PNNL-27638
RPT-DFTP-009, Rev. 0

Filtration of Hanford Tank AP-107 Supernatant

July 2018

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

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

Pacific Northwest National Laboratory
Richland, Washington 99352

Executive Summary

Bench-scale filtration testing of 9.5 liters of 241-AP-107 supernatant was conducted using two different crossflow filters (CFFs) and two different dead-end filters (DEF) in the hot cells of the Radiochemical Processing Laboratory at Pacific Northwest National Laboratory. The following filters were tested:

- Mott CFF Model 7610 (media grade 5)
- Mott CFF Model 7610 (media grade 0.1)
- Mott inline filter Model 6480 (media grade 5) (also known as the backpulsed dead-end filter, BDEF)
- Mott 70-mm disc filter (media grade 0.1) (also known as the dead-end filter, DEF)

The CFFs were tested in the cells unit filter (CUF) system in recycle mode and the DEFs (in-line and disc) were used to dewater the feed.

The media grade 5 CFF had high flux (~0.9 gpm/ft^2/psi) and exhibited very little flux decline during a 28-hour recycle test with 3.2 liters of AP-107 feed. It is believed that there were insufficient solids in the feed to challenge the filter and the results are not indicative of flux rates that may be expected with a higher feed volume to filter area ratio.

The media grade 0.1 CFF exhibited a flux decline consistent with previous testing and the flux after 28 hours of recycle testing was approximately 0.002 gpm/ft^2/psi. This value was approximately twice that measured in a similar testing conducted with AP-105.[1]

The BDEF test apparatus relied heavily on the CUF equipment to hold and agitate the feed, to deliver it at the targeted pressure, and to measure the BDEF permeate flux. The BDEF was effective at removing solids from the AP-107 feed and the filtration resistance measured during testing was used to estimate that the proposed Low-Activity Waste Pretreatment System filter will require backpulsing approximately every 10 hours when running AP-107 feed at a targeting feed rate of 10 gpm. The BDEF was backpulsed and the solids, concentrated in the back flush, were analyzed for particle size. The backflush fully restored the filter flux, indicating that the solids did not irreversibly foul the filter and the backpulse removed them from the filter system.

The DEF was used to filter the final 1.9 liters of AP-107 feed drained from the CUF. Because the goal of the DEF filtration is to capture solids, a 0.1 media grade filter was used. Solids were collected and analyzed and the results are reported. The solids concentration in AP-107 was measured to be 154 ppm based on solids recovered from the DEF. Additional DEF filtering was performed on 5.1 L of BDEF permeate in order to assess whether small particles that cause fouling in the 0.1 media grade filter passed through the 5 media grade filter. There was no indication of fouling and hardly any recoverable solids were seen on the filter.

After testing, the system was rinsed with water and the filters were backpulsed. Rinsing and backpulsing the filters restored the filtration flux to new filter condition, indicating that irreversible fouling of the

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[1] Geeting JGH, JR Allred, AM Rovira, RW Shimskey, CA Burns, and RA Peterson. 2018. *Crossflow Filtration of Hanford Tank AP-105 Supernatant*. PNNL-27085, Pacific Northwest National Laboratory, Richland, WA.

filters did not occur. Additionally, subsequent cleaning with 2 M HNO_3 resulted in modest improvement to the flux, and in the case of the 0.1 media grade filter actually resulted in a flux decline.

Acknowledgments

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

Acronyms and Abbreviations

AEA	alpha emissions analysis
ASR	analytical service request
AV	axial velocity
BDEF	backpulsed dead-end filter
CFF	crossflow filter
CUF	cells unit filter
CWF	clean water flux
DEF	dead-end filter
GEA	gamma emissions analysis
ICP	inductively coupled plasma
ICP-OES	inductively coupled plasma-optical emission spectroscopy
ID	internal diameter
IX	ion exchange
LAWPS	Low-Activity Waste Pretreatment System
OD	outside diameter
PNNL	Pacific Northwest National Laboratory
PSD	particle size distribution
QA	quality assurance
R&D	research and development
SEM	scanning electron microscope
TMP	transmembrane pressure
TOC	total organic carbon
WRPS	Washington River Protection Solutions
WWFTP	WRPS Waste Form Testing Program

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1.0 Introduction

1.1 Background

The Low-Activity Waste Pretreatment System (LAWPS) Project is undergoing major changes. Changes of particular interest for the work described herein include potentially changing the filtration method (dead-end vs. crossflow filtration) and filter media grade (5 vs. 0.1 media grade) (see Reynolds et al. 2018).

Filtration tests were conducted at the small-scale test platform [located at the Pacific Northwest National Laboratory (PNNL) Radiochemical Processing Laboratory] using approximately 9.5 liters of decanted AP-107 tank waste supernatant. The purpose of this testing was to a) demonstrate dead-end and crossflow filtration of AP-107 feed with a media grade 5 filter, b) demonstrate crossflow filtration of AP-107 feed with a media grade 0.1 filter (to provide direct comparison with filtration of AP-105 feed with a media grade 0.1 filter [see Geeting et al. 2018]), c) collect and characterize solids, and d) provide feed to ion exchange (IX) and vitrification (also part of the test platform). Subsequent reports will describe the IX and vitrification results.

1.2 Cells Unit Filter (CUF)

Figure 1.1 shows a schematic of the current CUF installed in the shielded analytical hot cells. The slurry feed was introduced into the CUF through the slurry reservoir. A rotary lobe pump (powered by an air motor) pumped the slurry through a recirculation loop containing a magnetic flow meter and filter assembly. Adjusting the air motor supply pressure (and thus the pump speed) and the throttle valve controls the axial velocity (AV) and transmembrane pressure (TMP).

Two crossflow filters (CFFs) (5 and 0.1 media grade) obtained from Mott Corp. (Farmington, CT) were used in testing. Both filters are composed of sintered stainless steel with a 1/2-in. internal diameter (ID), 5/8-in. outside diameter (OD), and 6-in. length. Both filters were new at the start of testing and were welded into a single assembly with valving to allow either filter to be used without the need to stop testing and install a new filter (see Figure 1.2).

Filtrate passed through the filter could be sent to the backpulse chamber (“Backpulsing”), 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 when operating in dewater mode.

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.

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.

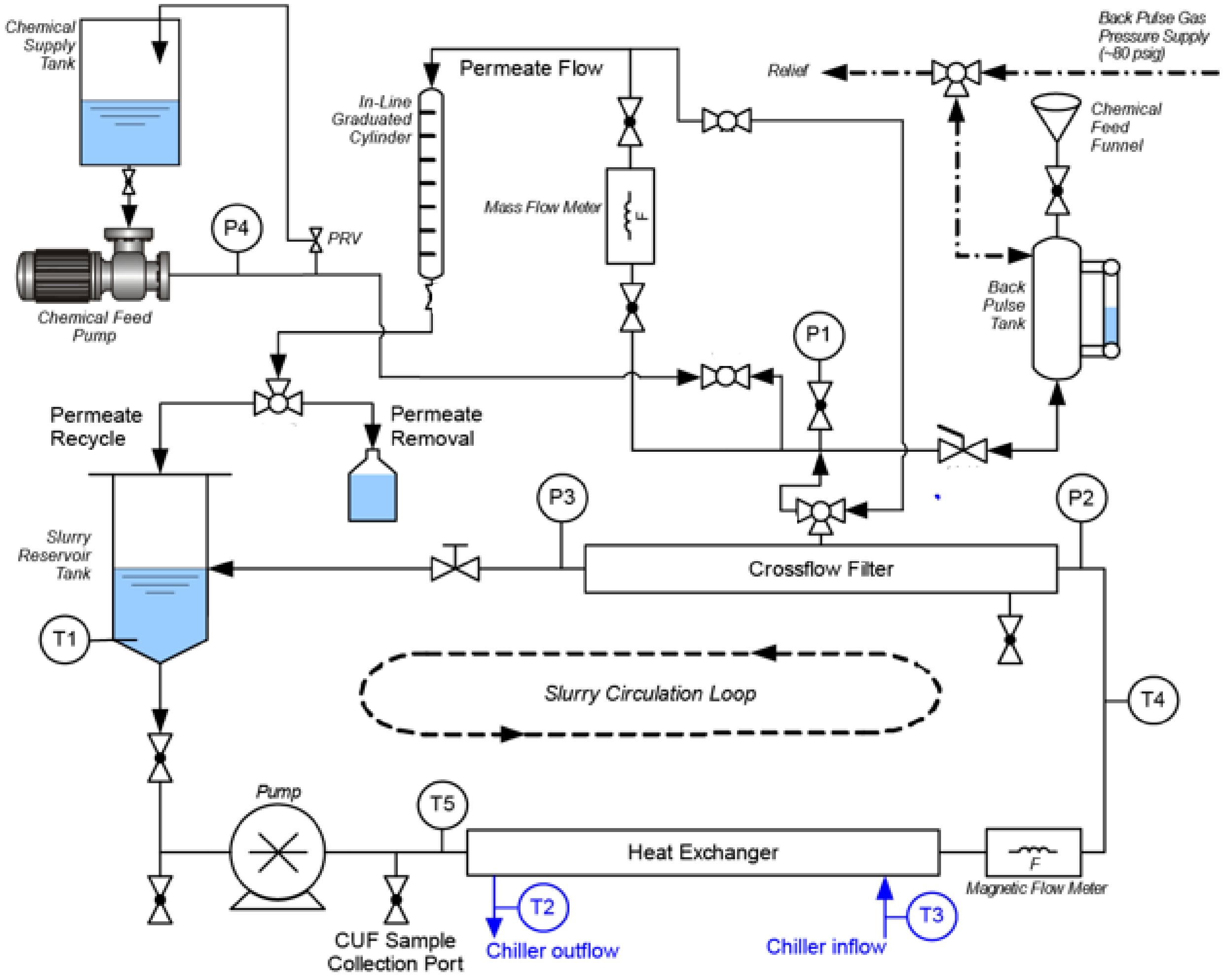

Figure 1.1. CUF Schematic

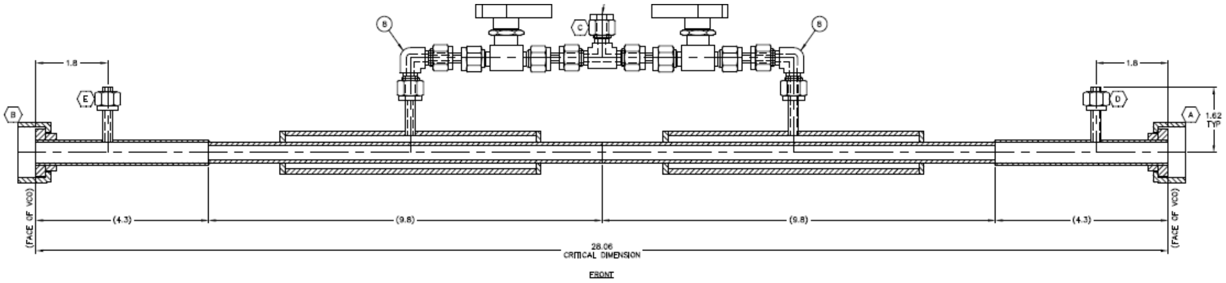

Figure 1.2. Double Filter Assembly

1.3 BDEF

The test filter for the backpulse dead-end filter (BDEF) was the Mott 6480-1/2-6-5, modified[1] to 2.75-in. filter active length. The filter element is fabricated from a seamless sintered stainless steel tube that is closed on one end. The other end is welded to a pipe reducing bushing. The filter is cylindrical with dimensions of 3/8-in. diameter x 2.75-in. length and has a filtration area of 3.24 in^2. Figure 1.3 shows an image of the Series 6480 filter.

The BDEF test apparatus relied on the CUF to hold and agitate the feed, deliver it at the targeted pressure, and measure the BDEF permeate flux. Figure 1.4 shows a schematic of the BDEF assembly. The CUF provides pressurized feed to the BDEF through a connection to the CUF sample collection port. The feed is filtered as it flows through the BDEF. Filtered feed is then introduced into the CUF permeate system to measure the flowrate through the glass flow meter and/or the Coriolis flowmeter. When the CUF is operating in “BDEF mode,” the CUF rotary lobe pump is operating in recirculation to provide pressurized feed to the BDEF, but the CUF CFF permeate valve is closed to prevent filtration. Thus, only filtrate from the BDEF is flowing through the CUF permeate metering and collection system. The BDEF may be backpulsed by closing the feed valve (Valve A), opening the drain valve (Valve B), and then using the CUF backpulse chamber and pressured air to backpulse the BDEF.

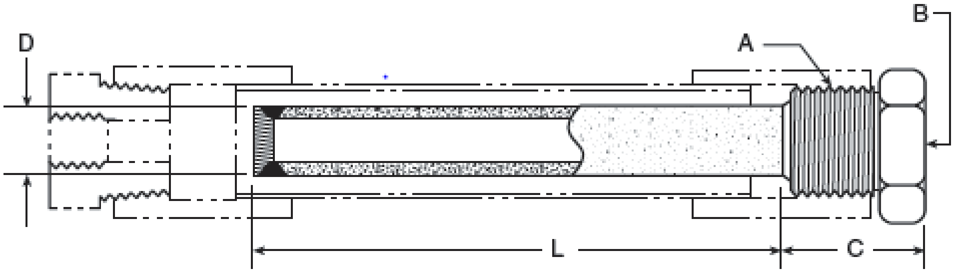

Figure 1.3. Mott 6480 Line Filter from https://mottcorp.com

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[1] The filter was cut approximately in half and a new non-porous end cap was welded on. The weld was inspected and approved prior to use.

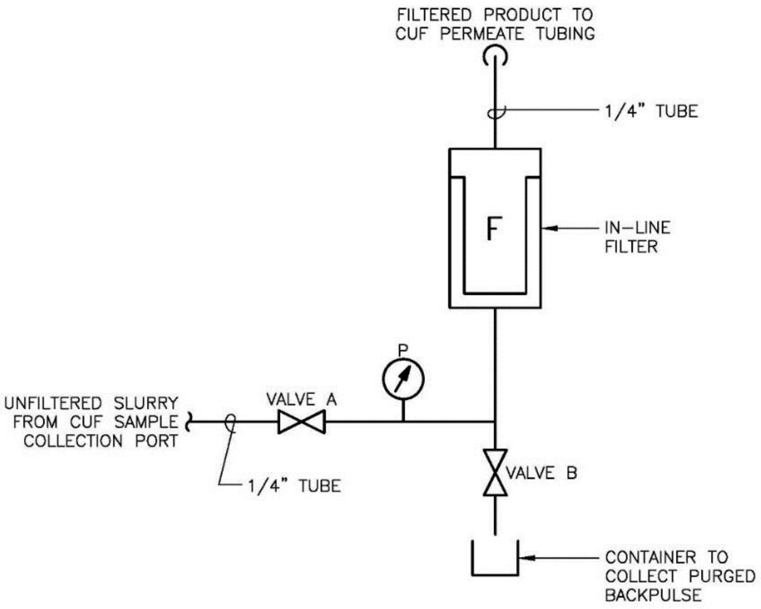

Figure 1.4. BDEF Schematic

1.4 DEF

The test filter for dead-end filtration is a Mott 70-mm disc test filter, which is a commercial off-the-shelf dead-end filter (DEF) designed for feasibility studies at the laboratory bench scale. The disc filter can operate at temperatures up to 250 °F and 150 psig. Figure 1.5 shows the configuration of the disc filter assembly. The 70-mm disc test filter is a barrier type filter with an available filter area of approximately 4.4 in^2. The Mott filter media is stainless steel sintered metal and available in various filter grades (0.1, 0.5, 1, 5, 10 μm). To capture as many solids as possible, we used the grade 0.1 filter media for testing.

Figure 1.6 shows a schematic of the DEF test apparatus. The dilute slurry feed is introduced to the system through the feed reservoir. Compressed air supplied to the top of the reservoir, at a controlled pressure, pushes the feed through the test filter. Filtrate passes through the filter and the TMP and filtrate mass are measured as a function of time. This filter system has the advantage of having no minimum volume necessary to operate. Therefore, the DEF was used to filter the final ~1.5 liters of AP-107 feed drained from the CUF, as the CUF requires >1.5 liters of feed to operate the pump.

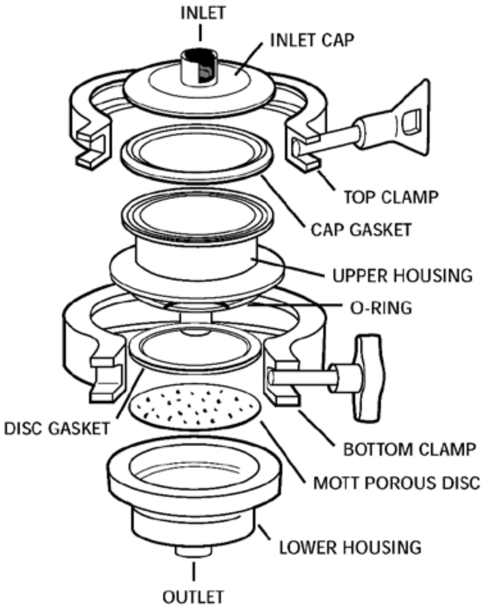

Figure 1.5. Mott 70-mm Disc Test Filter Assembly from https://mottcorp.com

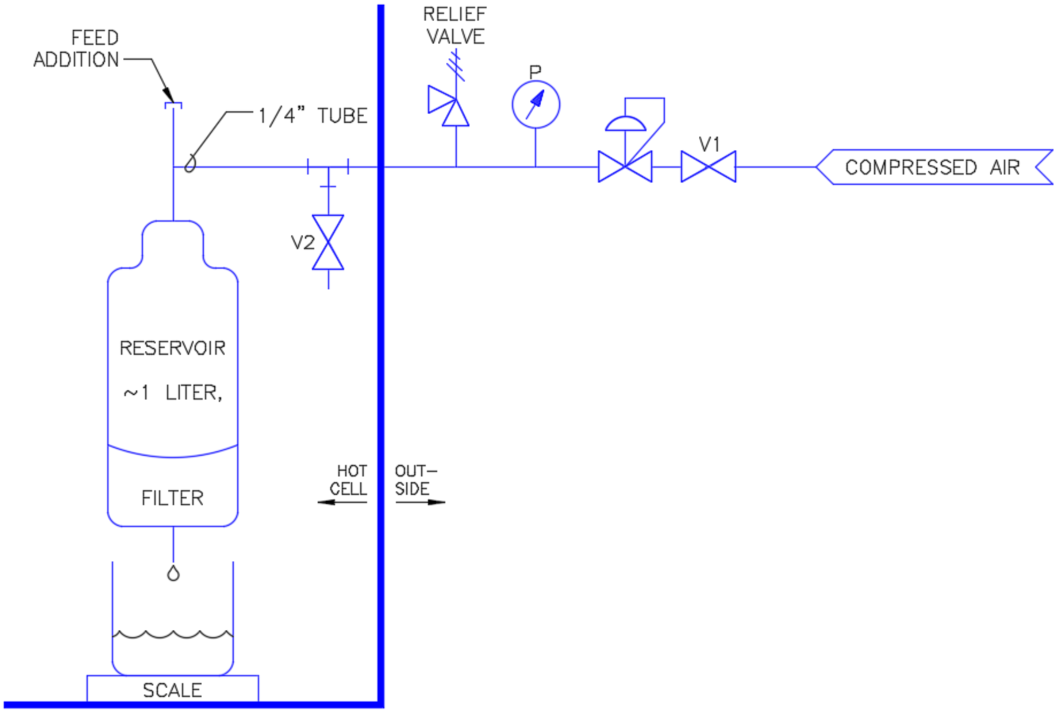

Figure 1.6. Schematic of Dead-End Filtration Test Setup

1.5 Quality Assurance

This work was conducted with funding from 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 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

Thirty-six supernatant samples (~250 mL each) were taken from Tank AP-107 in October 2017 and transferred to the Radiochemical Processing Laboratory. Filtration testing occurred in March 2018 and evaluated four new sintered metal Mott filters:

1. The **media grade 5 CFF** is a sintered metal Mott Model 7610 CFF with a 5/8-in. OD, 1/2-in. ID, 6-in. active length, and 9.4-in^2 (0.065 ft^2) filter area.
2. The **media grade 0.1 CFF** is a sintered metal Mott Model 7610 CFF with a 5/8-in. OD, 1/2-in. ID, 6-in. active length, and 9.4-in^2 (0.065-ft^2) filter area.
3. The **media grade 5 BDEF** is a sintered metal Mott Model 6480 in-line filter with a 3/8-in. OD, 1/4-in. ID, 2.75-in. active length,[1] and 3.24-in^2 (0.023-ft^2) filter area.
4. The **media grade 0.1 DEF** is a sintered metal Mott Grade 0.1 disc filter, 1/16-in. thick, 4.4 in^2 filter area.

