Wash; Valve SV-0609 Setting (for information only) Scale 0 to 2 Correlates to 0% and 100% Open, Respectively

Figure 4.40. TMP for Post-Caustic-Leach Slurry Wash

Figure 4.41. Tank T02A (TTK-0619) Temperature for Post-Caustic-Leach Slurry Wash

Figure 4.42. Permeate Production Rate for Post-Caustic-Leach Slurry Wash

4.6 Oxidative Leaching

Chromium-bearing slurry and AFA were added to the washed slurry before the sodium permanganate on 2/16/09. Chromium addition, although nonprototypic, was necessary because chromium in the simulant was found to oxidize to the $Cr⁶⁺$ state during the caustic leach.

Chromium slurry addition, 145 gallons, was started at 01:12 and ended at 01:23. Chromium solids were added (as slurry) in-line at the suction of recirculation pump UFP-PMP-T42A. The PJM nozzle velocity during this time was ~13.6 m/s, the steam ring air was off, and the upper and lower air spargers were set at 0.01 and 0.04 kg/min, respectively. See Table 4.5 for the chromium oxyhydroxide slurry composition; however, only Batch 1 was used for Integrated Test A.

Batch		CrOOH Test Batch 0				CrOOH Test Batch 1					CrOOH Final Batch	
Name		Cr-080623-250-SL				Cr-080729-2000-SL					Cr-080917-Final-SL	
Sample ID		Total Slurry Fraction			Total Slurry Fraction			Liquid Fraction Only			Total Slurry Fraction	
Parameter	μ g/mL	Mol/L	$\frac{0}{0}$	μ g/mL	Mol/L	$\frac{0}{0}$	μ g/mL	Mol/L	$\frac{0}{0}$	μ g/mL	Mol/L	$\frac{0}{0}$
Al	<3.5	0.0001	$< 0.0002\%$	< 7.1	< 0.0003	$< 0.0006\%$	1.40	0.0001	0.0001%	0.77	0.00003	0.0001%
$\, {\bf B}$	14	0.0013	0.0010%	4.15	0.0004	0.0003%	1.80	0.0002	0.0002%	3.3	0.0003	0.0003%
Ba	0.9	0.0000	0.0001%	92.1	0.0007	0.0077%	0.44	0.0000	0.0000%	13.4	0.0001	0.0011%
Bi	<17.6	0.0001	$< 0.0012\%$	<3.5	< 0.0000	$< 0.0003\%$	\leq 3	< 0.0000	$< 0.0003\%$	< 1.5	< 0.0000	$< 0.000\%$
Ca	44	0.0011	0.0030%	12.5	0.0003	0.0010%	< 1.2	< 0.0000	$< 0.0001\%$	19.1	0.0005	0.0016%
Cd	< 1.2	0.0000	$< 0.0001\%$	0.74	0.0000	0.0001%	0.22	0.0000	0.0000%	0.99	0.0000	0.0001%
Ce	< 5.9	0.0000	$< 0.0004\%$	< 1.2	< 0.0000	$< 0.0001\%$	1.00	0.0000	0.0001%	< 0.5	< 0.0000	$< 0.000\%$
Cr	48,800	0.9385	3.3388%	14,600	0.2808	1.2259%	33.7	0.0006	0.0029%	14,100	0.2712	1.1899%
Fe	23.3	0.0004	0.0016%	5.58	0.0001	0.0005%	0.37	0.0000	0.0000%	7.0	0.0001	0.0006%
K	270	0.0069	0.0185%	186	0.0047	0.0156%	89.8	0.0023	0.0077%	419	0.0107	0.0354%
La	2.3	0.0000	0.0002%	0.56	0.0000	0.0000%	< 0.28	< 0.0000	$< 0.0000\%$	0.94	0.0000	0.0001%
Mg	< 1.4	0.0001	$< 0.0001\%$	0.45	0.0000	0.0000%	< 0.23	< 0.0000	$< 0.0000\%$	0.72	0.0000	0.0001%
Mn	0.85	0.0000	0.0001%	0.33	0.0000	0.0000%	0.060	0.0000	0.0000%	0.23	0.0000	0.0000%
Na	322,000	14.0062	22.031%	90,550	3.9387	7.6029%	99,100	4.3106	8.4701%	$<$ 99,100	< 4.311	$< 8.363\%$
Nd	< 11.8	0.0001	$< 0.0008\%$	< 2.4	< 0.0000	$< 0.0002\%$	$<$ 2	< 0.0000	$< 0.0002\%$	< 1.0	< 0.0000	$< 0.000\%$
Ni	10	0.0002	0.0007%	0.68	0.0000	0.0001%	< 0.24	< 0.0000	$< 0.0000\%$	1.21	0.0000	0.0001%
${\bf P}$	$<$ 29.4	0.0009	$< 0.0020\%$	< 6.1	< 0.0002	$< 0.0005\%$	$<$ 5	< 0.0002	$< 0.0004\%$	< 2.5	< 0.0001	$< 0.000\%$
Pb	< 18.8	0.0001	$< 0.0013\%$	<3.8	< 0.0000	$< 0.0003\%$	<3.2	< 0.0000	$< 0.0003\%$	1.6	< 0.0000	$< 0.000\%$
${\bf S}$	< 94.1	0.0029	$< 0.0064\%$	22	0.0007	0.0018%	31.0	0.0010	0.0026%	30	0.0009	0.0025%
Si	33.8	0.0012	0.0023%	12.7	0.0005	0.0011%	10.5	0.0004	0.0009%	15	0.0005	0.0013%
Sr	0.13	0.0000	0.0000%	168.5	0.0019	0.0141%	0.054	0.0000	0.0000%	30.6	0.0003	0.0026%
Th	90.7	0.0004	0.0062%	27.8	0.0001	0.0023%	< 0.98	< 0.0000	$\leq 0.0001\%$	26.8	0.0001	0.0023%
Zn	11	0.0002	0.0008%	3.60	0.0001	0.0003%	1.60	0.0000	0.0001%	9.43	0.0001	0.0008%
Zr	< 0.6	0.0000	$< 0.0000\%$	< 0.1	< 0.0000	$< 0.0000\%$	< 0.11	< 0.0000	$\leq 0.0000\%$	0.073	0.0000	0.0000%

Table 4.5. Batch 1 Chromium Slurry Composition for Integrated Test A

It was necessary to remove excess liquid introduced by chromium solids slurry with five ultrafilter bundles. After dewatering, additional solids washing was performed to remove the excess hydroxide included in the chromium solids slurry and to achieve a hydroxide concentration of 0.25-M NaOH. The filter-loop flow rate decreased through the course of chromium washing; at 03:56, the filter-loop flow rate was 85 gpm (103 gpm at 02:51 2/16/09 [TI pg. 202]). The run sheet target was 109 ± 10 gpm (TI, pg. 204). The steam ring air and upper and lower spargers were off during post-chromium slurry washing. Leaks in UFP-VSL-T02A PJMs 1807 and 1801 vacuum lines were identified; by 10:00, they were still leaking after tightening the connections, (2/16/09, LRB 60230, pg. 98). At 13:18 on 2/16/09 (LRB 60230, pg. 100), leaking simulant was observed coming out the tops of the PJMs in Tank T02A. This was caused by PJM overdraw when vacuum was applied too long and drew slurry up into the vacuum delivery piping. The process of turning the UFP-VSL-T02A PJMs off and then restarting them one by one was initiated, and the PJM parameter adjustments were made as necessary. Refer to Figure 4.43 through Figure 4.46 for filtration activities during the reconcentration and washing of the slurry after chromium addition—where the prototypic UFP-VSL-T02A temperature was taken from TTK-0619.

Figure 4.43. Axial Velocity Based on Flow Measurements at the Suction to Pump T42A and the Discharge to Pump T43A for the Reconcentration and Wash Operations Following Chromium Slurry Addition; Valve SV-0609 Setting (for information only) Scale 0 to 2 Correlates to 0% and 100% Open, Respectively

Figure 4.44. TMP for the Reconcentration and Wash Operations Following Chromium Slurry Addition

Figure 4.45. Tank T02A (TTK-0619) Temperature for the Reconcentration and Wash Operations Following Chromium Slurry Addition

Figure 4.46. Permeate Production Rate for the Reconcentration and Wash Operations Following Chromium Slurry Addition

Permanganate additions started on 2/16/09 at 10:13 and ended at 10:21. The 1-M sodium permanganate addition, for a total of 37.2 gallons, was made to the suction side of pump UFP-PMP-T42A. Filter-loop flow rates increased (with no adjustment of the operating conditions) to the target flow rate during the sodium permanganate transfer, but declined soon after the transfer was complete.

Oxidative leaching started on 2/16/09 at 10:21 and ended at 16:23. The target filter-loop flow rate was 109 gpm; however, only 85 gpm maximum was achieved momentarily because of air entrainment. The steam-ring air purge and upper air spargers were kept off, per guidance, as opposed to the original run sheet targets of 0.14 and 0.01, respectively, in efforts to minimize entrained air in the filter loop. Although the lower air spargers were also supposed to be off to minimize entrained air in the filter loop, they were kept on at a rate of ~ 0.04 kg/min. The target temperature of 25 \degree C was maintained during the 6 hours of oxidative leaching; refer to Appendix G, "T02A temperatures" plots for specified times. The PJM nozzle velocity during oxidative leaching was \sim 12.1 m/s. Refer to Section 5.3 for the PJM peak average velocity summary.

4.7 Post-Oxidative Washing

Post-oxidative-leach washing followed 6 hours of oxidative leaching. There were a total of 86 IW additions of 41.6 kg each. The steam-ring air purge and the upper and lower air spargers were kept off, per guidance, as opposed to the original run sheet targets of 0.14, 0.01, and 0.04 kg/min, respectively. Refer to Figure 4.47 through Figure 4.50 for filtration activities during post-oxidative-leach washing--where the prototypic UFP-VSL-T02A temperature was taken from TTK-0619. Refer to

Section 8, Filtration, of report WTP-RPT-197 for TMP and temperature-corrected filter flux versus wt% solids plots—along with permeate production rates.

Figure 4.47. Axial Velocity Based on Flow Measurements at the Suction to Pump T42A and the Discharge to Pump T43A for Post-Oxidative-Leach Slurry Wash; Valve SV-0609 Setting (for information only) Scale 0 to 2 Correlates to 0% and 100% Open, Respectively

Figure 4.48. TMP for Post-Oxidative-Leach Slurry Wash

Figure 4.49. Tank T02A (TTK-0619) Temperature for Post-Oxidative-Leach Slurry Wash

Figure 4.50. Permeate Production Rate for Post-Oxidative-Leach Slurry Wash

Integrated Test A was put on hold after completing the post-oxidative-leach washing per the Test Director email 20:28 on 2/16/09. A list of tasks to perform after completing the wash was provided in the email. See discussion on the test suspension, Section 6.2.1.

4.8 Final Concentration

Integrated Test A was restarted on the late afternoon of 2/20/09. Final concentration to 20-wt% was done using all five filters on 2/20/09 from 21:24 to 21:40. The stable level in UFP-VSL-T02A before concentration was 45.4 (laser). Bubblers were turned off, per guidance (TI pg. 215), for the final concentration. The steam-ring air purge and the upper and lower air spargers were kept off, per guidance (to manage air entrainment), as opposed to the original run sheet targets of 0.14, 0.01, and 0.04 kg/min, respectively. The stable level in UFP-VSL-T02A after concentration was 38.9 inches (laser). In addition, the maximum attainable filter-loop flow rate was 73 gpm upstream from recirculation pump UFP-PMP-T42A (the flow rate on FI-0635, downstream from recirculation pump UFP-PMP-T43A, was higher) as opposed to the 109 ± 10 gpm run sheet target (TI Run Sheet). The flow meter performance was questionable at this time, as stated on the NCR list in Section 2. Refer to Figure 4.51 through Figure 4.54 for filtration activities during the final concentration—where the prototypic UFP-VSL-T02A temperature was taken from TTK-0619.

Figure 4.51. Axial Velocity Based on Flow Measurements at the Suction to Pump T42A and the Discharge to Pump T43A for Post-Oxidative-Leach Slurry Concentration; Valve SV-0609 Setting (for information only) Scale 0 to 2 Correlates to 0% and 100% Open, Respectively

Figure 4.52. TMP for Post-Oxidative-Leach Slurry Concentration

Figure 4.53. Tank T02A (TTK-0619) Temperature for Post-Oxidative-Leach Slurry Concentration

Figure 4.54. Permeate Production Rate for Post-Oxidative-Leach Slurry Concentration

Filter permeate and other flush solutions were transferred to permeate vessels UFP-VSL-T62A/B and/or totes. The High Solids Filter Test should have happened next, but due to the pump issues, it was postponed for a later date.

4.9 Flush Filter Loop

Simulant in the filter loop was drained directly to a tote without adding IW for later use: High Solids Filter Test, per Test Director guidance (TI pg. 225). Approximately 20 inches of simulant was also removed from UFP-VSL-T02A and put into totes to reach 20 inches in the vessel. Before test clean-up, a filter-loop study was performed on 2/20/09, 23:50, to evaluate how low the UFP-VSL-T02A tank level could reach, considering the filter-loop return nozzle location, while maintaining filter-loop flow rate targets. The filter-loop study indicated that the filter-loop flow rate dropped from 100 gpm at the 20-in. tank level to 83 gpm at an 18.3-in. level per upstream flow meter FI-0623 upstream from UFP-PMP-T42A recirculation pump. The laser was noted as not reading below the 17.8-in. vessel level. The PJMs were turned off during the filter-loop study so that the camera quality would not be compromised. The camera showed the simulant return line exposed approximately 2 inches at the 18.3-in. vessel level (TI pg. 224). Then IW was transferred to UFP-VSL-T02A and circulated through the filter loop with recirculation pumps UFP-PMP-T42A and UFP-PMP-T43A to allow a representative in-line sample of the filter-loop contents to be collected.

As stated earlier, slurry from UFP-VSL-T02A and the filter loop was transferred to totes for later use. In efforts to retain as much concentrated slurry as possible from Integrated Test A, the simulant and IW slurry in UFP-VSL-T02A was also transferred into a tote.

4.10 High-Solids Filter Test

The High-Solids Filter Test was to be done after performing the final concentration to 20-wt%, but due to the unstable filter-loop pump operations in previous testing processes, the High-Solids Filter Test was postponed until $3/20/09$, $00:31$, at the completion of Integrated Test B—caustic and oxidative leaching in UFP-VSL-T02A. The objective of the High-Solids Filter Test was to observe the solids concentration versus the filter flux.

Preparation for the High-Solids Filter Test started 3/20/09, 21:10 (LRB 60279, pg. 38), and ended 3/21/09, 00:31. Simulant from Integrated Test A stored in totes was added to UFP-VSL-T02A via transfer from UFP-VSL-T01B where it was mixed with PJM operation, and the steam ring was set to 0.20 kg/min before it was sent to UFP-VSL-T02A to achieve the target solids concentration. The UFP-VSL-T02A stable level at this point was 51.4 inches (laser). UFP-VSL-T02A was then filled with IW via in-line to the UFP-PMP-T42A suction side to reach the target vessel level. The stable level in UFP-VSL-T02A after IW addition was 63.9 inches (laser). A calculated 86-mL of concentrated AFA was added to account for the volume of IW used to reach the tank level target per the run sheet.

The High-Solids Filter Test started on 3/21/09, 01:37, and ended 3/21/09, 06:08. Filters were configured to flow slurry through all five filters, but permeate was withdrawn only through the first filter, Filter T01A. The permeate rate through Filter T01A before backpulsing was 3.2 kg/min. After the permeate valve was opened, Filter T01A was backpulsed consecutively five times as quickly as possible while sending permeate back into UFP-VSL-T02A. Standard backpulse parameters were used: a deadband pressure of 5 psi, an overpressure of 40 psi, and a pulse pot level of initiation of 10 inches. After backpulsing was completed, the permeate rate through Filter T01A was 3.5 kg/min. Slurry in UFP-VSL-T02A was then concentrated using only the first filter bundle; permeate was sent to UFP-VSL-T62B. The steam-ring air purge and upper and lower air spargers were kept off, per guidance, as opposed to the original run sheet targets of 0.14, 0.01, and 0.04 kg/min, respectively. The UFP-VSL-T02A stable level after concentration was 22.8 inches (laser). The High-Solids Filter Test results are reported in the filtration report, WTP-RPT-185. Refer to Figure 4.55 through Figure 4.58 for filtration performance during the High-Solids Filter Test—where the prototypic UFP-VSL-T02A temperature was taken from TTK-0619.

The temperature target for UFP-VSL-T02A was exceeded towards the end of the High-Solids Filter Test. By 05:15, 3/21/09 (LRB 60279, pg. 43), the heat exchanger UFP-HX-T02A, TIC-0513, temperature setpoint was decreased to 20° C because the temperature in the tank was 26.4° C, and the target was $23 \pm 2^{\circ}$ C. By 05:44, 3/21/09 (LRB 60279, pg. 43), the UFP-VSL-T02A heat exchanger temperature setpoint was set to manual operation in response to the increasing temperature in the UFP-VSL-T02A. This may have been influenced by the low level and high pump flow-rate operations. By 05:46, 3/21/09 (LRB 60271, pg. 4), the heat exchanger UFP-HX-T02A valve was set at 60% (open) in response to a vessel temperature of 28.5C. Temperature readings from RTD thermowells were not reliable as indicated on NCR 42402.1.

After completing the High-Solids Filter Test, the cesium tracer test was repeated as noted previously in Section 4.4.

Figure 4.55. Axial Velocity Based on Flow Measurements at the Suction to Pump T42A and the Discharge to Pump T43A for the High-Solids Scaling Test Conducted on 3/21/2009; Valve SV-0609 Setting (for information only) Scale 0 to 2 Correlates to 0% and 100% Open, Respectively

Figure 4.56. TMP for the High-Solids Scaling Test Conducted on 3/21/2009

Figure 4.57. Tank T02A (TTK-0619) Temperature for the High-Solids Scaling Test Conducted on 3/21/2009

Figure 4.58. Permeate Production Rate for the High-Solids Scaling Test Conducted on 3/21/2009

5.0 Analytical Results

Selected data obtained from the DAS during Integrated Test A have been plotted in 12-h intervals, and this is provided in Appendix G. Tables summarizing the results of sample measurements taken during testing are provided in Section 5.1 (analytical results) and Section 5.2 (physical-property results). The analysis of the analytical data is done in the final report, Sections 9, 10, 11, and 12, of the Pretreatment Engineering Platform Phase 1 Final Test Report, WTP-RPT-197. PJM peak average velocity and stroke length, tuned iteratively to target values during testing, are provided in Section 5.3. Data, including figures, in this section are NQA-1 compliant.

5.1 Analytical Data

This section provides analytical summary tables for key processes. Reporting limits and uncertainties are in the final analytical reports submitted by each analytical laboratory. These reports are identified for each sample in Appendix B, which provides a list of all samples collected, the disposition of each sample, and references to final electronic analytical reports.

In many cases, such as during caustic and oxidative leaching, the slurry samples taken from PEP were analyzed after phase separation was performed. The wet centrifuged solids and the supernatant liquid from the samples were analyzed separately. In other cases, data are presented in a more raw form: the species concentration in the supernatant, the concentration in the wet centrifuged solids, and the masses of tare sample vial, vial plus total slurry, and vial plus wet centrifuged solids. The latter three masses can be used to calculate the mass fraction of wet centrifuged solids in the slurry, which in turn can be used in calculating the total species concentration in the original sample slurry. See Section A.5 in Appendix A of WTP-RPT-186, Rev 0, for more information on calculating the species concentration in the slurry.

Table 5.1 through Table 5.6 provide analytical results for samples collected during the Batch 1 through Batch 6 caustic leach in Tank T02A. The slurry density results were performed on intact slurry submitted to the analytical laboratory. The wt% UDS, aluminum, and strontium slurry concentrations were calculated from samples phase-separated at PDL-W. The liquid analysis results are analytical results obtained from the phase-separated supernatants. Raman results, shown in brackets [], are provided as part of the liquid analyses and are presented in molar concentration (mole/liter).

Table 5.7 provides analytical results for samples collected during solids concentration of the caustic-leached slurry in Tank T02A. This concentration combined the leached solids from Batch 1 through 6 as described in Section 4. The wt% UDS and densities were obtained following analytical methods described in Section 3. An aliquot of the wet centrifuged solids was dried at 105°C to determine the percent solids that are reported with the reported analytes in the wet centrifuged solids.

Table 5.8 and Table 5.9 provide the cesium tracer analytical results for samples collected during Integrated Test A tracer tests using post-caustic-leached and concentrated slurry in Tank T02A.

			Slurry Analyses						Liquid Analyses				
							A ₁ $A1(OH)4-$	Free					
	UDS	Slurry Density	AI	Sr	Fe	Liquid Density	by Raman]	OH- [Raman]	C_2O_4 [Raman]	$Cl-$	NO ₃ [Raman]	NO ₂ [Raman]	SO_4 [Raman]
Sample location: Tank T02A, Condition:	Wt%	$g/mL^{(a)}$	μ g/g	μ g/g	μ g/g	$g/mL^{(a)}$	μ g/g [M]	M [M]	μ g/g [M]	μ g/g	μ g/g [M]	μ g/g [M]	μ g/g ſМ
After NaOH addition	2.91	1.362	14468	32.7	3614	1.330	4713 [0.263]	4.914 [5.153]	96.7 $\lceil n/d \rceil$	799	56966 [1.353]	13233 [0.429]	10266 [0.151]
88°C	2.35	1.326				1.312	6970 [0.354]	[4.609]	138 [<0.01]	731	52700 [1.240]	12200 [0.376]	9490 [0.139]
98° C—hour 0	2.38	1.323	13842	30.2	3289	1.308	7220 [0.347]	4.328 [4.522]	172 $\lceil n/d \rceil$	760	52650 [1.216]	11900 [0.360]	9330 [0.137]
98°C--hour 1	2.36	1.324				1.308	6950 [0.348]	$\overbrace{}$ [4.479]	177 [<0.01]	713	51100 [1.194]	12100 [0.372]	9420 [0.134]
98°C-hour 2	2.36	1.324				1.308	7390 [0.352]	$\overline{}$ [4.428]	155 [<0.01]	733	51600 [1.190]	12100 [0.368]	9390 [0.133]
98°C-hour 4	2.37	1.318	13051	30.1	3300	1.305	7270 [0.353]	[4.373]	166 [<0.01]	705	50900 [1.172]	11900 [0.364]	9270 [0.131]
98° C—hour 8	2.20	1.306	13215	29.7	3223	1.296	8050 [0.377]	4.185 [4.302]	249 [<0.01]	731	51000 $[1.163]$	11700 [0.363]	9190 [0.130]
98° C—hour 10	2.15	1.323				1.291	8000 [0.373]	$\overline{}$ [4.070]	187 [<0.01]	692	49400 [1.149]	11500 [0.348]	8990 [0.127]
98°C-hour 12	2.18	1.316	13010	29.3	3214	1.286	8410 [0.380]	3.956 $[4.222]$	251 [<0.01]	721	49400 [1.124]	11600 [0.356]	8910 [0.126]
98° C—hour 14	2.17	1.298				1.291	8190	$\hspace{0.1mm}-\hspace{0.1mm}$	215	677	48200	11300	8870
98°C-hour 16	2.05	1.319	12751	28.9	3151	1.288	[0.392] 8697	[4.170] 3.876	[<0.01] 244	703	[1.120] 48533	[0.355] 11367	[0.125] 8787
After cool down and transfer to UFP-VSL-T02A	2.14	1.303	$12472^{(b)}$	28.9	3178	1.281	[0.393] [0.389]	[4.095] [4.024]	[n/d] [<0.01]		[1.108] [1.101]	[0.351] [0.333]	[0.124] [0.123]

Table 5.1. Batch 1: Caustic Leach

(b) Liquid Al fraction calculated from Raman data.

— Analysis not required in test plan.

Bold numbers are calculated values.

Ca, Mg, Nd, and PO₄⁻ are not provided in this table because they were not used in the caustic-leach data analysis.

Raman results are provided as bracketed value [].

M Molarity.

n/d Triplicate samples with analysis results below method reporting limit.

			Slurry Analyses						Liquid Analyses				
							A ₁ [AI(OH) ₄]						
	UDS	Slurry Density	Al	$\rm Sr$	Fe	Liquid Density	by Raman]	Free OH- [Raman]	C_2O_4 [Raman]	$Cl-$	NO ₃ [Raman] [Raman]	NO ₂	SO_4 [Raman]
Sample location: Tank T02A, Condition:	Wt%	$g/mL^{(a)}$	μ g/g	μ g/g	μ g/g	$g/mL^{(a)}$	μ g/g [M]	M [M]	μ g/g [M]	μ g/g	μ g/g [M]	μ g/g [M]	μ g/g [M]
After NaOH addition	2.87	1.354	$14949^{(b)}$	32.9	3676	1.326	[0.262]	[5.167]	$\overline{}$ [n/d]		[1.348]	[0.425]	[0.150]
88° C	2.50	1.332				1.312	[0.352]	[4.677]	[<0.01]		[1.245]	[0.394]	[0.138]
98° C—hour 0	2.58	1.323	13768	29.5	3213	1.293	7180 [0.339]	4.186 [4.535]	167 [<0.01]	732	52200 [1.197]	12200 [0.380]	9380 [0.134]
98° C—hour 1	2.51	1.323				1.305	[0.349]	[4.424]	[<0.01]		[1.192]	[0.379]	[0.135]
98° C—hour 2	2.80	1.320				1.297	0.347	[4.349]	\equiv	$\overline{}$	[1.178]	[0.369]	[0.134]
98°C-hour 4	2.09	1.319	12668	27.6	3090	1.298	7310 [0.353]	[4.301]	252 [<0.01]	707	53800 [1.158]	1200 [0.359]	9350 [0.132]
98°C-hour 8	2.21	1.306	13398	30.6	3332	1.288	8420 [0.376]	3.925 [4.226]	234 [<0.01]	709	49490 [1.131]	11500 [0.354]	8990 [0.126]
98°C-hour 10, No samples pump repair													
98°C-hour 12	2.09	1.310	12801	28.5	3173	1.288	8300 [0.380]	3.812 [4.137]	286 [<0.01]	693	48400 [1.113]	11300 [0.346]	8830 [0.124]
98° C—hour 14	2.21	1.308				1.291	[0.393]	[4.191]	[<0.01]		[1.122]	[0.357]	[0.125]
98°C-hour 16	2.04	1.305	12927	28.9	3163	1.289	8807 [0.396]	3.810 [4.116]	261 $\lceil n/d \rceil$	684	48033 [1.109]	11333 [0.349]	8793 [0.124]

Table 5.2. Batch 2: Caustic Leach

(b) Liquid Al fraction calculated from Raman data.

— Analysis not required in test plan.

Bold numbers are calculated values.

Ca, Mg, Nd, and PO4- are not provided in this table because they were not used in the caustic leach data analysis.

Raman results are provided as bracketed value [].

			Slurry Analyses						Liquid Analyses				
Sample location: Tank T02A, Condition:	UDS Wt%	Slurry Density g/mL ^(a)	Al μ g/g	Sr μ g/g	Fe μ g/g	Liquid Density $g/mL^{(a)}$	A ₁ $\left[$ Al(OH) ₄ $\right]$ by Raman] μ g/g [M]	Free OH- [Raman] M [M]	C_2O_4 [Raman] μ g/g [M]	$Cl-$ μ g/g	NO ₃ [Raman] μ g/g [M]	NO ₂ [Raman] μ g/g [M]	SO_4 [Raman] μ g/g [M]
After NaOH addition	2.83	1.356	$14226^{(b)}$	31.3	3458	1.329	[0.227]	[5.430]	[n/d]		[1.297]	[0.411]	[0.145]
88°C	2.78	1.334			$\overline{}$	1.315	[0.347]	[4.856]	[<0.01]		[1.196]	[0.385]	[0.134]
98°C-hour 0	2.27	1.324	12293	28.4	3206	1.307	6750 [0.343]	$\overline{}$ [4.751]	181 [<0.01]	690	52600 [1.174]	11800 [0.373]	9200 $[0.132]$
98°C-hour 1	2.22	1.324				1.304	[0.339]	[4.684]	[<0.01]		[1.157]	[0.364]	[0.129]
98° C—hour 2	2.10	1.322				1.304	[0.346]	[4.658]	[<0.01]		[1.152]	[0.366]	[0.129]
98°C--hour 4	2.18	1.323	13120	32.2	3519	1.295	7210 [0.354]	[4.597]	232 [<0.01]	671	51300 [1.140]	11500 [0.362]	9020 [0.127]
98°C-hour 8	2.10	1.312	12324	30.0	3140	1.297	7600 [0.371]	[4.523]	220 [<0.01]	626	47700 [1.111]	10800 [0.360]	8470 [0.124]
98° C—hour 10	2.12	1.307			$\overline{}$	1.294	[0.378]	[4.448]	[<0.01]		[1.097]	[0.365]	[0.124]
98° C—hour 12	1.87	1.307	12097	26.3	3077	1.296	7920 [0.383]	[4.392]	243 [<0.01]	655	49600 [1.087]	11100 [0.356]	11100 $[0.122]$
98° C-hour 14	1.89	1.312			$\overline{}$	1.294	[0.384]	[4.288]	[<0.01]		[1.067]	[0.348]	$[0.120]$
98°C-hour 16	1.80	1.301	12018	26.2	3053	1.288	8257 [0.393]	[4.316]	266 $\lceil n/d \rceil$	645	49100 [1.061]	11000 [0.347]	8673 [0.119]

Table 5.3. Batch 3: Caustic Leach

(b) Liquid Al fraction calculated from Raman data.

— Analysis not required in test plan.

Bold numbers are calculated values.

Ca, Mg, Nd, and PO4- are not provided in this table because they were not used in the caustic leach data analysis.

Raman results are provided as bracketed value [].

			Slurry Analyses						Liquid Analyses				
Sample location: Tank T02A, Condition:	UDS Wt%	Slurry Density g/mL ^(a)	Al μ g/g	Sr μ g/g	Fe μ g/g	Liquid Density g/mL ^(a)	A ₁ [Al(OH) ₄] by Raman] μ g/g [M]	Free OH [Raman] M [M]	C_2O_4 [Raman] μ g/g [M]	$Cl-$ μ g/g	NO ₃ [Raman] μ g/g ſМ	NO ₂ [Raman] μ g/g [M]	SO_4 [Raman] μ g/g ſМl
After NaOH addition	2.48	1.364	$14012^{(b)}$	29.6	3378	1.341	[0.260]	[5.847]	$\lceil n/d \rceil$		[1.259]	[0.396]	[0.139]
88°C	2.18	1.347				1.321	[0.340]	[5.259]	[<0.01]		[1.185]	[0.378]	[0.131]
98° C—hour 0	2.12	1.334	12644	27.0	2974	1.315	6820 [0.336]	[5.162]	169 [<0.01]	705	52000 [1.156]	11600 [0.368]	8980 [0.128]
98°C-hour 1	2.12	1.340				1.317	[0.344]	[5.167]	[<0.01]		[1.151]	[0.366]	[0.129]
98° C—hour 2	2.14	1.334				1.315	[0.342]	[5.164]	[<0.01]		[1.132]	[0.359]	[0.126]
98°C-hour 4	2.03	1.325	12368	27.4	3074	1.307	7230 [0.363]	[5.062]	171 [<0.01]	650	48800 [1.142]	11000 [0.364]	8710 [0.127]
98°C-hour 8	1.88	1.327	12076	23.6	2688	1.305	7740 [0.378]	$\overline{}$ [4.919]	179 [<0.01]	664	51300 [1.119]	11400 [0.353]	9030 [0.125]
98° C—hour 10	1.95	1.323				1.310	[0.387]	[4.890]	[<0.01]		[1.098]	[0.348]	[0.123]
98°C-hour 12	1.92	1.315	12130	26.4	2955	1.305	8150 [0.385]	[4.803]	216 [<0.01]	617	46700 [1.085]	10500 [0.342]	8260 [0.122]
98° C—hour 14	1.88	1.317				1.300	[0.397]	[4.759]	[<0.01]		[1.075]	[0.336]	[0.121]
98°C-hour 16	1.72	1.310	11778	26.8	3056	1.302	8263 [0.402]	[4.697]	191 $\lceil n/d \rceil$	635	47867 [1.068]	10767 [0.337]	8423 [0.120]

Table 5.4. Batch 4: Caustic Leach

(b) Liquid Al fraction calculated from Raman data.

— Analysis not required in test plan.

Bold numbers are calculated values.

Ca, Mg, Nd, and PO4- are not provided in this table because they were not used in the caustic leach data analysis.

Raman results are provided as bracketed value [].

			Slurry Analyses						Liquid Analyses				
	UDS	Slurry Density	A ₁	Sr	Fe	Liquid Density	Al $[Al(OH)4]$ by Raman]	Free OH [Raman]	C_2O_4 [Raman]	$Cl-$	NO ₃ [Raman]	NO ₂ [Raman]	SO_4 [Raman]
Sample location: Tank T02A, Condition:	$Wt\%$	g/mL ^(a)	$\mu g/g$	μ g/g	$\mu g/g$	$g/mL^{(a)}$	μ g/g [M]	M [M]	$\mu g/g$ [M]	μ g/g	μ g/g [M]	μ g/g [M]	μ g/g [M]
After NaOH addition,	2.56	1.357	$13981^{(b)}$	29.3	3242	1.332	$\hspace{0.05cm}$ [0.255]	[5.830]	$\hspace{0.05cm}$ $\lceil n/d \rceil$		[1.262]	[0.400]	[0.141]
88° C	2.06	1.333				1.317	[0.337]	[5.243]	[<0.01]		[1.147]	[0.363]	[0.128]
98° C—hour 0	2.13	1.324	12516	27.3	2980	1.307	6580 [0.330]	[5.082]	177 [<0.01]	656	49800 [1.113]	11100 [0.354]	8730 [0.125]
98°C-hour 1	2.03	1.324				1.310	[0.346]	[5.112]	[<0.01]		[1.127]	[0.360]	[0.126]
98°C-hour 2	2.10	1.329				1.310	[0.346]	[5.013]	[<0.01]		[1.118]	[0.355]	[0.125]
98°C-hour 4	2.20	1.321	12260	26.7	2968	1.307	7220 [0.351]	[5.029]	172 [<0.01]	631	47400 [1.107]	10700 [0.351]	8380 [0.125]
98°C-hour 8	1.95	1.305	12235	27.9	3187	1.302	7530 [0.367]	[4.877]	164 [<0.01]	642	48600 [1.072]	10900 [0.341]	8590 [0.121]
98°C-hour 10	1.96	1.314				1.305	[0.376]	[4.797]	[<0.01]		[1.071]	[0.341]	[0.121]
98°C-hour 12	1.84	1.312	11920	27.0	3047	1.301	7870 [0.378]	[4.712]	221 [<0.01]	620	47400 [1.049]	10700 [0.332]	8330 [0.118]
98° C—hour 14	1.85	1.305				1.301	$\hspace{0.1cm}-\hspace{0.1cm}$ [0.388]	[4.624]	[<0.01]		[1.050]	[0.330]	[0.118]
98°C-hour 16	1.73	1.301	11311	24.2	2836	1.294	8107 [0.382]	[4.517]	263 $\lceil n/d \rceil$	622	46833 [1.024]	10500 [0.320]	8267 [0.115]

Table 5.5. Batch 5: Caustic Leach

(b) Liquid Al fraction calculated from Raman data.

— Analysis not required in test plan.

Bold numbers are calculated values.

Ca, Mg, Nd, and PO4- are not provided in this table because they were not used in the caustic leach data analysis.

Raman results are provided as bracketed value [].

			Slurry Analyses						Liquid Analyses				
	UDS	Slurry Density	AI	$\rm Sr$	Fe	Liquid Density	A ₁ [A(OH)4] by Raman]	Free OH [Raman]	C_2O_4 [Raman]	$Cl-$	NO ₃ [Raman]	NO ₂ [Raman]	SO_4 [Raman]
Sample location: Tank T02A, Condition:	Wt%	$g/mL^{(a)}$	μ g/g	μ g/g	$\mu g/g$	$g/mL^{(a)}$	μ g/g [M]	M [M]	μ g/g [M]	μ g/g	μ g/g [M]	μ g/g [M]	μ g/g [M]
After NaOH addition	2.65	1.368	13387	27.6	3349	1.338	[0.231]	5.680 [5.851]	73.2 [n/d]	735	52767 [1.273]	12333 [0.403]	9610 [0.141]
88° C	2.16	1.336				1.312	6790	4.970	114	694	50200	11700	9150
98°C-hour 0	2.15	1.343	12239	27.0	2955	1.315	6780 [0.341]	5.210 [5.086]	136 [<0.01]	672	50900 [1.147]	11500 [0.365]	9020 [0.130]
98° C—hour 1	2.12	1.336				1.313	6740	4.775	183	675	48700	11500	8880
98°C-hour 2	2.11	1.336				1.312	6900	4.765	179	670	48200	11400	8770
98°C-hour 4	2.13	1.334	12038	25.8	3048	1.313	7150	4.595	169	663	47400	11300	8810
98°C-hour 8	1.88	1.326	11930	25.8	3083	1.309	7660	4.180	181 $\overline{}$	649	47200	11100	8490
98° C—hour 10	1.83	1.318				1.308	7880	4.075	181	652	46700	11000	8480
98°C-hour 12	1.85	1.319	11696	25.0	3010	1.301	8070	3.760	212 $\hspace{0.05cm}$	639	46200	10900	8360
98° C—hour 14	1.82	1.319				1.300	8250	3.455	231	634	45800	10700	8260
98°C-hour 16	1.70	1.319	11633	25.6	3054	1.301	8383 [0.401]	4.587 [4.622]	229 $\lceil n/d \rceil$	634	48100 [1.059]	10733 [0.338]	8440 [0.119]

Table 5.6. Batch 6: Caustic Leach

— Analysis not required in test plan.

Bold numbers are calculated values.

Ca, Mg, Nd, and PO₄⁻ are not provided in this table because they were not used in the caustic leach data analysis.

Raman results are provided as bracketed value [].

		Slurry		Liquid	Wet Centrifuged Solids
Sample location: Tank T02A, UDS Density			Si	Density ¹	Si
Condition:	$Wt\%$	g/mL	μ g/g	g/mL	μ g/g
$T=4$ hours	2.54	1.299		1.255	
$T = 8$ hours	4.67	1.311		1.252	
$T=32$ hours	6.51	1.332		1.237	
$T = 34$ hours	6.06	1.334		1.257	
$T = 56$ hours	8.09	1.347		1.278	
$T=74$ hours	9.06	1.373		1.313	
$T=94$ hours	10.4	1.378		1.302	
$T=114$ hours	11.1	1.388		1.294	
$T=134$ hours	12.9	1.404		1.322	
$T=154$ hours	13.0	1.418		1.320	
$T=184$ hours	14.5	1.431		1.314	
$T = 204$ hours	15.4	1.431		1.266	
$T = 224$ hours	14.5	1.423		1.300	
$T = 244$ hours	15.5	1.432		1.295	
$T=264$ hours	16.1	1.457		1.303	
$T=265$ hours	! 16.9	1.467		1.313	
$T = 300$ hours	17.4	1.467		1.302	
After Concentration ^(c)	18.3	1.399	$68.2^{(a)}$	1.253	$689^{\text{(a, b)}}$

Table 5.7. Post-Caustic-Leach Concentration of Solids

— Analysis not required in Test Plan.

(a) Original intact slurry submitted to analytical lab so phase separation was performed at analytical laboratory.

(b) % Solids = 61.91 %.

(c) Analytical results duplicated in Analysis at Key Process Steps table column heading Initial Slurry Composition for Post-Caustic-Leach Wash.

Table 5.8. Evaluation By-Pass of Filter-Loop Jet within Tank T02A on 02/14/09

			Liquid Analyses
		Filtration loop	Outer Lower Port Tank T02A
		C _s	Cs.
TI Step	Condition:	μ g/g	μ g/g
	Before tracer addition	0.00975	
TI-65 12.1.4	1 min after tracer addition	299	44.9
TI-65 12.1.4	2 min after tracer addition	139	111
TI-65 12.1.4	4 min after tracer addition	112	108
TI-65 12 1 4	8 min after tracer addition	106	107
TI-65 12.1.4	16 min after tracer addition	107	107
TI-65 12.1.4	30 min after tracer addition	104	104
TI-65 12.1.4	40 min after tracer addition	103	103
TI-65 12.1.4	50 min after tracer addition	101	102
TI-65 12.1.4	60 min after tracer addition	101	101
- Analysis not required in test plan.			

		Liquid Analyses									
		Filtration loop	Outer Lower Port Tank T02A								
		Cs.	Cs								
TI Step	Condition:	μ g/g	μ g/g								
	Before tracer addition	71.5									
TI-65 12.1.4	1 min after tracer addition	112	65.3								
TI-65 12.1.4	2 min after tracer addition	110	107								
TI-65 12.1.4	4 min after tracer addition	107	106								
TI-65 12.1.4	8 min after tracer addition	105	100								
TI-65 12.1.4	16 min after tracer addition	102	104								
TI-65 12.1.4	30 min after tracer addition	104	103								
TI-65 12.1.4	40 min after tracer addition	98.6	104								
TI-65 12.1.4	50 min after tracer addition	99.3	104								
TI-65 12.1.4	60 min after tracer addition	97.4	$208^{(a)}$								
			$98^{(b)}$								
TI-65 12.1.4	70 min after tracer addition		101								

Table 5.9. Evaluation By-Pass of Filter-Loop Jet within Tank T02A on 3/21/09

— Analysis not required in Test Plan.

(a) Sample collected from UFP-VSL-T02A outer low CD port.

(b) Sample collected from UFP-VSL-T02A outer middle CD port.

Table 5.10 provides analytical results for samples collected during the post-caustic-leach washing in Tank T02A. Two separate slurry samples were collected for the wt% UDS and slurry density. The supernatant density is a higher accuracy density than can be provided by the UDS analysis. The silicon slurry concentrations were obtained by directed analysis of the slurry. The liquid analysis results are supernatant analytical results obtained from samples phase-separated in PDL-W. Raman results, shown in brackets [], are provided as part of the liquid analyses and presented in molar concentration (mole/liter).

Table 5.11 provides analytical results for samples collected during the oxidative leaching in Tank T02A. The slurry density results and silicon slurry concentrations were performed on intact slurry submitted to the analytical laboratory. The wt% UDS were calculated from samples phase separated at PDL-W. The liquid analyses results are supernatant analytical results obtained from samples phaseseparated at PDL-W. The solids analyses results are analytical results of rinsed solids obtained from sample phase separation and rinsing at PDL-W. The wet centrifuged solids were immediately rinsed after phase separation to remove the remaining supernatant from the wet solids in an attempt to interrupt further reaction. The rinsate results are analytical results of composited rinse solutions used during the rinsing of phase-separated solids. Raman results, shown in brackets, [], are provided as part of the liquid analyses and presented in molar concentration (mole/liter). An aliquot of the wet centrifuged solids was dried at 105°C to determine the percent solids that are reported with the reported analytes in the wet centrifuged solids.

		Slurry Analyses								Supernatant Analyses							
Sample	UDS	Slurry Density ^(b)	Si	TDS	Liquid Density ^(b)	A ₁ [Al(OH) ₄] by Raman	Free OH [Raman]	Na	Cr $[\mathrm{CrO_4}^{\text{-2}}$ by Raman]	C_2O_4 [Raman]	NO ₃ [Raman]	NO ₂ [Raman]	SO ₄ [Raman]	PO ₄ [Raman]		Cl PDMS PPG	
location: Tank T ₀₂ A	Wt%	g/mL	μ g/g	Wt %	g/mL	μ g/g [M]	M [M]	μ g/g	μ g/g [M]	μ g/g [M]	μ g/g [M]	μ g/g [M]	μ g/g [M]	μ g/g [M]		μ g/g mg/g	mg/g
After Wash 3			$\overline{}$			6730 [0.298]	3.31 [3.448]	101000	2.56 [<0.001]	387 [<0.01]	35600 [0.770]	9240 [0.247]	6500 [0.087]	2580 $[0.037]^{(e)}$	494		
After Wash 6	18.9	1.351	$\qquad \qquad -$	22.7	1.208	5790 [0.255]	2.84 [2.951]	87800	2.08 [<0.001]	577 [<0.01]	31300 [0.661]	8100 [0.211]	5840 [0.074]	2790 $[0.045]^{(e)}$	432		
After Wash 9	$\overline{}$		$\overline{}$			5190 [0.229]	2.52 [2.635]	77700	2.54 [<0.001]	763 [<0.01]	27900 [0.590]	7250 [0.187]	5100 [0.067]	3520 $[0.054]^{(e)}$	384		
After Wash 12!	18.9	1.305	$\qquad \qquad -$	18.6	1.168	4560 [0.196]	2.21 $[2.292]$	72300	2.20 [<0.001]	1030 [<0.01]	24600 [0.506]	6350 [0.161]	4500 [0.057]	4030 [0.064]	336		
After Wash 15	$\overline{}$					4050 [0.174]	2.04 [2.047]	65800	2.14 [<0.001]	1290 [<0.01]	22900 [0.447]	5700 [0.145]	4180 [0.050]	4950 [0.074]	299		
After Wash 19	19.2	1.261	$\overbrace{}$	14.3	1.133	3290 [0.138]	1.59 [1.656]	56700	1.60 [<0.001]	1810 [<0.01]	18300 [0.363]	4660 $[0.118]^{(e)}$	3410 [0.041]	6210 [0.081]	247		
After Wash 24	$\overline{}$		--			2650 [0.107]	1.30 [1.363]	48400	1.48 [<0.001]	2490 $[0.018]^{(e)}$	16300 [0.289]	3770 $[0.096]^{(e)}$	2810 [0.032]	8210 [0.115]	201		
After Wash 30	19.4	1.215		10.1	1.095	2010 [0.078]	1.04 [1.019]	38300	1.12 [<0.001]	3560 $[0.028]^{(e)}$	12400 [0.217]	2810 $[0.069]^{(e)}$	2120 [0.024]	7480 [0.086]	153		
After Wash 37	$\hspace{0.1mm}-\hspace{0.1mm}$					1570 $[0.059]^{(f)}$	0.79 $[0.745]^{(f)}$	30900	0.841 $[<0.001]^{(f)}$	5090 $[0.060]^{(e, f)}$	9300 $[0.171]^{(f)}$	2070 $[0.054]^{(e, f)}$	1590 $[0.018]^{(f)}$	5640 $[0.071]^{(f)}$	114		
After Wash 43	19.2	1.206	$\overline{}$	6.61	1.051	1250 $[0.039]^{(e, f)}$	0.58 $[0.549]^{(e, f)}$	25400	0.69 $[<0.001]^{(f)}$	6800 $[0.077]^{(f)}$	7060 $[0.129]^{(f)}$	1570 $[0.034]^{(e, f)}$	1220 $[0.013]^{(e, f)}$	4360 $[0.047]^{(e, f)}$	89.3		
After Wash 49	$\overline{}$		-			962 $[0.032]^{(e, f)}$	0.47 $[0.423]^{(e, f)}$	21200	< 0.978 $[<0.001]^{(f)}$	8950 $[0.095]^{(f)}$	5390 $[0.100]^{(f)}$	1210 $[0.025]^{(e, f)}$	957 $[0.010]^{(e, f)}$	3380 $[0.039]^{(\mathrm{e},\,\mathrm{f})}$	70.4		
After Wash 55	18.5	1.176		4.86	1.038	676 $[0.019]^{(e)}$	0.30 $[0.313]^{(e)}$	18300	0.330 [<0.001]	10900 [0.111]	3880 [0.075]	892 $[0.027]^{(e)}$	700 $[0.008]^{(e)}$	2490 $[0.027]^{(e)}$	56.5		
After Wash 61	$\overline{}$					529 $[0.017]^{(e)}$	0.28 $[0.244]^{(e)}$	16400	0.376 [<0.001]	12600 [0.132]	2970 [0.060]	690 $[0.030]^{(e)}$	548 $[0.007]^{(e)}$	1930 $[0.019]^{(e)}$	41.8		
After Wash 67	17.6	1.164		4.09	1.029	391 [<0.01]	0.22 $[0.182]^{(e)}$	14900	< 0.243 [<0.001]	14800 [0.159]	2270 [0.048]	540 [<0.03]	421 $[0.005]^{(e)}$	1510 $[0.016]^{(e)}$	44		
After Wash 73	$\overline{}$		$\overline{}$			307 [<0.01]	0.16 $[0.101]^{(e)}$	13700	< 0.248 [<0.001]	16100 [0.161]	1730 [0.040]	419 [<0.03]	324 $[0.004]^{(e)}$	1140 [<0.01]	28.1		
After Wash 79	17.1	1.135		3.01	1.029	231 [<0.01]	0.12 $[0.110]^{(e)}$	10500	< 0.247 [<0.001]	12100 [0.124]	1330 [0.033]	330 [<0.03]	247 $[0.004]^{(e)}$	878 [<0.01]	22.7		
After Wash 85	$\hspace{0.1mm}-\hspace{0.1mm}$					188 [<0.01]	0.10 $[0.092]^{(e)}$	8380	< 0.247 [<0.001]	9470 [0.100]	1040 [0.028]	263 [<0.03]	195 $[0.003]^{(e)}$	676 [<0.01]	19.9		
After Wash 90 17.2		1.131		1.88	1.020	132 [<0.01]	0.09 $[0.082]^{(e)}$	6820	< 0.249 [<0.001]	7620 [0.080]	840 [0.024]	222 [<0.03]	158 $[0.002]^{(e)}$	563 [<0.01]	19.6		

Table 5.10. Post-Caustic-Leach Wash

5.10

		Slurry Analyses								Supernatant Analyses							
						Al			Cr								
		Slurry			Liquid	$\left[$ Al(OH) ₄	Free OH		$\left[\text{CrO}_4\right]$ ⁻² by	C_2O_4	NO ₃	NO ₂	SO ₄	PO ₄			
Sample	UDS	Density ^(b)	Si	TDS	Density (b)	by Raman]	[Raman]	Na	Raman]	[Raman]	[Raman]	[Raman]	[Raman]	[Raman]	Сŀ	PDMS PPG	
location: Tank T ₀₂ A	Wt%	g/mL	μ g/g	Wt $\%$	g/mL	μ g/g [M]	M [M]	μ g/g	μ g/g [M]	μ g/g [M]	μ g/g [M]	μ g/g [M]	μ g/g [M]	μ g/g [M]	μ g/g	mg/g	mg/g
After Wash 95						140	0.07		< 0.244	6070	686	184	128	462			
						[<0.01]	[<0.04]	5550	[<,0.001]	[0.063]	[0.022]	[<0.03]	$[0.002]^{(e)}$	[<0.01]	19.4		
After Final $Wash^{(c)}$	17.5	1.116	wet solid ^(a) 1400 55.40% solids liquid ^(a) 7.2	1.21	1.015	107 [<0.01]	0.07 [<0.04]	4590	< 0.247 [<0.001]	4940 $[0.050]^{(e)}$	589 $[0.020]^{(e)}$	160 [<0.03]	108 $[0.002]^{(e)}$	382 [<0.01]		19.9 $n/d^{(d)}$	SX Failed

Table 5.10. Post-Caustic-Leach Wash

(a) Original intact slurry was submitted to the analytical laboratory, so phase separation was performed at the analytical laboratory.

