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Crossflow Filtration of LAWPS Simulants

January 2018

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U.S. DEPARTMENT OF
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Prepared for the U.S. Department of Energy
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Richland, Washington 99352

Executive Summary

The Low-Activity Waste Pretreatment System (LAWPS) will employ crossflow filtration (CFF) to separate solids from radioactive wastes. Design verification of CFF unit operations will involve engineering-scale demonstrations of the efficacy of liquid-solid separations with low-activity waste (LAW) simulants. Before execution of integrated engineering-scale verification tests, the LAW simulants developed to support design verification activities are to be evaluated in a laboratory-scale CFF apparatus to demonstrate simulant suitability, to help refine engineering-scale filtration and filter cleaning strategies prior to implementation, and to identify any anomalous filtration behaviors that could impact the conduct-of-operations at the engineering-scale test facility. This report provides the bench-scale test results for the four LAWPS simulants developed to support integrated testing and of the eight fouling simulants selected to evaluate the sustainability of LAWPS filtration and the effectiveness of LAWPS flux recovery technologies.

The bench-scale tests were performed using the Cells Unit Filter skid on site at Pacific Northwest National Laboratory. The skid uses a single 8-ft-long, 0.5-inch-diameter Mott Grade 0.1 sintered stainless steel filter element in conjunction with a slurry reservoir, recirculation loop, heat exchanger, permeate metering system and its associated instrumentation. The system was also equipped with a backpulse system, metered backflow system for delivery of chemicals for implementation of the proposed LAWPS cleaning protocol, and a data acquisition system. During testing, simulants were recirculated at nominal axial velocities of $14.7\,\mathrm{ft\,s^{-1}}$ and permeate was removed at constant temperature and pressure (as determined by the planned test conditions). Filter performance was investigated by observing the rate of change and magnitude of the permeate flux with time, as well as the response of the flux to changes in test conditions (pressure, temperature, or simulant materials) and recovery protocol (backpulsing or cleaning).

Flux data collected in these tests indicated that the LAWPS CFF system should be capable of meeting permeate production requirements over a period of a minimum of 120 hours if the waste materials in the feed resemble the simulants discussed in this report. The data also suggest that operating for more than 120 hours at the design rates of the LAWPS facility is achievable, particularly if certain operational strategies are implemented, i.e., operating at as low a transmembrane pressure as possible and use of periodic backpulsing to recover lost flux. If the CFF system is fouled significantly, the LAWPS cleaning protocol was observed to effectively recover flux and return the system to its initial performance level. Areas recommended for further study based on the information collected to-date include:

- characterizing the variability in filter performance with sodium oxalate solids
- determining the effect of solids concentration on the rate of fouling
- evaluating the effect of mixed (multi-component) solids phases on the rate and extent of fouling
- measuring filtration performance at the LAWPS upper viscosity limit
- determining the impact of waste simulant hold-up on LAWPS cleaning protocol efficacy
- evaluating the potential for precipitation due to acid/waste simulant contact and its impact on CFF performance

Acknowledgements

Development of this report and the test data underlying it were supported by a broad array of Pacific Northwest National Laboratory staff involved in the procurement of test materials and equipment, development and implementation of quality standards for collection and qualification of test data, execution of crossflow tests and physical analysis of samples, and analysis of the resultant test data. The authors would like to acknowledge Rick Shimskey and Justin Billing for helping guide us on and assist us with qualification of the Cells Unit Filter data acquisition system, for their support in the execution of crossflow filtration tests, and for their technical guidance during execution of tests. In addition, the authors thank Rick Shimskey and Matthew Wilburn for their thorough reviews of the final version of this report. The authors would also like to thank Jared Crum and Jared Kroll for their support with mineral phase identification of simulant materials through X-ray diffraction. The authors would like to acknowledge William Dey, who as project quality engineer provided critical guidance on resolution of qualify affecting issues. The authors would like to thank Reid Peterson for providing task-level feedback on the matrix of tests and selected test materials. The authors would also like to acknowledge Chrissy Charron and Veronica Perez for help with capturing project documentation and for their critical guidance on project standards and execution of work. Finally, the authors thank Matthew Landon of Washington River Protection Solutions and David Herman of Savannah River National Laboratory for providing key technical guidance and feedback on the execution of filtration tests and on this report.