The CFFs (1 and 2) were only tested in recycle mode.[2] All dewatering of AP-107 was performed with Filters 3 and 4. Testing was configured such that Filters 1-3 were all tested with fresh (i.e., new out of the AP-107 sample bottle) feed.

2.1 CUF and BDEF Testing

The CUF and BDEF equipment and testing was integrated, as Filters 1, 2, and 3 could be aligned or isolated by opening and closing a few valves (see Sections 1.2 and 1.3). Because the CUF/BDEF system requires fluid for the CUF pump to operate, filtration of the final 1.9 liters was performed with the DEF disc filter as described in Section 2.2. A more detailed description of the testing can be found in Appendix A.

The evolutions used to test the AP-107 are outlined below.

1. Performed clean water flux (CWF) tests, which served to measure the filter permeability (and verify the system had no leaks).
2. Composited AP-107 feed and performed recycle mode with media grade 5 CFF (Filter 1).[3]
    a. Composited AP-107 feed in CUF.
    b. Ran CUF in recycle (TMP: 1.7 psig; AV: 14.7 ft/s) for 28 hours with no backpulsing.
3. Aligned BDEF (Filter 3) in dewater mode and dewatered AP-107 to produce IX feed. Took sample of filtered permeate for total alpha testing.

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[1] The filter was modified from a standard 6-in. length at PNNL. The filter was cut approximately in half and a new end-cap was welded on the filter. The welds were inspected and approved prior to use in the hot cell.

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

[3] The goal of the Test Matrix with the 5 media grade filter was to match test conditions of the 0.1 the media grade CFF performed in Evolution 5. However, the filter flux with the 5 media grade filter was so high that the Coriolis and glass flowmeters were over-scaled at the targeted TMP. As a result, the test conditions were modified (see Section 3.1).

4. Composited AP-107 feed in CUF run in dewater mode with BDEF (Filter 3) to produce IX feed. Composited additional AP-107 feed and continued to dewater.
5. Composited AP-107 feed and performed with 0.1 media grade CUF (Filter 2).
    a. Composited AP-107 feed in CUF.
    b. Ran CUF in recycle (TMP: 10 psig; AV: 14.7 ft/s) for 7 hours.
    c. Backpulsed, then continued running in recycle (TMP: 15 psig; AV: 14.7 ft/s) for 7 hours.
    d. Backpulsed, then continued running in recycle (TMP: 20 psig; AV: 14.7 ft/s) for 7 hours.
    e. Backpulsed, then continued running in recycle (TMP: 10 psig; AV: 14.7 ft/s) for 7 hours.
6. Aligned BDEF (Filter 3) in dewater mode and dewatered AP-107 to produce IX feed.
7. Composited AP-107 feed in CUF. Backpulsed BDEF (saved sample for particle size distribution analysis) and then ran in dewater mode with BDEF (Filter 3) to produce IX feed.
8. Drained CUF and subsampled slurry. Took samples of drained AP-107. (Filter drained CUF material through DEF – see Section 2.2).
9. Backpulsed filters and rinsed system and performed CWF tests.
10. Cleaned system with 2 M HNO_3 and performed CWF tests.
11. Layed up CUF and BDEF.

Table 2.1 provides a mass balance for CUF/BDEF testing. A total of 12,111 g of material was added to the CUF during testing. A total of 11,871.5 g was accounted for, including 20 g estimated as lost during a transfer. The missing mass (239.5 g) is due to evaporation and material that wets the inside of the CUF and is not recoverable, and represents less than 2% of the initial feed.

2.2 DEF Testing

The evolutions used to test the AP-107 in the DEF are outlined below.

1. Performed CWF test.
2. Added AP-107 (drained from CUF) and filtered at 10 psig.
3. Added AP-107 (drained from CUF) and filtered at 20 psig.
4. Rinsed DEF system.

Table 2.2 provides a mass balance for DEF testing. A total of 2372.8 g of material was added to the DEF. A total of 2356.4 g was accounted. That left 16.4 of material unaccounted. The missing mass is material that wets the inside of the DEF and is not recoverable, and represents less than 0.7% of the feed.

Table 2.1. Mass Balance – CUF/BDEF

		IN (g)	OUT (g)				
Evolution	Description	AP-107	Feed to DEF	Dewater (to IX)	Permeate Sample	Slurry Sample	Transfer Loss
2, 3	Composite Feed, Test Matrix, and BDEF Dewater	4055.1	0	971.3	12.6	0	0
4.1	Composite Feed and BDEF Dewater	2377.2	0	2293.4	0	0	0
4.2	Composite Feed and BDEF Dewater	2326.6	0	2314.1	0	0	20
5, 6	Composite Feed, Test Matrix, and BDEF Dewater	2355	0	2437.6	0	0	0
7	Composite Feed and BDEF Dewater	997.1	0	1313.8	0	0	0
8	Drain CUF	0	2327.2	0	0	181.5	0
	Subtotals	12,111	2327.2	9330.2	12.6	181.5	20
	Total	12,111	11,871.5				

Table 2.2. Mass Balance-DEF

		IN (g)	OUT (g)	
Evolution	Description	AP-107 Fed to DEF	Permeate Collected (to IX)	Solids Recovered
2	Filtration of AP-107 (Batch 1)	1195.5	1185.7	
3	Filtration of AP-107 (Batch 2)	1177.3	1170.0	
5	Wet Solids Recovered from Filter			0.7
	Subtotals	2372.8	2355.7	0.7
	Total	2372.8	2356.4	

2.3 Filter Cleaning

Filters 1-3 were cleaned as indicated in this section. Filter 4 is considered a single-use filter and was disposed of after DEF testing and subsequent solids recovery.

1. The CUF/BDEF system was drained and rinsed with 2 liters of 0.01 M NaOH solution.
2. Filters 1, 2, and 3 were backpulsed.
3. The CUF/BDEF system was drained and filled with 2 liters of fresh 0.01 M NaOH solution.
4. The filter flux was measured for Filters 1, 2, and 3.
5. The CUF/BDEF system was drained and filled with 1.5 liters of 2 M HNO_3.
6. Filters 1, 2, and 3 were backpulsed twice with the acid solution.
7. The CUF/BDEF system was drained and filled with 2 liters of fresh 0.01 M NaOH solution and each of the filters (1, 2, and 3) was backpulsed.
8. The final CWF was measured for each filter.

Because the filter flux is so high for the media grade 5 filters (1 and 3), a single pressure was used for the CWF measurement. Higher pressures result in permeate flow rates above the range of the Coriolis and glass flowmeters. The CWF for the 0.1 media grade filter (Filter 2) was conducted for 5 minutes at 5, 10, 15, and 5 psig. In all cases, the recirculation rate of the CUF was 11 ft/s.

3.0 Results

3.1 Crossflow Filtration Results

The media grade 5 CFF was run for 28 hours in recycle mode.[1] Figure 3.1 shows the pressure normalized permeate flux under constant AV (14.7 ft/s) and TMP (1.7 psig). The plan was to test the filter at four TMP conditions of 10, 15, 20, and 10 psig, but the flux was beyond the scale of instrumentation at pressures above 2 psig. The flux rate was very high and averaged 0.9 gpm/ft^2/psi. The flux required no correction for temperature because the temperature remained constant at 24 +/-1 °C throughout this phase of testing.

It is believed that the filter was not challenged with the 3.2 liters of AP-107 supernatant used in this recycle test due to the lack of flux decline over the 28-hour testing period; i.e., there may have been insufficient solids in the 3.2 liters of feed to add any appreciable resistance to filtration. Because the permeability of the 5-micron grade filter is approximately 50 times higher than the 0.1-micron grade filter, fouling may not manifest without significantly more feed.

The very high flux rates and lack of evident fouling made us wonder whether small particles that cause fouling in the 0.1 media grade filter passed through the 5 media grade filter; however, six effluent bottles that were dewatered through the 5 media grade filter were re-filtered with the 0.1 media grade DEF and no recoverable solids were found. This possibly indicates that solids are getting trapped in the filter, but not enough volumetrically to appreciably impact the flux (as was seen with the 0.1 media grade filter).

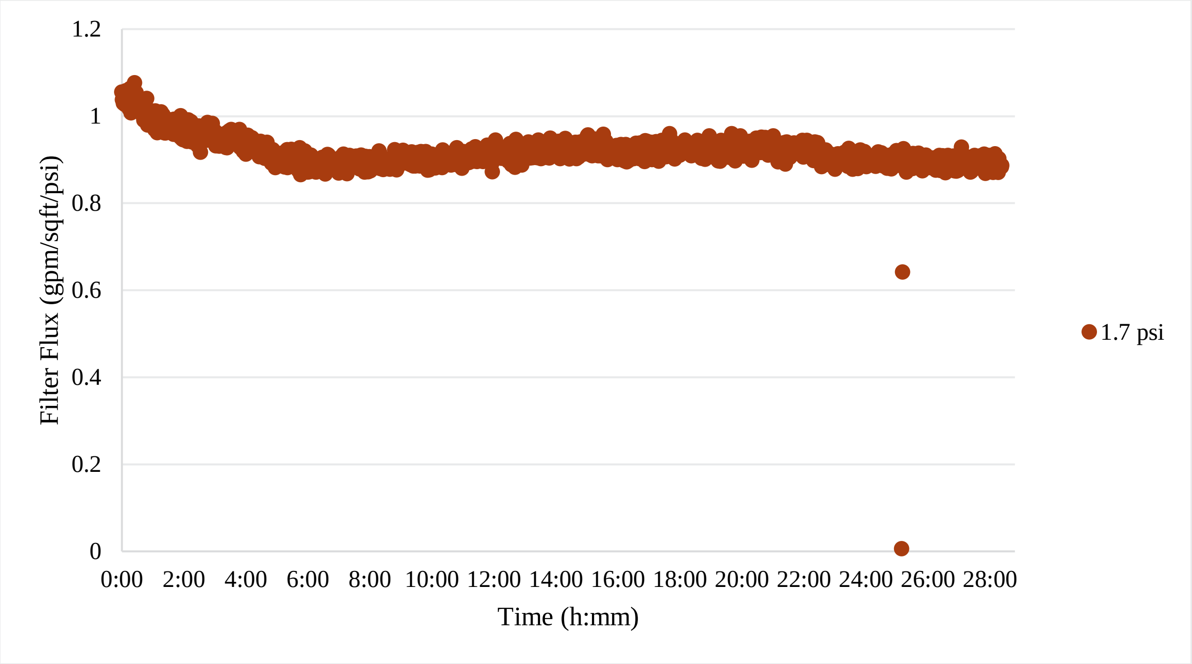

Figure 3.1. Recycle Test: 5 Media Grade Crossflow Filter (AP-107)

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

The CUF test matrix with the media grade 0.1 filter involved running the system in recycle mode for approximately 28 hours at 10, 15, 20, and 10 psi TMP and 14.7 ft/s AV. Figure 3.2 shows the pressure-normalized permeate flux under four test conditions at constant AV. Each of the data sets in Figure 3.2 started with a backpulse (Time 0) and ran for approximately 7 hours. Backpulses were conducted at approximately 80 psi. The normalized flux declines with increasing pressure. Backpulsing was effective in largely restoring the flux, and we note a small decline in flux between the first and last condition run at the same TMP.

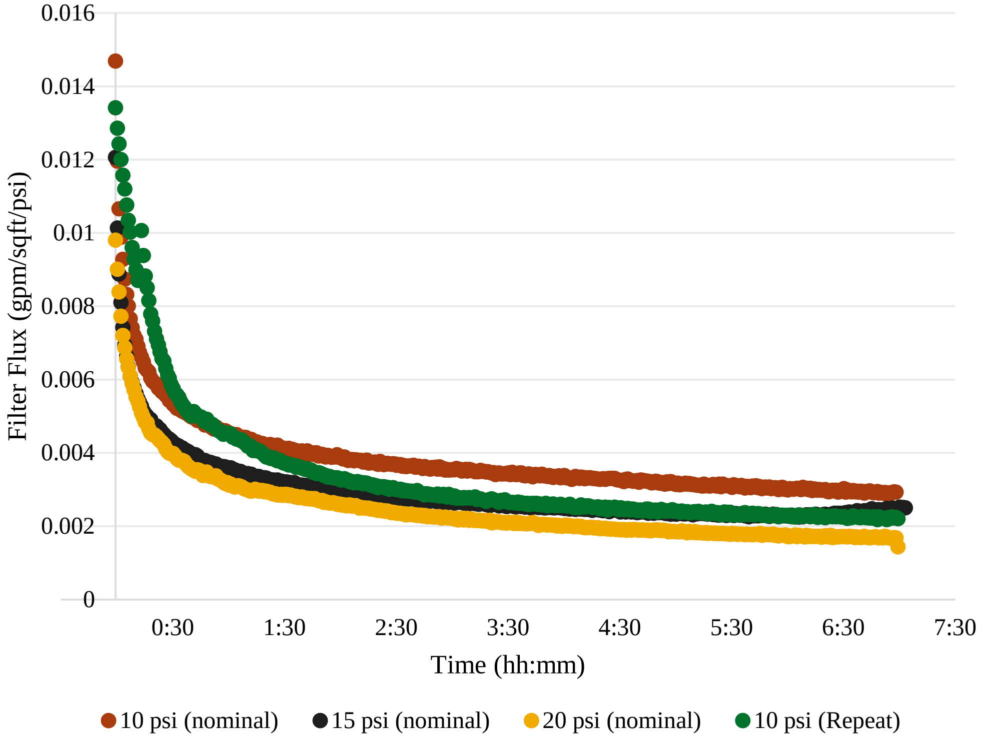

Figure 3.2. Recycle Test: 0.1 Media Grade Crossflow Filter (AP-107)

Figure 3.3 provides a plot of the 5 and the 0.1 media grade filter flux for AP-107. The filter flux of the media grade 5 filter was on average 200 times higher while still effectively filtering the solids.

Figure 3.4 compares the filtration rates of AP-107 and AP-105 (reported in Geeting et al. 2018), which were both tested with the media grade 0.1 CFF. The AP-107 filtration rate was approximately 2 times higher under similar test conditions[1], indicating that the AP-105 is a more difficult feed to filter. Additional validation comes from comparing AP-107 and AP-105 dead-end filtration results (see Section 3.3), where data also indicates AP-107 had ~2 times greater flux over AP-105.

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[1] Note AP-107 and AP-105 have different pressure profiles for the 21-28 hour segments, which may lead to different fouling characteristics.

