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PEP Integrated Test D Run Report— Caustic and Oxidative Leaching in UFP-VSL-T02A

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December 2009



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Work Authorization: WA# 2007-024
Test Plan: TP-RPP-WTP-506, Rev. 0.3; TP-WTP-PEP-044, Rev 0.1
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Test Scoping Statement(s): NA

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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:



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

12/11/09
Date

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BP Crume	K Miller	AD Woodstock
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RL Daubert	KE Parker	ST Yokuda
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Definitions and Acronyms

AFA	antifoam agent
APEL	Advanced Process Engineering Laboratory
ASO	Analytical Support Operations
ASR	Analytical Services Request
ASME	American Society of Mechanical Engineers
BNI	Bechtel National Inc.
BS	blank spike
CD	Coriolis densitometer
CFD	computational fluid dynamics
CFR	Code of Federal Regulations
CUF	Cell Unit Filter
CXP	cesium ion exchange process system
DAS	Data Acquisition System
DIW	deionized water
DOE	U.S. Department of Energy
EFRT	External Flowsheet Review Team
FEP	Feed Evaporator Process (vessel)
FRP	Feed Receipt Process (vessel)
GPC	gel permeation chromatography
HDI	“How Do I...?”
HLP	HLW lag storage and feed blending process system
HLW	high-level waste
HMI	human-machine interface
IC	ion chromatography
ICP	inductively coupled plasma
ICP-AES	inductively coupled plasma-atomic emission spectrometry
ICP-MS	inductively coupled plasma-mass spectroscopy
IW	inhibited water (0.01-M NaOH)
JTG	Joint Test Group
LCS	laboratory control sample
LTE	lead test engineer
LRB	laboratory record book
M&TE	measuring and test equipment
MS	matrix spike
NCR	nonconformance report

PDMS	polydimethylsiloxane
PB	preparation blank
PDL-W	Process Development Laboratory-West
PEP	Pretreatment Engineering Platform
P&ID	pipng and instrumentation diagram
PJM	pulse-jet mixer
PLM	polarized light microscopy
PNNL	Pacific Northwest National Laboratory
PPG	polypropylene glycol
PSD	particle-size distribution
PTF	Pretreatment Facility
QA	quality assurance
QAM	Quality Assurance Manual
QAP	Quality Assurance Plan
QARD	Quality Assurance Requirements and Descriptions
QC	Quality Control
Re	Reynolds number
R&T	research and technology
RPP	River Protection Project
RTD	resistance temperature detector
SBMS	Standards Based Management System
SEM	scanning electron microscopy
SLM	stable level measurement
SwRI	Southwest Research Institute
SPG	specific gravity
TAT	turn around time
TDS	total dissolved solids
TIC	total inorganic carbon
TOC	total organic carbon
THF	tetrahydrofuran
TMP	transmembrane pressure
UDS	undissolved solids
UFP	ultrafiltration process
VFD	variable frequency drive
VSL	vessel
wt%	weight percent
WTP	Hanford Tank Waste Treatment and Immobilization Plant
XRD	X-ray diffraction

Testing Summary

Pacific Northwest National Laboratory (PNNL) has been 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” of the External Flowsheet Review Team (EFRT) issue response plan.^(a) The PEP is a $1/4.5$ -scale test platform designed to simulate the WTP pretreatment caustic leaching, oxidative leaching, ultrafiltration solids concentration, and slurry washing processes.^(b) 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.

Two process flowsheets are currently being evaluated for the ultrafiltration process (UFP) and leaching operations. The baseline flowsheet (Integrated Test A) 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 (Integrated Tests B and D) has caustic leaching performed in the UFP-2 ultrafiltration feed vessels (i.e., vessel UFP-VSL-T02A^(c) in the PEP and vessels UFP-VSL-00002A and B [UFP-2] in the WTP PTF).

In Integrated Test D, 19-M sodium hydroxide (NaOH, caustic) was added to the waste slurry in the UFP-VSL-T02A vessel after the solids were concentrated to ~20% undissolved solids (UDS). The NaOH was added to leach solid aluminum compounds (e.g., gibbsite, boehmite). Caustic addition was followed by heating to 85°C using direct injection of steam to accelerate the leach process. The main difference of Integrated Test D compared to Integrated Test B is that the leach temperature was 85°C for 24 hrs as compared to 100°C for 12 hours. The other difference is the Integrated Test D simulant had Cr in the simulant from the start of processing and Integrated Test B had Cr added to adjust the simulant composition after aluminum leaching. Following the caustic leach, the UFP-VSL-T02A vessel contents were cooled using the vessel cooling jacket. The slurry was then concentrated to 17-wt% UDS and washed with inhibited water to remove NaOH and other soluble salts. Next, the slurry was oxidatively leached using sodium permanganate to solubilize chromium. The slurry was then washed to remove the dissolved chromium and concentrated.

This is one in a series of reports that summarize Phase 1 results from testing in the PEP located in the Process Development Laboratory-West (PDL-W) located in Richland, Washington. Separate run reports have been prepared for each Phase 1 test (i.e., Functional Testing [WTP-RPT-190], Integrated Tests A

-
- (a) 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.
 - (b) The scale of $1/4.5$ 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.
 - (c) 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).

[WTP-RPT-191], Integrated Test B [WTP-RPT-192], and Integrated Test D [WTP-RPT-193]). In addition, reports have been written that specifically address:

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 focuses on Integrated Test D and summarizes test conduct and results. It includes only very limited data analysis. Integrated Test D was conducted under Test Plan TP-RPP-WTP-506^(a) and in accordance with Test Instruction TI-WTP-PEP-082. Supporting data files and analytical reports have been supplied on transportable hard drives because of the large amount of data.

Objectives

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 D to meeting these objectives.

(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.

Table S.1. Summary of Test Objectives and Results

Test Objective	Objective Met?	Discussion
Caustic-leach process: Compare engineering- and laboratory-scale results to determine impact of scale-up.	Yes	Data from Integrated Test D were analyzed and compared with laboratory-scale data, with results presented in report WTP-RPT-197.
Oxidative-leach process: Compare engineering- and laboratory-scale results to determine impact of scale-up.	Yes	Data from Integrated Test D were analyzed and compared with laboratory-scale data, with results presented in report WTP-RPT-197.
Cross-flow Ultrafiltration: Monitor cross-flow filter performance at engineering- and laboratory-scale to determine scale-up.	NA	Data from Integrated Test D were analyzed and compared with laboratory-scale data, with results presented in report WTP-RPT-197.
Slurry wash process: Determine the post-caustic and oxidative leaching slurry wash efficiencies.	Yes	Data from Integrated Test D were used to determine washing efficiencies for several components after caustic and oxidative leaches, and the results are presented in WTP-RPT-197.
Process Integration: Evaluate the chemical addition, filter operation 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.	Yes	Supporting data from Integrated Test D are presented in this report, and results to meet this objective are discussed in WTP-RPT-197.
Monitor the performance of the recirculation system pumps, filters, and heat exchanger to support Engineering fabrication decisions for these components.	Yes	The data required to meet this objective were provided on Compact Discs transmitted in the letter from GH Beeman to H Hazen, "Subcontract No. 24590-QL-HC9-WA49-00001, Project No. 53569 (WA-024) Engineering Ties Data Transmittal: The 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.

Test Exceptions

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

Table S.2 Test Exceptions

Test Exceptions	Description of Test Exceptions
1) 24590-PTF-TEF-RT-08-00002, incorporated into ICN-1 to Test Plan TP-RPP-WTP-506.	<p>This test exception:</p> <ol style="list-style-type: none"> 1. Added a stage during the filter conditioning section of the Shakedown/functional Test where the simulant slurry was 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. 2. Documented the Joint Test Group (JTG) decision regarding the number of replicate samples to be collected at various processing times. 3. Revised the terminology specifying the Coriolis densitometer (CD) sample locations changed to be consistent with PEP operating procedures. Renamed the “center” array to “inner.” 4. The sampling specified in the low-solids filtration test over-specifies the 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-09-00001 incorporated into ICN-2 and ICN-3 to Test Plan TP-RPP-WTP-506.	<ol style="list-style-type: none"> 1. In several steps, the sampling location was changed from the filter-loop in-line location to a middle-low CD sample loop location in the UFP-T02A vessel. This change impacted sampling in the Shakedown/Functional and all Integrated tests (ref CCN 187749). 2. Added a step to the Shakedown/Functional Test (step A.1.31) to add sodium permanganate to UFP-VSL-T02A to assess possible foaming issue (ref CCN 187749). 3. Changed location of second sample for laboratory-scale Cells Unit 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). 4. Collected samples for laboratory-scale laboratory leaching test before and after caustic 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 (B1.2; Integrated Test A, B2.6; Integrated Tests B/D) (ref CCN 192734). 5. Deleted reconfiguration of the filter-loop to bypass UFP-VSL-T02A and circulate flush water with UFP-PMP-T02A and/or UFP-PMP-43A 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). 6. Eliminated step A.1.25 (filter-loop bypass test with tracer) from the Shakedown/Functional Test. This test was conducted after Integrated Test B was completed (ref CCN 187753). 7. Modified step A.1.29 (Shakedown/Functional Test) to eliminate the removal of solids from UFP-VSL-T02A before the High-Solids Filter Test. This step was not needed as the amount of solids was less than anticipated (ref CCN 187752). 8. Modified step A.1.30 (Shakedown/Functional Test) to include five filter backpulses before starting the High-Solids Filter Test (ref CCN 187752). 9. Modified step B.1.8 (Integrated Test A) to allow 80% of caustic to be added

Table S.2 Test Exceptions

Test Exceptions	Description of Test Exceptions
	<p>during in-line simulant transfers to UFP-VSL-T01B and 20% to be added directly to UFP-VSL-T01B (ref CCN 187748).</p> <ol style="list-style-type: none"> 10. Added a High-Solids Filter Test to the end of Integrated Test B to replace the High-Solids Filter Test from the simulant Shakedown/Functional Test. The test conducted during the Shakedown/Functional Test was hampered by pump cavitation, and the target solids concentration was not met (ref CCN 192734). 11. Eliminated Integrated Test C from the Test Plan (ref CCN 192735). 12. The requirement to record density using the CDs on the samplers in UFP-VSL-T02A was eliminated. The density function was not usable because of entrained air in the simulant. 13. Modified step B.2.6 (caustic addition in Integrated Test B/D) temperature limit to change from 60°C to “as specified in run sheet.” This temperature is calculated based on various other run parameters as specified in the run sheet. 14. Eliminated the monitoring of Integrated Test D permeate samples for 30 days to look for precipitation. This scope was deleted, and a revised scope was incorporated into the Test Plan (TP-WTP-PEP-044; Test Plan for PEP laboratory-scale laboratory testing). 15. Step B2.20 (Integrated Tests B and D) sampling of the heel in UFP-VSL-T01A was deleted. This sample was not needed since the heels were removed before follow-on testing. 16. Step B1.26 (Integrated Test A) sampling of heel in UFP-VSL-T01B was deleted. This sample was not needed since the heels were removed before follow-on testing. 17. Steps B1.25 (Integrated Test A) and B2.19 (Integrated Test B/D) were modified from the following: “transfer slurry from UFP-VSL-T02A to HLP-VSL-T27” to “transfer slurry from UFP-VSL-T02A to UFP-VSL-62A/B or to totes for storage as directed by the WTP test director.” The HLP-VSL-T27 vessel was no longer available for use since it served as the receipt vessel for the filter-loop pressure safety valves. 18. Added a second batch of leaching to Integrated Tests B/D in UFP-VSL-T02A. This additional leaching batch was needed to provide a sufficient quantity of solids to operate the UVP-VSL-T02A at prototypic levels for the steps following caustic leaching. 19. Added a filter bypass tracer test following the post-caustic-leach dewatering step in Integrated Test B. This test replaced the filter bypass tracer test that could not be conducted during the simulant Shakedown/Functional testing. 20. Deleted instructions to route permeate to a specific tank (i.e., UFP-VSL-T62A/B). There was no need to segregate various permeate streams. 21. Minor changes were made to make the Test Plan consistent with the approved run sheets.
<p>3) 24590-WTP-TEF-RT-09-00003 incorporated into ICN-1 to Test Plan TP-WTP-PEP-044.</p>	<p>This Test Exception specified activities to be performed with permeate samples obtained from Integrated Test D. The Integrated Test D permeate samples were originally stored in a temperature-controlled environment and then moved to a location with a reduced temperature where precipitation was likely to occur. The Test Exception requested that the approximate size distribution of the solids be measured in several (three or four) selected PEP samples from Integrated Test D using polarized light microscopy (PLM). Size-calibrated photographs should be provided along with the analysis. If possible, record the mineral identification of the solids phase(s) along with the particle-size distribution. WTP personnel will</p>

Table S.2 Test Exceptions

Test Exceptions	Description of Test Exceptions
	<p>select the samples in consultation with the subcontractor, and this will be based in part on observing which samples contain the most solids or appear to contain different types of solids. Repeat the size-distribution analysis approximately 1 week after the initial measurements to determine whether there was a significant change in crystal size, habit, or composition.</p> <p>Perform each size-distribution analysis by measuring the diameter (or length and width for elongated crystals) of approximately 100 individual particles in each sample. The size may be measured either on the microscope slide, using a calibrated ocular scale, or on the size-calibrated photographs. The program recognizes the limitations of the statistical significance of a size-distribution measurement based on such a small population. This Test Exception did not affect any of the existing Test Plan objectives.</p>
<p>4) 24590-WTP-TEF-RT-09-00002 Rev 0, incorporated into ICN-4 to Test Plan TP-RPP-WTP-506.</p>	<p>This Test Exception:</p> <ol style="list-style-type: none"> 1. Requests a report summarizing the lessons learned during scale-up, manufacture, and transport of the PEP simulant. 2. Specifies the sampling and analysis scope to be performed to complete the prototypic nitric acid PEP filter cleaning process. 3. Deletes the Engineering Ties report scope. 4. Specifies additional experimental and analytical work required to estimate the amount of excess caustic in caustic-leachate samples and post-caustic-leach wash solutions containing $\approx 3.5\text{-M Na}$.
<p>5) 24590-WTP-TEF-RT-09-00001 Rev 1 incorporated into ICN-2 to Test Plan TP-WTP-PEP-044.</p>	<p>This Test Exception specifies additional work to be conducted with caustic-leach solutions and post-caustic-leach washing permeate samples obtained from PEP Integrated Tests A, B, and D. It contains the following tasks:</p> <ol style="list-style-type: none"> 1. Determine precipitate mineralogy, precipitate phase compositions, and solution saturation composition. 2. Determine rate of approach to saturation concentrations. 3. Identify and characterize precipitates formed in post-caustic-leach filtrate. 4. Determine the dilution required to redissolve the precipitate. 5. Determine super-saturation in post-caustic-leach filtrates from Integrated Test B in the PEP. 6. Determine the effects of blending during the post-caustic-leach dewatering and wash cycle.

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

Table S.3. Deviations from Test Specification

Test Specification Reference	Exception Taken
<p>Section 6.4.4 “Analytical measurements will be made in conformance to the <i>Guidelines for Performing Chemical Physical, and Rheological Properties Measurements</i> (BNI 2002-3)^(a) as applicable.”</p>	<p>Three method exceptions are required under this Test Plan:</p> <ol style="list-style-type: none"> 1. Caustic-leach and oxidative-leach samples taken during this testing must be separated more quickly than the standard method using syringes. This testing will use a modified method with a shorter centrifuge time and will apply higher <i>g</i> forces (e.g., 4000 <i>g</i> vs. 1000 <i>g</i>). <i>Impact on results:</i> If the standard method were used, the longer time could very well lead to greater precipitation and inaccurate results. Laboratory testing will be conducted with simulants to confirm that this method of sample handling is adequate. 2. The 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. <i>Impact on results:</i> 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% UDS content of the slurries will in some cases be determined with 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. <i>Impact on results:</i> Both modifications are intended to decrease the time required to obtain results.

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 WTP computer process models projections of the waste processing campaign.
- Confirm the operability and functionality of UFP system components.

The 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

(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.

the success criteria covered by the scope of this report. The success criteria not addressed in this report are shaded in gray.

Table S.4. Success Criteria

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-2 projected leaching conditions at low process temperatures of nominally 100 and 80°C).	Aluminum leaching at 85°C was measured as a function of time under WTP UFP-2 projected leaching conditions during Integrated Test D. Results are presented in this report and in report 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 total NaOH is added directly to the tank.	Results to meet this success criterion are discussed in report WTP-RPT-197.
Measure chromium leaching performance in the PEP and laboratory systems as a function of time at the WTP projected conditions in UFP-2.	Chromium leaching was measured as a function of time under WTP UFP-2 projected leaching conditions during Integrated Test D. Results are presented in report WTP-RPT-197.
Evaluate the process control strategy for specifying 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. Results are presented in report WTP-RPT-197. 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 transmembrane pressures for the solids concentration and washing stages for the UFP-1 and UFP-2 aluminum leaching flowsheets.	Filter performance was measured at a nominal axial flow velocity of 15 ft/sec and a transmembrane pressure (TMP) of 40 psid during solids concentration and washing during Integrated Test D. Actual permeate flux and filtration conditions are provided in this report. A summary of filter system performance is provided in WTP-RPT-197.
Evaluate the control strategy for make-up additions from UFP-VSL-00001A/B to UFP-VSL-00002A/B during initial dewatering process.	The control strategy for make-up additions between UFP-VSL-T01A and UFP-VSL-T02A during the initial dewatering process was demonstrated during initial dewatering during Integrated Test D. 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 are presented 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.	Washing performance during Integrated Test D is presented in report WTP-RPT-197.

Table S.4. Success Criteria

Success Criteria	How Testing Did or Did Not Meet Success Criteria
Perform mass balances for selected constituents, including aluminum, chromium, manganese, sodium, hydroxide, oxalate, phosphate, sulfate, and water to evaluate leaching and washing process performance.	Results necessary for performing mass balances for selected constituents for Integrated Test D are presented in this report. Results to meet this success criterion are discussed for Cr in the oxidative leaching process for Integrated Tests A and B in report WTP-RPT-188 and are fully discussed for all tests and constituents in report WTP-RPT-197.
Measure solids distribution under scaled mixing conditions before and after caustic leaching evolutions.	Solids distribution before and after caustic leaching is presented in reports WTP-RPT-190 and WTP-RPT-197.
Measure the rheology of the slurry simulant and shear strength of the settled solids before and after each leaching and washing unit operation and following final concentration.	The rheology of the slurry simulant was measured for Integrated Test D and is provided in this report.
Estimate the quantity of excess hydroxide added in the process that may not be needed to keep aluminate in solution following filtration.	Results to meet this success criterion are expected to be discussed in report WTP-RPT-197.
Collect and retain permeate samples for extended precipitation studies (including permeate/simulated supernatant blended cases) from each concentration cycle.	Permeate samples were collected during Integrated Test D for precipitation studies. The results of the precipitation studies are discussed in WTP-RPT-197, WTP-RPT-200 and WTP-RPT-205.
UFP System Operability and Functionality	
Verify that the dual, in-series pump configuration is controllable and maintains the required slurry velocity and pressures for ultrafilter operation.	Pumps were operated in-series and were controllable. There were some air entrainment issues associated with the PEP system design that need to be evaluated for the WTP. The data required to meet this success criterion were provided on compact disks transmitted in the following reference: Letter from GH Beeman to H Hazen, "Subcontract No. 24590-QL-HC9-WA49-00001, Project No. 53569 (WA-024) Engineering Ties Data Transmittal: The 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.
Measure the operating characteristics for the cooling heat exchanger for the UFP-VSL-00002 filter recirculation loop (temperature changes as a function of flow to determine how to achieve the desired performance in the PTF analog).	The data required to meet this success criterion were provided on compact disks transmitted in the following reference: Letter from GH Beeman to H Hazen, "Subcontract No. 24590-QL-HC9-WA49-00001, Project No. 53569 (WA-024) Engineering Ties Data Transmittal: The 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.
Confirm whether the WTP process control strategies for ultrafilter system filling, 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	Results to meet this success criterion are expected to be discussed in report WTP-RPT-197.

Table S.4. Success Criteria

Success Criteria	How Testing Did or Did Not Meet Success Criteria
of restoring the filter permeate rates to ensure production throughput is maintained and whether operation of the backpulse system induces any process or equipment operations issues.	
Use only the process information and data available to the WTP PTF operating staff during WTP operations (e.g., caustic and permanganate addition volumes, permeate mass balances for solids concentration, etc.) to operate the PEP.	This success criterion was met by developing a run sheet of all the operating parameters (e.g., transfer volumes, reagent addition volumes, control levels, etc.) based on prototypic characterization data before the start of Integrated Test D. Changes to the run sheet made during the test itself were based only on data that would be available to the plant, and were not, for example, based on information from non-prototypic samples. This success criterion is fully discussed in WTP-RPT-197.
Confirm whether the elevated temperature PJM operating strategy is adequate for stable PEP and WTP operation.	Results to meet this success criterion are discussed in report WTP-RPT-197.
Measure the heat-up rate and controllability of the PEP UFP-VSL-00001 and UFP-VSL-00002 vessels and the cooling performance for UFP vessels.	The heat up, thermal control at 85°C, and cool-down performance of UFP-VSL-T02A was measured during Integrated Test D. Thermal profiles are provided in this report. This information for the other tests is discussed in the other run reports and summarized in WTP-RPT-197.
Measure the performance of the in-line addition of process chemicals into the simulated wastes and determine the extent of blending in the process vessels.	The blending of process chemicals added in-line is presented in report WTP-RPT-190 and WTP-RPT-197.
Monitor ultrafilter performance (to include visual inspection of the filter tubes, tube sheets, and heads from an ultrafilter for any evidence of flow mal-distribution and/or solids buildup at least once during Phase 1).	Ultrafilter performance was measured and the tubes were inspected after the completion of Integrated Test D. The inspections showed no evidence of flow mal-distribution, solids build-up, or corrosion.
Measure, record, and control ultrafiltration temperature, transmembrane pressure, and slurry flow during filter-loop operations.	Slurry flow rate, temperature, and TMP were recorded and controlled during Integrated Test D. Results are provided in this report.
Record any solids accumulations observed during any operating stage or maintenance evolution.	Results to meet this success criterion are discussed in report WTP-RPT-197.
Monitor the permeate production rate of each ultrafilter assembly in operation.	Permeate production rate of each ultrafilter was recorded during Integrated Test D. Results are provided in this report.
Record operating time of each ultrafilter assembly.	The operating time of each ultrafilter assembly was recorded during Integrated Test D. 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 WTP-RPT-197.
Record each ultrafilter assembly cleaning event (backpulse, flush, chemical cleaning, etc.).	At the completion of Integrated Test D the ultrafilters were cleaned with nitric acid and the results are provided in this report.

Table S.4. Success Criteria

Success Criteria	How Testing Did or Did Not Meet Success Criteria
Evaluation of the pulse-pot operation and backpulse operation strategies contained in Pretreatment Engineering Platform (PEP) Phase 1 Testing Process Description.	Significant backpulsing was not performed in Integrated Test D. Evaluation of backpulse operations is discussed in report WTP-RPT-197.
Evaluate permeate and permeate blends for precipitation of solids, particularly aluminum and oxalate solids.	Permeate samples were collected during Integrated Test D for precipitation studies. Results to meet this success criterion are discussed in reports WTP-RPT-197, WTP-RPT-200 and WTP-RPT-205.

Quality Requirements

The PNNL Quality Assurance 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.

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)*.

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

Following this procedure, a technical review verified 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.

Table S.5. R&T Test Conditions

List R&T Test Conditions	Were Test Conditions Followed?
General Requirements	
Perform mass balances for selected constituents, including aluminum, chromium, manganese, sodium, hydroxide, oxalate, phosphate, sulfate, and water, to evaluate leaching and washing process performance.	Necessary samples were taken to conduct a mass balance for Integrated Test B. Mass balance results are provided in WTP-RPT-197.
Evaluate ultrafilter performance (to include visual inspection of the filter tubes, tube sheets, and heads from an ultrafilter for any evidence of flow mal-distribution and/or solids buildup or evidence of potential failure).	Ultrafilter performance was measured, and the tubes were inspected after completing Integrated Test D. The inspections showed no evidence of flow mal-distribution, solids build-up, or corrosion. This R&T test condition is also discussed in report WTP-RPT-197.
Assess the blending achieved during in-line additions of leaching and washing solutions.	In-line addition of wash water during Integrated Tests D and B is discussed in WTP-RPT-187 and is fully discussed in report WTP-RPT-197.
Record any solids accumulations observed during any operating stage or maintenance evolution (e.g., photography, particle-size distribution).	This R&T test condition is discussed in report WTP-RPT-197.
Leaching Operations	
Maintain the caustic leaching temperature at the required setpoint and record the steam usage to remain in the temperature range.	The temperature during caustic leaching was maintained at setpoint with steam usage measured during Integrated Test D. Additional discussion of this condition is provided in WTP-RPT-197.
Maintain oxidative leaching temperature at the required setpoint.	The temperature during oxidative leaching was maintained at setpoint during Integrated Test D. Additional discussion of this condition is provided in WTP-RPT-197.
Obtain periodic samples during the leaching operations to monitor the amount of aluminum or chromium that has dissolved and concentrations of the reactants and products in the liquid fraction in the vessel.	This R&T condition was met for the caustic leaching tests discussed in this report. Additional discussion of this R&T condition is provided in WTP-RPT-188 and WTP-RPT-197.
Provide data to demonstrate the WTP process control strategy for the caustic and permanganate addition.	This R&T test condition is discussed in report WTP-RPT-197.
Measure the rheology of the slurry simulant and shear strength of the settled solids before and following each leaching unit operation.	This R&T test condition is discussed in report WTP-RPT-197.

Table S.5. R&T Test Conditions

List R&T Test Conditions	Were Test Conditions Followed?
Concentration Operations	
Monitor the permeate production rate of each ultrafilter assembly in operation.	The slurry rheology was measured with samples taken before and after each leaching unit operation. Results are provided in this report. Rheology results 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 B, with results discussed in report WTP-RPT-197.
Record each ultrafilter assembly “cleaning” event (backpulse, flush, chemical cleaning, etc.).	Ultrafilter cleaning events were recorded with results of the cleaning after Integrated Test D provided in this report. A summary of the filter cleaning events is provided in WTP-RPT-197.
Confirm pulse-pot operation and backpulse operation strategies.	This R&T test condition is discussed in report WTP-RPT-197.
Control ultrafiltration temperature, transmembrane pressure, and slurry flow as specified in test-specific run sheets.	Ultrafilter temperature, transmembrane pressure, and slurry flow rate were controlled. This R&T test condition is discussed in the run reports for the individual tests and WTP-RPT-197.
Collect and retain permeate samples for extended precipitation studies (including permeate/simulated supernatant blended cases) from each concentration cycle.	Samples were collected and retained for extended precipitation studies. The results of the precipitation studies are discussed in WTP-RPT-197, WTP-RPT-200, and WTP-RPT-205.
Demonstrate WTP ultrafiltration system control scheme in normal operating modes (e.g., fill and startup, operation, backpulsing, flush and drain, cleaning and return to service).	This R&T test condition is discussed in report WTP-RPT-197.
Washing Operations	
Wash slurries using a washing protocol to be specified in test-specific run sheets.	Slurries were washed as specified in the Integrated Test B run sheet. Washing results are discussed in reports WTP-RPT-187 and WTP-RPT-197.
Sample permeate immediately before each wash solution addition to monitor washing performance/efficiency.	Permeate was sampled and monitored during washing with results provided in report WTP-RPT-197.
Measure rheology of the washed solids.	The rheology of the 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 in WTP-RPT-197.

Simulant Use

The PEP testing program was conducted using 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 I 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 (Russell et al. 2009c).

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 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.

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

Discrepancies and Follow-on Tests

No discrepancies or follow-on tests were identified.

1.0 Introduction

This is one in a series of reports that summarize Phase 1 testing results from the Pretreatment Engineering Platform (PEP) located in the Process Development Laboratory-West (PDL-W) located in Richland, Washington. Separate run reports have been prepared for each Phase 1 test (i.e., Shakedown/Functional Testing [Josephson et al. 2009], Integrated Test A [Guzman-Leong 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 (Daniel et al. 2009)
2. Caustic Leaching (Mahoney et al. 2009)
3. Washing (Baldwin et al. 2009)
4. Oxidative Leaching (Rapko et al. 2009).

This report focuses on Integrated Test D and summarizes test conduct and results. It includes only very limited data analysis. Data files have been supplied in an electronic format because of the large amount of data. The test reported here was conducted according to TI-WTP-PEP-082, which was written in accordance with Test Plan TP-RPP-WTP-506, Rev. 0.4, which was written in response to Test Specification 24590-PTF-TSP-RT-07-001, Rev 2.

1.1 Purpose

The purpose of the M-12 testing program is to provide WTP with data to “...confirm the ultrafiltration process (UFP) system design and sludge treatment process flowsheet.”^(a) The WTP system design and treatment process flowsheet will be confirmed by evaluating the following operations on an engineering-scale integrated platform (the PEP):

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

Tests will be used to collect data needed for design input and to improve the 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.

(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.

1.2 Background

In October 2005, an External Flowsheet Review Team (EFRT) was assembled to provide a critical review of the design of the Hanford Tank Waste Treatment and Immobilization Plant (WTP). The review^(a) identified a number of issues and potential issues that if not addressed could impact the operation of the plant. 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 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 Bechtel National Inc. (BNI) and the U.S. Department of Energy (DOE) developed the “Issue Response Plan for Implementation of External Flowsheet Review Team (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 processes, and filtration scenarios.

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 (Barnes and Voke 2006, p. 5).
- Phase 2—Confirm the UFP system performance over a range of anticipated plant process conditions, including the principle 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 10/23/06 and 10/24/06, a WTP Project ultrafiltration leaching process technical review meeting was held to review the Issue Response Plan for M12.^(c) Participants included members of the DOE, EFRT, the WTP project, and Pacific Northwest National Laboratory (PNNL). At the meeting, PNNL made recommendations on the methodology and scale for the leaching processes.

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

-
- (a) JP Henschel. 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) 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.

- 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 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 Pretreatment Facility.
- Each ultrafilter assembly should contain 12 full-scale elements.
- Five ultrafilter assemblies should be provided (based on M13 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 $^{1/4.5}$ times their full-scale radius, and the operating height $^{1/4.5}$ that of full-scale. The tank volume will be $^{1/(4.5)^3}$ or $\sim^{1/90^{\text{th}}}$ of the plant.
- Filtration design is scaled by filter area. The total filtration area is $^{1/(4.5)^2}$ or $\sim^{1/20^{\text{th}}}$ that of the plant.
- 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 purposes 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 the *Pretreatment Engineering Platform (PEP) Testing (Phase I) Test Specification*^(a) identifies the requirements for engineering-scale tests. 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 Objectives

The objective of this report is to present the results of Integrated Test D 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 Requirements

The PNNL quality assurance (QA) program is based upon the requirements as defined in DOE Order 414.1C, *Quality Assurance* and 10 CFR 830, *Energy/Nuclear Safety Management*, Subpart A—*Quality Assurance Requirements* (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 through PNNL’s “How Do I...?” (HDI).^(a) PNNL implements the RPP-WTP quality requirements by performing work in accordance with the *River Protection Project—Waste Treatment Plant Support Program (RPP-WTP) Quality Assurance Plan* (RPP-WTP-QA-001, QAP). Work was performed to the quality requirements of NQA-1-1989, Part I, *Basic and Supplementary Requirements*, NQA-2a-1990, Part 2.7, and DOE/RW-0333P, Rev 13, *Quality Assurance Requirements and Descriptions (QARD)* 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.

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.

Key analytes in the laboratory control sample (LCS) and PEP control sample were plotted over time to look for anomalies. The PEP control sample is a project-provided material generated from material very similar to the initial simulant feed. In general, the plots constructed to date associated with the inductively coupled plasma (ICP) and ion chromatography (IC) analysis of solutions shows recovery within limits of 80% to 120%.

Table 2.1 shows list of nonconformance reports (NCRs) active during Integrated Test D.

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

Table 2.1. Description of NCRs Active During Integrated Test D

Nonconformance Report Number	Description
NCR 38767.1	<p>Measurement: Sparger flow rates were below the 0.5% accuracy range during Integrated Test D, although test results overall are not affected.</p> <p>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 ~4%.</p> <p>The following instruments were affected: FT-1973, FT-1977, FT-1981, FT-1901, and FT-1995.</p> <p>What it means: Recognize the greater uncertainty that accompanies data with air flow rates <0.090 kg/min.</p>
NCR 43398.1	<p>Measurement: TTK-427 one of several temperature measurements in vessel T01B.</p> <p>TTK-0427 failed at installation. All data from TTK-0427 are invalid and unusable.</p> <p>What it means: The sensor failed, but the test was not impacted since many other sensors exist in Vessel T01B.</p>
NCR 42402.1	<p>Measurement: Temperature measurement of the slurry in the filter-loop.</p> <p>RTD thermowells in the filter-loop do not extend into the process stream.</p> <p>TT-0513: HX-T02A (cooling) slurry outlet temperature. No data are to be used for quality-affecting work. Data may be used for qualitative purposes only.</p> <p>TT-0515: HX-T03A (heating) slurry outlet temperature. No data are to be used for quality-affecting work. Data may be used for qualitative purposes only.</p> <p>TT-0537: Filter #5 outlet temperature (HX02A inlet). No data are to be used for quality-affecting work. Data may be used for qualitative purposes only.</p> <p>TT-0791: Filter #1 inlet temperature. Data from this should not be used at all. Tank temperature data will be used for quality-affecting filter processing data.</p>
NCR 41589.1	<p>Measurement: On-line density measurement in T01B Coriolis sample station.</p> <p>Issue: DT-2101 failed. All readings after 12/23/2008 are not valid.</p> <p>What it means: All readings after 12/23/2008 are not valid. Actual sample density results should be used instead.</p>

3.0 Experimental Methods and Analysis

3.1 Pretreatment Engineering Platform Overview

The following section provides an overview of the PEP to orient the reader. This section does not provide details on PEP equipment or instrumentation. The interested reader is referred to the following documents for more details on the PEP:

- P&IDs and mechanical data books.
- Pretreatment Engineering Platform (PEP) Phase I Testing Process Description, 24590-WTP-RPT-PET-07-002, Rev 1.
- Functional Requirements for Pretreatment Engineering Platform (PEP), 24590-PTF-3YD-UFP-00002, Rev 1.