Abbreviations and Acronyms

AV	axial velocity
BP	backpulse
CFF	crossflow filtration
CUF	Cells Unit Filter
CWF	clean water flux
DAS	data acquisition system
DFLAW	Direct Feed Low-Activity Waste
DOE	U. S. Department of Energy
HDI	“How Do I. . . ?”
ITF	Integrated Test Facility
IX	ion exchange
LAW	low-activity waste
LAWPS	Low-Activity Waste Pretreatment System
MSS	modified sodium simulant
M&TE	measuring and test equipment
ORP	Office of River Protection
PNNL	Pacific Northwest National Laboratory
PSD	particle size distribution
QA	quality assurance
SEM	scanning electron microscopy
SOW	statement of work
sRF	spherical resorcinol-formaldehyde (resin)
TMP	transmembrane pressure
TS	total solids
UDS	undissolved solids
WRPS	Washington River Protection Solutions
WTP	Hanford Tank Waste Treatment and Immobilization Plant
XRD	X-ray diffraction

Notation

β	temperature coefficient for flux temperature correction (K)
δp_t	transmembrane pressure (kPa or psid)
$\delta p_{t,o}$	nominal (or set-point) transmembrane pressure (kPa or psid)
Δ_m	filter thickness (m or inch)
η	normalized flux (dimensionless)
$\dot{\gamma}$	shear rate ($\mathrm{s^{-1}}$)
λ	temperature coefficient for viscosity temperature correction (K)
ν	normalized volume (dimensionless)
μ	Newtonian viscosity (mPa s)
μ_o	reference viscosity for viscosity temperature correction (mPa s)
μ_w	viscosity of water (mPa s)
ρ	permeate density ($\mathrm{kg\,m^{-3}}$ or $\mathrm{g\,mL^{-1}}$)
τ	shear stress (Pa)
A	filter surface area ($\mathrm{m^2}$ or $\mathrm{ft^2}$)
c	suspension solids volume fraction (dimensionless)
c_g	gel-polarization volume fraction (dimensionless)
D	filter inner diameter (m or inch)
j	uncorrected permeate flux ($\mathrm{m\,s^{-1}}$ or $\mathrm{gpm\,ft^{-2}}$)
j_c	temperature-corrected permeate flux ($\mathrm{m\,s^{-1}}$ or $\mathrm{gpm\,ft^{-2}}$)
j_o	normalizing flux determined from Darcy's law ($\mathrm{m\,s^{-1}}$ or $\mathrm{gpm\,ft^{-2}}$)
$j_{p,c}$	temperature- and transmembrane pressure-corrected permeate flux ($\mathrm{m\,s^{-1}}$ or $\mathrm{gpm\,ft^{-2}}$)
k	gel-polarization coefficient ($\mathrm{m\,s^{-1}}$ or $\mathrm{gpm\,ft^{-2}}$)
L	filter length (m or ft)
m	mass (kg or g)
p_i	filter suspension-side inlet pressure (kPa or psi)
p_o	filter suspension-side outlet pressure (kPa or psi)
p_p	filter permeate-side pressure (kPa or psi)
Q_p	permeate production rate ($\mathrm{m^3\,s^{-1}}$ or gpm)
Q_s	suspension volumetric flow through CUF recirculation loop ($\mathrm{m^3\,s^{-1}}$ or gpm)
$r_{m,o}$	filter unit length resistance ($\mathrm{m^{-2}}$)
T	slurry reservoir temperature (°C or K)
T_o	nominal filtration or property reference temperature (°C or K)
u	suspension superficial axial flow velocity ($\mathrm{m\,s^{-1}}$ or $\mathrm{ft\,s^{-1}}$)
V_F	volume of permeate filtered ($\mathrm{m^3}$ or L)
V_{tank}	feed tank volume ($\mathrm{m^3}$ or L)
x	mass fraction of solids (mass fraction or wt%)

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1.0 Introduction

The primary mission of the U.S. Department of Energy Office of River Protection (DOE-ORP) is to retrieve and process approximately 56 million gallons of radioactive waste from 177 underground tanks located on the Hanford Site in Washington State. The Hanford waste tanks are currently operated and managed by Washington River Protection Solutions, LLC (WRPS). As part of tank farm operations, WRPS supports DOE-ORP's waste retrieval mission. An important element of the DOE-ORP mission is the construction and operation of the Hanford Tank Waste Treatment and Immobilization Plant (WTP). The WTP is tasked with separating the waste into low-activity waste (LAW) and high-level waste fractions and immobilizing these fractions by vitrification. The primary contractor supporting the construction of the WTP is Bechtel National, Inc.