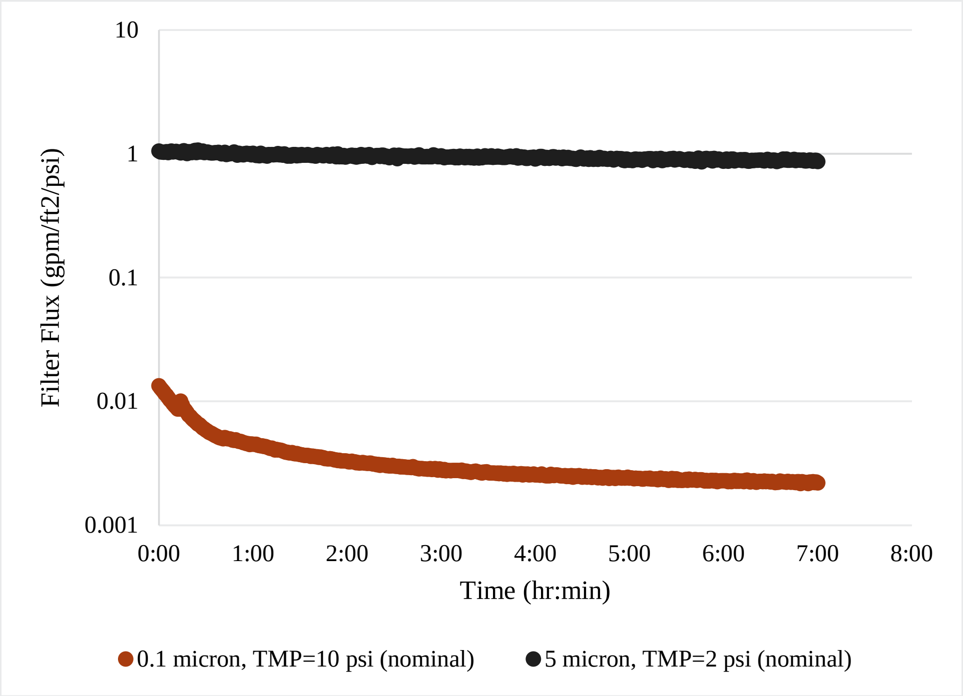

Figure 3.3. Recycle Test: 0.1 vs. 5 Media Grade Crossflow Filter (AP-107)

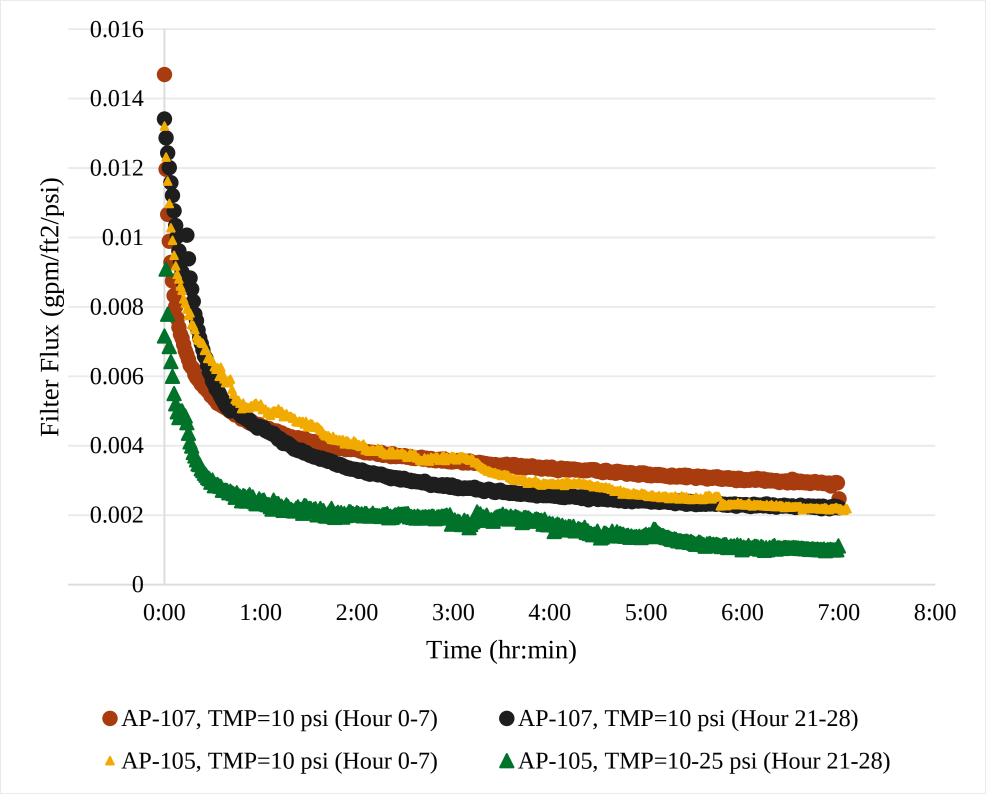

Figure 3.4. CUF Recycle Test: 0.1 Media Grade Crossflow Filter (AP-107 vs. AP-105)

3.2 BDEF Filtration Results

The BDEF was only run in dewatering mode.[1] The permeate flow was throttled to prevent the Coriolis flowmeter from being over ranged (range = 0-30 lph); as a result, the TMP floated during testing. That is, at the beginning of the test the TMP was about 5 psi, and as the solids loaded onto the filter it increased as indicated in Figure 3.5, which shows disparate BDEF dewatering events knitted together on a continuous time axis. The discontinuities in the data are when filtration was stopped to add more AP-107 feed to the CUF and change permeate containers. (The permeate was collected in a bottle that had to be changed when full.) Table 3.1 provides the key events and times relative to the figure, as well as event durations.

The BDEF filtration data observed during the initial 2 minutes of BDEF operation (and occurring immediately after CFF operation) is anomalous. Specifically, the flux remains constant following the media grade 5 CFF (Event a in Figure 3.5), indicating no solids building up on the filter. It appears that the 28-hour recycle test with the media grade 5 CFF removed solids from the system. It is not until fresh feed is added to the CUF/BDEF system (Event b in Figure 3.5) that the BDEF filtration flux declines with time.

Similarly, the 28-hour recycle test with the 0.1-µm CFF (Event f in Figure 3.5) also appears to have caused anomalies in the BDEF data (i.e., the flux after Event f (time 17:27 in figure) is higher than expected). It is suspected that during the testing of the CFF, the BDEF cake may have settled off of the surface of the filter on termination of BDEF flow, resulting in higher than expected normalized flux (and lower TMP) relative to filtration data just prior to the 0.1 media grade CFF test.

The BDEF was backpulsed at time 25:42 on the figure (Event h) and the flux rate was fully restored, indicating that the flush was effective at removing the filter cake. The backpulse was conducted at 30 psi, and 140 mL of flush fluid containing the concentrated solids were removed from the CUF/BDEF system. The concentrated solids from the backflush were removed from the CUF/BDEF system. Solids in the backflush were visible to the eye and were subsequently analyzed for particle size distribution (See Section 3.5.3.4).

The media grade 5 BDEF was effective at removing solids from the system. 5.1 liters of filtered permeate from the BDEF were subsequently refiltered with a media grade 0.1 filter on the DEF. No solids were recoverable on the DEF from this second filtration.

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[1] In dewater mode, the filtered permeate is removed from the CUF/BDEF system.

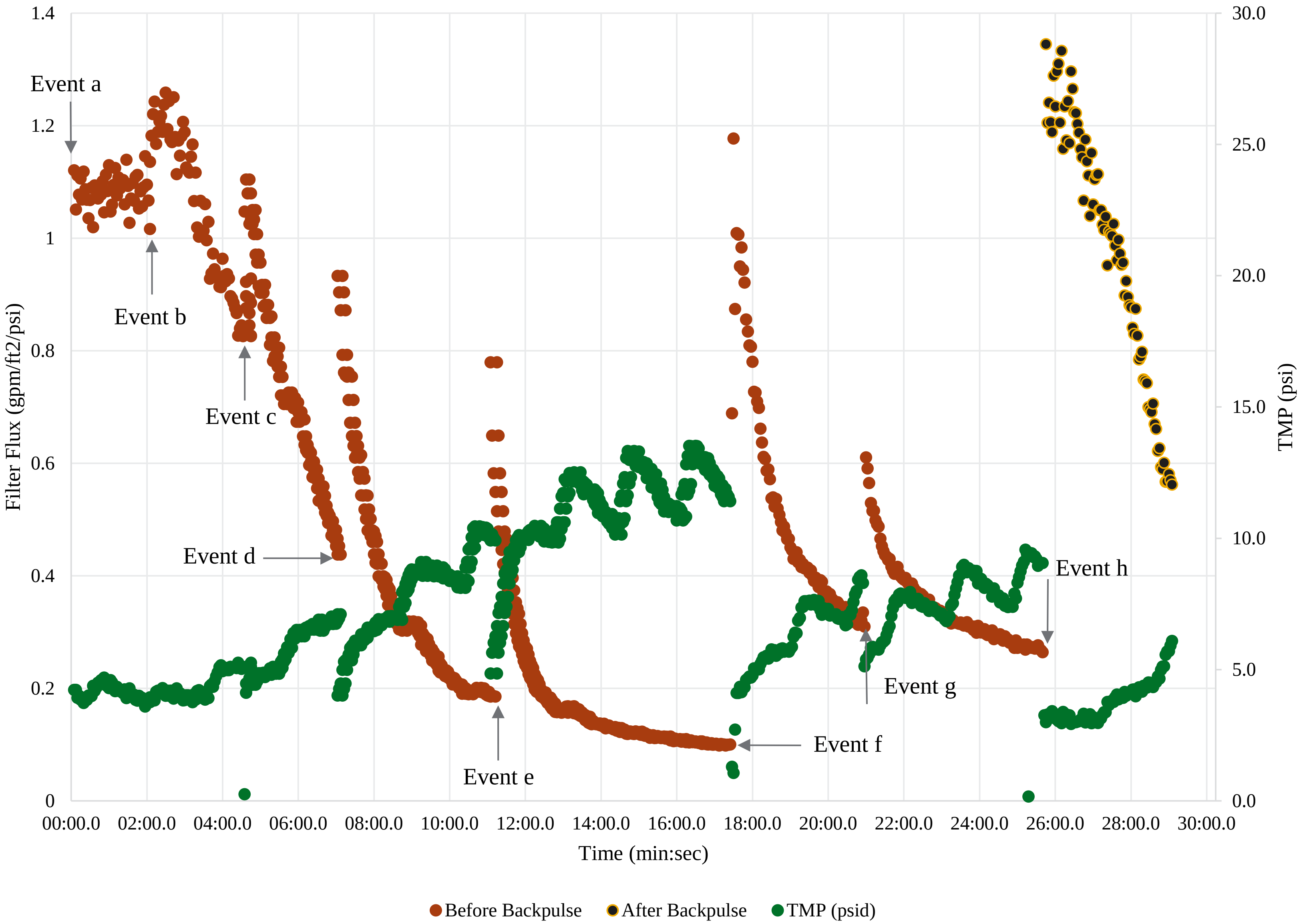

Figure 3.5. BDEF Filtration

Table 3.1. BDEF Key Events

Test Step (from Section 2.1)		Event	Time on Figure (min:sec)	Actual Duration of Event (hours:min)
2	a)	Recycle test with 5-µm CFF	<00:00	28:24
4	b)	Add more feed to CUF and change permeate bottle 1	02:07	1:36
4	c)	Change to bottle 2	04:37	0:26
4	d)	Add more feed to CUF and change to permeate bottle 3	07:10	1:31
4	e)	Change to bottle 4	11:15	0:19
5	f)	Add more feed to CUF, recycle test with 0.1-µm CFF, change to permeate bottle 5	17:27	30:30
6	g)	Change to bottle 6	20:57	0:06
7	h)	Add more feed to CUF, change to bottle 7 and backpulse	25:42	1:35

3.2.1 Projection for LAWPS Filtration

This section estimates how long the LAWPS filtration system may be expected to run between backpulses based on data obtained from the BDEF testing of AP-107.

The following LAWPS filtration information was provided by WRPS[1]:

- LAWPS filter area: 67 ft^2
- Targeted LAWPS filter flow: 10 gpm
- Maximum LAWPS TMP: 10 psig

Figure 3.6 shows the BDEF filtration data (before the backpulse)[2] plotted as filtration resistance as a function of volume filtered. Filtration resistance was determined using the following relationship shown in Eq. (1).

$$R_m = \frac{p_m}{\mu q} \tag{1}$$

Where R_m is medium resistance (1/m), p_m is filtration pressure (Pa), μ is liquid viscosity (kg/m-s), and q is equal to filtration rate ($m^3/m^2/s$).

A best fit line equation is shown for each data set. As indicated in the figure, the slope of the resistance increases with volume filtered (and TMP), indicating that a compressible cake is forming on the BDEF. The resistance (and slope) of the data after the 0.1 media grade CFF testing (circled in figure) doesn't

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[1] Email from Rose Russell (WRPS) to John Geeting (PNNL) dated May 17, 2018.

[2] Data immediately after the media grade 5 CFF test until fresh AP-107 feed is added to the CUF/BDEF system is excluded because it showed no flux decline with time, indicating there was no filter cake buildup.

match up well (lower resistance and slope than expected) and may indicate that cake sluffed off the filter during the 30-hour intermission between BDEF filtration events. As a result, the data (circled in Figure 3.6) were also excluded from further analysis.

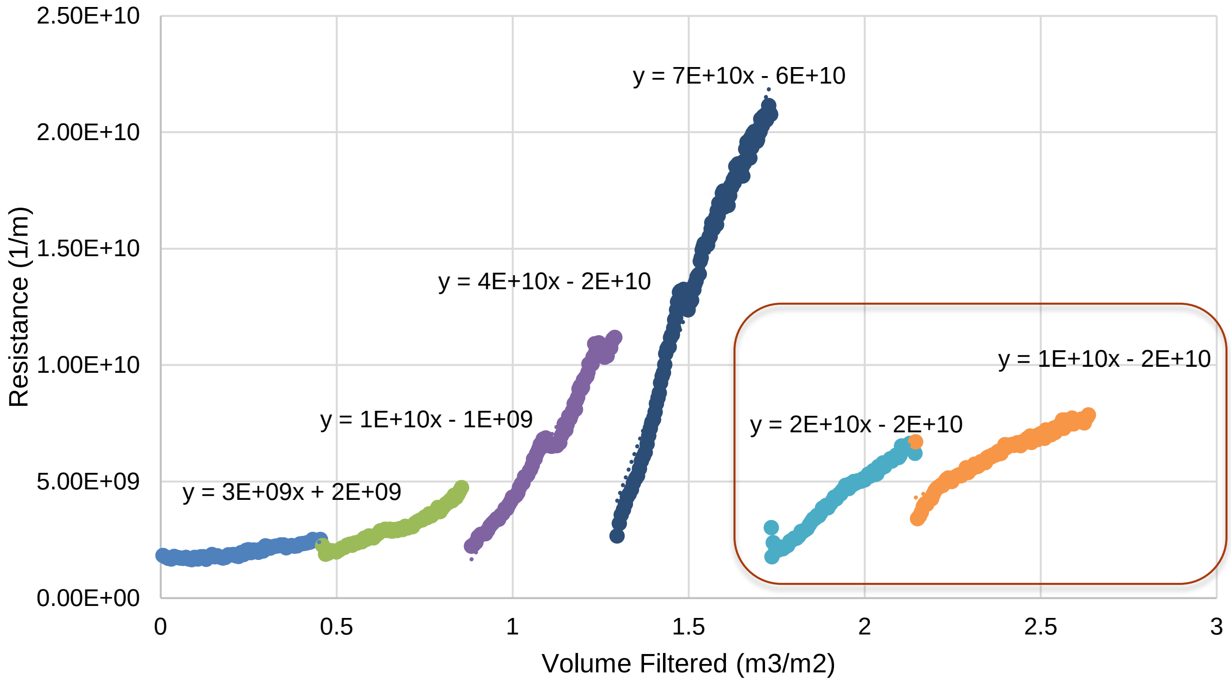

Figure 3.6. BDEF Filtration of AP-107 until Backpulse

Figure 3.7 shows a similar figure with the selected data along with data taken after the backpulse. The post-backpulse data match the pre-backpulse data well, indicating that the backpulse was effective at removing solids.

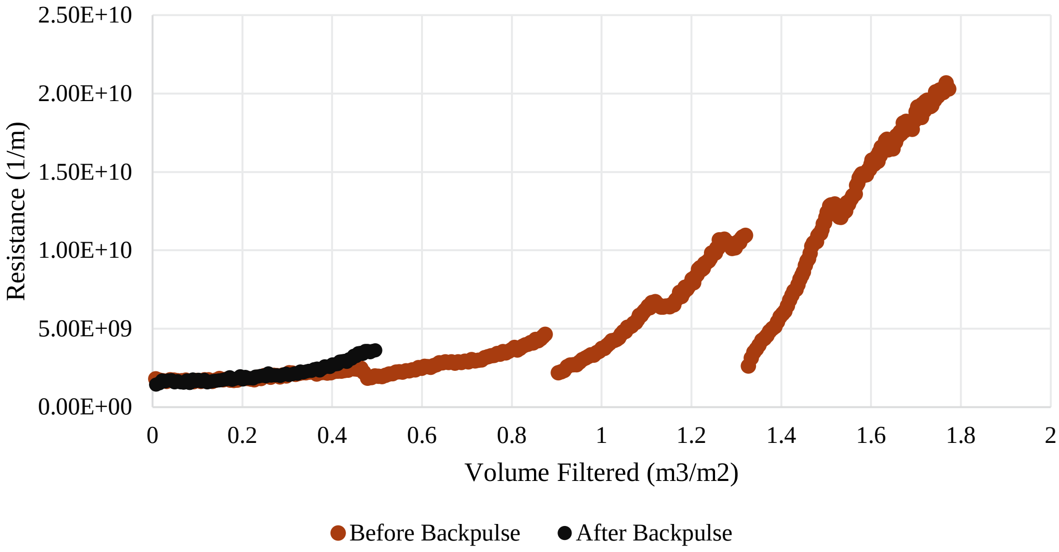

Figure 3.7. BDEF Filtration of AP-107 with Post-Backpulse Data Added

Figure 3.8 shows an empirical fit of the data. Data were excluded where the cake appears to reform (due to the discontinuities of testing) in order to improve the accuracy of the fit. This empirical fit was then used to estimate filtration resistance in the LAWPS until the correlation reached a slope of 70 x 10^9 ($1/m^2$), at which point this maximum slope was used to estimate the resistance. This slope was chosen as a maximum because it was the maximum measured (see Section 3.3 discussion on DEF filtration) during the filtration of AP-107. A maximum was also imposed on the correlation because the compressibility of the cake has a limit and it appears this limit was reached during the DEF filtration (conducted at 10 and 20 psi).