(b) If high-accuracy density results are available, then they are provided. If high-accuracy density results are not available, then density results from the UDS are provided.

(c) Analytical results were duplicated in Analysis at Key Process Steps table, column heading Post-Caustic-Leach, Concentration and Wash Sample.

(d) Single sample non-detect with analysis results below method reporting limit.

(e) Detected result is qualitative. Result > IDL but < EQL (estimated quantitation limit).

(f) Solids observed in the sample before and after centrifuging—results are subject to large error.

— Analysis not required in test plan.

Raman results are provided as bracketed value [].

M Molarity.

5.11

SX Failed—the organic phase failed to separate from the aqueous phase, and no results were obtained.

Table 5.10 (contd)

		Slurry Analyses	Liquid Analyses														
								Cr									
						OH	Cr	(rinsate)									
				Al	Al (rinsate)	[Free OH]	$\left[\text{CrO}_4\right]$ ⁻²	$\left[\text{CrO}_4\right]$ ⁻²									
		Slurry	Liquid	[Al(OH) ₄]	$\left[\text{Al}(\text{OH})_4 \right]$	by	by	by		Mn			C_2O_4		NO ₃	NO ₂	SO ₄
	UDS		Density ^(a) Density ^(a)	by Raman]	by Raman]	Raman ⁷	Raman]	Raman]	Mn	(rinsate)	PDMS	PPG	[Raman]	C _l -	[Raman]	[Raman]	[Raman]
Sample location: Tank T02A, Condition:	Wt%	g/mL	g/mL	μ g/g [M]	μ g/g [M]	M [M]	μ g/g [M]	μ g/g [M]	μ g/g	μ g/g	mg/g	mg/g	μ g/g [M]	μ g/g	μ g/g [M]	μ g/g [M]	μ g/g [M]
Before MnO ₄ Addition (b)	18.7	1.147	1.035	52.1 [n/d]	$22.2^{(e)}$ $\lceil n/d \rceil$	0.26 $[0.194]^{(d, e)}$	10.4 $\lceil n/d \rceil$	$14.2^{(e)}$ [n/d]	[n/d]	0.625	$n/d^{(f)}$	SX Failed	644 $\lceil n/d \rceil$	140	3617 $[0.054]^{(e)}$	69.9 $[0.029]^{(d, e)}$	32.9 $[0.001]^{(d, e)}$
After $MnO4$ Addition- Hr 0 $(5 \text{ minutes after MnO}_4)$ addition)	16.3	1.148	1.030	31.4 [<0.01]		0.05 $[0.062]^{(d)}$	7070 [0.134]		0.225				612 [<0.01]	131	3270 [0.064]	75.9 [<0.03]	148 $[0.002]^{(d)}$
After MnO ₄ Addition- Hr 1	16.7	1.155	1.025	39.6 [<0.01]		0.08 [<0.04]	6990 [0.130]		0.168				630 [<0.01]	136	3360 [0.064]	77.9 [<0.03]	147 $[0.002]^{(d)}$
After MnO ₄ Addition-Hr 2 16.6		1.149	1.026	44.6 [<0.01]		0.08 [<0.04]	6730 [0.124]		0.740				641 [<0.01]	133	3340 [0.064]	78.9 [<0.03]	140 $[0.002]^{(d)}$
After $MnO4$ Addition-Hr 3	16.7	1.150	1.024	46.6 [<0.01]		0.08 $[0.060]^{(d)}$	6450 [0.120]		0.682				657 [<0.01]	135	3400 [0.065]	81.9 [<0.03]	135 $[0.002]^{(d)}$
After MnO ₄ Addition- Hr 41	16.7	1.146	1.023	50.4 [<0.01]		0.09 $[0.057]^{(d)}$	6440 [0.119]		0.629				661 [<0.01]	137	3410 [0.066]	81 [<0.03]	134 $[0.002]^{(d)}$
After $MnO4$ Addition-Hr 5:	16.7	1.148	1.027	51.4 [<0.01]		0.08 $[0.086]^{(d)}$	6270 [0.118]		0.419				674 [<]0.01]	139	3470 [0.066]	81.2 [<,0.03]	134 $[0.002]^{(d)}$
After $MnO4$ Addition-Hr 6 (Final after Oxidative) Leach $)$ ^(c)	16.7	1.147	1.028	52.4 [n/d]	20.9 $\lceil n/d \rceil$	0.08 $[0.057]^{(d)}$	6300 [0.117]	1413 [0.027]	0.294	0.709	$n/d^{(f)}$	SX Failed	679 $\lceil n/d \rceil$	142	3490 [0.066]	81.3 [n/d]	137 $[0.002]^{(d)}$

Table 5.11. Oxidative Leach

(b) Analytical results were duplicated in the Analysis at Key Process Steps table column, heading Starting Slurry Composition Before Permanganate Addition, including wt% solids data.

(c) Analytical results were duplicated in the Analysis at Key Process Steps table column, heading Initial Slurry Composition for Post-Oxidative-Leach Wash, including weight percent solids data.

(d) Detected result is qualitative. Result > IDL but < EQL (estimated quantitation limit).

(e) One value is significantly different from the other two measured values.

(f) Single sample non-detect with analysis results below method reporting limit.

SX Failed— the organic phase failed to separate from the aqueous phase, and no results were obtained.

[Raman] results are provided as bracketed values [].

Bold numbers are calculated values.

— Analysis not required by Test Plan.

	Rinsed Wet Centrifuged Solids										
Sample location: Tank T02A,	Al	Si Sr Mn Сr μ g/g μ g/g μ g/g μ g/g μ g/g		PDMS	PPG						
Condition:						mg/g	mg/g				
Before $MnO4$ Addition ^(b)	118000	20433	1143	20433	Wet centrifuge solids $1570 - \mu g/g$ 51.82% solids Liquid fraction 70.2- μ g/g ^(g)	0.1	1.18				
After $MnO4$ Addition- Hr 0 $(5 \text{ minutes after MnO}_4 \text{ addition})$											
After $MnO4$ Addition—Hr 1											
After $MnO4$ Addition—Hr 2											
After MnO ₄ Addition-Hr 3											
After MnO ₄ Addition-Hr 4											
After MnO ₄ Addition-Hr 5											
After $MnO4$ Addition-Hr 6 (Final after Oxidative Leach $)^{(c)}$	122667	1170	1167	42100	Wet centrifuge solids $1420 - \mu g/g$ 50.61% solids Liquid fraction $25.2 - \mu g/g$	0.2	2.25				

Table 5.11 (contd)

Bold numbers are calculated values.

— Analysis not required by Test Plan.

(b) Analytical results were duplicated in the Analysis at Key Process Steps table column, heading Starting Slurry Composition Before Permanganate Addition, including wt% solids data.

(c) Analytical results were duplicated in the Analysis at Key Process Steps table, column heading Initial Slurry Composition for Post-Oxidative-Leach Wash, including wt% solids data.

(g) Original intact slurry was submitted to the analytical laboratory, so phase separation was performed at the analytical laboratory.

Table 5.12, *Post Oxidative Leach Wash,* provides analytical results for samples collected during the post-oxidative-leach washing in Tank T02A. Two separate slurry samples were collected for the weight percent UDS and slurry density. The supernatant density is a higher accuracy density than can be provided by the UDS analysis. The liquid analyses results are supernatant analytical results obtained from samples phase-separated in PDL-W. Raman results, shown in brackets, [], are provided as part of the liquid analyses and presented in molar concentration (mole/liter).

Table 5.13, *Full Analysis*, provides the concentration of key metal analytes, anions, total inorganic carbon (TIC), wt% UDS, and density at key process steps. Two separate slurry samples were collected for the wt% UDS and slurry density. The supernatant density is a higher accuracy density than can be provided by the UDS analysis. The concentrations of the metals and TIC in the slurry were calculated from samples phase-separated at PDL-W. The liquid analysis results are supernatant analytical results obtained from samples phase-separated in PDL-W. The wt% of wet centrifuged solids in the slurry can be used in calculating the total species concentration in the original samples slurry. See Section A.5 in Appendix A of WTP-RPT-186, Rev 0, for more information on calculating the species concentration in the slurry. An aliquot of the wet centrifuged solids was dried at 105°C to determine the percent solids that are reported with the reported analytes in the wet centrifuged solids.

Table 5.14, *PEP Testing Reagent Tables* (TDP-WTP-361), provides reagent analysis results. The reagent samples were shipped as collected. The analysis for contaminant metals and anions was performed on the initial samples. Contaminants were not detected in the 19-M NaOH above reporting limits with the exception of chloride present at 23.7-µg/g, which is slightly above the reporting limit of $16.9\text{-}\mu$ g/g. The 2-M HNO₃ showed multiple contaminants present. Several contaminants were detected in the NaMnO₄ at a concentration slightly above reporting limits. Sulfate was present at over 800-µg/g in the NaMnO4. This sulfate concentration is much higher than its reporting limit. For Integrated Test A, the sample of interest is *A 00C04 XXX XX 2610 OST 2*.
	Slurry Analyses		Liquid Analyses											
					Al		Cr		Free					
		Slurry		Liquid	$[A(OH)_4 by$		$[CrO4-2 by$		OH-	NO ₃	NO ₂	SO ₄	C_2O_4	
	UDS	Density ^(a)	TDS	Density ^(a)	Raman ⁷	Na	Raman]	Sr	[Raman]	[Raman]	[Raman]	[Raman]	[Raman]	$Cl-$
Sample location: Tank T02A, Condition:	Wt%	g/mL	wt $%$	g/mL	μ g/g [M]	μ g/g	μ g/g [M]	μ g/g	M [M]	μ g/g [M]	μ g/g [M]	μ g/g [M]	μ g/g [M]	μ g/g
After Wash 5	16.80	1.142	2.62	1.038	45.9 [<0.01]	7990	5130 [0.094]	< 0.165	0.08 [<0.04]	2820 [0.057]	75.9 [<0.03]	110 $[0.001]^{(c)}$	544 [<0.01]	114
After Wash 10					39.7 [<0.01]	6500	4190 [0.076]	< 0.249	0.07 [<,0.04]	2210 [0.048]	70 [<0.03]	94.7 $[0.002]^{(c)}$	447 [<0.01]	92.9
After Wash 15	17.00	1.135	1.76	1.022	33.9 [<0.01]	5460	3440 [0.062]	< 0.166	0.06 [<,0.04]	1800 [0.041]	65.6 [<0.03]	72.7 $[0.001]^{(c)}$	369 [<0.01]	73.4
After Wash 20					28.1 [<0.01]	4470	2730 [0.050]	< 0.244	0.04 [<,0.04]	1470 [0.035]	63.8 $[0.027]^{(c)}$	61.6 [<0.001]	295 [<0.01]	64.5
After Wash 25	17.10	1.128	1.21	1.029	21.9 [<0.01]	3720	2240 [0.041]	< 0.166	0.03 [<,0.04]	1190 [0.031]	64.1 $[0.029]^{(c)}$	46.9 [<0.001]	242 [<0.01]	53.6
After Wash 30					26.3 [<0.01]	3240	1870 [0.034]	< 0.250	0.05 [<,0.04]	991 [0.027]	64.8 [<0.03]	43.2 [<0.001]	202 [<0.01]	47.1
After Wash 35	17.30	1.147	0.83	0.996	18.9 [<0.01]	2730	1540 [0.028]	< 0.166	0.03 [<,0.04]	819 [0.024]	64.2 $[0.025]^{(c)}$	35.9 [<0.001]	164 [<0.01]	38.4
After Wash 40		1.125		1.009	17.3 [<0.01]	2330	1260 [0.023]	< 0.242	0.04 [<,0.04]	687 [0.021]	63.7 $[0.032]^{(c)}$	27.3 [<0.001]	137 [<0.01]	29.8
After Wash 48	17.40	1.144	0.56	0.989	14.8 [<0.01]	1830	929 [0.017]	< 0.166	0.03 [<0.04]	509 $[0.019]^{(c)}$	64.8 $[0.027]^{(c)}$	23.9 [<0.001]	96.2 [<0.01]	21.2
After Wash 54					13.7 [<0.01]	1580	759 [0.013]	< 0.246	0.03 [<,0.04]	428 $[0.017]^{(c)}$	64.4 [<0.03]	19.6 [<0.001]	80.1 [<0.01]	<19.6
After Wash 63	17.60	1.140	0.36	0.994	11.2 [<0.01]	1240	540 $[0.009]^{(c)}$	< 0.166	0.02 [<0.04]	319 $[0.015]^{(c)}$	65.4 [<0.03]	19.9 [<0.001]	59.2 [<0.01]	<19.9
After Wash 72					12.4 [<0.01]	1010	389 $[0.007]^{(c)}$	0.279	0.02 [<,0.04]	257 $[0.014]^{(c)}$	65.1 [<0.03]	19.8 [<0.001]	40.3 [<0.01]	<19.8
After Wash 78	17.60	1.144	0.19	1.000	10.8	884	308 $[0.005]^{(c)}$	0.508	0.02	220 $[0.013]^{(c)}$	65	19.8	32.3	<19.8
After Wash 84					[<0.01] 11.4	806	253	0.773	[<,0.04] 0.02	189	[<0.03] 63.1	[<0.001] 19.2	[<0.01] 36.1	<19.2
After Final Wash ^(b)	17.70	1.127	0.14	1.006	[<0.01] 11.4 [<0.01]	801	$[0.004]^{(c)}$ 233 $[0.004]^{(c)}$	0.868	[<,0.04] 0.02 < 0.04]	$[0.014]^{(c)}$ 180 $[0.013]^{(c)}$	$[0.029]^{(c)}$ 64.1 $[0.025]^{(c)}$	[<0.001] 19.5 [<0.001]	[<0.01] 36.3 [<0.01]	<19.5

Table 5.12. Post-Oxidative-Leach Wash

— Analysis not required in Test Plan.

[Raman] results are provided as bracketed values [].

M Molarity.

(a) If high-accuracy density results are available, then they are provided. If high-accuracy density results are not available, then density results from the UDS are provided.

(b) Analytical results were duplicated in the Analysis at Key Process Steps table, column heading Post Oxidative Leach, Concentration, and Wash Sample.

(c) Detected result is qualitative. Result > IDL but < EQL (estimated quantitation limit).

			Initial Slurry Composition for Post-Caustic-Leach		Post-Caustic-Leach, Concentration, and Wash			
		Simulant Feed Composition ^(g)	Wash		Sample			
	Slurry	Liquid Phase	wet centrifuged solid	Liquid Phase	wet centrifuged solid	Liquid Phase		
Analyte	$\mu g/g$	μ g/g	μ g/g	μ g/g	μ g/g	μ g/g		
A ₁	$20023 \pm 344^{\text{(a)}}$	2977 ± 52	84000 ± 5690	7010 ± 422	124000 ± 8380	107 ± 38		
Ca	154 ± 4	n/d	2060 ± 140	$<1.22 \pm 0.61$	3380 ± 262	$< 1.24 \pm 0.62$		
Ce	53.0 ± 1.1	0.0097 ± 0.0007	767 ± 52	0.038 ± 0.003	1270 ± 86	0.011 ± 0.003		
Cr	n/d	1.32 ± 0.03	48.1 ± 23.7	2.62 ± 0.20	63.3 ± 23.8	$< 0.247 \pm 0.124$		
Fe	4736 ± 94	n/d	68300 ± 4630	7.69 ± 2.49	109000 ± 7370	$<4.95 \pm 2.48$		
$\rm K$	1029 ± 17	1090 ± 20	306 ± 41	595 ± 40	$< 28.2 \pm 14.1$	8.69 ± 3.75		
La	40.0 ± 0.8	0.010 ± 0.001	575 ± 39	0.0325 ± 0.0031	952 ± 64	0.00950 ± 0.00254		
Mg	105 ± 3	n/d	1430 ± 97	$< 2.44 \pm 1.22$	2540 ± 183	$< 2.47 \pm 1.24$		
Mn	1027 ± 20	n/d	14400 ± 972	0.42 ± 0.07	22000 ± 1490	$< 0.124 \pm 0.062$		
Na	88920 ± 1372	91600 ± 1587	102000 ± 6910	108000 ± 6480	11600 ± 979	4590 ± 316		
Nd	108 ± 2	0.026 ± 0.001	1570 ± 106	0.08 ± 0.01	2620 ± 177	0.0246 ± 0.0029		
Ni	143 ± 3	n/d	1960 ± 132	0.16 ± 0.06	3370 ± 227	$< 0.124 \pm 0.062$		
\mathbf{P}	1784 ± 28	1873 ± 32	9680 ± 655	701 ± 43	1880 ± 130	144 ± 11		
Si ^(h)	$48.7 \pm 5.7^{(i)}$	$2.80 \pm 0.36^{(i)}$	519 ± 191	48.3 ± 2.9	1220 ± 205	7.08 ± 0.65		
Sr	43.8 ± 1.3	n/d	570 ± 40	$< 0.122 \pm 0.061$	897 ± 62	$< 0.124 \pm 0.062$		
$\mathop{\rm Zr}\nolimits$	136 ± 3	$0.737 \pm 0.022^{(i)}$	1830 ± 124	2.36 ± 0.15	3130 ± 211	$< 0.124 \pm 0.062$		
Cl		1137 ± 20		545 ± 34		$<19.9 \pm 10.0$		
NO ₂		18267 ± 329	$\qquad \qquad -$	10100 ± 607	$\qquad \qquad -$	160 ± 34		
NO ₃		79333 ± 1380	$\overline{}$	38900 ± 2459		589 ± 57		
PO ₄		5547 ± 96	\equiv	1990 ± 122	$\qquad \qquad =$	382 ± 38		
SO ₄		14167 ± 247	$\overline{}$	7110 ± 461	$\overline{}$	108 ± 12		
C_2O_4		625 ± 11	\equiv	276 ± 19	$\qquad \qquad$	4940 ± 357		
Wt% Solids			59.34		56.22	$\qquad \qquad$		
Free OH (µg/mL)	\equiv	$15239 \pm 660^{(d)}$	\equiv	$6.08E + 04$		$1.15E + 03$		
$TIC^{(b)}$	1510 ± 253	6905 ± 133	$\qquad \qquad$		$\overline{}$			
PDMS (mg/g)	$n/d^{(c,e)}$	$n/d^{(c)}$	$\qquad \qquad$	$\frac{1}{1}$	0.2	$n/d^{(c)}$		
PPG (mg/g)	$n/d^{(c,e)}$	$n/d^{(c)}$		$\overline{}$	2.65	SX Failed		
Density (g/mL)	1.278 ± 0.006	1.239 ± 0.001		1.253 ± 0.019		1.015 ± 0.016		
Wt% H ₂ O		72.7 ± 0.1	$\qquad \qquad$			$\overline{}$		
Slurry wt% UDS	5.52 ± 0.03	$\overline{}$	18.3 ± 0.3		$17.5 \pm .27$			
Slurry Density (g/mL)	1.278 ± 0.006		1.399 ± 0.022	$\overline{}$	1.116 ± 0.017	$\overline{}$		
Weight percent Centrifuged Solids (%)	14.6	$\overline{}$	41.6		30.9			
- Analysis not required in test plan.			(e) Analysis performed on wet centrifuged solids.					
Bold numbers are calculated values.								
n/d Triplicate samples with analysis results below method reporting limit.			(f) Liquid phase data contains only results from the decanted supernate. No rinsate results are included. (g) Uncertainty values reported for the Simulant Feed Composition are 1σ , instead of the 2σ values reported throughout					
SX Failed – the organic phase failed to separate from the aqueous phase and no results were obtained.			the rest of the table.					
(a) Fraction of solid-phase Al that is in gibbsite=0.435.			(h) Si results from sample named "ICP" rather than sample named "AFA".					
(b) TIC= Total Inorganic Carbon.			(i) The AFA slurry sample was phase separated at SwRI with the following information reported: wet centrifuged solids					
(c) Single sample non-detect with analysis results below method reporting limit.			had 590-µg/g Si; percent total solids in the wet centrifuged solids was 56.73%; liquid fraction of slurry sample had					
(d) Free OH given in units of mg/kg.			$3.07 - \mu g/g$ Si.					

Table 5.13. Analysis at Key Process Steps

	Initial Slurry Composition Before Permanganate			Initial Slurry Composition for Post Oxidative Leach	Post Oxidative Leach, Concentration, and Wash		
	Addition			Wash	Sample		
	rinsed wet centrifuged		rinsed wet centrifuged				
	solids	Liquid Phase ^(f)	solid	Liquid Phase ^(f)	wet centrifuged solid	Liquid Phase	
Analyte	$\mu g/g$	μ g/g	$\mu g/g$	$\mu g/g$	μ g/g	μ g/g	
Al	$\overline{118000 \pm 4608}$	52 ± 2	122667 ± 4787	52 ± 2	$\overbrace{\qquad \qquad }^{}$	11.4 ± 2.6	
Ca	2937 ± 128	n/d	2943 ± 108	n/d		9.60 ± 1.36	
Ce	1157 ± 45	n/d	1177 ± 46	0.0086 ± 0.0019	$\overline{}$	0.277 ± 0.017	
Cr	20433 ± 796	$\textbf{10.4} \pm \textbf{0.4}$	1170 ± 47	6300 ± 218	$\overline{}$	233 ± 14	
Fe	95300 ± 3724	n/d	97167 ± 3795	n/d	$\overline{}$	27.3 ± 3.0	
K	31 ± 8	n/d	107 ± 9	36.9 ± 3.1		$<14.8 \pm 7.4$	
La	864 ± 34	n/d	879 ± 34	0.0076 ± 0.0019	$\overline{}$	0.507 ± 0.031	
Mg	2013 ± 81	n/d	2050 ± 80	n/d		$<4.95 \pm 2.48$	
Mn	20433 ± 796	n/d	42100 ± 1641	0.294 ± 0.049	$\overline{}$	3.45 ± 0.24	
$\rm Na$	10140 ± 427	7610 ± 290	11767 ± 535	9657 ± 355		801 ± 57	
Nd	2367 ± 92	0.0102 ± 0.0019	2407 ± 94	0.0224 ± 0.0021	$\overline{}$	1.81 ± 0.11	
Ni	2740 ± 107	n/d	2790 ± 109	n/d	$\qquad \qquad -$	0.718 ± 0.251	
P	927 ± 36	64.5 ± 2.2	993 ± 39	62.6 ± 2.2	$\overline{}$	23.2 ± 1.5	
Si	1733 ± 127	36.93 ± 1.34	1757 ± 119	28.07 ± 1.05	$\qquad \qquad -$	8.25 ± 1.11	
Sr	1143 ± 45	n/d	1167 ± 46	n/d	$\overline{}$	0.868 ± 0.134	
Zr	2580 ± 101	n/d	2637 ± 103	n/d	$\qquad \qquad -$	0.706 ± 0.251	
C ₁		140 ± 7		142 ± 7		$<19.5 \pm 9.8$	
NO ₂		70 ± 19	$\overbrace{}$	81 ± 19	$\overbrace{}$	$<64.1 \pm 32.1$	
NO ₃		3283 ± 128	$\overline{}$	3490 ± 123	$\hspace{0.1mm}-\hspace{0.1mm}$	180 ± 45	
PO ₄		201 ± 19	$\hspace{0.1mm}-\hspace{0.1mm}$	208 ± 19		81.5 ± 30.2	
SO ₄		33 ± 6	$\overline{}$	137 ± 7	$\overline{}$	19.5 ± 9.8	
C_2O_4		644 ± 23		679 ± 24		36.3 ± 1.0	
Wt% Solids	54.43		55.02		$\overline{}$		
Free OH $(\mu$ g/mL)	$\qquad \qquad -$	$4.43E+03$	$\overline{}$	$1.38E+03$	$\overbrace{}$	3.38E+02	
$TIC^{(b)}$			$\overline{}$		$\overline{}$		
PDMS (mg/g)	0.1	$n/d^{(c)}$	0.2	$n/d^{(c)}$	$\hspace{0.1mm}-\hspace{0.1mm}$	$\hspace{0.1mm}-\hspace{0.1mm}$	
PPG (mg/g)	1.18	SX Failed	2.25	SX Failed	$\overline{}$		
Density (g/mL)	$\overline{}$	1.035 ± 0.014	$\overline{}$	1.028 ± 0.009	$\hspace{0.1mm}-\hspace{0.1mm}$	1.006 ± 0.016	
$Wt\% H_2O$			$\overline{}$		$\overline{}$	$\qquad \qquad$	
Slurry wt% UDS	18.7 ± 0.2	$\overline{}$	16.7 ± 0.1		17.7 ± 0.27	$\hspace{0.1mm}-\hspace{0.1mm}$	
Slurry Density (g/mL)	1.147 ± 0.010		1.147 ± 0.010	$\overline{}$	1.127 ± 0.018	$\qquad \qquad -$	
Weight percent Centrifuged Solids (%)	36.3	$\hspace{0.1mm}-\hspace{0.1mm}$	31.4	$\qquad \qquad -$	$\overline{}$	$\hspace{0.1mm}-\hspace{0.1mm}$	

Table 5.13 (contd)

— Analysis not required in test plan. **Bold** numbers are calculated values.

(f) Liquid phase data contain only results from the decanted supernate. No rinsate results are included. (h) Si results from sample named "ICP" rather than sample named "AFA."

n/d Triplicate samples with analysis results below method reporting limit.

SX Failed—the organic phase failed to separate from the aqueous phase, and no results were obtained.

(b) TIC= Total Inorganic Carbon.

(c) Single sample non-detect with analysis results below method reporting limit.

Table 5.14. PEP Testing Reagent Tables

M Molarity.

5.18

Table 5.15 provides analytical results for samples collected during the end of the testing when filters were being flushed, and slurry was transferred out of Tank T02A. The wt% UDS and densities were obtained following analytical methods described in Section 3 for slurry not phase-separated. The liquid analysis results are supernatant analytical results obtained from samples phase-separated in PDL-W.

Table 5.15. Filter Flush Analytical Results

	Slurry Analyses		Liquid Analyses								
		Slurry		$\left[\text{Al}(\text{OH})_{4} \right]$	$[\text{CrO}_4^{-2}$ by	[Free OH]	NO ₃	NO,	SO ₄	C_2O_4	
	UDS	Density i	TDS	by Raman]	Raman]	by Raman	[Raman]	[Raman]	[Raman]	[Raman]	
							μ g/g	μ g/g	μ g/g	μ g/g	
Sample Location	Wt%	g/mL	wt %	ſМ	ſМ	[M]	ſМl	[M]	ſМ	ſМl	μ g/g
5 min after circulating flush water in the loop with Tank T02A bypassed: Flushed loop contents	5.62	.034	0.147	$[<0.01]^{(a)}$	$[0.001]^{(a, b)}$	$[<,0.04]^{(a)}$	189 <63.3 [0.013] ^(a, b) $\left[<0.03\right]^{(a)}$		<19.2 [<,0.001]	111 \cdot [<0.01] ^(a) \leq 19	

— Analysis not required in Test Plan.

[Raman] results are provided as bracketed values [].

M Molarity.

(a) Solids observed in sample before and after centrifuging. Results are subject to larger error.

(b) Detected result is qualitative. Result > IDL but < EQL (estimated quantitation limit).

5.2 Physical Properties Data

This section summarizes the heat capacity, PSD, rheology, XRD, and SEM results. The interested reader is referred to Appendix H for additional information.

5.2.1 Heat Capacity

Table 5.16, *Heat-Capacity Measurements,* provides a summary of the heat capacity data obtained at process steps of interest at both 40 and 80°C. Heat capacity data over the temperature range 35 to 90°C for the samples listed can be found in Appendix H.

	Heat Capacity	Heat Capacity
Sample location: Tank T01A	$J/g \text{ }^{\circ}C$ @ 40 $^{\circ}C$	$J/g °C$ @ 80°C
Initial slurry material	3.32 ± 0.02	3.39 ± 0.06
$88C -$ Batch 1	3.35 ± 0.09	3.43 ± 0.10
98 °C—Start of Caustic Leach—Batch 1	3.48 ± 0.01	3.56 ± 0.07

Table 5.16. Heat-Capacity Measurements

5.2.2 Particle Size

Table 5.17, *Slurry Particle Size Distribution,* provides the PSD data at key process steps before and after sonication. A summary of the run conditions, sample preparation, and sample analysis is given in Appendix H.

		Pre-Sonication (microns)		Sonicated (microns)		
Process Step Description	d(0.100)	d(0.500)	d(0.900)	d(0.100)	d(0.500)	d(0.900)
Initial simulant characterization from HLP-VSL-T22	1.52	6.43	20.07	1.55	6.33	18.21
Post-caustic-leach concentration in Tank T02A - leached slurry	0.65	1.88	38.20	0.55	1.35	18.04
Post-caustic-leach water wash - unwashed slurry ^(a)	0.63	1.10	1.96	0.58	0.99	1.74
	0.57	1.20	2.68	0.50	0.93	1.73
Post-oxidative-leach concentration - final slurry	0.66	1.41	3.25	0.49	1.14	4.54
Duplicate sample analyzed. (a)						

Table 5.17. Slurry Particle-Size Distribution Results

5.2.3 Rheology

Table 5.18, *Integrated Test A Rheology Data,* provides a summary of rheology measurements performed at key points throughout Integrated Test A. For simplicity, only Bingham yield stress and consistency fits for the down ramps have been reported here. A more extensive analysis of the rheology measurements taken are provided in Appendix H with flow curve analysis using both the Bingham and Casson models for both the up and down ramps. Table 5.19, *Integrated Test A Shear-Strength Data*, provides addition shear-strength measurements.

				Bingham	Bingham	Supernate
			Shear Strength	Yield	Consistency	Viscosity
Process Test Step	Form	Measurement	$[Pa]^{(b)}$	$[Pa]^{(a)}$	$[mPa\cdot s]^{(a)}$	$[mPa\cdot s]^{(a)}$
Prior to transferring simulant from HLP-VSL-T22 to Tank T01A	Slurry	Flow Curve		$\overline{}$		4.244E-03
Prior to transferring simulant from HLP-VSL-T22 to Tank T01A	Slurry	Flow Curve	$\overline{}$	$\hspace{0.1mm}-\hspace{0.1mm}$	$\hspace{0.1mm}-\hspace{0.1mm}$	4.241E-03
Prior to transferring simulant from HLP-VSL-T22 to Tank T01A	Slurry	Flow Curve	$\hspace{0.05cm}$			4.199E-03
Tracer test-1 minute after tracer injection	Slurry	Flow Curve	$\hspace{0.1mm}-\hspace{0.1mm}$	2.685	15.33	
Tracer test—1 minute after tracer injection (repeat)	Slurry	Flow Curve	$\overline{}$	9.516	35.56	
Post-caustic-leach wash-initial slurry	Supernate	Flow Curve	$\hspace{0.1mm}-\hspace{0.1mm}$	$\overline{}$	$\hspace{0.1mm}-\hspace{0.1mm}$	4.144
Post-caustic-leach wash—initial slurry	Slurry	Flow Curve	$\overline{}$	2.769	14.84	
Post-caustic-leach wash-intermediate slurry	Supernate	Flow Curve	$\overbrace{}$			1.244
Post-caustic-leach wash—intermediate slurry	Slurry	Flow Curve		2.227	6.204	
Post-caustic-leach wash-final slurry	Supernate	Flow Curve	$\overline{}$	$\qquad \qquad -$		0.9893
Post-caustic-leach wash—final slurry	Slurry	Flow Curve		2.115	5.037	
Oxidative leach—before permanganate addition	Slurry	Shear Strength	7.623 12.36	$\hspace{0.1mm}-\hspace{0.1mm}$	$\overbrace{}$	
Oxidative leach-before permanganate addition	Slurry	Flow Curve	$\qquad \qquad \overline{\qquad \qquad }$	5.751	6.576	$\overline{}$
Oxidative leach-final slurry	Supernate	Flow Curve	$\hspace{0.05cm}$			1.021
Post-oxidative leach wash-intermediate slurry	Slurry	Flow Curve	$\overbrace{}$	2.232	5.135	
Post-oxidative leach wash-intermediate slurry	Supernate	Flow Curve	$\hspace{0.05cm}$	$\hspace{0.05cm}$	$\hspace{0.1mm}-\hspace{0.1mm}$	0.9838
Post-oxidative leach wash—final slurry	Supernate	Flow Curve	$\hspace{0.05cm}$	$\hspace{0.05cm}$		0.9693
Post-oxidative leach wash-final slurry	Slurry	Flow Curve	$\overline{}$	1.219	4.838	$\hspace{0.1mm}-\hspace{0.1mm}$
Post-oxidative leach concentration-final slurry	Slurry	Shear Strength	8.51 9.239	$\overline{}$		
Post-oxidative leach concentration-final slurry	Slurry	Flow Curve	$\hspace{0.1mm}-\hspace{0.1mm}$	4.515	6.121	$\hspace{0.1mm}-\hspace{0.1mm}$
Post-oxidative leach concentration-final slurry	Supernate	Flow Curve		0 ^(c)	0.9442	
Prepare for high solids test	Supernate	Flow Curve				0.999

Table 5.18. Integrated Test A Rheology Data Summary

— Analysis not required in Test Plan.

(a) All results derived from flow curve analysis correspond to the down-ramp.

(b) Shear-strength measurements correspond to a 72 hr gel time.

(c) Newtonian slurry treated as a Bingham Plastic with a zero yield stress.

			$Post$ —	$Post$ — $-$
	Oxidative Leach	Oxidative	Oxidative	Oxidative
	-before	Leach - before	Leach	Leach
	permanganate	permanganate	Concentration	Concentration
	addition	addition	--final slurry	—final slurry
Settling		Maximum	Maximum	Maximum
Time	Maximum Shear	Shear Stress	Shear Stress	Shear Stress
(hr)	Stress (Pa)	(Pa)	(Pa)	(Pa)
	5.747	4.988	3.902	4.072
$\overline{2}$	5.374	4.930	4.091	4.221
4	5.143	5.165	4.348	4.067
8	4.953	5.280	4.270	4.605
16	6.077	5.891	5.836	4.643
32	6.158	6.540	5.538	6.639
72	7.623	12.360	8.510	9.239

Table 5.19. Integrated Test A Shear-Strength Data

5.2.4 XRD

The results of the XRD analysis of the initial simulant characterization samples taken before the commencement of Integrated Test A, including the XRD pattern of the simulant used for Integrated Test A, are provided in Appendix H.

5.2.5 SEM

Appendix H contains the SEM micrograph along with information on the sample preparation, analysis, and further results.

5.3 Measured PJM Parameters

Table 5.20 summarizes the actual PJM peak average nozzle velocities and stroke lengths achieved at various portions of the test. Data files collected for the PJM peak average velocities and stroke lengths were collected at a frequency of 10 Hz. Figures 5.1 through 5.18 show the PJM peak average velocity curves during selected Integrated Test A operations.

Data Set						Actual PJM	Target Stroke	Actual Stroke		
Date:Time			PJM		Target PJM	Nozzle	Length	Length		Target PJM Actual PJM
(mm/dd/vr)	TI Step #		Operation	Process Step	Nozzle	Velocity	(in.)	(in.)		Cycle Time Cycle Time
(hr; min)	(TI p#)	Vessel	Mode	Description	Velocity (m/s)	(m/s)	(%)	(%)	(sec)	(sec)
1/31/09	$7.1.2.5.1$ (p67)	Tank T01A	Standard	Start of caustic leach for	4.8 ± 0.3	5.3	28.1 ± 1.7	29.8	35 ± 1	35.3
$12:52 - 13:13$				batch 1			$80\pm5\%$	85%		
1/31/09 $20:26 - 20:55$	$7.1.3.14.2$ (p74)	Tank T01A	Simple	Caustic leach @98°C for batch 1	4.8 ± 0.3	5.3	28.1 ± 1.7 $80 \pm 5 \%$	26.6 76%	35 ± 1	35.3
2/1/09 02:56	PJM tuning	Tank T01A	Simple	Caustic leach @98°C for batch 1	4.8 ± 0.3	4.7	28.1 ± 1.7 $80 \pm 5\%$	26.4 75%	35 ± 1	35.3
$2/1/09 - 2/2/09$ $22:01 - 01:36$	$7.1.4.7.1$ (p85)	Tank T02A	Standard	Dewatering for batch 1	7.3 ± 0.4	$7.6\,$	30.3 ± 1.8 $80 \pm 5\%$	28.8 76%	33 ± 1	33.2
1/31/09 $18:20 - 19:11$	$7.1.5.8.1$ (p90)	Tank T01B	Standard	Start of caustic leach for batch 2	4.8 ± 0.3	5.2	28.1 ± 1.7 $80 \pm 5\%$	28.6 82%	35 ± 1	35.1
2/1/09 ~104:02	7.1.6.15.2 (p97)	Tank T01B	Simple	Caustic leach @98°C for batch 2	4.8 ± 0.3	5.4	28.1 ± 1.7 $80 \pm 5\%$	21.2 60%	35 ± 1	35.1
2/1/09 07:51	PJM tuning	Tank T01B	Simple	Caustic leach @98°C for batch 2	4.8 ± 0.3	4.6	28.1 ± 1.7 $80 \pm 5\%$	29.3 84%	35 ± 1	35.1
2/2/09 $21:13 - 21:37$	$7.1.7.7.1$ (p107)	Tank T01A	Standard	Start of caustic leach for batch 3	4.8 ± 0.3	4.6	28.1 ± 1.7 $80 \pm 5\%$	25.4 72%	35 ± 1	35.3
2/3/09 02:54	PJM tuning	Tank T01A	Simple	Caustic leach @98°C for batch 3	4.8 ± 0.3	4.7	28.1 ± 1.7 $80 \pm 5\%$	25.6 73%	35 ± 1	35.3
2/3/09 ~104:40	$7.1.7.23.2$ (p115)	Tank T01A	Simple	Caustic leach @98°C for batch 3	4.8 ± 0.3	4.5	28.1 ± 1.7 $80 \pm 5\%$	26.7 76%	35 ± 1	35.3
2/4/09 ~16:06	$7.1.8.13.1$ (p126)	Tank T01B	Standard	Start of caustic leach for batch 4	4.8 ± 0.3	5.2	28.1 ± 1.7 $80 \pm 5\%$	$28\,$ 80%	35 ± 1	35.1
2/5/09 $01:43 - 02:00$	7.1.8.28.2 (p134)	Tank T01B	Simple	Caustic leach @98°C for batch 4	4.8 ± 0.3	4.6	28.1 ± 1.7 $80 \pm 5 \%$	28.2 80%	35 ± 1	35.1
2/7/09 $12:02 - 12:25$	$7.1.9.7.1$ (p146)	Tank T01A	Standard	Start of caustic leach for batch 5	4.8 ± 0.3	5.1	28.1 ± 1.7 $80 \pm 5\%$	30.7 87%	35 ± 1	35.3
2/7/09 18:49 - 19:41	$7.1.9.22.2$ (p154)	Tank T01A	Simple	Caustic leach @98°C for batch 5	4.8 ± 0.3	4.8	28.1 ± 1.7 $80 \pm 5 \%$	27.1 77%	35 ± 1	35.3
2/10/09 $06:16 - 06:44$	7.1.10.11.1 (p166)	Tank T01B	Standard	Start of caustic leach for batch 6	4.8 ± 0.3	4.6	28.1 ± 1.7 $80 \pm 5\%$	28.2 80%	35 ± 1	35.1
2/10/09 $13:59 - 17:15$	7.1.10.29.2 $(p174-175)$	Tank T01B	Simple	Caustic leach @98°C for batch 6	4.8 ± 0.3	$4.0\,$	28.1 ± 1.7 $80 \pm 5\%$	21.7 62%	35 ± 1	35.1
2/14/09 19:19	PJM tuning	Tank T02A	Simple	End of dewatering/ start of tracer test	12 ± 0.6	12.1	30.3 ± 1.8 $80 \pm 5\%$	31.3 83%	20 ± 1	20.8
2/14/09 $01:45 - 19:54$	7.1.12.1.3.1 (p190)	Tank T02A	Simple	End of dewatering/ start of tracer test	12 ± 0.6	12	30.3 ± 1.8 $80 \pm 5\%$	32.1 85%	20 ± 1	20.7

Table 5.20. PJM Parameters for Integrated Test A (TI-WTP-PEP-065)

T01A at 13:05 on 1/31/09: Peak Average = 5.3 (m/s)

Seconds

T01A at 20:48 on 1/31/09: Peak Average = 5.3 (m/s)

Figure 5.2. Tank T01A PJM Peak Average Velocity at the start of Batch 1 Caustic Leaching

T01A at 02:56AM on 2/1/09: Peak Average = 4.7 (m/s)

Figure 5.3. Tank T01A PJM Peak Average Velocity During Batch 1 Caustic Leaching

T02A at 00:46AM on 2/2/09: Peak Average = 7.6 (m/s)

Figure 5.4. Tank T02A Peak Average Velocity After Initial Transfer from UFP-VSL-T01A

T01B at 19:03 on 1/31/09: Peak Average = 5.2 (m/s)

T01B at 04:05AM on 2/1/09: Peak Average = 5.4 (m/s)

Figure 5.6. Tank T01B PJM Peak Average Velocity at the start of Batch 2 Caustic Leach

T01A at 21:30 on 2/2/09: Peak Average = 4.6 (m/s)

Figure 5.8. Tank T01A PJM Peak Average Velocity After Batch 3 Caustic Addition

T01A at 02:54AM on 2/3/09: Peak Average = 4.7 (m/s)

Figure 5.9. Tank T01A PJM Peak Average Velocity at the Start of Batch 3 Caustic Leach

T01A at 04:26AM on 2/3/09: Peak Average = 4.5 (m/s)

Figure 5.10. Tank T01A PJM Peak Average Velocity During Batch 3 Caustic Leach

T01B at 18:30 on 2/4/09: Peak Average = 5.2 (m/s)

T01B at 01:53AM on 2/5/09: Peak Average = 4.6 (m/s)

T01A at 19:27 on 2/7/09: Peak Average = 4.8 (m/s)

Figure 5.14. Tank T01A PJM Peak Average Velocity During Batch 5 Caustic Leaching

T01B at 6:36AM on 2/10/09:Peak Average=4.6 (m/s)

Figure 5.15. Tank T01B PJM Peak Average Velocity After Batch 6 Caustic Addition

T01B at 17:00 on 2/10/09: Peak Average = 4.0 (m/s)

Figure 5.16. Tank T01B PJM Peak Average Velocity During Batch 6 Caustic Leaching

T02A at 19:19 on 2/14/09: Peak Average = 12.1 (m/s)

T02A at 19:46 on 2/14/09: Peak Average=12.0 (m/s)

Figure 5.18. Tank T02A PJM Peak Average Velocity Before the Tracer Test

The loss coefficients evaluated in the RPP-WTP PEP testing are presented in Table 5.21. The plots used for the evaluations of the loss coefficients are given in Figure 5.19 through Figure 5.22.

	Loss Coefficient for	Loss Coefficient for	Target Velocity
Tank	Forward Flow	Backward Flow	(m/s)
Tank T01A	0.25	12	4 X
Tank T01B	0.38	1.3	4.8
Tank T02A	0.34	0.1	73
Tank T02A			

Table 5.21. Loss Coefficients Evaluated

T01A: K_f = 0.25 & K_b=1.2 for 4.9 (m/s)

Figure 5.19. Nozzle Velocity Distributions Used to Evaluate the Loss Coefficients for Tank T01A Tank at the Peak Average Velocity of 4.9 (m/s): k_f = 0.25 is the Loss Coefficient of the Forward Flow, and k $b = 1.2$ is the Loss Coefficient of the Backward Flow

Figure 5.20. Nozzle Velocity Distributions Used to Evaluate the Loss Coefficients for Tank T01B Tank at the Peak Average Velocity of 4.9 (m/s): k $f = 0.38$ is the Loss Coefficient of the Forward Flow, and $k_b = 1.3$ is the Loss Coefficient of the Backward Flow

Figure 5.21. Nozzle Velocity Distributions Used to Evaluate the Loss Coefficients for Tank T02A Tank at the Peak Average Velocity of 7.1 (m/s): $k_f = 0.34$ is the Loss Coefficient of the Forward Flow, and $k_b = 0.1$ is the Loss Coefficient of the Backward Flow

Figure 5.22. Nozzle Velocity Distributions Used to Evaluate the Loss Coefficients for Tank T02A Tank at the Peak Average Velocity of 11.5 (m/s): $k_f = 0.1$ is the Loss Coefficient of the Forward Flow, and $k_b = 0.1$ is the Loss Coefficient of the Backward Flow

6.0 Issues

Subsequent sections discuss the difficulties encountered during Integrated Test A, their work-arounds addressed, and in some cases, the resolutions. Refer to Appendix E for a timeline of events. Data, including figures, in this section are NQA-1 compliant.

6.1 Backpulse Description

Backpulsing was applied through the course of post-caustic-leach filtration to increase the permeate production rate. Initially per email, 2/4/09, 18:20 (Test Director), backpulsing was to be done only if pumps UFP-PMP-T42A and T43A had been running continuously for at least 6 hours, and the permeate rate dropped to or below 0.70 kg/min for at least 2 hours. Backpulse parameters were 40 psi overpressure (pressure above the average filter bundle tube side pressure), 5 psi deadband pressure (the pressure above the average filter bundle tube side pressure at which the backpulse is complete), and 10-in. backpulse initiation level (level drain setpoint). The first backpulse was performed on 2/5/09 at 00:07 (LRB 60108, pg. 130) using the standard backpulsing parameters noted above. A second backpulse was performed at 15:45 using the same backpulsing parameters (LRB 60229, pg. 121) per Test Director email, 2/5/09, 15:13.

By email (Test Director) 2/5/09, 17:14, backpulse parameters were changed, a number of consecutive backpulses were added, and backpulsing frequency was changed to every 12 hours. However, if the permeate rate dropped to 0.6 kg/min, the 6-hr continuous pump operation for UFP-PMP-T42A and Pump T43A still applied. However, by 2/6/09, the 2/5/09 backpulse instructions were revised because pulse pot volume changes, as found when using a 40 psi overpressure, could not be doubled using overpressures of either 20 or 60 psi. Backpulsing instruction changes included having the permeate rate to be at or below 0.60 kg/min before initiating a backpulse. While the deadband pressure was changed to achieve the target backpulse volume change in the pulse pot, the 10-in. level drain setpoint was kept the same for all backpulses.

Final backpulses performed were as shown in Table 6.1 (Test Director email, 2/6/09, 15:19).

Table 6.1. Initial Backpulses Performed During Post-Caustic-Leach Concentration

Reference	Date	Time	ID	Notes
LRB 60108, pg. 142 2/5/09		21:45		4 Perform 2 backpulses: overpressure 40 psi, deadband pressure 5 psi
LRB 60108, pg. 144 2/6/09		02:59		Perform 1 backpulse: overpressure 20 psi, deadband pressure 5 psi
LRB 60108, pg. 145	2/6/09	10:45		3 Perform 2 backpulses: overpressure 20 psi, deadband pressure 5 psi
LRB 60229, pg. 136 2/6/09		17:24		5a Perform 1 backpulse: overpressure 60 psi, deadband pressure 20.7 psi
LRB 60108, pg. 149 2/7/09		00:10		5b Perform 1 backpulse: overpressure 60 psi, deadband pressure 20.7 psi
LRB 60229, pg. 142 2/7/09		07:11		5c Perform 5 backpulses: overpressure 60 psi, deadband pressure 20.7 psi
LRB 60229, pg. 148 2/7/09		14:05		5d Perform 1 backpulse: overpressure 60 psi, deadband pressure 20.7 psi
LRB 60108, pg. 152 2/7/09		18:32	7	Perform 1 backpulse: overpressure 75 psi, deadband pressure 32.5 psi
				All backpulses used a level drain setpoint of 10 inches, and multiple backpulses were done back-to-back.

Refer to Figure 6.1 through Figure 6.8 to view the pressure drop across Filter T01A, the TMP (the average of Filter T01A inlet and outlet pressures minus the shellside pressure), the permeate rate, and the pulse pot level for each of the backpulses, noted by the corresponding identification number (ID), in Table 6.1. The TMP is reported in pounds-per square-inch differential pressure (psid). The pulse pot levels shown in Figure 6.1 through Figure 6.8 are for information only since these instruments were not calibrated to NQA-1 requirements.

Per phone conversation ~20:00 on 2/7/09 (LRB 60108, pg. 155), subsequent backpulses used the standard backpulse parameters.

