RPT-DFTP-005, Rev. 0
PNNL-27085

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# Crossflow Filtration of Hanford Tank AP-105 Supernatant

## February 2018

JGH Geeting
JR Allred
AM Rovira
RW Shimskey
CA Burns
RA Peterson

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

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# Executive Summary

A small-scale test platform to demonstrate the solids filtration, Cs removal, and low-activity waste (LAW) vitrification was constructed and installed at the Pacific Northwest National Laboratory (PNNL) 325 Building. Bench scale testing was conducted to demonstrate cross flow filtration using the cells unit filter (CUF) on approximately 14 liters of 241-AP-105 feed to support the confirmation of the Low-Activity Waste Pretreatment System (LAWPS) equipment performance and design basis for entrained solids removal process.

Despite the low solids content (~16 ppm) of the AP-105 feed, significant fouling of the filter was observed that required backpulsing of the filter to maintain the permeate flux within the LAWPS baseline design rate. The improvement from backpulsing was significant, but transitory, and lasted a few hours.

Increasing the transmembrane pressure was shown to have limited impact on the permeate flux in terms of magnitude and duration, especially compared with backpulsing. Because LAWPS must operate three to eight days to throughput requirements, testing demonstrated that backpulsing or chemical cleaning may need to be done every 7 to 14 hours when processing feed from AP-105. Because of this, backpulsing capability is strongly recommended for the baseline design.

The LAWPS prototypic filter cleaning was demonstrated at the end of testing and was shown to be very effective as it largely restored the filter permeability to near pre-test conditions.

# Acknowledgments

The authors gratefully acknowledge the help of hotcell technicians Jarrod Turner, LaWanda Grow, Mike Rojas, and Jason Cartwright in conducting this work.

# Acronyms and Abbreviations

| | |
|---|---|
| AEA | alpha emissions analysis |
| ASR | analytical service request |
| AV | axial velocity |
| CUF | cells unit filter |
| CWF | clean water flux |
| DEF | dead-end filtration |
| DFLAW | direct feed low-activity waste |
| DST | double shelled tank |
| EDS | energy dispersive spectrometer |
| FEG | field emission gun |
| GEA | gamma emissions analysis |
| HLW | high-level waste |
| ICP | inductively coupled plasma |
| ICP-OES | inductively coupled plasma-optical emission spectroscopy |
| ILAW | immobilized low-activity waste |
| IRC | Independent Review Committee |
| LAW | low-activity waste |
| LAWPS | Low-Activity Waste Pretreatment System |
| NIST | National Institute of Standards and Technology |
| PNNL | Pacific Northwest National Laboratory |
| QA | quality assurance |
| R&D | research and development |
| RPL | Radiochemical Processing Laboratory |
| SEM | scanning electron microscope |
| STEM | scanning transmission electron microscopy |
| TEM | transmission electron microscopy |
| TMP | transmembrane pressure |
| TOC | total organic carbon |
| WRPS | Washington River Protection Solutions |
| WTP | Waste Treatment Plant |
| WWFTP | WRPS Waste Form Testing Program |
| XRD | x-ray diffraction |

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# 1.0 Introduction

## 1.1 Background

The current plan for the disposal of Hanford tank wastes is through the Waste Treatment Plant (WTP), where both high-level waste (HLW) and low-activity waste (LAW) will be processed and made into glass. The Low-Activity Waste Pretreatment System (LAWPS) has been initiated to provide for the early production of Immobilized Low-Activity Waste (ILAW) by feeding LAW directly from tank farms to the WTP LAW Facility for immobilization. Prior to the transfer of feed to the WTP LAW Vitrification Facility, tank supernatant waste will be pretreated in the LAWPS to meet the WTP LAW waste acceptance criteria.

RPP-SPEC-56967 Rev. 6 (Ansolabehere 2016) provides the following line diagram (Figure 1.1) of the Low-Activity Waste Pretreatment System. The key process operations for treating the waste include solids filtration, cesium (Cs) removal, and vitrification of the LAW.

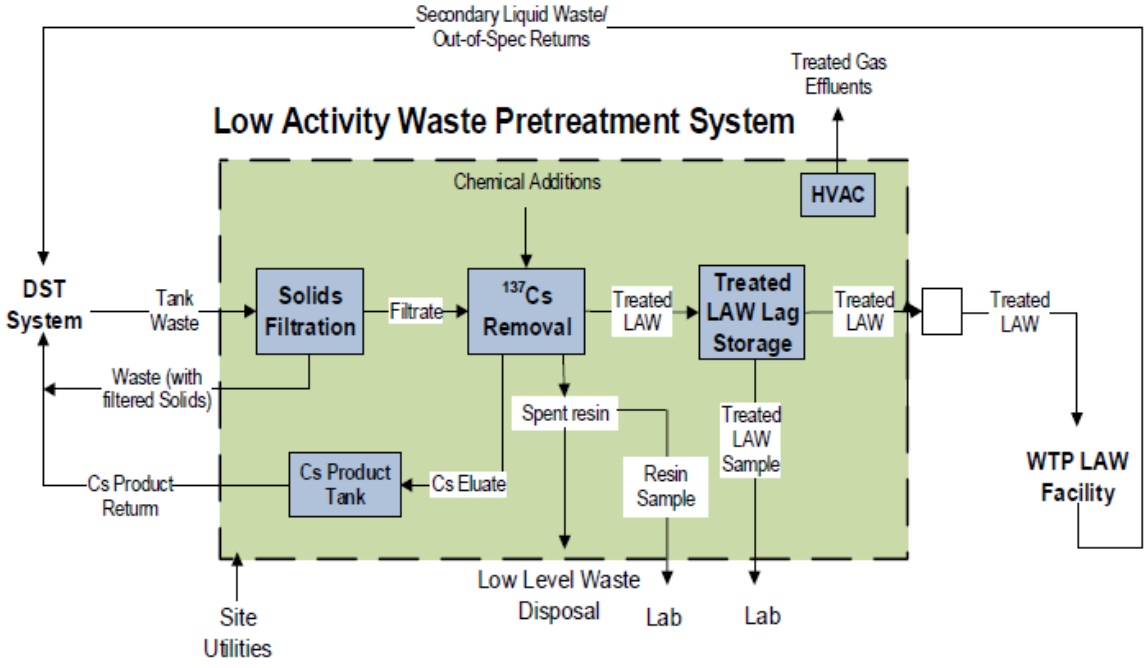


**Figure 1.1**. Low-Activity Waste Pretreatment System Diagram

LAWPS will receive tank supernatant waste from the double shelled tank (DST) system. Crossflow filtration will be used to separate the solids and the concentrate (with filtered solids) will be returned to the DST system. The filtrate will be treated by ion exchange to remove $^{137}Cs$, with the concentrated Cs being returned to the DST system. The treated waste will be stored in lag storage and sampled to confirm the WTP acceptance criteria are met. Treated waste meeting the acceptance criteria are sent to the WTP LAW facility for waste vitrification.

A small-scale test platform to demonstrate the solids filtration, Cs removal, and LAW vitrification was constructed and installed at the Pacific Northwest National Laboratory (PNNL) 325 Building. This report

describes the results of the crossflow filtration of Hanford Tank AP-105 supernatant. The filtered supernatant from this testing was subsequently used to demonstrate ion exchange and vitrification (pending) of that waste. Subsequent reports will describe the ion exchange and vitrification results.

## 1.2 Objectives

The objectives of the filtration testing are as follows.

1. Composite the 241-AP-105 feed samples and dilute to approximately 5.6 M sodium.
2. Demonstrate radioactive testing of the cells unit filter (CUF) ultrafiltration system on a sample of 241-AP-105 feed to support confirmation of the LAWPS equipment performance and design basis for entrained solids removal process.
3. Provide filtered feed for Ion Exchange.

## 1.3 Scaling Basis

Full-scale filtration typically uses bundles of filters that are 8 to 10 feet long. The filter bundles contain many filter tubes—typically more than 100 per bundle. Each tube is characterized by the internal diameter, wall thickness, and pore size rating. To avoid issues associated with differences in tube dimensions, the prototypic tube dimensions are used in bench-scale testing. However, shorter, single tubes are used in bench-scale processes. Previous work with simulants in the Pretreatment Engineering Platform has demonstrated that the performance for full-length bundles is approximately the same as for single 2-foot filter elements when represented as a filter flux (permeate rate/surface area) (Daniel et al. 2010).

The CUF matched the following filter characteristics planned for LAWPS.

1. Filter media (0.1-μm sintered stainless steel, 1/8-inch-thick filter media) – Matches filtration efficacy and filter media hydraulic resistance.
2. Filter internal diameter (0.5-inch) – Maintains the cross-sectional surface area and boundary layer filter cake effects at planned velocities.
3. Crossflow velocity (14.7 ft/s).

Filter lengths were not precisely scaled. The filter selected was based on standard filters available from Mott, which are available with 6- to 24-inch active lengths. The 6-inch length was selected to better match the filter feed/filter area ratio, as described below with reference to Table 1.1.

One key aspect of scaling filtration behavior is to attempt to maintain a similar ratio of feed vessel size (volume) to filter area (assuming similar solids concentrations at the different scales). This is balanced by the need to have a sufficiently long filter tube to avoid entrance and exit effects on filtration performance.

As indicated in Table 1.1, the 6-inch-long filter element more closely matches the full-scale filter feed tank-volume-to-filter-area ratio (66 vs. 50 $cm^3/cm^2$ ) and was selected for testing.

**Table 1.1**. Comparison of Feed Volumes and Filter Area at Different Scales

| Test Component | LAWPS Baseline Sizing | 1/9th Scale Test Configuration | CUF 6-inch Filter Configuration | CUF 2-foot Filter Configuration |
|---|---|---|---|---|
| Simulant tank (gal) | 1,000,000 | 6500 | NA | NA |
| Filter feed tank (gal) | 6467 | 700 | 1.1 | 1.1 |
| Filter area ($ft^2$) | 532 | 62.8 | 1/16 | 1/4 |
| Filter feed tank volume/filter area ($cm^3/cm^2$) | 50 | 45 | 66 | 16 |
| Prototypic filter flowrate* (gpm) | 9 | 1 | 0.001 | 0.004 |

* Based on the filter area and 0.016 gpm/$ft^2$ flux. Note that 0.001 gpm (prototypic flowrate using the 6-inch filter) is near the lower range of the CUF mass flowmeter.

## 1.4 Cells Unit Filter

A schematic of the current CUF installed in the shielded analytical hot cells is shown in Figure 1.2. The slurry feed was introduced into the CUF through the slurry reservoir. A rotary lobe pump (powered by an air motor) pumped the slurry from the slurry reservoir through the magnetic flow meter and the filter element. Adjusting the air motor supply pressure (and thus the pump speed) and the throttle valve controls the axial velocity and transmembrane pressure (TMP).

The filter was obtained from Mott Corp. (Farmington, CT). It is composed of stainless steel with a 1/2-in. internal diameter, a 5/8-in. outside diameter, and a 0.1-micron grade filter, and is 6-in. long. (Note that the CUF is designed to take up to a 24-inch-long filter element.)

Filtrate passed through the filter could be sent to the backpulse chamber, reconstituted with the slurry in the slurry reservoir ("Recycle mode"), or removed ("Dewater mode"). The filtrate flow rate was measured by a Coriolis mass flow meter and/or a fill and drain graduated glass flow monitor. Filtrate samples were taken at the three-way valve upstream from the slurry reservoir, the point at which the filtrate was removed.

Filter backpulsing (when deemed necessary) was conducted by filling half the backpulse chamber with filtrate, pressurizing the backpulse chamber with air, and forcing the filtrate in the backpulse chamber back through the filter. Additionally, filter cleaning agents (e.g., nitric acid) could be added directly to the backpulse chamber and sent back through the filter by similar means.

Feed compositing and dilution operations were performed inside the CUF's 4-L slurry reservoir. The slurry reservoir could be heated (if deemed necessary) using heat tape wrapped around the outside, and the temperature controlled by a controller external to the cell using a thermocouple in the slurry. The slurry was agitated by a mixer, and baffles were installed in the slurry reservoir to reduce vortex formations.

Prototypic filter cleaning was conducted by pumping the cleaning solution from a chemical supply tank backward through the filter and then out the CUF sample collection port.

During testing, the slurry temperature was maintained at 25 ± 5 °C by a 1000 W chiller that circulates chilled water through an in-line shell and tube heat exchanger. If elevated temperatures were required, the

chiller could be turned off or placed in heating mode where 1000 W of heating capability is available. The slurry temperature was measured by a thermocouple installed in a temperature well in the slurry reservoir.