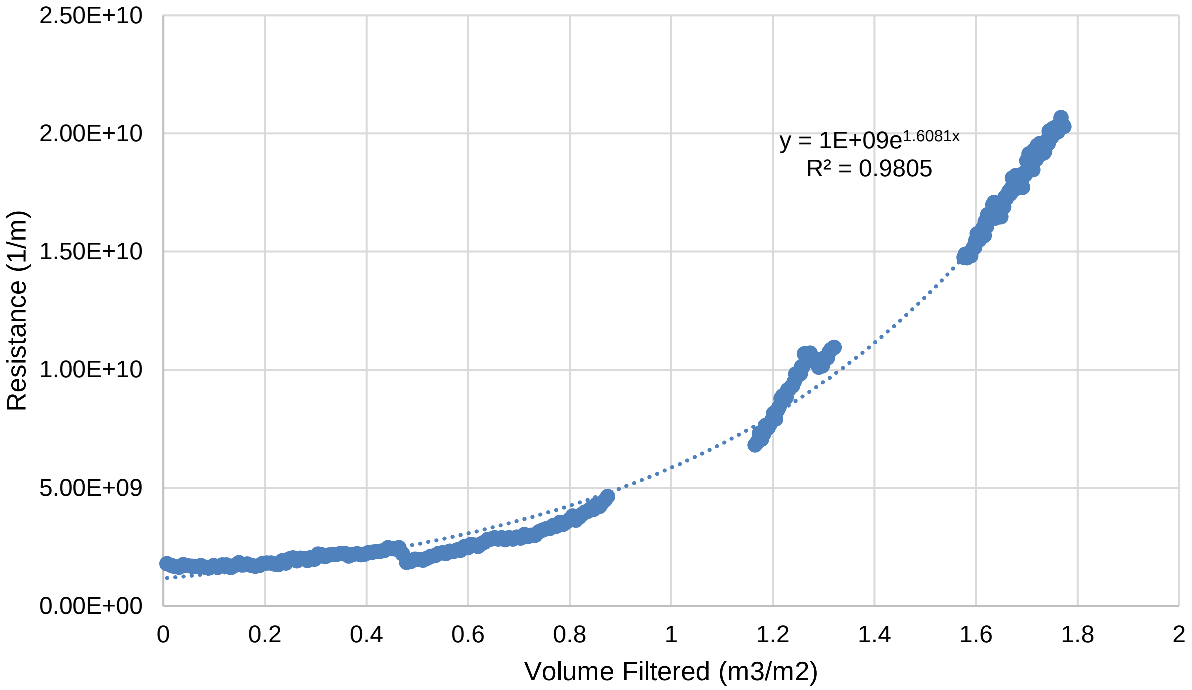

Figure 3.8. Empirical Fit of BDEF Data

Using this correlation, Figure 3.9 shows the projected TMP as a function of volume filtered for the 67-ft^2 LAWPS system. Because LAWPS plans to filter at a constant 10 gpm, the volume filtered is directly relatable to time of filtration as shown in Figure 3.10. Based on a maximum LAWPS TMP of 10 psi, this figure projects that backpulsing of the LAWPS filter should occur approximately every 10 hours when filtering AP-107. The following caveats should be noted:

1. The projection was made using 14 minutes of BDEF filtration data (at much higher rates than proposed for LAWPS filtration) vs. days of filtration performance required for the LAWPS filter.
2. The projection was made with a limited volume: 2.2 m^3/m^2 vs. 4.9 m^3/m^2 (BDEF data used in correlation vs. LAWPS minimum to make 12 hours between backpulses).
3. The projection was made at much smaller scale. The BDEF area is ~1/3000 the area of the proposed LAWPS filter (3.24 in^2 vs. 67 ft^2).

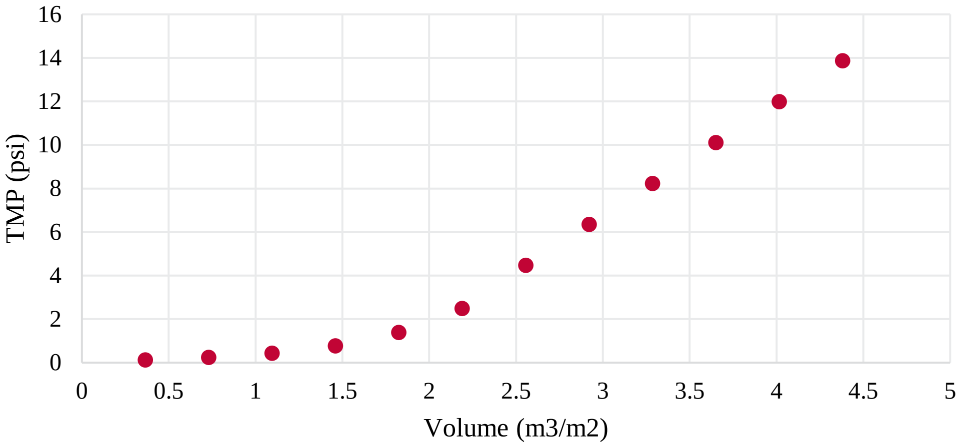

Figure 3.9. Projected LAWPS Filter TMP as a Function of Volume Filtered

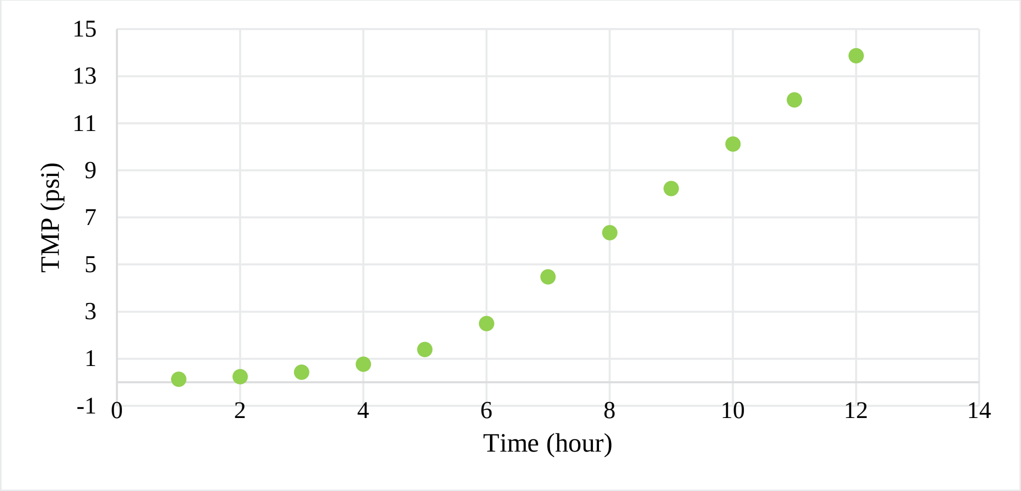

Figure 3.10. Projected LAWPS Filter TMP as a Function of Time (assuming 10-gpm filtration rate)

3.3 DEF Filtration Results

AP-107 drained from the CUF was filtered through the DEF and solids were collected. The AP-107 was filtered in two (~1-liter) batches, the first at 10 psi, and the second at 20 psi. Figure 3.11 shows flux data for this testing. For comparison, this figure also shows similar data taken for AP-105 DEF filtration (Geeting et al. 2018). It is apparent that as additional material is filtered, filter performance declines due filter cake building up on the surface of the filter. AP-107 has flux values roughly 2 times that of AP-105. This is consistent with what was seen with recycle flux values in the CUF with the 0.1 media grade CFF (see Section 3.1).

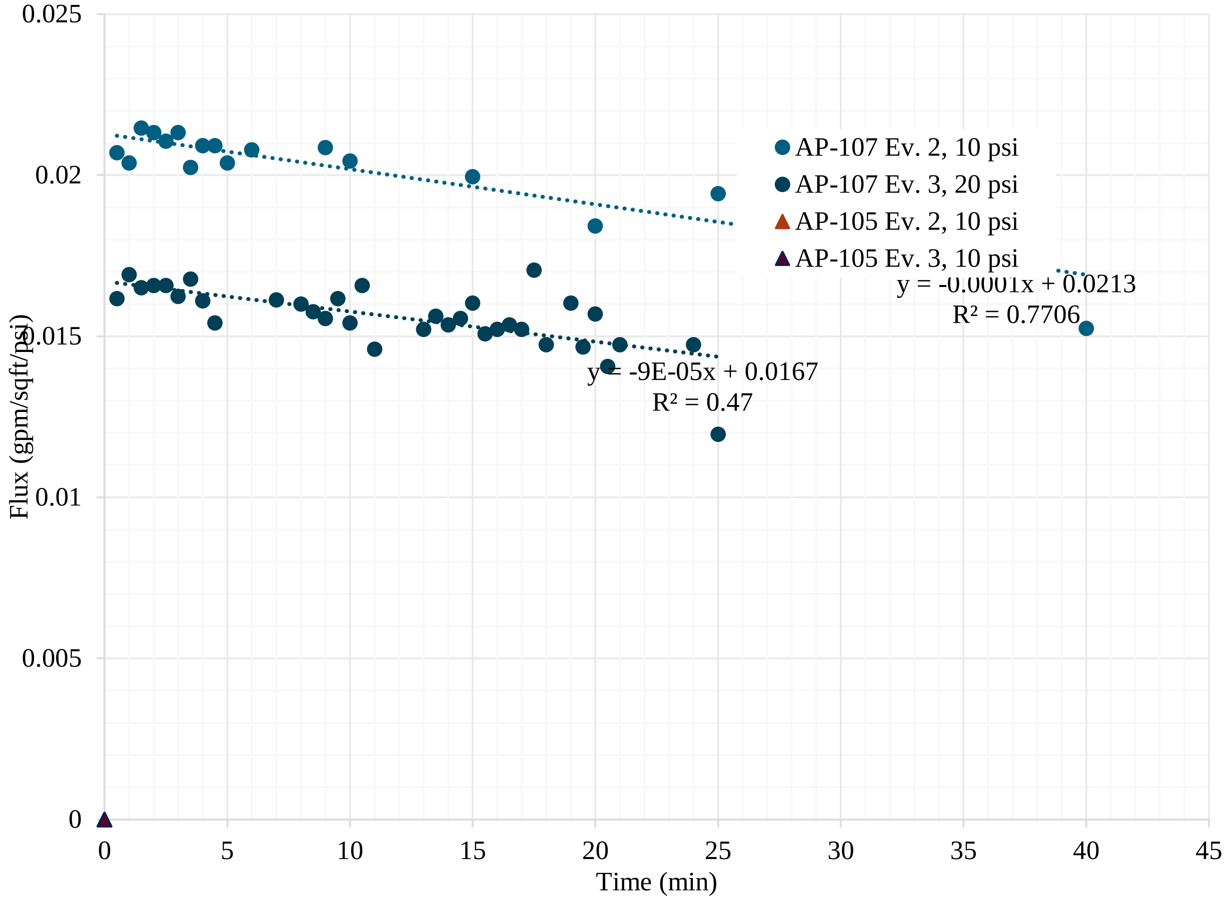

Figure 3.11. DEF Filtration (AP-107 and AP-105)

The solids collected from the filter appeared chalky and hydrophilic. Figure 3.12 is a picture of the solids on the filter after the DEF filtration, and Figure 3.13 is a picture with some of the solids scraped off the filter surface.

In addition to the AP-107 slurry, six bottles of CUF dewatered permeate were run through the DEF in order to collect additional solids that may have passed through the 5 media grade BDEF. Very minimal solids were found on the filter after passing the BDEF-filtered permeate through the DEF, indicating that the media grade 5 filter was effective at removing solids from the permeate.

Figure 3.12. Solids on Filter after DEF Filtration

Figure 3.13. Solids Scraped Off the Filter Surface after DEF

Figure 3.14 shows a graph of resistance vs. volume for the two DEF evolutions. The intercept of this graph represents the media resistance of the new clean filter. As the AP-107 is filtered, the resistance increases due to a cake formation on the filter surface. The slope of the resistance graph was maintained between the 10 and 20 psi data sets, suggesting that the cake layer has reached maximum compression at 10 psi and that the maximum slope is 7E+10 (1/m^2).

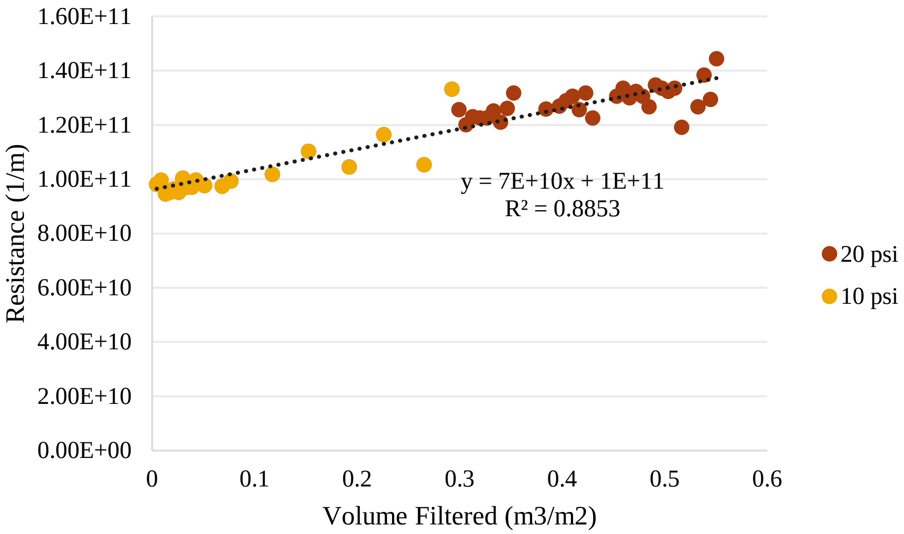

Figure 3.14. Resistance vs. Volume for DEF Evolutions 2 and 3

Given an estimated cake thickness at the conclusion of DEF testing of 0.5 mm, a solids permeability has been calculated to be 6 E-15 m^2. AP-107 feed is supernate sampled from tank farms with “no apparent” solids; however, small amounts of solids present have low permeability and, as indicated in Table 3.2 (Chen et. al 1996), are considered “difficult” to filter.

Table 3.2. Ease of Filtration with Direct Relationship to Filter Permeability

Ease of Filtration	Permeability (m^2)
Very easy	10^{-12}
Easy	10^{-13}
Moderate	10^{-14}
Difficult	10^{-15}
Very difficult	10^{-16}

3.4 Clean Water Flux

Figure 3.15 shows the CWF of the media grade 5 CFF before testing (New Filter), after testing (After AP-107 Filtration), and after acid cleaning with 2 M HNO_3 (Post Clean). The flux was high in all cases and shows some improvement after testing, which may be due to removal of fabrication residue in the high caustic feed. Acid cleaning resulted in very minor improvement and indicates that the media grade 5 filter did not foul during testing.

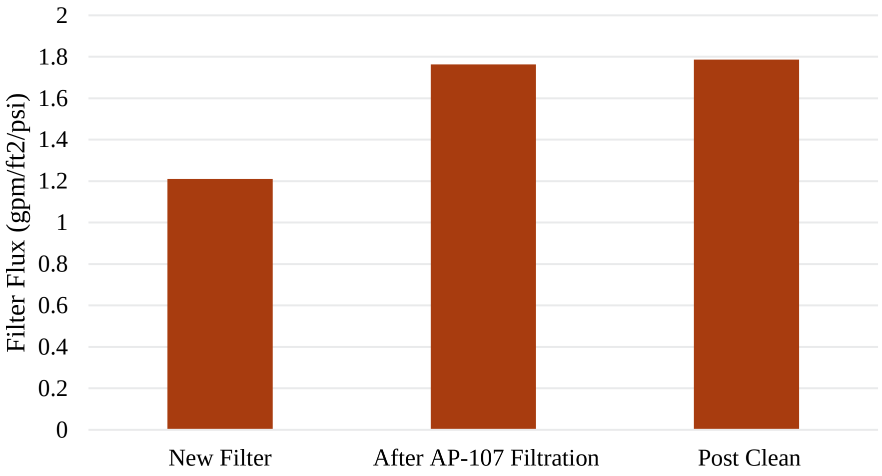

Figure 3.15. CWF Measurements for 5 Media Grade CUF at 1 psi

Figure 3.16 shows similar data for the media grade 5 BDEF (Mott in-line model 6480). Only very minor improvement in flux is measured after acid cleaning. Although the CFF and the BDEF had the same media grade, the flux rates for the BDEF were generally higher. Both filters are 1/16 in. thick, so the difference must be due to fabrication variability.

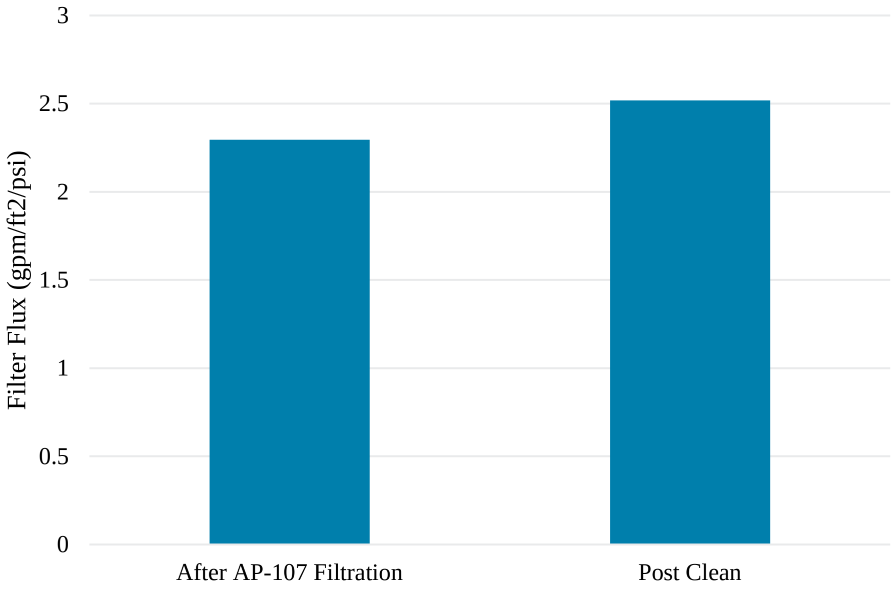

Figure 3.16. CWF Measurements for 5 Media Grade BDEF at 1.5 psi

In contrast to the media grade 5 filters, the media grade 0.1 filter indicates the CUF system had very small particulate that resulted in flux decline for the media grade 0.1 (but not the media grade 5) filter. Figure 3.17 shows the flux decline measured prior to testing, indicating that the CUF system was not completely clean. (After these measurements, the CFF was backpulsed and the CUF system rinsed, and the flux was restored to 0.044 gpm/ft^2/psi prior to initiating testing with AP-107.)

The post-testing CWF shows a significant improvement and we observe that higher pressure causes a lower normalized filtration rate. Acid cleaning the system actually resulted in a decline in the measured CWF for the first three conditions tested. Acid cleaning may decrease the size of particles in the CUF and make these smaller particles more prone to fouling the media grade 0.1 CFF. It is interesting to note that the final condition at 5 psi showed no flux decline (actually a small increase) relative to the first condition at 5 psi, suggesting that the CUF system had no further solids that impacted filter performance.

These tests generally show that the media grade 5 filter is more robust (in terms of flux decline) and may be better suited for the filtration of the very low solid concentrations found in AP-107.

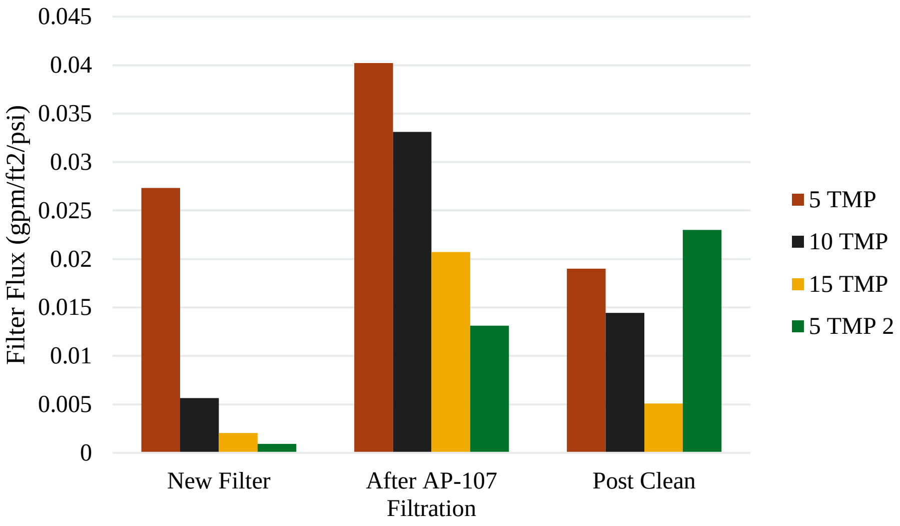

Figure 3.17. CWF Measurements for 0.1 Media Grade CUF

3.5 Sample Analysis

3.5.1 As-Received AP-107

Thirty-six samples were received from Tank AP-107. 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 the first and last of these bottles was obtained to validate the target feed composition. The target feed composition was validated, characteristics of these materials were averaged together in Table 3.3.