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, solids washing, chemical reagent addition and mixing, waste slurry heating and cooling, and waste chemical leaching. Figure 3.1 presents a simplified process diagram showing the vessels, pumps, heat exchangers, and filter systems associated with the PEP. Equipment that has been considered critical for evaluating the integrated system performance has been scaled to be prototypic. Specifically, the ultrafiltration feed preparation and feed vessels have been scaled to be geometrically similar to WTP with a $^{1/4.5}$ scale; the working height and diameter are scaled to $^{1/4.5}$. Pipe sizes are scaled to have approximately $^{1/4.5}$ the diameter, but the fluid velocity is to be approximately the same as the full-scale plant.

Waste simulant feed can be provided from three primary sources: High Level Waste (HLW) Feed Receipt Vessel (HLP-VSL-T22), Feed Receipt Process (FRP) Vessels (FRP-VSL-T01), or Feed Evaporator Process (FEP) Vessel (FEP-VSL-T01). Feed is received into the Ultrafiltration Feed Preparation Vessels UFP-VSL-T01A/B (henceforth Tank T01A and Tank T01B). In Integrated Test D, feed from Tanks T01A/B was used to provide simulant feed to UFP-VSL-T02A (henceforth Tank T02A) during simulant concentration. Simulant concentration, caustic leaching, oxidative leaching, and washing were conducted in Tank T02A and the associated filter-loop. The concentrated solids were transferred into totes while the liquid fraction (permeate) was stored in permeate collection vessels (UFP-VSL-T62A/B). Wash and leach solutions were sent to UFP-VSL-T62A/B.

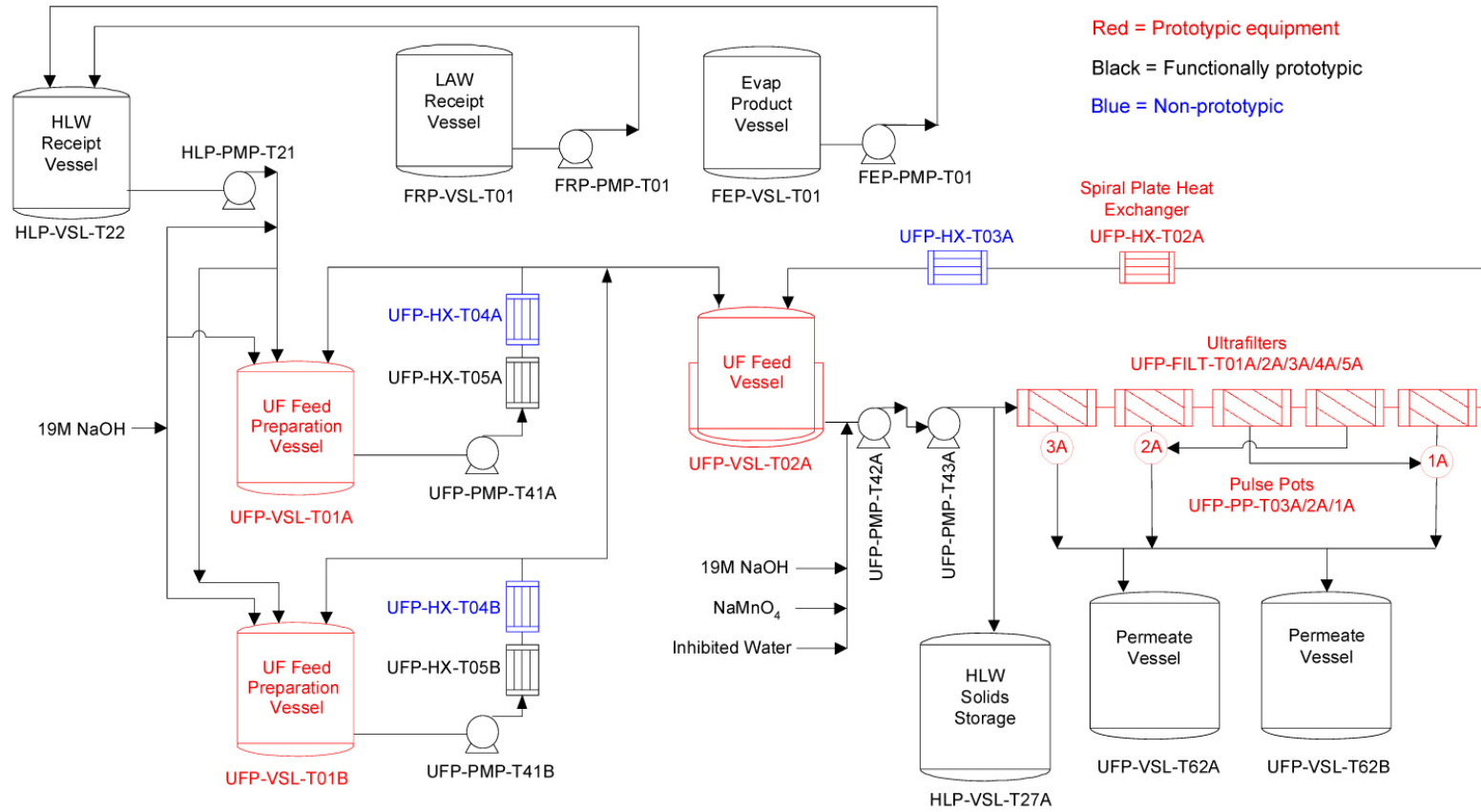


Figure 3.1. PEP Simplified Flow Diagram

3.2 PEP Filtration System

The PEP filtration system is composed of an ultrafiltration feed tank, Tank T02A, a slurry circulation and filtration loop, a permeate metering and collection system, and a filter backpulse and cleaning system. A simplified schematic of the filtration system is shown in Figure 3.2. The PEP filtration system was configured to measure the feed flow rate, temperatures, and axial and transmembrane pressure (TMP) for each filter. In addition, the system is configurable such that filters 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 filters 2 through 5. A summary of process instrumentation is provided in Appendix A. In the following paragraphs, key process equipment for slurry filtration operations are identified and discussed. Interested readers are referred to the documents listed in Section 3.1 if more information is needed.

3.2.1 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 using an array of six PJMs and the filter-loop return jet. Ancillary systems for Tank T02A include 1) air spargers, which aid in mixing and also limit inflammable gas hold-up in the actual waste treatment system, 2) bubblers to measure slurry density and level, 3) laser level sensors, and 4) an array of resistance temperature detectors (RTDs) to measure the tank temperature profile. Specific locations of the RTD arrays for Tanks T02A and T01A are available in laboratory record book (LRB) 59944, pages 12–17. Tank T02A is equipped with a water jacket to cool and a steam ring to heat the contained slurry.

3.2.2 Slurry Filtration Circulation Loop

The filtration loop contains process equipment key to slurry dewatering and washing operations. Two centrifugal slurry pumps, UFP-PMP-T42A and UFP-PMP-T43A (hereafter referred to as Pump T42A and Pump T43A, respectively), are operated in-series to provide the required slurry flow rate and pressure for the cross-flow filters. The suction to Pump T42A is fed from Tank T02A. In addition, the feed to Pump T42A is connected to process inhibited water^(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 filters. Pumps T42A and T43A are identical pumps rated at 150 gpm and 243 psi.

The cross-flow filter system is the core of slurry liquid-solid separations. It is composed of five filters connected in-series. These filters are designated as UFP-FILT-T01A to -T05A (hereafter referred to as Filters 1 through 5). The filter-loop is equipped with slurry bypass valves to allow slurry flow through Filter 1 and/or Filters 2 through 5.

(a) Inhibited water is 0.01-M NaOH.

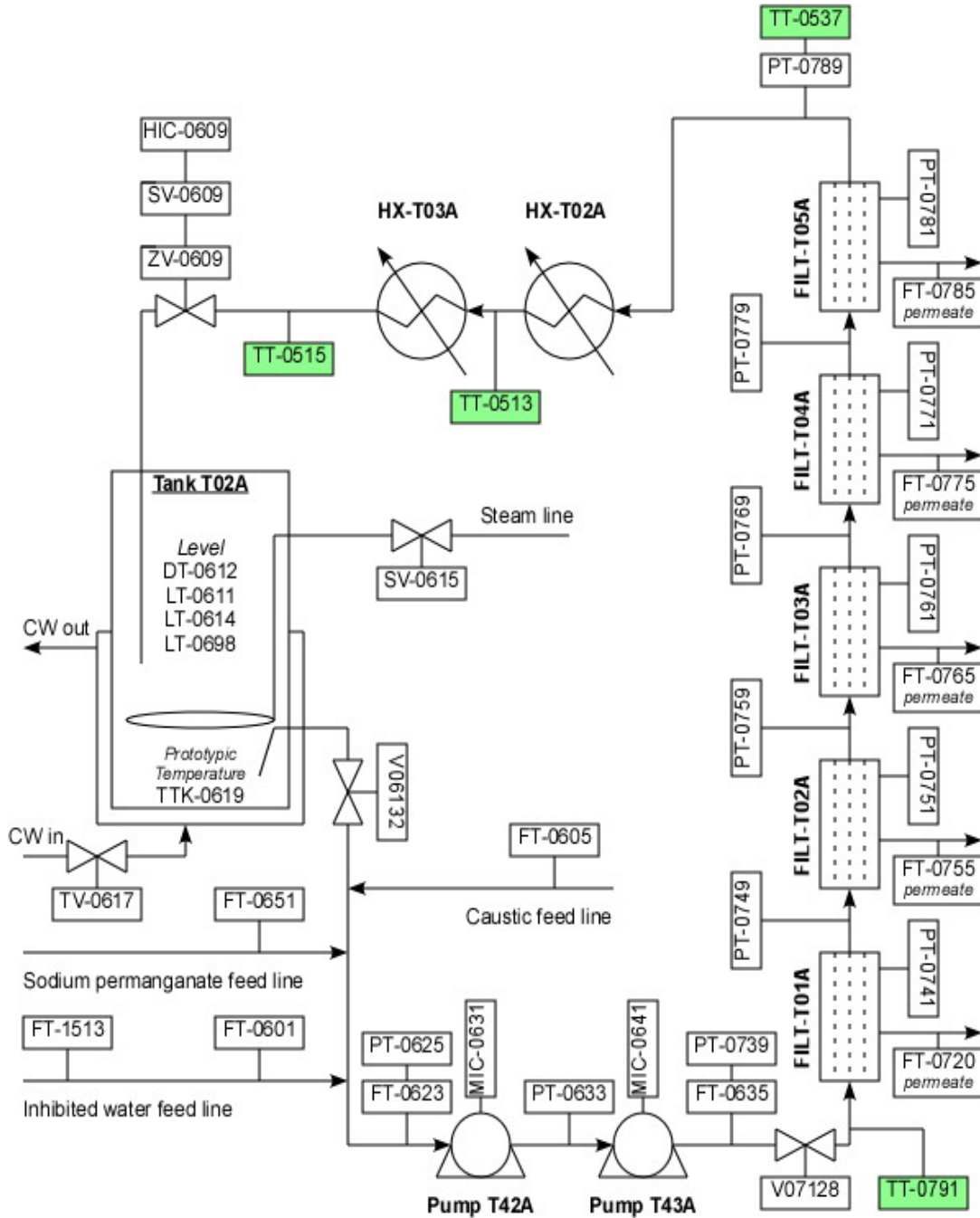


Figure 3.2. Schematic of Filtration Loop

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- μm . 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 or 120 inches. A summary of the geometries of the five filters is provided in Table 3.1.

The tubeside slurry flow rate and pressure are monitored by a series of flow meters and pressure transducers. As shown in Figure 3.2, 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 controlled with two in-line heat exchangers. The first heat exchanger, UFP-HX-T02A, is a (prototypic) spiral plate heat exchanger that uses chilled water to cool the circulating slurry. The second heat exchanger, UFP-HX-T03A, is a (nonprototypic) steam exchanger used 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 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 (ZV-0609) that can be adjusted in combination with the slurry pumps to provide adequate backpressure for permeate production. The filter-loop in-line sampler is located immediately downstream of ZV-0609. After passing through ZV-0609 and past the in-line sampler, the circulating slurry is recycled back into Tank T02A.

(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). The impact for testing was that during Integrated Test B, the temperature control for the cooling heat exchanger in the filter-loop, UFP-HX-T02A, was with a hybrid control method: The cooling water control valve (TV-0513) being adjusted (in automatic) to control the outlet slurry temperature TT-0513 to a targeted setpoint. However, the temperature setpoint was changed manually by an operator to try to maintain the temperature setting in Tank T02A..

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

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

3.2.2.1 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, three pulse-pots connected to high-pressure air supplies for backpulsing the filters, and permeate collection tanks.

Permeate (shellside) mass production rates from Filters 1 through 5 are monitored by Coriolis flow meters. Permeate flow from each of the filters is directed to three pulse-pots (designated as UFP-PP-T01A to UFP-PP-T03A). Pulse-pot UFP-PP-T03A serves Filter 1, pulse-pot UFP-PP-T02A serves Filters 2 and 4, and pulse-pot UFP-PP-T01A serves Filters 3 and 5. The pulse-pots are filled with a sufficient volume of collected permeate to backpulse the filters. Overflow from the pulse-pots may be directed to 1) permeate or process slurry collection tanks (UFP-VSL-T62A/B 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.

Table 3.2. Permeate Metering and Pulse-Pot Configurations for PEP

Filter Bundle No. / ID	Permeate Coriolis Meter	Associated Pulse 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

During backpulsing, one of the pulse-pots is isolated, drained to a specific level, and charged with high-pressure air until the pulse-pot pressure exceeds the slurry pressure at the filter inlet by a given amount (typically 40 psid). After the target pulse-pot pressure is reached, a large, fast-acting valve isolating the pulse-pot from the filter is opened, and a fraction of the liquid in the pulse-pot (typically less than 1 gal) flows back to the filter bundle, forcing permeate from the shellside to the tubeside of the filter

tubes. The backflow of permeate forces loosens any particles 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 Appendix B.

3.3 PJM Operational Modes

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 operating modes have the same basic cycle: Drive Phase, Vent Phase, and Vacuum Phase, followed by a Hold (or Vent) Phase. PJM mixing technology involves a pulse tube coupled with a jet nozzle. One 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 creates four operating phases for the pulse tube: 1) the Drive Phase, when pressure is applied to discharge the contents of the PJM tube through the nozzle, 2) the Vent Phase, when the pressure is vented to the atmosphere, 3) the Refill Phase, when vacuum is applied to refill the pulse tube (at temperatures above 60°C, the vacuum is disabled, and the pulse tubes refill by gravity.), and 4) the Hold Phase, when all valves to the PJMs are closed, causing the fluid level in each PJM to remain constant until the next Drive Phase. The PJM system uses these operating phases to produce a sequence of drive cycles that provide mixing in the vessel.

A timer set to the overall Cycle Time starts at the beginning of each Drive Phase. A new Drive Phase begins when either the cycle timer runs out, or the cycle enters the final Hold/Vent Phase, whichever is later. This condition exists to make sure the Vacuum Phase has completed before moving on to the next cycle.

PJM operations at PEP consisted of one of several control modes: standard mode (regular), standard mode (star pattern—Tank T02A only), standard mode (short cycle), and simple mode. However, PJM controls at PEP were not prototypic. During Integrated Test B, the standard mode (regular) was used whenever possible and the standard mode (star pattern) was used when the level was low, typically <23 inches. If the standard mode was nonfunctional (e.g., during caustic leaching, the PJM Drexelbrook level probes ceased functioning), the PJMs were operated in simple mode. For all tanks, if the PJMs were operating in standard mode, the short cycle function would automatically activate when the tank level dropped to 12 inches and would remain active until the tank level rose above 22 inches for Tank T01A and Tank T01B. The short cycle activation occurred automatically in Tank T02A when the level dropped to 11.5 inches and remained until the level returned to 16 inches.

3.3.1 Standard Mode (Regular) Description

Standard mode operations in PEP controls each PJM individually based on that PJM level instrument. The PJMs have one overall cycle time, which keeps all PJMs in synch at the beginning of each cycle. Each cycle is controlled by five main variables: Cycle Time, Drive Time Set Point, Vent Time Set Point, L1 Level, and L2 Level. L1 and L2 Levels are user-definable upper and lower level set points used by the control software to indicate an overblow. Additionally, each PJM could be enabled or disabled individually.

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

The Drive Phase is controlled by the time entered as the Drive Time Set Point (corrected for temperature if enabled). Each PJM begins the Drive Phase at the same instant, but can have different lengths of time specified for driving each PJM. If any PJM does not finish within 30 seconds of the PJM with the shortest drive time, that PJM is disabled, and the cycle continues to the next phase.

The Vent Phase is controlled by the Vent Time Set Point, which has no correction factors. Each PJM begins the Vent Phase at the same instant and can have different lengths of time specified for each PJM. If any PJM does not finish within 30 seconds of the PJM with the shortest vent time, that PJM will be disabled, and the cycle will 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 is controlled by the L1 Level, which is set at the top of the desired PJM stroke. All PJMs begin the Vacuum Phase at the same instant, but each PJM L1 Level is set separately. If any PJM does not reach the L1 Level within 30 seconds of the first PJM to reach its L1 Level, that PJM is disabled, and the cycle continues to the next phase. During high temperature ($>60^{\circ}\text{C}$) operations, the vacuum was disabled, and each PJM tube was gravity filled up to its L1 Level. If the tank level was less than 44 inches (T01A/B) or 46 inches (T02A), then the PJM would switch operation to Standard Mode--Short Cycle until either the temperature dropped below the 60°C threshold, or the level rose above the 44/46-in. threshold.

The final phase in standard mode is a Hold Phase. All valves to the PJM are closed, causing the fluid level in each PJM to remain constant at the L1 Level.

The standard mode operation also contains a temperature linearization variable that reduces the drive time to prevent overblows due to the difference in slurry properties as the temperature increases. The variable is expressed as a slope, the percent of drive time decrease per degree Celsius increase from zero degrees Celsius. This function was largely untested. A modest 0.25% linearization factor was used in early testing and adopted for the remainder of testing simply because it seemed to work, and further optimization was not conducted.

3.3.2 Standard Mode—Short Cycle

A Short Cycle consists of a Drive Phase in which each enabled PJM drives for 20% of the Drive Time Set Point followed by a 10-sec Vent Phase. The Short Cycle function would automatically activate when the tank level dropped to 12 inches and would remain active until the tank level rose above 22 inches for Tank T01A and Tank T01B. The Short Cycle activation occurred automatically in Tank T02A when the level dropped to 11.5 inches and remained until the level returned to 16 inches. Additionally, the Short Cycle would activate during high-temperature operations ($>60^{\circ}\text{C}$) if the tank level fell below 44 inches (T01A/B) or 46 inches (T02A) to confirm that there was enough fluid to gravity fill above the L1 Level for each PJM because the vacuum was disabled at high temperatures.

3.3.3 Standard Mode—Star Pattern

Tank T02A additionally can operate in standard mode—star pattern. The star pattern option follows the same rules as the regular standard PJM mode. The difference is that only two PJMs are enabled at a given time. When the operator sets the star pattern button on the human machine interface (HMI), the center PJM and one other PJM will be enabled for five cycles. Then the outer PJM will be disabled, and a different outer PJM will be enabled for another five cycles. The pattern of enabling and disabling the five outer PJMs forms a five point star. The disabled PJMs are vented.

The star pattern was developed by PEP test engineers in an effort to reduce the level fluctuations in Tank T02A and operate the PJMs in a manner that could be implemented in the PTF. During Integrated Test B, the star pattern was enabled when the level in Tank T02A was approximately 23 inches or when the filter-loop recirculation target could not be achieved. Enabling the star pattern increased the achievable filter-loop recirculation rate because the star pattern reduced the tank level fluctuations because fewer PJMs were filling and discharging in this mode.

3.3.4 Simple PJM Mode Description

As mentioned above, the simple mode operates substantially the same as the standard mode. Each cycle consists of Drive, Vent, Vacuum, and Hold/Vent phases. There are two main differences. The first difference is that all PJMs operate together as one unit. There are no individual levels used or individual enabling or disabling of PJMs. The second difference is that each phase is controlled by a time (instead of level). An operator sets the Cycle Time, Drive Time Set Point (uncorrected), Vent Time Set Point, and Vacuum Time Set Point (uncorrected). Additionally, the operator can choose whether the last phase holds the PJM level or vents (returning PJM level to the same as the tank level).

During Integrated Test B, the simple mode was used when the standard mode was not working. The standard mode did not work during leaching operations because the PJM level probe ceased to function at elevated temperatures in high-caustic solutions.

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 $(10,000 \text{ milliseconds}) * \{1.00 - [0.01/\text{inch} * (46-36 \text{ inches})]\} = 9000 \text{ milliseconds}$. The operator determines the tank level used in the calculation 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 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 $(10000 \text{ milliseconds}) * \{1.00 + [0.01/\text{inch} * (46-36 \text{ inches})]\} = 11000 \text{ milliseconds}$.

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 the 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 to travel all the way up into the hoses on top of the tank. Similarly, if the Vacuum Time was too low, the PJM fill level could decrease over successive cycles, eventually resulting in an overflow. If the Hold Phase was disabled, each PJM just vented to the tank level during the remainder of the PJM cycle.

3.4 Testing Overview

Nitric acid cleaning of the filters was performed before Integrated Test B but was not repeated before Integrated Test D. The filters were cleaned using nitric acid after Integrated Test D was complete. The LRBs used during testing to record testing activities were BNW-60272 and BNW-60280.

The following is a summary level overview of testing reported herein. Figure 3.3 shows a graphical schedule of events. Refer to the test narrative (Section 4), Test Instruction TI-WTP-PEP-082, and the LRBs listed above for additional details.

1. Transfer simulant from HLP-VSL-T22 to UFP-VSL-T01B.
2. Transfer simulant from UFP-VSL-T01B to UFP-VSL-T02A and concentrate solids to 20-wt% using one filter, UFP-FILT-T01A.
3. Maintain caustic-leach concentrated simulant at 85°C for 24 hours (Batch 1).
4. Transfer Batch 1 leached simulant to UFP-VSL-T01A for temporary storage.
5. Transfer simulant from HLP-VSL-T22 to UFP-VSL-T01B.
6. Transfer simulant from UFP-VSL-T01B to UFP-VSL-T02A and concentrate solids to 20-wt% using all five filters.
7. Maintain caustic-leach concentrated simulant at 85°C for 24 hours (Batch 2).
8. Concentrate leached solids (Batch 1 and 2) using all five filters.
9. Wash leached slurry with IW and remove permeate using all five filters.
10. Add permanganate and conduct oxidative-leach on slurry for 6 hours at 25°C.
11. Wash oxidative leached slurry using IW and all five filters and then concentrate.
12. Flush filter-loop with IW.
13. Drain system and clean filters with 2M nitric acid.

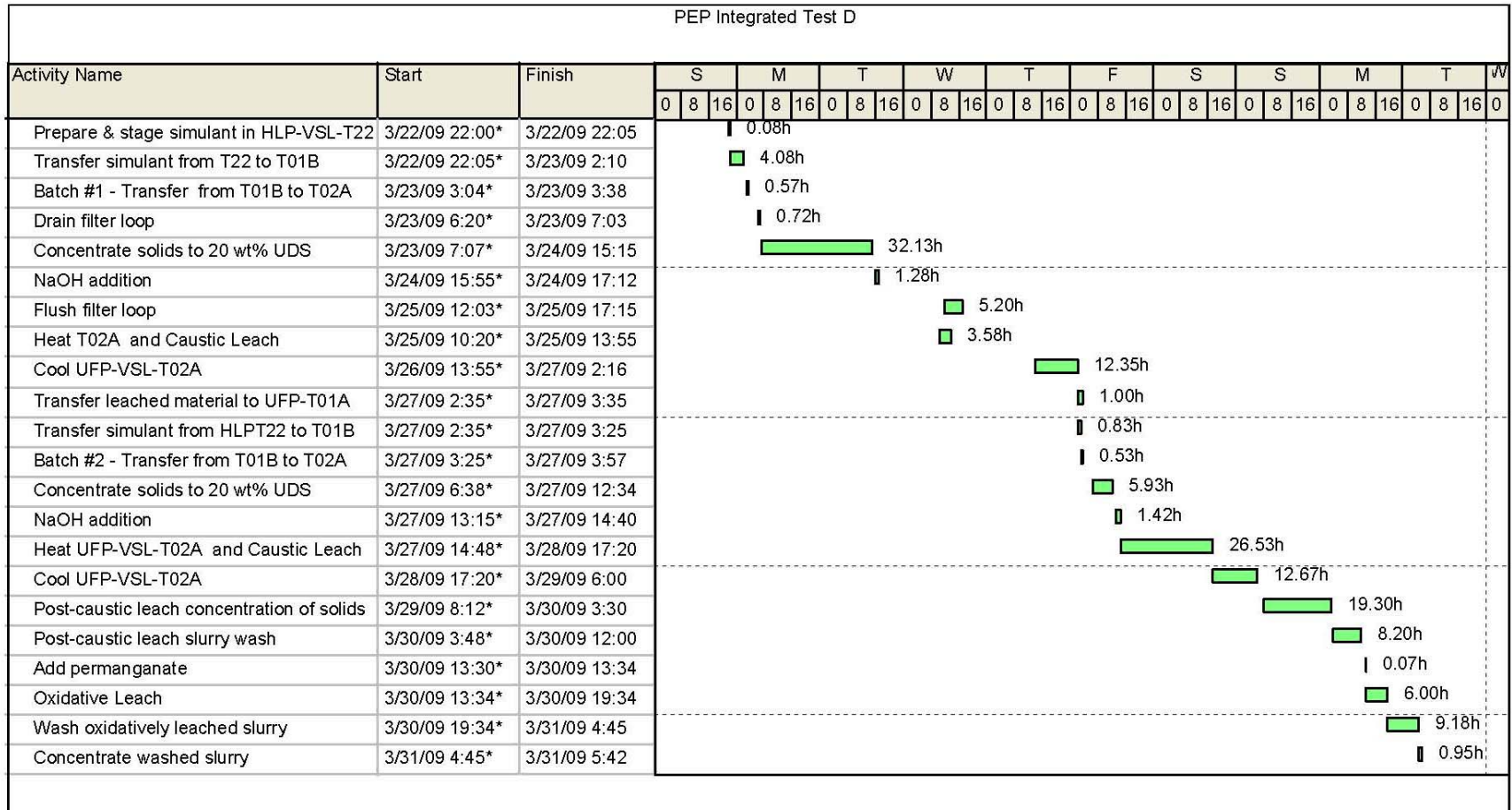


Figure 3.3. Integrated Test D Timeline

3.5 Simulant

The PEP process testing was performed with nonradioactive aqueous slurry of simulant waste solids and liquids. 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 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, 2008)
- Sodium oxalate
- Filtration simulant ((Russell et al. 2009c)
- Supernate.

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 certain 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 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.

(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, Washington.

- 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.

3.6 Sampling

A list of samples taken and their purpose is provided in Appendix C. Actual sample times were recorded in the Test Instruction.

In general, slurry samples were collected using either an 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 3.4. To obtain a sample, the downstream valve was fully opened, and then the upstream valve was opened sufficiently to allow samples to be safely obtained. The sample line and valves were purged with at least three line volumes before a sample was collected. The vent and the pressure gauge shown are for a different sampling method that would allow samples to be collected from the volume between the two main processing valves while being isolated from the process flow. This method was not implemented in Phase 1 testing.

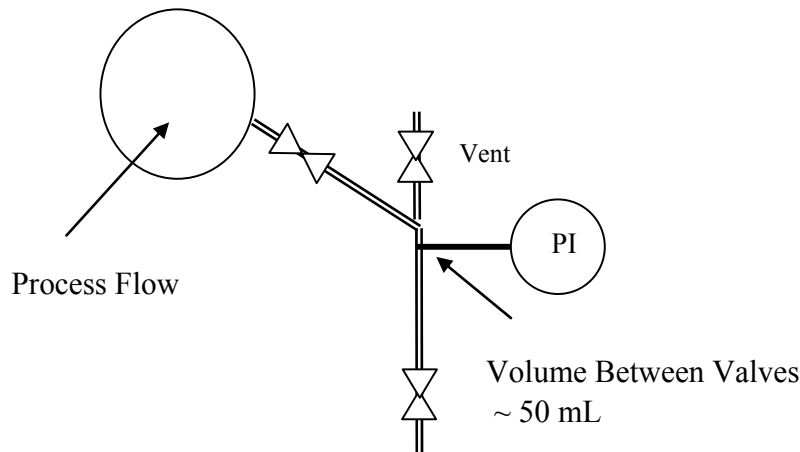


Figure 3.4. Simple In-Line Sampler

Figure 3.5 is a schematic of the in-tank sampling system for UFP-VSL-T01A/B and UFP-VSL-T02A. 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 line inlet 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.

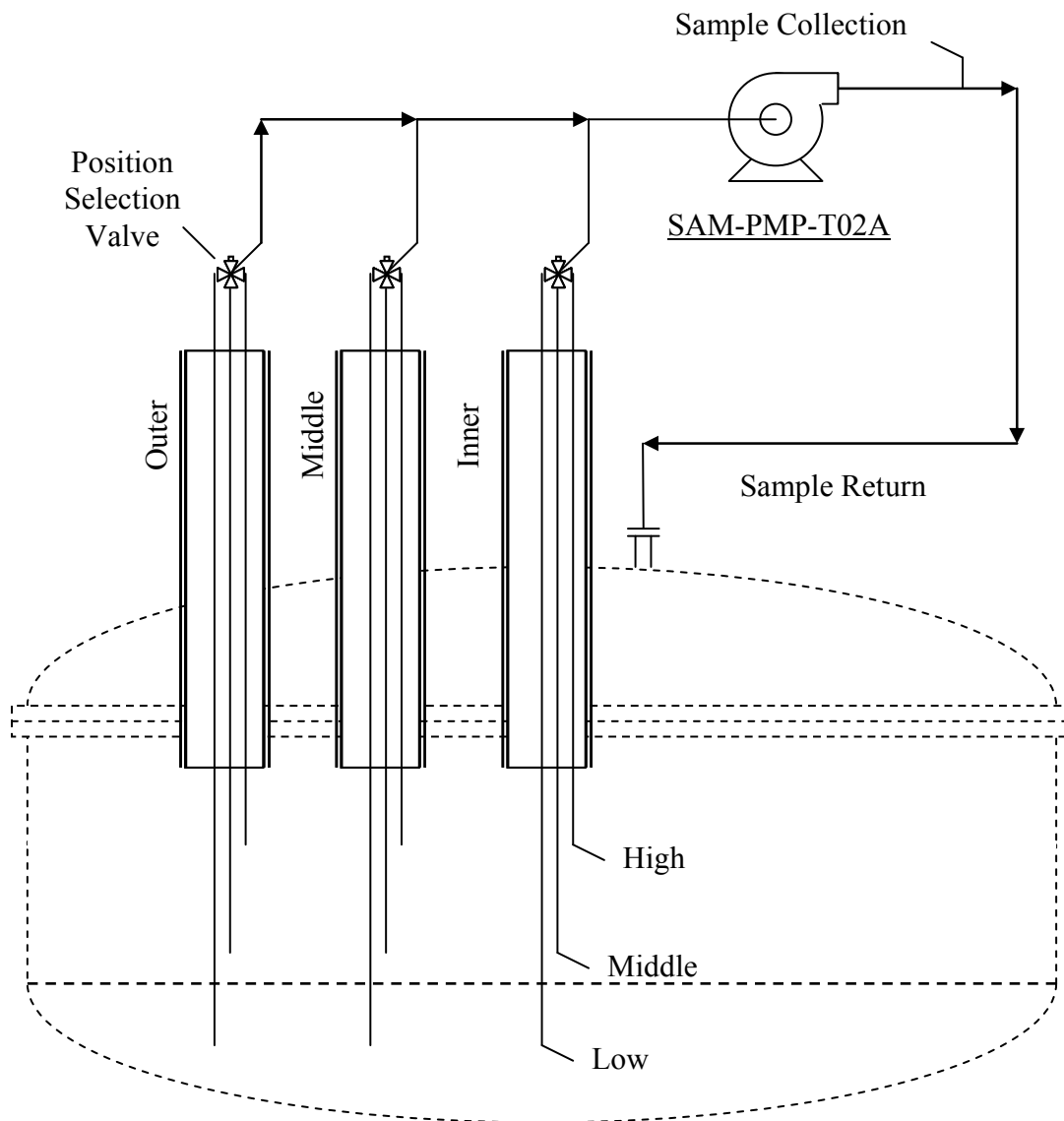


Figure 3.5. In-Tank Sampling Showing the Three Radial Positions at Three Heights and Sampling Flow Loop

3.7 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 3.6 through Figure 3.8 for samples collected and processed in PDL-W. 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.