The entire CUF was placed inside a series of drip pans to contain any leaks. A sight glass was installed on the backpulse chamber. The level in the slurry reservoir was viewed with an in-cell camera.

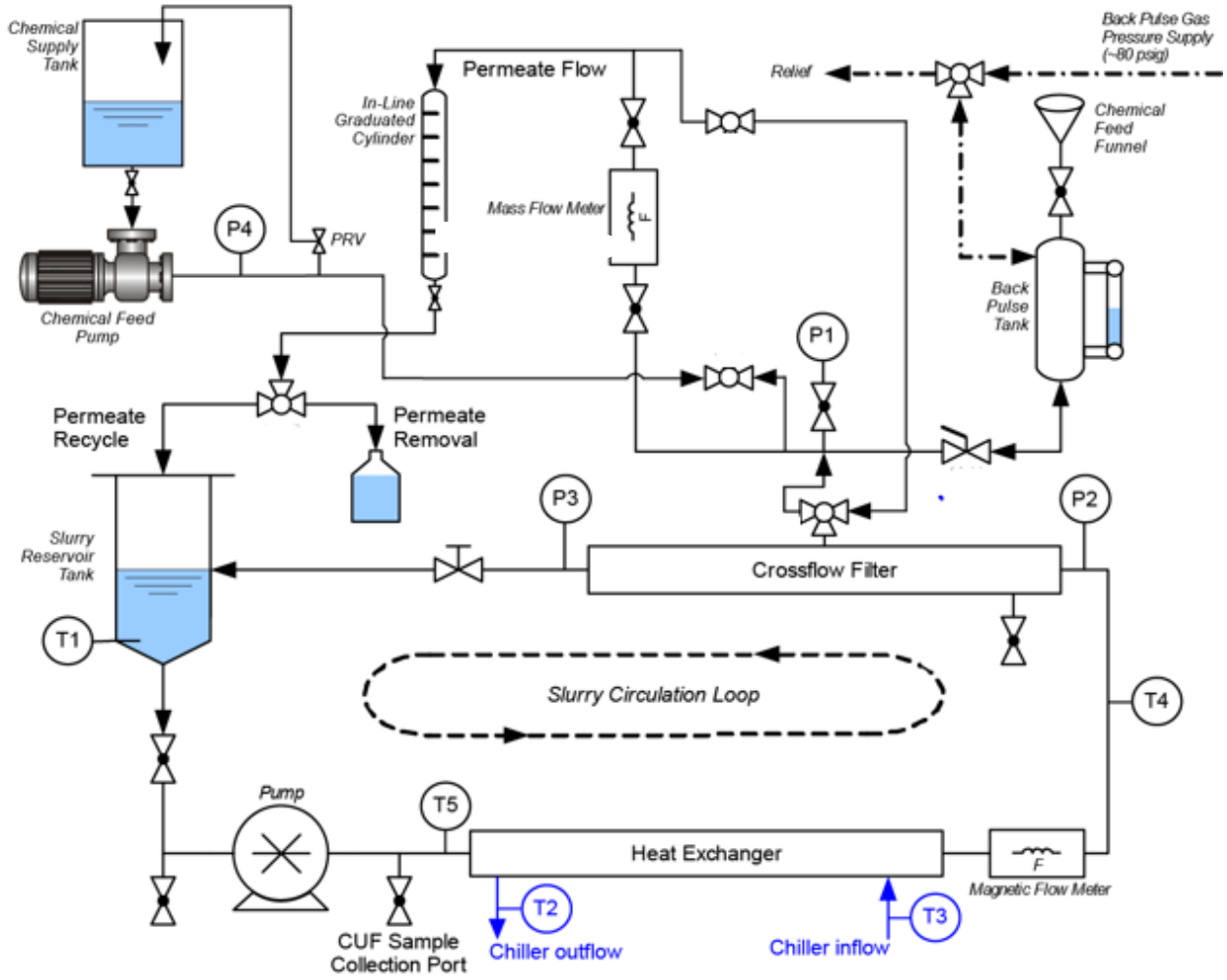


**Figure 1.2**. CUF Schematic

## 1.5 Quality Assurance

This work was conducted with funding from Washington River Protection Solutions (WRPS) under contract 292592, "*DFLAW Feed Qualification Maturation*." The work was conducted as part of PNNL Project 69833.

All research and development (R&D) work at PNNL is performed in accordance with PNNL's Laboratory-Level Quality Management Program, which is based on a graded application of NQA-1-2000, *Quality Assurance Requirements for Nuclear Facility Applications* (ASME 2000), to R&D activities. To ensure that all client quality assurance (QA) expectations were addressed, the QA controls of the WRPS

Waste Form Testing Program (WWFTP) QA program were also implemented for this work. The WWFTP QA program implements the requirements of NQA-1-2008, *Quality Assurance Requirements for Nuclear Facility Applications* (ASME 2008), and NQA-1a-2009, *Addenda to ASME NQA-1-2008* (ASME 2009), and consists of the WWFTP Quality Assurance Plan (QA-WWFTP-001) and associated QA-NSLW-numbered procedures that provide detailed instructions for implementing NQA-1 requirements for R&D work.

The work described in this report was assigned the technology level "Applied Research," and was planned, performed, documented, and reported in accordance with procedure QA-NSLW-1102, *Scientific Investigation for Applied Research*. All staff members contributing to the work received proper technical and QA training prior to performing quality-affecting work.

# 2.0 Test Conditions

## 2.1 Overview of Testing

CUF testing of the AP-105 supernatant started in June 2017 and was stopped short due to a failed weld that resulted in a leak. Testing was suspended after 18 of 36 bottles of the AP-105 feed had been processed. The leak was repaired and testing resumed in August 2017. Because of the leak, testing was broken into two campaigns that are described below. New Mott filters (6-in length, ½-in ID, 0.1µm grade) were used at the beginning of each campaign. A more detailed description of the testing may be found in Appendix A.

### 2.1.1 Campaign 1

The steps used to test the AP-105 in Campaign 1 are outlined below.

1. Performed clean water flux (CWF) tests.
2. Performed CUF Test Matrix
    a. Composited AP-105 feed in CUF and diluted to 5.6 M sodium.
    b. Subsampled slurry (for dead-end filtration (DEF) testing and analysis).[1]
    c. Ran CUF in recycle (TMP: 5 psid; AV 14.7 ft/s) for 7 hours.
    d. Backpulsed, then continued running in recycle (at TMP: 10 psid; AV: 14.7 ft/s) for 7 hours.
    e. Backpulsed, then continued running in recycle (at TMP: 15 psid; AV: 14.7 ft/s) for 4 hours.[2]
    f. Ran CUF in dewater mode to produce IX feed (at TMP: 10 psid; AV 14.7 ft/s).
3. Composited AP-105 feed in CUF and diluted to 5.6 M sodium and then ran CUF in dewater mode to produce IX feed (at TMP: 10 psid; AV 14.7 ft/s). Pump stops and restarts were performed as necessary to manage the leak. Backpulsing was conducted to maintain flowrate above 0.25 liter/hour.
4. Composited AP-105 feed in CUF and diluted to 5.6 M sodium and then ran CUF in dewater mode to produce IX feed (at TMP: 10 psid; AV 14.7 ft/s).[3] Pump stops and restarts were performed as necessary to manage the leak. Backpulsing was conducted to maintain flowrate above 0.25 liter/hour.
5. Drained CUF and subsampled slurry. (Drained CUF material filtered through DEF).

Note that no filter cleaning or final CWF was performed due to the leak.

A mass balance for Campaign 1 is shown in Table 2.1. A total of 8,953.5 g of material was added to the CUF during testing. A total of 8,805.4 g was accounted for, including 467 g (~300 mL) estimated lost due to the leak. Assuming the estimated leak size was accurate, there was still 150.5 g of material unaccounted for. The missing mass may be due to evaporation or material that wets the inside of the CUF

[1] See PNNL-26871 "Assessment of a Filtration Waste Qualification Method for LAWPS" for DEF testing results.
[2] Targeted time of 7 hours was cut short due to leak. The leak was reduced by use of epoxy on the weld joint. Testing resumed two days later and with the plan to only execute dewatering of the AP-105 to provide feed for IX.
[3] Testing secured at end of this evolution because leak rate became unmanageable.

and is not recoverable. This unrecoverable material is approximately the same as found in Campaign 2 (discussed below).

### 2.1.2 Campaign 2

The steps used to test the AP-105 in Campaign 2 are outlined below.

1. Performed CWF tests.
2. Performed CUF Test Matrix
    a. Composited AP-105 feed in CUF and diluted to 5.6 M sodium.
    b. Ran CUF in recycle (TMP: 10 psid; AV 14.7 ft/s) for 7 hours.
    c. Backpulsed, then continued running in recycle (at TMP: 15 psid; AV: 14.7 ft/s) for 7 hours.
    d. Backpulsed, then continued running in recycle (at TMP: 20[1] psid; AV: 14.7 ft/s) for 7 hours.
    e. Backpulsed, then continued running in recycle (at TMP: 10[1] psid; AV: 14.7 ft/s) for 7 hours.
3. Composited AP-105 feed in CUF and diluted to 5.6 M sodium and ran CUF in dewater mode to produce IX feed (at TMP: various;[2] AV 14.7 ft/s). Pump stops and restarts were also performed to manage the leak.
4. Composited AP-105 feed in CUF and diluted to 5.6 M sodium and ran CUF in dewater mode to produce IX feed (at TMP: various;[2] AV 14.7 ft/s). Pump stops and restarts were also performed to manage the leak.
5. Drained CUF. (Drained CUF material filtered through DEF).
6. Cleaned filter (using prototypic filter cleaning method, see Section 2.2).
7. CWF.

A mass balance for Campaign 2 is shown in Table 2.2. A total of 8,816.5 g of material was added to the CUF during testing. A total of 8,646.0 g was accounted for, including 28 g (~20 mL) spilled during transfer into the CUF. That left 170.5 g (131 mL) of material unaccounted for. The missing mass may be due to evaporation or material that wets the inside of the CUF and is not recoverable.

[1] Testing was started at the indicated pressure and maintained at that condition until the permeate flux fell to 0.25 liter/hr. When this occurred the TMP was increased.
[2] Testing was started at 10 psid and maintained at that condition until the permeate flux fell to 0.25 liter/hr. When this occurred the TMP was increased. The CUF was backpulsed only when an increase in pressure was unable to maintain the permeate flux. After a backpulse, the TMP was changed back to 10 psid and the process repeated.

**Table 2.1**. Mass Balance - Campaign 1

| CUF Test Step | Description | IN (g) | | OUT (g) | | | | |
|---|---|---|---|---|---|---|---|---|
| | | AP-105 | Dilution Water | Feed to DEF | Dewater (to IX) | PermeateSample | Slurry Sample | Leak |
| 2 | Composite Feed, Test Matrix, and Dewater | 2863.9 | 1071.3 | 847.0 | 371.9 | 130.6 | 11.0 | 467.3 |
| 3 | Composite Feed and Dewater | 1796.3 | 672.1 | 0.0 | 2437.7 | 0.0 | 0.0 | 0 |
| 4 | Composite Feed and Dewater | 1853.0 | 697.0 | 0.0 | 2392.9 | 0.0 | 0.0 | 0 |
| 5 | Drain CUF | 0.0 | 0.0 | 2052.0 | 0.0 | 14.7 | 80.3 | 0 |
| | **Subtotals** | 6513.2 | 2440.4 | 2899.0 | 5202.5 | 145.3 | 91.3 | 467.3 |
| | **Total** | 8953.5 | | 8805.4 | | | | |

**Table 2.2**. Mass Balance-Campaign 2

| CUF Test Step | Description | IN (g) | | OUT (g) | | |
|---|---|---|---|---|---|---|
| | | AP-105 | Dilution Water | Feed to DEF | Dewater (to IX) | Transfer Loss |
| 2 | Composite Feed, Test Matrix, and Dewater | 2688.3 | 1007.7 | 0.0 | 1279.2 | 0.0 |
| 3 | Composite Feed and Dewater | 1896.4 | 710.9 | 0.0 | 2497.1 | 28.1 |
| 4 | Composite Feed and Dewater | 1827.8 | 685.4 | 0.0 | 2617.9 | 0.0 |
| 5 | Drain CUF | 0.0 | 0.0 | 1989.1 | 235.0 | 0.0 |
| | **Subtotals** | 6412.5 | 2404.0 | 1989.1 | 6629.2 | 28.1 |
| | **Total** | 8816.5 | | 8646.0 | | |

## 2.2 Prototypic Filter Cleaning

The cleaning protocol used was similar to that planned for use in LAWPS and simulated the use of displacement and elution solutions fed through the IX columns and back through the filter (shell side to tube side). Table 2.3 provides the order of chemical delivery and the targeted volumes and flow rates of the cleaning protocol, which are based on scaling down (based on filter area) from the full-scale process values (provided by WRPS). The steps were as follows.