Table 3.3. Composition of Sample from 7AP-17-11 and 7AP-17-46

Analyte	Average (µg/L)	Molarity
Al	9825	0.364
K	3792.5	0.097
Na	129250	5.622
B	36.475	0.003
Ca	31.475	0.001
Cr	493.25	0.009
P	650.25	0.021
S	1697.5	0.053
Si	40.325	0.001

The density of as-received AP-107 was measured on samples 7AP-17-11 and 7AP-17-46 using a 10-mL A grade volumetric flask and an analytical balance. The density values obtained were 1.270 and 1.263 g/mL, respectively, at an ambient temperature of 26.6 °C.

3.5.2 Slurry and Permeate

Sample TI-031-E3-P was taken during evolution 3 and is permeate sampled from the 5 micron grade BDEF filter. Sample TI-031-E8-S1 is a sample of the back pulse collected from the BDEF filter taken during evolution 8. Alpha emitters were measured in both the permeate and slurry sample and comparison of the results suggest that the solids were dilute enough that they did not contribute significantly to the measured alpha activity in the slurry sample. (See Table 3.7 for alpha analysis of the solids.) The gross alpha results on these two samples have high uncertainty and the activity measured is lower than the sum of the activity from the radionuclides listed. The sum of the activity from the radionuclides has lower error and should be given precedence in any future analysis. Table 3.4 summarizes the results.

Table 3.4. AEA Results for ASR 0500, Slurry and Permeate: Measured Alpha Emitters, Bq/mL ± 1-σ

Sample	Gross Alpha	U-234+Np-237	Pu-239+240	Pu-238+Am-241	Cm-243+244	Cm-242
TI-031-E3-P	2.85E+1 ±26%	2.10E+0 ±11%	1.84E+1 ±4%	1.73E+1 ±4%	8.81E-1 ±4%	<1.E-1
TI-031-E3-P Duplicate	--	2.68E+0 ±10%	1.86E+1 ±4%	1.61E+1 ±4%	8.07E-1 ±17%	<1.E-1
RPD		24%	2%	7%	9%	
TI-031-E8-S1	2.55E+1 ±28%	2.14E+0 ±11%	1.83E+1 ±4%	1.52E+1 ±4%	6.11E-1 ±21%	<1.E-1

*RPD = relative percent difference

3.5.3 Solids

At the conclusion of CUF testing, the AP-107 slurry drained from the CUF was filtered through a dead-end filter and solids were collected. Solids collected off the dead-end filter weighed 0.5670 g. Using this mass, we can conclude the AP-107 feed had approximately 154 ppm solids (0.5670 g solids/3.68 kg AP-107 feed).

An effort was made to characterize the solids; the collected samples were analyzed by total organic carbon (TOC), inductively coupled plasma-optical emission spectrometry (ICP-OES), gamma emissions analysis (GEA), and alpha emissions analysis (AEA) under ASR 0500. Solids were acid-digested for analysis in the hotcell, in duplicate, and the digestates were surveyed out of the hotcell and delivered to the analytical lab with results indicated below.

Physical property characterization of the AP-107 feed with backpulsed filter solids was limited to particle size analysis of the collected solids.

3.5.3.1 TOC

One solids sample was submitted for organic carbon analysis. The analysis was performed by the furnace method, with 1.8 wt% organic carbon measured.

3.5.3.2 ICP-OES

For 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 K. The solids were not washed prior to analysis, so it is difficult to assess what fraction of the sodium present is from entrained supernate and what fraction is from the solids themselves. However, prior work has suggested that a significant fraction of this sodium is associated with the solids themselves. This suggests insoluble sodium salts. In addition, the presence of insoluble carbon suggests either insoluble carbonate or oxalate solids. This was not verified through XRD analysis. See Table 3.5 for ICP filter solid sample results.

Table 3.5. AP-107 ICP Filter Solids Characterization Results

Analyte	Sample 1 (µg/g)	Sample 2 (µg/g)	Average	Total µg	wt%
Al	30,200	26,900	28,550	16187.9	7.5%
Ca	724	608	666	377.6	0.2%
Cr*	1,730	1,480	1,605	910.0	0.4%
Fe	1,100	875	988	559.9	0.3%
K	10,200	8,810	9,505	5389.3	2.5%
Mg	523	395	459	260.3	0.1%
Na	324,000	327,000	325,500	184558.5	86.1%
P	1,810	1,790	1,800	1020.6	0.5%
S	3,920	5,300	4,610	2613.9	1.2%
Si*	2,290	2,130	2,210	1253.1	0.6%
B	244	244	244	138.3	0.1%
Ni	1,230	967	1,099	622.8	0.3%

*=QC issues arose during analysis. See ASR-0500 in Appendix B for full analysis report.

3.5.3.3 GEA and AEA

Activities for all gamma and alpha emitters detected are shown in Table 3.6 and Table 3.7. 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. The DEF solids sample TI-032-1 duplicates do not agree. The first sample results are consistently higher than the duplicate, well outside analytical uncertainty; thus, results should be used to determine trends as opposed to absolute values.

Table 3.6. GEA Results for ASR 0500, DEF Solids: Measured Activity, Bq/g ± 1-σ

Isotope	TI-032-1	TI-032-1 Duplicate	RPD
Co-60	1.32E+2 ±14%	1.03E+2 ±27%	25%
Cs-134	4.26E+2 ±18%	3.58E+2 ±20%	17%
Cs-137	1.46E+7 ±2%	1.27E+7 ±2%	14%
Eu-152	<1.7E+2	<1.6E+2	--
Eu-154	2.50E+3 ±4%	2.00E+3 ±7%	22%
Eu-155	<1.8E+3	<3.2E+3	--
Am-241	8.40E+3 ±28%	<1.0E+4	--
Am-243	9.90E+3 ±8%	1.60E+4 ±11%	47%
Pu-239	<6.0E+6	<1.0E+7	--

Comparison of the gross alpha measured in the solids with that of the slurry and permeate (Table 3.7 and Table 3.4, respectively) indicates approximately 3 orders of magnitude higher alpha measured in the solids sample.

Table 3.7. AEA Results for ASR 0500, DEF Solids: Measured Alpha Emitters, Bq/g ± 1-σ

Sample	Gross Alpha	U-234+Np-237	Pu-239+240	Pu-238+Am-241	Cm-243+244	Cm-242
TI-032-1	1.36E+4 ±3%	9.07E+1 ±13%	1.68E+3 ±4%	1.17E+4 ±2%	1.47E+3 ±4%	1.15E+2 ±11%
TI-032-1 Duplicate	1.04E+4 ±3%	4.81E+1 ±21%	1.24E+3 ±5%	8.55E+3 ±3%	1.21E+3 ±5%	7.89E+1 ±16%
RPD	26%	61%	30%	31%	19%	37%

*RPD = relative percent difference

3.5.3.4 Particle Size Analysis

Particle size characterization was accomplished using a Mastersizer 2000 (Malvern Instruments, Inc., Southborough, MA) with a Hydro µP wet dispersion accessory. The Mastersizer has a nominal size measurement range of 0.02 to 2000 µm. The actual range is dependent on the dispersion accessory used as well as the properties of the solids being analyzed. When coupled with the Hydro µP wet dispersion accessory, the nominal measuring range of the Mastersizer 2000 is typically reduced to 0.02 to 600 µm (dependent on material density). Although particle sizes above 600 µm can be observed with the Hydro µP, the volume contribution of solids above this size may not be accurately determined by the instrument. Measurement data were recorded and analyzed using Mastersizer 2000 software, Version 5.60. Table 3.8 provides a summary of basic information regarding the Mastersizer analyzer and accessory used for the current particle size measurements. All particle size distribution (PSD) measurements were performed with cesium-ion-exchanged AP-107 supernatant (filtered through a 0.45-µm syringe filter) as the carrier fluid.

Table 3.8. Summary of Malvern Mastersizer 2000 specifications

Parameter	Specification
Analyzer	Mastersizer 2000
Measurement Principle	Laser diffraction (Mie Scattering)
Analyzer Accessory	Hydro µP
Measurement Range	0.02-600 µm nominal
Type	Flow cell system with continuously variable and independent pump and ultrasound
Pump Speed	0-5000 rpm (variable, for analysis 2000 rpm)
Ultrasound Power	0-20 W (variable, for analysis used 0, 10, and 20 W)
Software Version/Date	5.60/1998-2009

Three sub-aliquots were analyzed for particle size distribution from the parent AP-107 slurry sample provided for analysis (i.e., AP-107 feed with backpulsed filter solids). Each sub-aliquot was measured under four conditions: 1) Pre-sonication, 2) 50% Ultrasonic power, 3) 100 % Ultrasonic power, and 4) Post-sonication. The results of the three sub-aliquots were then averaged to obtain a composite PSD for each condition evaluated. The composite PSDs of the collected AP-107 filter solids are shown in

Figure 3.18 with select percentiles given in Table 3.9. As is often the case upon sonication, weak to moderate agglomerates are broken, leading to an increase in the volume contribution of smaller particles. It should be noted that at 100 % ultrasonic power, a population of 300-700 µm "particles" appear. This population disappears once sonication is stopped, i.e., not observed in the post-sonication measurement. This population could either be weak agglomerates or an artefact of sonication.

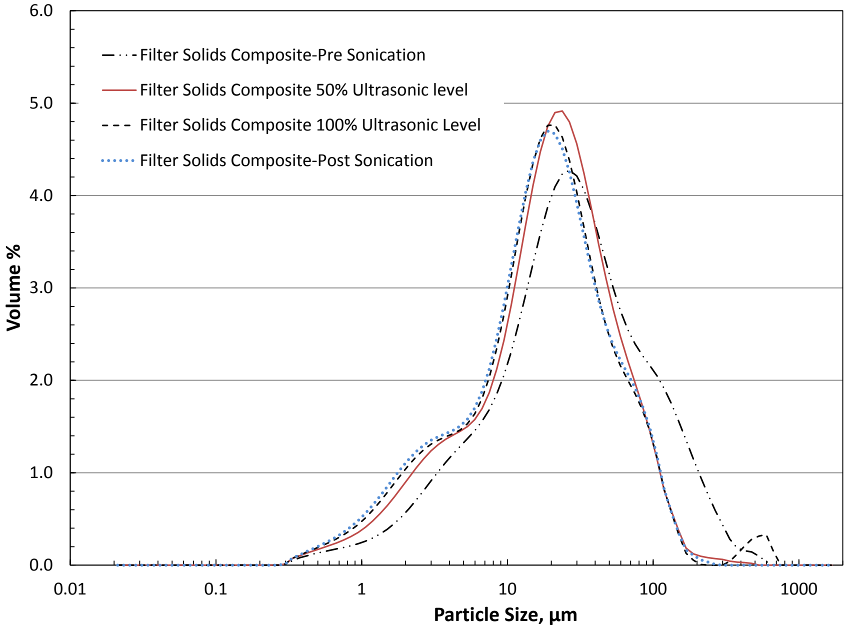

Figure 3.18. Composite PSDs of AP-107 collected filter solids measured in cesium-exchanged AP-107 supernatant.

Table 3.9. Selected Particle Size Percientiles for AP-107 Filter Solids

Percentile	d(0.05)	d(0.10)	d(0.25)	d(0.50)	d(0.75)	d(0.90)	d(0.95)
Filter Solids Composite-Pre-Sonication	2.50	4.44	12.0	26.4	57.6	122	175
Filter Solids Composite-50% Ultrasoinc Level	1.81	3.14	9.21	20.0	37.4	66.6	90.0
Filter Solids Composite-100% Ultrasoinc Level	1.59	2.73	8.25	18.3	35.3	68.9	96.2
Filter Solids Composite-Post-Sonication	1.51	2.57	7.76	17.5	34.1	65.0	88.0

4.0 Conclusions

Based on the results of filtration experiments on AP-107, the following observations and conclusions were made.

- The media grade 5 BDEF was effective at removing solids from the AP-107 feed.
- Filtration resistance measured during BDEF testing was used to estimate that the proposed filter for LAWPS will require backpulsing approximately every 10 hours when running AP-107 feed.
- AP-107 had compressible filter cake.
- The media grade 5 CFF had high flux (~0.9 gpm/ft^2/psi) and exhibited very little flux decline during a 28-hour recycle test with 3.2 liters of AP-107 feed. It is believed that there were insufficient solids in the feed to challenge the filter and the results are not indicative of flux rates with a higher feed to filter area ratio.
- The media grade 0.1 CFF exhibited a flux decline consistent with previous testing and the flux after 28 hours of testing was approximately 0.002 gpm/ft^2/psi. This value was approximately twice that measured in a similar testing conducted with AP-105.
- AP-107 solids collected on the 70-mm disc filter have a different nature than previously observed for AP-105: AP-107 solids appeared chalky and hydrophilic, whereas the AP-105 solids appeared hydrophobic and looked organic in nature.
- The AP-107 supernatant contains solids on the order of 154 ppm. These solids are suspended in the tank waste and are not visible to the naked eye.
- The AP-107 solids did not cause irreversible fouling of the filters, as filtration flux was restorable with rinsing and backpulsing.

5.0 References

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Appendix A

Description of Testing

A.1 Campaign 1

The cells unit filter (CUF) testing was divided into nine principal evolutions, as follows.

A.1.1 Evolution 1

This evolution involved a leak test and clean water flux (CWF) measurements. The leak test added ~2 L of 0.01 M NaOH solution to the CUF while checking for leaks with the system at static pressure, 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.1 for the 5 media grade filter and Table A.2 for the 0.1 media grade filter. At the conclusion of the 0.1 media grade filter run, the filter was backpulsed prior to draining the 0.01 M NaOH to ensure a clean filter for the start of Evolution 2 testing.

Table A.1. CUF Clean Water Flux Measurements for 5 Media Grade Filter

Test Transmembrane Pressure (TMP) (psig)	Initial Rate (L/h)	Final Rate (L/h)	Duration of Test (h:mm)
2	31.17	31.17	0:05

Table A.2. CUF Clean Water Flux Measurements for 0.1 Media Grade Filter

Test TMP (psig)	Initial Rate (L/h)	Final Rate (L/h)	Duration of Test (h:mm)
5.7	3.7	1.5	0:05
10.3	1.5	0.5	0:06
15.2	0.7	0.4	0:05
6.1	0.03	0.03	0:17

A.1.2 Evolution 2

This evolution involved the following steps: (1) composite approximately 3.5 L of feed,[1] and (2) execute the CUF test matrix through the 5 media grade filter.

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[1] All additions and removals of AP-107 to and from the CUF were tracked by mass.

Table A.3 summarizes the masses of samples added to the CUF at the start of this evolution.

Table A.3. Feed Material for Initial 5 Media Grade Filter CUF Testing

Sample ID	Mass Added to CUF (g)	Estimated Volume (mL)
7AP-17-11	310.8	244.7
7AP-17-12	335.7	264.3
7AP-17-13	339.2	267.1
7AP-17-14	345.6	272.1
7AP-17-15	342.4	269.6
7AP-17-16	343.7	270.6
7AP-17-17	337.4	265.7
7AP-17-18	343.3	270.3
7AP-17-19	340.6	268.2
7AP-17-20	339.9	267.6
7AP-17-21	339.6	267.4
7AP-17-22	336.9	265.3
Total	4055.1	3192.9

The CUF was operated with the recirculation of the permeate through the 5 media grade filter for the test condition outlined in Table A.4. Prior to running the system with the set condition, a backpulse was performed. Flux was relatively constant throughout the entire 28-hour run, so a backpulse was not conducted prior to dewatering in Evolution 3.

Table A.4. CUF Testing Conditions for Mott Grade 5 Filter Testing

TMP	Average Flux (gpm/ft^2/psi)	Average Pressure (psig)	Average Velocity (ft/s)	Duration (h:mm)	Density (g/mL)	Slurry Temperature (°C)
1.7 psig	0.916	1.8	14.1	28:23	1.269	24.2

A.1.3 Evolution 3

Dewatering for Evolution 3 was done through the 5 media grade backpulsed dead-end filter (BDEF) and began at a TMP of 4 psi. Dewatering produced one bottle of filtered permeate and was completed within minutes with no change in flux during the duration of the dewater. Table A.5 provides averaged data for the dewatering.

Table A.5. Operating Parameters of Evolution 3 for BDEF Dewatering

TMP	Average Flux (gpm/ft^2/psi)	Average Pressure (psig)	Average Velocity (ft/s)	Duration (h:mm)	Density (g/mL)	Slurry Temperature (°C)
4 psig	0.81	4.1	11.2	0:01	1.27	23.2

A.1.4 Evolution 4

Following the dewatering of Evolution 3, the CUF was loaded with additional AP-107 solution and operated to produce four bottles of clarified permeate for the subsequent ion exchange testing. This evolution involved the following steps: (1) composite approximately 2 L of feed,[1] (2) dewater two bottles (for ion exchange testing), (3) composite another 2 L of feed, and (4) dewater another two bottles. Table A.6 summarizes the masses of samples added to the CUF during this evolution.

Table A.6. Feed Material for CUF Testing

Sample ID	Mass Added to CUF (g)	Estimated Volume (mL)
7AP-17-23	340.2	267.9
7AP-17-24	340	267.7
7AP-17-25	344.2	271.0
7AP-17-26	337.9	266.1
7AP-17-27	341.9	269.2
7AP-17-28	334.6	263.5
7AP-17-29	338.4	266.5
7AP-17-30	331.3	260.9
7AP-17-31	335.4	264.1
7AP-17-32	328	258.3
7AP-17-33	336.6	265.0
7AP-17-34	326.6	257.2
7AP-17-35 (a)	337.6	265.8
7AP-17-36 (a)	331.1	260.7
Total	4703.8	3703.9

(a) Some loss in material in 7AP-17-35 and 36 occurred during transfer, estimated ~20 mL.