Figure 6.1. Filter T01A Backpulse (1) Using Deadband and Overpressures of 5 and 20 psi, 2/6/2009

Figure 6.2. Two Consecutive Backpulses (3) on Filter T01A Backpulses Using Deadband and Overpressures of 5 and 20 psi, 2/6/2009

Figure 6.3. Two Consecutive Backpulses (4) on Filter T01A Using Deadband and Overpressures of 5 and 40 psi, 2/5/2009

Figure 6.4. Filter T01A Backpulse (5a) Using Deadband and Overpressures of 20.7 and 60 psi, 2/6/2009

Figure 6.5. Filter T01A Backpulse (5b) Using Deadband and Overpressures of 20.7 and 60 psi, 2/7/2009

Figure 6.6. Five Consecutive Backpulses (5c) on Filter T01A Using Deadband and Overpressures of 20.7 and 60 psi, 2/7/2009

Figure 6.7. Filter T01A Backpulse (5d) Using Deadband and Overpressures of 20.7 and 60 psi, 2/7/2009

Figure 6.8. Filter T01A Backpulse (7) Using Deadband and Overpressures of 32.5 and 75 psi, $2/7/2009$

The permeate performance after backpulsing was the same for all backpulse variations. The peak permeate after the backpulse was complete was \sim 1.1 \pm 0.1 kg/min. However, permeate rates dropped below 1 kg/min within 10 minutes.

On 2/5/09, 21:58 (LRB 60108, pgs. 142 and 143), two consecutive backpulses were executed. During the second backpulse, it was observed that there was a level decrease pause when the overpressure was being applied and the pulse pot level was decreasing. Then, the level decrease started up again until the pulse pot level indicated 0 inches. This backpulse "hiccup" was observed on several occasions, and the reasons for such behavior were neither identified nor understood. This automatic second-backpulse behavior occurred again on 2/8/09, 06:59 (LRB 60229, pg. 157) and 08:08, 2/9/09 (LRB 60230, pg. 3).

6.2 Air Entrainment

Vibration in the filter-loop pumps and increasing axial pressure drop across the filters was first observed during PJM tuning for non-Newtonian slurry in preparation for the tracer test (2/14/09). Filter-loop flow rate and TMP unstable behavior were other symptoms of air entrainment that emerged at the start of post-caustic-leach washing (TI step 13.4.2). The tracer test was conducted at a tank level (laser) of 48.9 inches, per the most recent stable level measurement, and the post-caustic-leach washing was done at a tank level (laser) of 44.3 inches. Having checked that there were no leaks in the pump flanges and connections, such operational problems may have been caused by unintentionally introducing air sources into the filtration loop. Although there were PJM overblow observations noted for UFP-VSL-T02A during solids concentration, PJM pressure data examination did not provide clear confirmation of PJM overblows. The change in filter alignment, going from the alignment of UFP-FILT-T01A to the alignment of UFP-FILT-T02A through Filter T05A in preparation for the tracer test, may also have introduced air into the filtration loop. PJM tuning efforts to a higher velocity could also have resulted in PJM overblows. In addition to the operational problems, there was also a defective flow meter downstream from UFP-PMP-T43A as noted on NCR 42317.1. The air entrainment issue may have worsened when trying to increase the filter-loop flow rate based on the erroneous low value reported by the defective flow meter on the discharge line of UFP-PMP-T43A.

By 00:44, 2/15/09 (LRB 60230, pg. 59), post-caustic-leach washing proceeded with approval at a filter-loop flow rate of 88 gpm (run sheet target was 109 ± 10 gpm). This was in the midst of continuing unstable and below-target filter-loop flow rates, coupled with high axial pressure drops across most of the filters. Washing began at 01:13, 2/15/09 (LRB 60230, pg. 59), and TMPs were as high as 70 psi on Filters T02A and T03A intermittently at the beginning of washing.

Washing was suspended at 01:54, 2/15/09 (LRB 60230, pg. 60) because of unstable pump speed and TMPs.

Subsequent paragraphs will use instrument numbers associated with the UFP-VSL-T02A filter loop. Refer to Figure 6.9 for a layout of filter-loop instrument numbers.

Figure 6.9. Schematic of UFP-VSL-T02A Loop and Filtration. Note that UFP-HX-T03A was bypassed during the washing operations of Integrated Test A.^(a)

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⁽a) $DT =$ density transmitter, $HX =$ heat exchanger, $LT =$ level transmitter, $FT =$ flow transmitter (includes the matching FE or Flow Element), PT = pressure transmitter (includes the matching PI or Pressure Instrument), TT = temperature transmitter (included the matching TE or Temperature Element), MIC = Motor Indicating Controller, V= Valve, SV = Solenoid Valve, ZV = Positioning Valve, and HIC = Human Interface Control. Instruments with Nonconformance Reports issued during testing are shaded.

Filter-loop flow-rate investigations took place between 01:57, 2/15/09 (LRB 60230, pg. 60) and 14:26, 2/15/09 (LRB 60230, pg. 74). Several combinations of the following parameter settings were tried to achieve the target filter-loop flow rate:

- turning PJMs off, reducing steam-ring air flow rate, and turning lower spargers and PJMs off
- running with only one pump at a time (UFP-PMP-T42A and UFP-PMP-T43A) and ramping its speed incrementally with a fixed backpressure valve (SV-0609) position
- running both pumps at various speeds at a fixed backpressure valve position, etc.

Many setting combinations were tried to achieve the run sheet target flow rate of 109 gpm. The results were an ~40 gpm filter-loop flow rate difference between FT-0623 (upstream from UFP-PMP-T42A) and FT-0635 (downstream from UFP-PMP-T43A) and a maximum flow rate of \sim 90 gpm per FT-0635.

The 10:26, 2/15/09, 40-mL filter-loop sample was centrifuged to remove entrained air; this indicated a 3-mL level change (LRB 60230, pg. 73) or \sim 7.5% air retention in slurry. It was also postulated that there could be loose connections and air leaks within the pump flanges and surrounding piping; however, per 14:07, 2/15/09 (LRB 60230, pg. 74), no indication of air leaks for flanges between UFP-VSL-T02A and UFP-PMP-T42A were found.

To determine the rate of entrained gas release at room temperature and atmospheric pressure, a 2-L graduated cylinder was filled with 1800-mL of slurry, and a second graduated cylinder was filled with 53-mL of slurry (12:00, 2/15/09; LRB 60230, pg. 110). By 22:10 on 2/15/09, the stable level indicated a volume fraction change of 2.2% and 1.9% for the 2-L and second graduated cylinders, respectively (the level was monitored until 03:40 on 2/16/09). A second experiment was done to see whether adding AFA would improve the gas release rate (2/15/09, 15:11; LRB 60230, pg. 111). Two graduated cylinders were filled with 50-mL simulant (density was 1.34 g/mL), one with AFA and the other without AFA. The results showed that AFA had no effect on the gas release rate—both achieved a 49.5-mL stable level at the same time (2 hours and 39 minutes).

Between 16:02, 2/15/09 (LRB 60230, pg. 74) and 22:54, 2/15/09 (LRB 60230, pg. 88), post-caustic-leach washing commenced, per instruction indicated by the JTG (LRB 60235, pg. 9): proceed with whatever filter-loop flow rates that were achieved with upper and lower spargers, bubblers, and steam ring turned off (14:26, LRB 60230, pg. 74) and after waiting for \sim 2 hours. Filter-loop flow rates achieved were 116 gpm on FT-0623 and 78 gpm on FT-0635 (13:55, LRB 60230, pg. 74).

A camera put inside UFP-VSL-T02A showed vortexing of fluid, which may be likely the source contributing to air entrainment (23:52, 2/15/09, LRB 60230, pg. 88). Very little foam was observed (08:35, 2/16/09, LRB 60230, pg. 97). The vortex was around the filter loop return nozzle and appeared to draw the small quantity of foam present down into the slurry.

Pump Settings for the tracer were (LRB 60230, 56) as follows:

- MC-0631=95%
- MC-0641=51% (manual)
- \bullet HIC-0609=60%
- PI-0633=140 \pm 10 psi
- FT-0635=107 gpm (100 to 110 gpm).

Pump Settings for post-caustic-leach washing were (LRB 60230, pg. 74) as follows:

- MC-0631=85%
- MC-0641=42%
- \bullet HIC-0609=55%
- FT-0623=116-120 gpm
- FT-0635=78-80 gpm.

The return line was submerged at the elevations for which the tracer and washing took place, and with no leaks in the pump flanges and connections, air entrainment must have been a result of the simulant retaining air introduced by bubblers, steam air purge, PJMs, and spargers. The air bubbles were apparently small enough that they could not overcome the shear strength of the simulant (the simulant at this point was concentrated to \sim 17-wt%, high solids material); thus, they did not reach the surface and were therefore retained in the simulant. Air retention could also have been influenced if the air bubbles attached themselves to solids in the slurry. With the presence of AFA, surface retention is decreased, which restricts coalescence, thus preventing bubbles from merging, growing, and escaping to the surface. Slurry retention of small air bubbles was also enhanced when slurry traveled through the pumps, which shear the existing bubbles into smaller bubbles, thus decreasing the chance that the bubbles will ever reach the surface. Similar behavior was observed in WTP-RPT-147, where Savannah River National Laboratory conducted slurry experiments using impellers for mixing (Stewart et al. 2007). The impellers sheared the gas bubbles, thus decreasing their size, which prevented them from reaching the surface. PNNL gas retention and release investigations also confirmed that gas retention is increased with decreasing yield stress slurries containing AFA (Stewart et al. 2008). Samples (#2383 and #2384) collected before sodium permanganate addition on 2/16/09 indicate a shear strength range of 7.6 to 12.4 Pa, respectively, for a 72 hour settling time.

After chromium addition and before dewatering, higher filter-loop flow rates were achieved at the higher vessel level (01:57, 2/16/09, LRB 60230, pg. 90), and filter-loop flow rates dropped when dewatering was completed (03:30, 2/16/09, LRB 60230, pg. 92) due to the lower slurry level in Tank T02A. Another UFP-VSL-T02A recirculation loop sample (39.5-mL) was taken and centrifuged down for 20 minutes (4500 rpm); the final volume was 36.5 -mL, indicating air entrainment of ~7.6% (07:05, 2/16/09; LRB 60230, pg. 96).

Following the post-oxidative-leach washing, Integrated Test A was put on hold (midnight of 2/16/09). At the direction of the Test Director, a list of tasks were performed after completing the wash, however, only those associated with air entrainment are listed below:

- Chart filter-loop flow meters FT-0623 (pump UFP-PMP-T42A inlet) and 0635 (outlet of pump UFP-PMP-T43A), filter-loop pressure PT-0739 (inlet to filter UFP-FILT-T01A), and both pump speeds to understand the cause of low filter-loop flow rates and differences in flow meters.
- Retorque the mechanical connections from UFP-VSL-T02A to pump UFP-PMP-T42A in the event that there is a potential leak on the line.
6.2.1 Summary of Slurry Degassing and Filter-Loop Pump Exercises

The following paragraphs summarize slurry degassing methods employed during Integrated Test A in efforts to degass the air entrained in the Tank T02A slurry.

The stable level measurement after the slurry was concentrated and washed was 54.6 inches at 23:40 and then 53.0 inches at 23:53 per laser (2/16/09, LRB 60230, pg. 116), which could suggest evidence of foam. PJMs 1801 and 1807 in UFP-VSL-T02A were back in service at 12:30, 2/17/09 (LRB 60230, pg. 118).

On 17:12, 2/17/09, the spargers, steam ring, and bubblers were off in UFP-VSL-T02A, while UFP-VSL-T02A was recirculating, and UFP-VSL-T02A PJM nozzle velocities were tuned to 12 m/s. Samples were collected from UFP-VSL-T02A at the request of the program manager (19:37 and 21:31, 2/17/09, LRB 60230, pg. 121).

A video was taken to capture the effects of turning the lower spargers on at 0.4 kg/min and off during PJM operation, although image quality was poor and only got worse with time (LRB 60230, pg. 123-124). At 22:20, 2/17/09 (LRB 60230, pg. 121), the lower spargers were turned on to 0.4 kg/min:

Subsequent Sparger operation indicates lower spargers set to 0.4 kg/min, and PJM operation indicates 12 m/s and 80% stroke (as tuned).

Flow before spargers:

- FT-0623 ~107 gpm
- FT-0635 \sim 78 gpm.

Flow after spargers turned on:

- FT-0623 $~1$ 75 gpm
- FT-0635 $~5$ gpm.

A 5-in. tank level increase was seen with lower spargers ON (22:28, 2/17/09; LRB 60230, pg. 122).

The lower spargers were turned off at 00:10, 2/18/09 (LRB 60230, pg. 124), the filter-loop flow rate increased without making pump speed adjustments, and pump noise also stopped, pump vibration decreased, and a lower tank level was achieved. This improved filter loop pump performance indicates that some of the lower sparger air was being entrained in the pump intake.

Additional filter-loop flow rate exercises were conducted with PJMs ON and then with all sources (bubbler, steam ring, and spargers) off to see whether flow rates indicated by flow meters before UFP-PMP-T42A and after UFP-PMP-T43A converged at different pump speed settings and backpressure settings—08:31 through 12:48, 2/18/98 (LRB 60230, pgs. 126–128). The difference between flow meters stayed the same, 30 to 40 gpm, regardless of pump settings or with the PJMs on or off. At 09:06, 2/18/09 (LRB 60230, pg. 128), two 40-mL samples and one 100-mL sample from the UFP-VSL-T02A recirculation loop were collected. These permeate samples had long spindle crystals at the time of collection they appeared to be oxalate (LRB 60235, pg. 30).

Between 13:02 and 13:56, 2/18/09 (LRB 60230, pg. 129-130), exercises using a different number of spargers (total flow of 0.4 kg/min) were completed to see the effects on filter-loop flow rates. With the PJMs on, bubblers, upper spargers and steam ring air off, it was observed that turning three of the five lower spargers on (and when turning the other two of the five lower spargers on) caused the filter-loop flow rate to drop. When either three or the other two lower spargers were turned off, filter-loop flow rate increased without making pump setting adjustments (pump vibration and noise also decreased). The same result was seen when only the spargers on either side of Pump T42A inlet were turned on and off. The stable level per laser at 14:11 was 49.8 inches (LRB 60230, pg. 126-130). At 14:25, the laser level was 49.3 inches. After turning the pumps (UFP-PMP-T42A=65%, UFP-PMP-T43A=40%, HIC=0609=55%) and PJMs on, the stable level measurement at 15:15 was 49.1 inches per laser.

Figure 6.10 through Figure 6.18 show degassing operations listed in Table 6.2 through Table 6.5. Table 6.2 through Table 6.5 summarize mixing operations conducted to better understand air entrainment and air release in attempts to establish a gas-free simulant state.

Figure 6.10. UFP-VSL-T02A Gas Accumulation and Gas Release Operations Using PJM and Lower Spargers, in Reference to Table 6.2 and Table 6.3

Section 1. Test to see how much gas accumulation in the slurry is achieved with PJM and sparger operation.								
UFP-VSL-T02A								
Operations	PJMs	Spargers	Date	Time	LRB#	Page $#$	Laser Level (in.)	
Stable Level Measurement	OFF	OFF	2/18/09	15:15	60230	133	49.1	
$- -$	ΟN	ON	2/18/09	15:20	60230	133	Increasing tank levels	
Stable Level Measurement	OFF	OFF	2/18/09	15:48	60230	133	52.5	

Table 6.2. Gas Accumulation and Degassing Instructions per Guidance Section 1

Table 6.3. Gas Accumulation and Degassing Instructions per Guidance Section 2

Section 2. Test to see how well PJM and Sparger Operation enable coalescence in efforts to remove gas in the slurry.								
	UFP-VSL-T02A							
Operations	PJMs	Spargers	Date	Time	LRB#	Page #	Laser Level (in.)	
	ON	ON	2/18/09	15:49	60230	133	--	
Stable Level Measurement	OFF	OFF	2/18/09	16:16	60230	134	--	
	OFF	OFF	2/18/09	16:18	60230	134	53.3	
	OFF	ON	2/18/09	16:19	60230	134	--	
Stable Level Measurement	OFF	OFF	2/18/09	16:46	60230	134	--	
	OFF	OFF	2/18/09	16:48	60230	134	51.3	
	OFF	0 _N	2/18/09	16:48	60230	134	--	
Stable Level Measurement	OFF	OFF	2/18/09	17:15	60230	134	$- -$	
	OFF	OFF	2/18/09	17:17	60230	134	51.6	
	OFF	ON	2/18/09	17:17	60230	134	--	
Stable Level Measurement	OFF	OFF	2/18/09	17:45	60230	134	--	
	OFF	OFF	2/18/09	17:47	60230	134	52.9	
	OFF	ON	2/18/09	17:47	60230	134	--	
Stable Level Measurement	OFF	OFF	2/18/09	18:15	60230	134	--	
	OFF	OFF	2/18/09	18:44	60230	134	48.4	
	OFF	ON	2/18/09	18:44	60230	134	--	

Per Degassing Instructions provided on pg. 18 of LRB 60235; execution began at 20:17, 2/18/09.							
Operations		PJMs Spargers	Date		Time LRB $#$		Page # UFP-VSL-T02A laser Level, (in.)
1) Stable Level Measurement OFF		OFF	2/18/09	20:17	60230	134	
	OFF	OFF	2/18/09	20:47	60230	134	48.7
2) Pumps ON	ON	OFF	2/18/09 20:56		60230	135	--
3) Stable Level Measurement OFF		OFF	$2/18/09$ 22:16		60230	135	--
	OFF	OFF	2/18/09 22:46		60230	135	46.8
$4) -$	ON	ON	2/18/09 22:46		60230	135	--
5) Stable Level Measurement	OFF	OFF	2/19/09 00:45		60230	136	
	OFF	OFF			$2/19/09$ 01:13 LT-0614 average		47.6
$6) -$	ON	ON	$2/19/09$ 01:15		60235	20	
7) Stable Level Measurement OFF		OFF	2/19/09 03:15		60235	20	--
	OFF	OFF	2/19/09 03:45		60235	21	48.1
8) Pumps On	ON	OFF	2/19/09 03:50		60235	21	--
9) Stable Level Measurement	OFF	OFF	2/19/09 04:20		60235	21	
	OFF	OFF	2/19/09	04:50	60235	21	46.5

Table 6.4. Gas Accumulation and Degassing Instructions per Guidance

At 22:15, a recirculation loop sample was collected, and at 23:00, a slow leak at the flange where the hosing goes into the tank was observed on the south side of UFP-VSL-T02A. A pipe out of UFP-VSL-T02A was leaking chilled water, and a bucket was placed underneath for containment (2/18/09, LRB 60230, pg. 135).

An experiment was performed to assess the effect of dilution on the rate of degassing on 2/19/09. Three graduated cylinders contained \sim 500-mL of the following simulant dilutions: 1) 17-wt% simulant, 2) 17-wt% simulant diluted to 10-wt% and corresponding to 588-mL of simulant with 412 of IW (capped and shaken and then poured into a graduated cylinder), and 3) a dilution of the existing volume of 17-wt% simulant to the maximum operating level of UFP-VSL-T02A. This corresponded to 625-mL of 17-wt% simulant and 375-mL of deionized water, capped and shaken and then poured into the graduated cylinder (LRB 60230, pg. 136 and 143). The total volume and settled solids volume of each graduated cylinder (± 2-mL) was measured over a 7-hr period. It was observed that greater total volume and settled solids volume decreases occurred over time in the higher and lower weight percent samples, respectively. These results are shown in Table 6.5.

Hour	Graduated Cylinder $A - 17$ -wt%			Graduated Cylinder $B - 10$ -wt%	Graduated Cylinder C -10.6 -wt%	
2/19/09	Settled Solids	Total Volume	Settled Solids	Total Volume	Settled Solids	Total Volume
	Volume (mL)	(mL)	Volume (mL)	(mL)	Volume (mL)	(mL)
Start	440	455	440	490	440	490
	430	455	355	490	360	490
C	425	450	335	490	340	490
3	420	450	325	490	333	488
4	420	450	317	490	325	488
	415	450	310	490	320	488
6	412	450	305	490	315	488
	410	450	301	490	310	488

Table 6.5. Sample Dilution vs. Degassing

Figure 6.11. UFP-VSL-T02A Gas Release Operations, in Reference to Table 6.4, Steps 1 Through 5

Figure 6.12. UFP-VSL-T02A Gas Release Operations, in Reference to Table 6.4, Steps 6 Through 9

Figure 6.13. UFP-VSL-T02A Gas Release Operations, in Reference to Table 6.6

level, repeat degassing sparger and PJM operations.								
Operations	PJMs	Spargers	Date	Time	LRB#	Page #	UFP-VSL-T02A laser	
							Level, $(in.)$	
$4) -$	ON	ON	2/19/09	05:00	60235	21	--	
5) Stable Level	OFF	OFF	2/19/09	07:00	60235	22	--	
Measurement								
	OFF	OFF	2/19/09	07:30	60235	22	47.5	
$6) -$	ON	ON	2/19/09	07:30	60235	22	$\qquad \qquad -$	
7) Stable Level	OFF	OFF	2/19/09	09:30	60235	22	--	
Measurement								
	OFF	OFF	2/19/09	10:00	60235	22	48.0	
8) Pumps On	ON	OFF	2/19/09	10:00	60235	22	$-$	
9) Stable Level	OFF	OFF	2/19/09	10:31	60230	137	--	
Measurement								
	OFF	OFF	2/19/09	10:33	60230	137	45.8	
Pumps On	ON	OFF	2/19/09	10:36	60230	138	$- -$	
Stable Level Measurement	OFF	OFF	2/19/09	11:00	60230	138	--	
	OFF	OFF	2/19/09	11:30	60230	138	46.5	

Table 6.6. Degassing Instructions per Guidance, Part II

Per Degassing Instructions noted above (in Table 6.4), since last stable level is comparable to the smallest degassed

Between 11:36 and 13:45, 2/19/09 (LRB 60230, pgs. 138–140), PJMs and pumps were turned on; pump settings were adjusted to meet the target flow rate of 109 gpm. Stable level measurements at 13:30 still gave a 46.5-in. laser tank level. The pump settings were made at 13:45:

- MC-0631=71%
- MC-0641=65%
- HIC-0609=55%
- FT-0635= \sim 110 gpm
- PI-0739= \sim 142 psi
- PI-0789=55 psi
- PI-0625=-85 to -93 inches of water
- FT-0623=137 gpm.

Without making any adjustments to pump settings, a pressure drop was observed, and the flow on FT-0635 (downstream from UFP-PMP-T43A) increased to 130 gpm (LRB 60230, pg. 141). It seems as if a plug in the filter loop was released, but may have been attributed to a change in the quantity of air entrainment in the filter loop. After adjusting pump settings to bring the filter-loop level down to 109 gpm, the pump settings at 14:13 were as follows:

- MC-0631=71%
- MC-0641=45%
- \bullet HIC-0609=50%
- FT-0635=109 gpm
- PI-0739=110 psi
- PI-0789=50 psi
- PI-0625=-26 inches of water
- FT-0623=114 gpm.

(Note: there is no big difference between flow meters FT-0623 and FT-0635 at this time.)

UFP-VSL-T02A recirculation loop 40-mL samples were taken and centrifuged at the request of the Test Director at 16:15 and 16:32, 2/19/09 (LRB 60230, pg. 146). These samples had cloudy supernate. Similarly, there was an instance when permeate samples were taken and they too were cloudy, but white in appearance - like milk, but they cleared up in a few minutes. The cloudy appearance of the permeate samples indicates the presence of a large number of very small bubbles. After the samples were centrifuged, the supernate was a clear yellow color, and the sample volume decreased from 40 to \sim 38-mL, indicating a gas holdup of $\sim 6\%$.

By 18:50, the PJM and pumps were on, and pumps had similar settings obtained at 14:13 (LRB 60230, pg. 148). The JTG met and decided to proceed with Integrated Test A using the steps described in Table 6.7 (per Test Director email 2/19/09, 7:02 PM). The steps in Table 6.7 were performed using PJM settings as is, even though the stroke did not meet the target—this was approved by the Test Director (19:11, LRB 60235, pg. 24).

NOTE: *Mixing operations below indicate that upper spargers are OFF.*

Table 6.7. Attempts to Maintain Filter-Loop Target

Figure 6.14. Operations for Restoring Target Filter-Loop Flow Rate, in Reference to Table 6.7, Step 1

Figure 6.15. Operations for Restoring Target Filter-Loop Flow Rate, in Reference to Table 6.7, Step 2

Figure 6.16. Operations for Restoring Target Filter-Loop Flow Rate, in Reference to Table 6.7, Steps A Through F

Figure 6.17. Operations for Restoring Target Filter-Loop Flow Rate, in Reference to Table 6.8, Steps 2 and 3

Figure 6.18. Operations for Restoring Target Filter-Loop Flow Rate, in Reference to Table 6.8, Steps 4 Through 7

Instructions above (Table 6.7) were superseded by the following Test Director email, 2/20/09 (LRB 60230, pg. 160):

NOTE: *Mixing operations below indicate that upper spargers are OFF.*

More attempts were made to bring the filter-loop flow rate to the 109 gpm target; however, none were successful. On 2/20/2009 at19:22, facilities personnel tried verifying that flow meter FT-0635, downstream from UFP-PMP-T43A, was working within manufacturer's and PEP specifications. However, the flow meter FT-0635was found defective per NCR 42317.1. With approval, filtering was initiated having permeate going back in UFP-VSL-T02A. Backpulsing for each of the filters followed (2/20/09, LRB 60230, pg. 165).

With approval, staff then proceeded with the Test Instruction (2/20/09, LRB 60235, pg. 30).

Based on the flow rate divergence problems with flow meter FT-0635, its replacement was made after completing Integrated Test A on 2/26/09 (Operations Manager email, 16:55, 2/26/09) and Hanson email, 4/7/09, 17:25.

6.2.2 Air Entrainment Operational Guidance and Recommendations

Pump operation issues were minimized with the subsequent operating strategies exercised in Integrated Tests B (WTP-RPT-192) and D (WTP-RPT-193). Written guidance for UFP-VSL-T02A Mixing Systems Operation in the Presence of Air was provided as follows:^(a)

In the event that filter-loop flow rates cannot be maintained at the targeted 109 ± 10 gpm using the pump-control guidance, and there is evidence that there is significant entrained air in the slurry, the following actions are to be taken.

Tank T02A Systems Guidance

- 1. Turn level bubbler off.
- 2. Turn steam-ring air purge off.
- 3. Allow 30 minutes to determine if the Tank T02A recirculation system recovers the desired flow rate. If flow is recovered, proceed with testing.
- 4. If flow does not recover, implement the initial degassing protocol listed below.
- 5. If flow recovers, proceed with testing.
- 6. If flow does not recover, turn off lower air spargers.
- 7. Allow 30 minutes to see if the system recovers flow. If flow is recovered, proceed with testing.
- 8. If flow does not recover, implement initial degassing protocol.
- 9. If flow recovers, proceed with testing.
- 10. If flow does not recover, contact the on-call Test Director.
- 11. Perform the extended degassing protocol listed below as needed to fully degas the system.
- 12. Reinitiate testing.

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13. If flow rates cannot be maintained at the targeted 109 ± 10 gpm, perform the initial degassing protocol.

⁽a) E-mail communication from R Burk, Bechtel, to SM Barnes, Bechtel, and GB Josephson and DE Kurath, PNNL, RE: Written Guidance for T02A Mixing Systems Operation in the Presence of Air, Filterloop Pump Operation, and PJM Operation During Integrated Test B. 3/11/09, 16:12.

- 14. If flow does not recover, reduce the filter recirculation flow to 88 + 10 gpm filter-loop flow rate (gives \sim 12 ft/s axial velocity in the filter tubes).
- 15. If 88 gpm cannot be maintained, perform the extended degassing protocol as needed to fully degas Tank T02A.
- 16. Reinitiate testing.
- 17. If flow cannot be maintained at $109 + 10$ gpm or $88 + 10$ gpm, continue testing with the filter recirculation loop flow rate reduced to $67 + 10$ gpm. This filter-loop flow rate gives \sim 9 ft/s axial velocity in the filter tubes and power/volume scaled mixing from the return nozzle jet).
- 18. If none of these actions are successful, begin raising the Tank T02A level by diluting with IW. Raise the tank level in 2-in. increments, allowing at least 30 min between IW additions.

Initial Degassing Protocol

- 1. Turn off filter-loop pumps.
- 2. Take a stable level measurement.
- 3. Adjust lower air sparge flow rate to normal full flow $(0.40 \text{ kg/min}, \sim 2.4 \text{ scfm/tube})$ and turn on PJMs.
- 4. Allow this configuration to run for 50 min.
- 5. Turn off spargers and PJMs.
- 6. Wait for 10 min.
- 7. Take a stable level measurement.
- 8. Turn pumps on and attempt to achieve target filter-loop flow rate.

Restore Tank T02A mixing systems per the stage of the Tank T02A systems operating guidance above where the initial degassing process was referenced.

Extended Degassing Protocol

- 1. Turn off filter-loop pumps and take a stable level measurement.
- 2. Adjust lower air sparge flow rate to normal full flow (0.40 kg/min, \sim 2.4 scfm/tube) and turn on PJMs.
- 3. Allow this configuration to run for 4.5 hours.
- 4. Turn off spargers and PJMs.
- 5. Wait for 20 minutes.
- 6. Operate the filter-loop pumps for 10 minutes.
- 7. Take a stable level measurement.
- 8. Repeat steps 2 through 7 as needed to degas Tank T02A as determined by prior degassed level readings or by less than 5% gas volume holdup measured by centrifuging a slurry sample.
- 9. Turn pumps on, and attempt to achieve target filter-loop flow rate.
- 10. Restore Tank T02A mixing systems per the Tank T02A systems operating guidance above.

6.2.2.1 Recommendations

Significant air entrainment was encountered during conduct of Integrated Test A. The air entrainment resulted in apparent cavitation (actual cavitation is the formation of gas bubbles due to low pressure at the pump impeller, resulting in decreased pump performance) of the filter loop pumps and led to reduced pumping capacity and flow rates through the filter loop. The series of events presented in previous sections of this report indicate the following:

- PJM overblows may have contributed to air entrainment in some cases. With the filter loop in operation, overblow bubbles could have been entrained into the pump in-take. Overblows were reported by the operations staff but this could not be confirmed with the available DAS data. Implementation of a robust control system to minimize PJM overblows, especially when the filter loop pumps are operating, is suggested as a means of mitigating the contribution of PJM overblows to the air entrainment issue.
- Air from the air spargers also appears to have contributed to the air entrainment issue. Separate tests indicate that the spargers closest to the pump intake and the return nozzle had the greatest impact. Suggested mitigating actions include reevaluating the location of the air sparge outlets and reducing the air flow to some or all of the air spargers.
- The bubbler level measuring device was located near the pump intake and may have contributed to the air entrainment issue. Although the air flow rate through the bubbler was relatively low the close proximity to the pump intake may have allowed the air to be pulled into the pump intake. It is suggested that the location of the bubbler level measuring instrument be evaluated.
- Air from the steam ring air purge may have been pulled into the pump intake.
- The partial failure of the flow meter (FT-0635) at the outlet of the filter loop pumps may have contributed to the magnitude of the air entrainment problem. At the completion of Integrated Test A this flow meter was found to be reading low. In attempting to achieve the target flow rate of 109 gpm, operators may have achieved a higher flow rate which would exacerbate the air entrainment from the filter loop return nozzle.

Based on the air entrainment operational issues encountered in Integrated Test A, future plant operations should avoid prolonged operations with low tank levels. The filter-loop return line in UFP-VSL-T02A was exposed at 20.3 inches (Section 4.9); therefore, operating at tank levels lower than this may have introduced air in the slurry during filter-loop recirculation. Bubblers and steam-ring and sparger settings only exacerbated the air entrainment at low levels. In addition, it would be prudent to investigate optimum PJM stroke and average peak velocities at different UFP-VSL-T02A tank levels and slurry properties in efforts to minimize, or prevent, air entrainment caused by PJM overblowing.

To expedite degassing efforts in plant operations, it is suggested to do the following:

- Operate spargers and PJMs with no pumps for 1 to 2 hours.
- Wait 30 minutes to 1 hour before taking a stable level measurement.
- Turn PJMs and filter-loop pumps ON for at least 30 minutes to degas.
- Wait 30 minutes before taking a stable level measurement.
- Repeat PJM and pump operation until level stops decreasing.

6.3 Level Instruments

At several times during the test, there was no credible level instrument for status and level control in any of the ultrafeed vessels. Not having a reliable level instrument made filtering, filling a vessel to a target level, and auto batching confusing and problematic, not to mention the associated high- and low-level alarms interrupting steam injection, PJM, and pump operations. Please refer to Table E.1 in Appendix E for the sequential list of level instrumentation problems. The following paragraphs discuss the types of problems and work arounds, if any, for each of the level instruments: laser, bubbler, and Drexelbrook.

6.3.1 Lasers

Laser performance was unreliable throughout Integrated Test A efforts, but especially during the leaching and post-caustic-leach concentration. One of the symptoms of unstable laser level performance was high and low level spikes, which interrupted steam injection, PJM, and pump operation. When concentrating post-caustic-leach slurry, the UFP-VSL-T02A low-level spikes erroneously triggered batches from the feed vessel and/or shut down PJMs and filter-loop pumps:

- \bullet 15:58, 2/2/09 (LRB 60229, pg. 59). The 23rd auto batch from UFP-VSL-T01A was triggered by an erroneous laser level in UFP-VSL-T02A. The low-low level alarm was triggered in UFP-VSL-T02A; therefore, pumps and PJMs were turned off.
- (LRB 60229, pg. 60) 16:52, $2/7/09$ (LRB 60229, pg. 150). Two auto batches occurred within \sim 35 minutes of each other for unknown reason in UFP-VSL-T01B.

The UFP-VSL-T02A laser high-level spikes shut down PJMs and filter-loop pumps, and in one instance, this interrupted an autobatch transfer. Laser high-level spikes in vessels UFP-VSL-T01A and B interrupted steam injection, PJM, and pump operations, depending on the process at the time. Between 21:00 and 21:47, 2/10/09 (LRB 60230, pg. 22), high-level spikes in UFP-VSL-T01B caused steam injection, PJMs, and pumps in UFP-VSL-T01A/B to shut off—one of the instances was during an autobatch transfer from UFP-VSL-T01A to UFP-VSL-T02A in which the systems were restarted. Such autobatch transfer interruptions confused the operator because the batch counter does not distinguish between attempted batch transfers and completed batch transfers.

A second problem lasers encountered was level confusion as a result of foam and/or aerosol concentration in the vessel headspace. Simulant splashing, perhaps from overblowing or general PJM performance with high simulant levels in the tank levels, only exacerbated laser functionality at 01:14, 2/16/09 (LRB 60230, pg. 89). Turning the lower air spargers ON to idle flow rates at 00:50 increased the laser level from 51 to 54 inches, and foam seemed to appear; the filter pump speed was reduced to bring the laser level back to 51 inches. Aerosols, as indicated on emails between 2/4/09 and 2/5/09, caused the laser signal to stay stagnant ("flatline") in UFP-VSL-T02A—the error code associated with this behavior indicated that it was a weak signal (02:54, 2/3/09, LRB 60229, pg. 70). The UFP-VSL-T01B laser triggered level alarms, and then it stayed at a value of 55.5 inches. Consequently, the level control was switched to bubbler (09:55, 2/3/09, LRB 60229, pg. 78). The UFP-VSL-T01B laser no longer flatlined, but it still was not working.

A couple of things were done to investigate laser performance. Observations indicate having a vent improves laser performance. The laser vent system plugs in UFP-VSL-T01B were removed to allow air flow past the lens (11:25, 2/1/09, LRB 60229, pg. 25). The sample port return on UFP-VSL-T01B was also opened temporarily to see if this would reduce the high-level spikes on the laser trend graph (00:41, 2/14/09, LRB 60235, pg. 3). As shown in Figure 6.19, stable laser levels were achieved with the UFP-VSL-T01B vent open—there were no high-level spikes on the laser trend plot.

Figure 6.19. UFP-VSL-T01B Laser Performance Improved with Sample Port Open

With approval from testing and operations, the chemical addition vent flange was opened to improve laser performance in UFP-VSL-T02A—bleed air should flush vent space aerosols—refer to Figure 6.20 (13:48, 2/5/09, LRB 60229, pg. 119). By 13:52, UFP-VSL-T02A was changed to laser level control based on improved laser performance (09:28, 2/6/09, LRB 60229, pg. 130). On another occasion, the UFP-VSL-T02A funnel valve was opened fully to clear the headspace, which helped the laser read well. By 09:49, level control was back to the laser.

Figure 6.20. UFP-VSL-T02A Laser Performance Improved After Opening Chemical Addition Vent Flange

Operators searched for alternative ways to improve laser performance (LRB 60229, pg. 117, 12:28). When the laser lenses were cleaned, simulant was found on them, and cleaning did not always bring the laser back into service. By 22:29, 2/3/09 (LRB 60229, pg. 92), the UFP-VSL-T01B lasers were stagnant. By 23:03, the instrument technicians cleaned the lens, but the laser still did not work (low return signal); foam may be to blame (LRB 60108, pg. 127). The steam air purge flow rate was increased to reduce the simulant build up on the laser lens while minimizing over-heating issues (Operations Manager Email, 7/20/2009, 09:29).

Also, lasers may not have functioned consistently because of the high possibility that the laser temperature specifications were exceeded during caustic leaching. Facilities personnel discovered that the lasers had a high temperature sensor alarming at 60° C (LRB 60229, pg. 25, 10:55). Although the temperature in the vessel headspace was not measured, this could explain why lasers in UFP-VSL-T01A and B did not work soon after reaching 88° C during caustic leaching. At $18:29$, $01/31/09$, operations were switched from laser to bubbler level control in UFP-VSL-T01A because the laser was reading 96 inches, and the temperature in the vessel was ~88C (LRB 60229, pg. 9). On 2/1/09, 02:37, the UFP-VSL-T01B level control was switched from the laser to the bubbler because the laser was reading 95 inches; the temperature in the vessel was $\sim 88^{\circ}$ C (LRB 60229, pg. 14). Potentially, the temperature environment in the headspace exceeded the laser temperature specifications.

6.3.2 Bubblers

Bubblers were susceptible to plugging on a regular basis. Blowing high-pressure air through the bubbler piping sometimes left the bubblers operational, and sometimes this was followed by water pressure (steam condensate and IW were used for bubbler cleaning). After the bubblers were successfully cleaned, the duration of functionality was unpredictable. Cleaning the bubblers was also problematic. High- and low-level spikes in the bubbler would interrupt PJM, steam injection, and pump operation if the bubbler was the level control instrument. For instance, at 19:00, 2/1/09, the UFP-VSL-T01B bubblers were cleaned because the specific gravity was reading \sim 4. During the cleaning, the UFP-VSL-T01A pump was tripped (LRB 60229, pg. 41). In time, the level control was switched to the laser before cleaning the bubblers to prevent equipment shutdowns. At other times, the bubblers were not cleaned if the laser was working well. When neither lasers nor bubblers worked, then the bubblers were cleaned. To assist bubbler credibility, samples were taken, and their density was measured to validate bubbler density readings.

Another bubbler issue only experienced in UFP-VSL-T02A was that their performance was influenced by filter pump (UFP-PMP-T42A and UFP-PMP-T43A) operations because of their physical location in the tank and PJM drive operations. When the filter-loop pumps were on, the flow from the pumps caused pressure changes at the bubblers. Consequently, the bubbler level and density readings were unstable, and erroneous at times, when the filter-loop pumps were on.

6.3.3 Drexelbrook

The Drexelbrook in UFP-VSL-T01A worked rather well (2/1/09, 09:02 through 09:08, LRB 60229, pg. 20). Level control in UFP-VSL-T01A was switched back and forth between Drexelbrook, bubbler, and laser because the noise in the bubbler level values caused high-level alarms in the vessel. Level control was kept the most stable with the Drexelbrook. The Drexelbrook level trend was smoother with the steam-ring air off than when it was on (LRB 60229, pg. 22-24). The PJM Drexelbrooks, on the other hand, were not quite as reliable (01:33, 2/12/09, LRB 60230, pg. 29). All PJM Drexelbrooks in UFP-VSL-T01A and UFP-VSL-T02A, except one in UFP-VSL-T01A, were flatlining. At times, the PJM stroke correlated well with the tank level change produced by the laser and/or bubbler. However, there were times when the PJM stroke per the Drexelbrooks was much shorter than the corresponding tank level change (14:58 on 2/1/09, LRB 60229, pg. 35). PJM probes 1601, 1603, and 1605 were not responding to the UFP-VSL-T01A PJM drive phase; they stayed at value 48 inches or 38 inches for 1605.

The 08:32 PJM probes in UFP-VSL-T02A were not working properly on 2/2/09 (LRB 60229, pg. 47), and PJM drive pressure increased without having made adjustments. While PJM operations may have been hindered by prolonged vacuum (pulling the simulant too high in the PJM tubes and air supply lines) and/or drive times (not driving the simulant far enough in the PJM tubes), operational problems could be associated with the simulant "coating," which is simulant exposure on Drexelbrook that permanently influenced its performance.

At times, there was no reliable level instrument (08:59, 2/2/09, LRB 60229, pg. 48). No PJM stroke was observed in UFP-VSL-T01A, and by 09:13, there were no credible level readings from any source in UFP-VSL-T01A (LRB 60229, pg. 49). Creative level control techniques were used in Integrated Test A given the issues with the laser and bubblers. Late on 2/2/09, the UFP-VSL-T01A laser was used for level control, although it was not reading correctly. To control autobatch transfers, it was made certain that

batch transfers that were initiated at the proper tank level in UFP-VSL-T02A level control would be switched to the bubbler (21:20, 2/3/09, LRB 60229, pg. 90). Neither the UFP-VSL-T02A laser nor the bubbler was working. At 21:28, the bubbler was blown out, but with no success. While the bubbler was not accurate, its value was more stable than the laser values. When the estimated permeate had been collected, based on the permeate totalizer, the level control was then switched to the laser for triggering the next autobatch transfer. Once the transfer was complete, the level control was switched back to the bubbler.

In summary, obtaining credible level readings was a persistent problem in Integrated Test A. Having adequate venting in the vessel headspace appears to improve laser performance; it may also prevent splashing slurry on the lens. High-temperature application sensors should be selected, and perhaps it would be best to develop a laser self-cleaning system. It would be a good idea to use multiple sources for measuring levels.

7.0 Conclusions

Integrated Test A demonstrated caustic leaching in the ultrafilter feed preparation vessels UFP-VSL-T01A/B at 98° C. While the outlined process steps in the Test Plan, TP-RPP-WTP-506,^(a) were accomplished, there were several operational difficulties throughout Integrated Test A.

Air entrainment produced unstable filter-loop pump performance. Slurry entrainment in the UFP-VSL-T02A filter loop could have resulted from the use of bubblers, steam ring, and spargers coupled with possible PJM overblowing as well as low-level tank operations that exposed the simulant return line. It is recommended that plant operations avoid prolonged low tank level operations and have an interlock shut down the filter-loop recirculation pumps when a PJM overblow is detected. Future PJM investigations should consider optimizing PJM stroke and average peak velocities at different UFP-VSL-T02A tank levels and slurry properties in efforts to minimize, or prevent, air entrainment caused by PJM overblowing.

Integrated Test A level issues demonstrate that having multiple sources for level indication and control was convenient. However, bubblers plugged frequently, and lasers flatlined on numerous occasions because of aerosols in the vessel headspace and/or exposure to high temperatures. Having adequate venting in the vessel headspace appears to improve laser performance; it may also prevent splashing slurry on the lens. Using laser self-cleaning systems could have improved the laser functionality, and lasers may have worked better if they had been made for high-temperature applications. Bubbler readings were also impacted by the flow from the PJMs and the filter-loop return nozzle because of the bubbler physical placement in UFP-VSL-T02A. In addition, the level indication in PJMs was also problematic, especially under high-temperature conditions.

Backpulsing of the filters was found to be effective for at least temporarily increasing filter permeate flow during post-caustic-leach concentration.

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⁽a) G Josephson, O Bredt, J Young, and D Kurath. 2008. *Test Plan: Pretreatment Engineering Platform (PEP) Testing (Phase I).* TP-RPP-WTP-506, Rev. 0, Pacific Northwest National Laboratory, Richland, WA, 99352.

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Appendix A

Process Instrumentation Summary

Appendix A: Process Instrumentation Summary

Key components for measuring filtration performance at PEP include slurry and permeate flow meters, feed tank (Tank T02A) temperature sensors, and filter-loop pressure sensors. These sensors allow assessment of process conditions driving cross-flow filtration and of the performance of the filters in terms of permeate production. The process parameters of interest are 1) filter axial velocity (or slurry flow rate), 2) filter transmembrane and axial pressure drop, and 3) rate of permeate production. Permeate production is typically corrected for variations in slurry/supernate temperature (as well as variations in transmembrane pressure), and, as such, the process temperature is also of interest.

Table A.1 provides a summary of the PEP instrumentation used to assess slurry flow rates in the filtration loop. The two flow meters listed provide measurement of both suction and discharge flow rates from the circulation loop pumping system. Flow meter FT-0623 operates at the head pressure of the Tank T02A or lower, whereas flow meter FT-0635 operates at high pressure. Because of the pressure differential between flow meters, air entrained in the suction line is likely forced into solution on the discharge side. Some degree of air entrainment is expected from sparging of Tank T02A contents and slurry return. Because magnetic flow meters are sensitive to nonconductive phases like air, the reading on the discharge magnetic flow meter (FT-0635) is typically a few percent lower than that on the suction flow meter (FT-0623) for typical air entrainments observed during PEP runs. The divergence in flow meter readings may become severe if air entrainment becomes significant, but such behavior was not observed during the assessments of filter scaling effects.

Instrument ID	Description	Units
FT-0623	Instrument FT-0623 is a magnetic flow meter used to measure the volumetric flow rate of slurry entering Pump T42A. It is located at the suction to Pump $T42A$.	gpm
FT-0635	Instrument FT-0635 is a magnetic flow meter used to measure the volumetric flow rate of slurry exiting Pump T43A. It is located at the discharge to Pump T43A.	gpm

Table A.1. PEP Instrumentation Used to Assess Slurry Flow Rates Through the Filtration Circulation Loop

Determination of transmembrane and axial pressure differentials requires knowledge of the tubeside inlet/outlet pressures and of the shellside pressure for each filter bundle. Table A.2 provides a summary of the filter bundle instrumentation that provides this capability.

Filter Bundle	Instrument ID	Description	Units
1	PT-0739	PT-0739 provides measurement of the tubeside inlet pressure for filter bundle 1. It is located at the entrance to filter bundle 1.	psig
	PT-0749	PT-0749 provides measurement of the tubeside outlet pressure for filter bundle 1. It is located at the entrance to filter bundle 2.	psig
	PT-0741	PT-0741 provides measurement of the shellside pressure for filter bundle 1.	psig
$\overline{2}$	PT-0749	PT-0749 provides measurement of the tubeside inlet pressure for filter bundle 2. It is located at the entrance to filter bundle 2.	psig
	PT-0759	PT-0759 provides measurement of the tubeside outlet pressure for filter bundle 2. It is located at the entrance to filter bundle 3.	psig
	PT-0751	PT-0751 provides measurement of the shellside pressure for filter bundle 2.	psig
3	PT-0759	PT-0759 provides measurement of the tubeside inlet pressure for filter bundle 3. It is located at the entrance to filter bundle 3.	psig
	PT-0769	PT-0769 provides measurement of the tubeside outlet pressure for filter bundle 3. It is located at the entrance to filter bundle 4.	psig
	PT-0761	PT-0761 provides measurement of the shellside pressure for filter bundle 3.	psig
$\overline{4}$	PT-0769	PT-0769 provides measurement of the tubeside inlet pressure for filter bundle 4. It is located at the entrance to filter bundle 4.	psig
	PT-0779	PT-0779 provides measurement of the tubeside outlet pressure for filter bundle 4. It is located at the entrance to filter bundle 5.	psig
	PT-0771	PT-0771 provides measurement of the shellside pressure for filter bundle 4.	psig
5	PT-0779	PT-0779 provides measurement of the tubeside inlet pressure for filter bundle 5. It is located at the entrance to filter bundle 5.	psig
	PT-0789	PT-0779 provides measurement of the tubeside outlet pressure for filter bundle 5. It is located at the exit from filter bundle 5.	psig
	PT-0781	PT-0781 provides measurement of the shellside pressure for filter bundle 5.	psig

Table A.2. PEP Filter Bundle Pressure Sensing Instrumentation

Permeate mass flow rates are measured by Coriolis flow meters. A summary of these instruments is provided in Table A.3.

Filter Bundle	Instrument ID	Description	Units
	FT-0720	Coriolis flow meter FT-0720 measures permeate production rate	kg/min
		on filter bundle 1.	
	FT-0755	Coriolis flow meter FT-0755 measures permeate production rate	kg/min
		on filter bundle 2.	
3	FT-0765	Coriolis flow meter FT-0765 measures permeate production rate	kg/min
		on filter bundle 3	
4	FT-0775	Coriolis flow meter FT-0775 measures permeate production rate	kg/min
		on filter bundle 4.	
5	FT-0785	Coriolis flow meter FT-0785 measures permeate production rate	kg/min
		on filter bundle 5.	

Table A.3. PEP Coriolis Flow Meters for Permeate Production Rate Measurement

Corrections of permeate production rates for variations in process temperature are made against the temperature of Tank T02A. This vessel is fitted with an array of RTDs to determine the tank temperature profile. All permeate flux corrections were made using the prototypic temperature sensor TTK-0619. Other temperature sensors of interest are installed in thermowells located along the filter loop:

- TT-0791—indicates the inlet temperature to filter bundle 1
- TT-0537—indicates the outlet temperature from filter bundle 5
- TT-0513—indicates the outlet temperature for UFP-HX-T02A
- TT-0515—indicates the outlet temperature for UFP-HX-T03A.

The energy required to pump the slurry also causes temperature rise in the filtration loop. Although the spiral plate heat exchanger (UFP-HX-T02A) removes this heat, temperature differentials (of a few degrees, depending on slurry thickness) still exist between the filtration loop and Tank T02A. While it would be more appropriate to correct filter flux using the filtration loop temperature, temperature sensors in the filtration loop appear to be subject to potential stagnation of the slurry at the sensor thermowells (see the results for the High-Solids Scaling Test in WTP-RPT-185).^(a) Because of these concerns, the tank temperature was selected as the temperature reference for filtration calculations.