1. Isolated the slurry reservoir and pump from the rest of the process piping and the filter.
2. Loaded the chemical supply tank with the appropriate volume (or partial volume) of the chemical solution to flow through the filter.
3. Used a Fluid Metering Inc. QV metering pump to set the feed rate of the chemical solution to the nominal target values given in Table 2.3. The solution was passed through the shell side of the filter to the tube side, and then drained out into a collection vessel.
4. Repeated steps 2 through 3 until all the chemicals in Table 2.3 were delivered.

**Table 2.3**. LAWPS Cleaning Protocol Scaled for 6-in Crossflow Filter

| Step No. | LAWPS IX Step | Solution | Flowrate (mL/min) | Volume (liter) | Time (hr) |
|---|---|---|---|---|---|
| 1 | Displace | 0.1 M NaOH | 7.2 | 0.83 | 1.9 |
| 2 | Rinse | 0.01 M NaOH | 7.2 | 0.55 | 1.3 |
| 3 | Elution | 0.45 M HNO3 | 3.3 | 2.15 | 10.7 |
| 4 | Rinse | 0.01 M NaOH | 3.3 | 0.72 | 3.6 |
| | | | | **Total Time** | **17.5** |

Note: Elution volume is 50% of that planned, as the first 50% is to be sent directly to waste.

# 3.0 Results

## 3.1 Clean Water Flux

Campaign 1 and 2 both started with new filters. The first evolution of both campaigns was a CWF test, which served to measure the filter permeability (and verify the system had no leaks). Figure 3.1 indicates the initial permeate flux of these filters at the start of testing. During testing, the filters were exposed to AP-105 solids, which caused filter fouling. The ability to remove the solids that foul the filter was assessed using the LAWPS filter cleaning protocol (see Section 3.2 for description).

The filter cleaning was conducted at the conclusion of Campaign 2 testing, and as indicated in Figure 3.1, the filter permeability was largely restored after cleaning to near pre-test conditions. Based on these tests it was concluded that the LAWPS filter cleaning protocol is very effective at removing AP1-105 solids from the filter, as filter permeability was restored to essentially pre-test permeability.

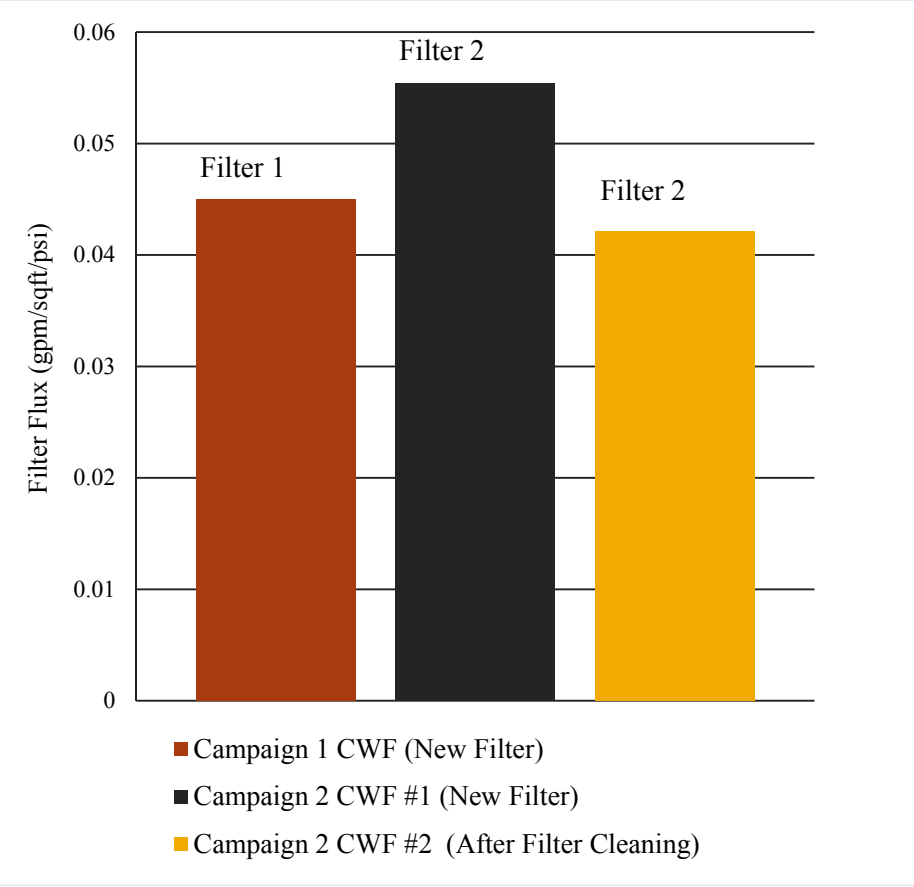


**Figure 3.1**. CWF Measurements for Campaigns 1 and 2 at 5 psi

## 3.2 Test Matrix in Recycle Mode

The first testing with AP-105 involved running a test matrix for approximately 18 hours in recycle mode.[1] Figure 3.2 shows the pressure-normalized permeate flux under three test conditions at constant axial velocity. Each of the data sets shown in Figure 3.2 start with a backpulse (Time 0). Inspection of the data suggests that some irreversible fouling of the filter occurred during this testing period, as evidenced by the lower normalized flux during the second and third portions of the testing (10 psi and 15 psi, respectively). It is also possible that there could be some compression of a filter cake; however, this feed had relatively low solids content, and it was not expected that a significant filter cake would develop. Also note that there was only a marginal difference between the second and third portions of the testing, which is consistent with what would be expected from irreversible fouling. Test conditions are summarized in Table 3.1. These fluxes required no correction for temperature, because the temperature remained constant at 25 +/-2 °C throughout this phase of testing.

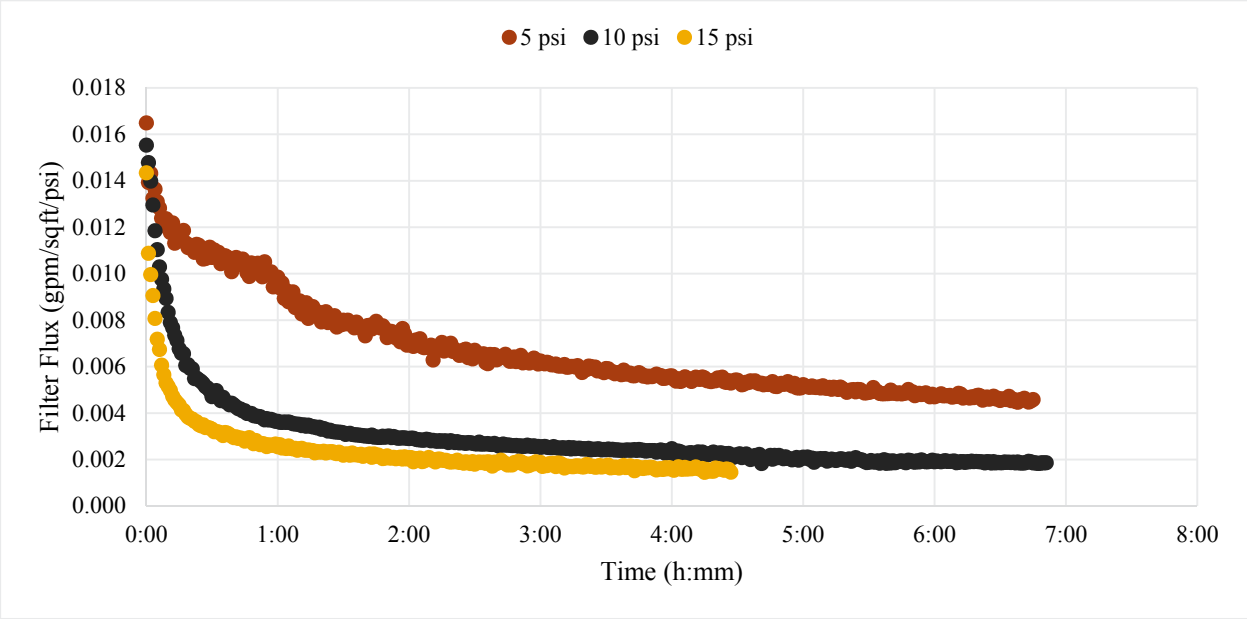


**Figure 3.2**. Campaign 1 CUF Testing in Recycle

**Table 3.1**. Operating Parameters of Campaign 1 for CUF Testing

| Test Condition | Average Flux (gpm/ft$^2$/psi) | Average Pressure (psig) | Average Velocity (ft/s) | Duration (h:mm) | Density (g/mL) | Slurry Temperature (°C) |
|---|---|---|---|---|---|---|
| Condition 1 – 5 psid | 0.0067 | 5.6 | 14.6 | 6:45 | 1.277 | 26.6 |
| Condition 2- 10 psid | 0.0029 | 11.0 | 14.6 | 6:51 | 1.276 | 25.1 |
| Condition 3 15 psid | 0.0024 | 15.5 | 15.0 | 4:27 | 1.276 | 24.9 |

[1] In recycle mode the filtered permeate is recycled back into the feed reservoir. This mode allows for longer filtration runs with limited feed.

Campaign 2 AP-105 testing also started with a matrix for 28 hours in recycle mode. Each of the data sets shown in Figure 3.3 started with a backpulse (Time 0). As shown in Figure 3.3, the permeate flux was generally lower for Campaign 2 despite the fact that both campaigns started with new filters. The lower flux may be due to residual solids from the first campaign or it may be due to higher pressures run during Campaign 2. Notice that the first condition for Campaign 2 was run at 10 psid TMP, compared to 5 psid for Campaign 1. It was observed that increased TMP resulted in more irreversible fouling and therefore only temporary increases in permeate flux.

During Campaign 2, the TMP was increased to maintain the flux above 0.25 liters/hour.

Test conditions are summarized in Table 3.2. As expected, the average filter flux was higher for the first test condition at the start of testing, as this test was performed with a clean filter. Both the 10 and 15 psi test conditions were run for the full 7 hours without increasing pressure. During the 20 psi test condition, the TMP was increased to 25 psi after ~4 hours, and increased again to 30 psi after the 6 hour mark. The second 10 psi test condition needed to be increased to 15 psi after 3 hours, 20 psi after 5 hours, and 25 psi after 6.5 hours due to low flux. These fluxes required no correction for temperature, because the temperature remained constant at 25 +/-2 °C throughout this phase of testing.