To support early production of immobilized LAW, the Direct Feed Low-Activity Waste (DFLAW) process has been proposed. In the DFLAW process, a nominally solid-free waste suspension that results from an in-tank settle and decant operation is sent to the Low-Activity Waste Pretreatment System (LAWPS) for filtration and cesium removal. The resultant treated waste is delivered to the WTP LAW Vitrification Facility for immobilization. The conceptual design and ongoing technology maturation of the LAWPS facility are being conducted by WRPS, but the DFLAW process also necessitates interfaces between WRPS and Bechtel National, Inc. to deliver the treated LAW from the LAWPS to the WTP for vitrification. Before the feed is transferred to the WTP LAW Vitrification Facility, tank supernatant waste will be pretreated in the LAWPS to meet the WTP LAW waste acceptance criteria. The key process operations for treating the waste include solids separation [by crossflow filtration (CFF)] and cesium removal [by ion exchange (IX)]. Figure 1.1 shows a general schematic of the anticipated process streams and unit operations.

To support LAWPS design selections prior to key project milestones [Critical Decisions[1]] and to improve the technology maturation level of the LAWPS, WRPS has planned both an integrated engineering-scale test facility using prototypic equipment and a full-scale test apparatus for the IX columns. To support these larger-scale facilities, WRPS identified five technical tasks in statements of work (SOWs)[2,3,4] to be performed by Pacific Northwest National Laboratory (PNNL). These tasks are distinct from the larger-scale test facilities and are intended to help achieve the following objectives:

- Provide technical information or data that either
  - (a) support refinements or simplifications of larger-scale test facilities; or
  - (b) provide expected performance of unit operations (guiding larger-scale operation or providing scale-up data).
- Support the safety basis of the planned LAWPS facility, specifically regarding hydrogen management.

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[1] Critical Decisions (CDs) are defined in DOE O 413.3B, Program and Project Management for the Acquisition of Capital Assets, dated 12/20/2016. The relevant CDs being supported as described in the text of this report are CD-2, Approve Performance Baseline, and/or CD-3, Approve Start of Construction/Execution.

[2] Statement of Work, July 29, 2015, Requisition 279909, LAWPS Integrated Support Testing, Rev. 1.

[3] Statement of Work, April 4, 2016, Requisition 279909, LAWPS Integrated Support Testing, Rev. 2.

[4] Statement of Work, August 31, 2016, Requisition 279909, LAWPS Integrated Support Testing, Rev. 3.