3.7.1 Non-Leaching Samples

During non-leaching steps, sample processing was not time sensitive, and several samples were shipped as 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 (2×1 -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-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 wt% 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 wt% UDS method described in Bechtel procedure, *Guidelines for Performing Chemical, Physical, and Rheological Properties Measurements* (24590-WTP-GPG-RTD-001 Rev 0.)^(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.

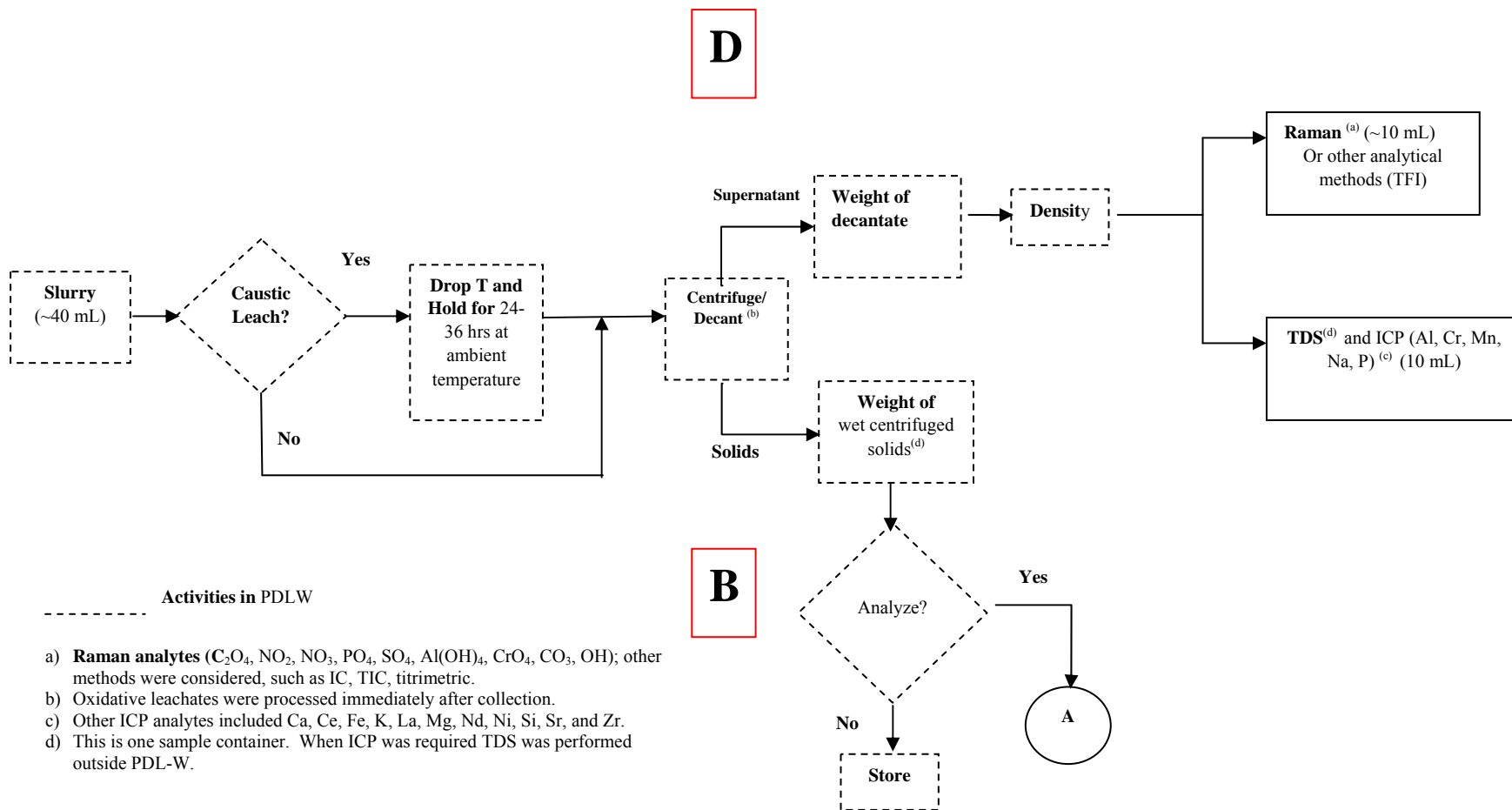
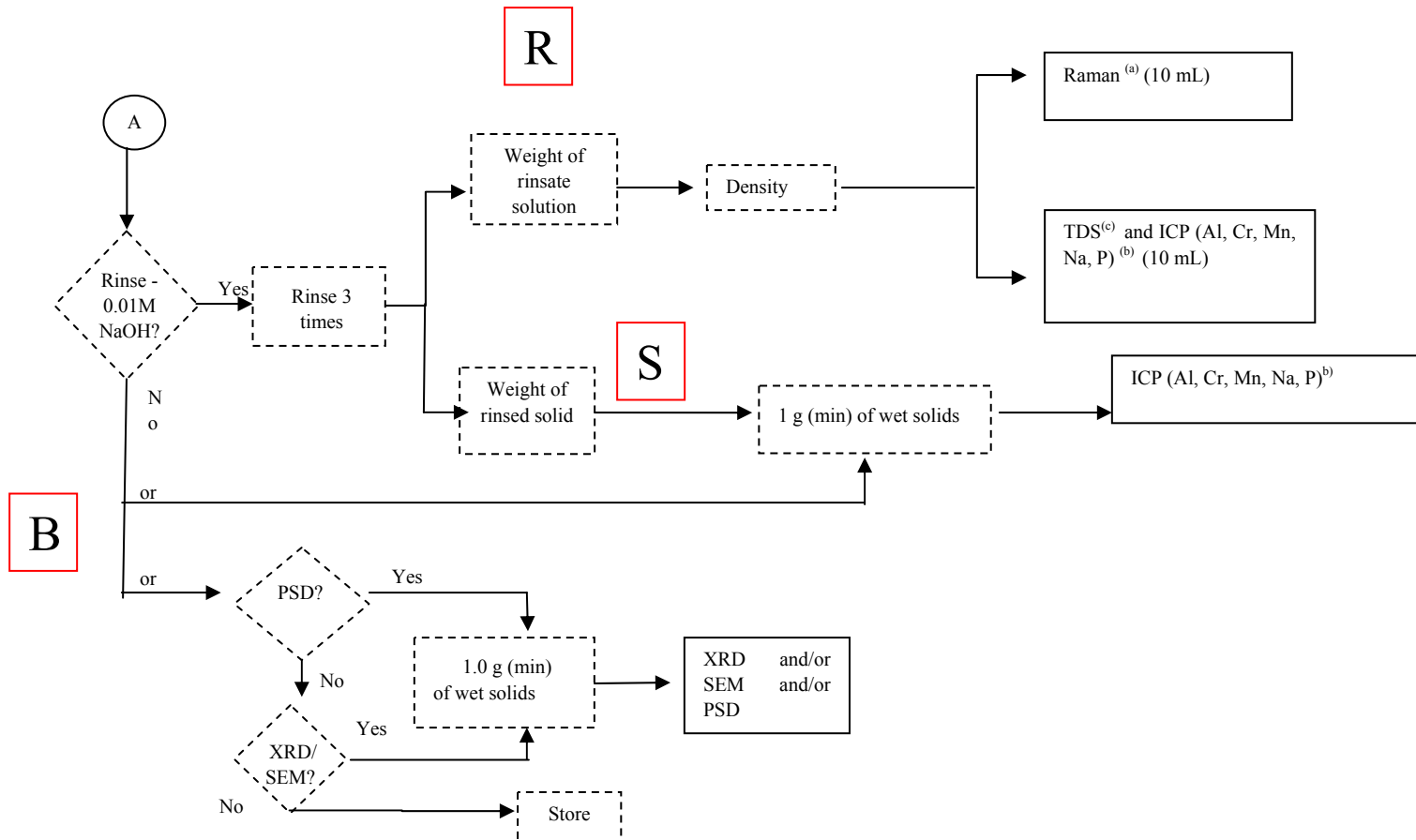


Figure 3.6. Sample Processing for Decantate Handling



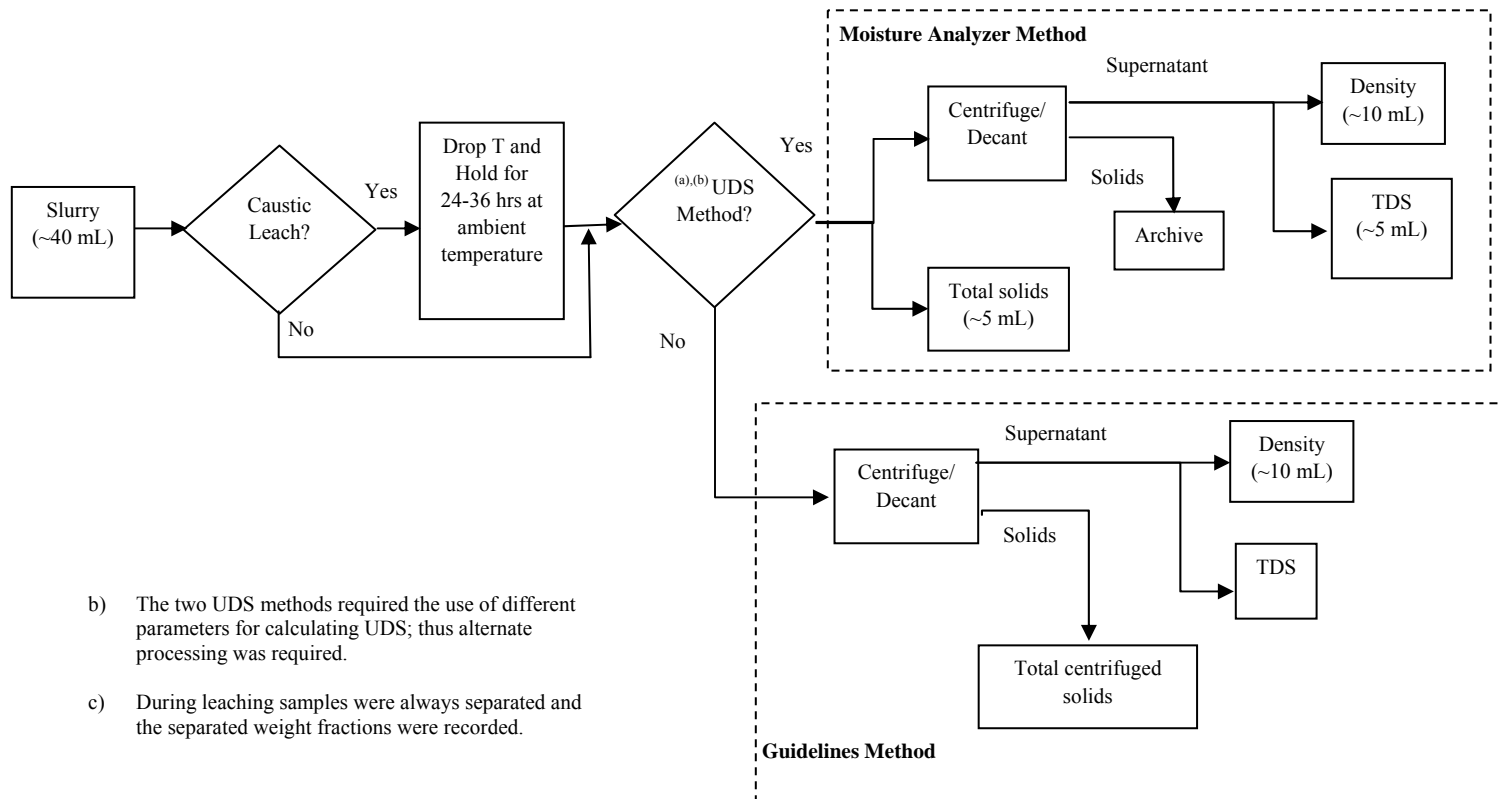
----- Activities in PDLW

(a) Raman analytes (C_2O_4 , NO_2 , NO_3 , PO_4 , SO_4 , $Al(OH)_4$, CrO_4 , CO_3 , OH); other methods were considered, such as IC, TIC, titrimetric.

(b) Other ICP analytes included Ca, Ce, Fe, K, La, Mg, Nd, Ni, Si, Sr, and Zr.

(c) This is one sample container. When ICP was required TDS was performed outside PDL-W.

Figure 3.7. Sample Processing for Solids



- b) The two UDS methods required the use of different parameters for calculating UDS; thus alternate processing was required.
- c) During leaching samples were always separated and the separated weight fractions were recorded.

Figure 3.8. 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. However, during some of the Integrated Test B and Integrated Test D process steps, phase separation was not achieved after 10 minutes, so the samples were centrifuged for an hour. The actual centrifuging time is recorded on the sample bench sheets. After centrifuging, the supernatant was decanted and submitted for some or all of the analyses listed below.

- inductively coupled plasma (ICP)
- total dissolved solids
- density
- ion chromatography (IC)
- total organic carbon (TOC)
- Raman
- free hydroxide.

Not all of these analyses were necessarily performed on every decanted supernatant. The specific 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, the density was determined by 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 antifoam agent (AFA) compounds by 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), XRD, and 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. 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 the Advanced Process Engineering Laboratory (APEL) by the next business day after sample collection.

3.7.2 Leaching Samples

The core critical analyses for leached samples were wt% 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 $20^{\circ} \pm 2^{\circ}\text{C}$ to decrease the sample temperature to $25 \pm 5^{\circ}\text{C}$ 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 NaMnO_4 were phase-separated immediately after collection. The oxidatively leached centrifuged solids from samples collected immediately before adding NaMnO_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 sub-samples 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.

3.7.2.1 Undissolved Solids

The sample processing for UDS is given in Figure 3.8. 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) L 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.

3.7.2.2 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.

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

3.8 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.

3.8.1 Preparation for ICP or ICP-MS

For the solutions, aliquots were diluted with hydrochloric acid and then analyzed. For the dilutions, a representative sample aliquot of approximately 2 grams was combined with 5-mL of 50% hydrochloric acid and then diluted to a final volume of 50-mL with deionized water (DIW).

For solids, two preparative techniques were used. 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.

3.8.2 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).

3.8.3 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).

3.8.4 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, process blank, LCS, and matrix spike (MS).

3.8.5 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 SwRI procedure 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, process blank, LCS, and MS.

3.8.6 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, process blank, and blank spike (BS).

3.8.7 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, process blank, BS, and MS.

3.8.8 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 then 3.0-mL toluene was added. 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- μ m PTFE syringe filters. Calibration verification was performed before sample analysis and after every 15 samples.

3.8.9 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- μ m guard column and a PLgel 5- μ m 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- μ m guard column and a PLgel 5- μ m 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°C. The quantity of PDMS and PPG was used to determine the amount of AFA, Dow Corning Q2-3183A Antifoam, in the samples. Since the AFA is a proprietary composition belonging to Dow Corning, they performed the analytical work. Calibration verification was performed before sample analysis and after every 15 samples.

3.9 Physical Properties

This section describes the methods used to determine the physical properties of the PEP simulant samples, the crystal form, and the 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.

3.9.1 Percent UDS and Density

Weight percent UDS, wt% dissolved solids, bulk slurry density, and supernatant density were determined following Bechtel procedure 24590-WTP-GPG-RTD-001, Rev 0.^(a) Some Bechtel 24590-WTP-GPG-RTD-001, Rev 0 steps 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 ~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.
- 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 pre-weighed 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 ~1000 gravities for 1 hour. Samples were centrifuged at ~4500 gravities for either 10 minutes or 1 hour. The centrifuge time is documented on the sample handling bench sheets 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 handling bench sheets 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 bench sheets. 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.

3.9.2 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.

(a) GL Smith and K Prindiville. May 20, 2002. *Guidelines for Performing Chemical, Physical, and Rheological Properties Measurements*. 24590-WTP-GPG-RTD-001, Rev 0, Bechtel National, Inc., Richland, Washington.

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

3.9.3 Rheology Measurements

Rheological testing was conducted on the solids in contact with the supernatant generated as part of the homogenization process. Testing was conducted according to 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 are considered: 1) incipient motion in settled tank waste solids (shear strength) and 2) non-elastic flow of tank waste slurries and supernates (flow curve).

3.9.3.1 Shear-Strength Testing

For tank waste slurries, 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 with operating constraints shown in Table 3.3. 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):

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

Here, τ_{ss} is the shear strength (N/m^2), M_{max} is the maximum torque ($\text{N}\cdot\text{m}$), 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 3.3. Vane Immersion Depth and Container Geometry Constraints for Shear-Strength Tests Using the Vane Technique

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

R = radius
H = height

3.9.3.2 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 shows 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,

$$\tau = \frac{M}{2\pi HR_l^2} \quad (3.2)$$

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 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,

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

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 (~1 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 B.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:

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

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 cP = 1 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

$$\tau = \eta \dot{\gamma} \quad (3.5)$$

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,

$$\tau = m \dot{\gamma}^n \quad (3.6)$$

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

$$\tau = \tau_o^B + k_B \dot{\gamma} \quad (3.7)$$

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,

$$(\tau)^{0.5} = (\tau_o^C)^{0.5} + (k_C \dot{\gamma})^{0.5} \quad (3.8)$$

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.

3.9.3.3 Rheology Instrumentation

Rheological characterization was accomplished with an Anton Parr Rheometer (MCR 301) for shear-rate measurements, and shear strength was determined on a HAAKE RS-600 Measuring System 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 × 16 mm (R × 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 3.4.

Table 3.4. Vane and Cup and Rotor Measuring System Dimensions

Measuring System	Vane/Rotor Radius (mm)	Vane/Rotor Height (mm)	Container Radius (mm)	Gap Width (mm)
Vane Tool	8	16	>16	>8
MV1	20.04	60	21	0.96

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 temperatures.

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.

3.9.3.4 Rheology Materials and Methods

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

3.9.3.5 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 8 × 16 mm (R to H) vane tool to a depth of 15 mm into the settled solids. The vane was slowly rotated at 0.3 rpm for 180 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.

3.9.3.6 Flow-Curve Testing

Each flow curve was measured over an 11-min 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 L.

3.9.4 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- μm . 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 3.5.

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.

Table 3.5. Properties Applied to Group 8 Test Materials

Property	
Material Selected for Optical Properties	Ferric Oxide Hydroxide
Refractive Index (RI)	2.94
Absorption	0
Analysis mode	General Purpose
Sensitivity	Normal
Suspending Phase	Water/0.01-M NaOH

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.

3.9.5 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, Al₂O₃), 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° 2 θ, with a step size of 0.02° 2 θ and count time of 2.0 seconds per step. Copper Kα 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 $\sim 35^\circ 2\theta$.

Details of the XRD analysis and results are provided in Appendix L.

3.9.6 Scanning Electron Microscopy

The specimen was vortexed, and a small volume of slurry was drawn up using a pipette and placed on an aluminum stub. This was done within the same sample processing as above for the XRD sample preparation, the final step just before the supernatant was decanted. 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 Appendix L as part of the Test Data Package, TDP-WTP-356.

4.0 Test Narrative

This section describes the execution of Integrated Test D that was run from 03/22/09 to 03/31/09. Testing was conducted in accordance with Test Instruction TI-WTP-PEP-082. Observations were written in this Test Instruction and in LRBs BNW-60272 and BNW-60280.

Integrated Test D started with loading the simulant that had chromium oxyhydroxide (CrOOH) already in the simulant, unlike previous tests. The slurry was kept at temperatures below 45°C until use to make sure that the Cr did not oxidize during transportation and storage.

The Operational Process Sheet section of the Test Instruction provided the Lead Test Engineer (LTE) with target or setpoint values for key process operations. A reproduction of the Operational Process Sheet, with actual values from the LTE or other data source, is provided in Appendix M.

Most of the data reported in the text of this section of the report are NOT NQA-1 because they were taken from the HMI during the conduct of the test. NQA-1 data are available in the graphs in Appendix J, selected values in the Operational Process Sheets, and the DAS files recorded as described in Section 3.6.

Caustic leaching in vessel UFP-VSL-T02A was demonstrated in Integrated Test D using nonradioactive simulant. To view the summary for sampling events, please refer to Appendix C. The subsequent paragraphs summarize the process steps executed for Integrated Test D. However, refer to Appendix K for start and stop times of process steps during Integrated Test D efforts.

Before starting Integrated Test B, all five filters were cleaned with 2-M nitric acid. No cleaning was performed between Integrated Tests B and D. At the conclusion of Integrated Test D, the filters were cleaned in a prototypic manner with 2-M nitric acid. The results are included in this report.

4.1 Feed Preparation

The slurry was delivered as simulant Batch 3 to PEP on 3/16/09, in a heated trailer. The delivery included three totes of slurry at a concentration of ~7.4-wt % undissolved solids (UDS) and two totes of supernate. The solids contained 1.6-wt% Cr as CrOOH. The slurry batch was mixed with previous Batch 2 slurry in the FRP vessel to produce slurry with ~5.0-wt% UDS. The FRP vessel held approximately 1009 gallons of Batch 2 slurry before adding Batch 3. All 720 gallons of Batch 3 slurry and 410 gallons of supernate were added on 3/16/09. The FRP was allowed to agitate for approximately 8 hours, and then 29 samples were taken. Twenty three of the samples were taken in the 50-mL containers for analysis to verify the UDS and Cr content. In addition, a larger 1-L sample was taken for a laboratory caustic-leaching test to determine how much Cr would be oxidized during the caustic-leaching process.^(a) This value was used to determine the quantity of NaMnO₄ to be added during the PEP oxidative-leach process.

(a) RD Scheele, GN Brown, and DE Kurath. 2009. *Scale-Up, Production, and Procurement of PEP Simulants*. WTP-RPT-204, Pacific Northwest National Laboratory, Richland Washington.

The HLP-VSL-T22 vessel received 2160 gallons of ~5-wt% solids from the FRP-VSL-T01 starting on 3/22/09 at 22:00 hours. For Integrated Test D, 1600 gallons of slurry were needed to make two batches of leached solids in Tank T02A.

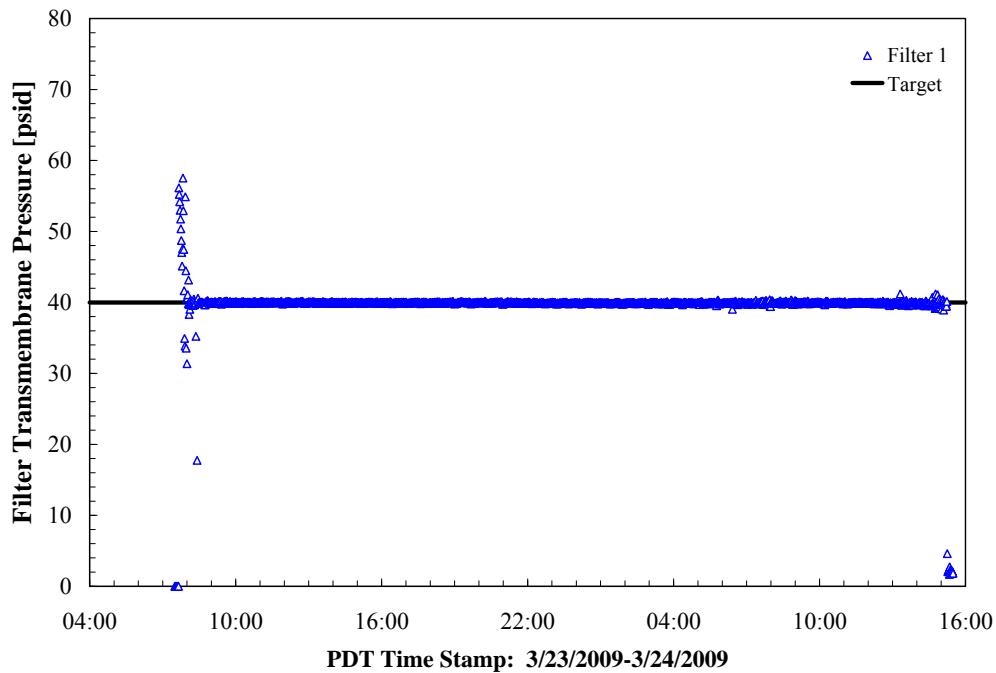
From the HLP-T22 vessel, 593 gallons of slurry were transferred on 3/23 at 02:10 hours to UFP-VSL-T01B at a rate of 7.4 gpm for the start of Batch 1. UFP-VSL-T01B was used as directed by the Test Instruction, although it was different than the Test Plan because one PJM in UFP-VSL-T01A was inoperable. During the transfer, samples were taken after 3, 200, and 400 gallons of slurry were transferred. The PJMs were started when the level in Tank T01B was 16 inches, and the pressure was adjusted later to obtain the correct nozzle velocity and stroke length. The final level in Tank T01B was 65.8 inches, based on the laser level monitor. The PJMs were tuned to obtain a 29-in. (80%) stroke and an average maximum velocity of 5.1 m/sec. The PJMs were operated with a 35-sec cycle time in the standard mode. One thousand mL of Dow Corning Q2-3183A Antifoam Agent (AFA) diluted with 3 parts water to 1 part AFA was added (target concentration of ~350 ppm) to minimize foaming.

From UFP-VSL-T01B, 218 gallons of slurry were transferred to UFP-VSL-T02A on 3/23/09 at 03:04 hours with the filter-loop inlet valve from Tank T02A closed. After filling the vessel to 50 inches, the PJMs in Tank T02A were operated with 7.7 m/sec nozzle velocity, a 32-in. (80 %) stroke and a 33-sec cycle time. The filter-loop was drained of inhibited water, and Filter T02A was backpulsed as a simple way to vent the system and increase the draining rate. After the filter-loop was drained, the filter-loop inlet valve V06132 was opened, and the filter-loop was filled with slurry. The automatic transfers from Tank T01B were set-up for 11-gallon batch transfers that occurred when the Tank T02A level was below 27 inches. Additional slurry was transferred to Tank T01B for the rest of leach Batch 1 and Batch 2 when the level was low. The laser level sensors in Tank T02A and T01B were the most reliable level sensors and were used most of the time.

4.2 Leach Batch #1 Slurry Concentration

The objective of the slurry concentration was to concentrate the ~5-wt% UDS slurry to 20-wt% by adding forty-six 11-gallon batches to Tank T02A from Tank T01B and concentrate Tank T02A down to 19 inches. The recirculation pumps were started for the first leach batch by setting Pump 42A to 25%, and then Pump 43A was ramped up to 65% to obtain a flowrate of 96 gpm at 07:50 hours. Pump 43A was then adjusted slowly to achieve the desired flow rate of 109 gpm. The lower air spargers and steam ring purge were set at 0.01 kg/min, and the upper spargers were off. Later the steam ring air purge was increased to 0.14 kg/min. The PJMs had a 33-sec cycle time, a nozzle velocity of 7.7 m/s, and stroke length of 32 inches. The filter permeate valves were opened, the slurry solids concentration was started on 3/23/09 at 07:17 hours, and the permeate was directed to UFP-VSL-T62A. Initially, all of the filters' permeate valves were opened for a short time to remove air from the system and allow stable filter-loop flow rates. Only Filter #1 was used to concentrate Tank T02A Batch #1. The filtration rate after Tank T01B transfer #9 (~3 hours) was 1.5 kg/min, and the automatic fill level was set at 27 inches based on the laser level probe. There was not enough slurry in Tank T01B to complete additions to Tank T02A to obtain the target 20-wt% solids for caustic-leach batch, so additional slurry was added to Tank T01B. The first refill of Tank T01B from HLP-T22 occurred on 3/23 when Tank T01B reached the 29.7-inch level. The transfer was 362 gallons at 7.4 gpm, and 612-mL of AFA was added to Tank T01B after the transfer. Figure 4.1 through Figure 4.4 illustrate TMP, axial velocity, temperature, permeate production, and Tank T02A tank level during the concentration of Batch 1.

After adding the 46 batches, the solids concentration continued, dropping the level in the Tank T02A vessel to 18.3 inches on 3/24/09 at 15:15 hours. When the level was below 25 inches, the PJM star mode was used. The concentration was performed with a slurry flow rate of 109 gpm, although the flow rate rose and fell a couple percent near the end because of tank level decreases, slurry rheology changes, and manual adjustments of the pump settings. When the level was low (<23 inches), the system had difficulty maintaining a flow rate of 109 gpm because of entrained air. The operations personnel adjusted the pumps to try and maintain the flow, but ultimately, the flow decreased to values less than 100 gpm. The TMP of 40 psid was maintained. The permeate rates were not significantly affected by the lower flow rates and the stable level measurements had larger temporary effects on the permeate flow rate. Stopping the pumps and restarting the pumps for stable level measurements caused some of the filter-loop flow fluctuations and flow disruptions. The final slurry UDS concentration for caustic leach Batch #1 in T02A was 23.4-wt% with the target being 20-wt%. Figure 4.5 provides the filter-loop flow rate level in Tank T02A and the axial pressure drop during Batch 1 concentration.