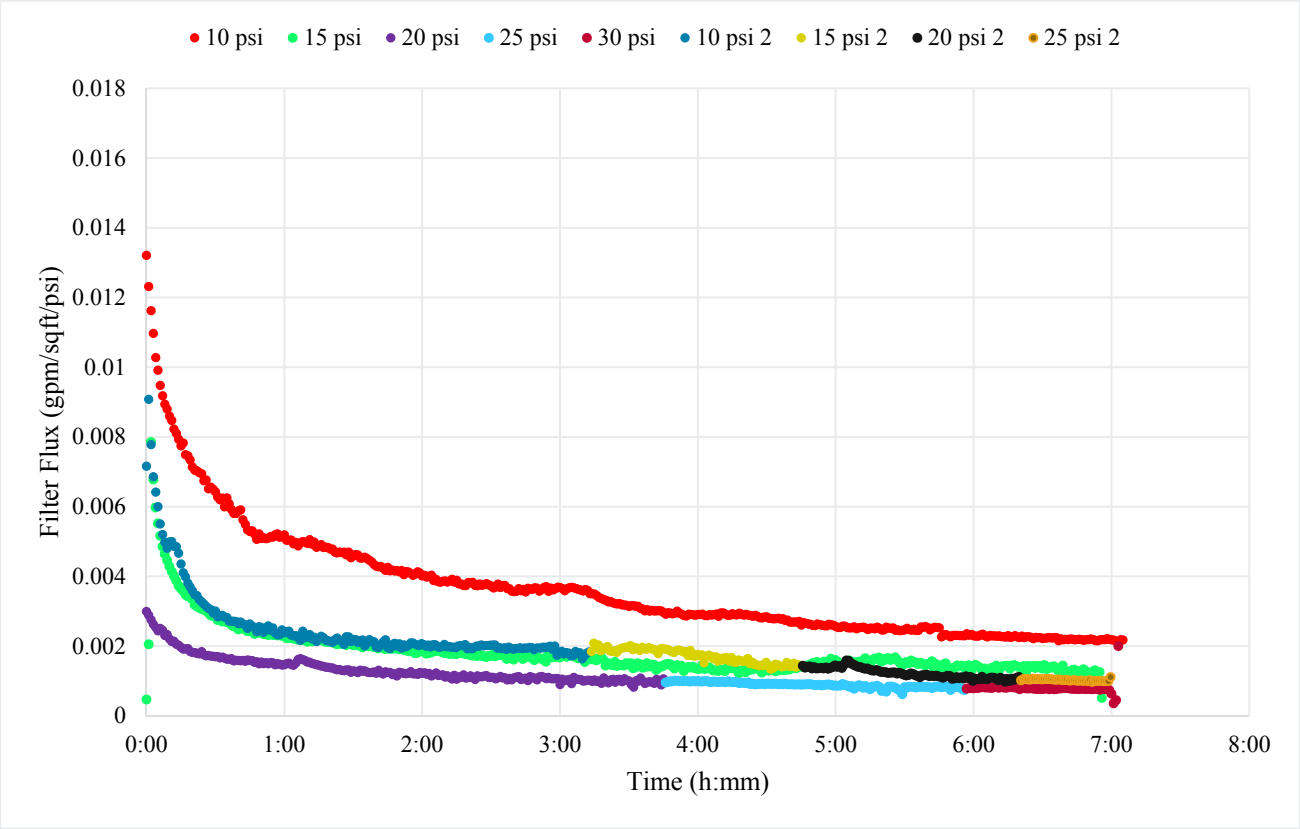


**Figure 3.3**. Campaign 2: CUF Testing in Recycle

**Table 3.2**. Operating Parameters of Campaign 2 for CUF Testing

| Test Condition | Average Flux (gpm/ft$^2$/psi) | Average Pressure (psig) | Average Velocity (ft/s) | Elapsed Time (h:mm) | Density (g/mL) | Slurry Temperature (°C) |
|---|---|---|---|---|---|---|
| Condition #1 - 10 psi | 0.0037 | 11.2 | 14.7 | 7:05* | 1.277 | 25.0 |
| Condition #2 - 15 psi | 0.0018 | 15.8 | 14.8 | 6:55* | 1.278 | 25.2 |
| Condition #3 - 20 psi | 0.0013 | 20.6 | 14.9 | 3:45 | 1.278 | 25.5 |
| Condition #3a - 25 psi | 0.0009 | 24.8 | 14.7 | 5:56 | 1.279 | 25.7 |
| Condition #3b - 30 psi | 0.0008 | 28.9 | 14.8 | 7:03 | 1.279 | 26.0 |
| Condition #4 - 10 psi | 0.0025 | 11.0 | 14.6 | 3:12 | 1.279 | 24.8 |
| Condition #4a - 15 psi | 0.0017 | 13.9 | 14.7 | 4:45 | 1.280 | 25.2 |
| Condition #4b - 20 psi | 0.0012 | 19.8 | 14.6 | 6:19 | 1.279 | 24.7 |
| Condition #4c - 25 psi | 0.0010 | 25.0 | 14.9 | 6:59 | 1.279 | 25.8 |

* Only single pressure tested for this condition so elapsed time is also the total duration of the test.

## 3.3 Dewatering

### 3.3.1 Campaign 1 - Dewatering

Campaign 1 dewatering[1] was conducted at a targeted constant TMP of 10 psid and AV of 14.7 ft/s, although operations during this segment of testing were frequently interrupted due to the leak that had developed.

Each of the dewatering segments (or trials) shown in Figure 3.4 started with a backpulse (Time 0) and then tracked the permeate flux as the feed was being filtered. The flux generally decreased during dewatering. Backpulsing provided a significant increase in the permeate flux; however, the benefit lasted only a few hours. The benefit of backpulsing had a diminishing impact (Trial 3 is lower than Trial 2 and Trial 1 after the backpulse), which suggests irreversible fouling was taking place. Stopping and restarting the pump (necessary to manage the leak) also provided some minor improvement to the permeate flux. Stopping and starting the pump can be identified by discontinuity of data sets and became more frequent as the dewatering progressed (compare Trial 1 and 3, for example). Dewatering was stopped when the leak rate became unmanageable. Table 3.3 provides averaged data for those three dewatering trials.

[1] In dewater mode the filtered permeate is is removed from the CUF.

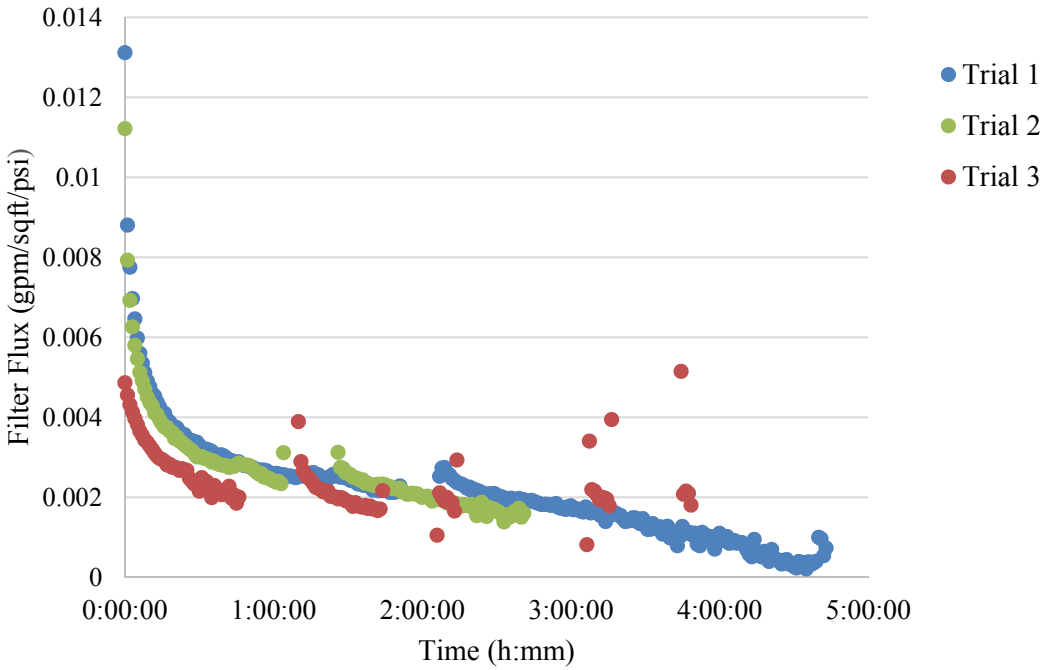


**Figure 3.4**. Campaign 1 – Dewatering

**Table 3.3**. Operating Parameters of Evolution 3 for CUF Testing

| | Average Flux ($gpm/ft^2/psi$) | Average Pressure (psig) | Average Velocity (ft/s) | Duration (h:mm) | Density (g/mL) | Slurry Temperature (°C) |
|---|---|---|---|---|---|---|
| First Dewatering Trial | 0.0021 | 10.97 | 14.3 | 4:43 | 1.285 | 25.7 |
| Second Dewatering Trial | 0.0027 | 11.40 | 14.6 | 2:41 | 1.300 | 25.2 |
| Third Dewatering Trial | 0.0024 | 10.68 | 13.7 | 3:48 | 1.296 | 25.1 |

A useful comparison is to look at the first and second dewaterings in relationship to the test matrix data. Figure 3.5 provides the data from these five filter tests. Inspection of the figure indicates that the filter performance for the last portion of the test matrix was nearly identical to the start of dewatering, indicating a slow down in the irreversible fouling with time. In addition, it is instructive to note that a significant increase in filter flux was achieved with each backpulse, but that this improvement rapidly decayed over the first hour of filtration performance, suggesting that more frequent backpulses would be beneficial to overall system performance.

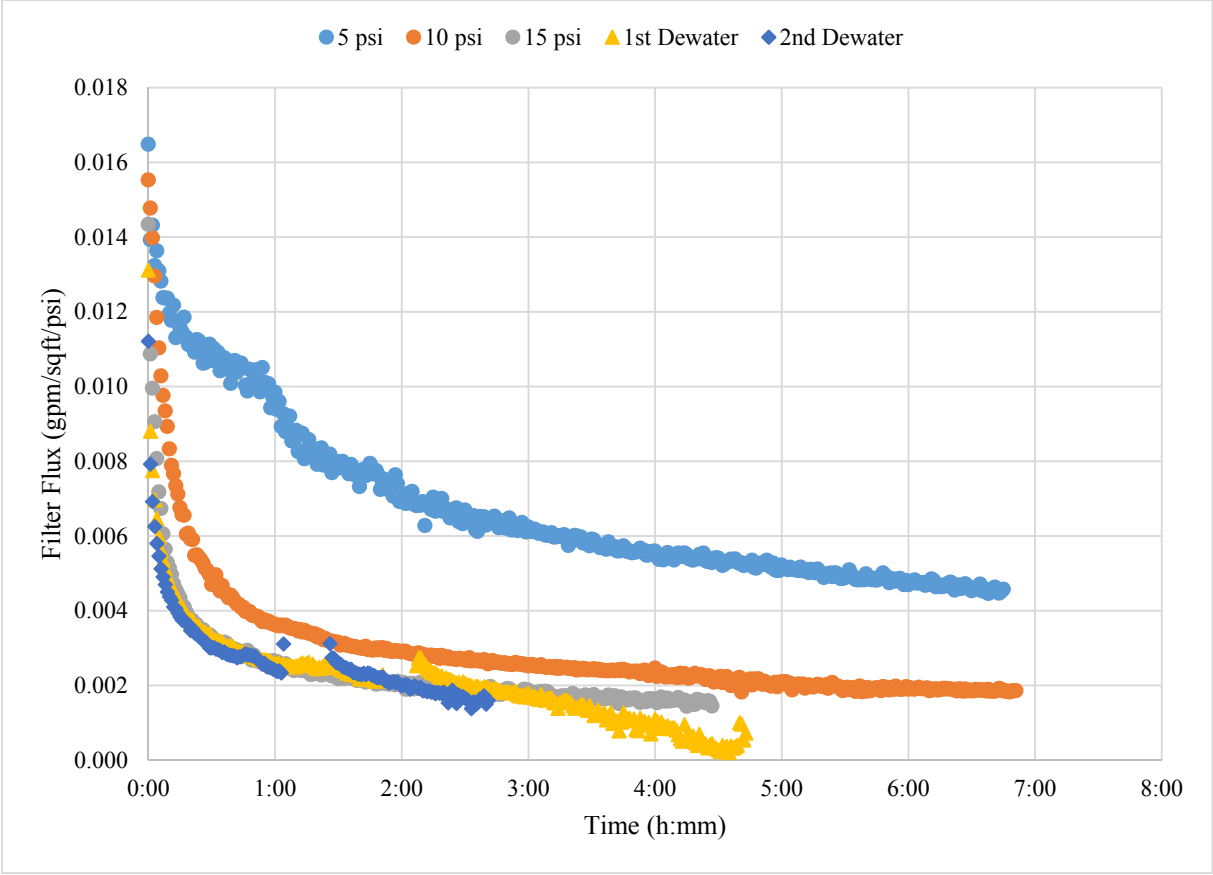


**Figure 3.5**. Comparison of Evolutions 2 and 3

### 3.3.2 Campaign 2 – Dewatering

Dewatering during Campaign 2 was peformed in three evolutions (D, E, and F). At the beginning of each evolution, the CUF was filled with AP-105 (diluted to 5.6 M Na) and backpulsed prior to dewatering. The evolution was concluded when the feed was dewatered close to the CUF's lower operating volume. The dewatering for Campaign 2, unlike Campaign 1, was not conducted at a constant TMP. Rather, the TMP was increased during dewatering to maintain a flux rate above 0.25 liter/hour. When increasing TMP was no longer effective, an additional backpulse was performed. Figure 3.6, Figure 3.7, and Figure 3.8 show the pressure-normalized flux for each of these dewatering evolutions. (The dewatering evolutions are shown in three separate figures because, if combined, the data become too hard to follow due to all of the pressure changes) Table 3.4, Table 3.5, and Table 3.6 provide averaged data for those three dewatering segments (or trials).