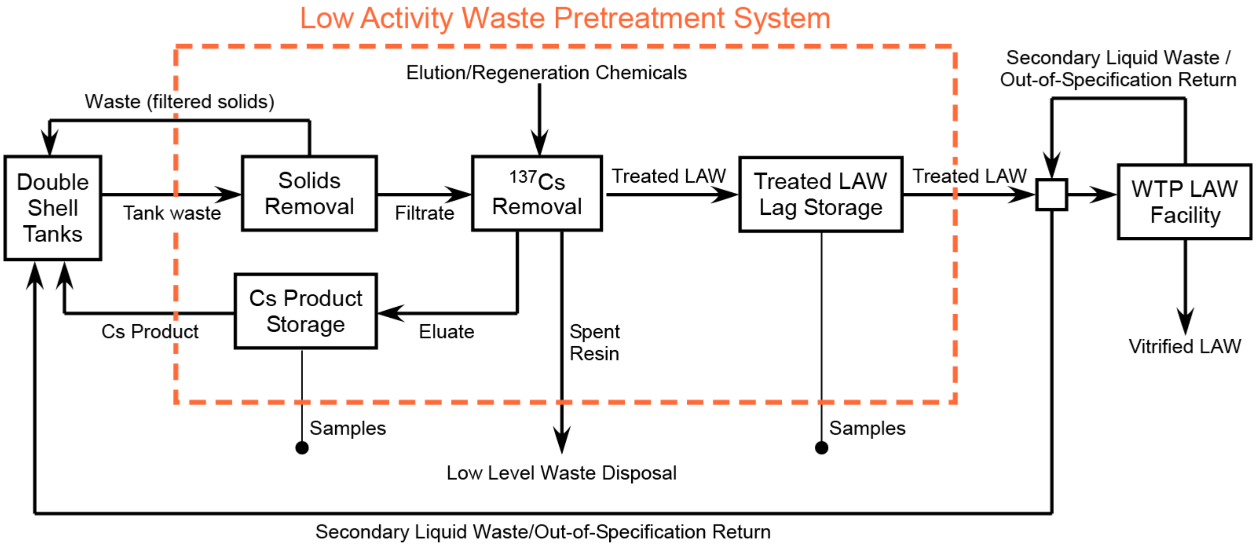

Figure 1.1. General conceptual schematic of the LAWPS facility unit operations and process streams.

The five PNNL technical tasks consist of the following focus areas:

1. Development of LAW waste simulants
2. Bench-scale CFF testing with simulants
3. Gas generation measurements in the presence of spherical resorcinol-formaldehyde (sRF) IX resin
4. Gas retention/release dynamics and fluidization of sRF IX resin
5. General technical support to the larger-scale testing

Tasks 1 and 2 are focused on providing technical information to inform the larger-scale test facilities, whereas Tasks 3 and 4 support the LAWPS facility safety basis. Task 5 supports the larger-scale testing directly. This report is concerned with addressing Task 2, CFF testing, which is described in more detail below.

CFF is a mature industrial technology for the separation of solids from liquids. As applied to treatment of nuclear wastes at the Hanford Site, CFF uses bundles of tubular porous filter elements encased in a permeate collection shell. Solid-liquid separation is effected by pressurizing the tube-side of the filter bundle, creating a transmembrane pressure (TMP) differential that causes liquid to flow from suspension flow through the tubular filters through the porous wall of the tube and into the permeate collection shell. The flux of liquid through the filter element drives the suspended solids to the tube-side surface of the filter element, where they would normally collect into a coherent filter cake that would reduce filter performance in the absence of crossflow. Under sufficient crossflow velocities, turbulent dispersion will re-entrain or sweep away solids collected on the filter surface, leading to sustained rates of permeate production. crossflow does not entirely prevent accumulation of solids on the surface filter and cannot remove solids that migrate into the pores of the filter element. The accumulation of solids on or in the filter leads to a time-dependent reduction in the rate of permeate production. The accumulation is referred to as filter fouling and the solids that build up on the filter surface are said to foul the filter. Evaluation and optimization of CFF operations consider the rate of filter fouling and the strategies for removing solids that have fouled the filter.

CFF will be employed in the LAWPS to remove solids from radioactive wastes and to support IX of the liquid waste fraction. WRPS has requested that PNNL assist in demonstrating the feasibility and sustainability of the CFF operations and to collect data needed to support CFF design verification activities at the engineering-scale Integrated Test Facility (ITF). To this end, PNNL undertook efforts to develop and evaluate the filtration performance of four LAW simulants (or simply the "LAWPS simulants") and eight "fouling" simulants. The applicable revised SOWs[1,2] governing these efforts outlined three CFF-specific testing objectives:

1. evaluate the CFF performance of the LAWPS simulants before the engineering-scale integrated test

2. evaluate various waste representative components and precipitates that will cause fouling

3. evaluate various chemical solutions for cleaning fouled filters, including the standard IX column eluent solution used during LAWPS operations (sRF resin bed elution)

To address these test objectives, three sets of CFF tests were conducted:

- **Series 1 (Permeate Production Rate) Testing** — evaluated the baseline permeate production rates of waste simulants at two temperatures (20 and 45 °C). Select replicate testing was performed to evaluate the reproducibility of baseline production rates between filter cleaning cycles. Filter conditions and production times were selected to be representative of those expected in the LAWPS treatment facility.