The output signal from each of the PEP sensors listed in Table A.1 to Table A.3 was recorded by and stored in the PEP data acquisition system (DAS). The analog-to-digital conversion system has been calibrated to accurately convert the instrument signals, and they were stored in a read-only data file to confirm the integrity of the process data from each test. The recorded data were time stamped by the DAS system so that it could be matched to process data sheets and logbooks. The raw, stored instrument outputs were converted to engineering data with a data interrogation program, which was technically reviewed, validated, and verified according to QA-RPP-WTP-SCP, *Software Contro*l. For simplicity, the system for recording, storing, and converting data will be referred to as the PEP DAS.

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⁽a) NCR 42402.1.

Appendix B

Integrated Test A Sample List
Final Electronic Report File Name CCP WTPSP 755 Int Test A UDS Data Analysis CCP WTPSP 755 Int Test A UDS Data Analysis R1 Shake and Test A and Test B Step001 Date 08 18 2009 CCP WTPSP 755 Int Test A UDS Data Analysis TDP WTP 359 Int Test A RHE Data on PEP Slurry CCP WTPSP 754 Shakedown UDS Data Analysis R1 Shake and Test A and Test B Step001 Date 08 18 2009

1 Shake and Test A and Test B Step001 Date 08 18 2009

TDP WTP 359 Int Test A RHE Data on PEP Slurry

TDP WTP 358 Int Test A PSD Data Analysis

R1 Shake and Test A and Test B Step001 Date 08 18 2009

Appendix B: Integrated Test A Sample List

Final Electronic Report File Name R1 Shake and Test A and Test B Step001 Date 08 18 2009 1 Shake and Test A and Test B Step001 Date 08 18 2009 R1 Shake and Test A and Test B Step001 Date 08 18 2009 1 Shake and Test A and Test B Step001 Date 08 18 2009 R1 Shake and Test A and Test B Step001 Date 08 18 2009 1 Shake and Test A and Test B Step001 Date 08 18 2009 090203-3 and 090203-4 ICP and ICPMS 090219-17 and 0902019-22 ICP and ICPMS 090203-3 and 090203-4 ICP and ICPMS 090219-17 and 0902019-22 ICP and ICPMS 090203-3 and 090203-4 ICP and ICPMS 090219-17 and 0902019-22 ICP and ICPMS 1 Shake and Test A and Test B Step001 Date 08 18 2009 R1 Shake and Test A and Test B Step001 Date 08 18 2009

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R1 Shake and Test A and Test B Step001 Date 08 18 2009

090203-5 and 090203-6 ICP and ICPMS

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090204-7 and 090205-20 ICP and ICPMS

ASR 8343 RAM rev 1

090204-7 and 090205-20 ICP and ICPMS

ASR 8343 RAM rev 1

090204-7 and 090205-20 ICP and ICPMS

TDP WTP 357 Int Test A XRD Data on PEP Slurry

TDP WTP 357 Int Test A XRD Data on PEP Slurry

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TDP WTP 363 Int Test B RHE Data on PEP slurry

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Integrated Test A Sample List

Appendix C

Sampling Event Summary

Integrated Test A Sampling Event Summary

Appendix C: Sampling Event Summary

ESPS Analytical Matrix (2/3/09), Integrated Test A Sampling Event Summary

The test instruction for Integrated Test A used the 1/27/09 version of the sample matrix. However, several changes followed sample matrix 1/27/09. The sampling matrix below summarizes the actual number of samples planned f collection during Integrated Test A. Some of the samples below were not collected as planned because the level in the tank was not high enough or the Coriolis sampling station was out of service—these events are summarized Appendix E.

Due to the unforeseen permeate rate from Filter T01A during post-caustic-leach concentration, the attached sample matrix does not include the additional CD sampling in UFP-VSL-T02A and (10) permeate samples for solids brea observation collected up until the 300th hour (the end of post-caustic-leach filtration). The frequency of post-caustic-leach permeate sampling also changed. After collecting the first permeate sample at the 32nd hour permeate samples were taken 24 hours thereafter. These changes affected process step B.1.7.

Integrated Test A Sampling Event Summary

NOTES:

Leach samples were cooled immediately to prevent further reaction of aluminum in the slurry.

Appendix D

Coriolis Sample Elevations

Appendix D: Coriolis Sample Elevations

'As installed' elevations of the Coriolis samplers were estimated by bubbling air through each sampler leg and measuring the bubbler pressure on the tank's lower bubbler leg calibrated pressure transmitter.

Method: After recording the pressure of the lowest bubbler leg the bubbler tubing was disconnected from the air supply and the supply attached to the Coriolis sampler stand via the low point drain and connecting Teflon tubing with Swagelock fittings. Air was bubbled through the tubing to each sampler leg and the pressure recorded. In UFP-1A and UFP-1B the PJMs were operating and the "lowest" pressure was recorded, when the PJMs were filled. Levels in UFP-1A, UFP-1B, and UFP-2A were measured on LT-0323, LT-0423, and LT-0611, respectively.

The measured pressures were compared with the measured differences in tubing lengths between the bottom-middle and middle-upper to make the final estimate sample elevations. Measured tubing differences were considered absolute and measured elevations were adjusted as much as an inch so that the offsets matched measured lengths.

Below are listed the "expected" sampler elevations based upon initial design and the results of this study:

Conclusions: The 'as installed' sampler levels agree quite well with the design with the following exceptions:

- The UFP-1B outer/middle and outer/high sampling tubing has been switched. (An NCR is being written and the tubing needs to be corrected and the correction date recorded. Samples taken until then are incorrect.)
- The inner low sampler tubes were cut shorter than originally anticipated. Instead of sampling very close (2") from the bottom of the vessel the sample is 10"-11" off the bottom.
- The middle and outer samplers in UFP-1B were installed about 4"-5" lower than initially intended.

The design of the UFP-2A middle/low position was designed to be 2 inches off the vessel bottom at the middle radial location. With a 2:1 elliptical head the elevation rise for the middle sampling location is estimated to be ~3.5" so the measured 6" elevation from the center tank bottom is actually very close.

Appendix E

Integrated Test A Timeline

ortable sampling station was cannibalized, it had no pump. In efforts lect pretest samples, an aluminum pipe was inserted in HLP-VSL-T22 isual observations indicated the pipe had partially dissolved. (LRB pg. 110) Associated Operations Shift Supervisor spoke to operators vent this from happening again and notifications were sent to team the affected samples numbers from the event.

um was used to collect the 2-40 mL pretest samples, by collecting 1L in a 1-Liter container. The 1-Liter was shaken, then an aliquot was d into 1-40 mL containers, the "shake and aliquot" method was red for the second 40-mL sample. (TI pg. 64).

Appendix E: Integrated Test A Timeline

The Test Plan process description Table E.1 is used to outline Integrated Test A. Test instruction and test parameter deviations, and problems are listed for each process step. Sampling events have been omitted from the In narrative for clarity given the large number of sampling events and sample containers. Sampling events and analyses are included on the Integrated Test A Sampling Event Summary, Appendix C. The times of events are based on unless noted otherwise.

Test Step	Test Step	Description/Purpose	Key Process Conditions	Sampling	
B.1.1		Prepare and stage simulant Prepare sufficient quantity of Phase 1 simulant	HLP-VSL-T22 agitator speed set so vessel	Note: The sampling and analyses at this step may have been completed at the start of	The pc
	in HLP-VSL-T22 Completed 1/31/09, 04:56	in HLP-VSL-T22 to complete tests.	is well mixed; recirculate the tank with the	functional testing.	to coll
		Characterize simulant for specification of all simulant-dependent process parameters (e.g., batch volumes, caustic and permanganate additions). Note: This characterization may have been completed at the start of functional testing.	pump.	Collect grab samples from HLP-VSL-T22 at 3 elevations.	and vis
				Analyze grab samples from HLP-VSL-T22 samples for \bullet wt% UDS. Circulate HLP-VSL-T22 with recirculation loop and take three (3) samples from recirculation line. The number of replicate samples was specified by the JTG with input from the data analysis team. Analyze recirculation line sample for $wt\%$ UDS slurry density shear strength (not in replicate) shear stress vs. shear rate (not in replicate) liquid density liquid viscosity (not in replicate) dissolved aluminum dissolved chromium	60108 to prev about 1 Vacuu 500-m poured repeato
				dissolved manganese dissolved oxalate dissolved carbonate free hydroxide dissolved metals liquid anion tracers dissolved AFA constituents (not in replicate) PSD (not in replicate) mineral forms (XRD) (not in replicate)	
				crystal morphology & habit (TEM/SEM) (not in replicate) total aluminum solids oxalate solids total metal solids AFA constituents in the solids solid metal tracers heat capacity (Integrated Test A only) Collect samples of 19M NaOH, 2 M HNO3 and 1 M NaMnO4 and analyze per step A.1.1 if this has not been completed or if fresh batches of chemicals are being used.	

Table E.1. Integrated Test A – Caustic Leaching in UFP-VSL-T01A/B and Oxidative Leaching in UFP-VSL-T02A

ampling just prior to transferring from HLP-VSL-T22 to SL-T01A only collected samples from middle and low elevations, the grab sample could not be collected with instrumentation used (TI).

es were made to the target transfer rates for the simulant and caustic on because the HLP-VSL-T22 flowmeter, FQI-0119, reports a ned transfer rate for both the simulant and caustic, where as the test tion treated these independently. TI and LRB 60108, pg. 109.

1, UFP-VSL-T01A, PJMs were turned on at a tank level of 40+ during the transfer from HLP-VSL-T22 to UFP-VSL-T01A while n Sheet stated to do this at a maximum tank level of 25 inches (TI: eet).

29, 01/31/09, switched from bubbler to laser level control in UFP-01A because laser was reading 96 inches, temperature in vessel is (LRB 60229, pg. 9).

level measurement taken at the end of initial heat for Batch 1 UFP-01A had vessel isolation valve V03129 open- deviation to TI. It was $~50$ minutes prior to reaching final target leach temperature per Run 18:50 on $01/31/09$). (TI).

controller TIC-0325 shut itself off, was put back onto automatic t 19:00, 1/31/09 (LRB 60108, pg. 112).

at end of heatup was 63.1, versus Run Sheet target of 61 ± 2 in (TI: heet).

UFP-VSL-T01A PJM tuning at 98°C was difficult because drive re was not stable. Drive pressure and time setpoint were adjusted

un Sheet PJM nozzle velocity were met $(21:13$ on $\overline{01/31/09}$. (TI).

09:02 through 09:08 (LRB 60229, pg. 20), level control in UFP-01A was switched back and forth between Drexelbrook, bubbler and ecause the noise in the bubblers level values caused high-level alarms lessel. Level control was kept with the Drexelbrook, the most stable. rexelbrook level trend was smoother with the steam-ring air off, then t was on (LRB 60229, pg. 22-24).

on 2/1/09 (LRB 60229, pg. 32) UFP-VSL-T01A pump was turned 1 after it was tripped by a high-high level alarm in UFP-VSL-T01B.

on 2/1/09 (LRB 60229, pg. 35) PJM probes 1601, 1603, and 1605 ot responding to the PJM drive phase, they stayed at value 48 inches or 38 inches for 1605.

ompleting initial transfer from UFP-VSL-T01A to UFP-VSL-T02A, PJMs in UFP-VSL-T02A could not begin because level was too With approval, the filter loop was filled, then proceeded to PJM in UFP-VSL-T02A. (TI).

 $2/1/09$ (LRB 60229, pg. 36) PJM drive time was reduced because vere overblowing.

 $(2/1/09)$; CD Samples were not collected from the Inner/High, Migh, Outer/High, and Inner/Middle sample ports because the CD could not stay primed. (TI).

2/1/09, UFP-VSL-T01B bubblers were cleaned because specific was reading $~4$, during the cleaning, UFP-VSL-T01A pump was , then it was restarted (LRB 60229, pg. 41).

2/1/09 (LRB 60229, pg. 43), UFP-VSL-T01A PJM drive pressures 01 and 1603 were lower than other drive pressures.

17:17:09 (LRB 60229 pg. 39), PJM flange 1805 had an air leak.

Iter-loop draining was completed at $20:40$, $2/1/09$. (TI).

19:00, Bubblers in UFP-VSL-T01B were cleaned because specific was reading 4.1. At 19:35 UFP-PMP-T41A was found off, then it started. The cleaning of the bubblers was believed to have caused a evel spike, which then turned UFP-VSL-T01A pump off (LRB , pg. 120).

on 2/2/09 (LRB 60229, pg. 45) UFP-VSL-T01B bubblers were d to address the high density alarm. At $01:00$, $2/2/09$ (LRB 60108 , $p₂$) UFP-PMP-T41A was turned back on – believed to have been I from the UFP-VSL-T01B bubbler cleaning.

 $0.2/2/09$ (LRB 60108, pg. 123) temperature setpoint for HX-T02A was decreased because temperature in the tank rose to

 $2/2/09$ (LRB 60108, pg. 123) temperature setpoint for UFP-HXwas increased because temperature in the tank was 22.1° C.

07:08, 2/2/09 (LRB 60229, pg. 46) UFP-VSL-T01A PJM settings were ed to meet Run Sheet targets.

PJM probes in UFP-VSL-T02A were not working properly on $2/2/09$, 60229, pg. 47) and PJM drive pressure increased without having adjustments.

 $2/2/09$ (LRB 60229, pg. 48) no PJM stroke was observed in VSL-T01A, by 09:13 there was no credible level readings from any e in UFP-VSL-T01A (LRB 60229, pg. 49).

on 2/2/09, PJM probes in UFP-VSL-T02A started working again 60229, pg. 49).

UFP-VSL-T02A PJM settings were adjusted because they were owing (LRB 60229, pg. 51).

Switched to laser level control in UFP-VSL-T01A (LRB 60229,

 $1/2/2/09$ (LRB 60229, pg. 54) PJM drive pressures for $VSL-T01A 1601$ and 1605 still looked abnormal (low).

13:36, 2/2/09 (LRB 60229, pg. 54) Filter-loop pumps shut off due to high bressure alarm for Filter T02A, pumps were restarted minutes later.

2/2/09 (LRB 60229, pg. 57) UFP-VSL-T01A PJMs sounded like vere overblowing; minutes later $(14:40)$, the drive time was changed.

 $1,2/2/09$ (LRB 60229, pg. 59) A 23rd auto batch from UFP-VSL-T01A iggered by an erroneous laser level in UFP-VSL-T02A.

16:38, 2/2/09 (LRB 60229, pg. 60) The low-low level alarm was triggered P-VSL-T02A, therefore pumps and PJMs were turned off.

/09, between 16:00 and 16:50 (LRB 60229, pg. 60) UFP-VSL-T02A as unstable, causing low-low level alarms, which then caused the and PJMs to turn off, which then triggered high transmembrane re (TMP) alarms- the TMP would decrease once pumps were ed. Low-low level alarms would occur whenever filter-loop flow rate ed 83 GPM (16:50, 2/2/09, LRB 60229, pg. 62). Auto batch ons were suspended until conditions stabilized.

 $2/2/09$ (LRB 60229, pg. 62). In response to the low low level alarms ²-VSL-T02A, the low-low interlock delay was changed to 3 seconds all the UFP vessels, and the laser out of range filter low to 0 inches.

2/09 UFP-VSL-T01A laser was used for level control although it t reading correctly. PJMs were turned off in UFP-VSL-T01A because of overblowing, level in tank was \sim 14 inches per laser. (LRB pg. 124).

es were made to the target transfer rates for the simulant and caustic an because the HLP-VSL-T22 flowmeter, FQI-0119, reports a ned transfer rate for both the simulant and caustic, where as the test tion treated these independently. (TI).

1/09, steam injection was inadvertently used to do the initial heating P-VSL-T01B Batch 2, instead of using UFP-HX-T04B. Forty-seven s of steam injection were completed, and luckily there was not more 3 inches (per laser) of condensation added during this time based on level measurements. UFP-HX-T04B was used to reach the target neat-up temperature per the Run Sheet (22:54 on 1/31/09, LRB pg. 117).

00:50 and at 02:43, Steam controller TIC-0425 shut itself off. It started and ramp time revised as appropriate (LRB 60108, pg. 118).

mples at 10° C below final heat leach temperature were only taken he low and middle elevations since the high elevation samples could ep the CD sample pump primed- could have been influenced by injection valve fully open $(02:31$ on $2/1/09)$. (TI).

02:37, UFP-VSL-T01B level control was switched from laser to r, because laser was reading 95 inches, temperature in vessel is (LRB 60229, pg. 14).

07:45 the inner high CD sample was not collected for $T=4$ hour ampling, not able to keep pump primed (LRB 60229, pg. 19).

on 2/1/09, LRB 60229, pg. 19 UFP-VSL-T01B CD sampler is not g at all.

/09, 10:33 (LRB 60229, pg. 22), the UFP-VSL-T01B CD sampling was handed over to facilities to fix the pump. The vacuum system he CD sampling station was used in the mean time to collect samples ad samples were for leach hours 5, 6, and 7 (LRB 60229, pg. 34). the repair, the vent was open, which did cause higher steam use than e vent closed (11:38, 2/1/09; LRB 60229, pg. 25). The 8 and 9 hour IFP-VSL-T01B CD samples were not taken because the CD sample pump was being repaired. Instead the 8-hour CD samples were taken α hour leach time, and an additional 8.7 L was collected from the niddle CD port in UFP-VSL-T01B. (TI).

on 2/1/09 (LRB 60229, pg. 32) UFP-VSL-T01A pump was turned after it was tripped by a high-high level alarm in UFP-VSL-T01B. bbler was flushed, but still gave a specific gravity of 1.09 (13:12, LRB 60229 pg 32).

13:10 (LRB 60229 pg. 32) UFP-VSL-T01B CD sample station it tag out was removed, and UFP-VSL-T01B vent valve was closed (LRB 60229, pg 29).

 $2/1/09$, (LRB 60229 pg. 32), The automatic temperature control reached 101.5°C in UFP-VSL-T01B.

2/1/09, (LRB 60229, pg. 33), Three sets of 2.9 Liter of inhibited were added to compensate for those during leach hours 8, 9, and 10.

PJM drive time was reduced since minutes earlier PJMs in UFP- $V01B$ were overblowing (LRB 60229, g. 36).

 $2/1/09$ (LRB 60229, pg 41) temperature in UFP-VSL-T01B dropped t_z , steam controller put back into "auto".

UFP-VSL-T01B bubblers were cleaned because specific gravity was ≈ 4 , during the cleaning, UFP-VSL-T01A pump was tripped, then it started (LRB 60229 , pg. 41).

19:00, Bubblers in UFP-VSL-T01B were cleared because specific was reading 4.10. At 19:35 UFP-PMP-T41A was found off, then it started. The cleaning of the bubblers was believed to have caused a vel spike, which then turned UFP-VSL-T01A pump off (LRB pg. 120).

If Liter of inhibited water was added $(19:10, 2/1/09)$ at the 16 hour me instead of the 15 hour leach time. (TI).

pg. 122 of LRB 601088, 23:45, laser in UPF-VSL-T01B appears to king now. Level in UFP-VSL-T01B at the end of final heat-up was es instead of 64 inch maximum level per Run Sheet; with approval ded with test (TI: Run Sheet).

none. post-caustic-leach cooling in UFP-VSL-T01B batch 2 to filtration temperature was accomplished by slowing the speed of UFP-PMP-T41B to 1.2 GPM instead of aligning and by-passing HX-T05B, per test instruction, as needed to meet cooling curve target. UFP-PMP-T41B speed was increased periodically to flush out settled $(TI, pg. 103)$.

RB 60229, pg. 43, UFP-VSL-T01B PJMs were left off until 20:45, aking a stable level measurement at 20:13.

on 2/2/09 (LRB 60229, pg. 45) UFP-VSL-T01B bubblers were d to address the high density alarm. At $01:00$, $2/2/09$ (LRB 60108 , 2) UFP-PMP-T41B was turned back on, was thought to have turned om the bubbler cleaning.

on 2/2/09 (LRB 60229, pg. 46) UFP-VSL-T01B bubblers were d again.

:39 through 18:50, 2/2/09 (LRB 60229, pg. 63) UFP-VSL-T02A bubblers were cleaned.

2/3/09 (LRB 60108, pg. 124), UFP-VSL-T01B PJM drive time reased because of overblow.

2/3/09 (LRB 60229, pg. 70) UFP-VSL-T01B laser triggered level rms and then it stayed at a value of 55.5 inches. Consequently, level ntrol was switched to bubbler.

 $(31, 2/3/09$ (LRB 60229, pg. 71) Drive time adjustments were made UFP-VSL-T01B PJMs because they were overblowing. After ustment, PJM 1715 produced a short stroke (07:02, 2/3/09, LRB 229 , pg. 73).

07:15 UFP-VSL-T02A bubblers were blown out to switch from laser to bbler autofill level control (no indication of switch occurring at this though). At 08:35, UFP-VSL-T02A bubblers were blown out again et to a high density alarm (LRB 60229, p. 74).

, 2/3/09 (LRB 60229, pg. 78) UFP-VSL-T02A laser was not working. 2/3/09 (LRB 60229, pg. 78) UFP-VSL-T01B laser no longer flined but still not working.

2/3/09 (LRB 60229, pg. 89) UFP-VSL-T01B laser was flatlining. $2/3/09$ (LRB 60229, pg. 89) UFP-VSL-T02A pumps were shut off to laser level spike.

 $2/3/09$ (LRB 60229, pg. 90) A batch transfer was mistakenly the gered because of low laser level in UFP-VSL-T02A.

2/3/09 (LRB 60229, pg. 90) Neither the UFP-VSL-T02A laser nor bubbler was working. At 21:28 the bubbler was blown out, but no success.

2/3/09 UFP-VSL-T02A pumps shut off, triggered by bubbler lowalarm –laser was reading high-high (LRB 60108 , pg. 126)-, and rger air was lost as well. Pumps were restarted after bubbler and er cleaning, 22:05 (LRB 60108, pg. 127).

:03, UFP-VSL-T02A laser was working after instrument technicians aned the lens (was dirty). During the cleaning, a batch transfer was t_{gered} mistakenly (LRB60229, pg. 91).

22:09, 2/3/09 (LRB 60229, pg. 91) UFP-VSL-T02A bubblers were cleaned. 29, 2/3/09 (LRB 60229, pg. 92), the UFP-VSL-T01B laser was

nt, by 23:03, instrument technicians cleaned the lens, but the laser l did not work (low return signal)—foam may be to blame (LRB 108, pg. 127). The level control was the bubbler. 0.2/4/09 (LRB 60108, pg. 129) The batch transfer counter read 34 ches although it was accidentally reset after making \sim 10 transfers.

See added rows below for subsequent batch descriptions

atch 3 UFP-VSL-T01A, PJM operation was initiated at a tank level of hes during transfer from HLP-VSL-T22 to UFP-VSL-T01A while heet prescribed turning them on at 25 inch maximum tank level. (TI: heet).

ges were made to the target transfer rates for the simulant and caustic on because the HLP-VSL-T22 flowmeter, FQI-0119, reports a ned transfer rate for both the simulant and caustic, where as the test ction treated these independently. (TI).

the number of samples collected ω "10 min after transfer is complete" into VSL-T01A second caustic batch in 01A were not immediately put in ter bath. Sampling Team Lead notified the team about affected e numbers and reminded staff how samples at elevated temperature I be put in water bath as soon as possible. Email by O. Bredt on

2/3/09 (LRB 60108, pg. 125) UFP-VSL-T01A bubblers were d.

atch 3 UFP-VSL-T01A steam-ring air purge rate was increased at a emperature of 84° C and not lower temperature target of 85° C per Run (TI: Run Sheet) However, LRB 60229, pg. 69 at 01:55 on 2/3/09, high temperature steam-ring setting would not be changed until final temperature was reached so that the heatup ramp time would not be \mathbf{d}

2/3/09 (LRB 60229, pg. 79) Level control in UFP-VSL-T01A was swed to Drexelbrook because (10:16) high high level alarms occurred used UFP-PMP-T41B to stop. Laser in UFP-VSL-T01A was le and bubblers were cleaned with hot steam condensate (10:58). setting changed the "high out of range" level to 85 inches in UFP-T01A to filter out erroneous laser readings.

2/3/09 (LRB 60229, pg. 83) UFP-VSL-T01A bubblers were cleaned.

2/3/09 (LRB 60229, pg. 90) UFP-VSL-T01A laser was flatlining. $2/3/9$ (LRB 60229, pg. 91) UFP-VSL-T01A bubblers were cleaned. 3 UFP-VSL-T01A, Stable level measurements and sample densities aken periodically to monitor tank level and validate bubbler levels ample densities – including an additional CD sample (inner/low).

3 UFP-VSL-T01A, during initial cool-down with UFP-HX-T05A, the

- d water valve was set to 40% open $@$ 20:55, 2/3/309. (TI pg. 121) to at exchanger.
- 2/4/09 (LRB 60229, pg. 97-98) UFP-VSL-T02A pumps shut off, a not known. Pumps were restarted. PJMs also shut off, although this served at 13:25.
- 13:11, 2/4/09 (LRB 60229, pg. 98) High vibration was noticed on UFP-T42A, pump speed was adjusted and vibration decreased.
- 2/4/09 (LRB 60229, pg. 99) The low-low alarm for UFP-VSL-T01B shut off UFP-PMP-T41B, while bubbler was on level control. switched to laser, pump shut off.
- 15:17:17, 2/4/09 (LRB 60229, pg. 100), There was a concern that the VSL-T01A auto batch transfer was not working, therefore it was reset.
- $1,2/4/09$ (LRB 60229, pg. 101), the UFP-VSL-T02A laser stopped ng, the pumps shut off, the level control switched to the bubbler, and mps were then restarted.
- 2/4/09 (LRB 60229, pg. 105) UFP-VSL-T01B laser was flatlining, level control was switched to the bubbler.
- 2/4/09, Test Director email: Since permeate rates during post**c-leach concentration - using the first filter bundle, were lower than expected (although they exceeded the design rate for the WTP), backpulsing was applied through the course of filtration. Backpulse guber 6 are evolved, however. Permeate performance after backpulsing was the same for all backpulse variations. See backpulse description,**
- 2/4/09 (LRB 60229, pg. 107) UFP-VSL-T02A laser was not working nd was not consistent in value readings.
- , 2/5/09 (LRB 60229, pg. 109) UFP-VSL-T01A PJM drive time was ed because of overblow.
- , 2/5/09 (LRB 60229, pg. 111), UFP-VSL-T01A was overblowing, ore PJM drive time and vacuum times were adjusted.
- 2/5/09 (LRB 60229, pg. 112), UFP-VSL-T02A laser started working, were agreeable with bubbler level.
- $12/5/09$ (LRB 60229, pg. 119) Chemical addition vent flange was to improve laser performance in UFP-VSL-T02A with approval esting and operations -bleed air should flush vent space aerosols. By UFP-VSL-T02A was changed to laser level control based on ved laser performance.
- 05:50, 2/6/09 (LRB 60229, pg. 129), UFP-VSL-T02A PJMs were restarted hutting down due to a low laser reading.
- 2/6/09 (LRB 60229, pg. 130), UFP-VSL-T02A high-high laser level alarms triggered switching the autofill level control to the bubblers.
- $0.2/6/09$ (LRB 60229, pg. 130) the chemical addition port flange was red to alleviate laser unstable readings.
- 09:28, 2/6/09 (LRB 60229, pg. 130) UFP-VSL-T02A valve V06149 was

- d fully to clear the headspace, which helped the laser read well. By level control was back to the laser.
- 2/6/09 (LRB 60229, pg. 130), UFP-VSL-T02A bubbler was cleared, nsity still read high.
- 10:24, 2/6//09 (LRB 60229, pg. 130), Filter T01A transmembrane pressure mporarily greater than 45 psi.
- 2/6/09 (LRB 60229, pg. 132), UFP-VSL-T02A plugged uppers ers were cleaned - a reduction in flow was observed the night before.
- 13:23, 2/6/09 (LRB 60229, pg. 132) Filter T01A permeate sample was ted, it was missed at 10:00.
- $12/6/09$ (LRB 60229, pg. 136), Backpulsing program change was eted. The change allows the user to enter desired dead band pressure.
- 2/6/09, (LRB 60229, pg. 138) UFP-VSL-T01A PJMs were turned off since level alarms (low) occurred. After several PJM parameter ments were made, PJMs were turned back ON and were ran about $\frac{1}{2}$ with vacuum slope at 0% and the hold phase re-enabled.
- $2/7/09$ (LRB 60108, pg. 150) After transferring the 49th batch from UFP-VSL-T01A to UFP-VSL-T02A, the PJMs were kept off for the last transfer, and UFP-VSL-T01A was recirculating at $~6$ GPM.
- 2/7/09 (LRB 60229, pg. 142) Ten gallons were transferred from UFP-VSL-T01A to UFP-VSL-T01B to bring level to 10 inches in UFP-VSL-T01A (while auto batching from UFP-VSL-T01B to $VSL-T02A$).
- atch 4 UFP-VSL-T01B, PJM operation was initiated at a tank level of hes during transfer from HLP-VSL-T22 to UFP-VSL-T01B while heet prescribed turning them on at 25 inch maximum tank level. (TI: Sheet).
- ges were made to the target transfer rates for the simulant and ic addition because the HLP-VSL-T22 flowmeter, FQI-0119, ts a combined transfer rate for both the simulant and caustic, as the test instruction treated these independently. (TI).
- 2/4/09 (LRB 60229, pg. 105) UFP-VSL-T01B laser was flatlining, level control was switched to the bubbler.
	- 19:49, 2/4/09 (LRB 60229, pg. 106) UFP-VSL-T01B PJM drive time was ed because of overblowing.
	- 4, 19:46 on 2/4/09 (LRB 60108, pg. 130) PJM drive time adjustments made because UPF-VSL-T01B PJMs were overblowing.
	- Batch 4, at 00:34, 2/5/09, (LRB 60229, pg. 108) UFP-PMP-T41A shut for no apparent reason, this also caused steam injection in $VSL-T01B$ to shut off, temperature dropped to 95.6°C- steam on was restarted at 00:44. A level spike, 09:14, in UFP-VSL-T01B e cause of the pump and steam injection shutdown (LRB 60229, pg.

2/5/09 (LRB 60229, pg. 111) UFP-VSL-T01B bubblers were

:31 2/5/09 (LRB 60229, pg. 115) UFP-VSL-T01B PJM condensate ak repair was completed, PJMs turned off and on during the repair.

ble to collect the inner/high CD sample for $T=12$ hour leach in UFP-T01B, level in vessel was not high enough to keep pump primed. $(LRB 60229, pg. 116).$

5/09, 11:07, steam pressure was lost by the boiler tripping offline due ozen sensing line. Pressure was restored by 11:14, lowest rature during the pressure loss was 96.8° C (LRB 60229, 116 and

2/7/09 (LRB 60229, pg. 140), UFP-VSL-T01B bubblers were

2/7/09 (LRB 60229, pg. 150) 2 Auto batches occurred within ~35 minutes of each other for unknown reason in UFP-VSL-T01B.

18:18, 2/7/09 (LRB 60229, pg. 151) UFP-VSL-T01B PJMs stuck in vent they were stopped and restarted.

2/7/09 (LRB 60229, pg. 152) UFP-VSL-T01B pump turned back on after a low-low laser level shutdown.

2/7/09 (LRB 60229, pg. 152) Level control was switched to bubbler in UFP-VSL-T01B.

 $2/7/09$ (LRB 60229, pg. 153) PJM drive time adjustments were made P-VSL-T01B because it was overblowing.

2/7/09 (LRB 60108, pg. 154) One of the lower spargers rotameters olated since flow rate was decaying with time, however target total vas kept.

2/8/09 (LRB 60229, pg. 155), It was observed that the HMI ew permeate totalizer sums permeate rates from all 5 filters, even only one (1A) was being used. Consequently the negative values yed on those not used produced a significant value over a long period of time. The individual Filter T01A totalizer should be used to determine ate permeate total. $(LRB 60108, pg. 156)$.

2/8/09 (LRB 60108, pg. 157) UFP-VSL-T02A PJMs were restarted hey were shut off for unknown reason.

2/8/09 (LRB 60229, pg. 156) PJM drive times adjustments were to UFP-VSL-T01B PJMs due to overblowing.

13:26, 2/8/09 (LRB 60229, pg. 161-162) Permeate rate was cycling, every μ inutes. By 16:20, pump vibration was heard (LRB 60229, pg. 163). speed and backpressure (valve 0609) adjustments were made up until $2/8/09$, to alleviate pump vibration. By 21:02, permeate rate was "normal", $(LRB 60229, pg. 164-167)$. Appears the "clog" broke free (1:14, 2/8/09, LRB 60108, pg. 160).

09:13, 2/9/09 (LRB 60230, pg. 3) UFP-PMP-T43A loose wire connection paired, the pump revolutions per minute appeared on the HMI again.

10:45, 2/9/09 (LRB 60230, pg. 3) UFP-VSL-T02A CD sampling station

ut back in service after completing gauge work (09:35).

2/9/09 (LRB 60230, pg. 4) UFP-VSL-T01B PJMs were turned off level is \sim 15 inches) to prevent low-low level alarm, relying on tank ulation for mixing.

Batch 4, 2/9/09, 16:24, P. Valdez email: The PJM simple mode code had a husing the vacuum time to remain at the last "corrected" value when hk level compensation feature is enabled (the code does not compute a orrected time when feature is disabled). The corrected code was l after completion of the Integrated Test A and in the meantime, the evel compensation should always be enabled but a value of zero entered to disable the feature.

2/9/09 (LRB 60230, pg. 6) Autofill was switched back to VSL-T01B as the source tank after transferring, by mistake, one batch JFP-VSL-T01A.

19:05, 2/9/09 (LRB 60230, pg. 7) UFP-VSL-T02A steam ring shows reddish moisture in it.

, 2/9/09 (LRB 60108, pg. 160) Steam-ring air purge flow rate was ed (0.194 kg/min) to meet target flow rate (0.14 kg/min).

2/9/09 (LRB 60230, pg. 7) 40 Liters of permeate samples were ted, per the Test Director request $(16:40, 2/9/09; LRB 60230, pg. 6)$.

atch 5 UFP-VSL-T01A, PJM operation was initiated at a tank level of thes during transfer from HLP-VSL-T22 to UFP-VSL-T01A while heet prescribed turning them on at 25 inch maximum tank level. (TI: heet).

ges were made to the target transfer rates for the simulant and caustic on because the HLP-VSL-T22 flowmeter, FQI-0119, reports a ned transfer rate for both the simulant and caustic, where as the test ction treated these independently. (TI).

5 UFP-VSL-T01A steam-ring air purge flow rate was adjusted to the emperature setting at \sim 1.5 hours of leaching. (18:58, 2/7/09, TI, pg.

Batch 5 UFP-VSL-T01A added 2.9 Liters of inhibited water before eting the 16 hour leach sampling, while the Run Sheet specified inhibited water at hours 1 through 15. (TI) .

5 14:16, 2/7/09 (LRB 60229, pg. 148) Drive time was reduced for UFP-VSL-T01A PJMs after an overblow was heard.

2/7/09 (LRB 60229, pg. 149) Steam injection was re enabled in UFP-VSL-T01A after it was found in manual mode.

2/7/09 (LRB 60229, pg. 150) UFP-VSL-T01A PJMs locked

the vent phase.

- 2/7/09 (LRB 60229, pg. 150) Leaching temperature in UFP-VSL-T01A was 103.8° C.
	- 17:40, 2/7/09 (LRB 60229, pg. 150) UFP-VSL-T01A PJMs locked up in the hase at 17:36, they were stopped and restarted.
	- 2/8/09 (LRB 60229, pg. 158) The inner/high CD sample in $SL-T01A$ was not collected at the leach $T=16$ hours, because prime ost in the CD pump.
	- 16:40, 2/9/09 (LRB 60230, pg. 6) Per the Test Director requests, 20-L and 40-L permeate samples from UFP-VSL-T01A to be collected and p carboys.
	- $2/9/09$ (LRB 60230, pg. 6) Per the Test Director request, a 20 Liter FP-VSL-T01A inner/middle CD port sample was collected.
	- 2/10/09 (LRB 60230, pg. 9) UFP-VSL-T01A PJM drive time nents were made due to overblowing.
	- 2/10/09 (LRB 60230, pg. 11) UFP-VSL-T02A PJM parameters were enly adjusted to values for UFP-VSL-T01B adjustments, this caused bowing in UFP-VSL-T02A, pump vibration, and unstable filter-loop tes. PJM parameters were corrected for both vessels.
	- 06:30, 2/10/09 (LRB 60230, pg. 13) UFP-PMP-T41A speed adjustments ade to achieve target transfer rate when auto batching to SL-T02A; night shift may have ran with below-target transfer rates.
	- 2/10/09 (LRB 60230, pg. 13) UFP-VSL-T02A PJMs overblew, g pump vibration (noted since 06:49). PJM vacuum time was
- 2/10/09 (LRB 60230, pg. 15) Confusion on the UFP-VSL-T01A batch counter value.
	- 2/10/09 (LRB, 60230, pg. 16) UFP-VSL-T01A PJM drive adjusted overblowing.
	- (LRB 60108, pg. 163) UFP-VSL-T01A PJM drive time was sed at $17:50$ and $20:50$ due to overblowing.
	- 2/10/09 (LRB 60230, pg. 18) Auto batch transfer UFP-VSL-T02A let a 47.3" due to a 2.6" level offset adjustments made to Run similar adjustments made to UFP-VSL-T01A/B).
	- 2/10/09 (LRB 60230, pg. 19) Electricians are working on ature sensor TI-0791 (upstream from Filter T01A).
	- 2/10/09 (LRB 60230, pg. 19) Drexelbrooks in both UFP-VSL-T01A FP-VSL-T02A were not working.
- 2/10/9 (LRB 60230, pg. 20) Both pumps on UFP-VSL-T01A/B have small oil leaks.
	- 2/10/09 (LRB, 60230, pg. 20) UFP-VSL-T01A PJM drive adjusted overblowing.
	- $2/10/09$ (LRB 60108, pg. 161), the following 20-Liter samples were ed: A_00PF1_007_XX_2758_CUF_4, through 2759 and

A_01AIM_13c_XX_2760_CUF_4.

40, 2/10/09 (LRB 60230, pg. 21) While on laser level control, there high-high level which shut down the pump, so then it was switched to r level control, which gave a low-low level pump shut down. At level control in UFP-VSL-T01A was switched to laser from bubbler bening V03109 to help laser readings. Technician was working on UFP-VSL-T01A laser.

2/10/09 (LRB, 60230, pg. 22) UFP-VSL-T01A PJM drive adjusted overblowing.

 $2/10/09$ (LRB, 60230 , pg. 22) Switched to bubbler level control in SL-T01A while technician made modification to laser panel. es later, autofill from UFP-VSL-T01A to UFP-VSL-T02A was ed for the laser modification. By 21:03, level control was back to laser and autofill re-enabled.

 $en 21:00$ and $21:47$, $2/10/09$ (LRB 60230, pg. 22) high-high level in UFP-VSL-T01B caused steam injection, PJMs, and pumps in SL-T01A/B to shut off-one of the instances was during an auto batch r from UFP-VSL-T01A to UFP-VSL-T02A; systems were restarted.

bressure guidance was provided by a Test Director via email on If at 09:51, because axial pressure drop was increasing through the of post-caustic-leach concentration $(26.8 \text{ psig is the WTP max})$ axial differential pressure). If and when the axial pressure drop s 25 psig, permeate production would resume, CD samples from SL-T02A should be taken and analyzed for rheology measurement, mple collection/analysis would be repeated at every 10% axial re drop increase of 25 psig.

1/09, 12:49 (LRB 60230, 26-27) - A problem was noted with the the totalizers on the HMI, specifically FQIC $\overline{0720}$ - when the $\text{er reached } 8192 \text{ kg}$, it stopped incrementing. Before fixing this ms users were told to log the value of the totalizer in the LRB when it ches 7500 kg, then reset the totalizer on the HMI. The DAS data is ted by this issue. This was fixed in early afternoon, same day.

 $2/11/09$ (LRB 60230, pg. 28) One of the spargers was plugged.

 $2/11/09$ (LRB 60108, pg. 166) All but one PJM Drexelbrook in each -VSL-T01A and UFP-VSL-T02A were flatlining.

 $2/12/09$ (LRB 60108, pg. 167) To investigate the PJM Drexelbrook ng, the vacuum slope compensation for UFP-VSL-T01A was set to en returned to $2.2\frac{\sqrt{6}}{n}$. While the tank level change did not change M cycle, the levels at which the change occurred were lower at 2.2%

 $2/12/09$ (LRB 60230, pg. 29) All PJM Drexelbrooks in UFP-VSLand UFP-VSL-T02A except one in UFP-VSL-T01A were flatlining.

6:14, 2/12/09 (LRB 60230, pg. 31) UFP-VSL-T01B pump was restarted, rned off by bubbler cleaning at 05:42 for UFP-VSL-T01A/B.

03:30, 2/12/09 (LRB 60230, pg. 30) sparger FIC-1943 was plugged.

07:05, 2/12/09 (LRB 60230, pg. 31) Sparger FIC-1943 was closed because plugged, the other rotameters were also flowing below Run Sheet

09:47, 2/12/09 (LRB 60230, pg. 33) UFP-VSL-T02A bubblers were blown an flushed after the 09:17 cleaning did not work.

11:20, 2/12/09 (LRB 60230, pg. 33) UFP-VSL-T02A sparger, nozzle 31, ushed to clear the plug.

2/12/09 (LRB 60230, pg. 33) UFP-VSL-T02A bubblers were blown

een 13:30 and 13:46, 2/12/09 (LRB 60230, pg. 34) Low level alarms P-VSL-T01A turned pump and PJMs off. Pump and PJMs were red and PJM parameter adjustments were made.

2/12/09 (LRB 60108, pg. 168) Filter-loop pumps were vibrating than normal and permeate rates are cycling, could indicate plugging.

 $12/12/09$ (LRB 60108, pg. 168) An alarm was added to all three ultra essels to signal when PJMs turn off due to a low level alarm per laser

 $1,2/12/09$ (LRB 60108, pg. 168) Pipefitters flushed UFP-VSL-T02A er nozzles 32 and 36 with \sim 100 mL (each) of steam condensate to nt plugging.

6, 04:08, 2/10/09 (LRB 60230, pg. 10), Caustic addition rate was ased to target level, from 61 kg/min to 15 kg/min. Batch 6 UFP-VSLexceeded initial heatup temperature after caustic addition to the top of ssel, therefore with approval, vessel contents were cooled using the xchanger to meet initial heatup temperature target per Run Sheet (TI for heating to initial temperature were not completed).

ges were made to the target transfer rates for the simulant and caustic on because the HLP-VSL-T22 flowmeter, FQI-0119, reports a ned transfer rate for both the simulant and caustic, where as the test ction treated these independently. (TI)

07:44, 2/12/09 (LRB 60230, pg. 31) All 3 steam-ring rotameters showed brown fluid in the line.

05:05, 2/10/09 (LRB 60230, pg. 11), UFP-VSL-T01B PJM parameters were ed due to overblowing.

, $2/10/09$ (LRB 60230, pg. 15) Level control in UFP-VSL-T01B was ed to laser from bubbler.

09:10, 2/10/09 (LRB 60230, pg. 16) UFP-VSL-T01B PJM drive ments were made due to overblowing.

ner/high CD sample was not collected because the sample port was bmerged for the 10°C below final leach temperature target sampling $(TI, pg. 281)$. (TI) .

2/10/09 (LRB 60230, pg. 16) The UFP-VSL-T01B bubbler was ed, but there was no sign of improvement.

12:11, 2/10/09 (LRB 60230, pg. 17) UFP-VSL-T01B stable level

rement at 98° C did not last 2 minutes as requested in test instruction.

2/10/09 (LRB 60230, pg. 17) UFP-VSL-T01B PJM vent valves were after they were closed at 12:45 for PJM tuning at 98° C.

2/10/09 (LRB 60230, pg. 21) UFP-VSL-T01B bubblers were , however this caused a high-high level alarm in UFP-VSL-T01Anp was restarted at 18:55.

2/10/09 (LRB 60230, pg. 21) UFP-VSL-T01B steam controller was manual since temperature reached to 103° C while it was on automatic

en 21:00 and 21:47, 2/10/09 (LRB 60230, pg. 22) High-high level in UFP-VSL-T01B caused steam injection, PJMs, and pumps in $VSL-T01A/B$ to shut off- one of the instances was during an auto ransfer from UFP-VSL-T01A to UFP-VSL-T02A; systems were

2/11/09 (LRB 60230, pg. 26) UFP-VSL-T01A level control switched aser (funnel valve was closed prior to switch) to Drexelbrook.

2/12/09 (LRB 60230, pg. 37) Cleared sparger nozzles 32 and 33.

 $2/13/09$ (LRB 60230, pg. 40) HMI operated quicker after a number ications were closed because it was operating slowly.

12:00, 2/13/09 (LRB 60230, pg. 40) UFP-VSL-T01B batch #10 transfer, evious transfers to UFP-VSL-T02A was done at a rate of 5.2 GPM, re UFP-VSL-T01B pump speed and recirculation valve adjustments hade to bring recirculation and transfer rates to targets, 5.5 and 6.9 respectively.

eak from Filters T02A and T04A permeate lines to pulse pot UFP-2A was observed on 2/4/09 (LRB 60229, pg. 94 at 08:15). an, per email on $2/13/09$, was to repair it during maintenance outage. 40 on $2/13/09$ (LRB 60230, pg. 42) via the filter sample pressure , it was observed that permeate lines for Filters T02A and T04A ≈ 200 psi, while others were ≤ 12 psi, hence the leaks. Per E. Dresel on 2/13/09: With approval, control valve from pulse pot UFP-PPto UFP-VSL-T62B was opened to bleed off the pressure. ently, the pulse pot UFP-PP-T02A manual isolation $(VV70164)$ and omatic valves were not isolating the air pressure source. Manual on valves VV70164 and VV70162 were closed to slow the leak (LRB pg. 43). Permeate line sample gauges were left aligned to monitor essure.

 $2/13/09$ (LRB 60235, pg. 3) The axial pressure drop was ntarily above 25 psi; therefore, a UFP-VSL-T02A middle/low CD was collected for rheology analysis per the Test Director tions: continue filtering even when axial pressure drop exceeds 25 Filter T01A, collect samples at 25 , 27.5 , 30 psi, and 10% of 25 psi. because was temporarily at 25 psi ω 21:50, but dropped back to 23.6 $21:55.$

 $2/14/09$ (LRB 60235, pg. 4) UFP-VSL-T01B vent was closed after it mporarily opened to verify that laser performance would improve,

- was alarming on high levels, this stopped with vent open-VSL-T01B on bubbler level-control (LRB 60230, pg. 44).
- 0.2/14/09 (LRB 60230, pg. 45) UFP-VSL-T02A upper spargers were d successfully.
- $0.2/14/09$ (LRB 60230, pg. 45) Brown fluid is seen in the 3 steam-ring rge rotameters, mostly in the UFP-VSL-T02A rotameter.
- 2/14/09 (LRB 60230, pg. 46) UFP-VSL-T02A bubblers were cd, filter-loop pump settings were changed to minimize vibration.
- en 08:00 and 08:17, 2/14/09 (LRB 60230, pg. 46 -47) VSL-T02A PJM and pump settings were adjusted to address potential low concerns causing instable filter-loop pump operations. However :54 bubbler cleaning was believed to have caused for pump vibration.
- $1/2/14/09$ (LRB 60230, pg. 51) Approval was given to proceed with testing after aligning, without draining, Filters T02A through T05A.
- approval, inhibited water was added to compensate for the greater xpected tank level decrease after aligning the five filter bundles in ration for the tracer test. (TI).
- loop flow rate was maintained at 90 ± 5 GPM, as opposed to the Run target of 109 ± 10 GPM. (TI: Run Sheet)Per LRB 60230, pgs. 50ter-loop pump vibration, unstable filter-loop flow rates, PJM tuning, ransmembrane and axial pressures- rheology sample taken at 19:11 he UVP-VSL-T02A middle/low port - 2/14/09 (LRB 60230, pg. 55) problematic for initiating the tracer test. Flowmeter FI-0635 stream from UFP-PMP-T43A) read $~107$ GPM while flowmeter am from UFP-PMP-T42A, FI-0623, read ~140 GPM (LRB 60230,). There was an instance, 21:58, 2/15/09 (LRB 60235, pg. 57), where spike of 150 GPM on FI-0635 lasted a few seconds, then flow rate returned to a stable rate of 90 to 103 GPM.
	- Irive time was decreased from the tuned setting to maintain filter-loop ate with Run Sheet targets. (TI).
	- target filter-loop flow rate was not achieved during the tracer test the tracer test was repeated after the High Solids Filter Test was eted on $2/21/09$.
	- -ring air purge, upper and lower air spargers were kept off, per nce, as opposed to original Run Sheet targets of 0.14, 0.01, and 0.04 n, respectively. (TI: Run Sheet).
	- pare samples were collected. An outer/middle CD sample was ted at $17:16$, then an outer/low CD sample was collected at $17:29$ -0 minutes of lower sparger operations at 0.4 kg/min. (LRB 60271, δ .
	- 23:13, 2/14/09 (LRB 60230, pg. 57) UFP-VSL-T02A bubblers were d, but not successfully.

Air entrainment (also includes small air bubbles trapped in simulant, was first recognized prior to washing post-caustic-leach concentrated slurry, on 2/15/09, even though symptoms surfaced during tracer test preparation. See discussion on air entrainment and slurry retained gas

dance, additional CD samples and a permeate sample from Filter were collected before post-caustic-leach washing than what was for in sample matrix, $2/14/09$ @ 23:54. (TI).

2/15/09, a 100-mL UFP-VSL-T02A filter-loop sample for rheology ken (LRB 60230, pg. 66).