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[1] All additions and removals of AP-107 to and from the CUF were tracked by mass.

Dewatering for Evolution 4 was done at varying TMPs while maintaining a constant flow rate of 11 ft/sec. Table A.7 provides averaged data for the dewatering.

Table A.7. Operating Parameters of Evolution 4 for Mott Grade 0.1 Filter Testing

Average Flux (gpm/ft^2/psi)	Average Pressure (psig)	Average Velocity (ft/s)	Duration (h:mm)	Density (g/mL)	Slurry Temperature (°C)
0.771	3.7	11.2	0:02	1.270	22.6
0.559	4.9	11.4	0:02	1.271	22.1
0.286	6.6	10.5	0:04	1.273	22.9
0.156	9.9	11.3	0:06	1.274	22.2

A.1.5 Evolution 5

Following the dewatering of Evolution 4, the CUF was loaded with an additional 2 L of AP-107 solution and operated with the recirculation of the permeate through the 0.1 media grade filter. Table A.8 summarizes the masses of samples added to the CUF at the start of this evolution.

Table A.8. Feed Material for Initial 0.1 Media Grade Filter CUF Testing

Sample ID	Mass Added to CUF (g)	Estimated Volume (mL)
7AP-17-37	336.7	265.1
7AP-17-38	339.7	267.5
7AP-17-39	334.6	263.5
7AP-17-40	341.3	268.7
7AP-17-41	336.9	265.3
7AP-17-42	332	261.4
7AP-17-43	333.8	262.8
Total	2355.0	1854.3

The CUF was then operated with recirculation of the permeate for the four test conditions outlined in Table A.9. Prior to running the system with Condition 1, a backpulse was performed. A backpulse was also performed at the conclusion of each condition..

Table A.9. Feed Material for 0.1 Media grade Filter CUF Testing

Test TMP	Average Flux (gpm/ft^2/psi)	Average Pressure (psig)	Average Velocity (ft/s)	Duration (h:mm)	Density (g/mL)	Slurry Temperature (°C)
11 psig	0.004	11.0	14.1	7:00	1.274	23.7
15.5 psig	0.003	16.2	14.6	7:04	1.276	24.9

20 psig	0.002	20.1	14.9	7:00	1.277	25.1
10.4 psig	0.003	10.8	14.0	7:00	1.277	23.5

A.1.6 Evolution 6

Dewatering for Evolution 6 was done through the 5 media grade BDEF. The TMP was not held constant and ranged between 4 and 8 psi during dewatering. Dewatering produced two bottles of filtered permeate for subsequent ion exchange testing. Table A.10 provides averaged data for the dewatering.

Table A.10. Operating Parameter of Evolution 6 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)
0.397	5.5	10.9	0:03	1.275	23.5
0.261	6.2	10.5	0:05	1.276	22.0

A.1.7 Evolution 7

Following the dewatering of Evolution 6, the CUF was loaded with the remaining ~1 L of AP-107 feed and operated to produce one bottle of clarified permeate. Table A.11 summarizes the masses of samples added to the CUF during this evolution.

Table A.11. Feed Material for CUF Testing

Sample ID	Mass Added to CUF (g)	Estimated Volume (mL)
7AP-17-44	344.3	271.1
7AP-17-45	331.6	261.1
7AP-17-46	321.2	252.9
Total	997.1	785.1

Dewatering for Evolution 7 was done through the 5 media grade BDEF and started at a TMP of 3 psi. Table A.12 provides averaged data for the dewatering.

Table A.12. Operating Parameters of Evolution 7 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)
0.756	3.2	11.5	0:03	1.275	23.2

A.1.8 Evolution 8

For Evolution 8, the BDEF system was backpulsed, collecting the solids and feed in a bottle for particle size distribution analysis. The CUF permeate slurry was drained into two bottles to be used for dead-end filter (DEF) testing. The system was then backpulsed and rinsed with 2 L of 0.01 M NaOH.

At the conclusion of testing, there were 11 bottles of material collected from the CUF, 8 bottles of permeate, 1 bottle of unfiltered AP-107 backpulsed from the BDEF, and 2 bottles of unfiltered AP-107 drained from the CUF for DEF testing, for a total of ~9.3 L of material (see Table A.13).

Table A.13. CUF Products for Ion Exchange/DEF Testing

Sample ID	Mass Removed (g)	Estimated Volume (mL)	Sample Type
AP7-IX-E3-1	971.3	765.4	Permeate
AP7-IX-E4-1	1216.4	957.8	Permeate
AP7-IX-E4-2	1077	847.4	Permeate
AP7-IX-E4-3	1123.1	880.9	Permeate
AP7-IX-E4-4	1191	934.1	Permeate
AP7-IX-E6-1	1102.6	864.8	Permeate
AP7-IX-E6-2	1335	1046.2	Permeate
AP7-IX-E7-1	1313.8	1034.5	Permeate
TI-031-E8-S2	177.26	139.6	Sample
DEF-E8-1	1198.2	943.5	Feed
DEF-E8-2	1179	928.3	Feed
Total	11884.7	9342.5	

A.1.9 Evolution 9

Evolution 9 consisted of the pre- and post-cleaning CWF measurements and filter cleaning. The CUF was rinsed with 2 L of inhibited water (0.01 M NaOH) and set to "Recycle Mode" through the 5 media grade filter. CWF measurements were taken for the CUF 5 media grade, 0.1 media grade, and the BDEF prior to filter cleaning to assess the filter cleaning method. The CWF measurement provides a comparison measurement to the initial CWF and final CWF after cleaning in order to compare filter resistance and efficacy of the cleaning method. Pre-cleaning CWF was conducted at conditions specified in Table A.14 for the CUF 5 media grade, Table A.15 for the CUF 0.1 media grade, and Table A.16 for the BDEF.

Table A.14. CUF Pre-Cleaning Clean Water Flux Measurements for 5 Media Grade Filter

Test TMP (psig)	Average Flux (gpm/ft^2/psi)	Average Pressure (psig)	Average Velocity (ft/s)	Duration (h:mm)	Density (g/mL)	Slurry Temperature (°C)
1	1.8	0.9	11.2	0:17	1.00	25.2

Table A.15. CUF Pre-Cleaning Clean Water Flux Measurements for 0.1 Media Grade Filter

Test TMP (psig)	Average Flux (gpm/ft^2/psi)	Average Pressure (psig)	Average Velocity (ft/s)	Duration (h:mm)	Density (g/mL)	Slurry Temperature (°C)
5	0.040	5.07	11.34	0:16	1.01	25.0
10	0.033	9.95	11.04	0:10	1.01	25.3
15	0.021	16.51	11.45	0:08	1.01	25.5
5	0.013	4.41	10.48	0:08	1.00	24.2

Table A.16. CUF Pre-Cleaning Clean Water Flux Measurements for BDEF

Test TMP (psig)	Average Flux (gpm/ft^2/psi)	Average Pressure (psig)	Average Velocity (ft/s)	Duration (h:mm)	Density (g/mL)	Slurry Temperature (°C)
1.5	2.3	1.5	10.5	0:08	1.01	25.3

After completion of the pre-cleaning CWF, the CUF was drained and filter cleaning was conducted. 1.5 L of 2 M HNO_3 was added to the slurry reservoir and backpulsed two times each through the 5 media grade, 0.1 media grade, and BDEFs. The cleaning was done to return the filters and system to pre-operation (clean) levels. Following the backpulses, the system was drained and rinsed again with an additional ~1.5 L of inhibited water and backpulsed through each filter to conclude the filter cleaning.

Final CWF measurements were taken through each filter following filter cleaning. The inhibited water was drained and 2 L of new inhibited water was added and set to “Recycle Mode” through the 5 media grade filter. Post-cleaning CWF was conducted at conditions specified in Table A.17 for the CUF 5 media grade, Table A.18 for the CUF 0.1 media grade, and Table A.19 for the BDEF.

Table A.17. CUF Post-Cleaning Clean Water Flux Measurements for 5 Media Grade Filter

Test TMP (psig)	Average Flux (gpm/ft^2/psi)	Average Pressure (psig)	Average Velocity (ft/s)	Duration (h:mm)	Density (g/mL)	Slurry Temperature (°C)
0.9	1.79	0.9	11.1	0:12	1.00	24.3

Table A.18. CUF Post-Cleaning Clean Water Flux Measurements for 0.1 Media Grade Filter

Test TMP (psig)	Average Flux (gpm/ft^2/psi)	Average Pressure (psig)	Average Velocity (ft/s)	Duration (h:mm)	Density (g/mL)	Slurry Temperature (°C)
5	0.019	4.9	11.1	0:06	1.00	24.9
10	0.014	10.4	11.0	0:03	1.00	25.3
15	0.005	15.3	10.0	0:08	1.00	25.1
5	0.000	5.8	11.6	0:06	1.01	24.4
5 repeat	0.023	4.6	11.5	0:05	1.00	24.8

Table A.19. CUF Post-Cleaning Clean Water Flux Measurements for BDEF

Test TMP (psig)	Average Flux (gpm/ft^2/psi)	Average Pressure (psig)	Average Velocity (ft/s)	Duration (h:mm)	Density (g/mL)	Slurry Temperature (°C)
1.3	2.52	1.43	11.2	0:12	1.00	25.1

Appendix B

Analytical Reports

Analytical reports provided by the Analytical Support Operations (ASO) are included in this appendix. In addition to the analyte results, they define the procedures used for chemical separations and analysis, as well as quality control sample results, observations during analysis, and overall estimated uncertainties. The analyses are grouped according to Analytical Services Request (ASR) number.

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Battelle PNNL/RPL/Inorganic Analysis ... ICP-OES Analysis Report
PO Box 999, Richland, Washington 99352

Project / WP#:	69832 / N79882
ASR#:	0395.01
Client:	S. Fiskum
Total Samples:	2 (liquids)

ASO Sample ID	Client Sample ID	Client Sample Description	Sample Weight (g)
18-0117	7AP-17-11	AP-107 Tank Supernate	NA
18-0118	7AP-17-46	AP-107 Tank Supernate	NA
DUP-0118	7AP-17-46	AP-107 Tank Supernate	NA

Sample Preparation: RPG-CMC-128, Rev. 1. "HNO3-HCl Acid Extraction of Liquids for Metals Analysis Using a Dry-Block Heater", performed by L. Darnell on Samples 18-0117, 18-0118, and DUP-0118 on 11/28/17. Simple dilution of "as received" samples in 5% v/v HNO_3 performed by J. Carter on 12/07/17.

Procedure: RPG-CMC-211, Rev. 4, "Determination of Elemental Composition by Inductively Coupled Argon Plasma Optical Emission Spectrometry (ICP-OES)."

Analyst:	J. Carter	**Analysis Date:**	12/07/2017	**ICP File:**	C0738

See Chemical Measurement Center 98620 file: ICP-325-405-3
(Calibration and Maintenance Records)

M&TE:		
	☒ PerkinElmer 5300DV ICP-OES	SN: 077N5122002
	☒ Mettler AT400 Balance	SN: 1113292667
	☒ Sartorius R200D Balance	SN: 39080042
	☒ SAL Cell 2 Balance	SN: 8033311209
	☒ Lab 201 Denver A-160 Balance	SN: 60568

Garrett N. Brown
Report Preparer

1/26/18
Date

Review and Concurrence

1/26/18
Date

Two aqueous samples submitted under Analytical Service Request (ASR) 0395.01 were analyzed by ICP-OES. Samples 18-0117 and 18-0118 were prepared following RPL procedure RPG-CMC-128 and diluted to approximately 25 mL. Sample 18-0118 was prepared in duplicate following RPL procedure RPG-CMC-128 and diluted to approximately 25 mL. All samples were further diluted in 5% HNO_3 prior to analysis. None of the samples were filtered.

All sample results are reported on a mass per unit volume basis (μg/mL) for each detected analyte. The data have been adjusted for instrument dilutions.

Analytes of interest (AOI) were specified in the ASR and are listed in the upper section of the attached ICP-OES Data Report. The quality control (QC) results for the AOI have been evaluated and are presented below. Analytes other than the AOI are reported in the bottom section of the report but have not been fully evaluated for QC performance.

Calibration of the ICP-OES was done following the manufacturer's recommended calibration procedure using multi-analyte custom standard solutions traceable to the National Institute of Standards and Technology (NIST). Midrange calibration verification standards (MCVA and MCVB) were used to verify acceptance of the two-point calibration curves obtained for each analyte and for continuing calibration verification.

The controlling documents were procedures RPG-CMC-211, Rev. 4, *Determination of Elemental Composition by Inductively Coupled Argon Plasma Optical Emission Spectrometry (ICP-OES)*, and ASO-QAP-001, Rev. 11, *Analytical Support Operations (ASO) Quality Assurance Plan*. Instrument calibrations, QC checks and blanks (e.g., ICV/ICB, CCV/CCB, LLS, ICS), matrix spike, post-digestion spikes, duplicate, reagent spike, blank spike, and serial dilution were conducted during the analysis run.

Preparation Blank (PB):

A preparation blank (reagents only) was prepared for the extraction process. The concentration of all AOI were within the acceptance criteria of <EQL (estimated quantitation level), ≤50% regulatory decision level, or ≤10% of the concentration in the samples.

Reagent Spike (RS)/Laboratory Control Sample (LCS):

A reagent spike (RS) sample (reagents and spikes) was prepared for the extraction process. Recovery values are listed for all analytes included in the RS that were measured at or above the EQL. Recovery values for the AOI meeting this requirement ranged from 103% to 107%, and were within the acceptance criterion of 80% to 120%.

Duplicate/Replicate Relative Percent Difference (RPD)/Relative Standard Deviation (RSD):

A duplicate of sample 18-0118 was prepared and analyzed. RPDs are listed for all analytes that were measured at or above the EQL. RPDs for the AOI meeting this requirement ranged from 1.0% to 7.2% and were within the acceptance criterion of ≤20% for liquid samples.

Matrix-Spike (MS) Sample:

A matrix spike (MS) of sample 18-0001 was prepared for the extraction process. Recovery values are listed for all analytes included in the MS that were measured at or above the EQL. Recovery values for the AOI meeting this requirement ranged from 106% to 116%, and were within the acceptance criterion of 75% to 125%.

Initial/Continuing Calibration Verification (ICV/CCV):

MCVA and MCVB solutions were analyzed immediately after calibration, after each group of not more than ten samples, and at the end of the analytical run. The concentrations of all AOI were within the acceptance criteria of 90% to 110% recovery, with the exception of potassium (110.1%) in the final CCV solution.

Initial/Continuing Calibration Blank (ICB/CCB):

The ICB/CCB solution (5% v/v HNO_3) was analyzed immediately after the ICV solutions and after the CCV solutions (after each group of not more than ten samples and at the end of the analytical run). The concentration of all AOI were within the acceptance criteria of <EQL, with the exception of sodium in the final three CCB solutions.

Low-Level Standard (LLS):

The LLS solution was analyzed immediately after the first CCB solution. The concentrations of all AOI were within the acceptance criteria of 70% to 130% recovery.

Interference Check Standard (ICS/SST):

The ICS solution was analyzed immediately after the first LLS solution and immediately prior to analyzing the final CCV solutions. The concentrations of all AOI were within the acceptance criteria of 80% to 120% recovery.

Serial Dilution (SD):

Five-fold serial dilution was conducted on sample 18-0018. Percent differences (%Ds) are listed for all analytes that had a concentration at or above the EQL in the diluted sample. The %Ds for the AOI meeting this requirement ranged from 3.5% to 6.9% and were within the acceptance criterion of $\leq$10%.

Post-Digestion Spike (PS-A)/Analytical Spike (AS-A) - Sample (A Component):

In addition to the BS sample, a post-digestion spike (A Component) was conducted on sample 18-0017. Recovery values are listed for all analytes in the spike that were measured at or above the EQL, and that had a spike concentration $\geq$25% of that in the sample. Recovery values for the AOI meeting this requirement ranged from 97% to 101%, and were within the acceptance criterion of 80% to 120%.

Post-Digestion Spike (PS-B)/Analytical Spike (AS-B) - Sample (B Component):

In addition to the MS sample, a post-digestion spike (B Component) was conducted on sample 18-0117. Recovery values are listed for all analytes in the spike that were measured at or above the EQL, and that had a spike concentration ≥25% of that in the sample. There were no AOI included in the spike B Component.

Other QC:

All other instrument-related QC tests for the AOI passed within their respective acceptance criteria.

Comments:

1) The "Final Results" have been corrected for all laboratory dilutions performed on the samples during processing and analysis, unless specifically noted.
2) Instrument detection limits (IDL) and estimated quantitation limits (EQL) shown are for acidified water and/or fusion flux matrices as applicable. Method detection limits (MDL) for individual samples can be estimated by multiplying the IDL by the "Process Factor" for that individual sample. The estimated quantitation limit (EQL) for each concentration value can be obtained by multiplying the EQL by the "Process Factor".
3) Routine precision and bias is typically ±15% or better for samples in dilute, acidified water (e.g. 5% v/v HNO_3 or less) at analyte concentrations > EQL up to the upper calibration level. This also presumes that the total dissolved solids concentration in the sample is less than 5000 µg/mL (0.5 per cent by weight). Note that bracketed values listed in the data report are within the MDL and the EQL, and have potential uncertainties greater than 15%. Concentration values < MDL are listed as "- -". Note, that calibration and QC standard samples are validated to a precision of ±10%.
4) Analytes included in the spike A component (for the AS/PS) are; Ag, Al, As, B, Ba, Be, Bi, Ca, Cd, Co, Cr, Cu, Fe, K, Li, Mg, Mn, Mo, Na, Ni, P, Pb, Sb, Se, Si, Sn, Sr, Ta, Ti, Tl, V, W, Y, Zn, and Zr. Analytes included in the spike B component are; Ce, Dy, Eu, La, Nd, Pd, Rh, Ru, S, Te, Th, and U.