- **Series 2 (Filter Fouling) Testing** — reviewed the simulant chemistries and historical filtration data with similar conditions to the planned LAWPS processing available in peer-reviewed journals and technical reports, with the goal of identifying the soluble and insoluble solids present in (or that could be added to) the LAWPS simulants that would be detrimental to sustained permeate production. Associated laboratory-scale tests were performed to directly evaluate the effect of a select subset of "worst-case" solids (precipitated or insoluble) on LAWPS filtration and permeate rate recovery operations.

- **Series 3 (Filter Cleaning) Testing** — evaluated the efficacy of different filter cleaning techniques and permeate production rate recovery strategies applied to crossflow filters fouled during Series 1 and Series 2 testing.

The following sections describe the CFF testing and summarize the results of the CFF series testing listed above.

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[1] Statement of Work, April 4, 2016, Requisition 279909, LAWPS Integrated Support Testing, Rev. 2.

[2] Statement of Work, August 31, 2016, Requisition 279909, LAWPS Integrated Support Testing, Rev. 3.

2.0 Quality Assurance

The work outlined in this report was performed in accordance with PNNL's Quality Management M&O Program Description, which is maintained as part of PNNL's "How Do I. . . ?" (HDI) system for deploying Laboratory-level requirements and procedures to PNNL staff. Details of this project's approach to assuring quality are contained in the LAWPS Testing Program Quality Assurance (QA) Plan (67535-QA-001, Rev. 0) and its associated procedures that implement the requirements of NQA-1-2008, *Quality Assurance Requirements for Nuclear Facility Applications*, and NQA-1a-2009, *Addendum to NQA-1-2008*. The work described in this report was performed at the technology level of Applied Research. The key procedures associated with the LAWPS QA program are as follows:

- All staff members contributing to the work specified in this report received proper technical and QA training before commencing quality-affecting work in accordance with QA-NSLW-0201, Training.

- The studies were conducted in accordance with QA-NSLW-1102, Scientific Investigation for Applied Research.

- The studies were planned and conducted in accordance with QA-NSLW-1104, Test Plans, and QA-NSLW-1107, Test Instructions.

- Test materials and samples were identified and controlled in accordance with QA-NSLW-0801, Item Identification and Sample Control.

- Measuring and test equipment (M&TE) used to generate quality-affecting data were properly procured, controlled, calibrated, handled, and maintained in accordance with QA-NSLW-1201, Calibration and Control of M&TE.

- All data and calculations used in the report were reviewed in accordance with QA-NSLW-1108, Data Entry and Data Review, QA-NSLW-0301, Management of Electronic Data, and QA-NSLW-0304, Calculations.

- Technical reports were generated in accordance with QA-NSLW-1109, Reporting, and were peer reviewed in accordance with QA-NSLW-0601, Document Preparation and Change, and QA-NSLW-0603, Independent Technical Review.

3.0 Test Methodology

To meet the objectives of filter performance and fouling tests discussed in Section 1,

- eight baseline performance CFF tests (denoted as Series 1 testing) were performed using the 4.0 M Na, 5.6 M (nominal) Na, and 8.0 M Na LAWPS simulants, and

- eight "fouling" tests (denoted as Series 2 testing) were run using a combination of soluble and insoluble foulants in a modified version of the 5.6 M (nominal) Na simulant.

These tests evaluate CFF performance under conditions relevant to LAWPS processing of Hanford wastes. Process metrics evaluated include, but are not limited to, permeate production rate (and rate loss caused by fouling of the filter elements) and flux recovery as a result of filter backpulse and chemical cleaning operations. All CFF tests employed filter equipment and test materials (both simulants and cleaning solutions) relevant to the LAWPS facility. This section describes the test approach, equipment, simulants, and cleaning solutions associated with Series 1 and 2 filtration and cleaning tests.

3.1 Definitions

Common terms that will be used to discuss filtration in this report are defined below.

Filtration: Filtration refers to a unit operation in which solids are separated from a liquid suspending phase by the sieving action of a porous membrane or element. In filtration, a suspension is separated into permeate (the "solid-free" or "solid-reduced" liquid) and solid concentrate fractions. Filtration is typically effected by application of a pressure differential across the membrane surface. The sieving efficiency of the membrane is determined by many factors, including the membrane pore size distribution and tortuosity, the suspension particle size distribution (PSD), and the flow field formed within the membrane.