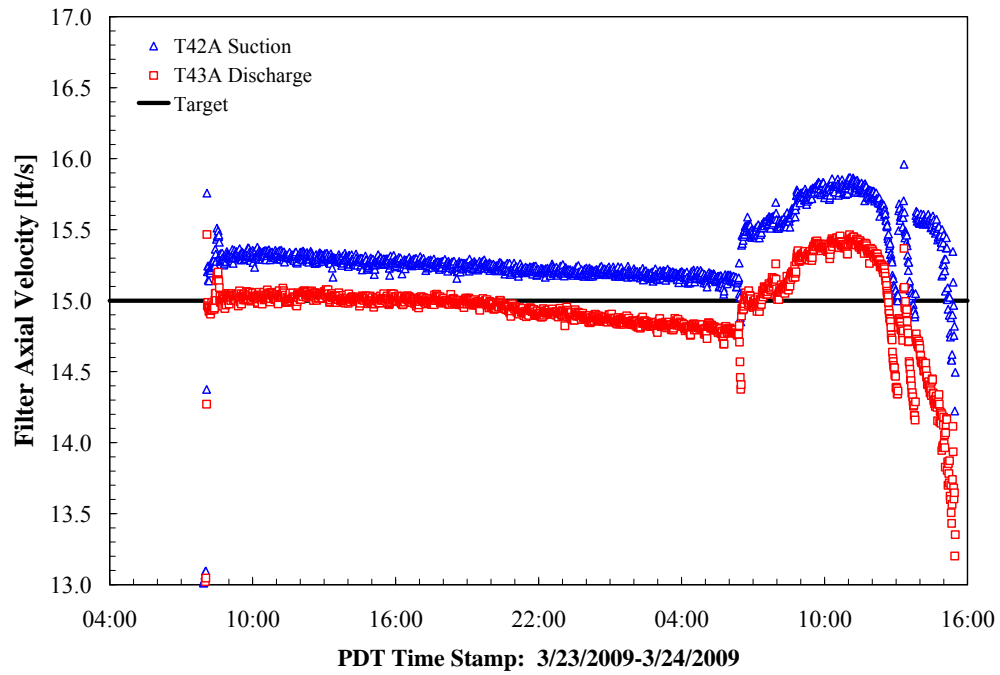


Figure 4.2. Axial Velocity Based on Flow Measurements at the Suction to T42A and the Discharge to T43A for Pre-Caustic-Leach Concentration, Simulant Batch #1

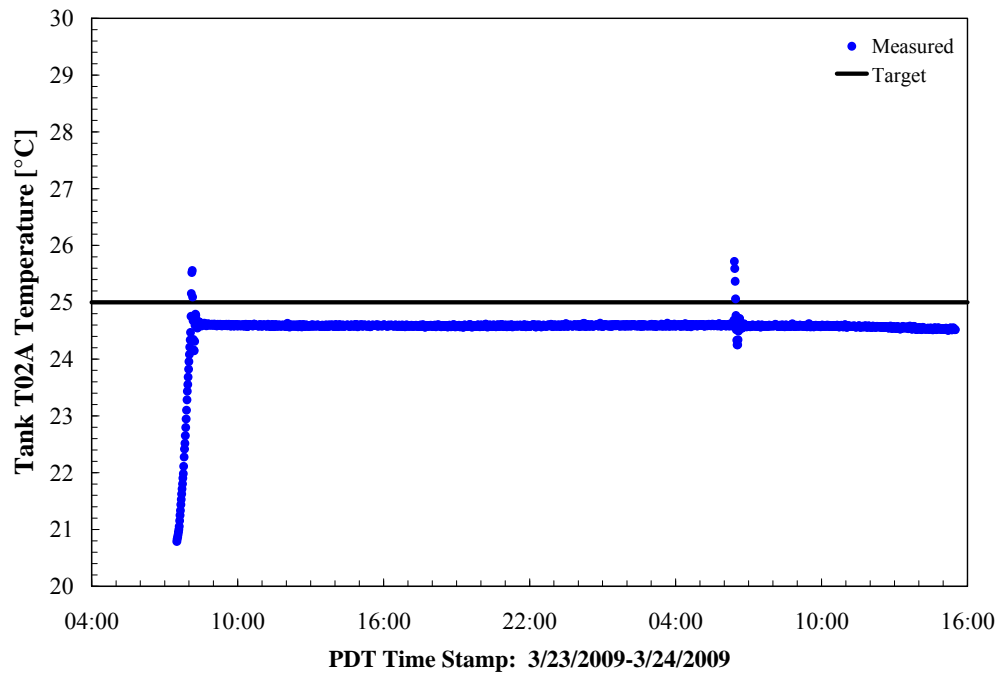


Figure 4.3. Tank T02A Temperature (TK-0619) for Pre-Caustic-Leach Concentration, Simulant Batch #1

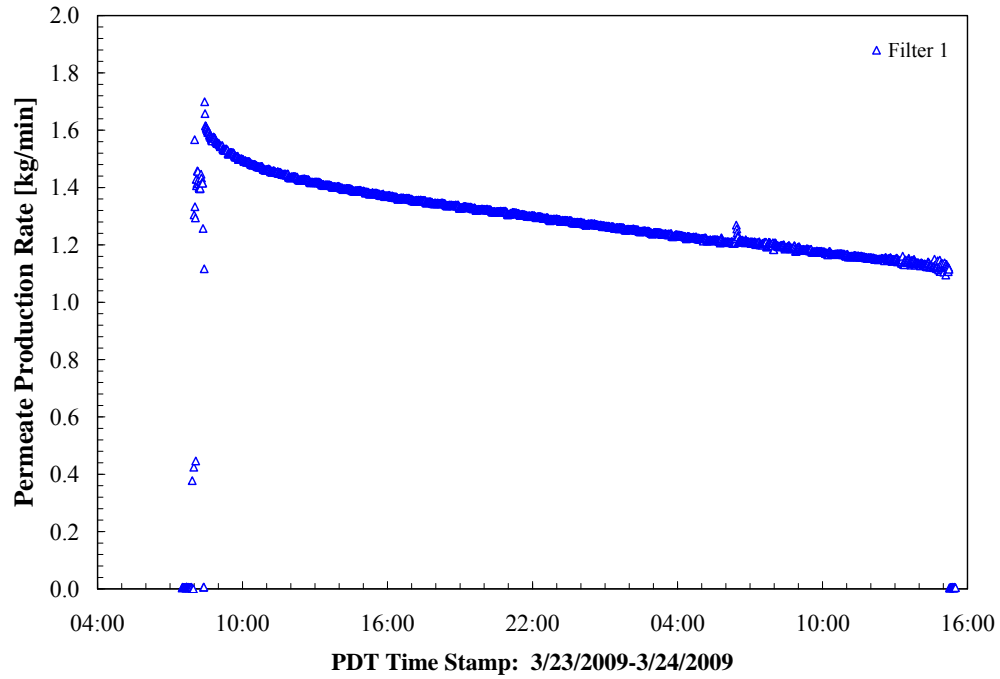


Figure 4.4. Permeate Production Rate for Pre-Caustic-Leach Concentration, Simulant Batch #1

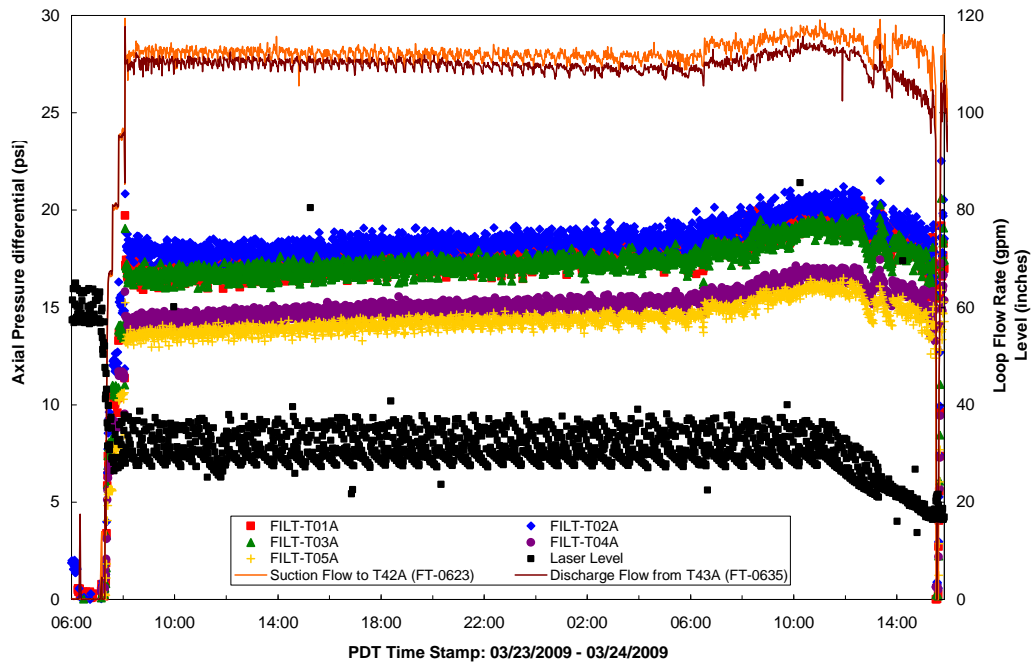


Figure 4.5. Filter-Loop Flow Rate, Level in Tank T02A, and Axial Pressure Drop During Batch #1 Concentration

4.3 Batch #1 Caustic Leaching

After concentration, 939 kg of 19-M NaOH was added on 3/24/09 at 15:49 hours to the inlet of the recirculation pumps at a rate of 11.5 kg/min. The NaOH flow control valve was manually set at 12.3% open, and the NaOH manual recirculation valve that could be adjusted to control the NaOH header pressure was 100% open. The filter-loop flow rate was ~109 gpm with the lower air spargers set at 0.01 kg/min, and the upper air spargers and steam purge were off. The PJMs were changed from star mode to standard mode at 16:07 hours with the same settings. The steam ring air purge was restored to 0.14 kg/min at 16:20 hours. The controller for the heat exchanger used to cool the filter-loop (UFP-HX-T02A) was adjusted to allow the temperature to increase to 65°C. After the NaOH was added over ~1.4 hours, the temperature of the slurry was ~65°C. The upper spargers were turned on at a rate of 0.1 kg/min at 17:09 hours when the Tank T02A level was ~50 inches with the pumps on. The Tank T02A level was 62.4 inches with SpG of 1.42 at the end of the caustic addition with the pumps on. The tank level was higher than expected, mainly because of air entrainment and/or foaming in the slurry. After caustic addition was complete, all air into the tank was turned off at 17:17 hours to perform an standard level measurement (SLM) and to reduce air entrainment. The PJMs were operated in simple mode with a 7.25-sec drive time.

After the cause of the high tank level was investigated and before filter-loop flushing, ~ 137 kg (25 gal) (LRB 60272, pg. 35–36) of slurry was removed from Tank T02A via the recirculation loop sampling port to lower the level and prevent overfilling the tank during leaching. The slurry was weighed in five batches to determine the amount removed. In addition, 2 liters of 4:1 diluted AFA was added on 3/25/09 at 00:08 hours to attempt to reduce the foaming in the tank. The slurry removed had a significant amount of bubbles as shown in the carboy sample in Figure 4.6. Additional 2 liter mixtures of AFA were added at 00:57 hours and 01:32 hours.

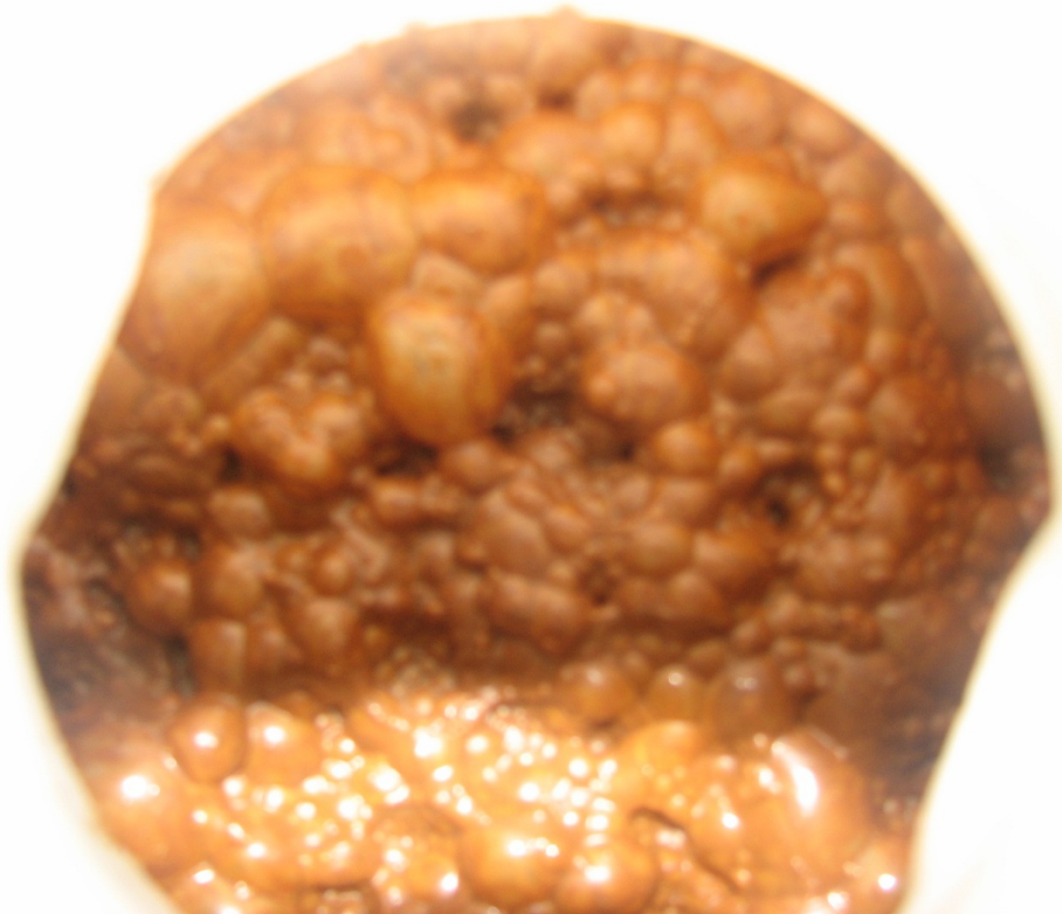


Figure 4.6. Surface of Slurry Sample in Carboy Drained from Tank T02A

After the tank level had been adjusted by removing slurry, a stable level measurement was performed, and various sparger rate settings and de-aeration hold times were performed to reduce entrained air and to obtain an accurate level measurement. A camera was also used to look in the tank to assess conditions. The camera view showed that the level appeared to match the laser sensor values, there was some foam, and that the laser level was aimed appropriately.

The filter loop had 74 kg of IW added to the loop at a rate of 75 kg/min on 3/25/09 at 12:03 hours, which caused ~ 74 kg of slurry to transfer back to Tank T02A. This purge was only a small portion of the ~400 kg in the filter-loop, but additional water was not used to prevent too much slurry in the tank. The TI instructions had not planned to transfer more of the filter-loop slurry back to the tank to make sure Tank T02A was not diluted if the flush did not occur as plug flow. After the initial IW addition, the rest of the filter-loop was isolated, recirculated, sampled, and transferred separately to T62A using portable diaphragm pumps.

The tank investigation and level adjustment took approximately 18 hours, and the slurry with caustic was held at temperatures between 50 and 65°C for that time. This time was not included in the leach time since the temperature was below 65°C.

After the IW addition to the filter-loop was complete and the Tank T02A isolated, steam was used to heat and hold the vessel contents at 85°C. The final SLM level before steam heating was 57.6 inches. The automatically controlled heating started on 3/25/2009 at 12:18 hours and reached 85°C at 13:52 about 1.6 hours later as specified in the Test Instruction. The Tank T02A temperature showing the heat-up rate while the NaOH was added and during steam heating is shown in Figure 4.7. The batch was leached for 24 hours before cooling started. The control system worked well, controlling the temperature between 83 and 87°C, except right at the start where an overshoot caused temperatures of ~89°C as was observed in previous test and may be attributed to the SLM performed just before. The SLM entails stopping the PJMs and reducing mixing for a short period of time. The tank level started at 57.6 inches after the NaOH was added, and flushing was completed. The level increased to 70.1 inches by the end of the leaching period shown in Figure 4.7. While holding the vessel temperature at 85°C, the condensate from direct steam injection increased the Tank T02A level approximately <0.3 in/hr. Many samples were taken to assess leaching progress. During heating, the PJM condensate drain valves were open when the Tank T02A temperature was above 65°C, and condensate from the PJM air lines was collected in a separate carboy.

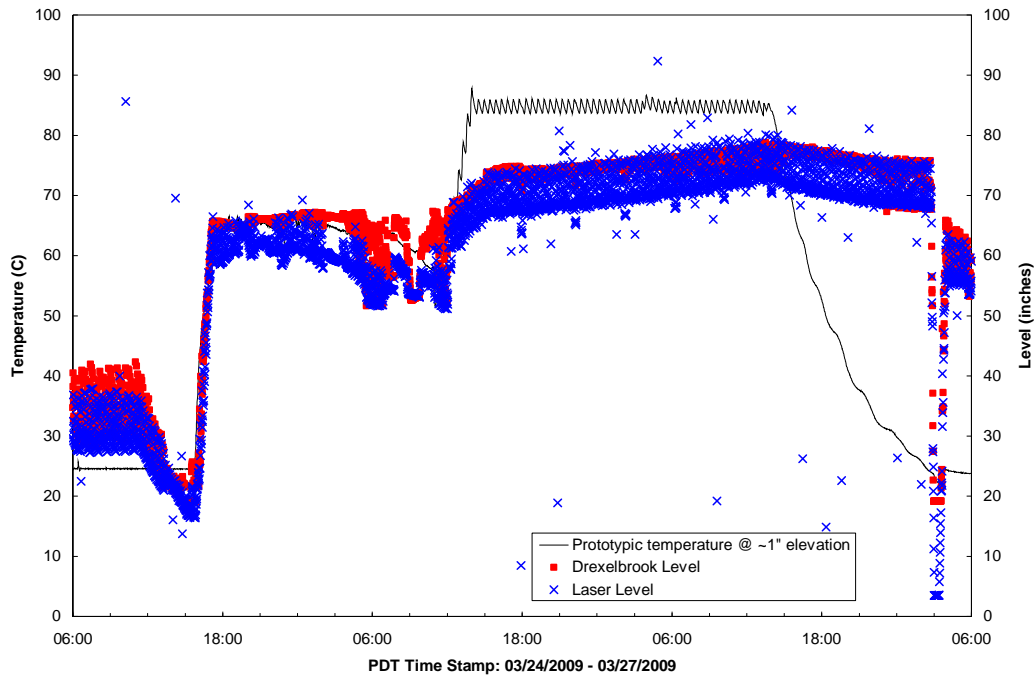


Figure 4.7. Tank T02A Level and Batch 1 Leach Temperatures

After the leaching process was complete, the Tank T02A vessel was cooled to 25°C over a period of 12.5 hrs as specified in the Test Instruction. The cooling process was fairly smooth as shown in Figure 4.7, and only the cooling jacket was used. The cooling control valve was slowly opened manually by the operator in 5% increments to control the tank cooling. A typical value at lower temperatures was 20% open.

During the leaching process on 3/26, the second refill of Tank T01B was performed by adding an additional 362 gallons of slurry, which was transferred from HLP-T22 to Tank T01B at a rate of 7.4 gpm.

AFA was also added in the amount of 612-mL undiluted. The AFA was always added in a 3 parts water to 1 part AFA mixture.

The first batch of leach solids was transferred to Tank T01A for storage until Batch 2 could be completed, and then the two batches were concentrated together. The UDS of the first leach batch was 5.02-wt%. The heel left in Tank T02A is about 5 gal.

4.4 Batch #2 Slurry Concentration

The second batch started by filling Tank T02A with 218 gallons of simulant from Tank T01B. The second batch concentration started on 3/27 0700 using five filters, and was almost the same as the first batch. The final tank level was 17 inches with the recirculation pumps operating. When the tank level is below ~20 inches, part of the filter-loop drains back into the tank when the pumps are off, so the stable level measurement was about 19 inches. The lower air spargers were set at 0.01 kg/min and the steam ring purge was 0.14 kg/min. The upper air spargers were off and the PJMs were set the same as Batch #1 with a 33 sec drive time, a 7.7 m/s nozzle velocity, and a 31.5 inch stroke. The filtering conditions were the same as Batch #1 with a 109 gpm flow rate and a 40 psid TMP. The permeate rates were slightly lower than Batch #1. The filtration time was 5.5 hours, including the pause to refill Tank T01B between 08:45 and 10:00. The final UDS concentration was 23.8-wt% and the target was 20-wt%. Figure 4.8 through Figure 4.11 illustrate TMP, axial velocity, temperature, and permeate production during the concentration of Batch 2. Figure 4.12 provides the filter-loop flow rate, level in Tank T02A, and axial pressure drop during Batch 2 concentration.

During the concentration in Tank T02A, the UFP-VSL-T01B was refilled the third time with 361 gallons at 10 gpm. Filtration was stopped while Tank T01B was being filled as noted in Figure 4.8. The target fill rate was 7.4 gpm, but the pump did not operate well at that rate. After the slurry addition, 612-mL of AFA was added to Tank T01B.

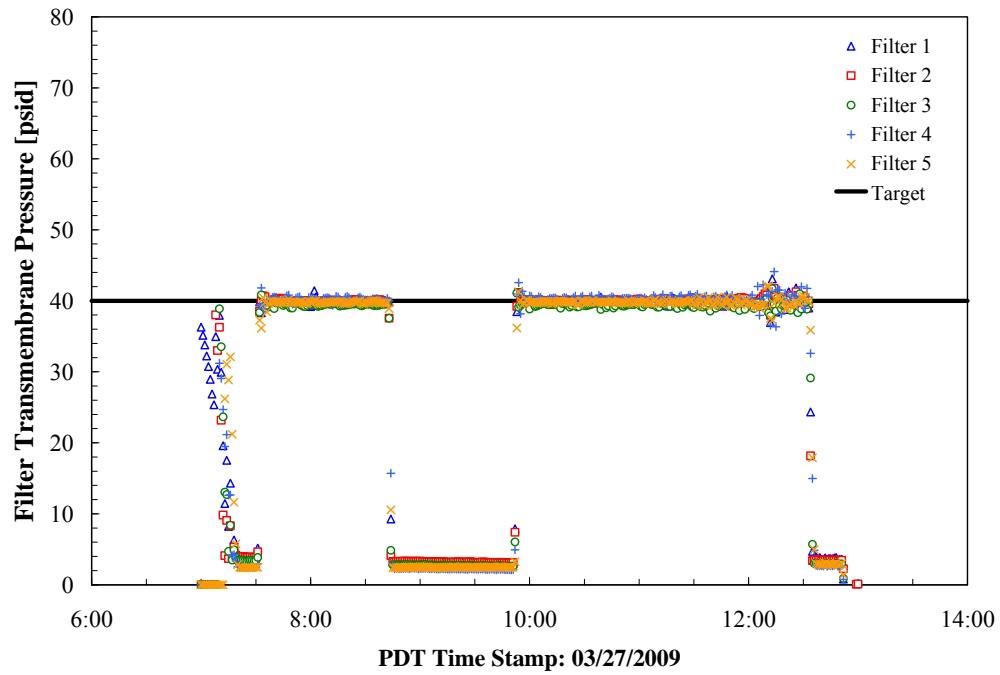


Figure 4.8. TMP for Pre-Caustic-Leach Concentration, Simulant Batch #2

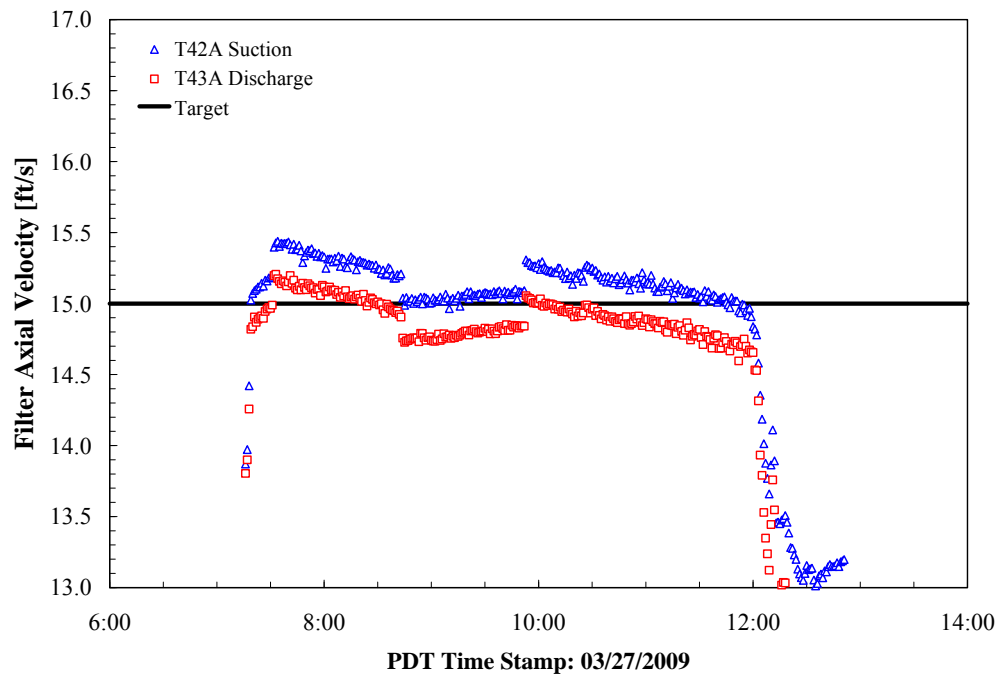


Figure 4.9. Axial Velocity Based on Flow Measurements at the Suction to T42A and the Discharge to T43A for Pre-Caustic-Leach Concentration, Simulant Batch #2

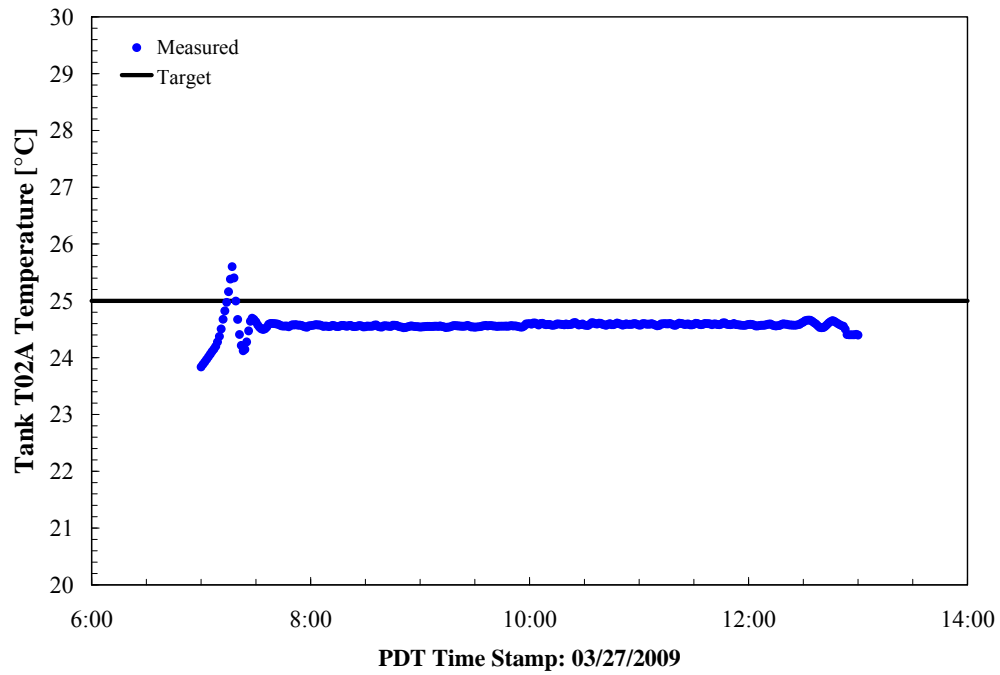


Figure 4.10. Tank T02A Prototypic Temperature for Pre-Caustic-Leach Concentration, Simulant Batch #2

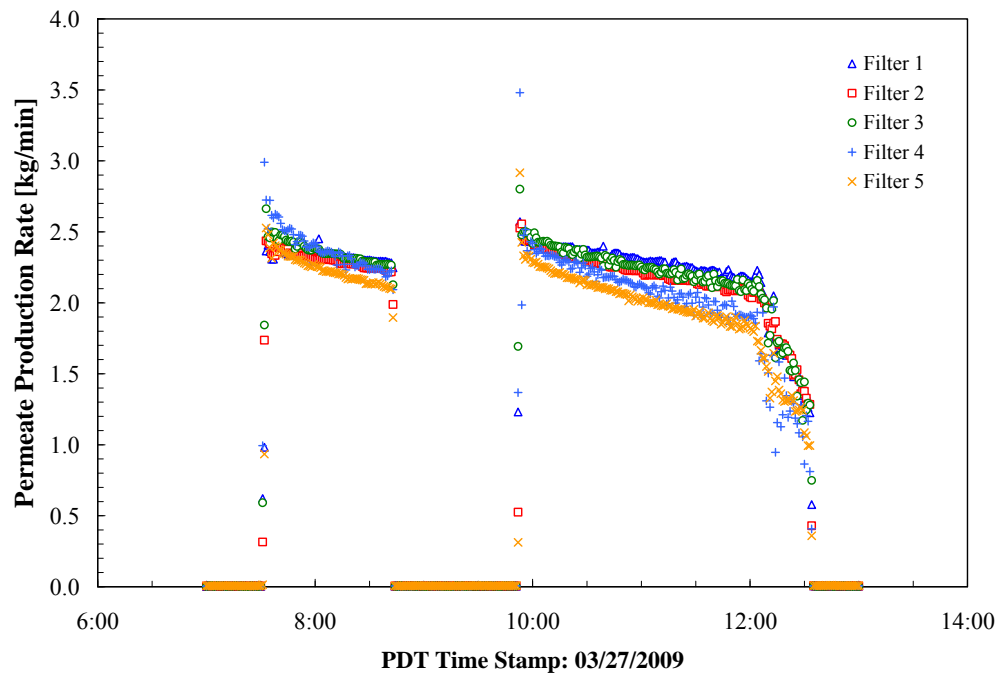


Figure 4.11. Permeate Production Rate for Pre-Caustic-Leach Concentration, Simulant Batch #2

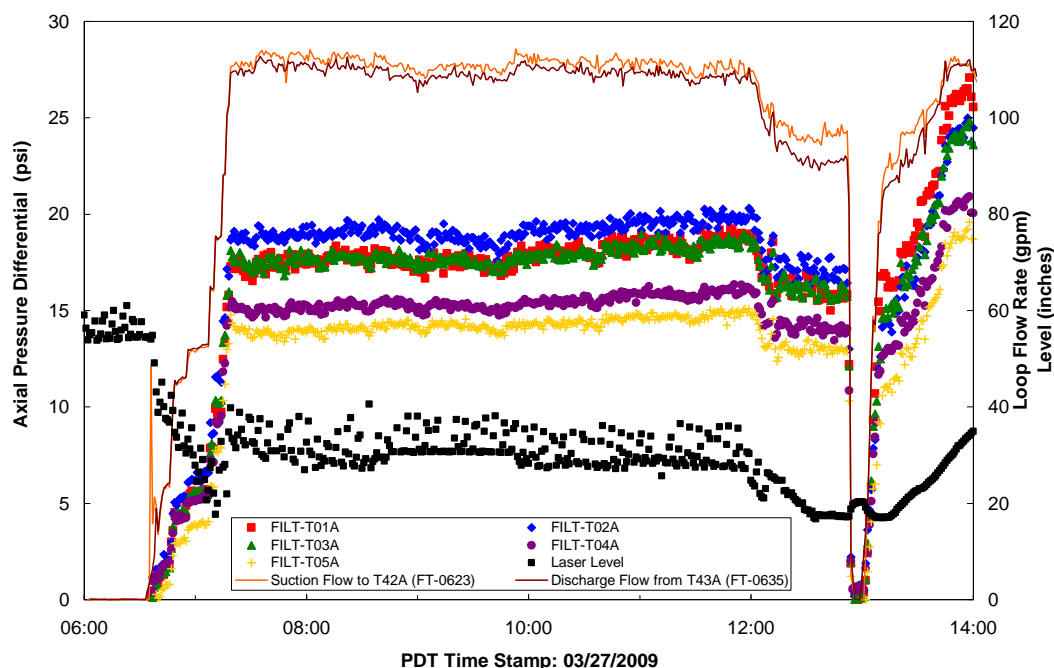


Figure 4.12. Filter-Loop Flow Rate, Level in Tank T02A, and Axial Pressure Drop During Batch 2 Concentration

4.5 Batch #2 Caustic Leaching

After concentration in Tank T02A, 928 kg of 19-M NaOH was added on 3/27 at 13:15 hours to the inlet of the recirculation pumps at a rate of 10.2 kg/min. The NaOH addition and initial heat-up rate of 0.47°C/min are shown in Figure 4.13. The heat from the NaOH reaction and the heat from pump were used to initially heat the slurry to 70°C. At the same time, the controller for heat exchanger used to cool the filter-loop (HX-02A) was adjusted to allow the temperature to increase to but not exceed 70°C. Also an additional 400-mL of diluted AFA was added to Tank T02A. The tank level after caustic addition was 57 inches. The target was 55 inches \pm 2. The level was within the target range, and the test proceeded. The filter-loop was not flushed back to Tank T02A because the test director was concerned the level would be too high at the end of the caustic-leach process. The PJMs were operated in simple mode with a 7.25-sec drive time after several adjustments. The slurry was heated with direct steam heating to raise the temperature of Tank T02A to 85°C in \sim 1.2 hours as shown in Figure 4.13. The heating time target from 70 to 85°C was 1.2 hrs.

The PJM stroke and high laser level readings caused the steam to turn off during the leaching process several times (3/28/2009 at 09:44, 16:50, and 17:05 hours), allowing the tank contents to cool slightly for part of the leach. The bubblers were flushed with hot water to try and obtain good level readings using the bubblers, but specific gravity was not stable enough to obtain good readings. The Drexelbrook level probe was used for tank level alarms and operator controls for awhile to avoid the high alarms that were possibly caused by foam. Eventually, an additional 2-L of diluted AFA was added on 3/27 at 20:30 hours and the level monitor control was switched back to the laser. Additional AFA appears to have alleviated some of the foam problems. A stable level measurement of 65.7 inches was made on 3/28 at 05:30 hours

with all air, steam, and PJMs off. The projected level increase from steam condensation was ~0.3 in./hr (1.2 gal/hr). On 3/28 at 19:45 and 19:49, it appeared some of the bottom spargers were plugging based on drops in the flow rate although the total flow was still 0.4 kg/min. No corrective actions were taken to clear the partially plugged sparger. Heating continued until 3/28 at 17:20 hrs, and the steam was turned off and cooling started. Cooling was similar to leach Batch 1, except cooling water was colder, and the initial temperature decrease was higher than planned. Intermittent opening and closing of the cooling water control valve was used for much of the remaining cooling period (22% to 0%) to control the cooling rate. The cooling took ~12.5 hrs as planned to reach 25°C. Samples were taken, and the stable level measurement was 66.5 inches from the laser and 70.2 inches from the bubblers. The final UDS concentration of leach Batch 2 was 5.4-wt%.

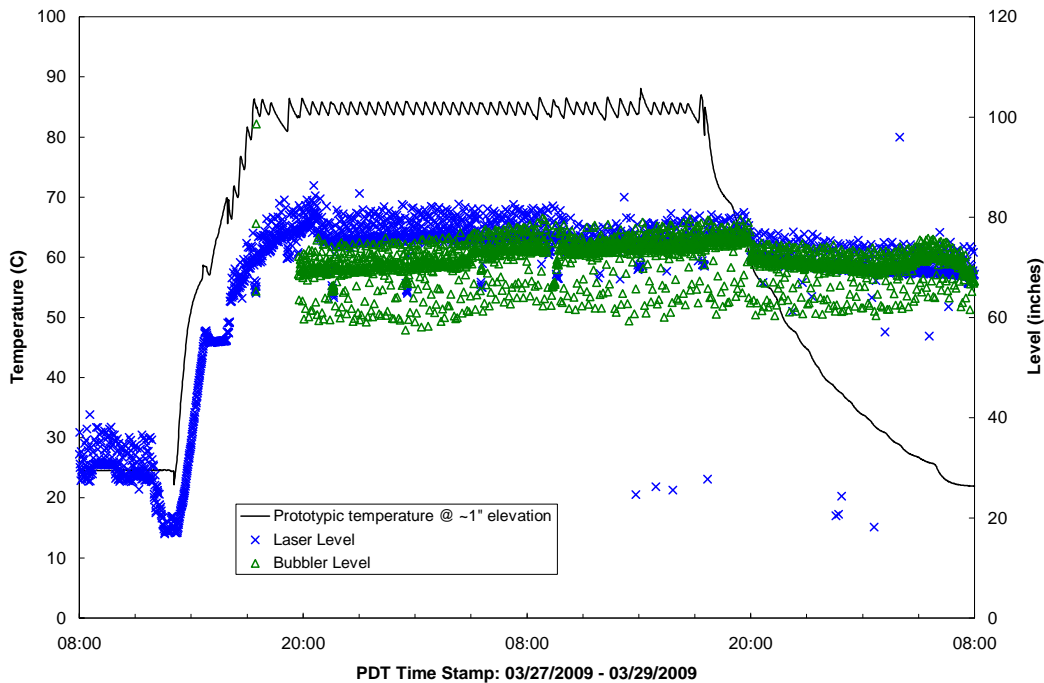


Figure 4.13. Batch #2 Leach: Tank T02A Level and Leach Batch #2 Temperatures

4.6 Leached Slurry Concentration

After the second batch was cooled, the leached solids were concentrated starting on 3/29/09 at 08:10 hours. When the Tank T02A tank level reached 53 inches, automatic transfers of the previous leached batch from Tank T01A were made. The upper and lower spargers in Tank T02A were set to 0.01 kg/min. The last couple of batches of slurry from Tank T01A were made with a portable pump so the tank could be drained completely. The filter-loop recirculation pumps were stopped during the portable pump transfers. The first transfer from the Tank T01A bottom drain to Tank T02A was started on 3/29/09 at 12:54 and completed at 13:40 hours. The second transfer was made on 3/29/09 at 15:24 hours as seen by disruptions in Figure 4.14 through Figure 4.17, including filter-loop temperatures. Turning off the filter-loop pumps caused small oscillations in the tank temperature as the controller tried to respond, and there were many PJM adjustment toward the end of the concentration to prevent aspiration or overblows. The leached solids were concentrated using five filters at an initial flow rate of

109 gpm and a TMP of 40 psid. Filter-loop flow rate changes occurred shortly after the first portable pump addition because of adjustments by the operations crew to obtain stable flow. The flow rate was also reduced near the end of the concentration as the tank level decreased, and the slurry became more concentrated. The pumps were turned off at ~ 22:10 hours to switch the Tank T02A PJMs to star mode. The pumps were turned off just in case of a PJM overblow, and the tank level was 22.5 inches. Towards the end of the concentration, the level in Tank T02A dropped below 20 inches, and the flow rate had to be reduced to 77.6 gpm because of air entrainment problems in the pumping system. The pumps were adjusted manually several times during the final concentration to try and maintain the filter-loop flow rate. The total permeate rate through all five filters started out at ~3.5 kg/min and slowed to <1.5 kg/min as the slurry became more concentrated. The UDS at the end of concentration was 18.8-wt%. Figure 4.14 through Figure 4.17 illustrate TMP, axial velocity, temperature, and permeate production during the concentration of the leached slurry.