Battelle PNNL/RPG/Inorganic Analysis ... ICPOES Data Report

		Run Date >	12/7/2017	12/7/2017	12/7/2017	12/7/2017	12/7/2017	12/7/2017
		Process Factor >	1.0	48.9	242.6	242.6	244.5	243.3
			405 diluent	BLK-0001	18-0117 @ 5x	18-0117 @ 5x rep	18-0118 @ 5x	Dup-0018 @5x
Instr. Det. Limit (IDL)	Est. Quant. Limit (EQL)	Client ID >	Lab Diluent	BLK-0001	7AP-17-11		7AP-17-46	
(µg/mL)	(µg/mL)	(Analyte)	(µg/mL)	(µg/mL)	(µg/mL)	(µg/mL)	(µg/mL)	(µg/mL)
0.0038	0.038	Al	--	--	9,850	9,750	9,750	9,950
0.0262	0.262	K	--	[4.6]	3,720	3,710	3,730	4,010
0.0075	0.075	Na	--	[3.3]	129,000	129,000	129,000	130,000
Other Analytes								
0.0014	0.014	Ag	--	--	--	--	--	--
0.0383	0.383	As	--	--	--	--	--	--
0.0032	0.032	B	[0.020]	[0.90]	32.6	30.8	35.1	47.4
0.0001	0.001	Ba	[0.0001]	0.580	1.17	1.16	0.934	0.844
0.0001	0.001	Be	--	--	[0.14]	[0.13]	[0.14]	0.165
0.0220	0.220	Bi	--	--	--	--	--	--
0.0054	0.054	Ca	--	6.86	31.2	30.9	28.3	35.5
0.0016	0.016	Cd	--	--	6.62	6.50	6.85	6.82
0.0052	0.052	Ce	--	--	--	--	--	--
0.0033	0.033	Co	--	--	--	--	--	--
0.0016	0.016	Cr	--	--	496	492	491	494
0.0023	0.023	Cu	--	--	[1.4]	[1.3]	[1.1]	[0.93]
0.0012	0.012	Dy	--	--	--	--	--	--
0.0006	0.006	Eu	--	--	--	--	--	--
0.0033	0.033	Fe	--	[0.26]	16.4	16.0	14.8	14.7
0.0010	0.010	La	--	--	--	--	--	[0.47]
0.0010	0.010	Li	--	[0.084]	[0.65]	[0.73]	[0.98]	[0.77]
0.0014	0.014	Mg	--	[0.23]	--	--	--	[1.6]
0.0003	0.003	Mn	--	--	--	--	--	[0.18]
0.0048	0.048	Mo	--	--	41.7	40.2	41.6	41.0
0.0095	0.095	Nd	--	--	[3.0]	--	--	--
0.0040	0.040	Ni	--	--	22.1	21.8	22.6	22.4
0.0369	0.369	P	--	--	648	638	677	638
0.0144	0.144	Pb	--	--	[11]	[12]	[11]	[10]
0.0082	0.082	Pd	--	--	[2.4]	[3.1]	[4.1]	[3.5]
0.0108	0.108	Rh	--	--	[2.7]	[2.9]	--	--
0.0068	0.068	Ru	--	--	[6.8]	[6.6]	[6.4]	[5.6]
0.0898	0.898	S	--	--	1,720	1,690	1,740	1,640
0.0569	0.569	Sb	--	--	--	--	--	--
0.0876	0.876	Se	--	--	[31]	--	--	--
0.0043	0.043	Si	[0.0076]	3.33	38.3	38.6	42.4	42.0
0.0195	0.195	Sn	--	--	[5.4]	[6.1]	--	[6.3]
0.0001	0.001	Sr	--	[0.020]	[0.092]	[0.086]	[0.090]	[0.099]
0.0109	0.109	Ta	--	--	--	--	--	--
0.0155	0.155	Te	--	--	--	--	--	--
0.0057	0.057	Th	--	--	[1.7]	--	[1.8]	[2.1]
0.0004	0.004	Ti	--	[0.021]	--	--	--	[0.28]
0.0310	0.310	Tl	--	--	--	--	--	--
0.0312	0.312	U	--	--	[14]	[18]	[17]	[20]
0.0016	0.016	V	[0.0039]	[0.23]	[1.2]	[1.0]	[1.2]	[1.3]
0.0187	0.187	W	--	--	67.2	62.8	65.4	66.7
0.0003	0.003	Y	--	--	--	--	--	--
0.0023	0.023	Zn	--	1.30	[1.4]	[0.81]	[1.0]	[0.76]
0.0013	0.013	Zr	--	--	[0.60]	[0.33]	[0.41]	[0.44]

1) "--" indicates the value is < MDL. The method detection limit (MDL) = IDL times the "multiplier" near the top of each column. The estimated sample quantitation limit = EQL (in Column 2) times the "multiplier". Overall error for values ≥ EQL is estimated to be within ±15%.

2) Values in brackets [ ] are ≥ MDL but < EQL, with errors likely to exceed 15%.

QC Performance 12/7/2017

Criteria >	≤ 20%	80%-120%	75%-125%	80%-120%	80%-120%	≤ 10%
QC ID >	18-0018 Dup	LCS/RS	18-0001 MS	18-0117 + AS-A	18-0117 + AS-B	18-0117 5-fold Serial Dil
Analytes	RPD (%)	%Rec	%Rec	%Rec	%Rec	%Diff
Al	2.0	103	106	97		3.5
K	7.2	104	116	101		3.6
Na	1.0	107	nr	nr		6.9
Other Analytes						
Ag				92		
As				102		
B	29.7	104	102	101		41.1
Ba	10.2	101	103	99		2.5
Be		100	101	97		
Bi		80	82	94		
Ca	22.5	108	115	103		
Cd	0.4	100	102	100		
Ce		99	98		96	
Co				99		
Cr	0.6	97	92	95		3.3
Cu		104	107	104		
Dy					96	
Eu					96	
Fe	0.3	101	100	100		
La		99	99		95	
Li		118	119	106		
Mg		103	105	102		
Mn		100	100	100		
Mo	1.3	98	94	97		
Nd		99	97		96	
Ni	0.9	101	101	102		
P	5.8	101	95	100		6.6
Pb		98	99	98		
Pd					91	
Rh					94	
Ru					94	
S	6.1	97	91		96	2.1
Sb				105		
Se				100		
Si	1.1	23	51	100		
Sn				94		
Sr		106	98	101		
Ta				98		
Te					98	
Th					95	
Ti		103	104	100		
Tl				91		
U		102	100		100	
V		100	98	97		
W	2.0	97	94	99		
Y				96		
Zn		97	98	99		
Zr		107	107	101		

Shaded results are outside the acceptance criteria.

nr = spike concentration less than 25% of sample concentration. Matrix effects can be assessed from the serial dilution.

Battelle PNNL/RPL/ASO Radiochemistry Analysis Report
P.O. Box 999, 902 Battelle Blvd., Richland, Washington 99352

Gamma Energy Analysis (GEA)

Project / WP#:	71274/ N96053
ASR#:	0500.00
Client:	J. Geeting
Total Samples:	1

RPL ID	Client Sample ID
18-1278	TI-032-1

Analysis Type:	GEA- for all positively measured or non-detected isotopes
Sample Processing Prior to Radiochemical Processing/Analysis	☐ None ☒ Digested as per RPG-CMC-129, Rev. 0 *HNO_3-HCl Acid Extraction of Solids Using a Dry Block Heater* ☐ Fusion as per RPG-CMC-115, *Solubilization of Metals from Solids Using a KOH-KNO_3 Fusion* ☐ Other: Preparation may also involve attaining a GEA geometry that is compatible with the calibration geometry.
Analysis Procedure:	RPG-CMC-450, Rev. 3, *Gamma Energy Analysis (GEA) and Low-Energy Photon Spectrometry (LEPS)*
Reference Date:	None
Analysis Date or Date Range:	May 14-18, 2018
Technician/Analyst:	T Trang-Le
Rad Chem Electronic Data File:	*18-1278 Geeting.xls*
ASO Project 98620 File:	*File Plan 5872, T4.4 Technical (Radiochemistry), Gamma Calibration, daily checks, and maintenance records; and T3 standard certificates and preparation. Also, balance calibration and performance check records.*
M&TE Number(s):	*Detectors E,G,N*

T Trang-le / 5/24/18
Prepare **Date**

LRGreenwood / 5-24-18
Reviewer **Date**

SAMPLE RESULTS

Activities for all gamma emitters detected in this sample are presented in an attached Excel spreadsheet for ASR 0500.00. All sample results for all target isotopes are reported in units of Bg/g with estimates of the total propagated uncertainty reported at the 1-sigma level. The client requested gamma activity measurements for Co-60, Cs-134, Cs-137, Eu Isotopes, Am-241 and Pu-239. Eu-154 was detected; however, Eu-152 and Eu-155 were not detected. Am-241 was seen weakly in the sample but could not be detected in the duplicate. Detection limits are listed for activities that could not be detected. Am-243 was tentatively detected in the samples. The peak at 74.7 keV cannot be resolved from the Pb x-ray peak at 75 keV. The high activity of Cs-137 may create x-rays in the lead shielding surrounding the gamma detector leading to the activity which we tentatively reported as Am-243 with low confidence.

The wet solids were initially dried in the SAL hot cell drying oven to constant weight prior to sub sampling for analyses. Two sub samples of the dried solids were obtained (Sample and Duplicate) and dissolved using acid digestion procedure RPG-CMC-129, Rev. 0, *"HNO_3-HCl Acid Extraction of Solids Using a Dry Block Heater"*. Due to the limited quantity of dried solids, a matrix spike sample was not prepared. The dried solids were completely dissolved with no visible residuals. Sub samples of the digestates were aliquoted and removed from the hotcell for further analyses.

ASO Project File, ASR 0500 has been created for this report including all appropriate supporting records which may include the Pipette Performance Check Worksheet form, standard certificates, laboratory bench records, Shielded Analytical Laboratory Bench Sheet, and Gamma Energy Analysis printouts. Detector calibration records, control charts and balance calibration records can be found in the ASO Records.

Sample Preparation, Separation, Mounting and Counting Methods

All samples were prepared by pipetting 2 mL of each sample into a 22 mL glass scintillation vial (calibrated geometry) and sent to the counting room for GEA analysis.

The quality control (QC) steps for direct GEA are discussed below.

QUALITY CONTROL RESULTS

Tracer:

Tracers are not used for ASO GEA methods.

Process Blank (PB):

The acid digestion process blank was analyzed with Cs-137 the only isotope detected in the list of target isotopes. The activity level of Cs-137 present in the blanks is well below 5% of the activity present in the samples with measurable activity meeting the acceptance criteria of less than 5% of the sample activity or less than the sample MDC.

The Cs-137 activity measured in the acid digestion blank is 3.11 E+01 Bq/g, greater than 5 E+05 times lower than the activity present in the samples.

Required Detection Limits

There is no required detection limits for these samples.

Blank Spike (BS)/Laboratory Control Sample (LCS)/ Matrix Spike (MS):

There are no BS, LCS or MS samples analyzed for ASO GEA analyses. Instrument performance is assessed by the analyses of daily control counts and weekly background counts, as discussed below.

Duplicate Relative Percent Difference (RPD):

Duplicate results are required to agree within ≤ 20% RPD. The ASO QAP further specifies that the two results agree within two standard deviations of the mean (Mean Difference). When sample results are above detection but duplicate results exceed 20% RPD, the mean difference evaluation is performed to determine if the results are statistically different at the 95% confidence level. Duplicate results were 17% RPD for Cs-134 and 14% RPD for Cs-137 thus meeting the ≤ 20% requirement. The duplicate results were 25% RPD for Co-60, 22% RPD for Eu-154 and 47% RPD for Am-243. Applying the mean difference calculation to the Co-60, Eu-154 and Am-243, the mean difference values for Co-60 is 0.44, for Eu-154 is 1.49 and for Am-243 is 1.55. All three of these mean difference results are below 1.96 thus the results are not statistically different and the 95% confidence level.

Instrument Calibration and Quality Control

Gamma detectors are calibrated using multi-isotope standards that are NIST-traceable and prepared in the identical counting geometry to all samples and detectors. Counter control sources containing Am-241, Cs-137 and Co-60 are analyzed daily before the use of each detector. Procedure RPG-CMC-450 requires that a counter control source is checked daily and must be within ±3 sigma or ±3% of the control value, whichever is greater. Gamma counting was not performed unless the control counts were within the required limits. Background counts are performed on all gamma detectors at least weekly for either an overnight or weekend count. The most recent background is subtracted from all sample counts.

Assumptions and Limitations of the Data

None

Interferences/Resolution

None.

Uncertainty

For gamma counting, the uncertainty in the counting data, photon abundance and the nuclear half-life, and efficiency are included in the calculation of the total uncertainty along with a systematic uncertainty for sample prep. The Canberra Genie software includes both random and systematic uncertainties in the calculation of the total uncertainties which are listed on the report. We conservatively estimate that 2% is the lowest uncertainty possible for our GEA measurements taking into account systematic uncertainties in gamma calibration standards.

Comments

None

Attachment: Data Report Sample Results for ASR 0500.

Pacific Northwest National Laboratory
Richland, WA
Radiochemical Sciences and Engineering Group

Filename: 18-1278 Geeting
5/24/2018

Client: Geeting
ASR: 0500

Project: 71274
WP: N96053

Prepared by: LRGreenwood 5/24/18

Technical Reviewer: T Trang-le 5/24/18

Procedures: RPG-CMC-450, Rev. 3 Gamma Energy Analysis (GEA) and Low-Energy Photon Spectrometry
M&TE: Gamma detectors E,G,N
Count dates: May 14-18, 2018

Measured Activity, Bq/g ± 1s

RPL ID:	18-1278-PB		18-1278-S		18-1278-D			
Sample ID:	TI-032-1		TI-032-1		TI-032-1			
Isotope							RPD	MD
Co-60	<2.8E+00		**1.32E+02**	**± 14%**	**1.03E+02**	**± 27%**	25%	0.44
Cs-134	<1.9E+00		**4.26E+02**	**± 18%**	**3.58E+02**	**± 20%**	17%	
Cs-137	**3.11E+01**	**± 2%**	**1.46E+07**	**± 2%**	**1.27E+07**	**± 2%**	14%	
Eu-152	<3.9E+00		<1.7E+02		<1.6E+02		-	
Eu-154	<2.7E+00		**2.50E+03**	**± 4%**	**2.00E+03**	**± 7%**	22%	1.49
Eu-155	<3.5E+00		<1.8E+03		<3.2E+03		-	
Am-241	<5.6E+00		**8.40E+03**	**± 28%**	<1.0E+04		-	
Am-243	<1.9E+00		**9.90E+03**	**± 8%**	**1.60E+04**	**± 11%**	47%	1.55
Pu-239	<1.2E+04		<6.0E+06		<1.0E+07		-	

Notes: Am-243 has significant interference from the lead x-ray at 75.0 keV which is unresolved from the main Am-243 gamma peak at 74.7 keV. This is especially a problem in the presence of the much higher activity from Cs-137, which will create x-rays from the lead shielding surrounding the gamma detector. Consequently, the Am-243 activity may be much lower than listed above.

The duplicate samples show acceptable agreement within a relative percent difference (RPD) below 20% for the Cs-134 and Cs-137 activities. In cases where the RPD of duplicate sample results exceeds 20%, the mean difference (MD) calculation can be performed to determine if the duplicate results agree at the 2-sigma level. An MD value below 1.96 indicates that the duplicate results agree at the 95% confidence level. The Co-60, Eu-154, and Am-243 results all pass the MD test.

Pacific Northwest National Laboratory
Richland, WA
Radiochemical Sciences and Engineering Group

filename 18-1278 Geeting
6/4/2018

Client: J. Geeting
ASR: 0500

Project: 71274
WP: N96053

Prepared by: T Trang-Le 6/4/18

Technical Reviewer: S Soderquist 6-4-2018

Procedures: RPG-CMC-129, Rev 0, HNO_3-HCL Acid Extraction of Solids Using a Dry-Block Heater
RPG-CMC-4001, Rev 1, Source Preparation For Gross Alpha and Gross Beta Analysis
RPG-CMC-408 Rev 2, Total Alpha and Total Beta Analysis
RPG-CMC-496, Rev 1, Coprecipitation mounting of actinides for alpha spectrometry
RPG-CMC-422, Rev 2, Solutions Analysis: Alpha spectrometry
M&TE: Ludlum, Alpha AEA
Count dates: May 14-18, 2018

Measured Activity, Bq/ml ± 1s

Sample ID	RPL ID	Gross Alpha	U-234+Np-237	Pu-239+240	Pu-238+Am-241	Cm-243+244	Cm-242
TI-031-E3-P	18-1276	**2.85E+1 ± 26%**	**2.10E+0 ± 11%**	**1.84E+1 ± 4%**	**1.73E+1 ± 4%**	**8.81E-1 ± 4%**	<1.E-1
	18-1276 DUP	--	**2.68E+0 ± 10%**	**1.86E+1 ± 4%**	**1.61E+1 ± 4%**	**8.07E-1 ± 17%**	<1.E-1
	RPD		24%	2%	7%	9%	
TI-031-E8-S1	18-1277	**2.55E+1 ± 28%**	**2.14E+0 ± 11%**	**1.83E+1 ± 4%**	**1.52E+1 ± 4%**	**6.11E-1 ± 21%**	<1.E-1
				Measured Activity, Bq/g ± 1s			
TI-032-1	18-1278-S	**1.36E+4 ± 3%**	**9.07E+1 ± 13%**	**1.68E+3 ± 4%**	**1.17E+4 ± 2%**	**1.47E+3 ± 4%**	**1.15E+2 ± 11%**
	18-1278-D	**1.04E+4 ± 3%**	**4.81E+1 ± 21%**	**1.24E+3 ± 5%**	**8.55E+3 ± 3%**	**1.21E+3 ± 5%**	**7.89E+1 ± 16%**
	RPD	26%	61%	30%	31%	19%	37%
	Matrix spike	115%		96%			
	Reagent spike	87%		98%			
	Lab blank	<2.1E-1	<8.E-4	<1.E-3	<1.E-3	<6.E-4	<7.E-2
	Hot cell blank	<2.1E+1	<1.E-1	2.11E-1 ± 26%	1.19E+0 ± 10%	5.99E-1 ± 13%	<5.E-4

Samples **TI-031-E3-P** and **TI-031-E8-S1** were surveyed out of the hot cell as raw sample and delivered to the analytical lab. The gross alpha results on these two samples have high uncertainty. The sum of the individual alpha emitters is a better estimate of the gross alpha activity. All alpha emitters found in the alpha spectrum appear on this report.

Sample **TI-032-1** was acid-digested for analysis in the hot cell, in duplicate, and the digestates were surveyed out of the cell and delivered to the analytical lab. The duplicates do not agree; the first is consistently higher than the second, well outside analytical uncertainty. The hot cell blank has readily measurable alpha activity, but is negligible compared to the sample. All alpha emitters found in the alpha spectrum appear in this report.

Several pairs of alpha emitters have nearly identical alpha energies, too close to resolve, and we report their sums.