**Table 3.4**. Operating Parameters of Evolution D for CUF Testing

| | Average Flux ($gpm/ft^2/psi$) | Average Pressure (psig) | Average Velocity (ft/s) | Elapsed Time (h:mm) | Density (g/mL) | Slurry Temperature (°C) |
|---|---|---|---|---|---|---|
| 10 psi | 0.0031 | 11.8 | 15.1 | 1:10 | 1.272 | 24.4 |
| 15 psi | 0.0016 | 16.2 | 14.0 | 2:06 | 1.279 | 24.6 |
| 20 psi | 0.0013 | 20.9 | 15.6 | 2:40 | 1.279 | 25.4 |

**Table 3.5**. Operating Parameters of Evolution E for CUF Testing

| | Average Flux (gpm/ft$^2$/psi) | Average Pressure (psig) | Average Velocity (ft/s) | Elapsed Time (h:mm) | Density (g/mL) | Slurry Temperature (°C) |
|---|---|---|---|---|---|---|
| 20 psi | 0.00176 | 19.5 | 15.1 | 0:37 | 1.277 | 24.6 |
| 25 psi | 0.00105 | 24.7 | 14.2 | 1:00 | 1.276 | 24.7 |
| 30 psi | 0.00087 | 30.9 | 14.7 | 1:40 | 1.276 | 25.6 |
| 35 psi | 0.00072 | 35.7 | 14.5 | 2:17 | 1.276 | 25.2 |
| 40 psi | 0.00067 | 42.1 | 14.8 | 2:43 | 1.278 | 25.8 |
| 45 psi | 0.00071 | 41.8 | 14.8 | 3:28 | 1.282 | 25.5 |
| 50 psi | 0.00050 | 51.3 | 13.6 | 4:33 | 1.281 | 26.2 |
| 15 psi | 0.00115 | 16.4 | 14.6 | 5:29 | 1.282 | 23.9 |
| 25 psi | 0.00083 | 27.3 | 14.7 | 5:59 | 1.282 | 26.2 |

**Table 3.6**. Operating Parameters of Evolution F for CUF Testing

| | Average Flux (gpm/ft$^2$/psi) | Average Pressure (psig) | Average Velocity (ft/s) | Elapsed Time (h:mm) | Density (g/mL) | Slurry Temperature (°C) |
|---|---|---|---|---|---|---|
| 10 psi | 0.0025 | 11.5 | 14.8 | 0:56 | 1.284 | 25.0 |
| 15 psi | 0.0013 | 16.2 | 15.2 | 1:31 | 1.285 | 25.9 |
| 20 psi | 0.0009 | 20.0 | 15.0 | 2:06 | 1.285 | 25.8 |
| 25 psi | 0.0007 | 25.2 | 14.8 | 2:31 | 1.285 | 25.4 |
| 30 psi | 0.0006 | 32.4 | 14.6 | 3:14 | 1.285 | 25.8 |
| 40 psi | 0.0005 | 33.9 | 14.3 | 3:21 | 1.285 | 25.4 |
| 11 psi | 0.0024 | 11.6 | 14.9 | 3:41 | 1.285 | 24.2 |
| 15 psi | 0.0015 | 15.7 | 15.0 | 3:54 | 1.285 | 25.0 |
| 20 psi | 0.0012 | 18.9 | 14.9 | 4:05 | 1.285 | 25.3 |
| 25 psi | 0.0010 | 25.3 | 15.1 | 4:13 | 1.285 | 25.3 |
| 30 psi | 0.0007 | 30.2 | 14.7 | 5:28 | 1.285 | 25.7 |
| 35 psi | 0.0006 | 34.8 | 14.8 | 5:44 | 1.286 | 26.1 |
| 11 psi | 0.0016 | 12.1 | 14.42 | 6:05 | 1.285 | 24.4 |
| 15 psi | 0.0016 | 14.6 | 14.87 | 6:17 | 1.285 | 25.2 |
| 20 psi | 0.0010 | 20.4 | 15.13 | 6:49 | 1.286 | 25.8 |
| 25 psi | 0.0008 | 27.2 | 15.00 | 7:11 | 1.286 | 25.8 |

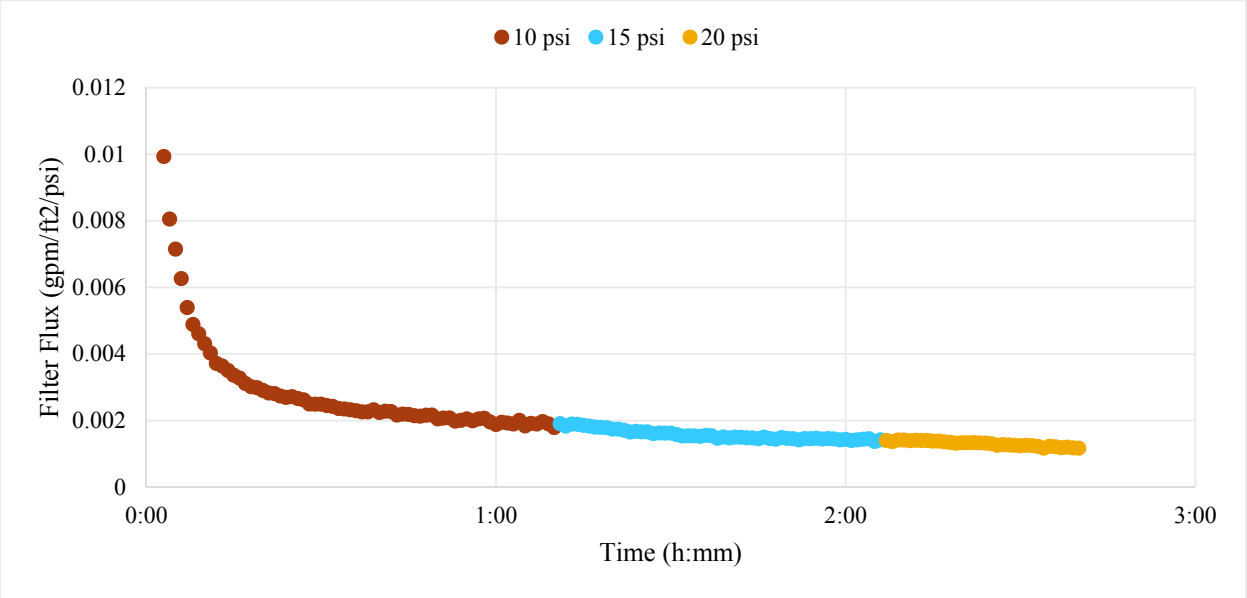


**Figure 3.6**. Dewatering – Evolution D Campaign 2

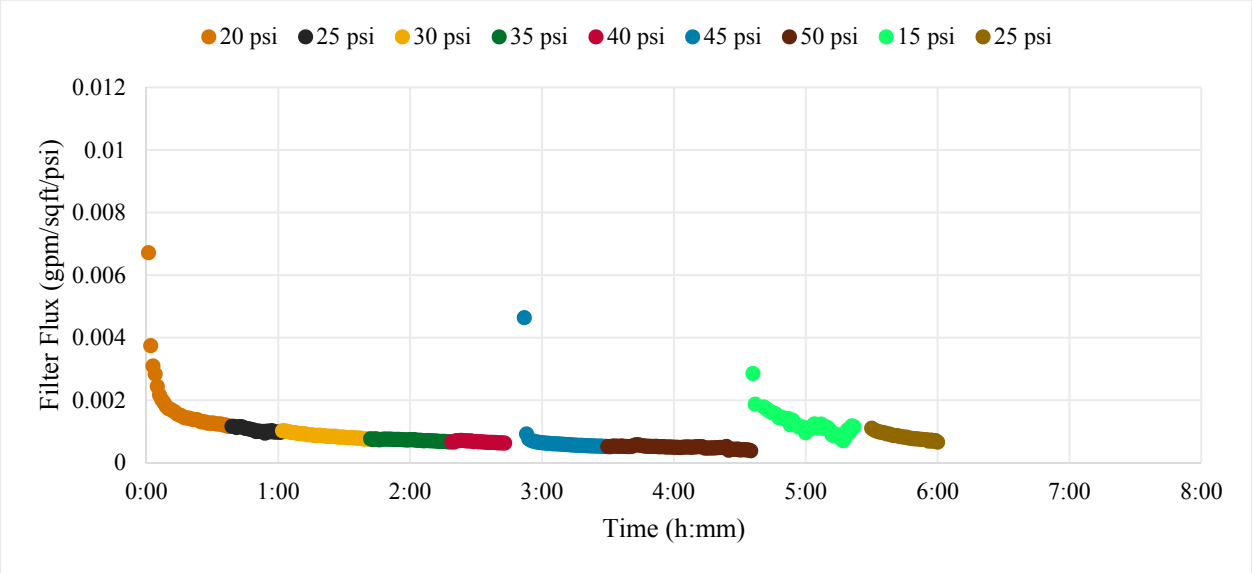


**Figure 3.7**. Dewatering Evolution E

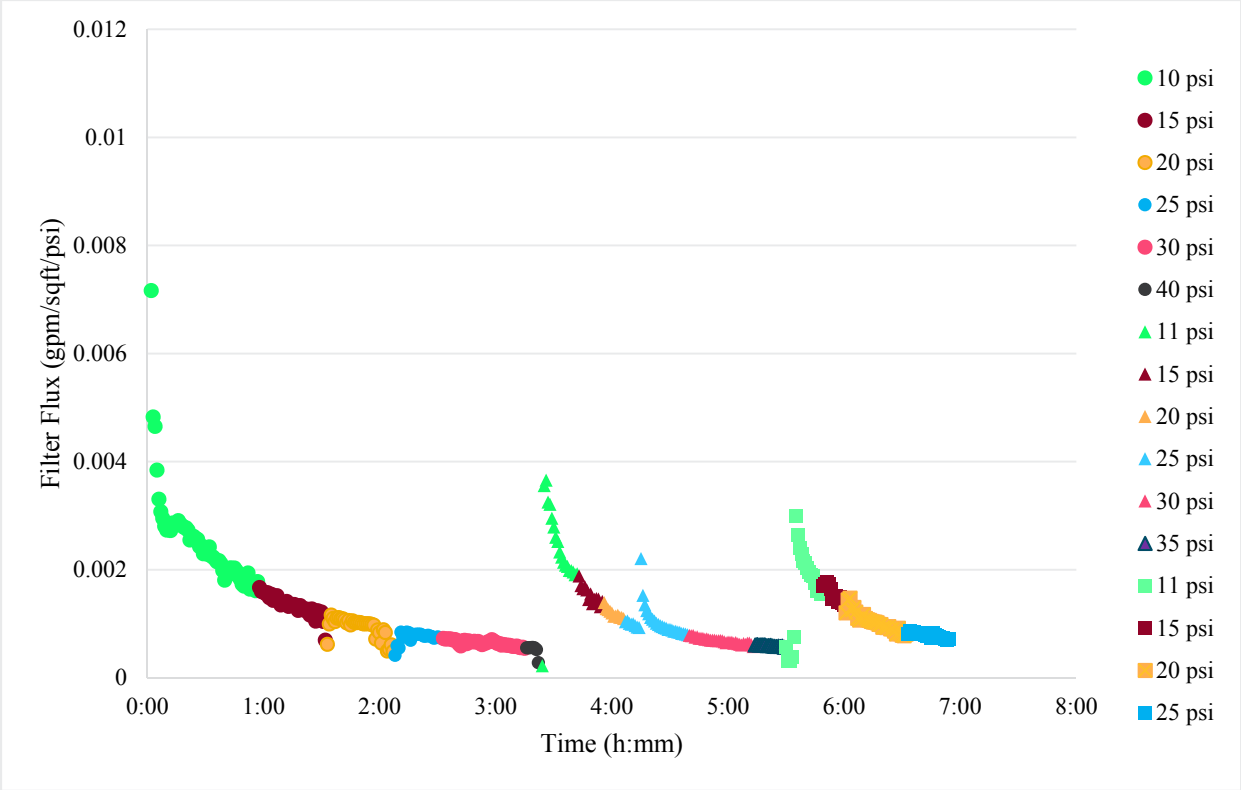


**Figure 3.8**. Dewatering Evolution F

It is constructive to examine the actual flux (not normalized for pressure) for a dewatering event. Recall that in this study, the TMP was increased as necessary to keep the flux above 0.025 gpm/ft$^2$ (lower measuring limit of the CUF mass flowmeter). Figure 3.9 plots the permeate flux and the operating TMP during dewatering of Evolution E. Also shown in Figure 3.9 is the LAWPS permeate flux upper design limit, lower design limit, and nominal target.

In order to compare the filter flux performance while backpulsing or while increasing TMP, an integral was taken under the flux curve. The area under the curve for each dewater represents the volume of feed that was passed through the filter before the flux dropped below the measuring limit of the Coriolis flowmeter. Prior to the start of dewatering at 20 psi, a backpulse was performed. Before opening the valve to the Coriolis flowmeter, the backpulse chamber was refilled with permeate. This is important to note, as any volume measured after the backpulse represents a net benefit of backpulsing. The integrated area under the curve from when dewater started to when the flux dropped to approximatel 0.02 gpm/ft$^2$ (Time 0:00 to 0:37) was found to be 0.08 gallons.