2/15/09, a 40-mL filter-loop sample was taken to assess void fraction after it was spun down for 20 minutes, a 3-mL volume change was ed \sim ~7.5% air entrainment (LRB 60230, pg. 69). Other nents were done to determine gas release rate and AFA effects on the ease rate, see section 6.2.

ring purge rate was set at 0.014 kg/min as opposed to Run Sheet of 0.14 kg/min – the 0.014 is a typo (TI: Run Sheet) \rightarrow steam-ring air te was off between 14:26 through 19:32:12, 2/15/09; LRB 60230, 81. Then from 19:32:12 to 20:16:03, 2/15/09, steam-ring flow rate 07 kg/min; then it was turned off for the rest of the washing (data and TI pg. 197).

oop flow rate ranged between 80 and 90 GPM as opposed to Run arget of 109 ± 10 GPM. (TI: Run Sheet).

bressure drop across the 5 filters was at 27 psig as opposed to Run arget of \leq 25 psig. (TI: Run Sheet).

aking the 12th CD Sample Event (wash #56), per approval, lower air rs were set to idle and steam-ring air purge rate was set to 0.07 to improve filter-loop flow rate. (TI).

aking the 13th CD Sample Event (wash #60), per approval, lower air rs were turned OFF for remainder of the wash to minimize pump on. They were set to idle after completing post-caustic-leach $ng.$ (TI).

 $2/16/09$ (LRB 60230 , pg. 89) Turning lower air spargers ON to idle tes at 00:50, increased laser level from 51 to 54 inches, appeared to h; filter pump speed was reduced to bring laser level back to 51

positively was 13.6 m/s as opposed to Run Sheet target of 12 ± 1 0.6 (TI: Run Sheet).

ring air purge rate was OFF, per Guidance, as opposed to original heet target of 0.14 kg/min. (TI: Run Sheet).

ring air purge, upper and lower air spargers were kept off, per ace, as opposed to original Run Sheet targets of 0.14, 0.01, and 0.04 , respectively. (TI: Run Sheet).

05:44, 2/16/09 (LRB 60230, pg. 95) During PJM normal operation, a "PJM inactive" alarm came on.

ed PJM stroke $@$ 03:16, 2/16/09, to be within target since it exceed stroke length. $(TI$ pg 203).

oop flow rate decreased through the course of chromium washing, at $2/16/09$ filter-loop flow rate was 85 GPM (was 103 GPM ω 02:51, $(71 \text{ pg}, 202)$; Run Sheet target was 109 ± 10 GPM.

er UFP-VSL-T02A recirculation loop sample (39.5-mL) was taken un down for 20 minutes (4500 rpm); final volume was 36.5 mL ing air entrainment of ~7.6% (07:05, 2/16/09; LRB 60230, pg. 96).

ring air purge, upper and lower air spargers were supposed to be off, idance, as opposed to original Run Sheet targets of 0.14, 0.01, and g/min, respectively. (TI: Run Sheet)However: Lower sparger flow as ON at the Run Sheet target of 0.04 kg/min, $08:30$ $2/16/09$ (TI pg.

tion, maximum attainable filter-loop flow rate was 85 GPM, opposed 109 ± 10 GPM Run Sheet target. (TI: Run Sheet).

2/16/09 (LRB 60230, pg. 97) Sampling folks noted great difficulty is ersing the centrifuged solids after decanting the supernate. The mixer alone was not getting the job done. An ultrasonic bath was ered, but more forcible manual mixing along with vortex mixing the problem.

Leaks on UFP-VSL-T02A PJMs 1807 and 1801 vacuum lines were $\text{ied, by } 10:00$, they were still leaking after tightening the connections, 09, LRB 60230, pg. 98).

ring air purge, upper and lower air spargers were kept off, per α ce, as opposed to original Run Sheet targets of 0.14, 0.01, and 0.04 , respectively. (TI: Run Sheet). However, lower sparger flow rate \overline{N} at ~0.04 kg/min, per Appendix G plots.

elocity was 13.6 m/s as opposed to the $12\pm.6$ m/s Run Sheet target. an Sheet).

 $2/16/09$ (LRB 60230, pg. 99) Filter-loop flow rates increased (by lves) to target flow rate during the sodium permanganate transfer, lined soon after transfer was complete.

 $18, 2/16/09$ (LRB 60230, pg. 100) Leaking simulant coming out the the PJMs in UFP-VSL-T02A was observed. PJM parameter and followed with several overblows. By 15:15, $2/16/09$ (LRB pg. 101) UFP-VSL-T02A PJMs 1801 and 1807 were taken out of The process of taking the UFP-VSL-T02A PJMs off then restarting one was initiated while listening for overblows, and making PJM eter adjustments as necessary.

ode of operation was switched to standard per the Test Director on the evening of $2/16/09$, 20:12.

ring air purge, upper and lower air spargers were kept off, per α ce, as opposed to original Run Sheet targets of 0.14, 0.01, and 0.04 , respectively. (TI: Run Sheet).

en 16:45 and 16:50 on 2/16/09 (TI pg. 209), Run Sheet PJM target eters were compromised, however, proceeded with testing with al. PJMs not functioning properly (scribe C. Brown email $2/17/09$,

level measurement $@$ 16:52, 2/16/09, before washing oxidated l slurry, indicated the bubbler in UFP-VSL-T02A was plugged, s gravity of 3.98 (TI pg. 210).

level measurement completed ω 00:12, 2/17/09, after washing ed leached slurry, indicated the bubbler in UFP-VSL-T02A was still rking, specific gravity of 0.95 (TI pg. 214). This stable level rement started at $23:40$, $2/16/09$, over this period of time, the laser ropped from 54.6 to 52.7 inches, indicating slurry was degassing (TI 4) - or foam was decreasing.

Integrated Test A was put on hold after completing the post oxidative leach washing per the Test Director email 20:28, 2/16/09. A list of tasks form after completing the wash was provided in the email. See **discussion on the test suspension, Section 6.2.**

ted Integrated Test A on the evening of $2/20/09$.

stable level measurement before commencing final concentration. ers were turned off, per guidance (TI pg. 215).

-ring air purge, upper and lower air spargers were kept off, per nce, as opposed to original Run Sheet targets of 0.14 , 0.01 , and 0.04 m. respectively. (TI: Run Sheet).

lition, maximum attainable filter-loop flow rate was 73 GPM am from UFP-PMP-T42A (flow rate on FI-0635, downstream from PMP-T43A was higher) as opposed to the 109 ± 10 GPM Run Sheet target. (TI: Run Sheet) Flowmeter performance is questionable at this

leted on $3/20/09$, after completing Integrated Test B. Simulant from ated Test A stored in totes was added to UFP-VSL-T02A, via transfer UFP-VSL-T01B, then UFP-VSL-T02A was filled with inhibited water 1 Sheet Target (TI, pg. 218). Refer to Instruction per the Test or, 3/20/09, 7:44 PM (LRB 60279, pg. 38).

At 22:00, 3/20/09, the filter-loop pumps turned off because of VSL-T02A PJM overblowing. PJMs were adjusted (LRB 60279, -41).

tions were run with the revised Run Sheet parameters:

ring, upper and lower spargers were OFF.

vessel temperature target is 23° C.

pressure drop ≤ 50 psi.

not listed on Run Sheet, a calculated 86 mL of concentrated AFA mL of diluted solution) was added to account for the volume of ted water used to reach tank level target per Run Sheet, $22:50$, $3/20/09$

f the 2-40 mL samples was not collected per guidance (the thought hat the post oxidative leached samples collected would provide the results as these samples; however, it was not foreseen that the High Test would be conducted at a later date using slurry from Integrated 3 and stored slurry from Integrated Test A (01:33, 3/21/09, TI).

-ring air purge, upper and lower air spargers were kept off, per nce, as opposed to original Run Sheet targets of 0.14, 0.01, and 0.04

n, respectively. (TI: Run Sheet).

05:15, 3/21/09 (LRB 60279, pg. 43) Heat exchanger, UFP-HX-T02A, TICemperature setpoint is decreased to 20°C because temperature in the s 26.4°C, target is 23 ± 2 °C.

05:44, 3/21/09 (LRB 60279, pg. 43) Heat exchanger, UFP-HX-T02A, TICemperature setpoint is set to manual operation, valve open $= 28\%$ se temperature is increasing fast (low level at high pump flow rate).

05:46, 3/21/09 (LRB 60271, pg. 4) Heat exchanger, UFP-HX-T02A valve is open = 60% , temperature in the vessel is 28.5° C

rocess step was not performed since contents of UFP-VSL-T02A on 9 were kept for later use, the simulant in the filter loop was drained y to a tote without adding inhibited water (TI pg. 225)

Iter-loop samples collected had UFP-VSL-T02A aligned (02:12, 9, TI pg. 226).

ximately 19 inches of simulant was also removed from VSL-T02A and put into totes in order to reach a 20 inch in the vessel. Iter-loop study indicated that filter-loop flow rate dropped from 100 at 20 inch tank level to 83 GPM at an 18.3 inch level per upstream heter FI-0623 upstream from UFP-PMP-T42A. The laser was noted reading below the 17.8" vessel level. The PJMs were turned off the filter-loop study so that the camera quality would not be comised. The camera showed the simulant return line exposed x imately 2 inches at the 18.3 inch vessel level. (TI pg 224).

mples were not collected (TI pg 303).

Appendix F

Sparger Operations
Appendix F: Sparger Operations

In addition to PJMs, air spargers were used in UFP-VSL-T02A to help with the mixing and suspension of solids. For Integrated Test A, target flow rates were idle ones, 0.01 kg/min (total flow) for the lower spargers and 0.04 kg/min (total) for the upper. While the data in this appendix are for information only, the figures are NQA-1 compliant.

UFP-VSL-T02A sparger air flow rates deviated from the target values in some instances during the tests. The upper spargers lost air flows on two occasions during dewatering of leached slurry. In both cases, the sparger nozzles were found plugged. The air flow rate came back to the target value after the spargers were cleaned.

- 1. The upper spargers air flow rate started fluctuating ~06:30, 2/5/09 and lost air flow from ~06:30 to \sim 12:00, 2/6/09. The upper spargers were cleaned at 11:49, 2/6/09.
- 2. The upper spargers air flow rate started fluctuating $\sim 04:00$, $\frac{2}{12/09}$ and lost air flow from 22:00, $\frac{2}{13}$ to 03:30, 2/14/09. The upper spargers were cleaned at 01:28, 2/14/09.

Sparging nozzles were clogged or plugged thus causing the lower sparger air flow rates lower than the target values. The spargers were cleaned occasionally to maintain the air flow rates within the target ranges. Air flow rates spiked during cleaning. Spargers were turned off per test instruction to take stable level measurements in UFP-VSL-T02A. On many occasions, sparging air was shut off for troubleshooting of abnormal operations, i.e., slurry air entrainments.

- 1. 21:57, 2/3/09. UFP-VSL-T02A pumps shut off, triggered by bubbler low-low alarm, and sparger air was lost as well. Spargers were restarted after cleaning of bubbler and laser ~22:05.
- 2. 21:52, 2/7/09. One of the lower sparger rotameters was isolated since flow rate was decaying with time. However, target total flow was kept.
- 3. 21:23, 2/11/09. One of the spargers was plugged.
- 4. 03:30, 2/12/09. Sparger, nozzle 31 was plugged and the associated flow meter FIC-1943 was closed at 07:05, 2/12/09.
- 5. 11:20, 2/12/09. Sparger nozzle 31, was flushed to clear the plug.
- 6. 19:50, 2/12/09. Cleared sparger nozzles 32 and 33.
- 7. 10:20, 2/15/09. After taking the 13th CD Sample Event (wash #60), per approval, lower air spargers were turned OFF for remainder of the wash to minimize pump vibration. They were set to idle after completing post-caustic-leach washing. (TI).
- 8. Between 01:57, 2/15/09 and 14:26, 2/15/09, lower spargers were turned on and off several times for filter-loop flow-rate investigations.
- 9. 00:50, 2/16/09. Turned lower air spargers ON to idle flow rates to see effect on pumps.
- 10. 13:02, 2/18/09. Exercises using different number of spargers (total flow of 0.4 kg/min) to see effects on filter-loop flow rate.
- 11. 15:20, 2/18/09 to 15:34, 2/20/09. Lower spargers were on and off many times, per Run Sheet, during slurry degassing and filter-loop pump exercises.

Figure F.1. Integrated Test A Air Sparge Flow Rate Summary 1/31/2009 through 2/21/2009

Figure F.2. Integrated Test A Air Sparge Flow Rates between 1/31/2009 and 2/16/2009

F.3

Figure F.3. Integrated Test A Air Sparge Flow Rates between 2/17/2009 and 2/21/2009

F.4

Appendix G

12-Hour Plots

Appendix G: Integrated Test A Data Plots

01/31/09 00:00 – 01/31/09 12:00

T01A level

T01B level

T01B temperatures

T02A level

Pump Pressures and Flow

Axial pressure drop

G.5

Permeate flow rates

T02A Inner Temperature Tree

T02A Outer Temperature Tree

T02A temperatures

Pump Operation

Pulsepot UFP-PP-T01A

Pulsepot UFP-PP-T02A

Pressure (psig)

Pressure (psig) **Pressure (psig)**

Pulsepot Levels

Level (in)

Filter UFP-FILT-T02A

G.13

Filter UFP-FILT-T04A

Chemical Flow

Chemical Flow

Air Flows

T02A Steam

T01A Steam

T01B Steam

Integrated Test A Data Plots 01/31/09 12:00 – 02/01/09 00:00

T01B level

T01B temperatures

T02A level

T02A and filter loop temperatures

Axial pressure drop

Permeate flow rates

T02A Inner Temperature Tree

T02A Outer Temperature Tree

T02A temperatures

T02A temperatures

T02A Heating and Cooling

Pump Operation

Pulsepot UFP-PP-T01A

Pulsepot UFP-PP-T02A

Pressure (psig) **Pressure (psig)**

Pulsepot UFP-PP-T03A

Pulsepot Levels

Level (in)
Filter UFP-FILT-T01A

Pressure (psig) **Pressure (psig)**

Filter UFP-FILT-T03A

Filter UFP-FILT-T05A

Chemical Flow

Chemical Flow

Air Flows

T02A Steam

T01A Steam

T01B Steam

Integrated Test A Data Plots 02/01/09 00:00 – 02/01/09 12:00

T01A level

T01A temperatures

T01B level

T01B temperatures

Level (in)

T02A level

T02A temperatures

Temperatures (C)

T02A and filter loop temperatures

Pump Pressures and Flow

Pressure (psig or in H2O)

PST

Axial pressure drop

Permeate flow rates

T02A Inner Temperature Tree

T02A Outer Temperature Tree

T02A temperatures

T02A temperatures

T02A temperatures

T02A temperatures

T02A Heating and Cooling

Pump Operation

Pulsepot UFP-PP-T01A

Pulsepot UFP-PP-T02A

PST

Pulsepot UFP-PP-T03A

Pulsepot Levels

Filter UFP-FILT-T01A

Filter UFP-FILT-T03A

Filter UFP-FILT-T05A

Chemical Flow

Chemical Flow

Air Flows

T02A Steam

T01A Steam

T01B Steam

Integrated Test A Data Plots 02/01/09 12:00 – 02/02/09 00:00

T01A level

T01A temperatures

T01B level

T01B temperatures

Temperatures (C) **Temperatures (C)**

T02A temperatures

T02A and filter loop temperatures

Pump Pressures and Flow

PST

Axial pressure drop

Permeate flow rates

T02A Inner Temperature Tree

T02A Outer Temperature Tree

T02A temperatures

T02A temperatures

T02A temperatures

T02A temperatures

T02A Heating and Cooling

Pump Operation

Pulsepot UFP-PP-T01A

Pulsepot UFP-PP-T03A

Pulsepot Levels

Filter UFP-FILT-T01A

Pressure (psig) **Pressure (psig)**

Pressure (psig) **Pressure (psig)** **Filter UFP-FILT-T03A**

Filter UFP-FILT-T05A

Chemical Flow

Chemical Flow

Air Flows

T02A Steam

T01A Steam

T01B Steam

Integrated Test A Data Plots 02/02/09 00:00 – 02/02/09 12:00

T01A level

T01A temperatures

T01B level

T01B temperatures

G.75

T02A level

T02A temperatures

Temperatures (C)

T02A and filter loop temperatures

Axial pressure drop

Permeate flow rates

PST

T02A Inner Temperature Tree

T02A Outer Temperature Tree

T02A temperatures

T02A temperatures

T02A temperatures

T02A temperatures

T02A Heating and Cooling

Pump Operation

Pulsepot UFP-PP-T01A

Pulsepot UFP-PP-T02A

Pressure (psig) **Pressure (psig)**

Pressure (psig) **Pressure (psig)**

Pulsepot UFP-PP-T03A

Pulsepot Levels

Filter UFP-FILT-T01A

Pressure (psig)

Pressure (psig)

Filter UFP-FILT-T03A

G.86

Filter UFP-FILT-T05A

Chemical Flow

Chemical Flow

Air Flows

T02A Steam

T01A Steam

T01B Steam

Integrated Test A Data Plots 02/02/09 12:00 – 02/03/09 00:00

T01A temperatures

T01B level

T01B temperatures

T02A level

T02A temperatures

T02A and filter loop temperatures

G.95

Axial pressure drop

Permeate flow rates

T02A Inner Temperature Tree

T02A Outer Temperature Tree

T02A temperatures

T02A temperatures

T02A temperatures

T02A temperatures

T02A Heating and Cooling

Pump Operation

Pulsepot UFP-PP-T01A

Pulsepot UFP-PP-T03A

Pulsepot Levels

Filter UFP-FILT-T01A

G.103

Filter UFP-FILT-T03A

PST

Filter UFP-FILT-T04A

Chemical Flow

PST

Chemical Flow

Air Flows

T02A Steam

T01A Steam

T01B Steam

Integrated Test A Data Plots 02/03/09 00:00 – 02/03/09 12:00

T01A level

T01A temperatures

T01B level

T01B temperatures

T02A level

T02A temperatures

T02A and filter loop temperatures

Axial pressure drop

T02A Inner Temperature Tree

T02A Outer Temperature Tree

T02A temperatures

T02A temperatures

T02A temperatures

T02A temperatures

T02A Heating and Cooling

Pump Operation

Pulsepot UFP-PP-T01A

Pulsepot UFP-PP-T02A

Pulsepot UFP-PP-T03A

Pulsepot Levels

Pressure (psig) **Pressure (psig)**

Filter UFP-FILT-T03A

Filter UFP-FILT-T05A

Chemical Flow

Pressure (psig) **Pressure (psig)**

Chemical Flow

Air Flows

T02A Steam

T01A Steam

T01B Steam

Integrated Test A Data Plots 02/03/09 12:00 – 02/04/09 00:00 **T01A level**

T01A temperatures

T02A level

T02A temperatures

T02A and filter loop temperatures

Axial pressure drop

Permeate flow rates

T02A Inner Temperature Tree

T02A Outer Temperature Tree

T02A temperatures

T02A temperatures

T02A temperatures

T02A temperatures

T02A Heating and Cooling

Pump Operation

Pulsepot UFP-PP-T01A

Pulsepot UFP-PP-T03A

Pulsepot Levels

Filter UFP-FILT-T01A

Pressure (psig) **Pressure (psig)**

Filter UFP-FILT-T03A

G.140

Filter UFP-FILT-T05A

Chemical Flow

Chemical Flow

Air Flows

G.142

T02A Steam

T01A Steam

T01B Steam

Integrated Test A Data Plots 02/04/09 00:00 – 02/04/09 12:00

T01A level

T01A temperatures

T01B level

T01B temperatures

T02A level

T02A temperatures

T02A and filter loop temperatures

G.149

Axial pressure drop

T02A Inner Temperature Tree

T02A temperatures

T02A temperatures

T02A temperatures

T02A temperatures

T02A Heating and Cooling

Pump Operation

Pulsepot UFP-PP-T01A

Pulsepot UFP-PP-T02A

Pulsepot UFP-PP-T03A

Pulsepot Levels

G.157

Filter UFP-FILT-T02A

Pressure (psig) **Pressure (psig)**

Pressure (psig) **Pressure (psig)**

Filter UFP-FILT-T05A

Chemical Flow

Chemical Flow

Air Flows

T02A Steam

T01A Steam

G.161

T01B Steam

Integrated Test A Data Plots 02/04/09 12:00 – 02/05/09 00:00

T01A temperatures

T01B level

T01B temperatures

T02A level

T02A temperatures

T02A and filter loop temperatures

Pump Pressures and Flow

Axial pressure drop

T02A Inner Temperature Tree

T02A Outer Temperature Tree

T02A temperatures

T02A temperatures

T02A temperatures

T02A temperatures

T02A Heating and Cooling

Pump Operation

Pulsepot UFP-PP-T01A

Pulsepot UFP-PP-T02A

PST

Pulsepot UFP-PP-T03A

Pulsepot Levels

Filter UFP-FILT-T01A

Pressure (psig) **Pressure (psig)**

Filter UFP-FILT-T03A

PST

Filter UFP-FILT-T05A

G.177

Chemical Flow

Air Flows

T02A Steam

T01A Steam

T01B Steam

Integrated Test A Data Plots 02/05/09 00:00 – 02/05/09 12:00

T01A level

T01A temperatures

T01B level

T01B temperatures

G.183

T02A level

T02A temperatures

Level (in)

T02A and filter loop temperatures

Pump Pressures and Flow

Axial pressure drop

Permeate flow rates

PST

T02A Inner Temperature Tree

T02A temperatures

T02A temperatures

T02A temperatures

T02A temperatures

T02A Heating and Cooling

Pump Operation

Pulsepot UFP-PP-T01A

Pulsepot UFP-PP-T03A

Level (in)

Filter UFP-FILT-T01A

G.193

Filter UFP-FILT-T03A

Filter UFP-FILT-T05A

Chemical Flow

Air Flows

T02A Steam

T01A Steam

T01B Steam

Integrated Test A Data Plots 02/05/09 12:00 – 02/06/09 00:00

T01A level

T01A temperatures

T01B level

T01B temperatures

T02A level

T02A temperatures

T02A and filter loop temperatures

Pump Pressures and Flow

G.203

Axial pressure drop

Permeate flow rates

T02A Inner Temperature Tree

T02A Outer Temperature Tree

T02A temperatures

T02A temperatures

T02A temperatures

T02A temperatures

T02A Heating and Cooling

Pump Operation

Pulsepot UFP-PP-T01A

Pulsepot UFP-PP-T03A

Pulsepot Levels

Filter UFP-FILT-T01A

G.211

Filter UFP-FILT-T03A

Filter UFP-FILT-T05A

Chemical Flow

Chemical Flow

Air Flows

T02A Steam

T01A Steam

T01B Steam

Integrated Test A Data Plots 02/06/09 00:00 – 02/06/09 12:00

T01A level

T01A temperatures

T01B temperatures

T02A level

T02A temperatures

T02A and filter loop temperatures

G.221

Axial pressure drop

Permeate flow rates

T02A Inner Temperature Tree

T02A Outer Temperature Tree

T02A temperatures

T02A temperatures

T02A temperatures

T02A temperatures

T02A Heating and Cooling

Pulsepot UFP-PP-T01A

Pulsepot UFP-PP-T02A

Pulsepot UFP-PP-T03A

Pulsepot Levels

Level (in)

Filter UFP-FILT-T03A

Filter UFP-FILT-T05A

Chemical Flow

Chemical Flow

Air Flows

G.232

T02A Steam

T01A Steam

T01B Steam

Integrated Test A Data Plots 02/06/09 12:00 – 02/07/09 00:00

T01A level

T01A temperatures

T01B temperatures

T02A level

T02A temperatures

T02A and filter loop temperatures

Axial pressure drop

Permeate flow rates

T02A Inner Temperature Tree

T02A Outer Temperature Tree

T02A temperatures

T02A temperatures

T02A temperatures

T02A temperatures

T02A Heating and Cooling

Pump Operation

Pulsepot UFP-PP-T01A

Pulsepot UFP-PP-T03A

Filter UFP-FILT-T01A

G.247

Filter UFP-FILT-T03A

PST

Filter UFP-FILT-T05A

PST

Chemical Flow

Air Flows

T02A Steam

T01A Steam

T01B Steam

Integrated Test A Data Plots 02/07/09 00:00 – 02/07/09 12:00

T01A level

T01A temperatures

T01B level

T01B temperatures

T02A level

T02A temperatures

T02A and filter loop temperatures

Axial pressure drop

Permeate flow rates

T02A Inner Temperature Tree

T02A Outer Temperature Tree

T02A temperatures

T02A temperatures

T02A temperatures

T02A temperatures

T02A Heating and Cooling

Pump Operation

Pulsepot UFP-PP-T01A

Pulsepot UFP-PP-T02A

PST

Pulsepot UFP-PP-T03A

Pulsepot Levels

Filter UFP-FILT-T01A

Filter UFP-FILT-T03A

Filter UFP-FILT-T05A

Chemical Flow

PST

Chemical Flow

Air Flows

T02A Steam

T01A Steam

T01B Steam

Integrated Test A Data Plots 02/07/09 12:00 – 02/08/09 00:00

T01B level

T01B temperatures

T02A level

T02A temperatures

T02A and filter loop temperatures

Axial pressure drop

Permeate flow rates

T02A Inner Temperature Tree

T02A Outer Temperature Tree

T02A temperatures

T02A temperatures

T02A temperatures

T02A temperatures

T02A Heating and Cooling

Pump Operation

Pulsepot UFP-PP-T01A

PST

Pulsepot UFP-PP-T03A

Pulsepot Levels

Pressure (psig) **Pressure (psig)**

Level (in)
Filter UFP-FILT-T01A

Pressure (psig)

Pressure (psig)

Filter UFP-FILT-T03A

G.284

Filter UFP-FILT-T05A

Chemical Flow

Pressure (psig) **Pressure (psig)**

Chemical Flow

Air Flows

PST

T02A Steam

T01A Steam

T01B Steam

Integrated Test A Data Plots 02/08/09 00:00 – 02/08/09 12:00

T01A level

T01A temperatures

T01B level

T01B temperatures

T02A level

T02A temperatures

Temperatures (C)

T02A and filter loop temperatures

Pressure (psig or in H2O)

Pressure (psig or in H2O)

Axial pressure drop

Permeate flow rates

T02A Inner Temperature Tree

PST

T02A temperatures

T02A temperatures

T02A temperatures

T02A temperatures

T02A Heating and Cooling

Pump Operation

Pulsepot UFP-PP-T01A

Pulsepot UFP-PP-T03A

Pulsepot Levels

Filter UFP-FILT-T01A

G.301

Pressure (psig)

Pressure (psig)

Filter UFP-FILT-T03A

Pressure (psig) **Pressure (psig)**

Pressure (psig) **Pressure (psig)**

Filter UFP-FILT-T05A

Chemical Flow

Pressure (psig) **Pressure (psig)**

Chemical Flow

Air Flows

T02A Steam

T01A Steam

T01B Steam

Integrated Test A Data Plots 02/08/09 12:00 – 02/09/09 00:00 **T01A level**

T01A temperatures

T01B level

T01B temperatures

T02A level

T02A temperatures

T02A and filter loop temperatures

Pump Pressures and Flow

G.311

Axial pressure drop

Permeate flow rates

T02A Inner Temperature Tree

T02A Outer Temperature Tree

T02A temperatures

T02A temperatures

T02A temperatures

T02A temperatures

T02A Heating and Cooling

Pump Operation

Pulsepot UFP-PP-T01A

Pulsepot UFP-PP-T02A

Pulsepot UFP-PP-T03A

Pulsepot Levels

Filter UFP-FILT-T01A

G.319

Filter UFP-FILT-T03A

G.320

Filter UFP-FILT-T05A

Chemical Flow

Chemical Flow

Air Flows

T02A Steam

T01A Steam

T01B Steam

Integrated Test A Data Plots 02/09/09 00:00 – 02/09/09 12:00 **T01A level**

T01A temperatures

T01B temperatures

T02A level

T02A temperatures

Temperatures (C)

T02A and filter loop temperatures

Pump Pressures and Flow

Permeate flow rates

T02A Inner Temperature Tree

T02A temperatures

T02A temperatures

T02A temperatures

T02A temperatures

T02A Heating and Cooling

Pump Operation

Pulsepot UFP-PP-T01A

Pulsepot UFP-PP-T02A

Pulsepot UFP-PP-T03A

Pulsepot Levels

Filter UFP-FILT-T01A

Pressure (psig) **Pressure (psig)**

Filter UFP-FILT-T03A

Filter UFP-FILT-T05A

Chemical Flow

Pressure (psig) **Pressure (psig)**

Chemical Flow

Air Flows

T02A Steam

T01A Steam

T01B Steam

Integrated Test A Data Plots 02/09/09 12:00 – 02/10/09 00:00 **T01A level**

T01A temperatures

T01B level

T01B temperatures

T02A level

T02A temperatures

Temperatures (C)

T02A and filter loop temperatures

Pump Pressures and Flow

Pressure (psig or in H2O)

Pressure (psig or in H2O)

Axial pressure drop

Permeate flow rates

T02A Inner Temperature Tree

T02A Outer Temperature Tree

T02A temperatures

T02A temperatures

T02A temperatures

T02A temperatures

T02A Heating and Cooling

Pulsepot UFP-PP-T01A

Pulsepot UFP-PP-T02A

PST

Pulsepot UFP-PP-T03A

Pulsepot Levels

Filter UFP-FILT-T01A

G.355

Filter UFP-FILT-T03A

Filter UFP-FILT-T05A

Chemical Flow

G.357

Pressure (psig) **Pressure (psig)**

Chemical Flow

Air Flows

PST

T02A Steam

T01A Steam

T01B Steam

Integrated Test A Data Plots 02/10/09 00:00 – 02/10/09 12:00

T01A level

T01A temperatures

T01B level

T01B temperatures

PST

T02A level

T02A temperatures

T02A and filter loop temperatures

PST

Axial pressure drop

Permeate flow rates

G.366

T02A Inner Temperature Tree

T02A Outer Temperature Tree

T02A temperatures

T02A temperatures

T02A temperatures

T02A temperatures

T02A Heating and Cooling

Pulsepot UFP-PP-T01A

Pulsepot UFP-PP-T02A

PST

Pulsepot UFP-PP-T03A

Pulsepot Levels

PST

Filter UFP-FILT-T01A

Pressure (psig) **Pressure (psig)** **Filter UFP-FILT-T03A**

Filter UFP-FILT-T05A

Chemical Flow

Air Flows

T02A Steam

T01A Steam

T01B Steam

Integrated Test A Data Plots 02/10/09 12:00 – 02/11/09 00:00

T01A level

T01A temperatures

T01B level

T01B temperatures

Temperatures (C) **Temperatures (C)**

T02A level

T02A temperatures

T02A and filter loop temperatures

Pump Pressures and Flow

Axial pressure drop

Permeate flow rates

T02A Inner Temperature Tree

T02A Outer Temperature Tree

T02A temperatures

T02A temperatures

G.386

T02A temperatures

T02A temperatures

T02A Heating and Cooling

Pulsepot UFP-PP-T01A

Pulsepot UFP-PP-T02A

Pulsepot UFP-PP-T03A

Pulsepot Levels

Filter UFP-FILT-T01A

Filter UFP-FILT-T02A

Pressure (psig) **Pressure (psig)**

Filter UFP-FILT-T03A

Filter UFP-FILT-T05A

Chemical Flow

Air Flows

T02A Steam

T01A Steam

T01B Steam

Integrated Test A Data Plots 02/11/09 00:00 – 02/11/09 12:00

T01A level

T01A temperatures

Level (in)

T01B level

T01B temperatures

T02A level

T02A temperatures

T02A and filter loop temperatures

Pump Pressures and Flow

PST

Axial pressure drop

Permeate flow rates

T02A Inner Temperature Tree

T02A Outer Temperature Tree

T02A temperatures

T02A temperatures

T02A temperatures

T02A temperatures

T02A Heating and Cooling

Pulsepot UFP-PP-T01A

Pulsepot UFP-PP-T03A

Pulsepot Levels

Filter UFP-FILT-T01A

G.409

Filter UFP-FILT-T03A

G.410

Filter UFP-FILT-T05A

Chemical Flow

Chemical Flow

Air Flows

T02A Steam

T01A Steam

T01B Steam

Integrated Test A Data Plots 02/11/09 12:00 – 02/12/09 00:00

T01A level

T01A temperatures

G.416

T01B temperatures

T02A level

T02A temperatures

T02A and filter loop temperatures

Pump Pressures and Flow

Pressure (psig or in H2O)

Pressure (psig or in H2O)

PST

Axial pressure drop

Permeate flow rates

T02A Inner Temperature Tree

T02A Outer Temperature Tree

T02A temperatures

T02A temperatures

T02A temperatures

T02A temperatures

T02A Heating and Cooling

Pump Operation

G.424

Pulsepot UFP-PP-T01A

Pulsepot UFP-PP-T02A

Pulsepot UFP-PP-T03A

Pulsepot Levels

PST
Filter UFP-FILT-T01A

Filter UFP-FILT-T03A

Filter UFP-FILT-T05A

Chemical Flow

PST

Chemical Flow

Air Flows

T02A Steam

T01A Steam

T01B Steam

Integrated Test A Data Plots 02/12/09 00:00 – 02/12/09 12:00

T01A temperatures

T01B level

T01B temperatures

T02A level

T02A temperatures

T02A and filter loop temperatures

T02A Inner Temperature Tree

T02A temperatures

T02A temperatures

T02A temperatures

T02A temperatures

T02A Heating and Cooling

Pulsepot UFP-PP-T01A

Pulsepot UFP-PP-T02A

Pulsepot UFP-PP-T03A

Pulsepot Levels

Filter UFP-FILT-T01A

Pressure (psig) **Pressure (psig)** **Filter UFP-FILT-T03A**

Filter UFP-FILT-T05A

Chemical Flow

Chemical Flow

Air Flows

T02A Steam

T01A Steam

PST

T01B Steam

Integrated Test A Data Plots 02/12/09 12:00 – 02/13/09 00:00

T01A temperatures

T01B temperatures

T02A level

T02A temperatures

T02A and filter loop temperatures

Axial pressure drop

Permeate flow rates

T02A Inner Temperature Tree

T02A Outer Temperature Tree

T02A temperatures

T02A temperatures

T02A temperatures

T02A temperatures

T02A Heating and Cooling

Pulsepot UFP-PP-T01A

Pulsepot UFP-PP-T02A

Pulsepot UFP-PP-T03A

Pulsepot Levels

Filter UFP-FILT-T01A

Pressure (psig) **Pressure (psig)**

Pressure (psig) **Pressure (psig)** **Filter UFP-FILT-T03A**

Filter UFP-FILT-T05A

Chemical Flow

PST

Chemical Flow

Air Flows

T02A Steam

T01A Steam

T01B Steam

Integrated Test A Data Plots 02/13/09 00:00 – 02/13/09 12:00

T01A level

T01A temperatures

T01B level

T01B temperatures

T02A level

T02A temperatures

Temperatures (C)

T02A and filter loop temperatures

G.473

Axial pressure drop

Permeate flow rates

T02A Inner Temperature Tree

T02A temperatures

T02A temperatures

T02A temperatures

T02A temperatures

Pulsepot UFP-PP-T01A

Pressure (psig) **Pressure (psig)**

Pressure (psig) **Pressure (psig)**

Pulsepot UFP-PP-T03A

Pulsepot Levels

Filter UFP-FILT-T01A

G.481

Filter UFP-FILT-T03A

PST

Filter UFP-FILT-T05A

Chemical Flow

Chemical Flow

Air Flows

T02A Steam

T01A Steam

T01B Steam

Integrated Test A Data Plots 02/13/09 12:00 – 02/14/09 00:00 **T01A level**

T01A temperatures

T01B level

T01B temperatures

T02A level

T02A temperatures

Temperatures (C)

T02A and filter loop temperatures

PST

Axial pressure drop

Permeate flow rates

T02A Inner Temperature Tree

G.493

T02A temperatures

T02A temperatures

T02A temperatures

T02A temperatures

T02A Heating and Cooling

Pulsepot UFP-PP-T01A

Pulsepot UFP-PP-T02A

Pressure (psig) **Pressure (psig)**

Pressure (psig) **Pressure (psig)**

Pulsepot UFP-PP-T03A

Pulsepot Levels

PST
Filter UFP-FILT-T01A

G.499

Filter UFP-FILT-T03A

G.500

Filter UFP-FILT-T05A

Chemical Flow

Chemical Flow

Air Flows

T02A Steam

T01A Steam

T01B Steam

Integrated Test A Data Plots 02/14/09 00:00 – 02/14/09 12:00

T01A level

T01A temperatures

T01B level

T01B temperatures

T02A level

T02A temperatures

T02A and filter loop temperatures

Axial pressure drop

Permeate flow rates

T02A Inner Temperature Tree

T02A temperatures

T02A temperatures

T02A temperatures

T02A temperatures

T02A Heating and Cooling

Pulsepot UFP-PP-T01A

Pulsepot UFP-PP-T03A

Pulsepot Levels

Filter UFP-FILT-T01A

G.517

Filter UFP-FILT-T03A

G.518

Filter UFP-FILT-T05A

Chemical Flow

Chemical Flow

Air Flows

T02A Steam

T01A Steam

T01B Steam

Integrated Test A Data Plots 02/14/09 12:00 – 02/15/09 00:00

T01A level

T01A temperatures

T01B temperatures

T02A level

T02A temperatures

T02A and filter loop temperatures

Permeate flow rates

T02A Inner Temperature Tree

T02A temperatures

T02A temperatures

G.530

T02A temperatures

T02A temperatures

T02A Heating and Cooling

Pulsepot UFP-PP-T01A

Pulsepot UFP-PP-T03A

Pulsepot Levels

Filter UFP-FILT-T01A

PST

G.535

Filter UFP-FILT-T03A

Pressure (psig) **Pressure (psig)**

Filter UFP-FILT-T05A

Chemical Flow

PST

Chemical Flow

Air Flows

T02A Steam

T01A Steam

T01B Steam

Integrated Test A Data Plots 02/15/09 00:00 – 02/15/09 12:00

T01A level

T01A temperatures

PST

T01B level

T01B temperatures

T02A level

T02A temperatures

T02A and filter loop temperatures

G.545

Permeate flow rates

T02A Inner Temperature Tree

T02A temperatures

T02A temperatures

T02A temperatures

T02A temperatures

T02A Heating and Cooling

Pulsepot UFP-PP-T01A

Pulsepot UFP-PP-T03A

Pulsepot Levels

Filter UFP-FILT-T01A

Filter UFP-FILT-T03A

G.554

Filter UFP-FILT-T05A

Chemical Flow

Chemical Flow

Air Flows

PST

T02A Steam

T01A Steam

T01B Steam

Integrated Test A Data Plots 02/15/09 12:00 – 02/16/09 00:00

T01A level

T01A temperatures

T01B level

T01B temperatures

T02A level

T02A temperatures

T02A and filter loop temperatures

Pump Pressures and Flow

G.563

Axial pressure drop

Permeate flow rates

T02A Inner Temperature Tree

T02A Outer Temperature Tree

T02A temperatures

T02A temperatures

T02A temperatures

T02A temperatures

T02A Heating and Cooling

Pulsepot UFP-PP-T01A

Pulsepot UFP-PP-T03A

Pulsepot Levels

Filter UFP-FILT-T01A

Filter UFP-FILT-T03A

Filter UFP-FILT-T04A

G.572

Filter UFP-FILT-T05A

Chemical Flow

PST

Chemical Flow

Air Flows

T02A Steam

T01A Steam

T01B Steam

Integrated Test A Data Plots 02/16/09 00:00 – 02/16/09 12:00

T01A level

T01A temperatures

PST

T01B level

T01B temperatures

T02A level

T02A temperatures

T02A and filter loop temperatures

Axial pressure drop

Permeate flow rates

PST

T02A Inner Temperature Tree

T02A Outer Temperature Tree

T02A temperatures

T02A temperatures

T02A temperatures

T02A temperatures

T02A Heating and Cooling

Pulsepot UFP-PP-T01A

Pulsepot UFP-PP-T03A

Pulsepot Levels

Filter UFP-FILT-T01A

PST

G.589

Filter UFP-FILT-T03A

Filter UFP-FILT-T04A

Filter UFP-FILT-T05A

Chemical Flow

Chemical Flow

Air Flows

T02A Steam

T01A Steam

T01B Steam

Integrated Test A Data Plots 02/16/09 12:00 – 02/17/09 00:00

T01A level

T01A temperatures

T01B level

T01B temperatures

T02A level

T02A temperatures

PST

T02A and filter loop temperatures

G.599

Axial pressure drop

T02A Inner Temperature Tree

T02A temperatures

T02A temperatures

T02A temperatures

T02A temperatures

T02A Heating and Cooling

Pulsepot UFP-PP-T01A

Pulsepot UFP-PP-T03A

Pulsepot Levels

Filter UFP-FILT-T01A

Filter UFP-FILT-T03A

G.608

Filter UFP-FILT-T05A

Chemical Flow

PST

Chemical Flow

Air Flows

T02A Steam

T01A Steam

T01B Steam

Integrated Test A Data Plots 02/17/09 00:00 – 02/17/09 12:00

T01A level

T01A temperatures

T01B level

T01B temperatures

T02A level

T02A temperatures

T02A and filter loop temperatures

Axial pressure drop

Permeate flow rates

G.618

T02A Inner Temperature Tree

T02A temperatures

T02A temperatures

T02A temperatures

T02A temperatures

T02A Heating and Cooling

Pump Operation

Pulsepot UFP-PP-T01A

PST

Pulsepot UFP-PP-T03A

Pulsepot Levels

Filter UFP-FILT-T01A

G.625

Filter UFP-FILT-T03A

G.626

Filter UFP-FILT-T05A

Chemical Flow

PST

Chemical Flow

Air Flows

PST

T02A Steam

T01A Steam

T01B Steam

Integrated Test A Data Plots 02/17/09 12:00 – 02/18/09 00:00

T01A level

T01A temperatures

PST

T01B level

T01B temperatures

T02A temperatures

T02A and filter loop temperatures

Axial pressure drop

Permeate flow rates

T02A Inner Temperature Tree

T02A temperatures

T02A temperatures

PST

T02A temperatures

T02A temperatures

T02A Heating and Cooling

Pump Operation

Pulsepot UFP-PP-T01A

PST

Pulsepot UFP-PP-T03A

Pulsepot Levels

Filter UFP-FILT-T01A

Filter UFP-FILT-T03A

Pressure (psig) **Pressure (psig)**

Pressure (psig) **Pressure (psig)**

Filter UFP-FILT-T05A

Chemical Flow

Chemical Flow

Air Flows

T02A Steam

T01A Steam

T01B Steam

Integrated Test A Data Plots 02/18/09 00:00 – 02/18/09 12:00

T01A level

T01A temperatures

T01B level

T01B temperatures

T02A temperatures

G.652

T02A and filter loop temperatures

Pump Pressures and Flow

Axial pressure drop

Permeate flow rates

T02A Inner Temperature Tree

T02A temperatures

T02A temperatures

T02A temperatures

T02A temperatures

T02A Heating and Cooling

Pump Operation

Pulsepot UFP-PP-T01A

Pulsepot UFP-PP-T03A

Pulsepot Levels

Filter UFP-FILT-T01A

G.661

Filter UFP-FILT-T03A

G.662

Filter UFP-FILT-T05A

Chemical Flow

PST

Chemical Flow

Air Flows

T02A Steam

T01A Steam

T01B Steam

Integrated Test A Data Plots 02/18/09 12:00 – 02/19/09 00:00

T01A level

T01A temperatures

T01B level

T01B temperatures

T02A temperatures

T02A and filter loop temperatures

PST

Axial pressure drop

Permeate flow rates

T02A Inner Temperature Tree

T02A temperatures

T02A temperatures

T02A temperatures

T02A temperatures

T02A Heating and Cooling

Pump Operation

Pulsepot UFP-PP-T01A

Pressure (psig) **Pressure (psig)**

Pulsepot UFP-PP-T03A

Pulsepot Levels

Filter UFP-FILT-T01A

Filter UFP-FILT-T03A

Pressure (psig)

Pressure (psig) **Pressure (psig)**

Filter UFP-FILT-T05A

Chemical Flow

G.681

Chemical Flow

Air Flows

T02A Steam

T01A Steam

T01B Steam

Integrated Test A Data Plots 02/19/09 00:00 – 02/19/09 12:00

T01A level

T01A temperatures

T01B level

T01B temperatures

T02A temperatures

Temperatures (C)

T02A and filter loop temperatures

Axial pressure drop

Permeate flow rates

T02A Inner Temperature Tree

T02A temperatures

T02A temperatures

T02A temperatures

T02A temperatures

T02A Heating and Cooling

Pump Operation

Pulsepot UFP-PP-T01A

Pulsepot UFP-PP-T02A

Pulsepot UFP-PP-T03A

Pulsepot Levels

Filter UFP-FILT-T01A

Filter UFP-FILT-T02A

Pressure (psig) **Pressure (psig)**

Filter UFP-FILT-T03A

Filter UFP-FILT-T05A

Chemical Flow

Chemical Flow

Air Flows

T02A Steam

T01A Steam

T01B Steam

Integrated Test A Data Plots 02/19/09 12:00 – 02/20/09 00:00

T01A level

T01A temperatures

T01B level

T01B temperatures

T02A level

T02A temperatures

T02A and filter loop temperatures

Pump Pressures and Flow

Axial pressure drop

Permeate flow rates

T02A Inner Temperature Tree

T02A Outer Temperature Tree

T02A temperatures

T02A temperatures

T02A temperatures

T02A temperatures

T02A Heating and Cooling

Pulsepot UFP-PP-T01A

Pulsepot UFP-PP-T03A

Pulsepot Levels

Filter UFP-FILT-T01A

Filter UFP-FILT-T02A

Filter UFP-FILT-T03A

Chemical Flow

Chemical Flow

Air Flows

T02A Steam

T01A Steam

T01B Steam

Integrated Test A Data Plots 02/20/09 00:00 – 02/20/09 12:00

T01A level

T01A temperatures

T01B level

T01B temperatures

T02A temperatures

T02A and filter loop temperatures

Pump Pressures and Flow

Axial pressure drop

Permeate flow rates

T02A Inner Temperature Tree

T02A temperatures

T02A temperatures

T02A temperatures

T02A temperatures

T02A Heating and Cooling

Pump Operation

Pulsepot UFP-PP-T01A

Pulsepot UFP-PP-T03A

Pulsepot Levels

Filter UFP-FILT-T01A

Pressure (psig)

Pressure (psig)

PST

Filter UFP-FILT-T03A

PST

Filter UFP-FILT-T05A

Chemical Flow

Chemical Flow

Air Flows

T02A Steam

T01A Steam

T01B Steam

Integrated Test A Data Plots 02/20/09 12:00 – 02/21/09 00:00

T01A level

T01A temperatures

T01B level

T01B temperatures

T02A level

T02A temperatures

T02A and filter loop temperatures

Axial pressure drop

Permeate flow rates

T02A Inner Temperature Tree

PST

T02A temperatures

T02A temperatures

T02A temperatures

T02A temperatures

T02A Heating and Cooling

Pump Operation Pump power T42A (JT-0629)* Pump power T43A (JT-0637)* 1800 200 Pump speed T42A (ST-0627) [right axis] Pump speed T43A (ST-0639) [right axis] Suction flow to T42A (FT-0623) 180 1600 Discharge Flow from T43A (FT-0635) *This data is For Information Only160 1400 140 1200 Pump Power (HP)
Pump Flow (gpm) **Pump Power (HP) Pump Flow (gpm)** 120 **Speed (rpm)** (rpm) 1000 100 Speed 800 80 600 60 400 40 200 20 0 Ω 02/20/2009 02/20/2009 02/20/2009 02/20/2009 02/20/2009 02/20/2009 02/21/2009 12:00:00 14:00:00 16:00:00 18:00:00 20:00:00 22:00:00 00:00:00

Pulsepot UFP-PP-T01A

Pulsepot UFP-PP-T02A

Pulsepot UFP-PP-T03A

Pulsepot Levels

Filter UFP-FILT-T01A

G.751

Filter UFP-FILT-T03A

G.752

Filter UFP-FILT-T05A

Chemical Flow

Chemical Flow

Air Flows

T02A Steam

T01A Steam

G.755

T01B Steam

Integrated Test A Data Plots 02/21/09 00:00 – 02/21/09 12:00

T01A level

T01A temperatures

PST

T01B level

T01B temperatures

T02A temperatures

T02A and filter loop temperatures

Pump Pressures and Flow

Axial pressure drop

Permeate flow rates

T02A Inner Temperature Tree

T02A temperatures

T02A temperatures

T02A temperatures

T02A temperatures

T02A Heating and Cooling

Pump Operation

Pulsepot UFP-PP-T01A

Pulsepot UFP-PP-T03A

Pulsepot Levels

Filter UFP-FILT-T01A

Filter UFP-FILT-T03A

Filter UFP-FILT-T05A

Chemical Flow

Chemical Flow

Air Flows

T02A Steam

T01A Steam

PST

T01B Steam

Integrated Test A Data Plots 02/21/09 12:00 – 02/22/09 00:00

T01A level

T01A level

Level (in)

T01B level

T01B temperatures

T02A temperatures

T02A and filter loop temperatures

Axial pressure drop

Permeate flow rates

T02A Inner Temperature Tree

T02A temperatures

T02A temperatures

T02A temperatures

T02A temperatures

T02A Heating and Cooling

Pulsepot UFP-PP-T01A

Pulsepot UFP-PP-T02A

Pulsepot UFP-PP-T03A

Pulsepot Levels

Filter UFP-FILT-T01A

Filter UFP-FILT-T02A

Filter UFP-FILT-T03A

Filter UFP-FILT-T04A

Pressure (psig) **Pressure (psig)**

Filter UFP-FILT-T05A

Chemical Flow

Chemical Flow

Air Flows

T02A Steam

T01A Steam

T01B Steam

Integrated Test A Data Plots 03/20/09 00:00 – 03/20/09 12:00

T01A level

T01A temperatures

T01B level

T01B temperatures

T02A level

T02A temperatures

T02A and filter loop temperatures

Axial pressure drop

Permeate flow rates

T02A Inner Temperature Tree

T02A temperatures

T02A temperatures

T02A temperatures

T02A temperatures

T02A Heating and Cooling

Pulsepot UFP-PP-T01A

Pressure (psig) **Pressure (psig)**

Pulsepot UFP-PP-T03A

Pulsepot Levels

G.804

Filter UFP-FILT-T01A

Filter UFP-FILT-T03A

Filter UFP-FILT-T05A

Chemical Flow

Air Flows

T02A Steam

T01A Steam

T01B Steam

Integrated Test A Data Plots 03/20/09 12:00 – 03/21/09 00:00

T01A level

T01A temperatures

PST

T01B level

T01B temperatures

T02A level

T02A temperatures

PST

T02A and filter loop temperatures

PST

Axial pressure drop

Permeate flow rates

T02A Inner Temperature Tree

T02A temperatures

T02A temperatures

T02A temperatures

T02A temperatures

Pump Operation Pump power T42A (JT-0629)* Pump power T43A (JT-0637)* 2000 140 Pump speed T42A (ST-0627) [right axis] Pump speed T43A (ST-0639) [right axis] Suction flow to T42A (FT-0623) 1800 Discharge Flow from T43A (FT-0635) 120 This data is For Information Only Ω 1600 100 1400 Pump Power (HP)
Pump Flow (gpm) **Pump Power (HP) Pump Flow (gpm)** 1200 80 Speed (rpm) **Speed (rpm)** 1000 60 800 600 40 400 20 200 0 Ω 03/20/2009 03/20/2009 03/20/2009 03/20/2009 03/20/2009 03/20/2009 03/21/2009 12:00:00 14:00:00 16:00:00 18:00:00 20:00:00 22:00:00 00:00:00

Pulsepot UFP-PP-T01A

Pulsepot UFP-PP-T03A

Pulsepot Levels

Filter UFP-FILT-T01A

Pressure (psig) **Pressure (psig)**

Filter UFP-FILT-T03A

Filter UFP-FILT-T05A

Chemical Flow

Chemical Flow

Air Flows

T02A Steam

T01A Steam

T01B Steam

Integrated Test A Data Plots 03/21/09 00:00 – 03/21/09 12:00

T01A level

T01A temperatures

PST

T01B level

T01B temperatures

T02A level

T02A temperatures

T02A and filter loop temperatures

T02A Inner Temperature Tree

T02A temperatures

T02A temperatures

T02A temperatures

T02A temperatures

T02A Heating and Cooling

Pump Operation

Pulsepot UFP-PP-T01A

G.839

Pulsepot UFP-PP-T03A

Pulsepot Levels

Filter UFP-FILT-T01A

Filter UFP-FILT-T03A

G.842

Filter UFP-FILT-T05A

Chemical Flow

Pressure (psig)

Flow (kg/min or gpm)

Chemical Flow

Air Flows

PST

T02A Steam

T01A Steam

T01B Steam

Integrated Test A Data Plots 03/21/09 12:00 – 03/22/09 00:00

T01A level

T01A temperatures

G.848

T01B level

T01B temperatures

T02A level

T02A temperatures

PST

T02A and filter loop temperatures

Pump Pressures and Flow

Axial pressure drop

Permeate flow rates

T02A Inner Temperature Tree

T02A Outer Temperature Tree

T02A temperatures

T02A temperatures

T02A temperatures

T02A temperatures

T02A Heating and Cooling

Pump Operation

G.856

Pulsepot UFP-PP-T01A

Pulsepot UFP-PP-T02A

Pulsepot UFP-PP-T03A

Pulsepot Levels

Filter UFP-FILT-T01A

PST

Filter UFP-FILT-T03A

Filter UFP-FILT-T04A

Chemical Flow

G.861

Chemical Flow

Air Flows

T02A Steam

T01A Steam

T01B Steam

Appendix H

Physical Properties

Appendix H: Physical Properties

H.1 Rheology

Specimens collected for the Integrated Test A were done under TI-065, Steps B.1.2, B.1.14, B.1.15, B.1.18, B.1.19, B.1.20, and B.1.21.