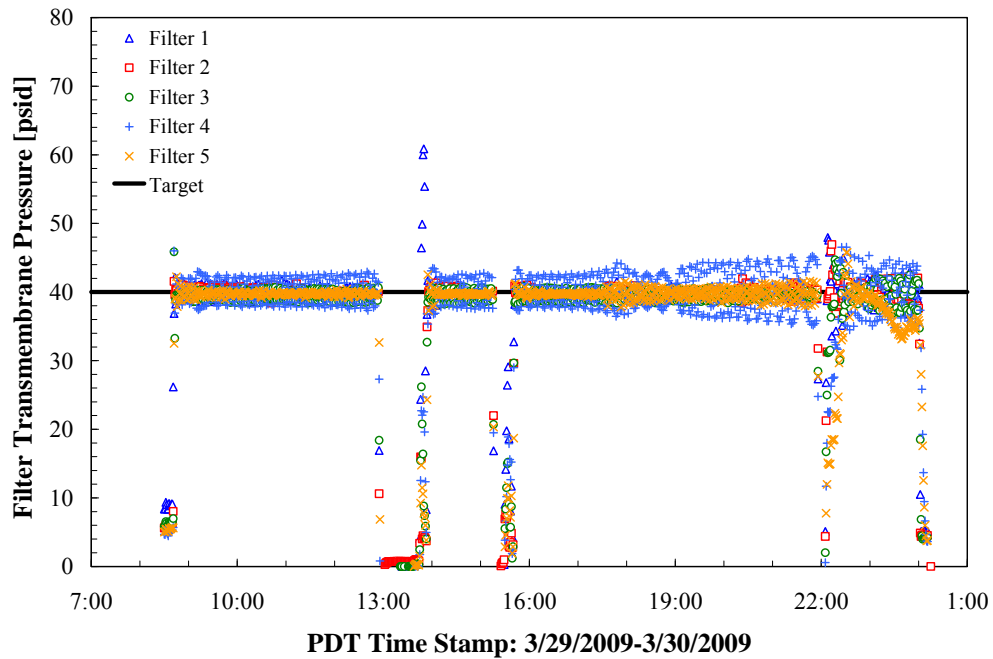


Figure 4.14. TMP for Post-Caustic-Leach Concentration

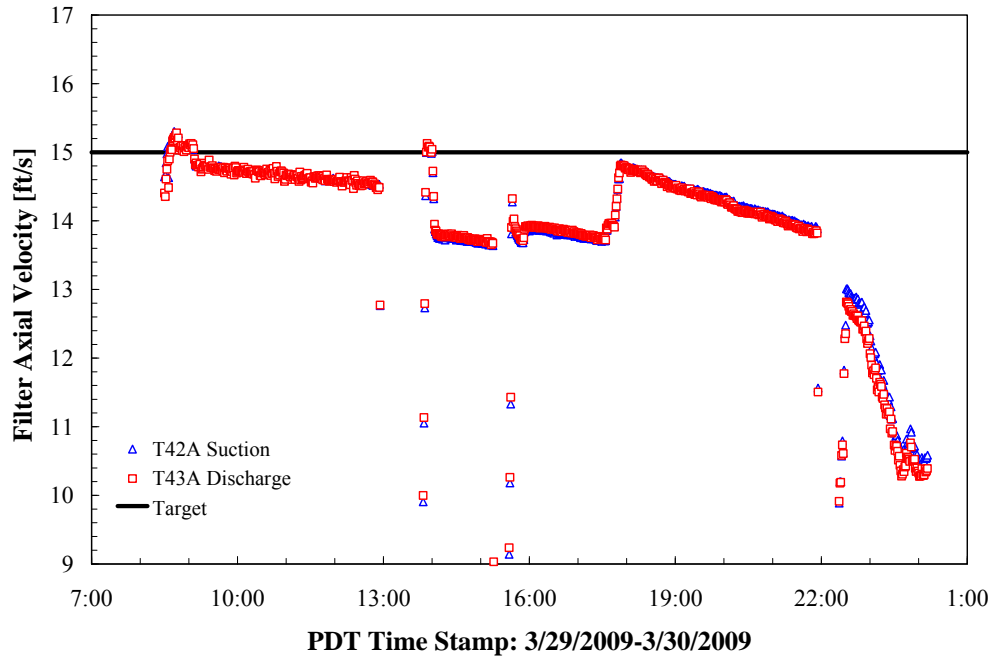


Figure 4.15. Axial Velocity Based on Flow Measurements at the Suction to T42A and the Discharge to T43A for Post-Caustic-Leach Concentration

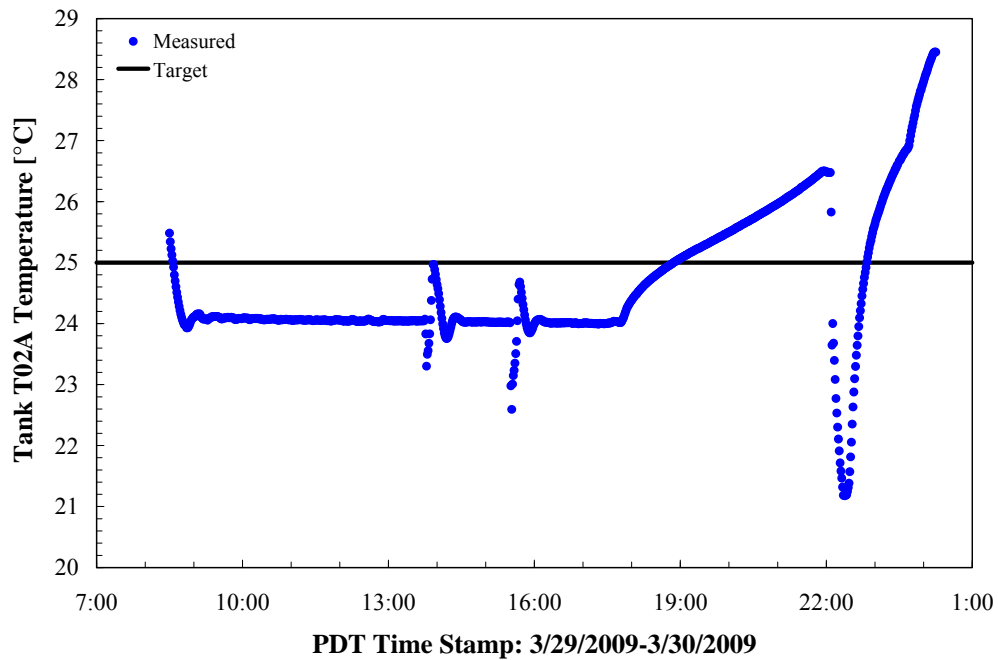


Figure 4.16. Tank T02A Temperature for Post-Caustic-Leach Concentration

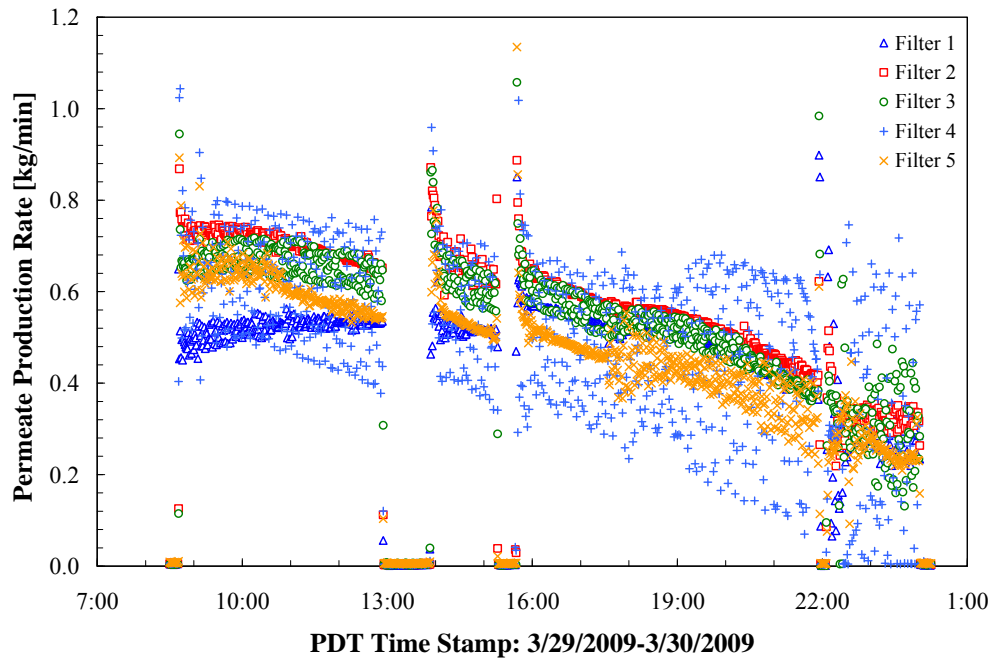


Figure 4.17. Permeate Production Rate for Post-Caustic-Leach Concentration

The final level in Tank T02A was 18.2 inches (17.2 inches with pumps on) with the pumps off or a total volume of ~168 gallons in the tank and the filter-loop. Figure 4.18 provides the filter-loop flow rate, Tank T02A tank level, and axial pressure drop during concentration. The post-caustic-leach concentration was completed on 3/30/09 at 00:15.

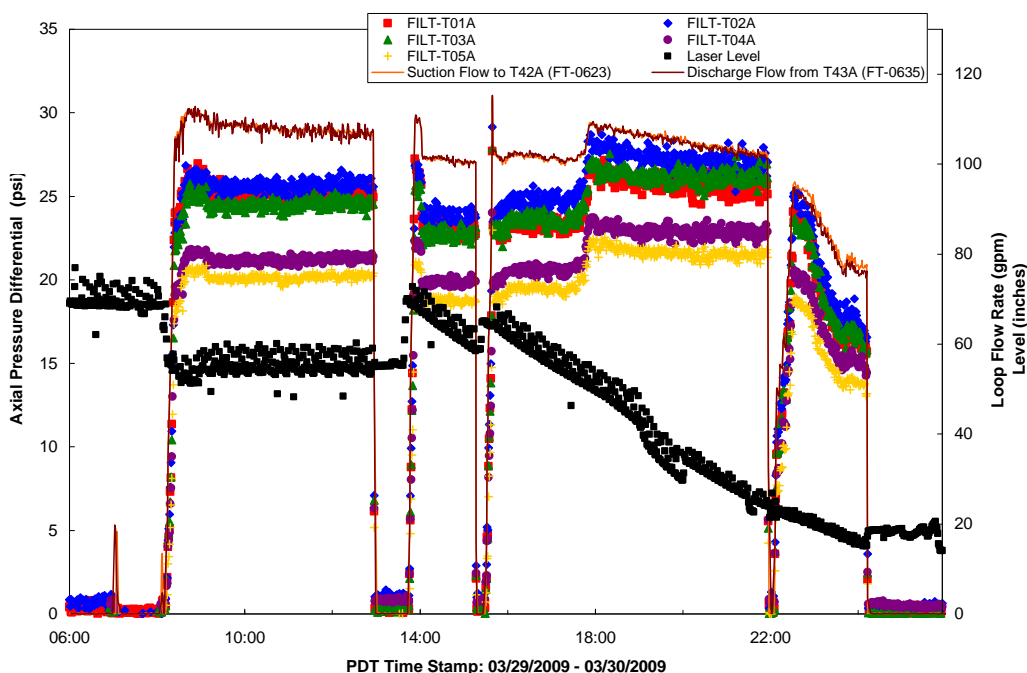


Figure 4.18. Integrated Test D Post-Leach Concentration Filter-Loop Flow Rate, Tank T02A Level, Axial Pressure Drop

4.7 Leached Slurry Washing

The leached and reconcentrated solids were washed with 2207 kg of IW using 53 batches starting on 3/30/09 at 03:48 hours. The batch size was 41.6 kg and was added when the tank level reached 17.4 inches. The IW used for washing was added at 28 kg/min, and the concurrent filtration was performed with five filters with a filter axial velocity of ~15 ft/sec as shown in Figure 4.19 and a TMP of 40 psid shown in Figure 4.20. The axial velocity slowly increased initially since the pumps were manually controlled, and the flow was reduced to ~13 ft/sec near the end of washing at the direction of Test Director Steve Barnes to head off pumping problems. Initially, the cooling water was having trouble maintaining the Tank T02A temperature and on 3/30/09 at 04:30 hours, the cooling water temperature was reduced from 42 to 40°C. Forty-four mL of AFA was added to every third batch to maintain the target AFA concentration on 350 ppm. As the washing progressed, the filtering rate improved significantly as expected as the dissolved solids decreased and the UDS increased. At the end of slurry washing with 50 batches of IW, the free hydroxide content was 0.16 M and the UDS content was 22.0-wt% Figure 4.20 through Figure 4.22 provide the temperature, and permeate production rates during the washing of the leached slurry.

The initial permeate rate per filter was 0.4, the maximum was 1.8, and the final was ~1.5 kg/min. The final tank level after washing was 17 inches with the pumps on.

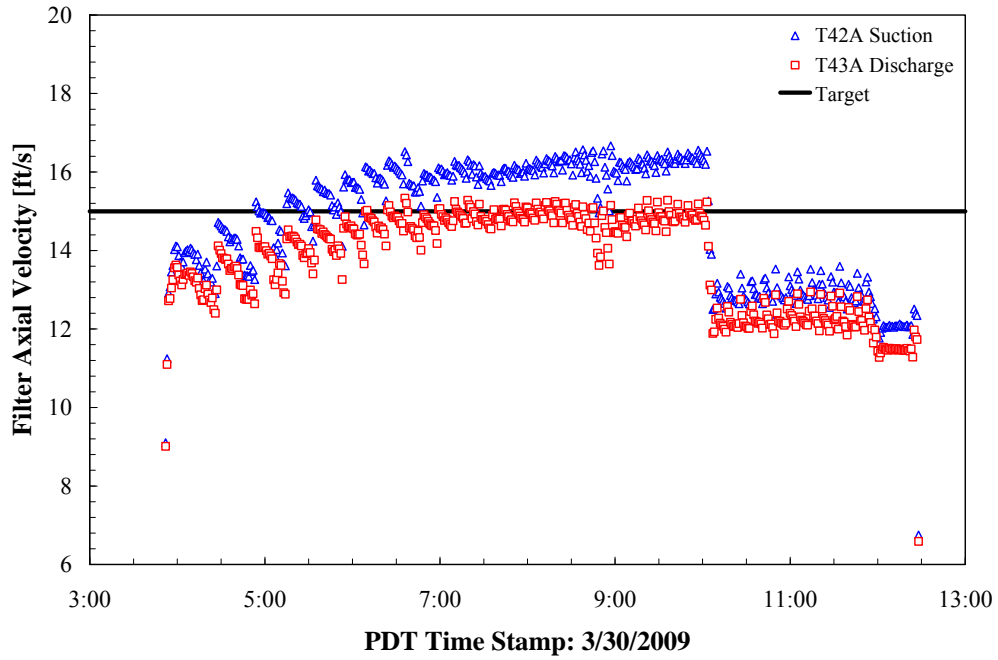


Figure 4.19. Axial Velocity Based on Flow Measurements at the Suction to T42A and the Discharge to T43A for Post-Caustic-Leach Wash

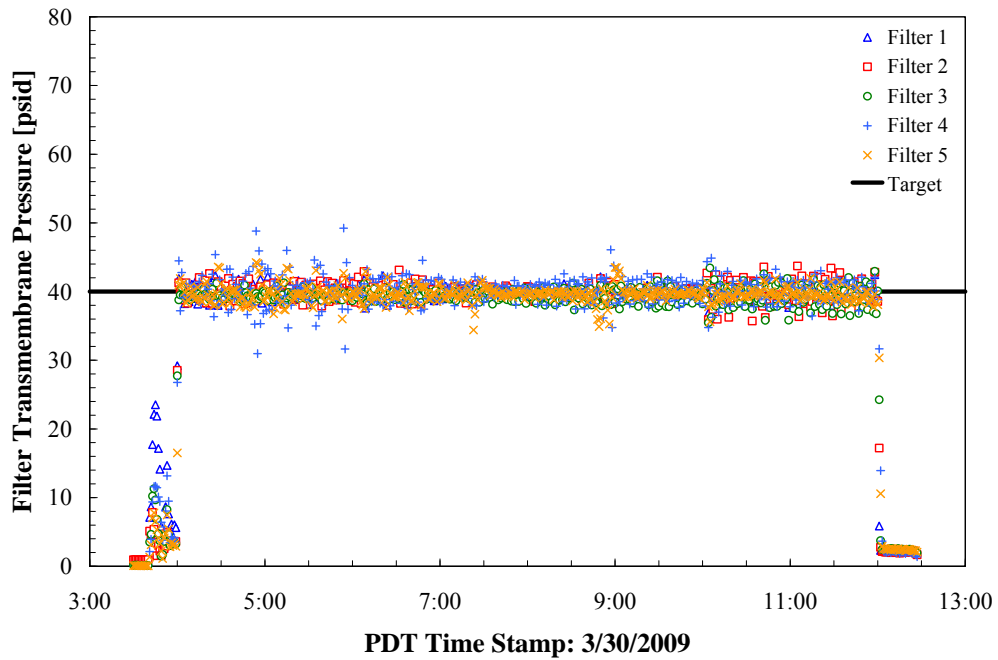


Figure 4.20. TMP for Post-Caustic-Leach Wash

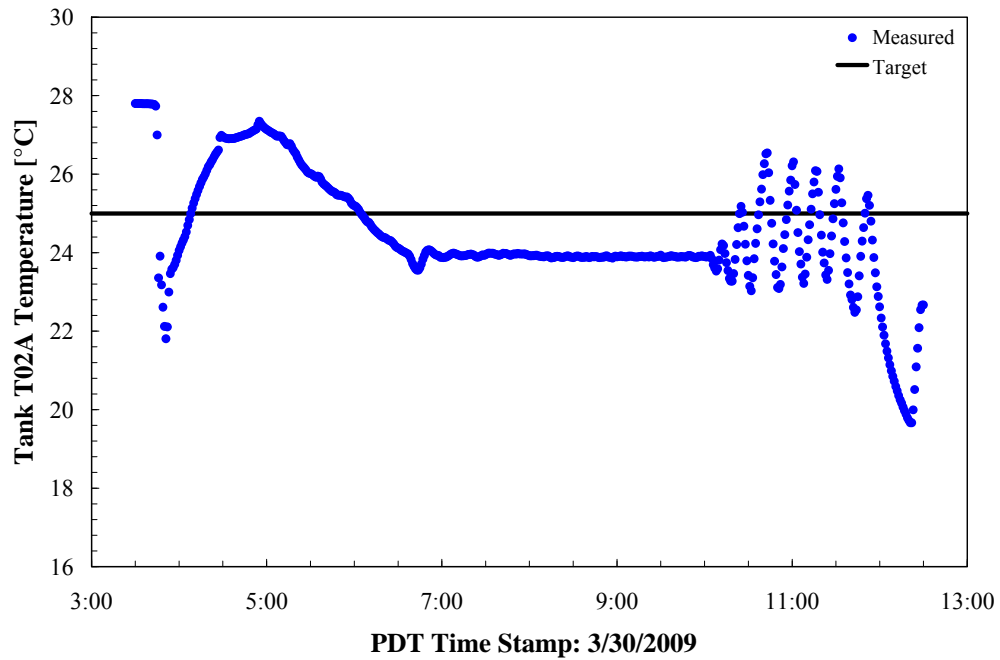


Figure 4.21. Tank T02A Temperature for Post-Caustic-Leach Wash

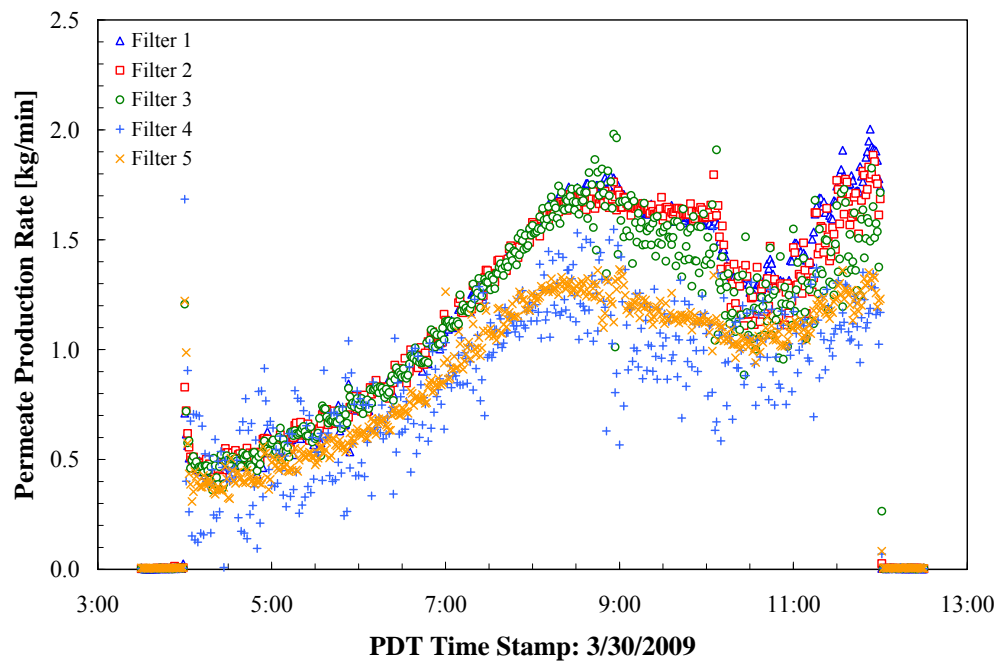


Figure 4.22. Permeate Production Rate for Post-Caustic-Leach Wash

4.8 Oxidative Leaching

The slurry was oxidatively leached after post-caustic-leach washing to remove chromium by adding 9.6 gal of 1M NaMnO₄ at 5 gpm to the filter-loop at 25°C. This volume was confirmed by the level change in the NaMnO₄ addition tank as well as the flow meter. The level in UFP-VSL-T02A increased more than it should have from adding 9.6 gal, and this is attributed to foaming. All air spargers and the steam ring purge were off. The PJMs were set in the star mode on 3/30/09 at 13:03 hours with a 20-sec cycle time, an 11.9 m/s nozzle velocity, and a 29.5 inch stroke length. The UFP-VSL-T02A temperature was controlled automatically using HX-T02A and the outlet RTD, but was manually adjusted to obtain a Tank T02A temperature of 23°C during the oxidative-leach process. The temperature fluctuated early in the test because pumps were turned off, and the tank level was low, which caused controller fluctuations during restart. During the leach, the filter axial velocity was ~13 ft/sec (~90 gpm filter flow rate), and the air spargers and the steam air purge were turned off to avoid pumping problems. The pumps were adjusted frequently between 13:55 and 14:23 hours and several times after that to try and maintain the filter-loop flow rate. The two flow meters on the filter-loop differed significantly, indicating that there was entrained air in the slurry. The filter-loop flow rate also dropped and rose (burped) for no apparent reason during the leach. The slurry was recirculated for 6 hours after the permanganate was added, and then the slurry was washed and dewatered. At the end of the oxidative-leach, the slurry UDS content was 21.2-wt%. The axial velocity in the filter-loop and the Tank T02A level is shown in Figure 4.23 and Figure 4.24.

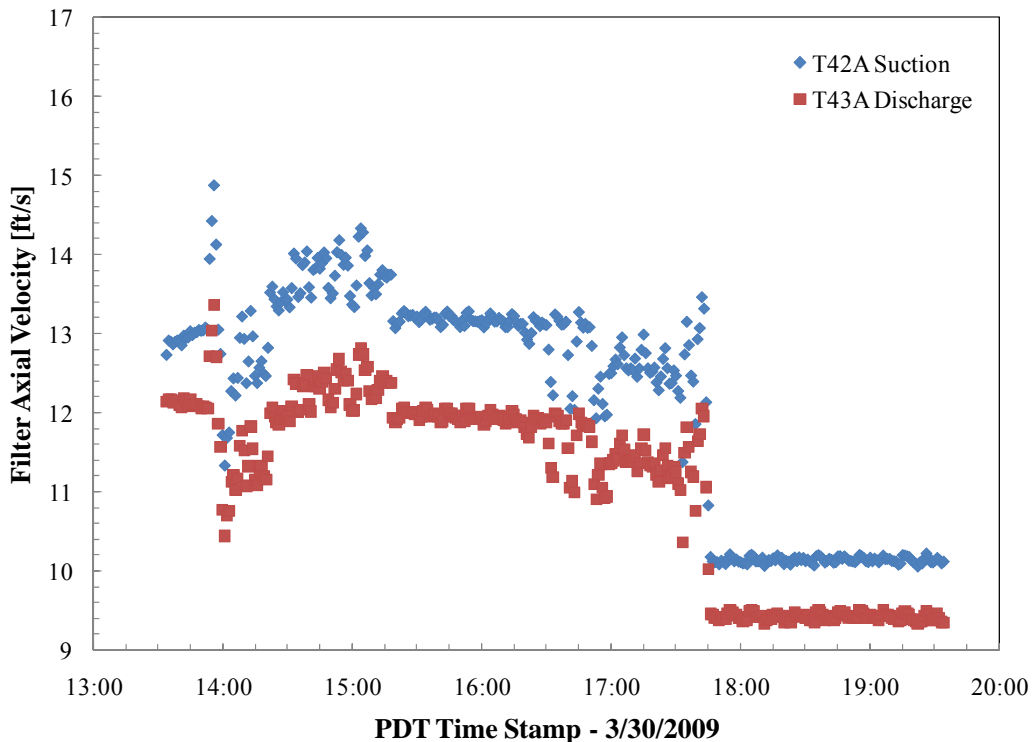


Figure 4.23. Filter Axial Velocity During Oxidative-Leach Operations, Integrated Test D

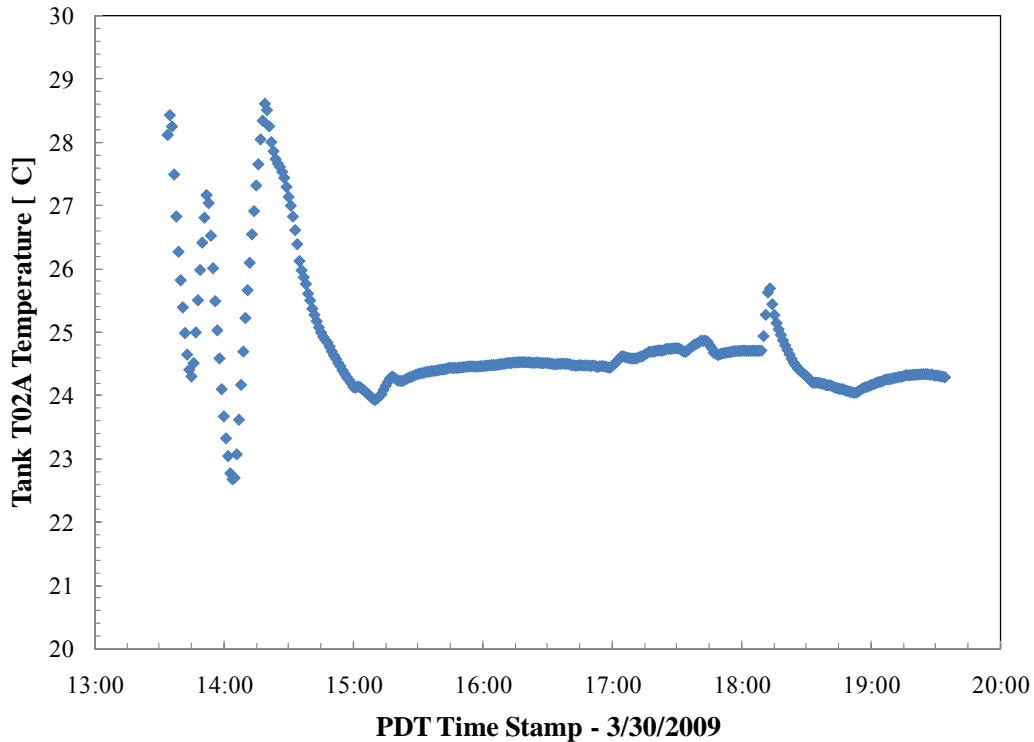


Figure 4.24. Tank T02A Temperature (TTK-0619) During Oxidative-Leach, Integrated Test D

4.9 Oxidative-Leach Washing

The washing and dewatering was started on 3/30/09 at 19:54 hours with five filters with automatic wash addition at a tank level of 19.4 inches. The wash water was added at a rate of 28 kg/min. The filtering temperature was 25°C in Tank T02A. A total of 1874 kg in forty-five 42 kg batches of IW were used to wash the slurry. An AFA mixture (1 part AFA in 3 parts IW) was added at a rate of 44-mL AFA every third wash batch to maintain the target AFA concentration of 350 ppm. The air spargers and steam ring purge were off during the washing and the PJMs were set to star mode at 19:56 hours.

Figure 4.25 through Figure 4.28 provide the TMP, axial velocity, temperature, and permeate production during the washing of the slurry. The instantaneous permeate rates during washing were erratic, so data in Figure 4.28 show 10-min averaged data. The suspected cause of the erratic operation was low permeate flow rates and relatively low loop pressures as indicated by the low TMP pressure for Filter T05A at the end of washing.

The final slurry concentration was completed on 3/31/09 05:42 hours with a final level of 15.6 inches according to the laser level probe. The slurry UDS content was 20.6-wt%. Figure 4.29 through Figure 4.32 provide the TMP, axial velocity, temperature, and permeate production during the final concentration of the slurry.

Finally, the filters were flushed back to Tank T02A with 340 kg of IW. UFP-VSL-T02A was drained to a tote, and Tank T02A was refilled with 400 kg of IW. The tank and filter-loop were recirculated, sampled, and drained to another tote. The filter-loop was filled with IW, and the test was complete on 3/31/09 at 10:28 hours.