Another integral was taken under the curve after the pressure was increased to 25 psi. The area under that curve (Time 0:38 to 1:00) was found to be 0.03 gallons. Comparing these two areas, it can be seen that when a backpulse was performed, the area under the curve was larger (i.e., more volume was passed through the filter before the flux dropped below the operating limit of the flowmeter). Therefore, the effect of backpulsing has a greater effect in terms of flux increase and duration of that increase than increasing TMP.

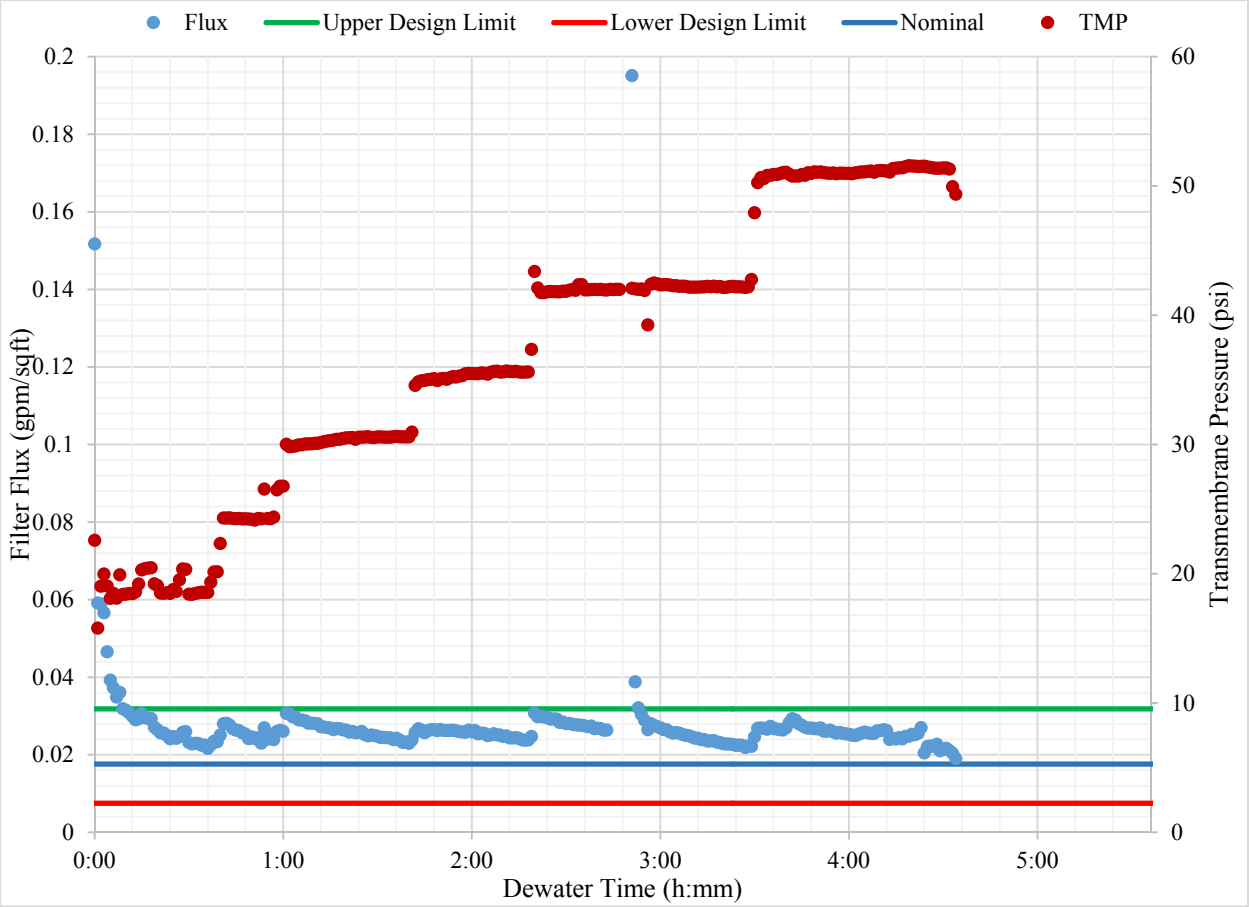


**Figure 3.9**. Permeate Flux and Operating TMP during Dewater

## 3.4 Sample Analysis

### 3.4.1 As-Received AP-105

Thirty-six samples were received from Tank AP-105. These samples were obtained in two sampling campaigns, but were all obtained from the upper supernate layer and thus were assumed to have the same composition. A sample of one of these bottles was obtained to validate the target feed composition. The characteristics of this material are given in Table 3.7.

**Table 3.7**. Composition of Sample from AP-105

| | µg/L | Molarity |
|---|---|---|
| Na | 196,000 | 8.53 |
| Al | 20,750 | 0.769 |
| K | 5525 | 0.141 |
| S | 1810 | 0.056 |
| P | 733.5 | 0.024 |
| Cr | 505 | 0.010 |
| W | 147.5 | 0.001 |
| Mo | 83.5 | 0.001 |
| B | 69.85 | 0.006 |
| Ni | 45.55 | 0.001 |

Data from CALC-DFTP-016

The density of the undiluted feed was measured at the start of Campaign 1 and Campaign 2 testing with the CUF Coriolis meter; the results were 1.390 and 1.387 g/mL, respectively.

Dilution of the feed material received from Tank AP-105 was based on the analytical results obtained from inductively coupled plasma optical emission spectrometry (given in Table 3.7). Dilution to 5.6 M Na concentration was performed multiple times using 0.01 M NaOH (i.e., "Inhibited Water"). The density of the diluted feed permeate was measured with the CUF Coriolis flowmeter during testing; the measurements ranged from 1.27–1.30 g/mL. Because the solids content in the feed was so low, these permeate densities also effectively represent the diluted AP-105 slurry density.

### 3.4.2 Permeate

The permeate viscosity was measured with a Haake M5-RV20 (equipped with an M5 measuring head and RC20 controller) and an MV1 rotor and cup measuring system. Temperature control was achieved using a combination of the standard measuring system temperature jacket and a Cole-Parmer® Polystat® Temperature-Controlled Recirculator, Model Number C-12920-00. This recirculator allows heating and cooling of recirculation fluid to the rheometer over -5° to 80º C with a stability of ±0.5° C. The permeate exhibited Newtonian properties; results are summarized in Table 3.8.

**Table 3.8**. AP-105 Permeate Rheology

| Temperature °C | Viscosity cP |
|---|---|
| 16 | 6.3 |
| 25 | 4.7 |
| 35 | 3.9 |

### 3.4.3 Slurry

At the end of Campaign 1 CUF testing, density was measured on a sample of the AP-105 retentate using a 10 mL A grade volumetric flask and an analytical balance. The density value obtained was 1.303 g/mL at an ambient temperature of 29.7 °C.

As expected (due to the low solids content of the feed), this measured density matched well with the density of the permeate measured with the Coriolis. In the future, it is suggested to forego the density measurement using the volumetric flask and rely on the Coriolis meter for ultra low-solids slurries.

The measured density of the diluted AP-105 slurry (1.303 g/mL) does not closely match the calculated density (1.256 g/mL).[1] This density difference suggests non-ideal volumetric mixing of the AP-105 feed and dilution water, with a 3.7% volume contraction (1.303 g/mL/1.256 g/mL).

At the conclusion of CUF testing, the AP-105 slurry was filtered through a dead-end filter and solids were collected. The dead-end filter test is described in Russell et al. (2017). The solids collected appeared hydrophobic and looked organic in nature. Figure 3.10a is a picture of the solids on the filter after DEF, with a small amount scraped off the filter surface. Figure 3.10b is a picture of the solids collected in a vial.

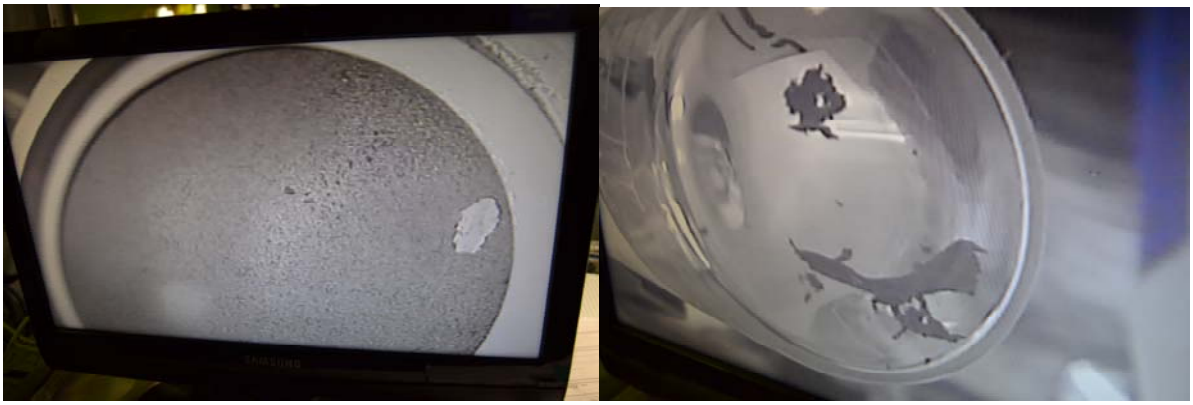

**Figure 3.10**. a) Picture of Solids on the Dead-end Filter, b) Picture of Solids in Vial after Collection

Solids collected off the dead-end filter from Campaign 1 and Campaign 2 weighed 0.0473 g and 0.0538 g, respectively. It was estimated that approximately half the solids present on the filter were captured and weighted. Assuming this estimate was correct, the original (undiluted) AP-105 feed had approximately 16 ppm solids (202 mg solids/12.9 kg undiluted feed).

An effort was made to identify the solids; the collected samples were analyzed by total organic carbon (TOC), ICP-OES, gamma emissions analysis (GEA), and alpha emissions analysis (AEA) under ASR 0323. Scanning electron microscope (SEM) and x-ray diffraction (XRD) examinations were also performed.

#### 3.4.3.1 TOC

The TOC analysis confirmed a high organic content as 9.2 wt% TOC was measured.

[1]Calculated density based on the mass of undiluted AP-105 and dilution volume (from Table 2.1) and measured density of the undiluted feed (from Section 3.4.1).

### 3.4.3.2 ICP-OES

For inductively coupled plasma-optical emission spectrometry (ICP-OES) analysis, the sample was prepared by combining two sub-samples, decomposing in $HNO_3$, and diluting to 10 mL with 0.5 M $HNO_3$. All results were reported on a mass per unit mass basis (µg/g) for each detected analyte to better understand the residual solid metal concentrations free of entrained supernatant components. The top three components measured were Na, Al, and Cr. See Table 3.9 for ICP filter solid sample results.

**Table 3.9**. AP-105 ICP Filter Solids Characterization Results

| Analyte | Sample 1 µg/g | Sample 2 µg/g | Average | Total µg | Wt% |
|---|---|---|---|---|---|
| Al | 35300 | 35800 | 35550 | 637.06 | 11.09 |
| B | 4420 | 4470 | 4445 | 79.65 | 1.39 |
| Ba | 740 | 726 | 733 | 13.14 | 0.23 |
| Ca | 1710 | 1810 | 1760 | 31.54 | 0.55 |
| Cd | 384 | 400 | 392 | 7.02 | 0.12 |
| Cr | 33500 | 33900 | 33700 | 603.90 | 10.51 |
| Cu | 1410 | 1400 | 1405 | 25.18 | 0.44 |
| Fe | 17900 | 18100 | 18000 | 322.56 | 5.61 |
| K | 4100 | 4340 | 4220 | 75.62 | 1.32 |
| Mg | 9600 | 9690 | 9645 | 172.84 | 3.01 |
| Mn | 1120 | 1140 | 1130 | 20.25 | 0.35 |
| Na | 201000 | 198000 | 199500 | 3575.04 | 62.23 |
| Ni | 6110 | 6190 | 6150 | 110.21 | 1.92 |
| Pb | 2270 | 2200 | 2235 | 40.05 | 0.70 |
| Si | 808 | 640 | 724 | 12.97 | 0.23 |
| Sr | 26.5 | 27.3 | 26.9 | 0.48 | 0.01 |
| Ti | 108 | 103 | 105.5 | 1.89 | 0.03 |
| Zn | 938 | 970 | 954 | 17.10 | 0.30 |

### 3.4.3.3 GEA and AEA

Samples received from the solids off the DEF filter were combined and decomposed using Optima grade $HNO_3$. The dried residue was dissolved in 0.5 M Optima grade $HNO_3$ to a 10 mL volume for both gamma and alpha analysis. Activities for all gamma and alpha emitters detected are shown below in Table 3.10 and Table 3.11. As expected, the predominant gamma activity was from Cs-137, which was likely from entrained supernatant. The predominant alpha activity was from Pu-238 and Am-241.