Multiple specimens of PEP slurry were delivered to APEL for rheological analysis between $2/8/09 - 2/23/09$. Specimens were analyzed to determine either their flow curves (i.e., plots of shear stress vs. shear rate) or shear strengths. The analysis was done using an Anton Parr Rheometer MCR 301 SN80371304 (for flow curve) and Haake RS-600 (for shear strength). Rheometer calibration verifications were done 3 times for Anton Parr Rheometer MCR 301 SN80371304 and 1 time for Haake RS-600 during this time period by Maria Luna in accordance with OA procedure TPR-WTP-PEP-049. Two Newtonian standards with different viscosities were used to check the calibration of these systems: a 9.4 cP Brookfield Standard Lot # 062408 and a 99.0 cP Brookfield Standard Lot #071008. All verifications were within the 10% of the reported values.

Figures 1-3 are examples of the calibration curves performed on the Anton Parr Rheometer MCR 301 SN80371304 with 9.4 cP and 99.0 cP Brookfield Standards and the Haake RS-600 with 99.0 cP Brookfield Standard, respectively. Note that the calibration for Haake RS-600 is only a performance check of the Haake RS-600 system, since the measurements performed on this system are shear strength measurements and Newtonian standards have a shear strength of zero.

Figure 2. Flow curve of the 99.0 cP Newtonian standard run on the Anton Paar rheometer

Figure 3. Flow curve of the 99.0 cP Newtonian standard run on the Haake RS-600 rheometer.

The analysis was performed in accordance with procedure TPR-WTP-PEP-049 and the data were collected digitally using the RHEOPLUS/32 V3.21 21003751-33024 software (for flow curve) and RHEOWIN V2.96 software (for shear strength) The same software was used to operate the corresponding rheometers. For the flow curve measurement, the specimens were prepared for analysis by shaking the bottle before extracting the sample. In order to obtain supernatant from the slurry, the specimen was centrifuged at 1000 times g for 1 hour. Experimental details were recorded in laboratory record book 60127.

Flow curve tests for both slurry and supernatant were done with the cup and bob sensor system CC27-SN13634. Shear stress was measured as a function of shear rate while the shear rate was ramped from 0 to 1000 1/sec over 5 min, followed by a 1 minute hold at 1000 1/sec and a ramp down to 0 1/sec over 5 minutes. Each specimen was generally measured three times. An initial measurement ("FRESH RUN") was made on a fresh sample taken from the specimen, that same sample was then re-run a second time ("RERUN"). The rheometer was cleaned, and a third measurement ("REPEAT FRESH RUN") was made by taking a second aliquot from the specimen. Note that the sample, A-02ARL-XXX-XX-2842-RHE-4, was measured only two times with two fresh samples due to suspected sample contamination. Samples were placed in the cup up to the designated fill line (about 8 to 10 mL). Settling cannot be prevented during the measurement if the particles are large and the viscosity low; however, no evidence of significant settling was seen in the data or the cup after testing.

Figure 4. Typical rheogram of a slurry sample with low shear strength.

Figure 4 is a typical rheogram of a slurry sample (sample A-02AOL-015-10-2319-RHE-4). There are three features that were typically characteristic of all of the slurry specimens: i) a yield stress at the onset of flow, (indicated by the blue arrow, "A"), ii) an approximately linear region where shear stress is linearly proportional to shear rate "B", and iii) a discontinuity where the slope changes and where there are more fluctuations in the data (starting about 700 1/sec in Figure 4, indicated by the red arrow, "C"). The data at very low shear rates was not included in the analysis to avoid overweighting the Bingham plastic and Casson curve fit calculations performed by the RHEOPLUS/32 V3.21 21003751-33024 software. Slope changes and data fluctuation as shown in "C" above made it difficult to fit the data to a single mathematical expression because such behavior is not included in existing fitting models. Several different factors might cause such behavior: the flow instability associated with laminar Taylor vortices and other disturbances such as cavitation, minor settling, or the release of entrained gas. Previously it was presumed that these flow instabilities were associated with Taylor vortices. The data analysis team evaluated these instabilities and decided to perform curve fit analysis using only the data prior to the discontinuity (e.g., up to 700 1/sec as shown above).

A simple hydrodynamic theory may provide a reasonable estimate and physical insights for instability behavior. The hydrodynamic transition from laminar Couette flow to laminar Taylor vortices occurs when the Reynolds number of the experiment exceeds a geometrically determined critical Reynolds number, Re_c. Assuming the infinite length of the fluid column, the critical Reynolds number in our case may be described by¹

^{1.} L. D. Landau and E. M. Lifshitz, "Course of Theoretical Physics - Volume 6: Fluid Mechanics," 2nd Ed., Butterworth-Heinemann, Oxford (1987).

$$
\text{Re}_c = 41.2 \sqrt{\frac{(R_o + R_i)}{2h}}
$$

where R_i and R_o denote bob radius and cup radius. Here h denotes a gap between the bob and cup, which is $R_o - R_i$. The Reynolds number of the experiment, Re, can be defined by $\rho h \Omega R_i / \mu_{app}$ where ρ , Ω , and μ_{app} represent the density of the sample, the angular velocity of the bob, and the apparent viscosity of the sample respectively. Using the geometries of the cup and bob sensor system CC27-SN13634, the critical Reynolds number is expected to be about 145. (It is also noteworthy that the transition to turbulent vortices occurs at a much higher Reynolds number). However, for our experimental set up, $Re \approx 100$ at 700 1/sec using sample density and apparent viscosity values of 1.2 $g/cm³$ and 10 cP, respectively (these values are typical for most slurry samples). Considering that the formula for the critical Reynolds number is based on a Newtonian fluid, one can expect that two Reynolds numbers are comparable and the hydrodynamic transition to laminar Taylor vortices occurs around 700 1/sec. Consequently, the slope changes and data fluctuation as shown in "C" may be due to the onset of the laminar Taylor vortices although other disturbances are possibly coupled. Applying this to the rheology of the supernatant samples, one may expect that the transition occurs at a much smaller shear rate since the viscosity of the supernatant is typically 10 times less than that of the slurry samples, which was indeed observed in our tests; such transition occurs typically about 120 1/sec.

The flow curves did not show significant hysteresis and the data were fit with Newtonian, Bingham Plastic, and Casson flow-curve descriptors using the RHEOPLUS curve fit software, depending on the samples' rheological behavior. The software fits the entire range of data collected. However, as explained previously, a partial range of data was used to obtain fitting parameters for most of samples in the Integrated Test A because of the slope changes and/or fluctuations. The partial ranges of data were typically 50-700 1/sec and 0-120 1/sec for slurry and supernatant samples respectively.

For shear strength test, a 1.6×1.6 cm vane shear spindle was used and an enough space (at least 1.6cm away from the top solid bed and the side wall and bottom of a test container) was provided for proper measurements. A and M factors for the tests are 116568.602 Pa/N·m and 1.0 (1/sec)(rad/sec) respectively indicated in RHEOWIN V2.96 software. Settling times for the test with the same sample are 1, 2, 4, 8, 16, 32, and 72 hrs. For each settling time, shear stresses were collected as a function of duration time under a constant shear rate (0.03 1/sec) during 2 minutes. Then, a maximum shear stress was obtained for each settling time.

The following table is a brief summary of samples and their specifications (chronological order). Note that all measurements were done at 25° C.

Summary of Results

Five samples, A-02AOL-018-XX-2383-RHE-4, A-02AOL-018-XX-2384-RHE-4, B-FRPGM-001-XX-2718-RHE-4 (identical to B-FRPGM-001-XX-2707-RHE-4), A-02AML-021-XX-2455-RHE-4, and A-02AML-021-XX-2456-RHE-4, were tested with a 1.6×1.6 cm vane shear spindle (Haake RS-600). Note that B-FRPGM-001-XX-2718-RHE-4 and B-FRPGM-001-XX-2707-RHE-4 were mixed to obtain sufficient precipitates to measure the shear strength after removing identical amounts of supernatants from each sample bottle, since the amount of precipitates from either sample was not enough for the measurement. All samples showed continuous increases in the maximum shear strength (within the experimental uncertainty) as the settling time increases and the complete asymptotes were not observed due to insufficient settling time. Considering both the limited settling time and an asymptotic behavior as settling time approaches 72 hrs, all samples except A-02AOL-018-XX-2384-RHE4 appear to be adequate for handling in the WTP (i.e. less than 30 Pa of the maximum shear strength). The shear strength measurement with a longer settling time might be needed to ensure an asymptotic behavior for A-02AOL-018-XX-2384-RHE4.

Some samples were run only as slurrics and some only as supernatants as was needed for the matrix support for that part of the test plan. Most slurry samples were reasonably well fitted with the Bingham plastic model. All supernatants did not exhibit a yield stress and Newtonian fits appear to be the best fit.

The average rheological parameters for flow curve measurements are presented in Appendix A. More detailed data for flow curve and shear strength measurements are presented in Appendix B and the actual rheograms for each test and the curve fits are presented in Appendix C.

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Appendix A

Average Rheological Parameters for Shear Stress vs. Shear Rate Measurements

H.2 Heat Capacity

PEP Slurry Sample Heat Capacity Report

Analysis

This report presents results of heat capacity measurements for four PEP simulant slurry samples delivered via Chain of Custody form numbers WTP-COC-063-048 on 03/13/2009 and WTP-COC-063-051 on 03/16/2009. The samples provided were:

- B 02AML 005 XX 2949 HTC 3
- B 02AML 007 XX 2965 HTC 3
- B 02AML 008 BT 2972 HTC 3
- B 02AML 008 00 2988 HTC 3

Three nominal 30 mg subsamples were taken from each of these samples as they were vigorously stirred using a pipette with an enlarged tip to assure that the slurry samples were representative. We analyzed each slurry sample in triplicate, recognizing that obtaining a 30 mg representative sample is challenging.

A Perkin-Elmer DSC7 differential scanning calorimeter (DSC) was used to determine the heat capacity of each subsample at temperatures between 20°C and 95°C. The DSC7 was temperature- and enthalpy-calibrated using a gallium standard (NIST SRM 2234) (m.p. 29.8°C) and/or an indium standard (NIST SRM 2232) (m.p. 156.6°C).

The method used for heat capacity measurement was as provided in the DSC7 operating manual. In this method, the heat capacity of the empty sample pan is measured to provide the "baseline"; the sample pan was a gold "volatile" sample pan designed to contain volatile samples such as the water in these PEP slurries. The heat capacity of a reference or standard material such as sapphire or high purity water is then determined; the purity of these water standards was determined by measuring their electrical conductivity. Finally the heat capacity of the sample is determined. The measured heat capacity of the sample is adjusted to remove the contribution of the empty pan. We further adjusted the reported heat capacity by applying a measured response factor for the high purity water.

Optimally the heat capacity measurement approach would include analyzing the standard or reference material in the same container as the sample would be analyzed; however, because analysis of these slurry samples require a hermetic seal which cannot be broken and resealed, a different but nearly equivalent gold volatile sample pan is used for the water. In addition, we characterized a sapphire standard to demonstrate instrument performance but unfortunately, the heat capacity of sapphire (0.8 J/g $^{\circ}$ C) is significantly lower than that of the PEP slurries (\sim 3.5 J/g) and does not provide an adequate calibration adjustment.

Results

The expected results for the measurements can be generated by considering what the theoretical heat capacity would be for slurries with solid fraction in the range that are expected in the samples. The slurry is mostly water, which at 20 \degree C is known to have a specific heat of 4.1818 J/g \degree C [1]. It is also known that at 20 $^{\circ}$ C, the heat capacity of boehmite is roughly 0.9 J/g $^{\circ}$ C [2]. So for slurries with mass fraction of 10%, 20%, and 30% boehmite in water, the theoretical heat capacities would be, respectively:

- \bullet 4.1818 J/g^oC * .9+ 0.9 J/g^oC * .1 = **3.85 J/g**^oC,
- 4.1818 J/g^oC * .80 + 0.9 J/g^oC * .2 = **3.52 J/g**^oC, and
- \bullet 4.1818 J/g^oC * .7+ 0.9 J/g^oC * .3 = **3.20 J/g**^oC.

Table 1 summarizes the results from these analyses by providing the mean measured heat capacities with their standard deviations. In general, the measured heat capacities are consistent with our expectations of hear capacities in the 3.2 to 3.8 J/g°C assuming solids contents ranging from 10 to 30 wt%. We used only those analyses found to be internally self-consistent, not erratic and as-such, we discarded one measurement for sample B 02AML 005 XX 2949 HTC 3, one measurement for sample B 02AML 007 XX 2965 HTC 3, and one from B 02AML 008 BT 2972 HTC 3, but replaced it with a re-analysis.

Temperature	B 02AML 005 XX	B 02AML 007 XX	B 02AML 008 BT	B 02AML 008 00
	2949 HTC 3	2965 HTC 3	2972 HTC 3	2988 HTC 3
	C_p (J/g ^o C)			
35° C	2.98 ± 0.00	3.21 ± 0.01	3.23 ± 0.03	3.22 ± 0.09
40° C	2.97 ± 0.00	3.22 ± 0.01	3.25 ± 0.02	3.23 ± 0.09
45° C	2.97 ± 0.00	3.24 ± 0.01	3.26 ± 0.03	3.23 ± 0.09
50° C	2.96 ± 0.02	3.25 ± 0.00	3.26 ± 0.03	3.23 ± 0.09
55° C	2.96 ± 0.02	3.25 ± 0.00	3.27 ± 0.02	3.24 ± 0.08
60° C	2.96 ± 0.02	3.26 ± 0.00	3.28 ± 0.02	3.23 ± 0.09
65° C	2.95 ± 0.03	3.27 ± 0.00	3.28 ± 0.02	3.23 ± 0.09
70° C	2.95 ± 0.04	3.28 ± 0.00	3.29 ± 0.02	3.23 ± 0.08
75° C	2.95 ± 0.04	3.28 ± 0.01	3.30 ± 0.02	3.23 ± 0.08
80° C	2.96 ± 0.05	3.29 ± 0.01	3.29 ± 0.02	3.23 ± 0.08
85°C	2.97 ± 0.05	3.29 ± 0.02	3.30 ± 0.02	3.22 ± 0.08
90° C	2.98 ± 0.05	3.30 ± 0.02	3.30 ± 0.02	3.22 ± 0.08

Table 1: Results of Heat Capacity Measurements.

The results from each sample measurement are presented in graphical form in Figures 1-4. In general, the heat capacity will increase nominally linearly as a function of temperature. In general, the average measured heat capacities do increase linearly with temperature, although some deviations do exist.

Figure 1: Heat Capacity Curve for Sample B_02AML_005_XX_2949_HTC_3

Figure 2: Heat Capacity Curve for Sample B_02AML_007_XX_2695_HTC_3

Figure 3: Heat Capacity Curve for Sample B_02AML_008_BT_2972_HTC_3

Figure 4: Heat Capacity Curve for Sample B_02AML_008_00_2988_HTC_3

Conclusions

The measured heat capacity values for the PEP slurry samples discussed in this report are well within the expected range.

References

[1] Lide, D. "CRC Handbook of Chemistry and Physics," $89th$ Edition, 2008-2009, 6-2.

[2] Hemingway, B.S., Robie, R.A., Apps, J.A., "Revised values for the thermodynamic properties of boehmite, AlO(OH), and related species and phases in the system Al-H-O," American Mineralogist, Volume 76, pages 445-457, 1991.

PEP Slurry Sample Heat Capacity Report Analysis

This report presents results of heat capacity measurements for four PEP simulant slurry samples delivered via Chain of Custody form numbers WTP-COC-063-020 on 02/02/2009 and WTP-COC-063-024 on 02/03/2009. The samples provided were:

- A 01AIM 002 XX 1519 HTC 3
- A 01AIM 003 00 1538 HTC 3
- A 01AIM 003 BT 1526 HTC 3
- B FRPGM 001 XX 2706 HTC 3

Three nominal 30 mg subsamples were taken from each of these samples as they were vigorously stirred using a pipette with an enlarged tip to assure that the slurry samples were representative. We analyzed each slurry sample in triplicate, recognizing that obtaining a 30 mg representative sample is challenging.

A Perkin-Elmer DSC7 differential scanning calorimeter (DSC) was used to determine the heat capacity of each subsample at temperatures between 20°C and 95°C. The DSC7 was temperature- and enthalpy-calibrated using a gallium standard (NIST SRM 2234) (m.p. 29.8°C) and/or an indium standard (NIST SRM 2232) (m.p. 156.6°C).

The method used for heat capacity measurement was as provided in the DSC7 operating manual. In this method, the heat capacity of the empty sample pan is measured to provide the "baseline"; the sample pan was a gold "volatile" sample pan designed to contain volatile samples such as the water in these PEP slurries. The heat capacity of a reference or standard material such as sapphire or high purity water is then determined; the purity of these water standards was determined by measuring their electrical conductivity. Finally the heat capacity of the sample is determined. The measured heat capacity of the sample is adjusted to remove the contribution of the empty pan. We further adjusted the reported heat capacity by applying a measured response factor for the high purity water.

Optimally the heat capacity measurement approach would include analyzing the standard or reference material in the same container as the sample would be analyzed; however, because analysis of these slurry samples require a hermetic seal which cannot be broken and resealed, a different but nearly equivalent gold volatile sample pan is used for the water. In addition, we characterized a sapphire standard to demonstrate instrument performance but unfortunately, the heat capacity of sapphire (0.8 J/g $^{\circ}$ C) is significantly lower than that of the PEP slurries (\sim 3.5 J/g) and does not provide an adequate calibration adjustment.

Results

The expected results for the measurements can be generated by considering what the theoretical heat capacity would be for slurries with solid fraction in the range that are expected in the samples. The slurry is mostly water, which at 20°C is known to have a specific heat of 4.1818 J/g°C [1]. It is also known that at 20 $^{\circ}$ C, the heat capacity of boehmite is roughly 0.9 J/g $^{\circ}$ C [2]. So for slurries with mass fraction of 10%, 20%, and 30% boehmite in water, the theoretical heat capacities would be, respectively:

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\bullet 4.1818 J/g<sup>o</sup>C * .9+ 0.9 J/g<sup>o</sup>C * .1 = 3.85 J/g<sup>o</sup>C,
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- $4.1818 \text{ J/g}^{\circ}\text{C}$ * $.80 + 0.9 \text{ J/g}^{\circ}\text{C}$ * $.2 = 3.52 \text{ J/g}^{\circ}\text{C}$, and
- \bullet 4.1818 J/g^oC * .7+ 0.9 J/g^oC * .3 = **3.20 J/g**^oC.

Table 1 summarizes the results from these analyses by providing the mean measured heat capacities with their standard deviations. In general, the measured heat capacities are consistent with our expectations of hear capacities in the 3.2 to 3.8 J/g°C assuming solids contents ranging from 10 to 30 wt%. We used only those analyses found to be internally self-consistent, not erratic and as-such, we discarded one measurement for sample A_01AIM_002_XX_1519_HTC_3, one measurement for sample A_01AIM_003_BT_1526_HTC_3 but replaced it with a reanalysis, and one measurement for Sample A_01AIM_003_00_1538_HTC_3.

Temperature	A 01AIM 002 XX	A 01AIM 003 00-	A 01AIM 003 BT-	001 FRPGM B	
	-1519 HTC 3	1538 HTC 3	1526 HTC 3	XX-2706 HTC 3	
	C_p (J/g ^o C)	C_p (J/g°C)	C_p (J/g ^o C)	C_p (J/g ^o C)	
35° C	3.32 ± 0.01	3.46 ± 0.00	3.33 ± 0.09	3.33 ± 0.02	
40° C	3.32 ± 0.02	3.48 ± 0.01	3.35 ± 0.09	3.33 ± 0.02	
45° C	3.34 ± 0.01	3.49 ± 0.01	3.36 ± 0.09	3.33 ± 0.02	
50° C	3.33 ± 0.01	3.51 ± 0.02	3.36 ± 0.09	3.34 ± 0.02	
55° C	3.34 ± 0.00	3.51 ± 0.03	3.37 ± 0.09	3.34 ± 0.02	
60° C	3.35 ± 0.01	3.53 ± 0.04	3.39 ± 0.09	3.34 ± 0.02	
65° C	3.36 ± 0.02	3.54 ± 0.05	3.40 ± 0.09	3.34 ± 0.02	
70° C	3.37 ± 0.03	3.54 ± 0.05	3.41 ± 0.09	3.35 ± 0.02	
75° C	3.38 ± 0.04	3.55 ± 0.06	3.43 ± 0.10	3.35 ± 0.02	
80° C	3.39 ± 0.06	3.56 ± 0.07	3.43 ± 0.10	3.35 ± 0.02	
85°C	3.40 ± 0.07	3.58 ± 0.09	3.44 ± 0.10	3.35 ± 0.03	
90° C	3.40 ± 0.08	3.59 ± 0.10	3.45 ± 0.10	3.36 ± 0.03	

Table 1: Results of Heat Capacity Measurements

The results from each sample measurement are presented in graphical form in Figures 1-4. In general, the heat capacity will increase nominally linearly as a function of temperature. In general, the average measured heat capacities do increase linearly with temperature, although some deviations do exist.

Figure 1: Heat Capacity Curve for Sample A_01AIM_002_XX_1519_HTC_3

Figure 2: Heat Capacity Curve for Sample A_01AIM_003_00_1538_HTC_3

Figure 3: Heat Capacity Curve for Sample A_01AIM_003_BT_1526_HTC_3

Figure 4: Heat Capacity Curve for Sample B_FRPGM_001_XX_2706_HTC_3

Conclusions

The measured heat capacity values for the PEP slurry samples discussed in this report are well within the expected range.

References

[1] Lide, D. "CRC Handbook of Chemistry and Physics," $89th$ Edition, 2008-2009, 6-2.

[2] Hemingway, B.S., Robie, R.A., Apps, J.A., "Revised values for the thermodynamic properties of boehmite, AlO(OH), and related species and phases in the system Al-H-O," American Mineralogist, Volume 76, pages 445-457, 1991.

H.3 Scanning Electron Microscopy

Two specimens were received for scanning electron microscopy (SEM) examinations in support of work being performed at the Pretreatment Engineering Platform (PEP) during the course of Integrated Test A. The morphologies of these specimens were analyzed using a JEOL 5900 SEM in accordance with procedure APEL-102-SEM, Rev. 1.

Sample Preparation

Once received from PEP, the specimens were cleaned of nitrates. This cleaning process included 3 cycles of adding 10 mL of fresh 0.01 M NaOH to the centrifuge tube, vortexing for 3 sets of 15 seconds, centrifuging at 4500 rpm for 10 minutes, decanting and then adding 10 mL of fresh 0.01 M NaOH. These washing steps were recorded in laboratory record book BNW-60176.

After the last rinse, the NaOH and the pellet were vortexed, a small volume of the slurry was drawn up using a pipette, and a drop was placed on the top of an aluminum SEM sample stub. It was then placed in an oven at approximately 100°C to dry overnight. Once it was dry, it was coated with a thin layer of Au/Pd using the Polaron Range plasma sputter coater in APEL/102. The Au/Pd layer was deposited using 800 V and 10 mA for 180 s. The coated specimen was placed in an SEM sample holder and was placed in the SEM for analysis.

Analysis

Scanning electron micrographs were collected for informational purposes only using the Scan 4 option with 160 s collection times using the settings as seen below in Table 1. See Table 2 for information regarding the person that performed the analysis as well as the date of analysis. Images at the four magnifications listed in Table 1 were compiled in a 2×2 arrangement to show a macroscopic to microscopic view of the particles. See APPENDIX A: Micrograph Collages.

Results

The micrographs collected for this report compare the morphology of undissolved solids in the PEP slurry at the beginning of the PEP treatment process (B_FRPGM_001_XX_2713_XSP_4_B; "B_FRPGM" for short) and after having completed the oxidative leaching step (A_02AML_021_XX_2460_XSP_4; "A_02AML" for short). The differences between these specimens were readily apparent. Specimen B_FRPGM appeared to be single phase, and consisted of particles that were somewhat tabular and faceted, with sizes ranging from 1 to 10 μ m (due to similar, average atomic numbers for boehmite and gibbsite, there is not a readily apparent difference in contrast or brightness between them when imaged in an SEM). Specimen A_02AML appeared to consist of two kinds of particles that were in somewhat of a bimodal size distribution. The smaller particles were approximately 180 nm in apparent diameter, and were equiaxed. The larger particles had distinct facets, and were approximately $1 \mu m$ in apparent

diameter. Morphologically, the larger particles appeared similar to the particles in B_FRPGM. Based on XRD results from a separate report, specimen B_FRPGM consisted of 51.6 wt% boehmite and 32.1 wt% gibbsite (balance was amorphous), and specimen A_02AML consisted of 38.4 wt% boehmite, and 1.1 wt% hematite (balance was amorphous). Thus, the micron-scale, faceted particles in A_02AML might be residual, crystalline boehmite (orthorhombic crystal structure) that remained after the treatment process, whereas the sub-micron, equiaxed particles could be the large fraction of amorphous material (~80%, as indicated by XRD analysis) which was the product from the PEP process. More definitive chemical information about these different particles is possible using energy dispersive spectroscopy, which is available, if desired. One can conclude that the PEP treatment process had a dramatic impact on the size, shape, composition, and the atomic structure (crystalline vs amorphous) of the undissolved solids in the slurry that was tested.

Sample ID	Voltage (KV)	Spot Size	Twore 1. I didn't color for explaining integes. Detector	Working Distance (mm)	Magnification
B FRPGM 001 XX 2713 XSP 4 B	20	30	SEI	12	$1,000\times$
B FRPGM 001 XX 2713 XSP 4 B	20	30	SEI	12	$2,500\times$
B FRPGM 001 XX 2713 XSP 4 B	20	25	SEI	12	$5,000\times$
B FRPGM 001 XX 2713 XSP 4 B	20	25	SEI	12	$10,000\times$
A 02AML 021 XX 2460 XSP 4	15	24	BSE	10	$1,000\times$
A 02AML 021 XX 2460 XSP 4	15	24	BSE	10	$2,500\times$
A 02AML 021 XX 2460 XSP 4	15	24	BSE	10	$5,000\times$
A 02AML 021 XX 2460 XSP 4	15	24	BSE	10	$10,000\times$

Table 1. Parameters used for capturing images.

Table 2. Analysis information.

Sample ID	Date of	Analysis	Signature	
	Analysis	Performed By		
B FRPGM 001 XX 2713 XSP 4 B	03/05/2009	Brian Riley		
A 02AML 021 XX 2460 XSP 4	03/18/2009	Brad Johnson		

APPENDIX A: Micrograph Collages

SEM micrographs from Pretreatment Engineering Platform (PEP) specimen B_FRPGM_001_XX_2713_XSP_4_B. The specimen, while in the original 50 mL centrifuge tube, was rinsed with 0.01 M NaOH, vortexed, centrifuged (4500 rpm for 10 minutes), and decanted $3\times$ to clean off the nitrates. After the final rinse, the contents of the tube were vortexed in order to resuspend the pellet, and a drop of the suspended particles was placed on an Al SEM stub and placed in an oven at approximately 105^oC to dry overnight. Once dry, the specimen was coated with Au/Pd and analyzed using the APEL/102 SEM (JEOL 5900).

Figure 1. Scanning electron micrographs collected for informational purposes only on B_FRPGM_001_XX_2713_XSP_4_B at different magnifications

SEM micrographs from Pretreatment Engineering Platform (PEP) specimen A_02AML_021_XX_2460_XSP_4. The specimen, while in the original 50 mL centrifuge tube, was rinsed with 0.01 M NaOH, vortexed, centrifuged (4500 rpm for 10 minutes), and decanted $3\times$ to clean off the nitrates. After the final rinse, the contents of the tube were vortexed in order to resuspend the pellet, and a drop of the suspended particles was placed on an Al SEM stub and placed in an oven at approximately 105°C to dry overnight. Once dry, the specimen was coated with Au/Pd and analyzed using the APEL/102 SEM (JEOL 5900).

Figure 2. Scanning electron micrographs collected for informational purposes only on A_02AML_021_XX_2460_XSP_4 at different magnifications

H.4 X-Ray Diffraction

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 * Optional Information

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O – Optional, comment resolution would provide clarification, but does not impact the integrity of the QA-F0604-01, Rev. 0

X-ray diffraction analysis was performed on a series of specimens in support of experimental work for the Pretreatment Engineering Platform tests. Sample analysis was carried out by the Pacific Northwest National Laboratory Advanced Processing and Application Group. The XRD instrument used was the Scintag PAD V X-ray Diffractometer, Property Number WD33356, located in lab 102 in the APEL building. The data range for the sample was 5° to 80° 2-theta, with a step size of 0.04° 2-theta and count time 2.0 - 4.0 seconds per step. Copper Ka X-rays were used. The X-ray tube operating conditions were 45 kV and 40 mA.

Sample Prep:

The as-received samples were rinsed 3 times with a 0.01M NaOH solution. Rinsing consisted of four steps: adding rinsate, votexing, centrifuging, and decanting of the rinsate. The process was repeated three times. The samples were then dried at 105°C overnight to drive off all liquid. The dried powder was placed into a tungsten carbide milling chamber and milled for 1 min in the angstrom shaker mill. A portion of the powder was weighted out to the nearest hundredth of a milligram. Then SRM 674b, TiO2 internal standard, was spiked in at a known mass, given in Table 1, with the dried powder and milled again for 2 min to insure a homogenous mixture of the two powders. The sample was then mounted into a sample holder. Replications were made and then summed for better counting statistics; the number of replications of each sample are shown in Table I.

Phase identification was done by use of the JADE search match routines (version 6.0, Materials Data Inc.) with comparison to the ICDD (International Centre for Diffraction Data) data base PDF-2 release 1999, which includes the ICSD (Inorganic Crystal Structure Database maintained by Fachinformationszentrum (FIZ), Karlsruhe, Germany). Searches were restricted to the PDF and ICSD Inorganic sections.

Table 2 summarizes the results of the XRD analysis to include; identified phases, mass %, and crystallite size of each phase. Balance of material in the sample is assumed to be amorphous. Boehmite is the major phase

except in the first and last samples or the series. Gibbsite was only observed in the second to last sample analyzed.

Sample ID	Boehmite		Gibbsite		Hematite		Other
	Amount $(wt\%)$	Size (A)	Amount $(wt\%)$	Size (\AA)	Amount $(wt\%)$	Size (A)	
A 01AIM 003 16 1607 XSP 4 B	48.5	336			2.9	609	FeOCl, trace
A 01AIM 13A XX 1880 XSP 4 B	47.4	336			3.9	609	
A 01AIM 13A 16 1871 XSP 4 B	58.1	380			3.7	525	
A 01AIM 13C 16 2124 XSP 4 B	49.3	335			1.6	555	
A 01AIM 006 XX 1631 XSP 4 B	78.2	390			1.2	699	
A 01AIM 13C XX 2134 XSP 4 B	51.2	336			2.2	751	
B FRPGM 001 XX 2713 XSP 4 B	51.6	414	32.1	160			
A 02AML 021 XX 2460 XSP 4	38.4	554			1.1	750	

Table 2. Measured Concentration and Estimated Crystallite Sizes of Identified Phases

Appendix A contains all of the individual reports for each sample analyzed. Individual reports contain information related to sample prep, scanning parameters, data plots, and PDF card information for phases identified.

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 ${\large \bf Appendix~A}$ Individual Reports of Each Sample

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XRD data from the samples identified in Table I have been examined for identification of crystalline phases present.

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Table I: Samples Examined in this Series

XRD File $ID(a)$		Count Time per	Number of	Internal Standard	
Identification		Step (Seconds)	Replications		
A 01 AIM 003 16 1607 XSP 4 B	020509B12			9.95 wt % TiO ₂	

(a) The letter appended to the XRD File ID identifies the first sample in the replication series. The letter has been dropped in subsequent text to indicate that the referenced file is the summation of data from replicate runs.

Sample analysis was carried out by the Pacific Northwest National Laboratory Advanced Processing and Application Group. The XRD instrument used was the Scintag PAD V X-ray Diffractometer, Property Number WD33356, located in lab 102 in the APEL building. The data range for the sample was 5° to 80° 2-theta, with a step size of 0.04° 2-theta and count time 4.0 seconds per step. Copper Ka X-rays were used. The X-ray tube operating conditions were 45 kV and 40 mA.

Sample 020509B12 was prepared with SRM 674b, TiO₂ internal standard. The dried powder was placed into a tungsten carbide milling chamber and milled for 2min in the angstrom shaker mill. The TiO₂ standard was then spiked in at 9.95 wt% with the dried powder and milled again for 2min to insure a homogenous mixture of the two powders. The sample was then mounted into standard sample holder.

Phase identification was done by use of the JADE search match routines (version 6.0, Materials Data Inc.) with comparison to the ICDD (International Centre for Diffraction Data) data base PDF-2 release 1999, which includes the ICSD (Inorganic Crystal Structure Database maintained by Fachinformationszentrum (FIZ), Karlsruhe, Germany).

The phase identification plots are shown using "stick figures" to indicate the phases present; peak positions and line intensities. The phase showing the greatest peak intensity is shown at the top of the stick figures display (except the internal standard, if used, is always the topmost). Phases present at lower peak area/height are shown in decreasing order down the display.

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The identified phases, ranked by relative peak intensities, are given below for each sample. Figure 1 shows the raw summed pattern. The background fitted pattern, along with the identified phases, are shown in figures 2, 3, and 4.

A_01AIM_003_16_1607_XSP_4_B, File: 020509B12

- Rutile, TiO₂, card# 21-1276
- Boehmite, syn, AlO(OH), card# 21-1307, Excellent overall fit. Peak locations and intensities are a \bullet good match to the pattern.
- Hematite, syn, Fe₂O₃, card# 64-6101, good fit as a minor phase. \bullet
- Iron(III) oxide chloride, FeOCl, card# 61-8704, possible fit to trace phase. \bullet
- \bullet Amorphous material, ~36° 2θ.

The pattern was also examined using RIQAS (release 4.0.0.26, 6/10/2002, Materials Data Inc.) rietveld analysis software. The phases identified above were input into the analysis along with a cubic spline function for the background and an amorphous hump at \sim 36° 20, with fitted pattern shown in figure 5.

- Boehmite, AlO(OH), 48.5 wt%, with a crystallite size of 336\AA
- Hematite, Fe₂O₃, 2.9 wt%, with a crystallite size of 609 Å \bullet
- The balance of the material is amorphous \bullet

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Appendix A2

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ICDD cards of interest for this sample

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PDF#21-1276: QM=Common(+); d=Other/Unknown; I=(Unknown) Rutile, syn TiO₂ Radiation=CuKa1 Lambda=1.5406 Filter= $I/Ic(RIR)=3.4$

Calibration= d-Cutoff= Ref: Level-1 PDF

4. 19

Tetragonal, P42/mnm(136) $Z=2$ $mp=$ CELL: 4.5933 x 4.5933 x 2.9592 <90.0 x 90.0 x 90.0> $P.S=$ Density(c)=4.23 Density(m)= $Mwt = Vol=62.4$ Ref: Ibid.

Strong Line: 3.25/X 1.69/6 2.49/5 2.19/3 1.62/2 1.36/2 0.82/1 1.35/1

PDF#21-1307: QM=Common(+); d=Other/Unknown; I=(Unknown) Boehmite, syn $AIO(OH)$ Radiation=CuKa1 Lambda=1.5406 Filter= Calibration= d-Cutoff= $1/1c(RIR)$ = Ref: Level-1 PDF

Orthorhombic, Amam(63) $Z=4$ $mn =$ CELL: 3.7 x 12.227 x 2.868 <90.0 x 90.0 x 90.0> $P S =$ Density(c)=3.07 Density(m)= Mwt= $Vol=129.8$ Ref: Ibid.

Strong Line: 6.11/X 3.16/7 2.35/6 1.86/3 1.85/3 1.45/2 1.31/2 1.66/1

PDF#64-6101: QM=Calculated; d=Other/Unknown; I=(Unknown) \bullet Hematite - synthetic, Hematite group Fe2 O3 Lambda=1.5406 Filter= Radiation=CuKa1 d -Cutoff= $I/Ic(RIR)=3.04$ Calibration= Ref: Calculated from FIZ#82134 (ICSD @08/01/01) by Jade 6.x

Hexagonal, R-3c(167) Z=6 mp=
CELL: 5.037 x 5.037 x 13.771 <90.0 x 90.0 x 120.0> P.S= Density(c)=5.258 Density(m)= $Mwt = Vol=302.6$ Ref: Ibid.

Strong Line: 2.70/X 2.52/6 1.70/4 1.84/3 1.45/3 1.49/3 2.21/2 3.68/2 NOTE: Effect of mechanical activation on the real structure and reactivity of iron(III) oxide with corundum-type structure, Journal of Solid State Chemistry [JSSCB] 123 (1996) 191-202, Sadykov V A, Isupova L A, Tsybulya S V, Cherepanova S V, Litvak G S, Burgina E B, Kustova G N, Kolomiichuk V N, Ivanov V P, Paukshtis E A, Golovin A V, Avvakumov E G, X-ray diffraction (powder). $R = 0.053$

PDF#61-8704: QM=Calculated; d=Other/Unknown; I=(Unknown) Iron(III) oxide chloride Fe O CI Lambda=1.5406 Filter= $\label{eq:radiation} \begin{aligned} \text{Radiation=} & \text{CuKal} \end{aligned}$ d-Cutoff= $I/Ic(RIR)=5.34$ Calibration= Ref: Calculated from FIZ#37158 (ICSD @08/01/01) by Jade 6.x

Orthorhombic, Pmnm(59)Z=2 $mp=$

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CELL: 3.75 x 7.65 x 3.3 <90.0 x 90.0 x 90.0>
Density(c)=3.764 Density(m)= Mwt= Vol=94.7 $P.S =$ Ref: Ibid.

 $\tilde{\mathbf{r}}$.

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Strong Line: $7.65/X$ 2.50/4 3.37/4 2.02/2 2.36/2 1.50/1 1.88/1 1.37/1
NOTE: Etude de quelques derives de l'oxyde ferrique (Fe O.OH, FeO2Na, FeOCl) determination de leurs structures.,
Bulletin de la Societe Francaise de Mi No R value given in the paper.

XRD data from the samples identified in Table I have been examined for identification of crystalline phases present.

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Table I: Samples Examined in this Series

Identification	XRD File $ID(a)$	Count Time per Step (Seconds)	Number of Replications	Internal Standard
A 01AIM 13a 1880 XX XSP 4 B	020509B10			8.85 wt % TiO ₂

(a) The letter appended to the XRD File ID identifies the first sample in the replication series. The letter has been dropped in subsequent text to indicate that the referenced file is the summation of data from replicate runs.

Sample analysis was carried out by the Pacific Northwest National Laboratory Advanced Processing and Application Group. The XRD instrument used was the Scintag PAD V X-ray Diffractometer, Property Number WD33356, located in lab 102 in the APEL building. The data range for the sample was 5° to 80° 2-theta, with a step size of 0.04° 2-theta and count time 4.0 seconds per step. Copper Ka X-rays were used. The X-ray tube operating conditions were 45 kV and 40 mA.

Sample 020509B10 was prepared with SRM 674b, TiO₂ internal standard. The dried powder was placed into a tungsten carbide milling chamber and milled for 2 min in the angstrom shaker mill. The TiO₂ standard was then spiked in at 8.85 wt% with the dried powder and milled again for 2 min to insure a homogenous mixture of the two powders. The sample was then mounted into standard sample holder.

Phase identification was done by use of the JADE search match routines (version 6.0, Materials Data Inc.) with comparison to the ICDD (International Centre for Diffraction Data) data base PDF-2 release 1999, which includes the ICSD (Inorganic Crystal Structure Database maintained by Fachinformationszentrum (FIZ), Karlsruhe, Germany). Searches were restricted to the PDF and ICSD Inorganic sections.

The phase identification plots are shown using "stick figures" to indicate the phases present; peak positions and line intensities. The phase showing the greatest peak intensity is shown at the top of the stick figures display (except the internal standard, if used, is always the topmost). Phases present at lower peak area/height are shown in decreasing order down the display.

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The identified phases, ranked by relative peak intensities, are given below for each sample. Figure 1 shows the raw summed pattern. The background fitted pattern, along with the identified phases, are shown in figures 2 , 3 , and 4 .

A 01AIM 13a XX 1880 XSP 4 B, File: 020509B10

- Rutile, $TiO₂$, card# 21-1276
- · Boehmite, syn, AlO(OH), card# 21-1307, Excellent overall fit. Peak locations and intensities are a good match to the pattern.
- \bullet Hematite, syn, Fe₂O₃, card# 64-6101, good fit as a minor phase.
- Iron(III) oxide chloride, FeOCl, card# 61-8704, possible fit to trace phase. \bullet
- Amorphous material, ~36° 2θ. \bullet

The pattern was also examined using RIQAS (release 4.0.0.26, 6/10/2002, Materials Data Inc.) rietveld analysis software. The phases identified above were input into the analysis along with a cubic spline function for the background and an amorphous hump at \sim 36° 20, with fitted pattern shown in figure 5.

- Boehmite, AlO(OH), 47.4 wt%, with a crystallite size of 336 Å \bullet
- Hematite, Fe₂O₃, 3.9 wt%, with a crystallite size of 609 Å
- The balance of the material is amorphous

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Appendix A3

 $\label{eq:2} \mathcal{L} = \mathcal{L} \mathcal{L}^{\text{max}}$

ICDD cards of interest for this sample

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PDF#21-1276: QM=Common(+); d=Other/Unknown; I=(Unknown) Rutile, syn TiO₂ Radiation=CuKa1 Lambda=1.5406 Filter= Calibration= d-Cutoff= $I/Ic(RIR)=3.4$ Ref: Level-1 PDF

Tetragonal, P42/mnm(136) $Z=2$ mp= CELL: 4.5933 x 4.5933 x 2.9592 <90.0 x 90.0 x 90.0> $P.S =$ Density(c)=4.23 Density(m)= $Mwt = Vol=62.4$ Ref: Ibid.

Strong Line: 3.25/X 1.69/6 2.49/5 2.19/3 1.62/2 1.36/2 0.82/1 1.35/1

PDF#21-1307: QM=Common(+); d=Other/Unknown; I=(Unknown) Boehmite, syn AlO(OH) Radiation=CuKa1 Lambda=1.5406 Filter= Calibration= d-Cutoff= $I/Ic(RIR)$ = Ref: Level-1 PDF

Orthorhombic, Amam(63) $Z=4$ $mp=$ CELL: 3.7 x 12.227 x 2.868 <90.0 x 90.0 x 90.0 > P.S= Density(c)=3.07 Density(m)= Mwt= $Vol=129.8$ Ref: Ibid.

Strong Line: 6.11/X 3.16/7 2.35/6 1.86/3 1.85/3 1.45/2 1.31/2 1.66/1

PDF#64-6101: QM=Calculated; d=Other/Unknown; I=(Unknown) ۰ Hematite - synthetic, Hematite group Fe2 O3 Radiation=CuKa1 Lambda=1.5406 Filter= Calibration= d-Cutoff= $I/Ic(RIR)=3.04$ Ref: Calculated from FIZ#82134 (ICSD @08/01/01) by Jade 6.x

Hexagonal, $R-3c(167)$ $Z=6$ $mp=$ CELL: 5.037 x 5.037 x 13.771 <90.0 x 90.0 x 120.0 > P.S= Density(c)=5.258 Density(m)= Mwt = Vol=302.6 Ref: Ibid.

Strong Line: 2.70/X 2.52/6 1.70/4 1.84/3 1.45/3 1.49/3 2.21/2 3.68/2 NOTE: Effect of mechanical activation on the real structure and reactivity of iron(III) oxide with corundum-type structure, Journal of Solid State Chemistry [JSSCB] 123 (1996) 191-202, Sadykov V A, Isupova L A, Tsybulya S V, Cherepanova S V, Litvak G S, Burgina E B, Kustova G N, Kolomiichuk V N, Ivanov V P, Paukshtis E A, Golovin A V, Avvakumov E G, X-ray diffraction (powder). $R = 0.053$

PDF#61-8704: QM=Calculated; d=Other/Unknown; I=(Unknown) \bullet Iron(III) oxide chloride Fe O Cl Lambda=1.5406 Filter= Radiation=CuKa1 d -Cutoff= Calibration= $I/Ic(RIR)=5.34$ Ref: Calculated from FIZ#37158 (ICSD $@08/01/01$) by Jade 6.x

Orthorhombic, Pmnm(59)Z=2 $mp=$

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CELL: 3.75 x 7.65 x 3.3 <90.0 x 90.0 x 90.0>
Density(c)=3.764 Density(m)= Mwt= Vol=94.7 $P.S =$ Ref: Ibid.

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Strong Line: 7.65/X 2.50/4 3.37/4 2.02/2 2.36/2 1.50/1 1.88/1 1.37/1
NOTE: Etude de quelques derives de l'oxyde ferrique (Fe O.OH, FeO2Na, FeOCl) determination de leurs structures.,
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XRD data from the samples identified in Table I have been examined for identification of crystalline phases present.

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Table I: Samples Examined in this Series

Identification	XRD File $ID(a)$	Count Time per Step (Seconds)	Number of Replications	Internal Standard	
A 01AIM 13a 16 1871 XSP 4	020909B			10.02 wt% TiO ₂	

(a) The letter appended to the XRD File ID identifies the first sample in the replication series. The letter has been dropped in subsequent text to indicate that the referenced file is the summation of data from replicate runs.

Sample analysis was carried out by the Pacific Northwest National Laboratory Advanced Processing and Application Group. The XRD instrument used was the Scintag PAD V X-ray Diffractometer, Property Number WD33356, located in lab 102 in the APEL building. The data range for the sample was 5° to 80° 2-theta, with a step size of 0.04° 2-theta and count time 4.0 seconds per step. Copper Ka X-rays were used. The X-ray tube operating conditions were 45 kV and 40 mA.