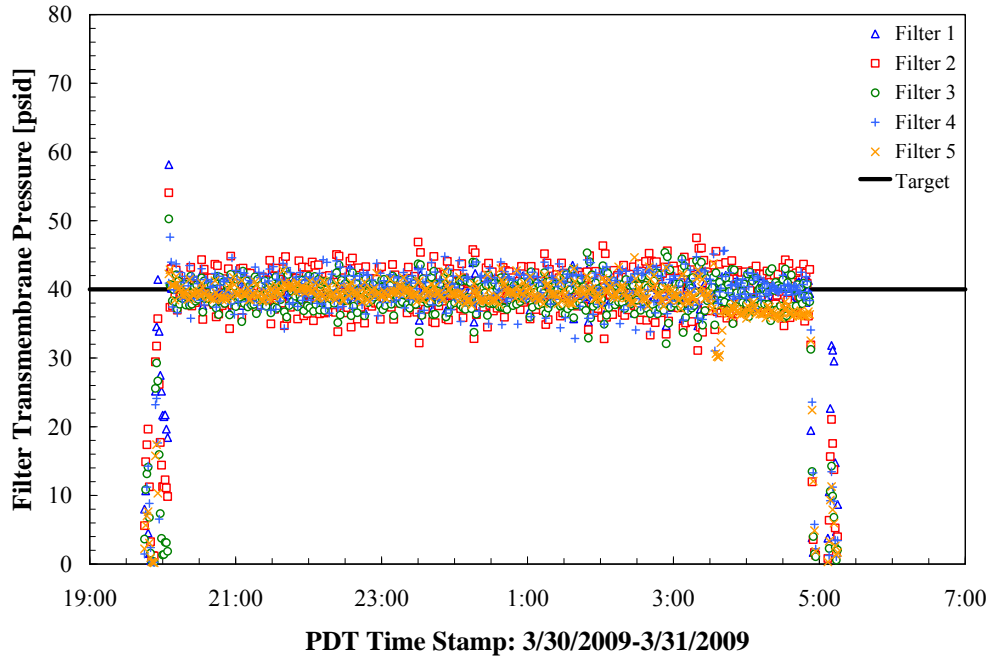


Figure 4.25. TMP for Post-Oxidative-Leach Wash

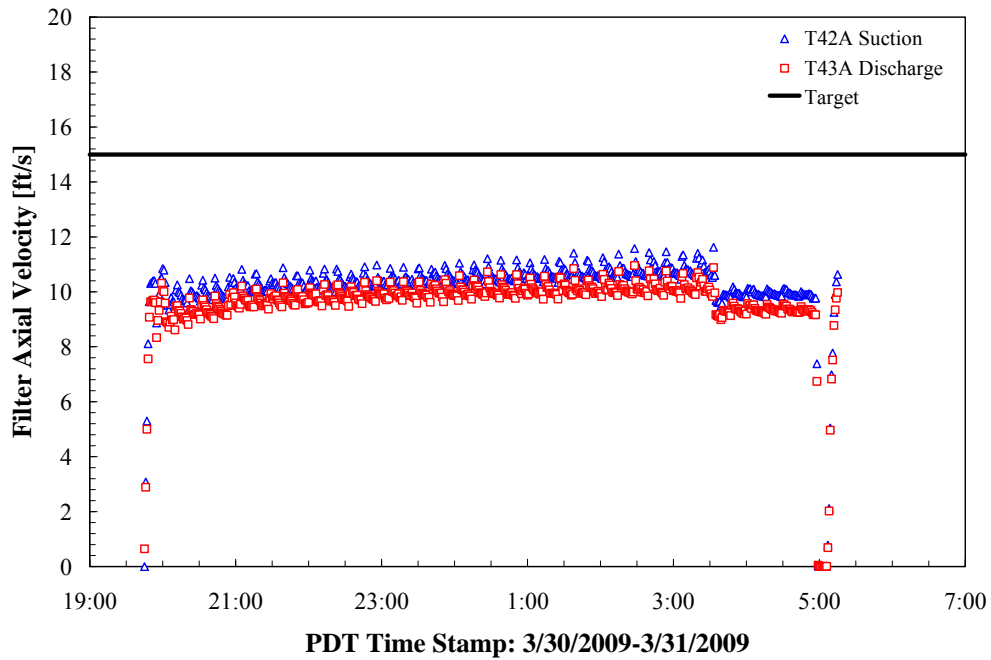


Figure 4.26. Axial Velocity Based on Flow Measurements at the Suction to T42A and the Discharge to T43A for Post-Oxidative-Leach Wash

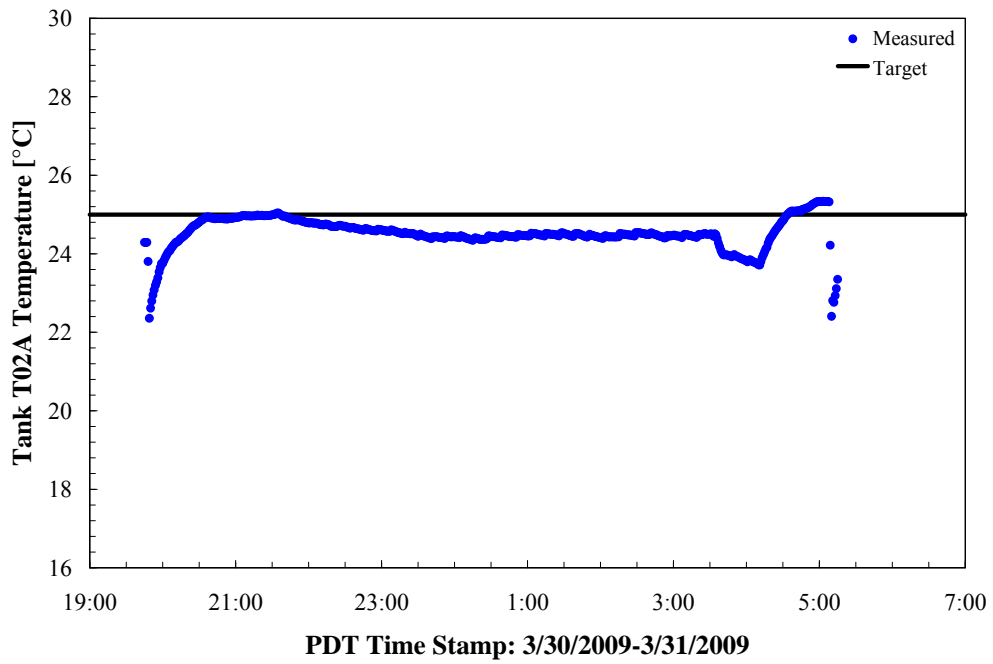


Figure 4.27. Tank T02A Temperature for Post-Oxidative-Leach Wash

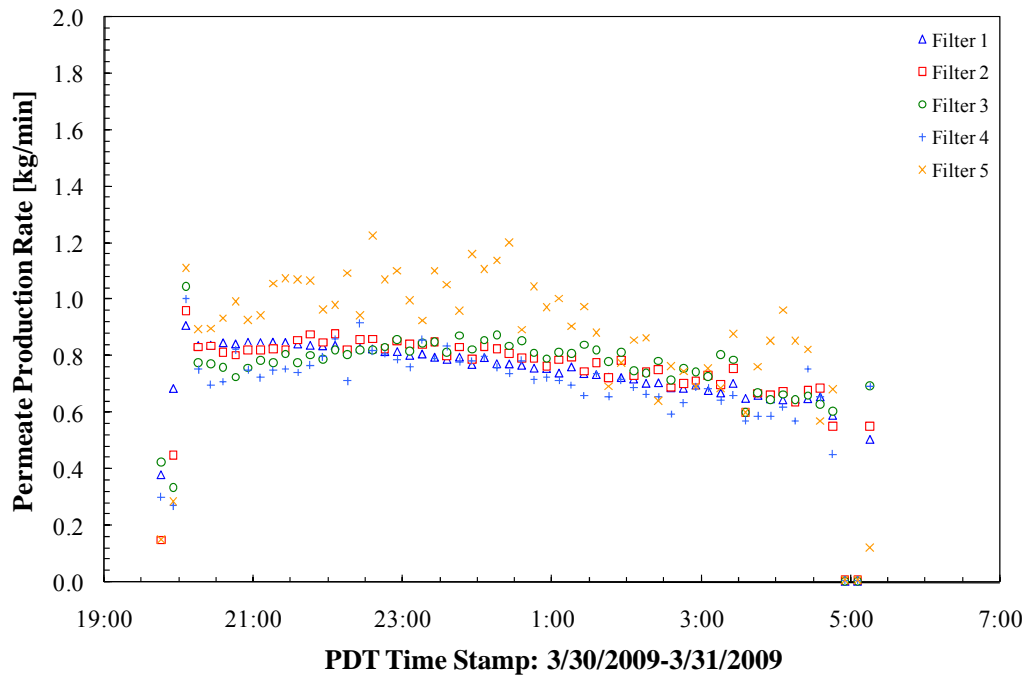


Figure 4.28. Permeate Production Rate for Post-Oxidative-Leach Wash

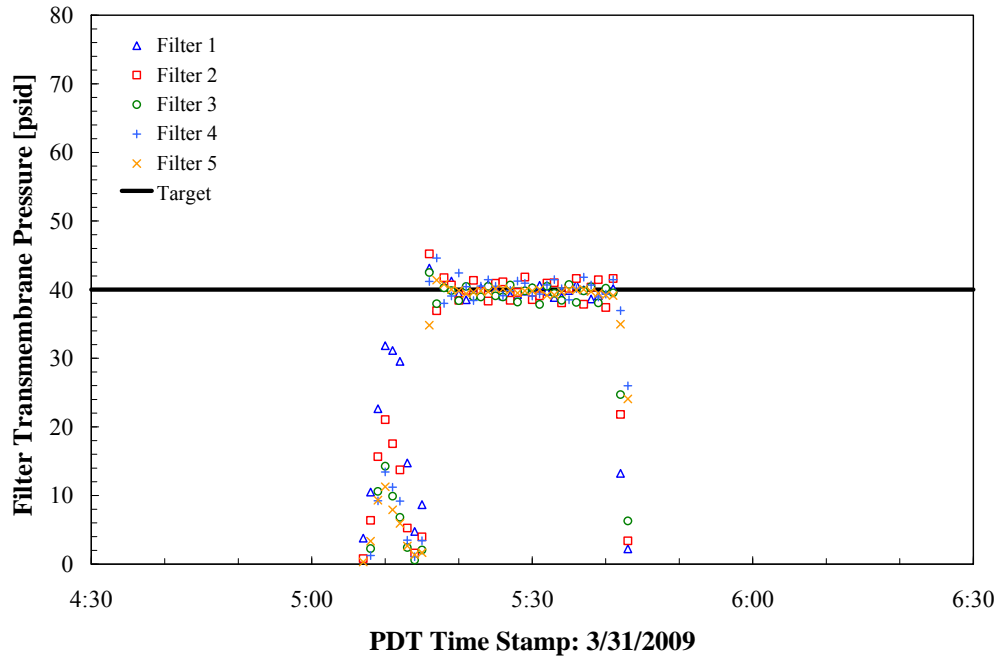


Figure 4.29. TMP for Post-Oxidative-Leach Concentration

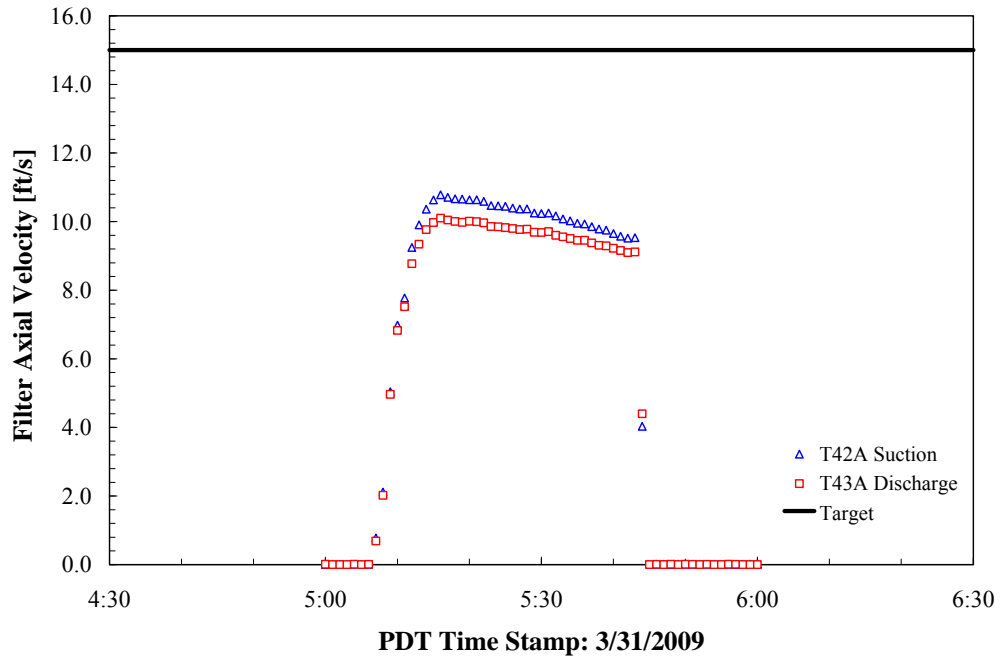


Figure 4.30. Axial Velocity Based on Flow Measurements at the Suction to T42A and the Discharge to T43A for Post-Oxidative-Leach Concentration

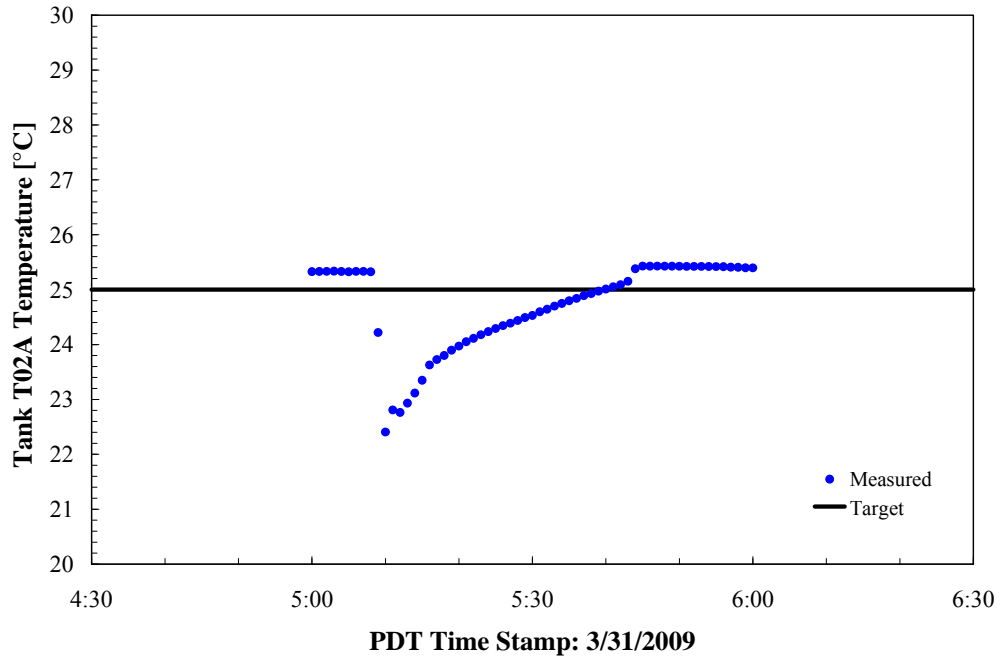


Figure 4.31. Tank T02A Temperature for Post-Oxidative-Leach Concentration

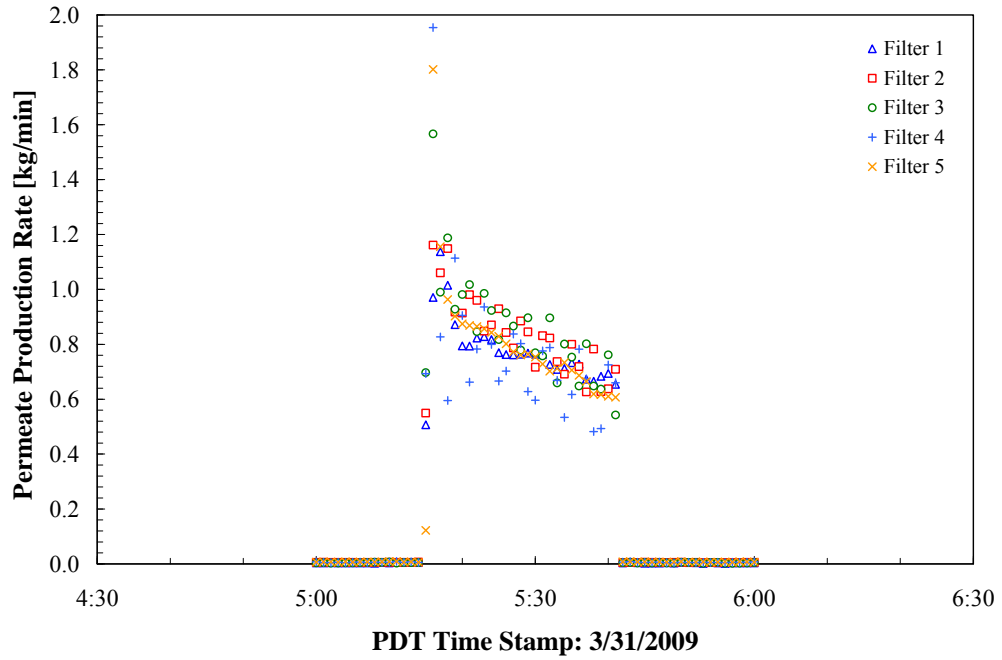


Figure 4.32. Permeate Production Rate for Post-Oxidative-Leach Concentration

4.10 Nitric Acid Filter Cleaning After Integrated Test D

At the conclusion of Integrated Test D, the tank, pumps, and filters were flushed out with IW twice to prepare for cleaning with nitric acid. The Tank T02A vessel was inspected before introducing the IW with a small video camera inserted through a flange in the top of the vessel (DVD titled “UFP-02A tank inspection” on 4/23/09). The video included inner tank walls, bottom head, upper vessel head, steam pipe and ring (top and bottom), and other internals (look for bathtub ring and donuts on internals, especially near upper simulant levels).

4.10.1 Visual Inspection Results

A visual inspection was performed at the inlet to ultrafilter UFP-FILT-T01A. The inspection was performed on 4/23/09, after Phase I, Integrated Test D. Valve V07128 was removed from the filter-loop to provide access to the inlet of Filter T01A.

The conditions of the filter-loop before the video were as follows:

- Simulant slurry remaining in the filter-loop at the end of Integrated Test D was drained to totes (3/31/09).
- A single 0.01-M IW flush of the filter-loop and Tank T02A was performed.
- The filters were laid up in 0.01-M IW after the flush.
- Filter T01A was drained just before the video.

The visual inspection consisted of two parts. The first part was performed using a Mirion C911 Series inspection camera. The camera was connected to a Dell flat-panel monitor and Panasonic DMR-EZ48V DVD recorder. This part of the inspection was recorded to a DVD. The camera was inserted into the filter inlet and positioned by hand to observe features of the filter head.

The second part of the inspection was performed using an Olympus 1F8S2-20 fiberscope and ILK-4 cold light supply. This part of the inspection was not recorded. The fiberscope was also inserted into the filter inlet and positioned by hand to observe features of the filter head. No unique observations were noted during the inspection with the fiberscope.

The inlet to Filter T01A (piping and filter head) had a light brown discoloration as shown in Figure 4.33 that was due to residual simulant film on the metal surfaces. Darker areas of heavier simulant deposition were also observed on the filter head. No significant solids buildup was observed at the filter inlet. The unused spare filter is pictured in Figure 4.34.

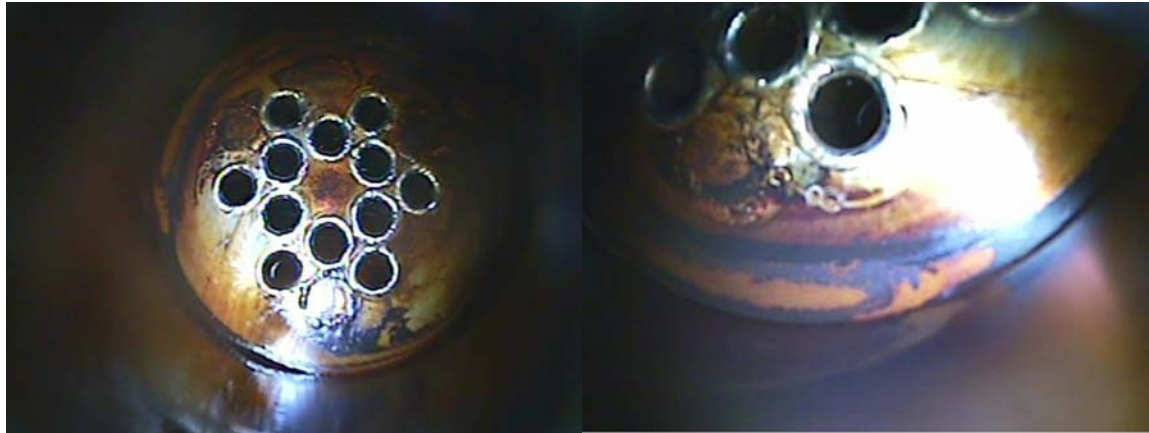


Figure 4.33. Inspections of Filter T01 Inlet

Several small (approximately $\frac{1}{2}$ to 1 mm) reflective beads were observed near the inlet of the individual ultrafilter tubes. The beads observed near the inlet to the individual ultrafilter tubes appear to be stray weld material from the fabrication process and do not appear to be large enough to affect filter performance. The 8-ft spare ultrafilter shown in Figure 4.34 was inspected for comparison and also found to have reflective beads near the ultrafilter tube inlet.



Figure 4.34. Inspections of 8-ft Spare Ultrafilter

In conclusion, no significant solids were observed at the inlet to UFP-FILT-T01A during the 4/23/09 inspection and the visual inspection did not identify any indications of flow mal-distribution or evidence of potential filter failure.

4.10.2 Cleaning

After the inspection, the tank was filled with 300 gallons of IW to obtain baseline filtration rates.

After the IW rinses, the IW drained from the loop appeared to be dirty, and samples were taken for analyses. The UDS analyses showed 0.25-wt% solids. The solution was dark brown when shaken, and when settled, there was a ~2% layer of solids on the bottom. The oven-dried solids were 10.8-wt% of the centrifuged solids. The permeate sample from Filter T01A was yellow with little visible solids, but the

sample from the shellsides of the other filters was brown and contained visible solids. After the shellside samples were allowed to settle, the supernatant was yellow, and there was a thin layer of solids on the bottom of a 1-L container as shown in Figure 4.36.

The nitric acid cleaning started with approximately 200 gallons in Tank T02A, and this proceeded as described in the *Steps for PEP Prototypic Filter Cleaning* instructions below. Samples were taken from the filter-loop during each cleaning evolution and analyzed. The third cleaning solution turned brown because of particulate precipitation when neutralized with IW and 2M NaOH. The first two cleaning solutions had visible solids while the third one had very little solids. The UDS, pH, and total solids results are provided in Table 4.1. The slurry samples were also analyzed by ICP, and the results are shown in Figure 4.37 and Figure 4.38.

Table 4.1. Filter-Cleaning Sample Results

Sample Description	UDS wt%	Ave pH	Total Solids wt%	Observations
Dirty water filter-loop sample	0.25	12.59	1.52	Significant layer of solids on bottom of sample container
Permeate sample dirty water	0	12.57	1.45	Permeate yellow, no visible solids
Permeate sample filter shell sample after dirty-water flux	0.01	12.56	1.61	Permeate yellow, thin layer of settle solids on the bottom of 1-L container
1st acid wash filter-loop sample	0.09	0.12	0.56	Settled solids visible in sample carboy. Solution dark brown when mixed.
2nd acid wash filter-loop sample	0.02	0.06	0.13	Solids visible in sample carboy.
3rd acid wash filter-loop sample	0.00	0.05	0.10	Yellow supernatant with no visible solids in large carboy. Solids visible when neutralized.
Before final pH adjustment filter-loop sample	0.00	1.86	1.6	
Before final pH adjustment permeate sample	0.00	1.83	1.63	
pH ~13 filter-loop sample	0.04	12.07	2.03	
pH ~13 permeate sample	0	12.08	1.99	



Figure 4.35. Picture of Filter Shellside After Settling

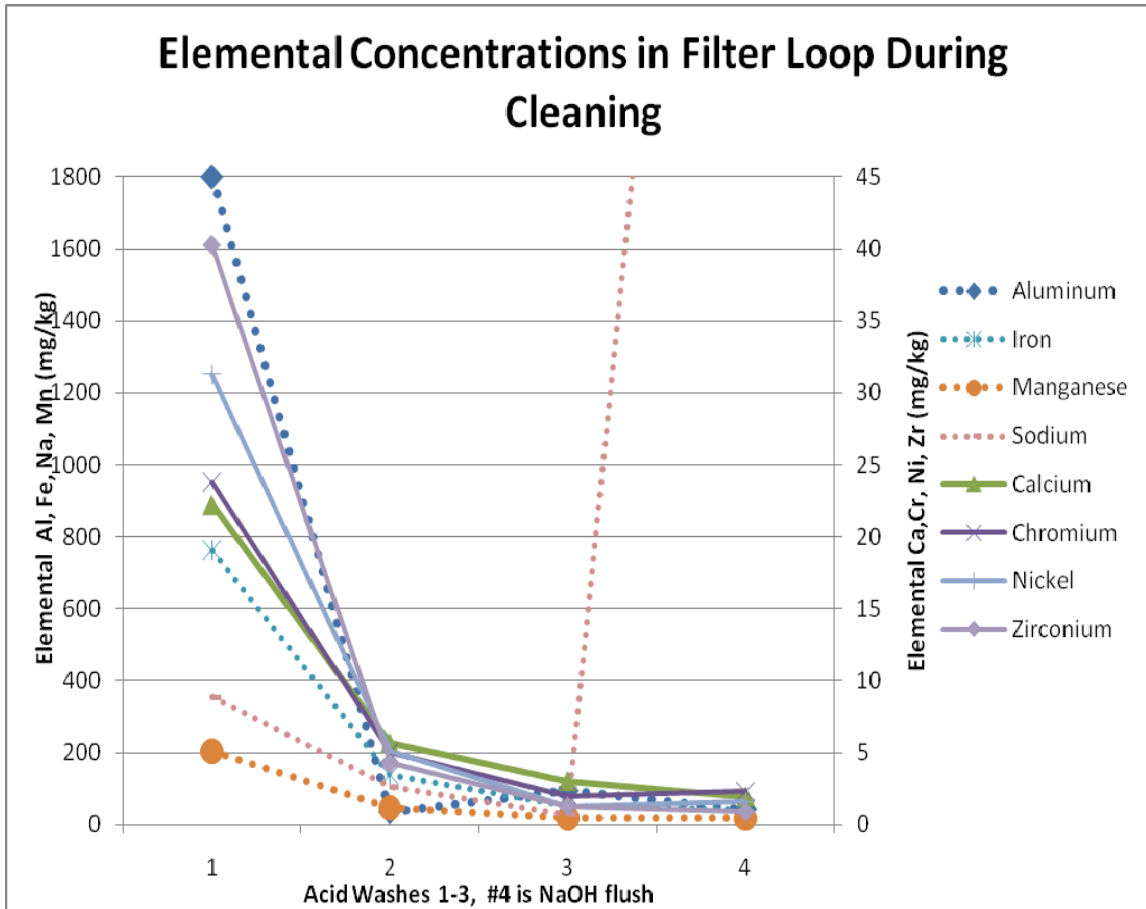


Figure 4.36. Elemental Concentrations During Filter Cleaning

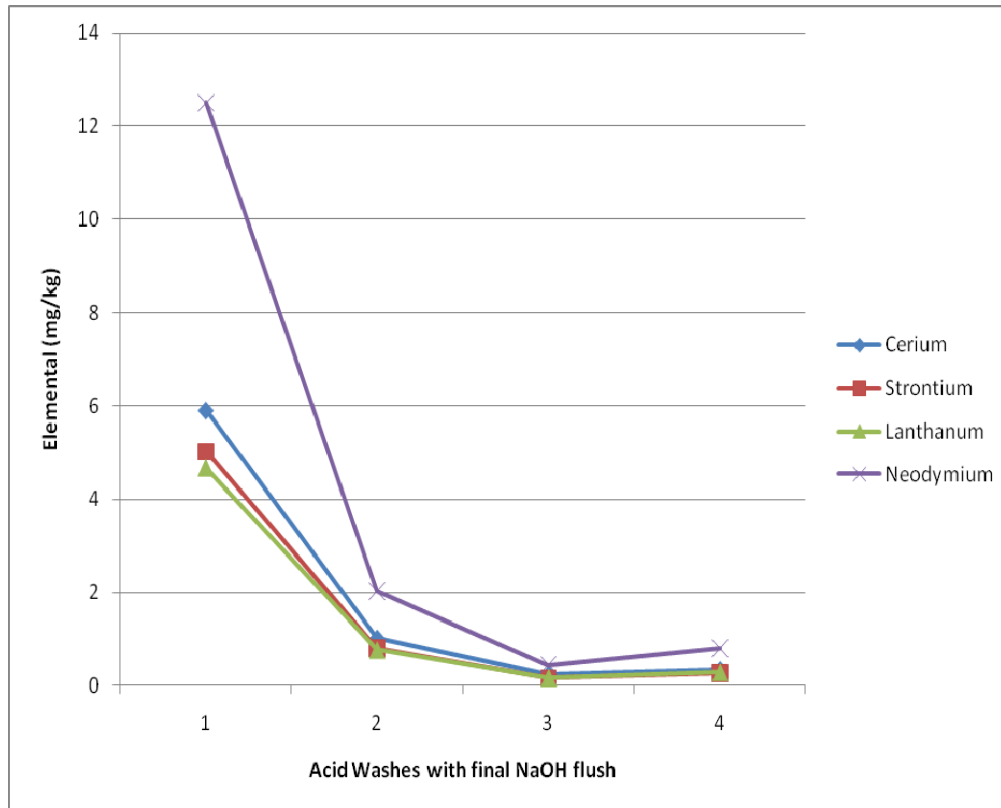


Figure 4.37. Trace Element Concentrations During Filter Cleaning

Temperature corrected Filter flux

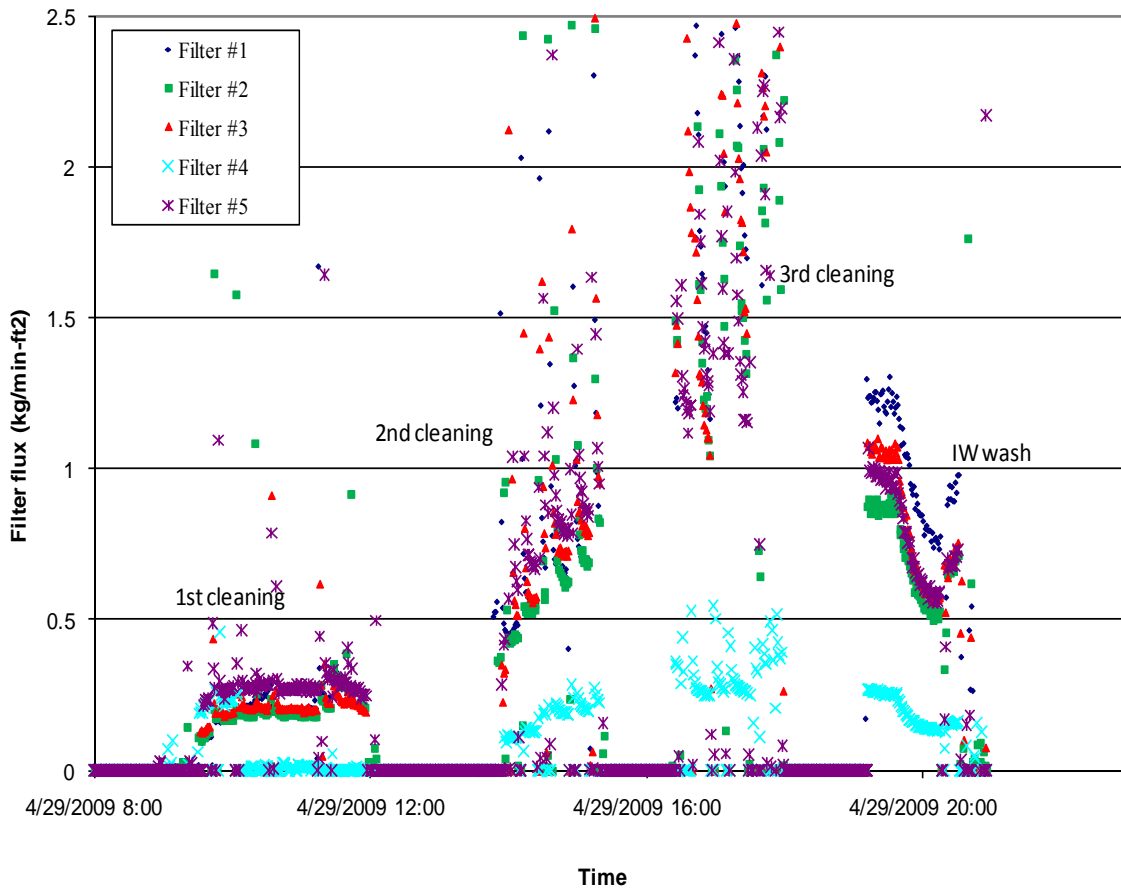


Figure 4.38. Temperature and TMP-Corrected Filter Flux

Selected centrifuged solids collected from the first IW filter-loop sample (6501) and the filter shell sample (6524) were also analyzed. The solids material from the filter shellside was mainly manganese with quantities of zirconium, and zinc significantly above the solids concentration of material in the filter-loop as shown in Table 4.2. It is probable that these solids are oxalates that formed during washing activities and did not redissolve. The solids may have deposited in dead legs. The particle size distribution for the solids collected from the permeate sample before cleaning started was significantly larger than the leached slurry and more comparable to the original slurry. The values are provided in Table 5.18.

The UDS content in the material collected from the filter-loop during the three washes decreased during each successive wash but overall composition did not change. The solids were mainly aluminates estimated as >90% of the total solids weight. There were also small amounts of chromium, iron, silicon, and zirconium. The overall quantity of solids was small as indicated by the UDS measurements.

Table 4.2. Draining Results

Section of Filter-Loop Drained	Amount Drained
Tubeside	518 lbs
Spiral Heat Exchanger	559 lbs cumulative
After 1 st and 2 nd shellside drain	1226 lbs cumulative
After 2 nd filter-loop side drain	1288 (154 gal) cumulative
Spiral HX volume	4.9 gallons drained
Pulse-pots and shell volume	79.6 gallons drained
Tubeside volume	69.2 gallons
Filter-loop bypass drained (not part of cumulative total)	5.4 gallons

The cleaning worked well with permeate rates increasing from the baseline IW rate of 0.1 kg/min-ft² to values approaching 0.2, 0.8, and 1.5 kg/min-ft², respectively, for each wash as shown in Figure 4.38. The final IW water rate was approximately 0.8 kg/min-ft². Filter #4 rates were low, but it appears that the backpulse valve was leaking or the flow meter was incorrect, creating low flows through the flow meter. The leak was indicated by the calculated permeate rates based on the pulse-pot filling rate after a backpulse and the similarity of filling rate compared to the other pulse pots.

During the cleaning, the PJMs were off (except when specified), the lower air spargers, and the steam ring air purge were off during all cleaning steps. Initially, the lower air spargers were on at idle flow, but filter-loop pumping difficulties caused them to be turned off. The cooling heat exchanger had a 45-degree outlet setpoint. After refilling the filter-loop and pulse-pots, the tank level was ~16 inches (WTP=72 inches). The TMP was set at 40 psi, but if 40 psi could not be met, a lesser TMP was acceptable. The backpulsing pressure set point was 40 psi above the tubeside. The filter-loop backpressure valve started at 50% open but was adjusted to 1% to obtain 110 psi pressure and 100 gpm.