Dead-end Filtration: Dead-end filtration refers to filtration in which the bulk suspension flow is normal to the membrane surface (see Figure 3.1), such that the entire suspension either passes through the filter or is collected on the surface of the filter. In dead-end filtration, all suspension solids (except those small enough to pass through the membrane) will collect on the membrane surface (or possibly within the membrane's pores), forming a filter "cake" that grows indefinitely with time. The filter cake acts as a membrane itself, creating additional sieving of solids and increasing the pressure needed to maintain filtrate production rates.

Crossflow Filtration: Crossflow filtration refers to filtration in which the bulk suspension flow is tangential to the membrane surface (see Figure 3.1). Although solids still accumulate on the filter surface, bulk suspension flow tends to sweep the solids off the filter surface such that cake formation is arrested or completely eliminated. Filter cake mitigation allows CFF operations to sustain much greater long-term permeate production rates relative to dead-end filtration.

Filter or Filter Membrane: A filter or filter membrane is a porous solid, collection of particles, or mesh used for solid-liquid separation by retaining solids from suspensions that flow through the membrane (generally by application of a pressure difference across the membrane).

Axial Velocity: In CFF, axial velocity (AV) refers to the superficial flow velocity of suspension in the flow channel. Common units for AV are $\mathrm{m\,s^{-1}}$ and $\mathrm{ft\,s^{-1}}$.

Transmembrane Pressure: Transmembrane pressure is the pressure difference across the filter membrane that effects filtration. Common units for TMP are kPa and psid (pounds per square inch differential).

Permeate Production Rate: The volumetric flow per unit time of permeate produced by filtration. Common units for permeate production rate are $\mathrm{m^3\,s^{-1}}$ and gpm (gallons per minute).

Filter Flux: Filter flux refers to the permeate production rate per unit area of filter. Common units for filter flux are $\mathrm{m\,s^{-1}}$ and $\mathrm{gpm\,ft^{-2}}$.

Clean Water Flux (CWF): A standard reference measurement of flux using a solid-free 0.01 M NaOH solution to gauge the cleanliness of the filter. CWF measurements are typically done at the start of a filtration test or after chemical cleaning to verify the filter is clean. Use of a standard CWF test solution (0.01 M NaOH) allows comparison of current CWF measurements to historical ones.

Fouling: Fouling generally refers to a reduction in the rate of permeate production that results from accumulation of solids on the surface of the filter (i.e., build-up of a filter cake) or blockage (either partial or complete) of the filter pores or mesh by solids particles.

Backpulse (or Backflush): As applied to the study of Hanford waste filtration, a backpulse (BP) is a permeate production rate recovery operation in which permeate is flushed through the filter in the direction opposite of normal flow. Backflow of liquid disrupts the filter cake (should one exist) and dislodges particles trapped in the filter pores or mesh. Backflow is effected by reversing the pressure differential across the filter. Usage of the term "backpulse" for Hanford operations differs from that in the peer-reviewed literature. In the latter, backpulsing refers to application of a high-frequency oscillatory pressure across the filter to disrupt the filter cake. To describe a prolonged reversal of flow, the peer-reviewed literature typically uses the term "backflush".