Sample 020909B was prepared with SRM 674b, TiO₂ internal standard. The dried powder was placed into a tungsten carbide milling chamber and milled for 2 min in the angstrom shaker mill. The $TiO₂$ standard was then spiked in at 10.02 wt% with the dried powder and milled again for 2 min to insure a homogenous mixture of the two powders. The sample was then mounted into standard sample holder.

Phase identification was done by use of the JADE search match routines (version 6.0, Materials Data Inc.) with comparison to the ICDD (International Centre for Diffraction Data) data base PDF-2 release 1999, which includes the ICSD (Inorganic Crystal Structure Database maintained by Fachinformationszentrum (FIZ), Karlsruhe, Germany). Searches were restricted to the PDF and ICSD Inorganic sections.

The phase identification plots are shown using "stick figures" to indicate the phases present; peak positions and line intensities. The phase showing the greatest peak intensity is shown at the top of the stick figures display (except the internal standard, if used, is always the topmost). Phases present at lower peak area/height are shown in decreasing order down the display.

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The identified phases, ranked by relative peak intensities, are given below for each sample. Figure 1 shows the raw summed pattern. The background fitted pattern, along with the identified phases, are shown in figures 2 , 3 , and 4 .

A 01AIM 13a 16 1871 XSP 4 B, File: 020909B

- Rutile, $TiO₂$, card# 21-1276
- · Boehmite, syn, AlO(OH), card# 21-1307, Excellent overall fit. Peak locations and intensities are a good match to the pattern.
- \bullet Hematite, syn, Fe₂O₃, card# 64-6101, good fit as a minor phase.
- Iron(III) oxide chloride, FeOCl, card# 61-8704, possible fit to trace phase. \bullet
- Amorphous material, ~36° 20. \bullet

The pattern was also examined using RIQAS (release 4.0.0.26, 6/10/2002, Materials Data Inc.) rietveld analysis software. The phases identified above were input into the analysis along with a cubic spline function for the background and an amorphous hump at \sim 36° 20, with fitted pattern shown in figure 5.

- Boehmite, AlO(OH), 58.1 wt%, with a crystallite size of 380 Å \bullet
- Hematite, Fe₂O₃, 3.7 wt%, with a crystallite size of 525 Å ٠
- The balance of the material is amorphous \bullet

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PDF#21-1276: QM=Common(+); d=Other/Unknown; I=(Unknown) Rutile, syn TiO₂ Radiation=CuKa1 Lambda=1.5406 Filter= Calibration= d-Cutoff= $I/Ic(RIR)=3.4$ Ref: Level-1 PDF

Tetragonal, P42/mnm(136) $Z=2$ $mp=$ CELL: 4.5933 x 4.5933 x 2.9592 <90.0 x 90.0 x 90.0 > $P.S=$ Density(c)=4.23 Density(m)= $Mwt = Vol=62.4$ Ref: Ibid.

Strong Line: 3.25/X 1.69/6 2.49/5 2.19/3 1.62/2 1.36/2 0.82/1 1.35/1

PDF#21-1307: QM=Common(+); d=Other/Unknown; I=(Unknown) Boehmite, syn $AIO(OH)$ Radiation=CuKa1 Lambda=1.5406 Filter= d-Cutoff= Calibration= $I/Ic(RIR)$ = Ref: Level-1 PDF

Orthorhombic, Amam(63) $Z=4$ $mp=$ CELL: 3.7 x 12.227 x 2.868 <90.0 x 90.0 x 90.0> $P.S=$ Density(c)=3.07 Density(m)= $Mwt = Vol=129.8$ Ref: Ibid.

Strong Line: 6.11/X 3.16/7 2.35/6 1.86/3 1.85/3 1.45/2 1.31/2 1.66/1

PDF#64-6101: QM=Calculated; d=Other/Unknown; I=(Unknown) Hematite - synthetic, Hematite group Fe2 O3 Radiation=CuKa1 Lambda=1.5406 Filter= d -Cutoff= $I/Ic(RIR)=3.04$ Calibration= Ref: Calculated from FIZ#82134 (ICSD @08/01/01) by Jade 6.x

Hexagonal, $R-3c(167)$ $Z=6$ $mp=$ CELL: 5.037 x 5.037 x 13.771 <90.0 x 90.0 x 120.0 > P.S= Density(c)=5.258 Density(m)= Mwt= $Vol=302.6$ Ref: Ibid.

Strong Line: 2.70/X 2.52/6 1.70/4 1.84/3 1.45/3 1.49/3 2.21/2 3.68/2 NOTE: Effect of mechanical activation on the real structure and reactivity of iron(III) oxide with corundum-type Structure, Journal of Solid State Chemistry [JSSCB] 123 (1996) 191-202, Sadykov V A, Isupova L A, Tsybulya S V,
Cherepanova S V, Litvak G S, Burgina E B, Kustova G N, Kolomiichuk V N, Ivanov V P, Paukshtis E A, Golovin A V Avvakumov E G, X-ray diffraction (powder). $R = 0.053$

PDF#61-8704: QM=Calculated; d=Other/Unknown; I=(Unknown) Iron(III) oxide chloride Fe O Cl Radiation=CuKa1 Lambda=1.5406 Filter= Calibration= d -Cutoff= $I/Ic(RIR)=5.34$ Ref: Calculated from FIZ#37158 (ICSD @08/01/01) by Jade 6.x

Orthorhombic, Pmnm(59)Z=2 $mp=$

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CELL: 3.75 x 7.65 x 3.3 <90.0 x 90.0 x 90.0> $\mathbf{P}.\mathbf{S}$ Density(c)=3.764 Density(m)= Mwt= Vol=94.7
Ref: Ibid.

 $\label{eq:1} \mathbf{u} = \left[\begin{array}{cc} \mathbf{u} & \mathbf{v} \\ \mathbf{v} & \mathbf{v} \end{array} \right] \mathbf{v} \quad ,$

 \mathcal{N}

Strong Line: $7.65/X$ 2.50/4 3.37/4 2.02/2 2.36/2 1.50/1 1.88/1 1.37/1
NOTE: Etude de quelques derives de l'oxyde ferrique (Fe O.OH, FeO2Na, FeOCl) determination de leurs structures.,
Bulletin de la Societe Francaise de Mi No R value given in the paper.

February 16, 2009 Date:

Crum

Johnson

To: O. Bredt

Reviewe

From J. V. Crum

 $\overline{\mathbf{R}}$

XRD Examination of: A 01AIM 13c 16 2124 Subject: Analyst: Date

Date

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XRD data from the samples identified in Table I have been examined for identification of crystalline phases present.

Table I: Samples Examined in this Series

XRD File $ID(a)$		Count Time per	Number of	Internal Standard
Identification		Step (Seconds)	Replications	
A 01AIM 13c 16 2124 XSP 4	021309B			8.92 wt % TiO ₂

(a) The letter appended to the XRD File ID identifies the first sample in the replication series. The letter has been dropped in subsequent text to indicate that the referenced file is the summation of data from replicate runs.

Sample analysis was carried out by the Pacific Northwest National Laboratory Advanced Processing and Application Group. The XRD instrument used was the Scintag PAD V X-ray Diffractometer, Property Number WD33356, located in lab 102 in the APEL building. The data range for the sample was 5° to 80° 2-theta, with a step size of 0.04° 2-theta and count time 4.0 seconds per step. Copper Ka X-rays were used. The X-ray tube operating conditions were 45 kV and 40 mA.

Sample 021309B was prepared with SRM 674b, TiO₂ internal standard. The dried powder was placed into a tungsten carbide milling chamber and milled for 2 min in the angstrom shaker mill. The $TiO₂$ standard was then spiked in at 8.92 wt% with the dried powder and milled again for 2 min to insure a homogenous mixture of the two powders. The sample was then mounted into standard sample holder.

Phase identification was done by use of the JADE search match routines (version 6.0, Materials Data Inc.) with comparison to the ICDD (International Centre for Diffraction Data) data base PDF-2 release 1999, which includes the ICSD (Inorganic Crystal Structure Database maintained by Fachinformationszentrum (FIZ), Karlsruhe, Germany). Searches were restricted to the PDF and ICSD Inorganic sections.

The phase identification plots are shown using "stick figures" to indicate the phases present; peak positions and line intensities. The phase showing the greatest peak intensity is shown at the top of the stick figures display (except the internal standard, if used, is always the topmost). Phases present at lower peak area/height are shown in decreasing order down the display.

The identified phases, ranked by relative peak intensities, are given below for each sample. Figure 1 shows the raw summed pattern. The background fitted pattern, along with the identified phases, are shown in figures 2 , 3 , and 4 .

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A 01AIM 13c 16 2124 XSP 4 B, File: 021309B

- Rutile, $TiO₂$, card# 21-1276
- · Boehmite, syn, AlO(OH), card# 21-1307, Excellent overall fit. Peak locations and intensities are a good match to the pattern.
- Hematite, syn, $Fe₂O₃$, card# 64-6101, good fit as a minor phase.
- Amorphous material, \sim 36° 20.

The pattern was also examined using RIQAS (release 4.0.0.26, 6/10/2002, Materials Data Inc.) rietveld analysis software. The phases identified above were input into the analysis along with a cubic spline function for the background and an amorphous hump at \sim 36° 20, with fitted pattern shown in figure 5.

- Boehmite, AlO(OH), 49.3 wt%, with a crystallite size of 335 Å \bullet
- Hematite, Fe₂O₃, 1.6 wt%, with a crystallite size of 555 Å \bullet
- The balance of the material is amorphous \bullet

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Appendix A5

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PDF#21-1276: QM=Common(+); d=Other/Unknown; I=(Unknown) Rutile, syn TiO₂ Lambda=1.5406 Filter= $\label{eq:radiation} \begin{aligned} \text{Radiation=} & \text{CuKa1} \end{aligned}$ Calibration= d-Cutoff= $I/Ic(RIR)=3.4$ Ref: Level-1 PDF

Tetragonal, P42/mnm(136) $Z=2$ $mp=$ CELL: 4.5933 x 4.5933 x 2.9592 <90.0 x 90.0 x 90.0> $P.S =$ Density(c)=4.23 Density(m)= $Mwt = Vol=62.4$ Ref: Ibid.

Strong Line: 3.25/X 1.69/6 2.49/5 2.19/3 1.62/2 1.36/2 0.82/1 1.35/1

PDF#21-1307: QM=Common(+); d=Other/Unknown; I=(Unknown) Boehmite, syn $AIO(OH)$ Radiation=CuKa1 Lambda=1.5406 Filter= Calibration= d-Cutoff= $I/Ic(RIR)$ = Ref: Level-1 PDF

 $Z=4$ Orthorhombic, Amam(63) $mp=$ CELL: $3.7 \times 12.227 \times 2.868$ <90.0 x 90.0 x 90.0 > $P.S=$ Density(c)=3.07 Density(m)= $Mwt = Vol=129.8$ Ref: Ibid.

Strong Line: 6.11/X 3.16/7 2.35/6 1.86/3 1.85/3 1.45/2 1.31/2 1.66/1

PDF#64-6101: QM=Calculated; d=Other/Unknown; I=(Unknown) \bullet Hematite - synthetic, Hematite group Fe2 O3 Radiation=CuKa1 La:
Calibration= d-Cutoff= Lambda=1.5406 Filter=
 $\text{H} = \text{I/IC(RIR)} = 3.04$ Ref: Calculated from FIZ#82134 (ICSD @08/01/01) by Jade 6.x

Hexagonal, $R-3c(167)$ Z=6 $mp=$ CELL: 5.037 x 5.037 x 13.771 <90.0 x 90.0 x 120.0 > P.S= Density(c)=5.258 Density(m)= $Mwt = Vol=302.6$ Ref: Ibid.

Strong Line: 2.70/X 2.52/6 1.70/4 1.84/3 1.45/3 1.49/3 2.21/2 3.68/2 NOTE: Effect of mechanical activation on the real structure and reactivity of iron(III) oxide with corundum-type structure, Journal of Solid State Chemistry [JSSCB] 123 (1996) 191-202, Sadykov V A, Isupova L A, Tsybulya S V, Cherepanova S V, Litvak G S, Burgina E B, Kustova G N, Kolomiichuk V N, Ivanov V P, Paukshtis E A, Golovin A V, Avvakumov E G, X-ray diffraction (powder). $R = 0.053$

February 17, 2009 Date: O. Bredt To: From J. V. Crum XRD Examination of: A_01AIM_006_XX_1631 Subject:

 C_{rum}

 $B.R$

Analyst:

Reviewer:

Date: ung Date: Johnson

XRD data from the samples identified in Table I have been examined for identification of crystalline phases present.

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Table I: Samples Examined in this Series

Identification	XRD File $ID(a)$	Count Time per Step (Seconds)	Number of Replications	Internal Standard
A 01AIM 006 XX 1631 XSP 4	021609A6			9.17 wt % TiO ₂

(a) The letter appended to the XRD File ID identifies the first sample in the replication series. The letter has been dropped in subsequent text to indicate that the referenced file is the summation of data from replicate runs.

Sample analysis was carried out by the Pacific Northwest National Laboratory Advanced Processing and Application Group. The XRD instrument used was the Scintag PAD V X-ray Diffractometer, Property Number WD33356, located in lab 102 in the APEL building. The data range for the sample was 5° to 80° 2-theta, with a step size of 0.04° 2-theta and count time 4.0 seconds per step. Copper Ka X-rays were used. The X-ray tube operating conditions were 45 kV and 40 mA.

Sample 021609A6 was prepared with SRM 674b, TiO₂ internal standard. The dried powder was placed into a tungsten carbide milling chamber and milled for 2 min in the angstrom shaker mill. The TiO₂ standard was then spiked in at 9.17 wt% with the dried powder and milled again for 2 min to insure a homogenous mixture of the two powders. The sample was then mounted into standard sample holder.

Phase identification was done by use of the JADE search match routines (version 6.0, Materials Data Inc.) with comparison to the ICDD (International Centre for Diffraction Data) data base PDF-2 release 1999, which includes the ICSD (Inorganic Crystal Structure Database maintained by Fachinformationszentrum (FIZ), Karlsruhe, Germany). Searches were restricted to the PDF and ICSD Inorganic sections.

The phase identification plots are shown using "stick figures" to indicate the phases present; peak positions and line intensities. The phase showing the greatest peak intensity is shown at the top of the stick figures display (except the internal standard, if used, is always the topmost). Phases present at lower peak area/height are shown in decreasing order down the display.

The identified phases, ranked by relative peak intensities, are given below for each sample. Figure 1 shows the raw summed pattern. The background fitted pattern, along with the identified phases, are shown in figures 2, 3, and 4.

A 01 AIM 006 XX 1631 XSP 4 B File: 021609A6

- Rutile, TiO₂, card# 21-1276
- Boehmite, syn, AlO(OH), card# 21-1307, Excellent overall fit. Peak locations and intensities are a good match to the pattern.
- Hematite, syn, $Fe₂O₃$, card# 64-6101, good fit as a minor phase.
- Amorphous material, $\sim 36^{\circ} 2\theta$.

The pattern was also examined using RIQAS (release 4.0.0.26, 6/10/2002, Materials Data Inc.) rietveld analysis software. The phases identified above were input into the analysis along with a cubic spline function for the background and an amorphous hump at $\sim 36^{\circ}$ 20, with fitted pattern shown in figure 5.

The results of the analysis for the crystalline phases are:

- Boehmite, AlO(OH), 78.2 wt%, with a crystallite size of 390 Å
- Hematite, Fe₂O₃, 1.2 wt%, with a crystallite size of 699 Å \bullet
- The balance of the material is amorphous \bullet

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Appendix A6

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PDF#21-1276: QM=Common(+); d=Other/Unknown; I=(Unknown) Rutile, syn TiO₂ Radiation=CuKa1 Lambda=1.5406 Filter= Calibration= d-Cutoff= $I/Ic(RIR)=3.4$ Ref: Level-1 PDF

Tetragonal, P42/mnm(136) $Z=2$ mp= CELL: 4.5933 x 4.5933 x 2.9592 <90.0 x 90.0 x 90.0> $P.S =$ Density(c)=4.23 Density(m)= $Mwt = Vol=62.4$ Ref: Ibid.

Strong Line: 3.25/X 1.69/6 2.49/5 2.19/3 1.62/2 1.36/2 0.82/1 1.35/1

PDF#21-1307: QM=Common(+); d=Other/Unknown; I=(Unknown) Boehmite, syn AlO(OH) Radiation=CuKa1 Lambda=1.5406 Filter= Calibration= d-Cutoff= $I/Ic(RIR)$ = Ref: Level-1 PDF

 \bullet

Orthorhombic, Amam(63) $Z=4$ mp= CELL: 3.7 x 12.227 x 2.868 <90.0 x 90.0 x 90.0 > P.S= Density(c)=3.07 Density(m)= $Mwt = Vol=129.8$ Ref: Ibid.

Strong Line: 6.11/X 3.16/7 2.35/6 1.86/3 1.85/3 1.45/2 1.31/2 1.66/1

PDF#64-6101: QM=Calculated; d=Other/Unknown; I=(Unknown) Hematite - synthetic, Hematite group Fe2 O3 Radiation=CuKa1 Lambda=1.5406 Filter= Calibration= d-Cutoff= $I/Ic(RIR)=3.04$ Ref: Calculated from FIZ#82134 (ICSD @08/01/01) by Jade 6.x

Hexagonal, $R-3c(167)$ $Z=6$ $mp=$ CELL: 5.037 x 5.037 x 13.771 <90.0 x 90.0 x 120.0 > P.S= Density(c)=5.258 Density(m)= $Mwt = Vol=302.6$ Ref: Ibid.

Strong Line: 2.70/X 2.52/6 1.70/4 1.84/3 1.45/3 1.49/3 2.21/2 3.68/2 NOTE: Effect of mechanical activation on the real structure and reactivity of iron(III) oxide with corundum-type structure, Journal of Solid State Chemistry [JSSCB] 123 (1996) 191-202, Sadykov V A, Isupova L A, Tsybulya S V, Cherepanova S V, Litvak G S, Burgina E B, Kustova G N, Kolomiichuk V N, Ivanov V P, Paukshtis E A, Golovin A V, Avvakumov E G, X-ray diffraction (powder). $R = 0.053$

XRD data from the samples identified in Table I have been examined for identification of crystalline phases present.

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Table I: Samples Examined in this Series

Identification	XRD File ID	Count Time per Step (Seconds)	Number of Replications	Internal Standard
A 01AIM 13c XX 2134 XSP 4	021609A7			8.94 wt % TiO ₂

Sample analysis was carried out by the Pacific Northwest National Laboratory Advanced Processing and Application Group. The XRD instrument used was the Scintag PAD V X-ray Diffractometer, Property Number WD33356, located in lab 102 in the APEL building. The data range for the sample was 5° to 80° 2-theta, with a step size of 0.04° 2-theta and count time 4.0 seconds per step. Copper K α X-rays were used. The X-ray tube operating conditions were 45 kV and 40 mA.

Sample 021609A7 was prepared with SRM 674b, TiO₂ internal standard. The dried powder was placed into a tungsten carbide milling chamber and milled for 2 min in the angstrom shaker mill. The TiO₂ standard was then spiked in at 8.94 wt% with the dried powder and milled again for 2 min to insure a homogenous mixture of the two powders. Note: During transfer of the sample + TiO2 standard to the milling chamber, static charge caused a tiny amount of the sample and standard to leave the wax paper, used for weighing and transferring. This caused the weights to be suspect to a minor extent. Estimates of the material lost are 0.01g of the total that is equal to 0.7469g. This does not impact the ability to identify phases in the sample only the quantification of the phases to a minor extent. The sample was then mounted into standard sample holder.

Phase identification was done by use of the JADE search match routines (version 6.0, Materials Data Inc.) with comparison to the ICDD (International Centre for Diffraction Data) data base PDF-2 release 1999, which includes the ICSD (Inorganic Crystal Structure Database maintained by Fachinformationszentrum (FIZ), Karlsruhe, Germany). Searches were restricted to the PDF and ICSD Inorganic sections.

The phase identification plots are shown using "stick figures" to indicate the phases present; peak positions and line intensities. The phase showing the greatest peak intensity is shown at the top of the stick figures display (except the internal standard, if used, is always the topmost). Phases present at lower peak area/height are shown in decreasing order down the display.

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The identified phases, ranked by relative peak intensities, are given below for each sample. Figure 1 shows the raw summed pattern. The background fitted pattern, along with the identified phases, are shown in figures 2, 3, and 4.

A 01AIM 13c XX 2134 XSP 4 B File: 021609A7

• Rutile, TiO₂, card# 21-1276

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- · Boehmite, syn, AlO(OH), card# 21-1307, Excellent overall fit. Peak locations and intensities are a good match to the pattern.
- Hematite, syn, $Fe₂O₃$, card# 64-6101, good fit as a minor phase.
- Amorphous material, $\sim 36^{\circ} 2\theta$.

The pattern was also examined using RIQAS (release 4.0.0.26, 6/10/2002, Materials Data Inc.) rietveld analysis software. The phases identified above were input into the analysis along with a cubic spline function for the background and an amorphous hump at $\sim 36^{\circ}$ 20, with fitted pattern shown in figure 5.

The results of the analysis for the crystalline phases are:

- Boehmite, AlO(OH), 51.2 wt%, with a crystallite size of 336 Å
- Hematite, Fe₂O₃, 2.2 wt%, with a crystallite size of 751 Å \bullet
- The balance of the material is amorphous \bullet

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PDF#21-1276: QM=Common(+); d=Other/Unknown; I=(Unknown) Rutile, syn TiO₂ Lambda=1.5406 Filter= Radiation=CuKa1 Calibration= d-Cutoff= $I/Ic(RIR)=3.4$ Ref: Level-1 PDF

Tetragonal, P42/mnm(136) $Z=2$ mp= CELL: 4.5933 x 4.5933 x 2.9592 <90.0 x 90.0 x 90.0> $P.S=$ Density(c)=4.23 Density(m)= Mwt= $Vol=62.4$ Ref: Ibid.

Strong Line: 3.25/X 1.69/6 2.49/5 2.19/3 1.62/2 1.36/2 0.82/1 1.35/1

PDF#21-1307: QM=Common(+); d=Other/Unknown; I=(Unknown) Boehmite, syn AlO(OH) Radiation=CuKa1 Lambda=1.5406 Filter= Calibration= d-Cutoff= $I/Ic(RIR)$ = Ref: Level-1 PDF

Orthorhombic, Amam(63) $Z=4$ mp= CELL: 3.7 x 12.227 x 2.868 <90.0 x 90.0 x 90.0 > P.S= Density(c)=3.07 Density(m)= Mwt= $Vol=129.8$ Ref: Ibid.

Strong Line: 6.11/X 3.16/7 2.35/6 1.86/3 1.85/3 1.45/2 1.31/2 1.66/1

PDF#64-6101: QM=Calculated; d=Other/Unknown; I=(Unknown) Hematite - synthetic, Hematite group Fe2 O3 Radiation=CuKa1 Lambda=1.5406 Filter= Calibration= d-Cutoff= $I/Ic(RIR)=3.04$ Ref: Calculated from FIZ#82134 (ICSD @08/01/01) by Jade 6.x

Hexagonal, $R-3c(167)$ Z=6 $mp=$ CELL: 5.037 x 5.037 x 13.771 <90.0 x 90.0 x 120.0 > P.S= Density(c)=5.258 Density(m)= $Mwt = Vol=302.6$ Ref: Ibid.

Strong Line: 2.70/X 2.52/6 1.70/4 1.84/3 1.45/3 1.49/3 2.21/2 3.68/2 NOTE: Effect of mechanical activation on the real structure and reactivity of iron(III) oxide with corundum-type structure, Journal of Solid State Chemistry [JSSCB] 123 (1996) 191-202, Sadykov V A, Isupova L A, Tsybulya S V, Cherepanova S V, Litvak G S, Burgina E B, Kustova G N, Kolomiichuk V N, Ivanov V P, Paukshtis E A, Golovin A V, Avvakumov E G, X-ray diffraction (powder). $R = 0.053$

Date: February 06, 2009 To: O. Bredt J. V. Crum From Subject: XRD Examination of: B FRPGM 001 XX 2713 XSP 4 B Analyst: Date: C_{num} Reviewer: Date: Johnsor

XRD data from the samples identified in Table I have been examined for identification of crystalline phases present.

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Table I: Samples Examined in this Series

Identification	XRD File $ID(a)$	Count Time per Step (Seconds)	Number of Replications	Internal Standard
B FRPGM 001 XX 2713 XSP 4 B	031309A11			4.93 wt $\%$ TiO ₂

(a) The letter appended to the XRD File ID identifies the first sample in the replication series. The letter has been dropped in subsequent text to indicate that the referenced file is the summation of data from replicate runs.

Sample analysis was carried out by the Pacific Northwest National Laboratory Advanced Processing and Application Group. The XRD instrument used was the Scintag PAD V X-ray Diffractometer, Property Number WD33356, located in lab 102 in the APEL building. The data range for the sample was 5° to 80° 2-theta, with a step size of 0.04° 2-theta and count time 4.0 seconds per step. Copper Ka X-rays were used. The X-ray tube operating conditions were 45 kV and 40 mA.

Sample 031309A11 was prepared with SRM 674b, TiO₂ internal standard. The dried powder was placed into a tungsten carbide milling chamber and milled for 2 min in the angstrom shaker mill. The TiO₂ standard was then spiked in at 4.93 wt% with the dried powder and milled again for 2 min to insure a homogenous mixture of the two powders. The sample was then mounted into standard sample holder.

Phase identification was done by use of the JADE search match routines (version 6.0, Materials Data Inc.) with comparison to the ICDD (International Centre for Diffraction Data) data base PDF-2 release 1999, which includes the ICSD (Inorganic Crystal Structure Database maintained by Fachinformationszentrum (FIZ), Karlsruhe, Germany). Searches were restricted to the PDF and ICSD Inorganic sections.

The phase identification plots are shown using "stick figures" to indicate the phases present; peak positions and line intensities. The phase showing the greatest peak intensity is shown at the top of the stick figures display (except the internal standard, if used, is always the topmost). Phases present at lower peak area/height are shown in decreasing order down the display.

The identified phases, ranked by relative peak intensities, are given below for each sample. Figure 1 shows the raw summed pattern. The background fitted pattern, along with the identified phases, are shown in figures 2, 3, and 4.

B FRPGM 001 XX 2713 XSP 4 B, File: 031309A11

- Rutile, $TiO₂$, card# 21-1276
- Boehmite, syn, AlO(OH), card# 21-1307, Excellent overall fit. Peak locations and intensities are a \bullet good match to the pattern.
- Gibbsite, Al(OH)₃, card# 60-2702, Good fit. \bullet
- Amorphous material, ~36° 20. \bullet

The pattern was also examined using RIOAS (release 4.0.0.26, 6/10/2002, Materials Data Inc.) rietveld analysis software. The phases identified above were input into the analysis along with a cubic spline function for the background and an amorphous hump at $\sim 36^{\circ}$ 20, with fitted pattern shown in figure 5.

The results of the analysis for the crystalline phases are:

- Boehmite, AlO(OH), 51.6 wt%, with a crystallite size of 414 Å
- Gibbsite, Al(OH)₃, 32.1 wt% with a crystallite size of 160 Å \bullet
- The balance of the material is amorphous $\ddot{\bullet}$

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PDF#21-1276: QM=Common(+); d=Other/Unknown; I=(Unknown) Rutile, syn $TiO₂$ Lambda=1.5406 Filter= Radiation=CuKa1 Calibration= d-Cutoff= $I/Ic(RIR)=3.4$ Ref: Level-1 PDF

Tetragonal, P42/mnm(136) $Z=2$ $mp=$ CELL: 4.5933 x 4.5933 x 2.9592 <90.0 x 90.0 x 90.0 > $P.S=$ Density(c)=4.23 Density(m)= Mwt= Vol=62.4 Ref: Ibid.

Strong Line: 3.25/X 1.69/6 2.49/5 2.19/3 1.62/2 1.36/2 0.82/1 1.35/1

PDF#21-1307: QM=Common(+); d=Other/Unknown; I=(Unknown) Boehmite, syn AlO(OH) Radiation=CuKa1 Lambda=1.5406 Filter= Calibration= d-Cutoff=
Ref: Level-1 PDF $Ulc(RIR)$ =

Orthorhombic, Amam(63) $Z=4$ mp= CELL: 3.7 x 12.227 x 2.868 <90.0 x 90.0 x 90.0> $P.S=$ $Mwt = Vol=129.8$ Density(c)=3.07 Density(m)= Ref: Ibid.

Strong Line: 6.11/X 3.16/7 2.35/6 1.86/3 1.85/3 1.45/2 1.31/2 1.66/1

PDF#60-2702: QM=Calculated; d=Other/Unknown; I=(Unknown) Gibbsite $AI(OH)₃$ Radiation=CuKa1 Lambda=1.5406 Filter= Calibration= d-Cutoff= I/Ic(RIR)=1.67 Ref: Calculated from FIZ#6162 (ICSD @08/01/01) by Jade 6.x

 \bullet

Monoclinic, $P21/n(14)$ Z=8 $mp=$ CELL: 8.684 x 5.078 x 9.736 <90.0 x 94.54 x 90.0> P.S= Density(c)=2.421 Density(m)= Mwt= Vol=428.0 Ref: Ibid.

Strong Line: 4.85/X 4.38/4 2.39/2 4.33/2 2.05/2 1.81/1 1.69/1 1.75/1 NOTE: Refinement of the crystal structure of gibbsite, Al (O H)₃, Zeitschrift fuer Kristallographie, Kristallgeometrie, Kristallphysik, Kristallchemie (-144,1977) [ZEKGA] 139 (1974) 129-135, Saalfeld H, Wedde M, Dx=2.4. M PDF 33-18. $R = 0.032$.

XRD data from the samples identified in Table I have been examined for identification of crystalline phases present.

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Table I: Samples Examined in this Series

Identification	XRD File $ID(a)$	Count Time per Step (Seconds)	Number of Replications	Internal Standard
A 02AML 021 XX 2460 XSP 4	031709A11			5.0 TiO ₂

(a) The letter appended to the XRD File ID identifies the first sample in the replication series. The letter has been dropped in subsequent text to indicate that the referenced file is the summation of data from replicate runs.

Sample analysis was carried out by the Pacific Northwest National Laboratory Advanced Processing and Application Group. The XRD instrument used was the Scintag PAD V X-ray Diffractometer, Property Number WD33356, located in lab 102 in the APEL building. The data range for the sample was 5° to 80° 2-theta, with a step size of 0.04° 2-theta and count time 4.0 seconds per step. Copper K α X-rays were used. The X-ray tube operating conditions were 45 kV and 40 mA.

Sample 031709A11 was prepared with SRM 674b, TiO₂ internal standard. The dried powder was placed into a tungsten carbide milling chamber and milled for 1min in the angstrom shaker mill. The TiO₂ standard was then spiked in at 5.0 wt% with the dried powder and milled again for 2 min to insure a homogenous mixture of the two powders. The sample was then mounted into an off-axis quartz sample holder (zero background holder).

Phase identification was done by use of the JADE search match routines (version 6.0, Materials Data Inc.) with comparison to the ICDD (International Centre for Diffraction Data) data base PDF-2 release 1999, which includes the ICSD (Inorganic Crystal Structure Database maintained by Fachinformationszentrum (FIZ), Karlsruhe, Germany). The chemistry provided for Group 7, in order of decreasing concentration, was Fe, Na, U, P, Ca, Al, Si, Bi, Sr and Mg. Phase identifications first done without chemistry restrictions. Searches were restricted to the PDF and ICSD Inorganic sections.

The phase identification plots are shown using "stick figures" to indicate the phases present; peak positions and line intensities. The phase showing the greatest peak intensity is shown at the top of the stick figures display (except the internal standard, if used, is always the topmost). Phases present at lower peak area/height are shown in decreasing order down the display. Sample identification is below the scan or

shown in the top most left of the plot figures. Scan running conditions are shown to the right on the identification.

The identified phases, ranked by relative peak intensities, are given below for each sample. Figure 1 shows the raw summed pattern. The background fitted pattern along with the identified phases are shown in figures 2, 3, and 4.

A 02AML 021 XX 2460 XSP 4, File: 031709A11

- Rutile, $TiO₂$, card# 21-1276
- Boehmite, syn, AlO(OH), card# 21-1307, Excellent overall fit. Peak locations and intensities are a good match to the pattern.
- Hematite, Fe₂O₃, card# 64-6101, Good fit. \bullet

The pattern was also examined using RIQAS (release 4.0.0.26, 6/10/2002, Materials Data Inc.) rietveld analysis software. The phases identified above were input into the analysis along with a polynomial background and an amorphous hump at \sim 35 $^{\circ}$ 20, with fitted pattern shown in figure 5.

The results of the analysis are:

- Boehmite, AlO(OH), 38.4 wt%, with a crystallite size of 554 Å
- Hematite, $Fe₂O₃$, 1.1 wt%, with a crystallite size of 750 Å

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Appendix A

X t_{∞}

ICDD cards of interest for this sample

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PDF#21-1276: QM=Common(+); d=Other/Unknown; I=(Unknown) Rutile, syn TiO₂ Lambda=1.5406 Filter= Radiation=CuKa1 Calibration= d-Cutoff= $I/Ic(RIR)=3.4$ Ref: Level-1 PDF

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Tetragonal, P42/mnm(136) $Z=2$ mp= CELL: 4.5933 x 4.5933 x 2.9592 <90.0 x 90.0 x 90.0> $P.S=$ Density(c)=4.23 Density(m)= Mwt= Vol=62.4 Ref: Ibid.

Strong Line: 3.25/X 1.69/6 2.49/5 2.19/3 1.62/2 1.36/2 0.82/1 1.35/1

PDF#21-1307: QM=Common(+); d=Other/Unknown; I=(Unknown) Boehmite, syn $AIO(OH)$ Lambda=1.5406 Filter= Radiation=CuKa1 Calibration= d-Cutoff= $I/Ic(RIR)$ = Ref: Level-1 PDF

Orthorhombic, Amam(63) $Z=4$ mp= CELL: 3.7 x 12.227 x 2.868 <90.0 x 90.0 x 90.0> $P.S=$ Density(c)=3.07 Density(m)= $Mwt = Vol=129.8$ Ref: Ibid.

Strong Line: 6.11/X 3.16/7 2.35/6 1.86/3 1.85/3 1.45/2 1.31/2 1.66/1

PDF#64-6101: QM=Calculated; d=Other/Unknown; I=(Unknown) Hematite - synthetic, Hematite group Fe2 O3 Radiation=CuKa1 Lambda=1.5406 Filter= Calibration= d -Cutoff= $I/Ic(RIR)=3.04$ Ref: Calculated from FIZ#82134 (ICSD @08/01/01) by Jade 6.x

Hexagonal, R-3c(167) $Z=6$ $mp=$ CELL: 5.037 x 5.037 x 13.771 <90.0 x 90.0 x 120.0 > P.S= Density(c)=5.258 Density(m)= Mwt = Vol=302.6 Ref: Ibid.

Strong Line: 2.70/X 2.52/6 1.70/4 1.84/3 1.45/3 1.49/3 2.21/2 3.68/2 NOTE: Effect of mechanical activation on the real structure and reactivity of iron(III) oxide with corundum-type structure, Journal of Solid State Chemistry [JSSCB] 123 (1996) 191-202, Sadykov V A, Isupova L A, Tsybulya S V, Cherepanova S V, Litvak G S, Burgina E B, Kustova G N, Kolomiichuk V N, Ivanov V P, Paukshtis E A, Golovin A V, Avvakumov E G, X-ray diffraction (powder).

H.3 Particle-Size Distribution

MEMORANDUM

Six samples were delivered for analysis under WP# H14568. These samples were to be analyzed per instructions provided in a 02/17/2009 Letter to Analyst (LTA). Copies of the particle distribution plots for the samples are attached in addition to a copy of the LTA. The particle size distribution plots for the performance standard calibration checks are attached as well. They were collected on $11/25/2008$ and $03/09/2009$ using the manufacturer specified material, QAS3001B, part No. CRM00016.

SAMPLE PREP

All 6 samples were analyzed as provided using a Malvern MS2000 Analyzer with Hydro 2000S particle dispersion unit. Settled samples delivered in conical bottom centrifuge tubes, were initially agitated with a Scientific Industries, Vortex-T, Genie 2 vibrator to the discretion of the analyst. After the initial agitation, the samples were shaken by hand and the slurry transferred to the Malvern MS2000 using a poly transfer pipette. The transfer pipette was also used to keep the sample material suspended in the centrifuge tubes during transfer.

The requester of service indicated concern over potential agglomeration issues and requested that each sample, in addition to the as-received form, be analyzed after sonication. A transfer pipette was used to mix the slurry and transfer the required amount to the analyzer. Nominally 1 to 5 drops of each sample/sub-sample were placed in the Hydro 2000S particle dispersion unit using 0.01 M NaOH as the dispersion fluid. Sample A 02AOL 015 2809 XSP 0 was analyzed using A PF1 permeate as the dispersion fluid. Sample A 02AML 007 XX 2694 UDS 2 utilized both 0.01 M NaOH and A PF1 permeate as dispersion fluids. The dispersion unit has an internal sonication device with a variable speed pump/agitator. Unless noted differently, the pump/agitator was operated at a setpoint of 3000 and the sonicator operated at 0% (as-received) or 50% maximum setpoint. Sonicator setpoints of up to 100% maximum were an option at the analyst's discretion.

The amount of sub-sample required for analysis varied with the actual concentration of the sample. Neither the amount of sample nor the amount of 0.01 M NaOH dispersion solution was critical to the analytical process since the analytical software indicates graphically the amount of sample needed for analysis. The actual amounts of sample and water did not need to be, nor were they measured or recorded. Any additional sonication required to de-agglomerate sample(s) was noted on the respective particle size distribution data plot.

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ANALYSIS

Analysis was performed per procedure TPR-RPP-WTP-626, Rev. 0, Particle Size Analysis using Malvern MS2000 (current Revision) and per the LTA provided by the responsible scientist. The ID of the Malvern MS2000 Analyzer is property# PT20096 (serial number MAL1019545 listed on the attached data plots). The pump/stirrer speed of the Hydro 2000S particle dispersion unit (ID property# PT20083) integral to the analyzer was set at 3000. Samples were allowed to circulate/sonicate in the Hydro 2000S for at least 30 seconds prior to analysis.

The Malvern MS2000 Analyzer does not have the capability of calibration adjustment, and other than cleaning the lens or the sample chamber, or returning the equipment to the vendor, there are no adjustments, other than pump/stirrer speed and % sonication, that will affect the operation of the equipment. Per the LTA, a QAS3001B, part No. CRM00016 standard was used to verify the analyzer performance on 11/25/2008 and 03/09/2009. Procedure TPR-RPP-WTP-626 requires that the performance analysis be repeated every 90 days. The following parameters were included in the standard analysis setup as per Malvern Instruction Sheet MRK704-0407-2007:

Setup name: Glass Beads (typical) Dispersant Name: DI Water Sensitivity: Enhanced Analysis model: Single mode spherical Particle refractive index: 1.520 Dispersant refractive index: 1.33

Successful performance of the Malvern MS2000 as defined by the LTA, requires that standard D_{50} analysis results fall within +/- 10% of the manufacture's D_{50} specifications. The glass standard microspheres were analyzed on 11/25/2008 and 03/09/2009 as spherical particles and the D_{50} values were 62.395 and 62.206 microns respectively, which fell within $+/$ -10% of the manufacture specified volume weighted D_{50} value of 60.59 microns. However, it was noted that the 03/09/2009 standard was inadvertently analyzed using incorrect settings (Senstivity = Normal, and Analysis model = General Purpose). The raw data was re-analyzed with the Mastersizer 2000 analytical software on 04/13/2009 using the correct settings. The average D₅₀ results (see LRB BNW 60175, page 51) from the repeat analysis of the raw data was 63.167 microns. This D_{50} value still fell within the +/- 10% bracket on the vendor specified D_{50} value. The re-analyzed results are included in Table 1 and a single typical data plot, record 2950, is included in the Performance Evaluation section of this report.

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Table 1. Standard Analysis Results from 11/25/2008 & 03/09/2009 MV and D_{50} results in microns

PEP specimens were sub-sampled a minimum of 2 times (noted as aliquots) to allow duplicate analysis of each sample. Per instructions on the LTA, 3 additional subsamples were to be analyzed if the initial duplicate MV (volume weighted mean diameter) values of the as-received or the sonicated sub-samples differed by more than 15%. All duplicate analysis results varying by more than 100% were reviewed by the cognizant scientist. Any additional analysis requirements could be waived if the cognizant scientist agrees that agglomeration issues may cause results to be suspect.

The following parameters were included in the sample analysis setup:

Setup name: Ferric Oxide Hydroxide Dispersant Name: 0.01 M NaOH Sensitivity: Normal Analysis model: General purpose Particle refractive index: 2.94 Dispersant refractive index: 1.33

For samples using A PF1 permeate as the dispersion fluid:

Setup name: Ferric Oxide Hydroxide Dispersant Name: A_PF1 Permeate Sensitivity: Normal Analysis model: General purpose Particle refractive index: 2.94 Dispersant refractive index: 1.33

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COMMENTS AND OBSERVATIONS

Prior to completion of the Test A PSD sample analysis, discussions were initiated by the customer with regards to the effect of performing the analysis using A_PF1 Permeate as the dispersion fluid versus using 0.01M NaOH. The analysis of slurried samples versus the analysis of centrifuged solids was also discussed. Typically samples were received after the slurry form had been rinsed in 0.01 M NaOH, centrifuged, and the supernatant liquid decanted. The undissolved solids in the bottom of the conical centrifuge tube (referred to as centrifuged solids) were re-suspended in 0.01 M NaOH and pipetted into the Malvern Hydro 2000S particle dispersion unit. Prior to these discussions, samples B FRPGM 001 XX 2713 XSP B and A T22GM 002 XX 1507 XSP 4 B had already been delivered for analysis in the centrifuged solids form. To help understand the effects of analysis with the A_PF1 Permeate and the analysis of the slurried form, multiple tests were performed on sample A_02AML_007_XX_2694_UDS_2: 1) slurried with filtered A_PF1 permeate dispersion fluid, 2) slurried with un-filtered A_PF1 permeate dispersion fluid, 3) slurried in 0.01M NaOH dispersion fluid, and 4) washed (centrifuged 4 times replacing 0.01 M NaOH wash solution for each wash), using 0.01 M NaOH to re-suspend the centrifuged solids, and using 0.01 M NaOH as the PSD dispersion fluid. The results are shown in Table 2. NOTE that only 1 analysis, at the direction of the customer, was performed on each of the 4 pre-described conditions instead of the typical duplicate analysis.

Diameter and D50 results in microns Volume weighted **Ultrasonic** mean Record **Sample Name** D_{50} level **Analysis Condition** diameter number A_02AML_007_XX_2694_UDS_2 -Un-washed, FilteredA_PF1 1177 Average 1.882 11.259 Ω A 02AML 007 XX 2694 UDS 2 15.994 $\pmb{0}$ Un-washed, A_PF1 2.26 1201 Average A 02AML_007_XX_2694_UDS_2 Un-washed, 0.01 M NaOH Average 1.298 8.57 Ω 1231 A 02AML 007 XX 2694 UDS 2 1.892 $\pmb{0}$ Washed, 0.01 M NaOH 1.25 1255 Average A 02AML 007 XX 2694 UDS 2-50 Un-washed, FilteredA_PF1 1.346 5.411 1183 Average A 02AML 007 XX 2694 UDS 2-4.968 Un-washed, A_PF1 1207 Average 1.413 50 A_02AML_007_XX_2694_UDS_2 Un-washed, 0.01 M NaOH 1.113 6.052 50 1237 Average A_02AML_007_XX_2694_UDS_2 -50 Washed, 0.01 M NaOH 1.031 1.485 1261 Average

Table 2. Dispersion Fluid Evaluation Results

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It was observed that the particle size distribution (PSD) plots for the samples dispersed in permeate showed a distinct bi-modal size distribution, whereas the PSD plots for samples dispersed in 0.01M NaOH typically showed only a single prominent peak.

As a result of the A 02AML 007 XX 2694 UDS 2 analysis, the customer decided to analyze the pre-wash slurried sample A 02AOL 015 XX 28009 XSP 0 in A PF1 permeate, and the post-wash slurried sample A 02AOL 015 XX 28011 XSP 0 in 0.01 M NaOH. All other samples were analyzed in 0.01 M NaOH.

Both slurried samples analyzed with the 0.01 M NaOH dispersion fluid produced results that were outside of the +/- 15% duplicate "acceptance" requirement and therefore 3 additional aliquots for each sample were analyzed. The slurried sample results analyzed with the A PF1 permeate dispersion fluid fell within the +/- 15% duplicate "acceptance" requirement as did the centrifuged solids samples. Results are shown in Table 3.

In addition to 0% sonication and 50% sonication, most samples were analyzed at 100% sonication to observe the effects of sonication on the breakdown of potentially agglomerated particles. With the technical input of the equipment custodian, it was decided to report only the 0% and 50% sonicated results.

Table 3. Sample Analysis Results

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Table 3. Sample Analysis Results (continued)

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Table 3. Sample Analysis Results (continued)

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Appendix I

PEP Operational Control Systems

Appendix I: Operational Control Systems for PEP

The operational control systems for the Pretreatment Engineering Platform are extensive and include instrumentation, utility interfaces, chemical mixing and addition, process operations, valve alignment, and maintenance. The system is documented in the Tessenderlo Kerley Services Mechanical Data Book(s) Volumes I through XVIII as supplied by Bechtel National Inc.^(a) Testing processes were controlled by the Test Plan,^(b) the specific Test Instruction for the test being conducted, and the operating procedures for the PEP. The summary of the main operational procedures is listed below.

Administrative Procedures

OP-601 Surveillance Report Procedure OP-602 Turnover Checklist Procedure

System Startup and Shutdown Procedures

OP-101 System Startup Procedure OP-102 System Standby Shutdown Procedure OP-103 System Cold Shutdown Procedure

Utilities Procedures

OP-201 Reverse Osmosis System Procedure OP-202 Demineralized Water Procedure SOP-203 Chilled Water System Operating Procedure (F&O) SOP-204 Compressed Air Operating Procedure (F&O) OP-205 Air Dryer Operating Procedure cancelled SOP-206 Vacuum System Operating Procedure (F&O) OP-207 Vessel Vent System Procedure SOP-208 Boiler Operating Procedure (F&O) OP-209 Data Acquisition System (DAS) Operating Procedure

Chemical Reagents Procedures

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OP-301 Simulant Addition Procedure OP-302 19M NaOH Operating Procedure OP-303 Inhibited Water Procedure OP-304 2M Caustic Operating Procedure OP-305 Acid Procedure OP-306 Sodium Permanganate (NaMnO4) Operating Procedure OP-307 Miscellaneous Chemical Addition Procedure

Processing Procedures

OP-401 Sampling Instructions Procedure OP-402 Tank Transfer Procedure OP-403 Tank Heating and Cooling Procedure OP-404 Pulse Jet (PJM) Operating Procedure OP-405 T02A Sparging Procedure OP-406 Leaching Procedure OP-407 T02A Leaching Procedure OP-408 Tank T02A Recirculation Procedure OP-409 Ultrafiltration (Dewatering) Procedure OP-410 Solids Washing Procedure OP-411 Tank Drain Operating Procedure OP-412 Waste Transfer Procedure OP-413 Flushing and Transfer of Inhibited Water Procedure OP-414 UFP-VSL T01A & B Recirculation Procedure OP-415 Stable Level Measurements Procedure

Process Maintenance Procedures

OP-501 Filter Chemical Cleaning Procedure cancelled – requires revision OP-502 Pump Seal Pot Operating Procedure OP-503 Spill Response Procedure OP-504 Inspection of Vessel Interiors Using Video Camera or Borescope

⁽a) H Hazen, Letter April 15, 2009, "*Contract No. DE-AC27-01RV14136 – Hanford Tank Waste Treatment and Immobilization Plant, Memorandum of Agreement (MOA), 24590-QL-HC9-WA49-00001, PNNL R&T Support, Approval to use Subcontractor Supplied Documents for the Pretreatment Engineering Platform(PEP)*", CCN 196243, Bechtel National Inc., Richland, Washington.

⁽b) GB Josephson, OP Bredt, JK Young, and DE Kurath. 2009. Test Plan for Pretreatment Engineering Platform (PEP) Testing (Phase I). TP-RPP-WTP-506, Rev. 0.4, Pacific Northwest National Laboratory, Richland, Washington.

System Valve Alignment Procedures

Z001 HLP-VSL-T22 System Alignment Checklist Z002 FRP-VSL-T01 System Alignment Checklist Z003 UFP-VSL-T01A System Alignment Checklist Z004 UFP-VSL-T01B System Alignment Checklist Z005 UFP-HX System Alignment Checklist Z006 UFP-VSL-T02A System Alignment Checklist Z007 Ultrafilters System Alignment Checklist Z008 UFP-VSL-T62A/B System Alignment Checklist Z009 FEP-VSL-T01 System Alignment Checklist Z010 HLP-VSL-T27 System Alignment Checklist Z011 Vessel Vent System Alignment Checklist Z012 Vacuum and Compressed Air System Alignment Checklist F&O Z013 Boiler System Alignment Checklist F&O Z014 Chemical System Alignment Checklist Z015 Chemical System Alignment Checklist Z016 UFP-VSL-T01A PJM System Alignment Checklist Z017 UFP-VSL-T01B PJM System Alignment Checklist Z018 UFP-VSL-T02A PJM System Alignment Checklist Z019 Sparger System Alignment Checklist Z020 UFP-VSL-T01A Coriolis Densitometer System Alignment Checklist Z021 UFP-VSL-T01B Coriolis Densitometer System Alignment Checklist Z022 UFP-VSL-T02A Coriolis Densitometer System Alignment Checklist Z023 Pump Seal Pot System Alignment Checklist Z024 Pump Seal Pot System Alignment Checklist Z025 Chilled Water System Alignment Checklist F&O Z026 Reverse Osmosis Unit System Alignment Checklist **Appendix J**

Operational Process Sheets, Integrated Test A Parameters

Appendix J: Operational Process Sheet, Integrated Test A Parameters

The Integrated Test A Test Instruction, TI-WTP-PEP-065, included a Run Sheet that provided the LTE target or setpoint values for key process operations. The LTE recorded the actual values obtained for the target setpoint values during testing from available data on the Run Sheet. Table J.1 is comprised of operational process sheets summarizing the actual target values obtained during Integrated Test A. The "actual values" column of data comes from various data sources. Majority of the times, these data were taken from the HMI, which is not NQA-1 qualified and were "real-time" values, rather than an average. Consequently, Table J.1 is for information only.