Steps for PEP Prototypic Filter Cleaning

1. Fill UFP-VSL-T02A with IW to the 300-gallon level. *This step is not prototypic. In the WTP, the vessel would be filled with IW to rinse out the solids, but not after sitting for a long period of time.* **Completed.**
2. Verify that all five filters and the cooling heat exchanger are aligned. Open V06132 and recirculate UFP-VSL-T02A contents at 70 gpm, with all five permeate valves open and aligned to UFP-VSL-T02A. Backpulse as soon as the pulse-pots are full. Recirculation should continue until the permeate rate through each filter has stabilized. This is the baseline flux. *This step is not prototypic.* **Completed.**
3. Close the permeate valves and turn off pumps. *This step is prototypic.*
4. Isolate UFP-VSL-T02A by closing V06132. *This step is non-prototypic.*
5. Drain the filter-loop into the empty tote to measure the effectiveness (volume) of the drain. The filter-loop drain should include the slurry (tubeside) of filters, the spiral heat exchanger via the sample port on the north end of the skid, pulse-pots, and shellsides of the filters. Record the tube side/spiral HX volume as one total volume and the filter shell and pulse-pot volume as another total. *This step is prototypic.*
6. The volume of the filter-loop, pulse-pots, and shells is 182 gallons. Subtract the volume in the tote from 182 gallons to obtain the volume remaining in the filter-loop. Subtract the volume remaining in the filter-loop from 33 gallons, and leave this volume in the tank after draining UFP-VSL-T02A. The sum of the volume in the tank and the volume in the filter-loop should equal ~33 gallons. *This step will give prototypic nitric acid dilution, as expected, in the WTP.* **The actual calculated volume left in the filter-loop was 28 gallons.**
7. Fill UFP-VSL-T02A with 207 gallons of 2-M nitric acid. *This step is prototypic.* **Completed.**
8. Take stable level measurement. *This step is not prototypic.* The stable tank level should be approximately 49 inches. **The level measurement was 50.4 inches and 212 gallons. NAR vessel indicates 199 gallons transferred.**
9. Run PJMs for 1 hour at ~7.3 m/s, with a 33-sec cycle time and an 80% stroke. Take a sample from UFP-VSL-T02A to determine nitric acid concentration and UDS. **Samples taken.**
10. Turn PJMs off, open V06132, and begin recirculating the nitric acid at 70 gpm, with all five permeate valves open and aligned to UFP-VSL-T02A, for 1.5 hours. *This step is prototypic.* During this step, the permeate rates should be monitored. Each filter should be backpulsed as soon as filtration begins (when pulse-pot level is >10 inches) and at 0.5, 1, and 1.5 hours. Take a 5-gallon sample near the end of 1.5 hours from the filter-loop to determine nitric acid concentration and UDS, and complete possible precipitation study. **Nitric acid recirculated at 109 gpm.**
11. Close the permeate valves and turn off the pumps. *This step is prototypic.*
12. Isolate UFP-VSL-T02A by closing V06132. *This step is non-prototypic.* **Completed.**
13. Take stable level measurement. *This step is non-prototypic. The difference in stable level measurements between steps 12 and 17 should equal the volume drained during step 5.* **Completed.**
14. Drain UFP-VSL-T02A (leaving the same volume in the tank as was left in Step 6), and drain the filter-loop. The pulse-pots and shellsides of the filters should be drained. *This step is prototypic.* **Completed.**
15. Fill UFP-VSL-T02A with 207 gallons of 2-M nitric acid. *This step is prototypic.* **Filled with 229 gallons.**

16. Take stable level measurement. *This step is not prototypic.* The stable tank level should be approximately 49 inches. **Level at 54.1 inches.**
17. A second nitric acid wash will begin. Recirculate the nitric acid at 70 gpm, with all five permeate valves open and recycled to UFP-VSL-T02A for 1.5 hours. *This step is prototypic.* (Note: A third nitric acid step may be needed after looking at the permeate flow rates during the first two nitric acid steps.) During this step, the permeate rates should be monitored. Each filter should be backpulsed as soon as filtration begins (when the pulse-pot level is >10 inches) and at 0.5, 1, and 1.5 hours. Take a 5-gallon sample near the end of 1.5 hours from the filter-loop to determine nitric acid concentration and UDS, and complete the possible precipitation study. **The recirculation rate was 100 to 110 gpm, and a sample was obtained.**
18. Close the permeate valves and turn off pumps. *This step is prototypic.*
19. Isolate UFP-VSL-T02A by closing V06132. *This step is non-prototypic.*
20. Take stable level measurement. *This step is non-prototypic.* The difference in stable level measurements between steps 20 and 24 should equal the volume drained during step 5. **Level 18.4 inches (69 gallons).**
21. Drain UFP-VSL-T02A (leaving the same volume in the tank as was left in Step 6) and drain the filter-loop. The pulse-pots and shellsides of the filters should be drained. The contents can be drained to UFP-VSL-T62A or B. *This step is prototypic.*
22. Fill UFP-VSL-T02A with 207 gallons of 2-M nitric acid. *This step is prototypic.* **Added 219 gals--level was 52 inches.**
23. Take stable level measurement. *This step is not prototypic.* The stable tank level should be approximately 49 inches.
24. A third nitric acid wash will begin. Recirculate the nitric acid at 70 gpm, with all five permeate valves open and recycled to UFP-VSL-T02A for 1.5 hours. *This step is prototypic.* During this step, the permeate rates should be monitored. Each filter should be backpulsed as soon as filtration begins (when the pulse-pot level is >10 inches) and at 0.5, 1, and 1.5 hours. Take a 5-gal sample near the end of 1.5 hours from the filter-loop to determine the nitric acid concentration and UDS, and complete the possible precipitation study. **Recirculated solution at ~109 gpm, and samples were taken.**
25. Close the permeate valves, and turn off the pumps. *This step is prototypic.*
26. Isolate UFP-VSL-T02A by closing V06132. *This step is non-prototypic.*
27. Take stable level measurement. *This step is non-prototypic.* **Tank T02A level was 17.4 inches (64 gallons).**
28. Drain UFP-VSL-T02A (leaving the same volume in the tank as was left in Step 6, and drain the filter-loop. The pulse-pots and shellsides of the filters should be drained. The contents can be drained to UFP-VSL-T62A or B. *This step is prototypic.* **Completed.**
29. Fill UFP-VSL-T02A with 173 gallons of IW and 34 gallons of 2-M sodium hydroxide. *This step is prototypic.* **Completed—Tank T02A level was 51.4 inches (217 gallons).**
30. Recirculate the contents at 70 gpm, with all five permeate valves open and aligned to UFP-VSL-T02A, for 1 hour. *This step is prototypic.* During this step, the permeate rates should be monitored. **Do not backpulse.** Sample the permeate every 10 minutes, test, and record the pH using pH paper. Take a sample from the filter-loop and each of the permeate sample ports at the end to determine the pH and the UDS. The pH target is to be greater than 12. **Recirculated solution at ~109 gpm, and samples were taken. The pH started out at 13, but decreased to 2 in 40 minutes.**

An additional 50 kg of 2-M NaOH was added to the tank, and the pH was >12. The sample results of the filter cleaning are provided in Table 4.1.

31. Close the permeate valves, and turn off the pump. Leave the filter-loop full and wetted.
32. Isolate vessel UFP-VSL-T02A (close V06132), and drain the Tank T02A vessel to UFP-VSL-T62A or B. **The SLM was 16.2 inches before draining.**
33. Inspect the vessel, and take a video of the tank, steam ring, and all internals to determine the effectiveness of cleaning tank internals. **Complete.**

5.0 Data

Selected data obtained from the DAS during Integrated Test D have been plotted in 12-hour intervals and are provided in Appendix J tables summarizing the results of sample measurements taken during testing. These data 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 PEP Phase 1 Final Test Report, WTP-RPT-197. The PJM peak average velocity and stroke length, tuned iteratively to target values during testing, are provided in Section 5.3.

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 C, 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 the tare sample vial, the vial plus total slurry, and the vial plus wet centrifuged solids or the calculated value of the wt% 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 slurries of the original samples. 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 and Table 5.2 provide the wt% UDS for slurry samples collected during the concentration of the first and second batch of slurry (respectively) before caustic leaching in Tank T02A. The wt% wet centrifuged solids in the slurry can be used in calculating the total species concentration in the slurries of the original samples. 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 5.3, Batch 1: Leach, and Table 5.4, Batch 2: Leach, respectively, provide analytical results for samples collected during the Batch 1 and Batch 2 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. The wt% wet centrifuged solids in the slurry can be used in calculating the total species concentration in the slurries of the original samples. 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.

Raman results, shown in Table 5.5 and Table 5.6, are provided as part of the liquid analyses and presented in molar concentration (mole/liter).

Table 5.1. Caustic-Leach Batch 1: Initial Solids Concentration to 20-wt%

Sample Location: Filter-Loop Condition: CD Pump Repair	wt% UDS (slurry)	Cr in liquid (µg/g)	Cr in Wet Centrifuged Solids (µg/g)
Transfer to Tank T01B	5.00	—	5493^(b)
Prior to Concentration	6.69	—	—
After Batch 3	6.41	—	—
After Batch 6	6.98	—	—
After Batch 9	7.78	—	—
After Batch 12	8.81	—	—
After Batch 15	9.23	—	—
After Batch 18	9.88	—	—
After Batch 21	10.7	—	—
After Batch 24	11.5	—	—
After Batch 27	12.2	—	—
After Batch 30	13.0	—	—
After Batch 33	13.7	—	—
After Batch 36	14.5	—	—
After Batch 39	15.2	—	—
After Batch 42	16.0	—	—
After Batch 45	16.7	—	—
After Concentration ^(a)	23.4	43.6	8307^(c)
After Caustic Addition and Filter Flush	8.00	566	4770 ^(d)

(a) Analytical results duplicated in Analysis at Key Process Steps table in column Post Concentration of Solid Batch #1.

(b) The wt% centrifuged solids value for this step is 14.8%.

(c) The wt% centrifuged solids value for this step found in Analysis at Key Process Steps table in column Post Concentration of Solid Batch #1.

(d) The wt% centrifuged solids value for this step found in Batch 1: Caustic Leach in row after NaOH.

— Analysis not required by Test Plan.

Bold numbers are calculated values.

Table 5.2. Caustic-Leach Batch 2: Initial Solids Concentration to 20-wt%

Sample Location: Filter-Loop Condition: CD Pump Repair	wt% UDS (slurry)	Cr in liquid (µg/g)	Cr in Wet Centrifuged Solids (µg/g)
Transfer to Tank T01B	5.21	—	5040^(b)
Prior to Concentration	6.35	—	—
After Batch 3	6.44	—	—
After Batch 6	7.44	—	—
After Batch 9	8.60	—	—
After Batch 12	8.58	—	—
After Batch 15	9.16	—	—
After Batch 18	9.99	—	—
After Batch 21	10.40	—	—
After Batch 24	11.40	—	—
After Batch 27	12.30	—	—
After Batch 30	13.00	—	—
After Batch 33	13.60	—	—
After Batch 36	14.70	—	—
After Batch 39	15.20	—	—
After Batch 42	15.40	—	—
After Batch 45	16.30	—	—
After Concentration ^(a)	23.83	46.9	7240^(c)
After Caustic Addition and Filter Flush	6.63	179	4320 ^(d)

(a) Analytical results duplicated in Analysis at Key Process Steps table in column Post Concentration of Solid Batch #2.

(b) The wt% centrifuged solids value for this step is 15.3%.

(c) The wt% centrifuged solids value for this step found in Analysis at Key Process Steps table in column Post Concentration of Solid Batch #2.

(d) The wt% centrifuged solids value for this step found in Batch 2: Caustic Leach in row After NaOH.

— Analysis not required by Test Plan.

Bold numbers are calculated values.

Table 5.3. Batch 1: Caustic-Leach^(a)

Sample Location: Tank T02A, Condition:	Slurry Analyses						Liquid Analyses								
	UDS wt%	Slurry Density g/mL	Al µg/g	Fe µg/g	Sr µg/g	Wt-% Cent Solids wt%	Liquid Density g/mL	Al µg/g	Cr µg/g	Free OH ⁻ M	C ₂ O ₄ µg/g	Cl ⁻ µg/g	NO ₃ µg/g	NO ₂ µg/g	SO ₄ µg/g
After NaOH	8.00	1.468	31913	9485	91	23.83	1.383	13800	566	8.78	<19.0	434	37200	8780	800
75°C	7.76	1.450	—	—	—	23.45	1.391	12500	557	8.37	21.8	422	35900	8450	1440
85°C - hour 0	7.07	1.439	29599	8950	86	22.66	1.372	11933	617	8.03	30.2	403	34600	8113	2403
85°C - hour 1	6.63	1.436	—	—	—	22.74	1.377	12300	742	7.83	27.4	403	34300	8110	2570
85°C - hour 2	6.82	1.435	—	—	—	21.88	1.377	12500	825	7.81	28.6	394	34000	8030	2700
85°C - hour 4	6.79	1.434	29025	8969	85	21.30	1.378	12600	976	7.48	30.5	403	33900	8060	3080
85°C - hour 8	6.32	1.428	29064	8758	83	19.64	1.373	13100	1220	7.56	37.0	391	32600	7650	3680
85°C - hour 10	6.06	1.422	—	—	—	18.79	1.360	13300	1300	7.38	29.5	389	32900	7710	4030
85°C - hour 12	5.85	1.405	27947	8507	80	18.74	1.366	13800	1390	7.12	37.5	381	32300	7630	4430
85°C - hour 14	5.54	1.402	—	—	—	17.61	1.364	12500	1270	6.81	30.5	372	31300	7420	5110
85°C - hour 16	5.61	1.398	27057	8415	78	16.80	1.362	13200	1360	7.30	33.3	376	31500	7400	5460
85°C - hour 18	5.18	1.406	—	—	—	16.95	1.355	13500	1350	7.04	34.2	363	31000	7320	5820
85°C - hour 20	4.98	1.400	—	—	—	16.29	1.349	13800	1350	6.79	32.4	358	30500	7200	6200
85°C - hour 22	5.09	1.404	—	—	—	16.50	1.359	13900	1340	6.75	33.3	363	30600	7200	6330
85°C - hour 24	5.03	1.394	26505	8007	75	15.55	1.356	14067	1347	6.84	34.2	358	30267	7120	6253
After cool down to 25°	5.02	1.402	27805	8358	79	15.09	1.357	14800	1400	6.78	31.0	374	30900	7290	6040

(a) Ca, Mg, and Nd are not provided in this table because they were not used in the caustic-leach data analysis.
 — Analysis not required by Test Plan.
Bold numbers are calculated values.
 M—Molarity.

Table 5.3 (contd)

Sample Location: Tank T02A, Condition:	Wet Centrifuged Solids Analyses					Solids Content %
	Al μg/g	Cr μg/g	Fe μg/g	Na μg/g	Sr μg/g	
After NaOH	89800	4770	39800	157000	380	62.79
75°C	—	—	—	—	—	—
85°C - hour 0	89900	4283	39500	144333	377	63.64
85°C - hour 1	—	—	—	—	—	—
85°C - hour 2	—	—	—	—	—	—
85°C - hour 4	89700	3210	42100	133000	398	64.94
85°C - hour 8	94400	2210	44600	137000	422	60.73
85°C - hour 10	—	—	—	—	—	—
85°C - hour 12	89300	1460	45400	126000	427	61.92
85°C - hour 14	—	—	—	—	—	—
85°C - hour 16	95700	1170	50100	114000	465	60.82
85°C - hour 18	—	—	—	—	—	—
85°C - hour 20	—	—	—	—	—	—
85°C - hour 22	—	—	—	—	—	—
85°C - hour 24	94067	1087	51500	119667	485	57.92
After cool down to 25°	101000	1120	55400	115000	524	59.26

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

— Analysis not required by Test Plan.

Bold numbers are calculated values.

M—Molarity.

Table 5.4. Batch 2: Caustic-Leach^(a)

Sample Location: Tank T02A, Condition:	Slurry Analyses						Liquid Analyses								
	UDS wt%	Slurry Density g/mL	Al μg/g	Fe μg/g	Sr μg/g	Wt-% Cent Solids wt%	Liquid Density g/mL	Al μg/g	Cr μg/g	Free OH ⁻ M	C ₂ O ₄ μg/g	Cl ⁻ μg/g	NO ₃ μg/g	NO ₂ μg/g	SO ₄ μg/g
After NaOH	6.63	1.489	30289	8864	83	32.12	1.418	12400	179	9.64	26.6	366	31200	7340	1080
75°C	6.69	1.474	—	—	—	30.96	1.403	12900	229	9.06	<19.5	364	32000	7550	1650
85°C - hour 0	6.36	1.462	30816	8920	84	27.25	1.392	13367	381	8.83	34.4	374	31667	7430	1337
85°C - hour 1	6.14	1.452	—	—	—	27.16	1.392	13600	541	6.72	45.6	374	31300	7330	1540
85°C - hour 2	6.19	1.427	—	—	—	26.44	1.378	13600	663	8.64	27.6	369	31200	7340	1690
85°C - hour 4	5.89	1.421	30888	9077	86	24.40	1.377	14100	957	8.45	31.6	367	31100	7300	1920
85°C - hour 8	5.76	1.441	29001	8425	80	23.47	1.379	13300	1180	8.25	27.4	351	30200	7140	2450
85°C - hour 10	5.89	1.433	—	—	—	21.78	1.379	13600	1300	7.91	27.0	328	28700	6790	2620
85°C - hour 12	5.96	1.432	29083	8691	82	20.99	1.380	13800	1380	7.51	29.7	333	28800	6780	2900
85°C - hour 14	5.69	1.425	—	—	—	20.44	1.373	13900	1390	7.46	26.7	327	28400	6620	3140
85°C - hour 16	5.48	1.421	28356	8245	79	19.40	1.376	14000	1390	7.90	28.5	315	27400	6500	3460
85°C - hour 18	5.23	1.415	—	—	—	19.29	1.371	14300	1400	7.56	24.4	322	28400	6690	3930
85°C - hour 20	4.90	1.415	—	—	—	18.65	1.365	14200	1360	7.26	33.5	308	27100	6390	4120
85°C - hour 22	4.42	1.407	—	—	—	18.62	1.365	14300	1350	7.31	30.2	307	27100	6380	4540
85°C - hour 24	4.74	1.399	27398	8190	77	17.13	1.365	14433	1337	7.64	32.1	310	26733	6323	5013
After cool down to 25°	5.40	1.411	27947	8487	79	17.04	1.360	14500	1340	6.90	25.4	314	27200	6340	4350

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

— Analysis not required by Test Plan.

Bold numbers are calculated values.

M—Molarity.

Table 5.4 (contd)

Sample Location: Tank T02A, Condition:	Wet Centrifuged Solids Analyses					Solids Content %
	Al μg/g	Cr μg/g	Fe μg/g	Na μg/g	Sr μg/g	
After NaOH	68100	4320	27600	162000	258	57.82
75°C	—	—	—	—	—	—
85°C - hour 0	77400	4640	32733	148667	308	59.38
85°C - hour 1	—	—	—	—	—	—
85°C - hour 2	—	—	—	—	—	—
85°C - hour 4	82900	3420	37200	142000	353	58.51
85°C - hour 8	80200	2090	35900	141000	340	61.56
85°C - hour 10	—	—	—	—	—	—
85°C - hour 12	86600	1450	41400	136000	389	61.25
85°C - hour 14	—	—	—	—	—	—
85°C - hour 16	88000	1210	42500	131000	409	63.47
85°C - hour 18	—	—	—	—	—	—
85°C - hour 20	—	—	—	—	—	—
85°C - hour 22	—	—	—	—	—	—
85°C - hour 24	90100	1177	47800	122333	450	56.83
After cool down to 25°	93400	1180	49800	127000	462	57.77

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

— Analysis not required by Test Plan.

Bold numbers are calculated values.

M—Molarity.

Table 5.5. Batch 1: Caustic Leach—Raman Results

Sample Location: Tank T02A, Condition:	Al(OH) ₄ ⁻ [M]	Free OH- [M]	CrO ₄ ⁻² [M]	C ₂ O ₄ [M]	NO ₃ [M]	NO ₂ [M]	SO ₄ [M]	PO ₄ [M]
85°C—hour 0	0.471	8.238	0.018	<0.01	0.774	0.273	0.033	0.036 ^(a)
	0.478	8.282	0.018	<0.01	0.782	0.270	0.033	0.034 ^(a)
	0.475	8.236	0.018	<0.01	0.784	0.264	0.034	0.039 ^(a)
85°C—hour 12	0.536	7.385	0.041	<0.01	0.724	0.239	0.062	0.031 ^(a)
85°C—hour 16	0.539	7.112	0.041	<0.01	0.695	0.244	0.074	0.026 ^(a)
	0.561	6.686	0.040	<0.01	0.668	0.220	0.086	0.028 ^(a)
	0.570	6.652	0.040	<0.01	0.674	0.224	0.086	0.029 ^(a)
85°C—hour 24	0.569	6.740	0.040	<0.01	0.677	0.230	0.086	0.018 ^(a)

(a) Detected, results are qualitative: results > IDL but < EQL (estimated quantitation limit)

M—Molarity.

Table 5.6. Batch 2: Caustic-Leach—Raman Results

Sample Location: Tank T02A, Condition:	Al(OH) ₄ ⁻ [M]	Free OH- [M]	CrO ₄ ⁻² [M]	C ₂ O ₄ [M]	NO ₃ [M]	NO ₂ [M]	SO ₄ [M]	PO ₄ [M]
85°C—hour 0	0.528	9.002	0.011	<0.01	0.710	0.248	0.018	0.036 ^(a)
	0.536	9.027	0.011	<0.01	0.713	0.250	0.018	0.034 ^(a)
	0.527	9.069	0.011	<0.01	0.706	0.246	0.018	0.036 ^(a)
85°C—hour 12	0.581	8.022	0.047	<0.01	0.661	0.220	0.041	0.033 ^(a)
85°C—hour 16	0.595	7.694	0.047	<0.01	0.642	0.217	0.050	0.028 ^(a)
	0.611	7.226	0.044	<0.01	0.608	0.203	0.070	0.028 ^(a)
	0.608	7.238	0.044	<0.01	0.604	0.197	0.070	0.029 ^(a)
85°C—hour 24	0.620	7.266	0.045	<0.01	0.617	0.192	0.071	0.032 ^(a)

(a) Detected, results are qualitative: results > IDL but < EQL (estimated quantitation limit).

M—Molarity.

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 and 2 as described in Section 4. The wt% UDS and densities were obtained following analytical methods described in Section 3 for slurry not phase-separated. The wt% 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 5.7. Post-Caustic-Leach Concentration of Solids

Sample Location: Tank T02A, Condition:	Slurry			Liquid
	UDS wt%	Density ^(a) g/mL	Si μg/g	Density g/mL
Prior to Concentration	5.10	1.389	—	1.313
After Batch 2	5.50	1.394	—	1.304
After Batch 4	5.58	1.420	—	1.311
After Batch 6	6.07	1.418	—	1.320
After Batch 8	6.32	1.405	—	1.325
After Batch 10	6.69	1.401	—	1.294
After Batch 12	6.94	1.409	—	1.313
After Batch 14	7.29	1.413	—	1.338
After Batch 16	7.50	1.420	—	1.333
After Batch 18	8.03	1.418	—	1.327
After Batch 19	9.67	1.414	—	1.289
After Concentration ^(c)	18.8 ^(d)	1.496	855 ^(b)	1.351

(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 analysis are provided.

(b) Wet centrifuged solids. Slurry sample was phase separated at SwRI with the following information reported: percent total solids in the wet centrifuged solids was 59.41%; liquid fraction of slurry sample had 240-μg/g Si.

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

(d) Analysis rerun on sample D 02AML 012 XX 5362 SOX 2
— Analysis not required by Test Plan.

M—Molarity.

Table 5.8 through Table 5.10 provide 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 analyses. 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. 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. Raman results, shown in Table 5.10, are provided as part of the liquid analyses and presented in molar concentration (mole/liter).

Table 5.8. Post-Caustic-Leach Wash

Sample Location: Tank T02A	Slurry Analyses		Liquid Analyses										
	UDS wt%	Slurry Density (a) g/mL	Liquid Density (a) g/mL	Al μg/g	Cr μg/g	Free OH ⁻ M	Na μg/g	C ₂ O ₄ μg/g	NO ₃ μg/g	NO ₂ μg/g	SO ₄ μg/g	PO ₄ μg/g	TDS ^(b) wt%
After Wash 3	—	—	1.287	12300	1120	5.60	130000	88.1	23800	5650	6540	1550	31.00
After Wash 6	18.3	1.390	1.239	9320	883	4.35	104000	253	19000	4550	5280	2030	26.30
After Wash 8	—	—	1.180	8950	807	3.71	93500	414	16700	4020	4690	2050	22.50
After Wash 11	20.5	1.309	1.176	6590	627	2.92	74300	805	16400	3440	3750	1700	19.10
After Wash 13	—	—	1.130	6310	571	2.51	66000	1070	12600	3010	3340	1510	16.20
After Wash 16	22.0	1.238	1.123	4670	450	1.92	52000	1680	9880	2320	2610	1170	13.70
After Wash 19	—	—	1.081	4170	378	1.58	43500	2320	7980	1860	2140	936	10.80
After Wash 22	22.5	1.240	1.076	3000	293	1.22	34300	3410	6150	1440	1670	737	9.30
After Wash 24	—	—	1.050	2840	265	1.04	31200	4070	5320	1250	1470	640	8.13
After Wash 27	22.6	1.214	1.055	2090	199	0.79	24700	5440	3940	948	1100	461	7.00
After Wash 29	—	—	1.033	2050	181	0.65	23000	6360	3470	836	977	416	6.24
After Wash 32	21.8	1.199	1.042	1470	140	0.56	19100	8090	2590	635	747	312	5.42
After Wash 35	—	—	1.020	1380	120	0.46	17700	9910	2120	527	613	254	4.98
After Wash 37	22.0	1.187	1.069	1060	95.5	0.36	15600	11200	1700	430	495	208	4.83
After Wash 40	—	—	1.022	1020	86.0	0.36	15000	12100	1500	382	436	184	4.49
After Wash 43	21.3	1.165	1.026	718	60.1	0.23	14000	15100	1010	271	290	132	4.16
After Wash 45	—	—	1.016	744	58.8	0.22	13400	14500	967	256	279	118	4.10
After Wash 48	20.5	1.176	1.017	534	41.8	0.17	9310	10400	713	196	207	94.3	3.19
After Wash 50	—	—	1.009	541	40.9	0.16	9470	10300	681	187	193	79.8	2.88
After Final Wash ^(c)	22.0	1.167	1.016	397	30.2	0.12	6800	6970	523	149	142	65.4	2.19

(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) TDS results determined during UDS analysis when available. Otherwise, TDS results determined from supernate samples phase separated at PDL-W.

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

— Analysis not required by Test Plan.

M—Molarity.

Table 5.9. Post-Caustic-Leach Wash—Wet Centrifuged Solids

Sample Location: Tank T02A	Slurry Analyses				Wet Centrifuged Solids Analyses									
	UDS wt%	Slurry Density ^(a) g/mL	PDMS mg/g	PPG mg/g	Al μg/g	Cr μg/g	Fe μg/g	Na μg/g	P μg/g	S μg/g	Si ^(b) μg/g	Sr μg/g	Solids Content %	
After Final Wash ^(c)	22.0	1.167	8.8E-02	3.0	157000	504	91500	11100	216	60.5	wet solid ^(b) 1170; 50.22% solids liquid ^(b) 17.7	865	56.27	

(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) Original intact slurry submitted to analytical laboratory, so phase separation was performed at analytical laboratory.
 (c) The wt% centrifuged solids value for this step found in Analysis at Key Process Steps table in column Post-Caustic-Leach, Concentration, and Wash Sample.

Table 5.10. Post-Caustic-Leach Wash—Raman Analysis

Sample Location: Tank T02A	Al(OH) ₄ ⁻ [M]	Free OH- [M]	CrO ₄ ⁻² [M]	C ₂ O ₄ [M]	NO ₃ [M]	NO ₂ [M]	SO ₄ [M]	PO ₄ [M]
After Wash 3	0.499	5.728	0.031	<0.01	0.504	0.152	0.085	0.012 ^(a)
After Wash 27	0.074	0.900	0.004 ^(a)	0.052 ^(a)	0.076	0.036 ^(a)	0.013 ^(a)	<0.01
After Wash 50	0.016 ^(a)	0.229 ^(a)	<0.001	0.104	0.021	<0.03	0.003 ^(a)	<0.01

(a) Detected, results are qualitative: results > IDL but < EQL (estimated quantitation limit).
 M—Molarity.

5.11

Table 5.11 and Table 5.12 provide 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 phase-separated 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. 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. Raman results, shown in Table 5.12, are provided as part of the liquid analyses and presented in molar concentration (mole/liter).

Table 5.11. Oxidative-Leach

Sample Location: Tank T02A, Condition:	Slurry Analyses		Liquid Analyses													
	UDS wt%	Slurry Density ^(a) g/mL	Liquid Density ^(a) g/mL	Al μg/g	Al (rinsate) μg/g	Free OH ⁻ M	Cr μg/g	Cr (rinsate) μg/g	Si μg/g	PDMS mg/g	PPG mg/g	C ₂ O ₄ μg/g	Cl ⁻ μg/g	NO ₃ μg/g	NO ₂ μg/g	SO ₄ μg/g
Before MnO ₄ Addition ^(e)	22.0	1.173	1.017	354	132	0.12	27.6	7.60	17.7	n/d ^(b)	SX Failed	7050	n/d	514	150	142
After MnO ₄ Addition- Hr 0	20.7	1.165	1.022	473	—	0.12	135	—	—	—	—	7830	<19.3	809	116	258
After MnO ₄ Addition- Hr 1	20.9	1.165	1.025	454	—	0.15	164	—	—	—	—	7820	<19.9	748	197	254
After MnO ₄ Addition- Hr 2	21.0	1.165	1.021	452	—	0.14	177	—	—	—	—	7940	<19.9	782	205	269
After MnO ₄ Addition- Hr 3	21.0	1.167	1.020	466	—	0.15	186	—	—	—	—	7850	<20.0	813	212	267
After MnO ₄ Addition- Hr 4	21.1	1.171	1.028	473	—	0.15	196	—	—	—	—	7870	<19.8	807	211	268
After MnO ₄ Addition- Hr 5	21.2	1.159	1.018	490	—	0.16	201	—	—	—	—	7900	<20.0	830	218	272
After MnO ₄ Addition-Hr 6 (Final after Oxidative-Leach) ^(f)	21.2	1.159	1.022	506	119	0.17	207	40.6	12.1	n/d ^(b)	SX Failed	7960	<19.9	842	222	276

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

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

(d) The wt% centrifuged solids value for this step found in Analysis at Key Process Steps table in column Starting Slurry Composition Before Permanganate Addition.

(e) The wt% centrifuged solids value for this step found in Analysis at Key Process Steps table in column Initial Slurry Composition for Post-Oxidative-Leach Wash.

— Analysis not required by Test Plan.

Bold numbers are calculated values.

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

SX Failed—Analysis failed due to solvent extraction issue.

M—Molarity.

Table 5.11 (contd)

Sample Location: Tank T02A, Condition:	Rinsed Wet Centrifuged Solids Analyses							
	Al μg/g	Cr μg/g	Fe μg/g	Na μg/g	Si μg/g	Sr μg/g	PDMS mg/g	PPG mg/g
Before MnO ₄ Addition ^(d)	152667	503	92667	8357	wet solid ^(c) 1210 50.29% solids liquid ^(c) 17.7	859	0.13	2.4
After MnO ₄ Addition- Hr 0	—	—	—	—	—	—	—	—
After MnO ₄ Addition- Hr 1	—	—	—	—	—	—	—	—
After MnO ₄ Addition- Hr 2	—	—	—	—	—	—	—	—
After MnO ₄ Addition- Hr 3	—	—	—	—	—	—	—	—
After MnO ₄ Addition- Hr 4	—	—	—	—	—	—	—	—
After MnO ₄ Addition- Hr 5	—	—	—	—	—	—	—	—
After MnO ₄ Addition-Hr 6 (Final after Oxidative- Leach) ^(e)	151000	297	89400	10100	wet solid 1250 53.89% solids liquid 12.1	842	0.04	1.5

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

(d) The weight percent centrifuged solids value for this step found in Analysis at Key Process Steps table in column Starting Slurry Composition Before Permanganate Addition.

(e) The weight percent centrifuged solids value for this step found in Analysis at Key Process Steps table in column Initial Slurry Composition for Post-Oxidative-Leach Wash.

— Analysis not required by Test Plan.

Bold numbers are calculated values.

Table 5.12. Oxidative-Leach—Raman Results

Sample Location: Tank T02A, Condition:	Al(OH) ₄ ⁻ [M]	Al(OH) ₄ ⁻ (rinsate) [M]	Free OH ⁻ [M]	CrO ₄ ⁻² [M]	CrO ₄ ⁻² (rinsate) [M]	C ₂ O ₄ [M]	NO ₃ [M]	NO ₂ [M]	SO ₄ [M]	PO ₄ [M]
Before MnO ₄ Addition	<0.01	<0.01	0.203 ^(a)	<0.001	<0.001	0.078	0.019 ^(a)	0.025 ^(a)	0.002 ^(a)	<0.01
	<0.01	<0.01	0.191 ^(a)	<0.001	<0.001	0.076	0.019 ^(a)	0.029 ^(a)	0.003 ^(a)	<0.01
	<0.01	<0.01	0.163 ^(a)	<0.001	<0.001	0.074	0.019 ^(a)	0.026 ^(a)	0.003 ^(a)	<0.01
After MnO ₄ Addition- Hr 0	<0.01	—	<0.04	<0.001	—	0.016 ^(a)	0.128	<0.03	<0.001	<0.01
After MnO ₄ Addition-Hr 6 (Final after Oxidative-Leach)	0.012 ^(a)	<0.01	0.201 ^(a)	0.002 ^(a)	<0.001	0.081	0.024	<0.03	0.004 ^(a)	<0.01

(a) Detected, results are qualitative: results > IDL but < EQL (estimated quantitation limit).