Battelle - Pacific Northwest National Laboratory
Radiochemical Science and Technology
TOC Report – Furnace Method
PO Box 999, Richland, Washington 99352

Project Number:	71274
Charge Code:	N96053
ASR Number:	0500
Client:	J. Geeting
Total Samples:	1 solid

	Sample
RPL Numbers	18-1278
Client IDs	TI-032-1, AP-107 Solids

Analysis Procedure	RPG-CMC-386 Rev. 1, "Carbon Measured in Solids, Sludge, and Liquid Matrices"
Prep Procedure	None
Analyst	C. Rutherford
Analysis Date	June 1, 2018
CCV Standards	Alpha-D-Glucose CMS 510048
BS/LCS/MS Standards	Dextrose Anhydrous CMS 510050
Excel Data File	ASR 0500 Geeting.xlxs
M&TE Numbers	Carbon System (WD36639, RPL/701)
	Balance : Sartorius R200D, S/N 30809774
All Analysis Records	System File TC-18-3

Crystal Rutherford 6/5/18

Prepared By **Date**

Karl N. Pool 6/5/18

Reviewed By **Date**

Table 1: TOC Results for ASR 0500

Results	
TOC in Sample **18-1278** (mg C/kg):	17963
Standard Deviation (mg C/kg):	10963
Relative Standard Deviation (%):	61.0

Sample Analysis/Results Discussion

One solid sample was submitted under Analytical Service Request (ASR) 0500 for organic carbon analysis. The analysis was performed by the furnace method, with the results summarized in Table 1. The furnace method uses high temperature combustion in a flowing oxygen atmosphere to convert organic and inorganic forms of carbon to carbon dioxide. The combustion temperature of 600°C was selected for TOC analysis. The analyses were performed following the QA Plan ASO-QAP-001, Rev. 11 and procedure RPG-CMC-386, Rev. 1.

The one sample was analyzed in duplicate and with an analytical spike. The sample result is corrected for contribution from the system blank, as per procedure RPG-CMC-386, Rev. 1. All data are reported as μg C/g of sample.

Data Limitations

None

Quality Control Discussion

The calibration and QC sample standard for the TOC initial/continuing calibration verification check (ICV/CCV) is Alpha-D glucose. The identification of the standard and its Chemical Management System (CMS) number is included on the raw data bench sheets for traceability.

The QC for the method includes, as applicable, analysis of initial and continuing calibration verification samples (ICV/CCV), initial and continuing calibration blanks (ICB/CCB), laboratory duplicates for each sample, a laboratory control sample/blank spike (LCS/BS), and an analytical spike (AS).

Two blanks run at the beginning of a batch are required to be < EQL. For this run, the method IDL of 55 ug C was calculated from the blanks run within the batch. EQL is normally 5 to 10x the IDL. The two blanks processed at the beginning of the run are 3.59μg C and 3.20μg C, both below the EQL and meet the acceptance criterion. The blank analyzed at the end of the batch run has a value of 32.70, below the EQL.

Calibration Check Verification Standards: The calibration of the coulometer analysis system was checked by calibration verification standards analyzed at the beginning and end of the analysis run. TOC results for the two ICVs were 91 and 99% recovery, within the acceptance criterion of 90% to 110% and for the CCV, was 98% recovery, with the acceptance criterion of 85% to 115%.

Laboratory Control Sample/Blank Spike: A LCS/BS was analyzed with the samples and recovered 108% for TOC, meeting the acceptance criteria of 75% to 125%.

Duplicate: Precision of the carbon measurements is demonstrated by the relative percent difference (RPD) between sample and duplicate/replicate. Unfortunately, the sample and duplicate did not show very good agreement with an RPD of 61%. The acceptance criterion for duplicate results is ≤ 20% RPD when the results are 10x greater than the IDL. The sample and duplicate result variability may be due to the small sample sizes used based on limited sample available. The final reported result is the average of the sample and duplicate results.

Analytical Spike (AS): The analytical spike of the sample was analyzed and recovered 75% for TOC, meeting the acceptance criteria of 75-125%.

Deviation from Procedure

None.

General Comments

1) Routine precision and bias are typically ±15% or better for non-complex samples that are free of interferences.
2) The estimated quantitation limit (EQL) is defined as 5 times the MDL. Results <5xMDL have higher uncertainties, and RPDs (or RSDs, if applicable) are not calculated if the results are <5xMDL.
3) For the TOC, the analysis MDL is based on the standard deviation calculated from the number (n) of system blanks analyzed with the batch of samples, with the standard deviation multiplied by the Student's t values for n-1 degrees of freedom to establish the daily analysis.
4) Where applicable, the reported "Final Results" have been corrected for any dilution performed on the sample prior to analysis.

Battelle PNNL/RPL/Inorganic Analysis ... ICP-OES Analysis Report

PO Box 999, Richland, Washington 99352

Project / WP#:	71274 / N96053
ASR#:	0500
Client:	J. Geeting
Total Samples:	1 (solid)

ASO Sample ID	**Client Sample ID**	**Client Sample Description**	**Sample Weight (g)**
18-1278-S-129	TI-032-1	AP-107 Solids	0.1668
18-1278-D-129	TI-032-1	AP-107 Solids	0.1003

Sample Preparation: RPG-CMC-129, Rev. 0. "HNO3-HCl Acid Extraction of Solids Using a Dry-Block Heater", performed by L. Grow from 05/02/18 to 05/03/18. Simple dilution of "as received" samples in 5% v/v HNO_3 performed by J. Carter on 05/18/18.

Procedure: RPG-CMC-211, Rev. 4, "Determination of Elemental Composition by Inductively Coupled Argon Plasma Optical Emission Spectrometry (ICP-OES)."

Analyst:	J. Carter	**Analysis Date:**	05/18/2018	**ICP File:**	C0756

See Chemical Measurement Center 98620 file: ICP-325-405-3
(Calibration and Maintenance Records)

M&TE:	☒	PerkinElmer 5300DV ICP-OES	SN: 077N5122002
	☐	Sartorius ME414S Balance	SN: 22406373
	☐	Mettler AT400 Balance	SN: 1113292667
	☐	Sartorius R200D Balance	SN: 39080042
	☒	OHaus PA224C Balance	SN: B725287790
	☐	Mettler AT201 Balance	SN: 192720-92
	☒	SAL Cell 2 Balance	SN: 8033311209
	☐	Lab 309 Balance	SN: 10803210

Jennifer Carter
Report Preparer

5.25.18
Date

Garrett N. Brown
Review and Concurrence

5/24/18
Date

One solid sample submitted under Analytical Service Request (ASR) 0500 was analyzed by ICP-OES. Sample 18-1278 was prepared in duplicate following RPL procedure RPG-CMC-129 and diluted to 10.47 and 8.82 mL, respectively. All samples were further diluted in 5% HNO_3 prior to analysis. None of the samples were filtered.

All sample results are reported on a mass per unit mass basis (µg/g) for each detected analyte. The data have been adjusted for instrument dilutions.

Analytes of interest (AOI) were specified in the ASR and are listed in the upper section of the attached ICP-OES Data Report. The quality control (QC) results for the AOI have been evaluated and are presented below. Analytes other than the AOI are reported in the bottom section of the report, but have not been fully evaluated for QC performance.

Calibration of the ICP-OES was done following the manufacturer's recommended calibration procedure using multi-analyte custom standard solutions traceable to the National Institute of Standards and Technology (NIST). Midrange calibration verification standards (MCVA and MCVB) were used to verify acceptance of the two-point calibration curves obtained for each analyte and for continuing calibration verification.

The controlling documents were procedures RPG-CMC-211, Rev 4, *Determination of Elemental Composition by Inductively Coupled Argon Plasma Optical Emission Spectrometry (ICP-OES)*, and ASO-QAP-001, Rev. 11, *Analytical Support Operations (ASO) Quality Assurance Plan*. Instrument calibrations, QC checks and blanks (e.g., ICV/ICB, CCV/CCB, LLS, ICS), post-digestion spikes, duplicate, blank spike, and serial dilution were conducted during the analysis run.

Preparation Blank (PB):

A preparation blank (reagents only) was prepared for the extraction process. The concentration of all AOI were within the acceptance criteria of <EQL (estimated quantitation level), ≤50% regulatory decision level, or ≤10% of the concentration in the samples. In addition to the PB, a diluent blank from the ICP-OES laboratory (5% HNO_3) was analyzed and the concentration of all AOI were within the acceptance criteria of <EQL.

Blank Spike (BS)/Laboratory Control Sample (LCS):

A blank spike (BS) sample was prepared during the sample preparation process. Recovery values are listed for all analytes included in the BS that were measured at or above the EQL. Recovery values for the AOI meeting this requirement ranged from 97% to 105%, and were within the acceptance criterion of 80% to 120%.

Duplicate/Replicate Relative Percent Difference (RPD)/Relative Standard Deviation (RSD):

Duplicate (18-1278-S-129 and 18-1278-D-129) samples were prepared and analyzed. RPD are listed for all analytes that were measured at or above the EQL. RSD for the AOI meeting this requirement ranged from 1.0% to 29.9% and were within the acceptance criterion of ≤35% for solid samples. Instrument replicate analysis on sample 18-1278-S-

129 resulted in RPD ranging from 0.1% to 3.6%, suggesting that most of the variation was due to the sample preparation process.

Matrix-Spike (MS) Sample:

No matrix spike sample was required to be prepared for the samples.

Initial/Continuing Calibration Verification (ICV/CCV):

MCVA and MCVB solutions were analyzed immediately after calibration, after each group of not more than ten samples, and at the end of the analytical run. The concentrations of all AOI were within the acceptance criteria of 90% to 110% recovery.

Initial/Continuing Calibration Blank (ICB/CCB):

The ICB/CCB solution (5% v/v HNO_3) was analyzed immediately after the ICV solutions and after the CCV solutions (after each group of not more than ten samples and at the end of the analytical run). The concentration of all AOI were within the acceptance criteria of <EQL.

Low-Level Standard (LLS):

The LLS solution was analyzed immediately after the first CCB solution. The concentrations of all AOI were within the acceptance criteria of 70% to 130% recovery.

Interference Check Standard (ICS/SST):

The ICS solution was analyzed immediately after the first LLS solution and immediately prior to analyzing the final CCV solutions. Recovery values are listed for all analytes included in the ICS that were measured at or above the EQL. Recovery values for the AOI meeting this requirement were within the acceptance criterion of 80% to 120%. Not all AOI were present in the ICS.

Serial Dilution (SD):

Five-fold serial dilution was conducted on sample 18-1278-S-129. Percent differences (%Ds) are listed for all analytes that had a concentration at or above the EQL in the diluted sample. The %Ds for the AOI meeting this requirement ranged from 2.0% to 6.5% and were within the acceptance criterion of ≤10%.

Post-Digestion Spike (PS-A)/Analytical Spike (AS-A) - Sample (A Component):

In addition to the BS sample, a post-digestion spike (A Component) was conducted on sample 18-1278-S-129. Recovery values are listed for all analytes in the spike that were measured at or above the EQL, and that had a spike concentration ≥25% of that in the sample. Recovery values for the AOI meeting this requirement ranged from 101% to 180%, and, with the exception of sodium (123%) and silicon (180%), were within the acceptance criterion of 80% to 120%.

Post-Digestion Spike (PS-B)/Analytical Spike (AS-B) - Sample (B Component):

In addition to the BS sample, a post-digestion spike (B Component) was conducted on sample 18-1278-S-129. Recovery values are listed for all analytes in the spike that were measured at or above the EQL, and that had a spike concentration ≥25% of that in the sample. The recovery value for the AOI meeting this requirement was 109% (sulfur) and was within the acceptance criterion of 80% to 120%.

Other QC:

All other instrument-related QC tests for the AOI passed within their respective acceptance criteria.

Comments:

1) The "Final Results" have been corrected for all laboratory dilutions performed on the samples during processing and analysis, unless specifically noted.
2) Instrument detection limits (IDL) and estimated quantitation limits (EQL) shown are for acidified water and/or fusion flux matrices as applicable. Method detection limits (MDL) for individual samples can be estimated by multiplying the IDL by the "Process Factor" for that individual sample. The estimated quantitation limit (EQL) for each concentration value can be obtained by multiplying the EQL by the "Process Factor".
3) Routine precision and bias is typically ±15% or better for samples in dilute, acidified water (e.g. 5% v/v HNO_3 or less) at analyte concentrations > EQL up to the upper calibration level. This also presumes that the total dissolved solids concentration in the sample is less than 5000 µg/mL (0.5 per cent by weight). Note that bracketed values listed in the data report are within the MDL and the EQL, and have potential uncertainties greater than 15%. Concentration values < MDL are listed as "- -". Note, that calibration and QC standard samples are validated to a precision of ±10%.
4) Analytes included in the spike A component (for the AS/PS) are; Ag, Al, As, B, Ba, Be, Bi, Ca, Cd, Co, Cr, Cu, Fe, K, Li, Mg, Mn, Mo, Na, Ni, P, Pb, Sb, Se, Si, Sn, Sr, Ta, Ti, Tl, V, W, Y, Zn, and Zr. Analytes included in the spike B component are; Ce, Dy, Eu, La, Nd, Pd, Rh, Ru, S, Te, Th, and U.

Battelle PNNL/RPG/Inorganic Analysis ... ICPOES Data Report

		Run Date >	5/18/2018	5/18/2018	5/18/2018	5/18/2018
		Process Factor >	1.0	89.9	627.7	879.4
			405 diluent	18-1278-129-PB	18-1278-129-S @ 10x	18-1278-129-D @ 10x
Instr. Det. Limit (IDL)	Est. Quant. Limit (EQL)	Client ID >	Lab Diluent	PB	TI-032-1	TI-032-1
(µg/mL)	(µg/mL)	(Analyte)	(µg/mL)	(µg/g)	(µg/g)	(µg/g)
0.0038	0.038	Al	--	[0.53]	30,200	26,900
0.0054	0.054	Ca	--	[1.6]	724	608
0.0016	0.016	Cr	--	--	1,730	1,480
0.0033	0.033	Fe	--	[1.2]	1,100	875
0.0262	0.262	K	--	27.0	10,200	8,810
0.0014	0.014	Mg	--	--	523	395
0.0075	0.075	Na	--	29.8	324,000	327,000
0.0369	0.369	P	[0.047]	--	1,810	1,790
0.0898	0.898	S	--	[28]	3,920	5,300
0.0043	0.043	Si	[0.0061]	[1.6]	2,290	2,130
Other Analytes						
0.0014	0.014	Ag	[0.0015]	[0.21]	[6.4]	[5.8]
0.0383	0.383	As	--	--	--	--
0.0032	0.032	B	[0.012]	[1.9]	244	244
0.0001	0.001	Ba	--	[0.062]	21.6	15.0
0.0001	0.001	Be	--	--	0.816	0.592
0.0220	0.220	Bi	[0.048]	[3.5]	[37]	[54]
0.0016	0.016	Cd	--	--	48.5	45.8
0.0052	0.052	Ce	--	--	[11]	[12]
0.0033	0.033	Co	--	--	[3.6]	[5.3]
0.0023	0.023	Cu	--	--	32.5	26.9
0.0012	0.012	Dy	--	--	--	[1.4]
0.0006	0.006	Eu	--	--	--	[1.2]
0.0010	0.010	La	--	--	9.31	[5.6]
0.0010	0.010	Li	--	--	6.41	[4.9]
0.0003	0.003	Mn	--	--	65.7	48.0
0.0048	0.048	Mo	--	--	118	102
0.0095	0.095	Nd	--	--	[46]	[30]
0.0040	0.040	Ni	--	--	1,230	967
0.0144	0.144	Pb	--	--	114	[89]
0.0082	0.082	Pd	[0.0091]	--	[8.0]	[19]
0.0108	0.108	Rh	--	--	[7.1]	[11]
0.0068	0.068	Ru	--	--	[16]	[20]
0.0569	0.569	Sb	--	--	--	--
0.0876	0.876	Se	--	--	--	--
0.0195	0.195	Sn	--	[3.3]	[24]	[33]
0.0001	0.001	Sr	--	--	4.33	3.41
0.0109	0.109	Ta	--	--	[6.9]	--
0.0155	0.155	Te	--	[1.6]	[11]	[19]
0.0057	0.057	Th	--	--	35.9	[21]
0.0004	0.004	Ti	--	--	8.98	6.46
0.0310	0.310	Tl	--	[4.0]	--	--
0.0312	0.312	U	--	--	[93]	[91]
0.0016	0.016	V	[0.0017]	[0.24]	[6.3]	[7.9]
0.0187	0.187	W	--	--	171	[160]
0.0003	0.003	Y	--	--	[1.4]	[1.4]
0.0023	0.023	Zn	--	6.23	91.1	99.6
0.0013	0.013	Zr	--	[0.12]	9.81	[8.3]

1) "--" indicates the value is < MDL. The method detection limit (MDL) = IDL times the "multiplier" near the top of each column. The estimated sample quantitation limit = EQL (in Column 2) times the "multiplier". Overall error for values ≥ EQL is estimated to be within ±15%.

2) Values in brackets [ ] are ≥ MDL but < EQL, with errors likely to exceed 15%.

QC Performance 5/18/2018

Criteria >	≤ 35%	80%-120%	75%-125%	80%-120%	80%-120%	≤ 10%
QC ID >	18-1278 Dup	LCS/BS	MS (None)	18-1278 + PS-A	18-1278 + AS-B	18-1278 5-fold Serial Dil
Analytes	RPD (%)	%Rec	%Rec	%Rec	%Rec	%Diff
Al	11.6	99		101		2.2
Ca	17.5	101		102		2.0
Cr	15.4	97		104		2.8
Fe	23.0	100		104		1.5
K	14.5	98		109		6.5
Mg	27.9	97		105		2.2
Na	1.0	105		123		2.6
P	1.2	102		107		5.6
S	29.9	101			109	2.6
Si	7.3	99		180		2.0
Other Analytes						
Ag				99		
As				110		
B	0.0	107		106		21.6
Ba	35.7	99		102		11.8
Be	31.9	95		104		
Bi		85		99		
Cd	5.7	99		108		8.8
Ce		99			106	
Co				108		
Cu	19.0	103		107		
Dy					104	
Eu					105	
La		98			104	
Li		112		113		
Mn	31.2	99		104		15.3
Mo	15.0	100		107		
Nd		100			105	
Ni	23.9	100		108		4.2
Pb		100		108		
Pd					102	
Rh					101	
Ru					104	
Sb				112		
Se				107		
Sn				104		
Sr	23.8	102		113		16.4
Ta				110		
Te					109	
Th					106	452.8
Ti	32.5	100		104		
Tl				99		
U		103			110	
V		102		104		
W		100		108		
Y				104		
Zn	8.9	99		109		10.9
Zr		104		105		

Shaded results are outside the acceptance criteria.

nr = spike concentration less than 25% of sample concentration. Matrix effects can be assessed from the serial dilution.

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*All distribution will be made electronically as PDF files.

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