The "comments" column indicates whether the value is "for information only" or NQA-1. Values are "for information only" if taken from the HMI, valve lineup verifications, variable frequency drive (VFD) and antifoam agent or inhibited water additions. Where NQA-1values resulting from previously published reports and data analysis efforts are available, these values are provided instead. In some instances, target parameters were changed by the Test Directors during the test; these changes are shown as text strike-outs. Cross-hatched patterned cells on the operational process sheets indicate the test instruction steps associated with the target parameters were not performed per Test Director guidance.

NOTE:

HMI values are for information only.

VFD—this value was taken from the VFD readout, which was not calibrated.

- The steam ring air purge and the air sparge values were read locally from the instrument (Coriolis gas flowmeter). These local readouts were calibrated and are NQA-1.
- WTP-RPT-XXX—If a report is cited as the data source, the value was evaluated and checked via a computational computer program (CCP) traceable through the report referenced.

				Integrated Test A Run sheet (TI-065 Rev. 0)			
		Operation	Target or Setpoint Value	Range $(-/+)$	Units	Actual Value	Comments
	Simulant storage in	HLP-VSL-T22 Operation					
	HLP-VSL-T22 (Test Instructions) step 7.1.1)	Minimum volume of simulant in HLP-VSL-22 to complete Test A.	2,600		gal	3040	Value given includes allowance for 500-gal heel. Value obtained from the HMI
		Agitator speed	68	$-10/+10$	rpm	60	Variable Frequency Drive, Information Only
	Transfer simulant from HLP-VSL-T22 to	Total slurry transfer volume (endpoint)	388 ^a 501	$-5/+5$	gal	491	Assumes no previous heel in UFP-VSL-T01A. WTP-RPT-186
	UFP-VSL-T01A and add 17.9 M	Slurry transfer rate	7.49.6	$-1/+1$	gpm	10.1	WTP-RPT-186
	caustic in-line (Test Instructions) steps $7.1.2.1$ to	Total 17.9 M caustic addition (in-line after HLP-PMP-T21)	639	$-10/+10$	kg	637	WTP-RPT-186
Leach Batch 1	7.1.2.7)	17.9 M caustic addition rate	12.7	$-1/+3$	kg/min	14	Caustic addition should finish 1 to 2 min before slurry transfer finishes to flush caustic from transfer line. WTP-RPT-186
		UFP-VSL-T01A Operation					
		Volume of antifoam to add to UFP-VSL-T01A	1,000	$-20/+20$	mL	1000	This value is concentrate. Dilute with 3 parts IW to 1 part concentrate. Information Only
		Level to initiate PJM operation	15	$-0/10$	in	$40+$	Value obtained from the HMI
		PJM jet velocity	4.8	$-0.3/+0.3$	m/s	5.3	NQA-1
		PJM stroke (80%)	29.0	$-2/+2$	in	29.8 (85%)	NQA-1
		PJM cycle time	35	$-1/+1$	${\bf S}$		NQA-1

Table J.1. Integrated Test A Operational Process Sheet

^a Target parameters were changed by the Test Directors during the test; these changes are shown as text strike-outs, typical for Table J.1.

				Integrated Test A Run sheet (TI-065 Rev. 0)				
		Operation	Target or Setpoint Value	Range $(-/+)$	Units	Actual Value	Comments	
		add to get prototypic condensate					collected at 1, 2, 3, 13, 14, and 15 hrs after start of leach. Information Only	
		UFP-VSL-T01A Operation						
		PJM jet velocity	4.8	$-0.3/+0.3$	m/s	5.3	$NQA-1$	
		PJM stroke (80%)	29.0	$-2/+2$	in	26.6 (76%)	$NQA-1$	
		PJM cycle time	35	$-1/+1$	S	35.3	$NQA-1$	
		Steam ring air purge flowrate (automatic)	0.13	$0.02/+0.02$	kg/min	$0.1 - 0.14$	\sim 4.0 scfm, purge will turn off when steam exceeds 6 cfm and will turn back on when steam flow drops below 4 cfm WTP-RPT-186	
		Level at end of leach	66	$-5/+5$	in	67.2	Y (init.) \sim 593 gal WTP-RPT-186	$\mathbf N$ STOP
	Cool-down of UFP-VSL-T01A by UFP-HX- T ₀₅ A	Initial cool-down temperature using external chiller UFP- HX-T05A (endpoint)	60	$-2/+2$	$\rm ^{\circ}C$	60	Value obtained from the HMI	
	(Test Instructions steps 7.1.3.20 to	Initial cool-down duration	2.75	$-0.5/+0.5$	hr	2.75	Value obtained from the HMI	
	7.1.3.26	UFP-VSL-T01A Operation						
		Temperature to increase steam ring air purge rate	90	$-5/+2$	$\rm ^{\circ}C$	~10	NQA-1	
		Steam ring air purge flowrate	0.20	$0.02/+0.02$	kg/min	0.17	6.1 scfm below 90°C in UFP-VSL-T01A Local Calibrated Instrument, NQA-1	
		Temperature to resume PJM suction refill	60	$-2/+2$	$^{\circ}\mathrm{C}$	yes	Value obtained from the HMI	
Concentrat e Leach Batch	Transfer and cool from	leached simulant Initial transfer volume	275	$-3/+3$	gal	275	Transferred while filter loop is still isolated from UFP-VSL-T02A. Value obtained from the HMI	
		UFP-VSL-T01A Initial transfer rate	6.9	NA	gpm	6.9	Value obtained from the HMI	

Table J.1. Integrated Test A Operational Process Sheet

			Integrated Test A Run sheet (TI-065 Rev. 0)				
	Operation	Target or Setpoint Value	Range $(-/+)$	Units	Actual Value	Comments	
to UFP-VSL-T02A	Make-up batch transfer volume	11	$-0.2/+0.2$	gal	11	Value obtained from the HMI	
and concentrate solids	Make-up batch flowrate	6.9	NA	gpm	6.4	Value obtained from the HMI	
(Test Instructions steps $7.1.4.1$ to $7.1.4.8$ and steps $7.1.11.1$ to	UFP-VSL-T01A level (endpoint to stop batch transfers)	10	$-2/+2$	in	$\sim\!\!10$	Y (init.) Estimate 23 make-up batches from UFP-VSL-T01A. $NQA-1$	${\bf N}$ STOP
7.1.11.8	UFP-VSL-T02A level to initiate each make- up batch	44.7	$-1/+1$	in	44.7	Value obtained from the HMI	
	UFP-VSL-T02A Operation						
	Level to initiate PJM operation	20	$-2/10$	in	~100	NQA-1	
	PJM jet velocity	7.3	$-0.4/+0.4$	m/s	7.6	$NQA-1$	
	PJM stroke (80%)	31.5	$-2/+2$	in	28.8 (76%)	NQA-1	
	PJM cycle time	33	$-1/+1$	S	33.2	$NQA-1$	
	Steam ring air purge flowrate	0.14	$0.02/+0.02$	kg/min	0.14	4.1 scfm below 90°C in UFP-VSL-T02A Local Calibrated Instrument, NQA-1	
	Total flowrate of 5 lower air spargers (idle)	0.04	0.005/ $+0.005$	kg/min	0.04	\sim 13 scfh for each sparge tube Local Calibrated Instrument, NQA-1	
	Flowrate of upper air sparger (idle) Ultrafiltration Loop	0.01	0.005/ $+0.005$	kg/min	0.01	\sim 16 scfh (record field transmitter value) Local Calibrated Instrument, NQA-1	
	Operation	\blacksquare					
	Number of filter bundles					Y (init.)	$\mathbf N$
	(UFP-FLT-T01A should be aligned)	1	$-0/0+$		1	Information Only	STOP
	Filter loop flowrate	109	$-10/+10$	gpm	109	Value obtained from the HMI	
	Trans-membrane pressure	40	$-4/+4$	psi	40	Value obtained from the HMI	

Table J.1. Integrated Test A Operational Process Sheet

			Integrated Test A Run sheet (TI-065 Rev. 0)				
	Operation	Target or Setpoint Value	Range $(-/+)$	Units	Actual Value	Comments	
(Non-prototypic using external exchanger) (Test Instructions steps $7.1.6.1$ to 7.1.6.9	Time to heat to initial heat-up temperature	$<$ 3	NA	hr	1.5	Heating loop is \sim 9 gallons and will be lost (unprototypic). Target and range is not important. Should be complete in under 3 hours. NQA-1	
Final heat-up UFP-VSL-T01B	Target final temperature (endpoint)	98	$-2/+2$	$\rm ^{\circ}C$	~108	Y (init.) $NQA-1$	$\mathbf N$ STOP
(Test Instructions	Heat-up time	3.8	$-0.5/+0.5$	hr	3.8	NQA-1	
steps $7.1.6.10$ to 7.1.6.15)	UFP-VSL-T01B Operation						
	Temperature to turn off PJM suction refill	60	$-2/+2$	$\rm ^{\circ}C$	61	Value obtained from the HMI	
	Temperature to reduce steam ring air purge rate	90	$-2/+8$	$\rm ^{\circ}C$	92	Value obtained from the HMI	
	High-temperature steam ring air purge flowrate	0.13	$0.02/+0.02$	kg/min	0.13	4.0 scfm above 90°C in UFP-VSL-T01A Local Calibrated Instrument	
	Level at end of heat-up	62	$-2/+2$	in	64.2	Y (init.) Anticipate accumulation of ~62 gal of condensate during heat-up. NQA-1	${\bf N}$ STOP
Caustic leach in UFP-VSL-T01B	Caustic leach duration (endpoint)	16	$-0.1/+1$	hr	16.2	NQA-1	
(Test Instructions)	Temperature	98	$-2/+2$	$\rm ^{\circ}C$	~108	$NQA-1$	
steps 7.1.6.16 to 7.1.6.19	Volume of water to add to get prototypic condensate	2.9	$-0.1/+0.1$	L	2.9	This quantity added after samples are collected at $1, 2, 3, \ldots 13, 14$, and 15 hrs after start of leach. Information Only	
	UFP-VSL-T01B Operation						
	PJM jet velocity	4.8	$-0.3/+0.3$	m/s	5.4	NQA-1	
	PJM stroke (80%)	29.0	$-2/+2$	in	$21.2(60\%)$	NQA-1	

Table J.1. Integrated Test A Operational Process Sheet

			Integrated Test A Run sheet (TI-065 Rev. 0)				
	Operation	Target or Setpoint Value	Range $(-/+)$	Units	Actual Value	Comments	
and add 17.9 M caustic in-line	addition (in-line after HLP-PMP-T21)						
(Test Instructions steps $7.1.7.1$ to 7.1.7.9)	17.9 M caustic addition rate	15.516.7	$-1/+310$	kg/min	\sim 22	Caustic addition should finish 1 to 2 min before slurry transfer finishes to flush caustic from transfer line. Value obtained from the HMI	
	UFP-VSL-T01A Operation						
	Volume of antifoam to add to UFP-VSL-T01A	890	$-20/+20$	mL	890	This value is concentrate. Dilute with 3 parts IW to 1 part concentrate. Information Only	
	Level to initiate PJM operation	15	$-0/10$	in	30	Value obtained from the HMI	
	PJM jet velocity	4.8	$-0.3/+0.3$	m/s	4.6	NQA-1	
	PJM stroke (80%)	29.0	$-2/+2$	in	25.4 (72%)	NQA-1	
	PJM cycle time Steam ring air purge	35	$-1/+1$	S	35.3	$NQA-1$ 6.1 scfm below 90°C in UFP-VSL-T01A	
	flowrate	0.20	$0.02/+0.02$	kg/min	0.168	Local Calibrated Instrument, NQA-1	
Initial heat-up of UFP-VSL-T01A	Initial heat-up temperature (endpoint)	57	$-1/+3$	$^{\circ}C$	~ 57	Y (init.) NQA-1	N STOP
(Non-prototypic using external exchanger) (Test Instructions steps $7.1.7.10$ to 7.1.7.18	Time to heat to initial heat-up temperature	$<$ 3	NA	hr	1.5	Heating loop is \sim 9 gallons and will be lost (unprototypic). Target and range is not important. Should be complete in under 3 hours. NQA-1	
Final heat-up UFP-VSL-T01A	Target final temperature (endpoint)	98	$-2/+2$	$\rm ^{\circ}C$	~108	Y (init.) NQA-1	N STOP
(Test Instructions Heat-up time)		3.8	$-0.5/+0.5$	hr	3.7	$NQA-1$	
steps 7.1.7.19 to 7.1.7.22	UFP-VSL-T01A Operation			$\overline{}$			
	Temperature to turn off PJM suction refill	60	$-2/+2$	$\rm ^{\circ}C$	60	Value obtained from the HMI	

Table J.1. Integrated Test A Operational Process Sheet

			Integrated Test A Run sheet (TI-065 Rev. 0)				
	Operation	Target or Setpoint Value	Range $(-/+)$	Units	Actual Value	Comments	
	PJM jet velocity	4.8	$-0.3/+0.3$	m/s	5.2	$NQA-1$	
	PJM stroke (80%)	29.0	$-2/+2$	in	28 (80%)	$NQA-1$	
	PJM cycle time	35	$-1/+1$	S	35.1	$NQA-1$	
	Steam ring air purge flowrate	0.20	$0.02/+0.02$	kg/min	0.2	6.1 scfm below 90°C in UFP-VSL-T01A Local Calibrated Instrument, NQA-1	
Initial heat-up of UFP-VSL-T01B	Initial heat-up temperature (endpoint)	57	$-1/+3$	$\rm ^{\circ}C$	59	Y (init.) $NQA-1$	N STOP
(Non-prototypic using external exchanger) (Test Instructions steps 7.1.8.15 to 7.1.8.23	Time to heat to initial heat-up temperature	$<$ 3	NA	hr	1.3	Heating loop is \sim 9 gallons and will be lost (unprototypic). Target and range is not important. Should be complete in under 3 hours. NQA-1	
Final heat-up UFP-VSL-T01B	Target final temperature (endpoint)	98	$-2/+2$	$\rm ^{\circ}C$	~108	Y (init.) $NQA-1$	N STOP
(Test Instructions	Heat-up time	3.8	$-0.5/+0.5$	hr	3.6	$NQA-1$	
steps 7.1.8.24 to 7.1.8.27	UFP-VSL-T01B Operation	$\overline{}$					
	Temperature to turn off PJM suction refill	60	$-2/+2$	$\rm ^{\circ}C$	60	Value obtained from the HMI	
	Temperature to reduce steam ring air purge rate	90	$-2/+8$	$\rm ^{\circ}C$	90	Value obtained from the HMI	
	High-temperature steam ring air purge flowrate	0.13	$0.02/+0.02$	kg/min	0.13	4.0 scfm above 90°C in UFP-VSL-T01A Local Calibrated Instrument, NQA-1	
	Level at end of heat-up	61	$-2/+2$	in	58.7	Y (init.) Anticipate accumulation of $~62$ gal of condensate during heat-up. $NQA-1$	${\bf N}$ STOP
Caustic leach in UFP-VSL-T01B	Caustic leach duration (endpoint)	16	$-0.1/+1$	hr	15.9	NQA-1	

Table J.1. Integrated Test A Operational Process Sheet

			Integrated Test A Run sheet (TI-065 Rev. 0)				
	Operation	Target or Setpoint Value	Range $(-/+)$	Units	Actual Value	Comments	
(Test Instructions	Temperature	98	$-2/+2$	\overline{C}	~100	$NQA-1$	
steps 7.1.8.28 to 7.1.8.32)	Volume of water to add to get prototypic condensate	2.9	$-0.1/+0.1$	L	2.9	This quantity added after samples are collected at $1, 2, 3, \ldots 13, 14$, and 15 hrs after start of leach. Information Only	
	UFP-VSL-T01B Operation	\blacksquare			\overline{a}		
	PJM jet velocity	4.8	$-0.3/+0.3$	m/s	4.6	$NQA-1$	
	PJM stroke (80%)	29.0	$-2/+2$	in	28.2 (80%)	$NQA-1$	
	PJM cycle time	35	$-1/+1$	S	35.1	$NQA-1$	
	Steam ring air purge flowrate (automatic)	0.13	$0.02/+0.02$	kg/min	0.13	\sim 4.0 scfm, purge will turn off when steam exceeds 6 cfm and will turn back on when steam flow drops below 4 cfm Local Calibrated Instrument, NQA-1	
	Level at end of leach	66	$-5/+5$	in	62.8	Y (init.) \sim 593 gal $NQA-1$	${\bf N}$ STOP
Cool-down of UFP-VSL-T01B by UFP-HX-T05A (Test Instructions)	Initial cool-down temperature using external chiller UFP-HX-T05B (endpoint)	60	$-2/+2$	$\rm ^{\circ}C$	~ 60	NQA-1	
steps 7.1.8.32 to 7.1.8.36	Initial cool-down duration	2.75	$-0.5/+0.5$	hr	2.8	Table 4.4 of the Run Report Value obtained from the HMI	
	UFP-VSL-T01B Operation	\sim		\overline{a}	\blacksquare		
	Temperature to increase steam ring air purge rate	90	$-5/+2$	$\rm ^{\circ}C$	~10	NQA-1	
	Steam ring air purge flowrate	0.20	$-0.02/$ $+0.02$	kg/min	0.16	6.1 scfm below 90 \degree C in UFP-VSL-T01A Local Calibrated Instrument, NQA-1	
	Temperature to resume PJM suction refill	60	$-2/+2$	$\rm ^{\circ}C$	60	$NQA-1$	

Table J.1. Integrated Test A Operational Process Sheet

				Integrated Test A Run sheet (TI-065 Rev. 0)				
		Operation	Target or Setpoint Value	Range $(-/+)$	Units	Actual Value	Comments	
	leached simulant volume	Transfer and cool Make-up batch transfer	11	$-0.2/+0.2$	gal	11	Value obtained from the HMI	
	from UFP-VSL-T01B	Make-up batch flowrate	6.9	NA	gpm	6.9	Value obtained from the HMI	
	to UFP-VSL-T02A and concentrate solids	UFP-VSL-T01B level (endpoint to stop batch transfers)	10	$-2/+2$	in	~10	Y (init.) Estimate 47 make-up batches from UFP-VSL-T01B. $NQA-1$	N STOP
	steps $7.1.11.6$ to 7.1.11.8	(Test Instructions UFP-VSL-T02A level to initiate each make- up batch	44.7	$-1/+1$	in	44.7	Value obtained from the HMI	
		UFP-VSL-T02A Operation						
		PJM jet velocity	7.3	$-0.4/+0.4$	m/s	$- -$	Not recorded	
		PJM stroke (80%)	31.5	$-2/+2$	in	$\overline{}$	Not recorded	
		PJM cycle time	33	$-1/+1$	$\mathbf S$	33	Value obtained from the HMI	
		Steam ring air purge flowrate	0.14	$0.02/+0.02$	kg/min	0.14	4.1 scfm below 90°C in UFP-VSL-T02A $NQA-1$	
Concentrate Leach Batch 4		Total flowrate of 5 lower air spargers (idle)	0.04	$-0.005/+$ 0.005	kg/min	0.04	\sim 13 scfh for each sparge tube NQA-1	
		Flowrate of upper air sparger (idle)	0.01	$-0.005/+$ 0.005	kg/min	0.01	\sim 16 scfh (record field transmitter value) $NQA-1$	
		Ultrafiltration Loop Operation						
		Number of filter				--		
		bundles (UFP-FLT-T01A should be aligned)		$-0/0+$		1	Information Only	
		Filter loop flowrate	109	$-10/+10$	gpm	110	Value obtained from the HMI	
		Temperature	25	$-2/+2$	$\rm ^{\circ}C$	25.5	Value obtained from the HMI	
		Axial ΔP for one filter	<25	\overline{a}	psi	20.5	Y (init.)	N

Table J.1. Integrated Test A Operational Process Sheet

				Integrated Test A Run sheet (TI-065 Rev. 0)				
		Operation	Target or Setpoint Value	Range $(-/+)$	Units	Actual Value	Comments	
		bundle					Value obtained from the HMI	STOP
	Transfer simulant from	Total slurry transfer volume (endpoint)	305 415	$-5/+5$	gal	415	Assumes previous heel in UFP-VSL-T01A. Value obtained from the HMI	
	HLP-VSL-T22 to	Slurry transfer rate	7.4	$-2/+2$	gpm	7.4	Value obtained from the HMI	
	UFP-VSL-T01A and add 17.9 M caustic in-line	Total 17.9 M caustic addition (in-line after HLP-PMP-T21)	625	$-5/+5$	kg	625	Value obtained from the HMI	
	(Test Instructions) steps $7.1.9.1$ to 7.1.9.9)	17.9 M caustic addition rate	15.9 17.2	$-1/+310$	kg/min	17.2	Caustic addition should finish 1 to 2 min before slurry transfer finishes to flush caustic from transfer line. Value obtained from the HMI	
		UFP-VSL-T01A Operation						
5		Volume of antifoam to add to UFP-VSL-T01A	871	$-20/+20$	mL	871	This value is concentrate. Dilute with 3 parts IW to 1 part concentrate. Information Only	
Leach Batch		Level to initiate PJM operation	15	$-0/10$	in	42	Value obtained from the HMI	
		PJM jet velocity	4.8	$-0.3/+0.3$	m/s	5.1	$NQA-1$	
		PJM stroke (80%)	29.0	$-2/+2$	in	30.7 (87%)	$NQA-1$	
		PJM cycle time	35	$-1/+1$	$\,$ S	35.3	$NQA-1$	
		Steam ring air purge flowrate	0.20	$0.02/+0.02$	kg/min	0.17	6.1 scfm below 90°C in UFP-VSL-T01A Local Calibrated Instrument, NQA-1	
	Initial heat-up of UFP-VSL-T01A	Initial heat-up temperature (endpoint)	57	$-1/+3$	$\rm ^{\circ}C$	57.3	Y (init.) $NQA-1$	${\bf N}$ STOP
	(Non-prototypic using external exchanger) (Test Instructions) steps 7.1.9.10 to 7.1.9.18	Time to heat to initial heat-up temperature	$<$ 3	NA	hr	1.1	Heating loop is \sim 9 gallons and will be lost (unprototypic). Target and range is not important. Should be complete in under 3 hours. NQA-1	
	Final heat-up UFP-VSL-T01A	Target final temperature (endpoint)	98	$-2/+2$	$\rm ^{\circ}C$	~108	Y (init.) NQA-1	N STOP

Table J.1. Integrated Test A Operational Process Sheet

	Integrated Test A Run sheet (TI-065 Rev. 0)								
	Operation	Target or Setpoint Value	Range $(-/+)$	Units	Actual Value	Comments			
(Test Instructions Heat-up time		3.8	$-0.5/+0.5$	hr	3.6	NQA-1			
steps 7.1.9.19 to 7.1.9.21)	UFP-VSL-T01A Operation			\blacksquare					
	Temperature to turn off PJM suction refill	60	$-2/+2$	$\rm ^{\circ}C$	60	Value obtained from the HMI			
	Temperature to reduce steam ring air purge rate	90	$-2/+8$	$\rm ^{\circ}C$	95	Value obtained from the HMI			
	High-temperature steam ring air purge flowrate	0.13	$0.02/+0.02$	kg/min	0.13	4.0 scfm above 90°C in UFP-VSL-T01A Local Calibrated Instrument, NQA-1			
	Level at end of heat-up	61	$-2/+2$	in	59.8	Y (init.) Anticipate accumulation of ~62 gal of condensate during heat-up. $NQA-1$	N STOP		
Caustic leach in UFP-VSL-T01A	Caustic leach duration (endpoint)	16	$-0.1/+1$	hr	16.0	NQA-1			
(Test Instructions)	Temperature	98	$-2/+2$	$\rm ^{\circ}C$	~108	$NQA-1$			
steps $7.1.9.21$ to 7.1.9.27	Volume of water to add to get prototypic condensate	2.9	$-0.1/+0.1$	\mathbf{L}	2.9	This quantity added after samples are collected at $1, 2, 3, \ldots 13, 14$, and 15 hrs after start of leach. Information Only			
	UFP-VSL-T01A Operation	\blacksquare							
	PJM jet velocity	4.8	$-0.3/+0.3$	m/s	4.8	$NQA-1$			
	PJM stroke (80%)	29.0	$-2/+2$	in	27.1(77%)	$NQA-1$			
	PJM cycle time	35	$-1/+1$	${\bf S}$	35.3	$NQA-1$			
	Steam ring air purge flowrate (automatic)	0.13	$0.02/+0.02$	kg/min	0.13	\sim 4.0 scfm, purge will turn off when steam exceeds 6 cfm and will turn back on when steam flow drops below 4 cfm Local Calibrated Instrument, NQA-1			
	Level at end of leach	66	$-5/+5$	in	64.4	Y (init.)	${\bf N}$		

Table J.1. Integrated Test A Operational Process Sheet
Integrated Test A Run sheet (TI-065 Rev. 0)								
		Operation	Target or Setpoint Value	Range $(-/+)$	Units	Actual Value	Comments	
							\sim 593 gal $NQA-1$	STOP
	Cool-down of UFP-VSL-T01A by UFP-HX- T ₀₅ A (Test Instructions)	Initial cool-down temperature using external chiller UFP-HX-T05A (endpoint)	60	$-2/+2$	$^{\circ}\mathrm{C}$	60	Value obtained from the HMI	
	steps $7.1.9.28$ to 7.1.9.34	Initial cool-down duration	2.75	$-0.5/+0.5$	hr	$\overline{3}$	Value obtained from the HMI	
		UFP-VSL-T01A Operation				\blacksquare		
		Temperature to increase steam ring air purge rate	90	$-5/+2$	$\rm ^{\circ}C$	90	Value obtained from the HMI	
		Steam ring air purge flowrate	0.20	$0.02/+0.02$	kg/min	0.164	6.1 scfm below 90°C in UFP-VSL-T01A Local Calibrated Instrument, NQA-1	
		Temperature to resume PJM suction refill	60	$-2/+2$	$^{\circ}C$	60	Value obtained from the HMI	
	Transfer and cool leached simulant	Make-up batch transfer volume	11	$-0.2/+0.2$	gal	11	Value obtained from the HMI	
	from UFP-VSL-T01A	Make-up batch flowrate	6.9	NA	gpm	6.9	Value obtained from the HMI	
Concentrate Leach Batch 5	to UFP-VSL-T02A and concentrate solids	UFP-VSL-T01A level (endpoint to stop batch transfers)	10	$-2/+2$	in	9.8	Y (init.) Estimate 47 make-up batches from UFP-VSL-T01A. Value obtained from the HMI	${\bf N}$ STOP
	(Test Instructions steps $7.1.11.6$ to	UFP-VSL-T02A Operation						
	7.1.11.8	UFP-VSL-T02A level to initiate each make- up batch	44.747.3	$-1/+1$	in	44.7	Value obtained from the HMI	
		PJM jet velocity	7.3	$-0.4/+0.4$	m/s	7.5	Value obtained from the HMI	
		PJM stroke (80%)	31.5	$-2/+2$	in	77%	Value obtained from the HMI	

Table J.1. Integrated Test A Operational Process Sheet

Integrated Test A Run sheet (TI-065 Rev. 0)								
		Operation	Target or Setpoint Value	Range $(-/+)$	Units	Actual Value	Comments	
		PJM cycle time	33	$-1/+1$	S	33	Value obtained from the HMI	
		Steam ring air purge flowrate	0.14	$0.02/+0.02$	kg/min	0.14	4.1 scfm below 90°C in UFP-VSL-T02A Local Calibrated Instrument, NQA-1	
		Total flowrate of 5 lower air spargers (idle)	0.04	$-0.005/+$ 0.005	kg/min	0.04	\sim 13 scfh for each sparge tube Local Calibrated Instrument, NQA-1	
		Flowrate of upper air sparger (idle)	0.01	$-0.005/+$ 0.005	kg/min	0.01	\sim 16 scfh (record field transmitter value) Local Calibrated Instrument, NQA-1	
		Ultrafiltration Loop Operation				$\overline{}$		
		Number of filter bundles (UFP-FLT-T01A should be aligned)	1	$-0/0+$	$\overline{}$	-- 1	Information Only	
		Filter loop flowrate	109	$-10/+10$	gpm	109	Value obtained from the HMI	
		Temperature Axial ΔP for one filter bundle	25 $<$ 25	$-2/+2$ \blacksquare	$\rm ^{\circ}C$ psi	25 22.7	Value obtained from the HMI Y (init.) Value obtained from the HMI	N STOP
	from	Transfer simulant Slurry transfer volume (endpoint)	305393	$-5/+5$	gal	393	Assumes previous heel in UFP-VSL-T01B. Value obtained from the HMI	
		HLP-VSL-T22 to Slurry transfer rate	7.4	$-2/+2$	gpm	$7 - 15$	Value obtained from the HMI	
	UFP-VSL-T01B and add 17.9 M caustic in-line	17.9 M caustic addition (in-line after) HLP-PMP-T21)	499	$-5/+5$	kg	499	Value obtained from the HMI	
Leach Batch 6	(Test Instructions steps 7.1.10.1 to 7.1.10.13)	In-line 17.9 M caustic addition rate	12.713.8	$-1/+310$	kg/min	$11 - 60$	Caustic addition should finish 1 to 2 min before slurry transfer finishes to flush caustic from transfer line. Value obtained from the HMI	
		17.9 M caustic addition to top of UFP-VSL-T01B $(in-tank)$	125	$-2/+2$	kg	124.8	NQA-1	

Table J.1. Integrated Test A Operational Process Sheet

Integrated Test A Run sheet (TI-065 Rev. 0)								
			Target or					
		Operation	Setpoint Value	Range	Units	Actual Value		
		Temperature to		$(-/+)$			Comments	
		increase steam ring air	90	$-5/+2$	$\rm ^{\circ}C$	85	Value obtained from the HMI	
		purge rate						
		Steam ring air purge	0.20	$0.02/+0.02$	kg/min	0.16	6.1 scfm below 90°C in UFP-VSL-T01A	
		flowrate					Local Calibrated Instrument, NQA-1	
		Temperature to resume PJM suction refill	60	$-2/+2$	$^{\circ}{\rm C}$	60	Value obtained from the HMI	
		Transfer and cool Make-up batch transfer						
	leached simulant volume from		11	$-0.2/+0.2$	gal	~11	$NQA-1$	
		Make-up batch						
	UFP-VSL-T01B	flowrate	6.9	NA	gpm	--	Not recorded	
	to	UFP-VSL-T01B level					Y (init.)	${\bf N}$
	UFP-VSL-T02A and concentrate solids (Test Instructions)	(endpoint to stop batch	41	$-2/+2$	in	\sim 35	Estimate 22 make-up batches from	STOP
\circ		transfers) UFP-VSL-T02A level					UFP-VSL-T01B. NQA-1	
		to initiate each make-	44.747.3	$-1/+1$	in	44.7	Value obtained from the HMI	
Concentrate Leach Batch	steps $7.1.11.6$ to	up batch						
	7.1.11.8	UFP-VSL-T02A						
		Operation						
	Steps 7.1.11.6 to 8 are not related	PJM jet velocity	7.3	$-0.4/+0.4$	m/s	7.6	Value obtained from the HMI	
	to PJM	PJM stroke (80%)	31.5	$-2/+2$	in	32.5	Value obtained from the HMI	
	operations.	PJM cycle time Steam ring air purge	33	$-1/+1$	S	33	Value obtained from the HMI 4.1 scfm below 90°C in UFP-VSL-T02A	
		flowrate	0.14	$0.02/+0.02$	kg/min	0.14	Local Calibrated Instrument, NQA-1	
		Total flowrate of 5						
		lower air spargers	0.04	$-0.005/+$ 0.005	kg/min	0.04	\sim 13 scfh for each sparge tube Local Calibrated Instrument, NQA-1	
		(idle)						
		Flowrate of upper air	0.01	$-0.005/+$	kg/min	0.01	\sim 16 scfh (record field transmitter value)	
		sparger (idle)		0.005			Local Calibrated Instrument, NQA-1	
		Ultrafiltration Loop						

Table J.1. Integrated Test A Operational Process Sheet

				Integrated Test A Run sheet (TI-065 Rev. 0)				
			Target or					
			Setpoint	Range				
		Operation	Value	$(-/+)$	Units	Actual Value	Comments	
		PJM stroke (80%)	31.5	$-2/+2$	in	$\overline{}$	Not recorded	
		PJM cycle time	20	$-1/+1$	$\,$ s	20	Value obtained from the HMI	
		Steam ring air purge flowrate	0.14	$0.02/+0.02$	kg/min	0.137	4.1 scfm below 90°C in UFP-VSL-T02A WTP-RPT-187	
		Total lower air sparger flow rate with recirculation (idle)	0.04	$-0.005/+$ 0.005	kg/min	0.008	\sim 12.6 scfh for each sparger tube WTP-RPT-187	
		Upper air sparger flow rate (idle)	0.01	$-0.005/+$ 0.005	kg/min	0.01	\sim 15.6 scfh Local Calibrated Instrument, NQA-1	
		Ultrafiltration Loop Operation						
		Number of filter bundles	5	$-0/0-$		5	Y (init.) Information Only	${\bf N}$ STOP
		Filter loop flowrate	109	$-10/+10$	gpm	$-88.5 - 133.9$	WTP-RPT-187	
		Trans-membrane pressure	40	$-4/+4$	psi	39.4	Lower range not applicable if back pressure valve is 100% open. WTP-RPT-187	
		Temperature	25	$-2/+2$	$\rm ^{\circ}C$	24.3	WTP-RPT-187	
		Axial ΔP for one filter bundle	<25	\blacksquare	psi	24.2	Y (init.) WTP-RPT-187	${\bf N}$ STOP
	Add chromium slurry (Test Instructions	Volume of chromium solids slurry to add (endpoint)	144	$-1/+1$	gal	145	Chromium slurry vessel may be washed out with as much as 20 gal of inhibited water. WTP-RPT-188	
	step $7.1.14.1$)	Chromium slurry addition flowrate	No targeted rate					
Chromium Slurry Addition and Wash		Volume of antifoam to add to UFP-VSL-T02A UFP-VSL-T02A Operation	230	$-5/+5$	g mL	230	Antifoam is added to account for dilution by the Cr slurry addition. Information Only	
		PJM jet velocity	12	$-0.6/+0.6$	m/s	13.6	Value obtained from the HMI	
		PJM stroke (80%)	31.5	$-2/+2$	in	29.5	Value obtained from the HMI	
		PJM cycle time	20	$-1/+1$	${\bf S}$	20	Value obtained from the HMI	

Table J.1. Integrated Test A Operational Process Sheet

Integrated Test A Run sheet (TI-065 Rev. 0)							
	Operation	Target or Setpoint Value	Range $(-/+)$	Units	Actual Value	Comments	
	Steam ring air purge flowrate	0.14	$0.02/+0.02$	kg/min	$\boldsymbol{0}$	4.1 scfm below 90°C in UFP-VSL-T02A Local Calibrated Instrument, NQA-1	
	Total lower air sparger flow rate with recirculation (idle)	0.04	$-0.005/+$ 0.005	kg/min	0.04	\sim 12.6 scfh for each sparge tube Local Calibrated Instrument, NQA-1	
	Upper air sparger flow rate (idle)	0.01	$-0.005/+$ 0.005	kg/min	0.01	\sim 15.6 scfh Local Calibrated Instrument, NQA-1	
	Ultrafiltration Loop Operation						
	Number of filter bundles	5	$-0/0$		5	Y (init.) Information Only	${\bf N}$ STOP
	Filter loop flowrate	109	$-10/+10$	gpm	103	Value obtained from the HMI	
	Permeate valves	CLOSED		\blacksquare	Closed	Y (init.) Information Only	
	Temperature	25	$-2/+2$	$\rm ^{\circ}C$	24	Value obtained from the HMI	
	Axial ΔP for one filter bundle	< 25		psi	<25	Y (init.) Value obtained from the HMI	$\mathbf N$ STOP
Wash post- chromium slurry (Test Instructions	UFP-VSL-T02A level to initiate wash water batch addition	42-44.3	$-1/+1$	in	44.3	Value obtained from the HMI	
step 7.1.14.2 to 7.1.14.9	Total wash water	1,749-1540	$-10/+10$	kg	1593	462 gal of wash water in 42 wash water batches $NQA-1$	
	Wash water batch flow rate	28.0	$-3/+3$	kg/min	$28\,$	\sim 7.4 gpm Value obtained from the HMI	
	Wash water batch size	41.6	$-1/+1$	kg	41.6	Value obtained from the HMI	
	Volume of antifoam to add initially and after every 3rd 11-gal batch to UFP-VSL-T02A	44	$-2/+2$	mL	44	Antifoam $(\sim]$ 14 ml per 11 gallon wash batch) is added to account for wash water dilution. This value is concentrate. Dilute with 3 parts IW to 1 part concentrate. Information Only	
	UFP-VSL-T02A Operation						
	PJM jet velocity	12	$-0.6/+0.6$	m/s	$\overline{}$	Not recorded	

Table J.1. Integrated Test A Operational Process Sheet

			Integrated Test A Run sheet (TI-065 Rev. 0)				
	Operation	Target or Setpoint Value	Range $(-/+)$	Units	Actual Value	Comments	
	Total lower air sparger flow rate with recirculation (idle)	0.040	$-0.005/+$ 0.005	kg/min	$1.13 - 1.25$ scfm	\sim 12.6 scfh for each sparge tube WTP-RPT-188	
	Upper air sparger flow rate (idle)	$0.01 - 0$	$-0.005/+$ 0.005	kg/min	$\boldsymbol{0}$	\sim 15.6 scfh WTP-RPT-188	
	Ultrafiltration Loop Operation						
	Number of filter bundles	5	$-0/+0$		5	Y (init.) Information Only	${\bf N}$ STOP
	Filter loop flowrate	109	$-10/+10$	gpm	114	WTP-RPT-188	
	Permeate valves	CLOSED			Closed	Value obtained from the HMI	
	Temperature	25	$-2/+2$	$\rm ^{\circ}C$	25	Value obtained from the HMI	
	Axial ΔP for one filter bundle	<25		psi	$16 - 20$	Y (init.) Value obtained from the HMI	${\bf N}$ STOP
Conduct oxidative leach	Oxidative leach duration (endpoint)	6	$-0.1/+0.5$	hr	6	May be extended to accommodate sampling. WTP-RPT-188	
(Test Instructions)	Temperature	25	$-2/+2$	$\rm ^{\circ}C$	$24.4 - 27.8$	WTP-RPT-188	
step 7.1.14.14)	UFP-VSL-T02A Operation						
	PJM jet velocity	12	$-0.6/+0.6$	m/s	12.1	WTP-RPT-188	
	PJM stroke (80%) PJM cycle time	31.5 20	$-2/+2$ $-1/+1$	in S	82% 20.8	WTP-RPT-188 WTP-RPT-188	
	Steam ring air purge flowrate	0.1400	$0.02/+0.02$	kg/min	$\boldsymbol{0}$	4.1 scfm below 90°C in UFP-VSL-T02A WTP-RPT-188	
	Total lower air sparger flow rate with recirculation (idle)	$0.040 - 1.19$ scfm	$-0.005/+$ 0.005	kg/min	1.18 scfm	\sim 12.6 scfh for each sparge tube WTP-RPT-188	
	Upper air sparger flow rate (idle)	$0.010 - 0$	$-0.005/+$ 0.005	kg/min	$\boldsymbol{0}$	\sim 15.6 scfh WTP-RPT-188	
	Ultrafiltration Loop Operation						
	Number of filter bundles	5	$-0/+0$		5	Y (init.) Information Only	N STOP

Table J.1. Integrated Test A Operational Process Sheet

				Integrated Test A Run sheet (TI-065 Rev. 0)				
			Target or Setpoint	Range				
		Operation	Value	$(-/+)$	Units	Actual Value	Comments	
		Filter loop flowrate	109	$-10/+10$	gpm	89	WTP-RPT-188	
		Permeate valves	CLOSED					
		Temperature	25	$-2/+2$	$\rm ^{\circ}C$	$24.9 - 27.4$	WTP-RPT-188	
		Axial ΔP for one filter bundle	<25	\overline{a}	psi	$18 - 21$	Y (init.) $NQA-1$	${\bf N}$ STOP
	Wash post- oxidative leach slurry	UFP-VSL-T02A level to initiate wash water batch addition	50.9-52.5	$-1/+1$	in	~ 52	231 gal in UFP-VSL-T02A + 86 gal in filter loop $NQA-1$	
	(Test Instructions step 7.1.15.1 to	Total wash water	3,622-3581	$-20/+20$	kg	3657	87 batches Value obtained from the HMI	
	7.1.15.3.6.3)	Wash water batch flow rate	28.0	$-3/+3$	kg/min	\sim 25	\sim 7.4 gpm $NQA-1$	
		Wash water batch size	41.6	$-1/+1$	kg	42.5	WTP-RPT-187	
Post Oxidative Leach Wash and Dewater		Volume of antifoam to add initially and after every 3rd 11-gal batch to UFP-VSL-T02A	44	$-2/+2$	mL	44	Antifoam (~14 ml per 11 gallon wash batch) is added to account for wash water dilution. This value is concentrate. Dilute with 3 parts IW to 1 part concentrate. Information Only	
		Estimated total permeate mass	3,600-3500	$-100/+200$	kg	3792	Estimate is rounded down to nearest hundred. Value obtained from the HMI	
		UFP-VSL-T02A Operation						
		PJM jet velocity	12	$-0.6/+0.6$	m/s	12	Value obtained from the HMI	
		PJM stroke (80%)	31.5	$-2/+2$	in	$\overline{}$	Not recorded	
		PJM cycle time	20	$-1/+1$	S	\overline{a}	Not recorded	
		Steam ring air purge flowrate	0.14	$0.02/+0.02$	kg/min	Off	4.1 scfm below 90°C in UFP-VSL-T02A Local Calibrated Instrument, NQA-1	
		Total lower air sparger flow rate with recirculation (idle)	0.04	$-0.005/+$ 0.005	kg/min	Off	\sim 12.6 scfh for each sparge tube Local Calibrated Instrument, NQA-1	
		Upper air sparger flow rate (idle)	0.01	$-0.005/+$ 0.005	kg/min	Off	\sim 15.6 scfh Local Calibrated Instrument, NQA-1	
		Ultrafiltration Loop						

Table J.1. Integrated Test A Operational Process Sheet

Integrated Test A Run sheet (TI-065 Rev. 0)								
	Operation	Target or Setpoint Value	Range $(-/+)$	Units	Actual Value	Comments		
	Operation							
	Number of filter bundles	5	$-0/0+$		5	Y (init.) Information Only	N STOP	
	Filter loop flowrate	109	$-10/10$	gpm	151.9	$FI-0625 = 73$ WTP-RPT-187		
	Trans-membrane pressure	40	$-4/+4$	psi	39.8	Lower range not applicable if back pressure valve is 100% open. WTP-RPT-187		
	Temperature	25	$-2/+2$	$\rm ^{\circ}C$	23.3	WTP-RPT-187		
	Axial ΔP for one filter bundle	< 25		psi	16.3	Y (init.) WTP-RPT-187	${\bf N}$ STOP	
Concentrate post- oxidative leach	UFP-VSL-T02A level (endpoint)	$37.3 - 40$	$-1/+1$	in	38.9	Remove \sim 226 kg of permeate. Value obtained from the HMI		
slurry (Test Instructions)	UFP-VSL-T02A Operation				\blacksquare			
step 7.1.15.3.6.4	PJM jet velocity	12	$-0.6/+0.6$	m/s	12	Value obtained from the HMI		
to $7.1.15.3.6.8$)	PJM stroke (80%)	31.5	$-2/+2$	in	31.5	Value obtained from the HMI		
	PJM cycle time	20	$-1/+1$	S	20	Value obtained from the HMI		
	Steam ring air purge flowrate	0.14	$0.02/+0.02$	kg/min	Off	4.1 scfm below 90°C in UFP-VSL-T02A Local Calibrated Instrument, NQA-1		
	Total lower air sparger flow rate with recirculation (idle)	0.04	$-0.005/+$ 0.005	kg/min	Off	\sim 12.6 scfh for each sparge tube Local Calibrated Instrument, NQA-1		
	Upper air sparger flow rate (idle)	0.01	$-0.005/+$ 0.005	kg/min	$\boldsymbol{0}$	\sim 15.6 scfh Local Calibrated Instrument, NQA-1		
	Ultrafiltration Loop Operation				\blacksquare			
	Number of filter bundles	5	$-0/0+$	\sim	5	Y (init.) Information Only	$\mathbf N$ STOP	
	Filter loop flowrate	109	$-10/10$	gpm	73 $(FI-0623)$	$FI-0625 = 123$ Value obtained from the HMI		
	Trans-membrane pressure	40	$-4/+4$	psi	40	Lower range not applicable if back pressure valve is 100% open. Value obtained from the HMI		

Table J.1. Integrated Test A Operational Process Sheet

	Integrated Test A Run sheet (TI-065 Rev. 0)									
			Target or							
			Setpoint	Range						
		Operation	Value	$(-/+)$	Units	Actual Value	Comments			
		Temperature	$\overline{25}$	$-2/+2$	\overline{C}	25	Value obtained from the HMI			
	Partial transfer from	Target level in UFP- VSL-T02A (endpoint)	20-22.5	$-1/+3$	in		<u> 1995 - Johann Hermann, Amerikaansk ferfinger om de formannelse om de formannelse om de formannelse om de for</u> <u> Kanada (Kanada (Kana</u>			
	UFP-VSL-T02A	Transfer rate	No set point	\blacksquare						
	(Test Instructions step 7.1.16.1 to 7.1.16.3)	UFP-VSL-T02A Operation								
		PJM jet velocity	12	$-0.6/+0.6$	m/s					
		PJM stroke (80%)	31.5	$-2/+2$	in					
		PJM cycle time	20	$-1/+1$	S					
		Steam ring air purge flowrate	0.14	$0.02/+0.02$	kg/min					
Prepare and Run High-Solids Filter Flux Test		Total lower air sparger flow rate with recirculation (idle)	0.04	$-0.005/+$ 0.005	kg/min					
		Upper air sparger flow rate (idle)	0.01	$-0.005/+$ 0.005	kg/min		<u> Kabupatèn Bandaran Indonesia (Kabupatèn Bandaran Indonesia (Kabupatèn Bandaran Indonesia (Kabupatèn Bandaran</u>			
		Ultrafiltration Loop Operation								
		Number of filter bundles	$\mathbf{1}$	$-0/0-$			<u>ta kalendari ka</u>	en de la familie de la fam De la familie de la famili <u>tin dhe që përfshën për përfsh</u>		
		Permeate valves	CLOSED							
		Filter loop flowrate	109	$-10/+10$	gpm					
		Temperature	25	$-2/+2$	$\rm ^{\circ}C$					
		Axial ΔP for one filter bundle	<25	\blacksquare	psi		<u> Kabupatèn Bandaran Indonesia Bandaran Indonesia Bandaran Indonesia Bandaran Indonesia Bandaran Indonesia Ba</u>	en de la familie de la fam De la familie de la famili <u>estas por estas por</u>		
	Dilute UFP-VSL-T02A	Target UFP-VSL- T02A level (endpoint)	62.565.2	$-5/+5$	in	63.9	UFP-VSL-T02A volume \sim 283 gal Value obtained from the HMI			
	slurry with inhibited water	Inhibited water addition rate	No set point			$~10~\text{kg/min}$	$NQA-1$			
	(Test Instructions step 7.1.16.4 to	Collect sample for parallel testing				Yes	Y (init.) Value obtained from the HMI	${\bf N}$ STOP		
	7.1.16.6									

Table J.1. Integrated Test A Operational Process Sheet

Integrated Test A Run sheet (TI-065 Rev. 0)										
	Operation	Target or Setpoint Value	Range $(-/+)$	Units	Actual Value	Comments				
step $7.1.17$)	rate	achievable flow				<u>ka maa maana maraa marka ma</u>				
	UFP-VSL-T02A Operation									
	PJM jet velocity	12	$-0.6/+0.6$	m/s						
	PJM stroke (80%)	31.5	$-2/+2$	in						
	PJM cycle time	$20\,$	$-1/+1$	${\bf S}$						
	Steam ring air purge flowrate	0.14	$0.02/+0.02$	kg/min						
	Total lower air sparger flow rate with recirculation (idle)	0.04	$-0.005/+$ 0.005	kg/min		<u> 1999 - Johann Strauss, fransk konge og formanne</u>				
	Upper air sparger flow rate (idle)	0.01	$-0.005/+$ 0.005	kg/min						
	Ultrafiltration Loop Operation									
	Number of filter bundles	5	$-0/+0$			<u> Kabupatèn Bandaran Indonesia Bandaran Indonesia Bandaran Indonesia Bandaran Indonesia Bandaran Indonesia Ba</u> en de la familie de la fam Constituit de la familie d <u>i seria di seria di</u>				
	Filter loop flowrate	OFF								
	Permeate valves	CLOSED								
Transfer from	Transfer rate	No set point	N.A.	gpm		<u> 1995 - Johann Steffenson, fransk forsk</u>				
UFP-VSL-T02A (Test Instructions	UFP-VSL-T02A Operation									
step 7.1.17)	Level to stop PJM operation	15	$-5/+5$	in						
	Level to stop steam ring air purge	10	$-5/+0$	in						
	Level to turn off air sparge	13	$-5/+5$	in						
	Ultrafiltration Loop Operation									
	Number of filter	5	$-0/+0$							

Table J.1. Integrated Test A Operational Process Sheet

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