— Analysis not required by Test Plan.

M—Molarity

Table 5.13 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 Table 5.14, are provided as part of the liquid analyses and presented in molar concentration (mole/liter).

Table 5.13. Post-Oxidative-Leach Wash

Sample Location: Tank T02A, Condition:	Slurry Analyses		Liquid Analyses											
	UDS wt%	Slurry Density ^(a) g/mL	Liquid Density ^(a) g/mL	Al μg/g	Na μg/g	Cr μg/g	Sr μg/g	Free OH ⁻ M	NO ₃ μg/g	NO ₂ μg/g	SO ₄ μg/g	C ₂ O ₄ μg/g	Cl ⁻ μg/g	TDS ^(b) wt%
After Wash 3	19.5	1.198	1.008	413	7660	165	<0.124	0.14	687	190	222	6120	<20.1	2.30
After Wash 6	—	—	1.002	402	7140	151	<0.124	0.13	645	181	203	5500	<20.1	2.11
After Wash 9	19.5	1.171	1.031	273	5000	105	<0.125	0.09	466	136	138	3860	<20.3	1.70
After Wash 12	—	—	1.000	255	4590	94	<0.123	0.09	425	127	123	3510	<20.6	1.40
After Wash 15	19.5	1.211	1.001	193	3520	71	<0.124	0.07	336	105	98.1	2610	<20.1	1.22
After Wash 18	—	—	0.999	181	3170	62	<0.124	0.06	301	98.2	92.6	2260	<20.2	0.931
After Wash 21	—	—	0.996	130	2300	44	<0.117	0.05	231	81.6	58.7	1570	<20.3	0.672
After Wash 24	—	—	0.990	113	2020	37	<0.116	0.05	208	75.0	50.5	1310	<20.2	0.606
After Wash 27	19.3	1.181	1.001	98.0	1740	31	<0.125	0.04	182	68.1	45.2	1070	<18.7	0.631
After Wash 30	—	—	0.994	86.7	1540	26	<0.117	0.04	174	66.5	37.2	908	<19.7	0.503
After Wash 33	19.3	1.154	1.000	81.1	1460	24	<0.123	0.04	168	<64.9	34.9	851	<19.7	0.426
After Wash 36	—	—	1.000	68.4	1220	19	<0.115	0.03	142	<62.3	23.3	625	<18.9	<0.5
After Wash 39	19.8	1.142	1.000	53.9	978	14	<0.122	0.03	132	<66.3	20.3	474	<20.1	0.284
After Wash 42	—	—	1.001	45.8	852	11	<0.123	0.02	121	<66.5	<20.2	365	<20.2	—
After Wash 44	18.9	1.161	1.000	45.4	815	10	<0.119	0.02	116	<62.8	<19.1	329	<19.1	0.288
After Final Wash	20.6	1.129	1.003	47.3	812	10	<0.120	0.02	127	<66.6	<20.2	316	<20.2	0.225

(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) TDS results determined during UDS analysis when available. Otherwise, TDS results determined from supernate samples phase-separated at PDL-W.

— Analysis not required by Test Plan.

M—Molarity

Table 5.14. Post-Oxidative-Leach Wash—Raman Results

Sample Location: Tank T02A, Condition:	Al(OH) ₄ ⁻ [M]	Free OH ⁻ [M]	CrO ₄ ⁻² [M]	C ₂ O ₄ [M]	NO ₃ [M]	NO ₂ [M]	SO ₄ [M]	PO ₄ [M]
After Wash 3	0.011 ^(a)	0.194 ^(a)	0.003 ^(a)	0.060 ^(a)	0.022	<0.03	0.004 ^(a)	<0.01
After Wash 21	<0.01	0.096 ^(a)	<0.001	0.015 ^(a)	0.013 ^(a)	0.030 ^(a)	0.002 ^(a)	<0.01
After Wash 44	<0.01	0.07 ^(a)	<0.001	<0.01	0.011 ^(a)	0.033 ^(a)	0.002 ^(a)	<0.01

(a) Detected, results are qualitative: results > IDL but < EQL (estimated quantitation limit).

M—Molarity.

Table 5.15 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. Table 5.16 provides the composition of the final slurry in T02A. The weight percent 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 5.15. Analysis of Key Process Steps

Analyte	Simulant Feed Composition ^(e)		Post Concentration of Solid Batch #1		Post Concentration of Solid Batch #2		Initial Slurry Composition for Post-Caustic-Leach Wash		Post-Caustic-Leach, Concentration, and Wash Sample	
	Wet Centrifuged		Wet Centrifuged		Wet Centrifuged		Wet Centrifuged		Wet Centrifuged	
	Solids μg/g	Liquid Phase μg/g	Solids μg/g	Liquid Phase μg/g	Solids μg/g	Liquid Phase μg/g	Solids μg/g	Liquid Phase μg/g	Solids μg/g	Liquid Phase μg/g
Al	106000 ± 2066^(a)	3230 ± 56	146333 ± 5699	2957 ± 103	132000 ± 5148	3230 ± 114	110000 ± 7430	14900 ± 895	157000 ± 10600	397 ± 24
Ca	881 ± 17	n/d	1310 ± 51	n/d	1407 ± 57	1.24 ± 0.35	1870 ± 135	1.4 ± 0.6	2910 ± 221	<1.23 ± 0.62
Ce	328 ± 6	0.006 ± 0.001	562 ± 22	0.0327 ± 0.0018	522 ± 20	0.0977 ± 0.0037	708 ± 48	0.0973 ± 0.0064	1100 ± 74	0.0134 ± 0.0026
Cr	5253 ± 102	30.30 ± 0.53	8307 ± 162	43.6 ± 1.5	7240 ± 141	46.9 ± 1.8	1130 ± 76	1380 ± 83	504 ± 34	30.2 ± 1.8
Fe	29200 ± 569	n/d	50900 ± 1984	4.60 ± 0.72	43100 ± 1681	12.6 ± 0.8	57500 ± 3880	31.5 ± 2.3	91500 ± 6180	<2.45 ± 1.23
K	590 ± 15	1083 ± 20	483 ± 28	961 ± 35	546 ± 27	982 ± 36	175 ± 32	425 ± 32	35.2 ± 14.2	11.2 ± 5.0
La	250 ± 5	0.006 ± 0.001	426 ± 17	0.029 ± 0.002	398 ± 16	0.0851 ± 0.0033	535 ± 36	0.0969 ± 0.0063	845 ± 57	0.0100 ± 0.0025
Mg	532 ± 11	n/d	959 ± 44	n/d	993 ± 41	3.01 ± 0.70	1340 ± 95	<2.49 ± 1.25	1800 ± 137	<2.45 ± 1.23
Mn	6550 ± 128	n/d	10400 ± 406	0.171 ± 0.035	8743 ± 341	0.624 ± 0.041	13900 ± 938	2.83 ± 0.18	20900 ± 1410	0.288 ± 0.064
Na	70967 ± 1393	99900 ± 1733	62033 ± 2442	88100 ± 3054	61267 ± 2403	95500 ± 3311	117000 ± 7920	160000 ± 9600	11100 ± 751	6800 ± 408
Nd	680 ± 13	0.016 ± 0.001	1183 ± 46	0.079 ± 0.003	1097 ± 43	0.218 ± 0.008	1480 ± 100	0.222 ± 0.014	2190 ± 148	0.0289 ± 0.0030
Ni	952 ± 19	n/d	1683 ± 66	0.496 ± 0.039	1357 ± 53	2.04 ± 0.08	1790 ± 121	0.494 ± 0.069	2910 ± 197	<0.245 ± 0.123
P	1270 ± 30	2057 ± 36	1103 ± 46	1740 ± 60	1290 ± 51	1773 ± 62	1440 ± 101	648 ± 40	216 ± 15	14.8 ± 0.9
Si ^(g)	511 ± 15	5.70 ± 0.41	1080 ± 48	22.7 ± 0.9	1133 ± 50	46.1 ± 5.8	864 ± 68	262 ± 19	1430 ± 104	7.99 ± 0.69
Sr	282 ± 6	n/d	462 ± 18	n/d	471 ± 19	0.124 ± 0.035	590 ± 40	0.184 ± 0.064	865 ± 59	<0.123 ± 0.062
Zr	872 ± 17	0.588 ± 0.020	1607 ± 63	0.786 ± 0.044	1533 ± 60	1.060 ± 0.079	1980 ± 134	5.47 ± 0.82	2630 ± 178	<0.123 ± 0.062
Cl	—	1023 ± 18	—	1013 ± 36	—	1067 ± 37	—	350 ± 23	—	<19.7 ± 9.9
NO ₂	—	18200 ± 363	—	18233 ± 728	—	19400 ± 692	—	6920 ± 438	—	149 ± 34
NO ₃	—	75633 ± 1332	—	76200 ± 2684	—	82800 ± 2877	—	29400 ± 1774	—	523 ± 54
PO ₄	—	5023 ± 87	—	5100 ± 177	—	5250 ± 182	—	1790 ± 111	—	65.4 ± 30.4
SO ₄	—	14033 ± 249	—	14200 ± 504	—	14733 ± 513	—	4880 ± 296	—	142 ± 13
C ₂ O ₄	—	619 ± 11	—	697 ± 25	—	679 ± 24	—	76.7 ± 9.7	—	6970 ± 421
% Solid	52.83	—	65.69	—	59.68	—	68.67	—	56.27	—
Free OH (μg/mL)	—	13983 ± 211^(c)	—	1.70E+04	—	1.65E+04	—	1.25E+05	—	2.03E+03
TIC ^(b)	—	5766 ± 116	—	—	—	—	—	—	—	—
PDMS (mg/g)	n/d ^(d)	n/d ^(d)	—	—	—	—	n/d ^(d)	n/d ^(d)	8.8E-02	n/d ^(d)
PPG (mg/g)	9.0E-02	1.0E-03	—	—	—	—	1.4E-01	9.8E-02	3.0	SX Failed
Density (g/mL)	—	1.236 ± 0.005	—	1.233 ± 0.011	—	1.249 ± 0.011	—	1.351 ± 0.021	—	1.016 ± 0.016
Wt% H ₂ O	—	73.1 ± 0.12	—	—	—	—	—	—	—	—
Slurry wt% UDS	4.97 ± 0.02	—	23.5 ± 0.2	—	23.9 ± 0.2	—	18.8 ± 0.3 ^(h)	—	22.0 ± 0.3	—
Slurry Density (g/mL)	1.276 ± 0.006	—	1.419 ± 0.013	—	1.427 ± 0.013	—	1.496 ± 0.023	—	1.167 ± 0.018	—
Weight Percent Centrifuged Solids (%)	14.7	—	45.4	—	52.4	—	46.9	—	40.6	—

(a) Fraction of solid-phase Al that is in gibbsite = 0.435.

(b) TIC = total inorganic carbon.

(c) Free OH is expressed in mg/kg.

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

(e) Uncertainty values reported for the Simulant Feed Composition are 1 σ, instead of the 2-σ values reported throughout the rest of the table.

SX Failed Analysis failed due to solvent extraction issue.

(f) Liquid phase data contains only results from the decanted supernate. No rinsate results are included.

(g) Si results from sample named “ICP” rather than sample named “AFA”.

(h) Analysis rerun on sample D 02AML 012 XX 5362 SOX 2

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

— Analysis not required by Test Plan.

Bold numbers are calculated values.

Table 5.15 (contd)

Analyte	Starting Slurry Composition Before Permanganate Addition		Initial Slurry Composition for Post-Oxidative-Leach Wash		Post-Oxidative-Leach and Wash Sample		Post-Oxidative-Leach, Wash, and Concentration Sample	
	Wet Centrifuged Solids	Liquid Phase ^(f)	Wet Centrifuged Solids	Liquid Phase ^(f)	Wet Centrifuged Solids	Liquid Phase	Wet Centrifuged Solids	Liquid Phase
	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g
Al	152667 ± 5947	354 ± 12	151000 ± 10200	506 ± 30	—	47.3 ± 3.1	142000 ± 9590	662 ± 40
Ca	2833 ± 121	n/d	2640 ± 178	<1.25 ± 0.63	—	<1.20 ± 0.60	2730 ± 185	<1.24 ± 0.62
Ce	1080 ± 42	n/d	1070 ± 72	<0.005 ± 0.003	—	<0.0048 ± 0.0024	1030 ± 70	<0.00497 ± 0.00249
Cr	503 ± 20	27.6 ± 1.0	297 ± 21	207 ± 12	—	9.83 ± 0.60	472 ± 32	72.7 ± 4.4
Fe	92667 ± 3613	n/d	89400 ± 6040	<2.5 ± 1.3	—	<4.81 ± 2.41	87600 ± 5910	<4.97 ± 2.49
K	41 ± 8	11.4 ± 2.8	105 ± 16	15.0 ± 5.1	—	<7.21 ± 3.61	150 ± 36	<7.46 ± 3.75
La	828 ± 32	n/d	827 ± 56	<0.005 ± 0.003	—	<0.0048 ± 0.0024	775 ± 52	<0.00497 ± 0.00249
Mg	1760 ± 75	n/d	1600 ± 108	<2.5 ± 1.3	—	<2.4 ± 1.2	1700 ± 115	<2.49 ± 1.25
Mn	20867 ± 812	n/d	27200 ± 1840	165 ± 10	—	<0.120 ± 0.060	25000 ± 1690	<0.124 ± 0.062
Na	8357 ± 327	6563 ± 227	10100 ± 683	9120 ± 547	—	812 ± 51	13600 ± 962	8330 ± 500
Nd	2180 ± 85	n/d	2140 ± 144	0.00594 ± 0.00253	—	0.00946 ± 0.00247	2140 ± 144	<0.00497 ± 0.00249
Ni	2857 ± 112	n/d	2750 ± 186	<0.25 ± 0.13	—	<0.12 ± 0.06	2900 ± 196	<0.124 ± 0.062
P	193 ± 8	15.8 ± 0.6	192 ± 13	35.6 ± 2.2	—	3.83 ± 0.38	246 ± 17	42.0 ± 2.5
Si ^(g)	1473 ± 62	6.84 ± 0.37	1450 ± 106	10.2 ± 0.8	—	3.38 ± 0.52	1740 ± 121	14.4 ± 1.0
Sr	859 ± 34	n/d	842 ± 57	<0.125 ± 0.063	—	<0.12 ± 0.06	831 ± 56	<0.124 ± 0.062
Zr	2547 ± 99	n/d	2470 ± 167	<0.125 ± 0.063	—	<0.24 ± 0.12	2590 ± 175	<0.249 ± 0.125
Cl	—	n/d	—	<19.9 ± 10.0	—	<20.2 ± 10.1	—	28.2 ± 10.0
NO ₂	—	150 ± 20	—	222 ± 35	—	<66.6 ± 33.5	—	366 ± 39
NO ₃	—	514 ± 31	—	842 ± 67	—	127 ± 46	—	1400 ± 95
PO ₄	—	69 ± 18	—	124 ± 31	—	<61.9 ± 31.0	—	153 ± 32
SO ₄	—	142 ± 8	—	276 ± 19	—	<20.2 ± 10.2	—	378 ± 25
C ₂ O ₄	—	7050 ± 246	—	7960 ± 480	—	316 ± 22	—	1800 ± 108
% Solid	57.26	—	55.99	—	—	—	58.43	—
Free OH (µg/mL)	—	1.99E+03	—	2.88E+03	—	4.18E+02	—	5.02E+03
TIC ^(b)	—	—	—	—	—	—	—	—
PDMS (mg/g)	1.3E-01	n/d ^(d)	4.0E-02	n/d ^(d)	—	—	1	n/d ^(d)
PPG (mg/g)	2.4	SX Failed	1.5	SX Failed	—	—	1.6	SX Failed
Density (g/mL)	—	1.017 ± 0.009	—	1.022 ± 0.016	—	1.003 ± 0.016	—	1.055 ± 0.016
Wt% H ₂ O	—	—	—	—	—	—	—	—
Slurry wt% UDS	22.0 ± 0.2	—	21.2 ± 0.3	—	20.6 ± 0.3	—	20.7 ± 0.3	—
Slurry Density (g/mL)	1.173 ± 0.010	—	1.159 ± 0.018	—	1.125 ± 0.017	—	1.217 ± 0.019	—
Weight Percent Centrifuged Solids (%)	39.2	—	37.6	—	—	—	40.6	—

(a) Fraction of solid-phase Al that is in gibbsite = 0.435.

(b) TIC = total inorganic carbon.

(c) Free OH is expressed in mg/kg.

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

(e) Uncertainty values reported for the Simulant Feed Composition are 1 σ, instead of the 2-σ values reported throughout the rest of the table.

(f) Liquid phase data contains only results from the decanted supernate. No rinsate results are included.

(g) Si results from sample named “ICP” rather than sample named “AFA.”

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

— Analysis not required by test plan.

Bold numbers are calculated values.

SX Failed—Analysis failed due to solvent extraction issue.

Table 5.16. Final Slurry Composition Before Transfer to Waste Tank

Sample Location	Slurry Analyses						Liquid Analyses						
	UDS wt%	Slurry Density g/mL	Al μg/g	Na μg/g	Cr μg/g	Sr μg/g	Free OH ⁻ M	NO ₃ μg/g	NO ₂ μg/g	SO ₄ μg/g	C ₂ O ₄ μg/g	Cl ⁻ μg/g	TDS wt%
Prior to transfer of slurry from Tank T02A to UFP-VSL-T62A/B	20.5	1.163	68.3	1100	13	<0.121	0.03	178	<66.5	33.4	373	<20.2	0.453

M—Molarity.

Table 5.17 provides the composition of the flush of the filter-loop with Tank T02A bypassed after 5 minutes of recirculation.

Table 5.17. Analysis of Material in Filter-Loop

Sample Location	Slurry Analyses						Liquid Analyses						
	UDS wt%	Slurry Density g/mL	Al μg/g	Na μg/g	Cr μg/g	Sr μg/g	Free OH ⁻ M	NO ₃ μg/g	NO ₂ μg/g	SO ₄ μg/g	C ₂ O ₄ μg/g	Cl ⁻ μg/g	TDS wt%
Batch 1: 5 min after circulating flush water in the loop with Tank T02A bypassed: Flushed loop contents	4.46	1.300	7780	116000	306	<0.115	5.25	23800	5660	4720	137	280	29.0
Batch 2: 5 min after circulating flush water in the loop with Tank T02A bypassed: Flushed loop contents	5.91	1.398	—	—	—	—	—	—	—	—	—	—	38.4
Post-oxidative-leach: 5 min after circulating flush water in the loop with Tank T02A bypassed: Flushed loop contents	4.62	1.050	139	3910	7.92	<0.121	0.05	2430	609	444	764	42.9	1.56

M—Molarity.

5.2 Physical Properties

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

5.2.1 Particle Size

Table 5.18, *Slurry Particle-Size Distribution*, provides the PSD data at key process steps, before and after sonication. The average particle size, i.e., the average equivalent diameter, is defined as the diameter where 50 mass % (of the particles) have a larger equivalent diameter, and the other 50 mass % have a smaller equivalent diameter. Hence, the average particle size is denoted as equivalent d(0.500). The definition of d(0.100) is the equivalent diameter where 10 mass % (of the particles) has a smaller diameter (and hence the remaining 90% is coarser). The definition of d(0.900) can be derived similarly. A summary of the run conditions, sample preparation, and sample analysis is given in Appendix L.

Table 5.18. Slurry Particle-Size Distribution

Process Step Description	Pre-Sonication (microns)			Sonicated (microns)		
	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	1.636	7.485	27.558	1.572	6.704	19.661
Initial simulant characterization at test start from HLP-VSL-T22	1.819	8.585	31.923	1.685	7.49	22.199
Batch #1—pre-leach concentration in Tank T02A—final slurry	0.872	4.186	14.68	1.008	4.512	14.943
	0.822	4.419	14.097	0.95	4.642	13.967
Batch #1—pre-leach concentration in Tank T02A—final slurry	1.263	6.549	19.721	1.404	6.637	19.271
Post-leach concentration—initial unwashed slurry	1.066	3.81	15.996	1.034	3.645	14.015
	1.075	3.722	13.03	1.046	3.491	11.262
	0.787	2.364	15.531	0.772	2.669	25.546
Post-leach concentration—concentrated unwashed slurry	0.769	2.005	5.787	0.704	1.849	6.054
	0.757	1.981	5.794	0.705	1.856	6.014
Post-leach concentration—final washed slurry	0.697	1.761	4.245	0.592	1.349	3.644
Permeate sample before filter cleaning	1.537	4.498	36.335	1.076	4.159	52.445

5.2.2 Rheology

Table 5.19 provides a summary of rheology measurements performed at key points throughout Integrated Test D. 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 L with flow curve analysis using both the Bingham and Casson models for both the up and down ramps. Table 5.20 provides additional shear-strength results.

Table 5.19. Integrated Test D Rheology Data Summary

<i>Process Test Step</i>	Form	Shear Strength [Pa] ^(b)	Bingham Yield [Pa] ^(a)	Bingham Consistency [mPa·s] ^(a)	Supernate Viscosity [mPa·s] ^(a)
Feed UFP-VSL-FRP	Slurry	11.61	—	—	—
Feed UFP-VSL-FRP	Slurry	—	0 ^(c)	4.272	—
Feed UFP-VSL-FRP	Supernate	—	—	—	2.689
Feed HLP-VSL-T22	Slurry	—	0 ^(c)	4.264	—
Batch #1—Pre-leach concentration in Tank T02A—initial slurry	Slurry	—	0 ^(c)	5.011	—
Batch #1—Pre-leach concentration in Tank T02A—final slurry	Slurry	—	2.831	11.18	—
Batch #2—Pre-leach concentration in Tank T02A—initial slurry	Slurry	—	0 ^(c)	5.503	—
Batch #2—Pre-leach concentration in Tank T02A—final slurry	Slurry	—	5.98	14.05	—
Post-caustic-leach concentration in Tank T02A—initial slurry	Slurry	—	0 ^(c)	19.84	—
Post-caustic-leach concentration in Tank T02A—initial slurry	Supernate	—	—	—	11.85
Post-caustic-leach concentration in Tank T02A—final slurry	Slurry	—	12.107	41.51	—
Post-caustic-leach concentration in Tank T02A—final slurry	Supernate	—	—	—	10.75
Post-caustic-leach wash—intermediate slurry	Slurry	—	16.966	9.702	—
Post-caustic-leach wash—intermediate slurry	Supernate	—	—	—	1.245
Post-caustic-leach wash—final slurry	Slurry	—	24.034	8.976	—
Post-caustic-leach wash—final slurry	Supernate	—	—	—	1.027
Oxidative-leach—before permanganate addition	Slurry	—	23.982	9.05	—
Oxidative-leach—before permanganate addition	Slurry	34.73	—	—	—
Oxidative-leach—final slurry	Supernate	—	—	—	1.064
Post-oxidative-leach wash—intermediate slurry	Slurry	—	15.443	6.939	—
Post-oxidative-leach wash—intermediate slurry	Supernate	—	—	—	0.9851
Post-oxidative-leach wash—final slurry	Slurry	—	15.608	6.737	—
Post-oxidative-leach wash—final slurry	Supernate	—	—	—	0.9847
Post-oxidative-leach concentration—final slurry	Slurry	—	26.613	8.987	—
Post-oxidative-leach concentration—final slurry	Supernate	—	—	—	0.9719
Post-oxidative-leach concentration—final slurry	Slurry	37.96	—	—	—

— Analysis not required by 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.

Table 5.20. Integrated Test D Rheology Data Summary

Settling Time (hr)	Feed UFP-VSL-FRP Maximum Shear Stress (Pa)	Oxidative leach—before permanganate addition Maximum Shear Stress (Pa)	Post-oxidative-leach concentration—final slurry Maximum Shear Stress (Pa)
1	1.734	17.830	16.400
2	1.785	13.350	15.150
4	1.837	17.390	16.370
8	2.899	17.680	16.540
16	3.193	19.500	19.310
32	5.369	22.000	21.530
72	11.610	34.730	37.960

5.2.3 XRD and SEM

Slurry samples for XRD and SEM analysis were taken before testing and after the oxidative-leach. Results are provided in Appendix L.

5.3 PJM Tuning and Parameters

In the PJM operation of the RPP-WTP PEP testing, air pressure was used to drive liquid inside the PJM tubes to mobilize and mix tank contents. The data obtained in the test runs included liquid surface levels in steady periodic vertical motion inside the PJM tubes and the PJM tank, and the steady periodic driving PJM tube pressures. The measured liquid levels in the PJM tubes and the PJM tank as well as the driving pressure in the PJM tubes were used to determine the PJM jet velocity (peak average velocity). The measured liquid levels in the PJM tubes and the PJM tank were used to determine the average PJM stroke length. In the PEP testing, the drive pressures and cycle times were iteratively adjusted to meet the target drive velocities and stroke lengths specified by the Test Instructions. Table 5.21 shows the PJM parameters for Integrated Test D, and Appendix F provides details on how the PJM peak average velocities were determined.

Table 5.21. PJM Parameters for Integrated Test D (TI-082)

Data Set Date:Time (mm/dd/yr) (hr:min)	TI step # (TI p#)	Vessel	PJM Operation Mode	Process Step Description	Target PJM Nozzle Velocity (m/s)	Actual PJM Nozzle Velocity (m/s)	Target Stroke Length (in.) (%)	Actual Stroke Length (in.) (%)	Target PJM Cycle Time (sec)	Actual PJM Cycle Time (sec)
3/23/09 02:25–02:44	7.2.7.1 (p 33)	Tank T01B	Standard	After transfer from HLP-VSL-T22 to Tank T01B for Batch 1	4.8±0.3	4.3	28.1±1.7” 80±5%	28.3” 81%	35±1	35.1
3/23/09 05:47–06:05	7.3.5.1 (p 37)	Tank T02A	Standard	After transfer from Tank T01B to Tank T02A for Batch 1	7.3±0.4	5.9	30.3±1.8” 80±5%	30.2” 80%	33±1	33.2
3/25/09 ~15:50	7.6.21 (p 59)	Tank T02A	Simple	Caustic-leach @85°C for Batch 1	7.3±0.4	7.1	30.3±1.8” 80±5%	29.3” 78%	33±1	33.2
3/27/09 05:33–05:44	7.8.4.1 (pp 75–76)	Tank T01A	Simple	After transfer from Tank T02A to Tank T01A for Batch 1	4.8±0.3	5.5	28.1±1.7” 80±5%	26.8” 76%	35±1	33.2
3/26/09 04:04–04:51	7.9.6 (pp 78-79)	Tank T01B	Standard	After transfer from HLP-VSL-T22 to Tank T01B for Batch 2	4.8±0.3	4.5	28.1±1.7” 80±5%	27.8” 79%	35±1	35.1
3/27/09 05:33–05:44	7.10.5.1 (p 82)	Tank T02A	Simple	After transfer from Tank T01B to Tank T02A for Batch 2	7.3±0.4	6.3	30.3±1.8” 80±5%	28.7” 76%	33±1	33.2
3/27/09 ~18:20	7.13.21 (p 103)	Tank T02A	Simple	Caustic-leach @85°C for Batch 2	7.3±0.4	6.8	30.3±1.8” 80±5%	30.1” 80%	33±1	33.2
3/30/09 00:19–03:22	7.15.17.1 (p 127)	Tank T02A	Standard	Post-Caustic-Leach Tank T02A	12±0.6	11.9	30.3±1.8” 80±5%	29.1” 77%	20±1	20.2

5.23

6.0 Issues

No improvements to the test system or operational changes were made between Integrated Test B and Integrated Test D. Issues for Integrated Test B also occurred in Integrated Test D and are repeated here.

6.1 Maintaining Targeted Flow Rate in Filter-Loop

The flow rate in the filter-loop was unable to meet the targeted rate of 109 gpm when the level in the tank was below ~22 inches. This is the approximate level that the return leg of the slurry coming back into UFP-VSL-T02A is exposed to (i.e., the slurry in the tank is lower than the return leg). The result is air entrainment into the slurry, which causes apparent cavitation and decreased performance of the pumps. (Actual cavitation is the formation of gas bubbles due to low pressure at the pump impeller, resulting in decreased pump performance.) However, the degassing protocol applied in Integrated Test D did not result in lower levels or increased pumping rates. Additional AFA provided some benefit.

6.2 PJM Operation

Standard Mode—The Drexelbrook level instruments in the PJMs (which determined how long pressure and vacuum to the PJMs would be applied) did not work at elevated temperatures in caustic solution. As a result, while heating during the leach, the PJMs would have to be switched from standard mode to simple mode. (See Section 3.4 for PJM operational details.)

Simple Mode—In simple mode, pressure and vacuum are applied for predetermined time intervals. These time intervals are modified by tank level and temperature. These corrections generally worked, but operators would have to closely monitor the PJMs as these correction factors did not work perfectly. The PJMs would overblow (that is, pressurized air would be on too long, causing the air to come out the bottom of the PJM) and overdraw (that is, vacuum being applied too long and drawing slurry up into the vacuum delivery piping). Both issues caused operational problems.

In UFP-VSL-T02A, air from PJM overblows was drawn into the pump suction causing severe pumping problems. Overblows were heard for PJM-1801 during Integrated Test D, and drive pressures and times were reduced. Because the pump flow rate in UFP-VSL-T01A/B was much lower, PJM overblows in these tanks did not present an operational issue.

Overdraws did not cause immediate problems, but could eventually be observed if there were any leaks in the air delivery piping where the slurry was present. During Integrated Test B, simulant was observed leaking from the air line outside the tank. Also, it is expected that over long durations, the overdraws would cause plugging issues.

PJM-1605 was inoperative during Integrated Test D. During testing, it was observed that PJM-1605 did not fill as high as other PJMs, and as a result, it was prone to overblows during the Drive Phase. No plug was found during Post-Phase 1 testing inspection. However, tanks were rinsed with water, and PJMs operated before they were inspected.

6.3 Tank Level Measurement

6.3.1 Lasers

The issues with the laser reported in Integrated Test A were largely corrected for Integrated Tests B and D. Two software filters were used to remove spurious readings. The first was an out-of-range filter that ignored any high or low readings that were out of range. The second filter ignored readings when the rate of change was greater than 3 inches in 0.5 seconds.

The spurious readings may have been generated for a variety reasons including aerosol, condensation on the lens, and foam. The issue with aerosols was largely corrected by implementing two fixes initiated in Integrated Test A. The first was to open a tank vent, which allowed air to sweep through the tank head, effectively reducing the aerosol concentration and may have also changed the foam on the surface. The second was to remove the laser vent plugs, which allowed air to flow directly past the lens. Previously air flowed around the protective tube attached to the lens but not directly on the lens. It was also possible that some condensation on the lenses caused spurious readings.

6.3.2 Bubblers

Bubblers were susceptible to plugging on a regular basis. Blowing high pressure air through the bubbler piping, which was sometimes followed by water (steam condensate and inhibited water were used for bubbler cleaning), left bubblers operational—sometimes. When bubbler cleaning was successful, the duration of functionality was unpredictable.

Another issue with the bubbler is that the readings in Tank UFP-VSL-T02A were affected when the filter-loop pumps (UFP-PMP-T42A and/or UFP-PMP-T43A) were running. The flow from these pumps would cause pressure changes at the bubbler dip legs. Consequently, the bubbler level and density readings were wrong when the filter-loop pumps were on. The bubblers in Tanks UFP-VSL-T01A/B were not influenced by the recirculation pumps in these tanks because of lower flow or better placement within the tank.

The lesson learned on the bubblers is that they need to be equipped with cleaning capability, and the placement of the dip legs should be chosen so as not to be influenced by moving liquid within the tank.

6.3.3 Drexelbrook

The Drexelbrook level indicators did not function properly in the PJM s at elevated temperatures and elevated caustic solutions. This may have due to the narrow annulus in the air line and contact with the wall. The Drexelbrook probes used in the tanks worked much better. During the temperature ramp from ambient to 85°C in both batches, the PJMs quit functioning in standard mode (which relied on the Drexelbrook level sensors in the PJMs to signal when the slurry reach the Drive Level Set Points) and had to be switched to simple mode (which did not rely on the Drexelbrook level sensors).

6.3.4 Other Issues for Integrated Test D

Other issues for Integrated Test D were the excessive oxidation of Cr during the caustic leaching step before the oxidative-leach step was started and foaming in T02A during caustic addition.

The oxidation of Cr was affected since the starting level was much lower than planned. In addition, the vessel T02A sampler pump failed during the tests, and samples were taken from the recirculation loop. However, this should not affect the sample results.

Foaming in Integrated Test D during the NaOH addition caused high-level readings in T02A and a concern that the level would be too high during the leaching process. The caustic addition was higher than in previous tests and could have contributed to the air entrainment/foaming issue. Slurry was removed from T02A during Batch 1, additional AFA was added, air sparging was reduced, and the loop was not flushed back to the tank to alleviate the level concern. In Batch 2, no slurry was removed, but additional AFA was added, and the tank-level high alarms had to be monitored closely during leaching to prevent the steam from automatically turning off. It is believed that these activities did not have a significant impact on the process. However, there was slightly less slurry leached than originally planned, and the mixing in the vessel was altered.

7.0 References

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