**Table 3.10**. GEA Results for ASR 0323: Measured Gamma Activity, µCi per g ± 1-σ

| Sample | Co-60 | Cs-134 | Cs-137 | CePr-144 | Eu-154 | Am-241 |
|---|---|---|---|---|---|---|
| TI-012-3 & -4 | 1.60E-02 ±4% | 2.53E-02 ±12% | 1.68E+02 ±2% | 2.59E-01 ±8% | 3.01E-01 ±2% | 9.84E-01 ±6% |

**Table 3.11**. AEA Results for ASR 0323: Measured Alpha Emitters, µCi per g ± 1-σ

| Sample | Pu-238 + Am-241 | Pu-239 + 240 | Cm-242 | Cm-243 + 244 |
|---|---|---|---|---|
| TI-012-3 & -4 | 1.03E+00 ±2% | 2.46E-01 ±3% | 4.60E-03 ±13% | 1.88E-01 ±3% |
| TI-012-3 & -4 Duplicate | 1.14E+00 ±2% | 2.66E-01 ±3% | 5.63E-03 ±11% | 2.13E-01 ±3% |
| RPD | 11% | 8% | 20% | 13% |

*RPD = Relative Percent Difference

#### 3.4.3.4 SEM Analysis

The collected solids were analyzed with a SEM with the objective of determining the composition of the solid and identification of the phases. There were many iron-rich phases in the waste as well as sodium salt phases. The iron-rich phases appeared to be Fe-Cr-Ni steel-derived particles. Al-silicates were found as well as a wispy Cr-oxide phase. Boehmite was also identified in the waste. There was evidence for the attachment of Si around the boehmite particles. Boehmite phases were found in close association with Cr phases, however, iron was not co-located in these instances. Additional details regarding the SEM analysis are provided in Buck (2017).

#### 3.4.3.5 XRD Analysis

The sample was amorphous in nature and it was difficult to identify any crystalline species, other than small amounts of thermonatrite ($Na_2CO_3$ $H_2O$). Of particular interest was that no crystalline oxalate species were identified.

# 4.0 Conclusions

Based on the results of CUF filtration experiments on AP-105, the following observations and conclusions were made.

- The AP-105 supernatant contains solids on the order of 16 ppm. These solids are suspended in the tank waste and are not separable by settling nor are they visible with the naked eye.
- Despite the low solids content of the AP-105 feed, significant fouling of the filter was observed and backpulsing of the filter was required to keep the permeate flux within the LAWPS baseline design rate.
- The improvement from backpulsing was significant, but transitory, and lasted a few hours.
- Increasing TMP was shown to have a limited impact on permeate flux in terms of magnitude and duration, especially compared to backpulsing.
- The use of backpulsing is recommended for the operation of a cross flow filter under the tested waste conditions. Bench scale testing shows backpulsing or chemical cleaning would need to be done every 7 to 14 hours to maintain throughput in processing waste from AP-105. Chemical cleaning takes approximately 34 hours and backpulsing takes <1 hour.
- The LAWPS filter cleaning protocol was very effective, as it was able to largely restore the filter permeability to near pre-test conditions.

# 5.0 References


1. Ansolabehere A. 2016. *Project T5L01 Low Activity Waste Pretreatment System Specification*. RPP-SPEC-56967, Rev. 6, Washington River Protection Solutions, Richland Washington.

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3. ASME. 2000. NQA-1-2000, *Quality Assurance Requirements for Nuclear Facility Applications*. American Society of Mechanical Engineers, New York, New York.

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6. Russell RL, JGH Geeting, JR Allred, and RA Peterson. 2017. *Assessment of a Filtration Waste Qualification Method for LAWPS*. PNNL-26871, Pacific Northwest National Laboratory, Richland, Washington.

7. Buck EC. 2017. SEM Report – *Electron Microscopy Characterization of Solids from Hanford tank 241-AP-105*. PNNL-27065, Pacific Northwest National Laboratory, Richland, Washington.

# Appendix A

# Description of Testing

# Appendix A

# Description of Testing

## A.1 Campaign 1

The CUF testing was divided into three principal evolutions, as follows.

### A.1.1 Evolution 1

This evolution involved CWF measurements. CWF measurements provide a baseline measurement of the filter resistance and were conducted at conditions indicated in Table A.1.

**Table A.1**. CUF Clean Water Flux Measurements

| Test TMP (psig) | Initial Flux (L/h) | Final Flux (L/h) | Duration of Test (min) |
|---|---|---|---|
| 5.4 | 3.9 | 3.1 | 5.0 |
| 10.1 | 6.0 | 6.3 | 5.0 |
| 14.8 | 8.3 | 9.5 | 5.0 |
| 6.0 | 2.9 | 3.8 | 5.0 |

### A.1.2 Evolution 2

This evolution involved the following steps: (1) composite approximately 4 L of feed[1] (diluted to achieve 5.6 M Na), (2) subsample for DEF testing, (3) execute the CUF test matrix, and (4) dewater (for ion exchange testing).

Table A.2 summarizes the masses of samples and water added to the CUF at the start of this evolution.

**Table A.2**. Feed Material for Initial CUF Testing

| Sample ID | Mass Added to CUF (g) | Estimated Volume (mL) |
|---|---|---|
| 5-AP-16-01 | 309 | 220 |
| 5-AP-16-02 | 307.1 | 220 |
| 5-AP-16-03 | 385.1 | 281 |
| 5-AP-16-04 | 384.6 | 281 |
| 5-AP-16-05 | 334.4 | 250 |
| 5-AP-16-06 | 384.4 | 270 |
| 5-AP-16-07 | 373.5 | 260 |
| 5-AP-16-08 | 385.8 | 270 |
| IW-NaOH-1 | 80.2 | 80.2 |
| IW-NaOH-2 | 991.1 | 991.1 |
| **Total** | **3935.2** | **3123.3** |

[1] All additions and removals of AP-105 (including dilution water to achieve targeted sodium molarity) to and from the CUF were tracked by mass.

After mixing, 750 mL of the composited material was removed using a small pump and large pipette and set aside for DEF testing. An additional sample was removed for ion exchange batch contacts. Table A.3 summarizes all of the samples removed from the CUF.

**Table A.3**. Samples Removed from CUF

| Sample ID | Mass (g) | Approximate Volume (mL) |
|---|---|---|
| DEF-E2 | 847.0 | 671.7 |
| IX-E2-1 | 371.9 | 290.5 |
| TI-010-E2-P | 130.6 | 102.0 |
| TI-010-E2-S | 11.0 | 8.7 |
| **Total** | **1360.5** | **1072.9** |

The CUF was then operated with recirculation of the permeate for the three test conditions outlined in Table A.4. At the conclusion of Condition 1 and Condition 2, a backpulse was performed. Condition 3 was stopped prematurely due to a leak that developed in one of the fittings of the CUF. At the end of Condition 3, the CUF was placed in standby mode and epoxy was applied to the leak site and allowed to cure. After 50 hours and 12 minutes of elapsed down time, the CUF was restarted. The leak persisted at a low, manageable rate at the start of Evolution 3. Note that the average density remained constant as measured by the Coriolis flow meter.

**Table A.4**. CUF Testing Conditions for Filter Testing

| Test Condition | Average TMP (psig) | Average Axial Velocity (ft/s) | Duration (h:mm) | Average Density (g/mL) | Average Slurry Temperature (°C) |
|---|---|---|---|---|---|
| Condition #1 – 5 PSI target | 5.6 | 14.6 | 6:45 | 1.277 | 26.6 |
| Condition #2 – 10 PSI target | 11.0 | 14.6 | 6:51 | 1.276 | 25.1 |
| Condition #3 – 15 PSI target | 15.5 | 15.0 | 4:27 | 1.276 | 24.9 |

### A.1.3 Evolution 3

Upon the restart of operations, the CUF was operated to produce clarified permeate for the subsequent ion exchange testing. Because of the leak described above, the objective of this phase of testing was adjusted to produce the maximum amount of filtrate possible. This evolution lasted 19 hours until the leak became too large and testing was halted. Below is a timeline of events.

- Time 0 – Restarted operations with permeate removal. Permeate density was 1.284 g/mL.
- Time 0:41 – Completed initial dewater, stopped filtration to add more feed (shown in Table A.5).

**Table A.5**. Solutions Added to CUF during Evolution 3

| Sample ID | Mass Added (g) | Estimated Volume (mL) |
|---|---|---|
| 5AP-16-09 | 302.2 | 217.0 |
| 5AP-16-10 | 364.5 | 262.2 |
| 5AP-16-11 | 365.9 | 263.0 |
| 5AP-16-12 | 381.4 | 274.4 |
| 5AP-16-13 | 382.3 | 275.0 |
| IW-NaOH-1 | 614.7 | 614.7 |
| IW-NaOH-3 | 57.3 | 57.3 |
| Subtotal | 2468.3 | 1963.6 |
| Slurry from Previous Test | - | 1690 |
| Total | - | 3653.6 |
| Measured (dipstick) | - | 3561 |

- Time 3:29 – Filter flux had declined to the lower detection limit of the Coriolis flow meter; a backpulse was performed.
- Time 5:21 – Filter stopped to add back liquid collected from the leak site.
- Time 5:37 – Filter restarted.
- Time 8:13 – Filter operation stopped for addition of feed (see Table A.6– this is designated Evolution 3.1, a subset of Evolution 3).

**Table A.6**. Solutions Added to CUF during Evolution 3.1*

| Sample ID | Mass Added (g) | Estimated Volume (mL) |
|---|---|---|
| 5AP-16-14 | 380.7 | 273.9 |
| 5AP-16-15 | 374.4 | 269.4 |
| 5AP-16-16 | 368.7 | 265.3 |
| 5AP-16-17 | 366.4 | 263.6 |
| 5AP-16-18 | 362.8 | 261.0 |
| IW-NaOH-1 | 438.7 | 438.7 |
| IW-NaOH-3 | 258.3 | 258.3 |
| Subtotal | 2550 | 2030.2 |
| Slurry from previous test (dipstick) | - | 1592 |
| Total | - | 3622.2 |
| Measured (dipstick) | - | 3675 |

* Evolution 3.1 represents a subset of Evolution 3 associated with the addition of fresh feed to the CUF.

- Time 11:21 – Filter restarted. Permeate density of 1.30 g/mL.
- Time 11:35 – Backpulse performed.
- Time 12:41 – Filter stopped to add back liquid collected from the leak site.
- Time 13:03 – Filter restarted.
- Time 14:18 – Filter stopped to add back liquid collected from the leak site.
- Time 15:09 – Filter restarted and backpulse performed. Permeate density of 1.296 g/mL.
- Time 15:56 – Filter stopped to add back liquid collected from the leak site.

- Time 16:20 – Filter restarted.
- Time 16:54 – Filter stopped to add back liquid collected from the leak site.
- Time 17:16 – Filter restarted.
- Time 17:24 – Filter stopped to add back liquid collected from the leak site.
- Time 18:17 – Filter restarted.
- Time 18:27 – Filter stopped to add back liquid collected from the leak site.
- Time 18:55 – Filter restarted.
- Time 18:59 – Evolution 3 stopped.

At the conclusion of testing, there were six bottles of material collected from the CUF, four bottles of permeate, and four bottles of material drained from the CUF for a total of ~5.4 L of material (see Table A.7).

**Table A.7**. CUF Products for Ion Exchange/DEF Testing

| Sample ID | Mass Removed (g) | Estimated Volume (mL) | Sample Type |
|---|---|---|---|
| IX-E3-1 | 1290.9 | 1014.1 | Permeate |
| IX-E3-2 | 1146.8 | 898.6 | Permeate |
| IX-E3-3 | 1310.2 | 1023.0 | Permeate |
| IX-E3-4 | 1082.7 | 854.7 | Permeate |
| DEF-E4 | 951 | 730.8 | Feed |
| DEF-E9-2 | 1101 | 844.6 | Feed |
| TI-010-E8-P3 | 14.7 | 11.5 | P-sample |
| **Total** | **6897.3** | **5377.2** | |

## A.2 Campaign 2

The evolutions in Campaign 2 were designated with letters, A through I. Evolutions B and C were not performed because a new filter was used in this testing. These two evolutions called for filter cleaning (Evolution B) and a subsequent CWF (Evolution C). The CUF testing was divided into four principal evolutions as follows.

### A.2.1 Evolution A

This evolution involved a leak test and CWF measurements. The leak test added ~1.5 L of 0.1 M NaOH solution to the CUF while checking for leaks with the system at static, and with increasing pump speeds and permeate pressures. This ensured there were no leaks in the system prior to the CWF. CWF measurements provide a baseline measurement of the filter resistance and were conducted at conditions specified in Table A.8.

**Table A.8**. CUF Clean Water Flux Measurements

| Test TMP (psig) | Initial Flux (mL/min) | Final Flux (mL/min) | Avg. TMP (psig) | Duration of Test (min) |
|---|---|---|---|---|
| 5.1 | 3.9 | 3.4 | 4.7 | 10 |
| 9.7 | 3.5 | 2.6 | 8.9 | 5.0 |
| 14.4 | 5.5 | 4.7 | 15.4 | 5.0 |
| 4.8 | 1.2 | 1.1 | 5.5 | 5.0 |

### A.2.2 Evolution D

This evolution involved the following steps: (1) composite approximately 2 L of feed[1] (diluted to achieve 5.6 M Na), (2) execute the CUF test matrix, and (3) dewater (for ion exchange testing). Table A.9 summarizes the masses of samples and water added to the CUF at the start of this evolution.

**Table A.9**. Feed Material for Initial CUF Testing

| Sample ID | Mass Added to CUF (g) | Estimated Volume (mL) |
|---|---|---|
| 5-AP-16-19 | 150.6 | 108.6 |
| 5-AP-16-20 | 380.9 | 274.6 |
| 5-AP-16-21 | 380.1 | 274.0 |
| 5-AP-16-22 | 371 | 267.5 |
| 5-AP-16-23 | 376.5 | 271.4 |
| 5-AP-16-24 | 352 | 253.8 |
| 5-AP-16-25 | 315.4 | 227.4 |
| 5-AP-16-26 | 361.8 | 260.9 |
| IW-NaOH-4 | 987.4 | 987.4 |
| IW-NaOH-3 | 20.3 | 20.3 |
| **Total** | **3696** | **2945.9** |

The CUF was then operated with recirculation of the permeate for the four test conditions outlined in Table A.10. Prior to running the system with Condition 1, a backpulse was performed. At the conclusion of Condition 1 and Condition 2, a backpulse was also performed. During Condition 3, the TMP was increased twice in 5 psi increments due to low flux. At the conclusion of Condition 3, a backpulse was performed. During Condition 4, the TMP was increased three times in 5 psi increments due to drops in the permeate flux. At the conclusion of Condition 4, a backpulse was performed and dewatering began.

[1] All additions and removals of AP-105 (including dilution water to achieve targeted sodium molarity) to and from the CUF were tracked by mass.

**Table A.10**. CUF Testing Conditions for Filter Testing

| Test Condition | Average Flux (gpm/ft$^2$/psi) | Average Pressure (psig) | Average Velocity (ft/s) | Duration (h:mm) | Density (g/mL) | Slurry Temperature (°C) |
|---|---|---|---|---|---|---|
| Condition #1 - 10 psi | 0.0037 | 11.2 | 14.7 | 7:05 | 1.277 | 25.0 |
| Condition #2 - 15 psi | 0.0018 | 15.8 | 14.8 | 6:55 | 1.278 | 25.2 |
| Condition #3 - 20 psi | 0.0013 | 20.6 | 14.9 | 3:45 | 1.278 | 25.5 |
| Condition #3a - 25 psi | 0.0009 | 24.8 | 14.7 | 5:56 | 1.279 | 25.7 |
| Condition #3b - 30 psi | 0.0008 | 28.9 | 14.8 | 7:03 | 1.279 | 26.0 |
| Condition #4 - 10 psi | 0.0025 | 11.0 | 14.6 | 3:12 | 1.279 | 24.8 |
| Condition #4a - 15 psi | 0.0017 | 13.9 | 14.7 | 4:45 | 1.280 | 25.2 |
| Condition #4b - 20 psi | 0.0012 | 19.8 | 14.6 | 6:19 | 1.279 | 24.7 |
| Condition #4c - 25 psi | 0.0010 | 25.0 | 14.9 | 6:59 | 1.279 | 25.8 |

Dewatering for Evolution D began at a TMP of 10 psi, after ~1 hour the TMP was increased to 15 psi and after another hour, increased to 20 psi where it ran for an additional 30 minutes. Table A.11 provides averaged data for the dewatering.

**Table A.11**. Operating Parameters of Evolution D for CUF Testing

| | Average Flux (gpm/ft$^2$/psi) | Average Pressure (psig) | Average Velocity (ft/s) | Elapsed Time (h:mm) | Density (g/mL) | Slurry Temperature (°C) |
|---|---|---|---|---|---|---|
| 10 psi | 0.0031 | 11.8 | 15.1 | 1:10 | 1.272 | 24.4 |
| 15 psi | 0.0016 | 16.2 | 14.0 | 2:06 | 1.279 | 24.6 |
| 20 psi | 0.0013 | 20.9 | 15.6 | 2:40 | 1.279 | 25.4 |

### A.2.3 Evolution E

Following the dewatering of Evolution D, the CUF was loaded with an additional 0.01 M NaOH and AP-105 solution, mixed for 10 minutes, and operated to produce two bottles of clarified permeate for the subsequent ion exchange testing. Table A.12 summarizes the masses of samples and water added to the CUF at the start of this evolution.

**Table A.12**. Solutions Added to CUF during Evolution E

| Sample ID | Mass Added (g) | Estimated Volume (mL) |
|---|---|---|
| 5AP-16-27 | 358.9 | 255.4 |
| 5AP-16-28 | 383.9 | 273.2 |
| 5AP-16-29 | 382.8 | 272.5 |
| 5AP-16-31 | 385.6 | 274.4 |
| 5AP-16-30 | 385.2 | 274.2 |
| IW-NaOH-3 | 260.4 | 260.4 |
| IW-NaOH-5 | 450.5 | 450.5 |
| Subtotal | 2607.3 | 2060.6 |
| Slurry from Previous Test | - | 1655 |
| Total | - | 3715.6 |
| Measured (dipstick) | - | 3058 |

During the dewatering of Evolution E, the TMP was increased up to 50 psid in order to maintain adequate permeate flow into the permeate bottles. There was no backpulse performed prior to dewatering. Table A.13 provides averaged data for the detwatering.

**Table A.13**. Operating Parameters of Evolution E for CUF Testing

| | Average Flux ($gpm/ft^2/psi$) | Average Pressure (psig) | Average Velocity (ft/s) | Elapsed Time (h:mm) | Density (g/mL) | Slurry Temperature (°C) |
|---|---|---|---|---|---|---|
| 20 psi | 0.00176 | 19.5 | 15.1 | 0:37:00 | 1.277 | 24.6 |
| 25 psi | 0.00105 | 24.7 | 14.2 | 1:00:00 | 1.276 | 24.7 |
| 30 psi | 0.00087 | 30.9 | 14.7 | 1:40:00 | 1.276 | 25.6 |
| 35 psi | 0.00072 | 35.7 | 14.5 | 2:17:00 | 1.276 | 25.2 |
| 40 psi | 0.00067 | 42.1 | 14.8 | 2:43:00 | 1.278 | 25.8 |
| 45 psi | 0.00071 | 41.8 | 14.8 | 3:28:00 | 1.282 | 25.5 |
| 50 psi | 0.00050 | 51.3 | 13.6 | 4:33:00 | 1.281 | 26.2 |

### A.2.4 Evolution F

Following the dewatering of Evolution E, the CUF was loaded with an additional 0.01 M NaOH and AP-105 solution and dewatered to produce additional permeate for the subsequent ion exchange testing. Prior to the dewatering of Evolution F, a backpulse was performed. Table A.14 summarizes the masses of samples and water added to the CUF at the start of this evolution. Table A.15 provides averaged data for the dewatering.

**Table A.14**. Solutions Added to CUF during Evolution F

| Sample ID | Mass Added (g) | Estimated Volume (mL) |
|---|---|---|
| 5AP-16-32 | 371.7 | 264.6 |
| 5AP-16-33 | 378.8 | 269.6 |
| 5AP-16-34 | 364.7 | 259.6 |
| 5AP-16-35 | 361 | 256.9 |
| 5AP-16-36 | 351.6 | 250.2 |
| IW-NaOH-5 | 543.7 | 543.7 |
| IW-NaOH-6 | 141.7 | 141.7 |
| Subtotal | 2513.2 | 1986.3 |
| Slurry from previous test (dipstick) | - | 1000 |
| Total | - | 2986.3 |
| Measured (dipstick) | - | 3524 |

**Table A.15**. Operating Parameters of Evolution F for CUF Testing

| | Average Flux ($gpm/ft^2/psi$) | Average Pressure (psig) | Average Velocity (ft/s) | Elapsed Time (h:mm) | Density (g/mL) | Slurry Temperature (°C) |
|---|---|---|---|---|---|---|
| 10 psi | 0.0025 | 11.5 | 14.8 | 0:57:00 | 1.284 | 25.0 |
| 15 psi | 0.0013 | 16.2 | 15.2 | 1:32:00 | 1.285 | 25.9 |
| 20 psi | 0.0009 | 20.0 | 15.0 | 2:07:00 | 1.285 | 25.8 |
| 25 psi | 0.0007 | 25.2 | 14.8 | 2:32:00 | 1.285 | 25.4 |
| 30 psi | 0.0006 | 32.4 | 14.6 | 3:15:00 | 1.285 | 25.8 |
| 40 psi | 0.0005 | 33.9 | 14.3 | 3:22:00 | 1.285 | 25.4 |
| 11 psi | 0.0024 | 11.6 | 14.9 | 3:42:00 | 1.285 | 24.2 |
| 15 psi | 0.0015 | 15.7 | 15.0 | 3:55:00 | 1.285 | 25.0 |
| 20 psi | 0.0012 | 18.9 | 14.9 | 4:06:00 | 1.285 | 25.3 |
| 25 psi | 0.0010 | 25.3 | 15.1 | 4:14:00 | 1.285 | 25.3 |
| 30 psi | 0.0007 | 30.2 | 14.7 | 5:13:00 | 1.285 | 25.7 |
| 35 psi | 0.0006 | 34.8 | 14.8 | 5:29:00 | 1.286 | 26.1 |

### A.2.5 Evolution G

For Evolution G, the CUF permeate slurry was drained into two bottles to be used for DEF testing. The system was the backpulsed and rinsed with 0.1 M NaOH.

At the conclusion of testing, there were seven bottles of material collected from the CUF, five bottles of permeate, and two bottles of unfiltered AP-105 drained from the CUF, for a total of ~5.4 L of material (see Table A.16).

**Table A.16**. CUF Products for Ion Exchange/DEF Testing

| Sample ID | Mass Removed (g) | Estimated Volume (mL) | Sample Type |
|---|---|---|---|
| IX-ED-1 | 1279.2 | 994.8 | Permeate |
| IX-EE-1 | 1314.5 | 1016.8 | Permeate |
| IX-EE-2 | 1182.6 | 922.2 | Permeate |
| IX-EF-1 | 1654 | 1282.5 | Permeate |
| IX-EF-2 | 963.9 | 751.2 | Permeate |
| DEF-EG-1 | 1191.7 | 916.7 | Feed |
| DEF-EG-2 | 797.4 | 613.4 | Feed |
| **Total** | **8383.3** | **6497.6** | |

### A.2.6 Evolution H

Prototypic filter cleaning was conducted by pumping the cleaning solution from a chemical supply tank backward through the filter and then out the CUF sample collection port. The cleaning was done to return the CWF to pre-operation (clean) levels. Cleaning started by loading the supply tank with ~0.83 L of 0.1 M NaOH. The tank was then supplied with 0.55 L of inhibited water. Following the inhibited water, ~2.0 L of 0.45 M $HNO_3$ was added and rinsed again with an additional ~0.72 L of inhibited water to conclude the filter cleaning.

### A.2.7 Evolution I

Evolution I was the final CWF measurement. The CUF was rinsed with 0.1 M NaOH and drained. An additional 2.0 L of 0.1 M NaOH was added to the slurry reservoir and set to “Recycle Mode”. The final CWF measurement provides a comparison measurement to the initial CWF in order to compare filter resistance and authenticity of the prototypic filter cleaning method. Final CWF was conducted at conditions specified in Table A.17.

**Table A.17**. Final CUF Clean Water Flux Measurements

| Test TMP (psig) | Initial Flux (mL/min) | Final Flux (mL/min) | Duration of Test (min) |
|---|---|---|---|
| 5 | 3.9 | 3.8 | 3.0 |
| 10 | 6.0 | 6.6 | 5.0 |
| 15 | 10.5 | 10.5 | 4.0 |
| 5 | 2.8 | 2.5 | 4.0 |

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