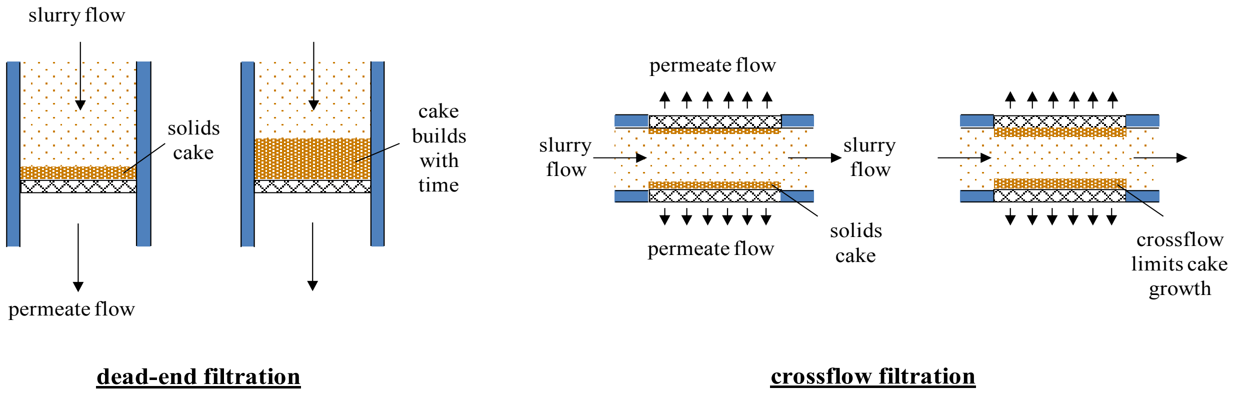

Figure 3.1. An illustration of dead-end and crossflow filtration.

3.2 Test Equipment

All filter and filter cleaning tests employed PNNL's Cells Unit Filter (CUF). The CUF is a bench-top filter system that allows up to 25 L of a simulant waste solution to be circulated through a tubular filter. A tube-in-shell heat exchanger, installed on the main slurry circulation loop, cools or heats the feed solution during filtration operations. The CUF is equipped with sensors and metering devices that can measure filter feed flow rates, filtrate flow rates, system pressures, and temperatures simultaneously. Readings from the sensors are recorded at 0.4 Hz (and subsequently time-averaged to a 1-minute reporting interval) by a LabVIEW data acquisition system (DAS). The individual CUF system components are discussed in greater detail in the sections that follow.

3.2.1 Crossflow Filter and Filter Assembly

The crossflow filter element used in testing was supplied by the Mott Corporation (Farmington, CT) and is nearly equivalent to the filters planned for use in the LAWPS. Specifically, the CUF filter is an 8-foot-long, 0.5-inch-inner-diameter Mott Grade[1] 0.1, 316L sintered stainless steel symmetric element. All CUF filter element specifications are equivalent to those used in LAWPS save for filter length — LAWPS will use 10-foot-long filter elements. Figure 3.2 shows a scanning electron microscopy (SEM) image of a typical filter element cross-section and illustrates both the pore size and tortuosity of the filter media, which allow it to retain submicron particles with high efficiency. Analysis of manufacturer[2] and literature data (Rubow and Jha 1999), including isopropanol bubble point and filter flux data and SEM images, indicates that the Mott Grade 0.1 sintered stainless steel filter elements have an effective hydrodynamic pore size in the range of 1 to 3 µm.

Use of the Mott filter elements in testing requires modification and installation of the element into a larger filter assembly. To create the filter assembly, four 2-ft-long Mott filter elements are welded together to form a semicontinuous 8-foot-long porous filter element. Next, two non-porous 0.5-inch spacers are welded to the ends of the combined 8-foot-long Mott element. The porous section of the resulting tube is enclosed in a shell of sufficient diameter to create an annular spacing of 1/8 inch between tube and shell. This permeate collection shell has two outlets (created by punching holes into the shell and welding 3/8-inch-diameter stainless steel tube pieces with Swagelok fittings in place of those holes). One outlet is placed in the center of the assembly to collect filtrate and pass it to flow metering and filtrate collection devices. Another output is placed near the inlet of the filter to function as a shell-side drain. Pressure ports (1/4-inch stainless steel tubing) are then installed on the non-porous inlet and outlet spacers to measure the filter inlet and outlet pressures. Finally, O-ring face seal (Swagelok VCO) fittings purchased from the Swagelok Company (Solon, OH) are placed on the inlet and outlet filter feed tube connections to facilitate installation of the filter assembly in the slurry circulation loop. Figure 3.3 shows a diagram of the filter assembly; Figure 3.4 shows an image of the final filter assembly. For CFF testing, a single filter element assembly (ID: Filter 8-ft, 85598-000, 7610982-001, LAWPS-03-2016) was used for the entire series of tests described in this report.

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[1] The Mott media grade refers to the nominal particle diameter (in micrometers) corresponding to approximately 90% particle removal efficiency in liquid. While many publications include units of "µm" on the media grade, the Mott Corporation does not appear to do this in their own product literature. It should be noted that the media grade does not reflect the average pore size of the filter medium.

[2] Owsiany M. 2007. "Mott HyPulse LSX Data Summary — 24590-MOTT-FDP02." Mott Corporation, Farmington, Connecticut.

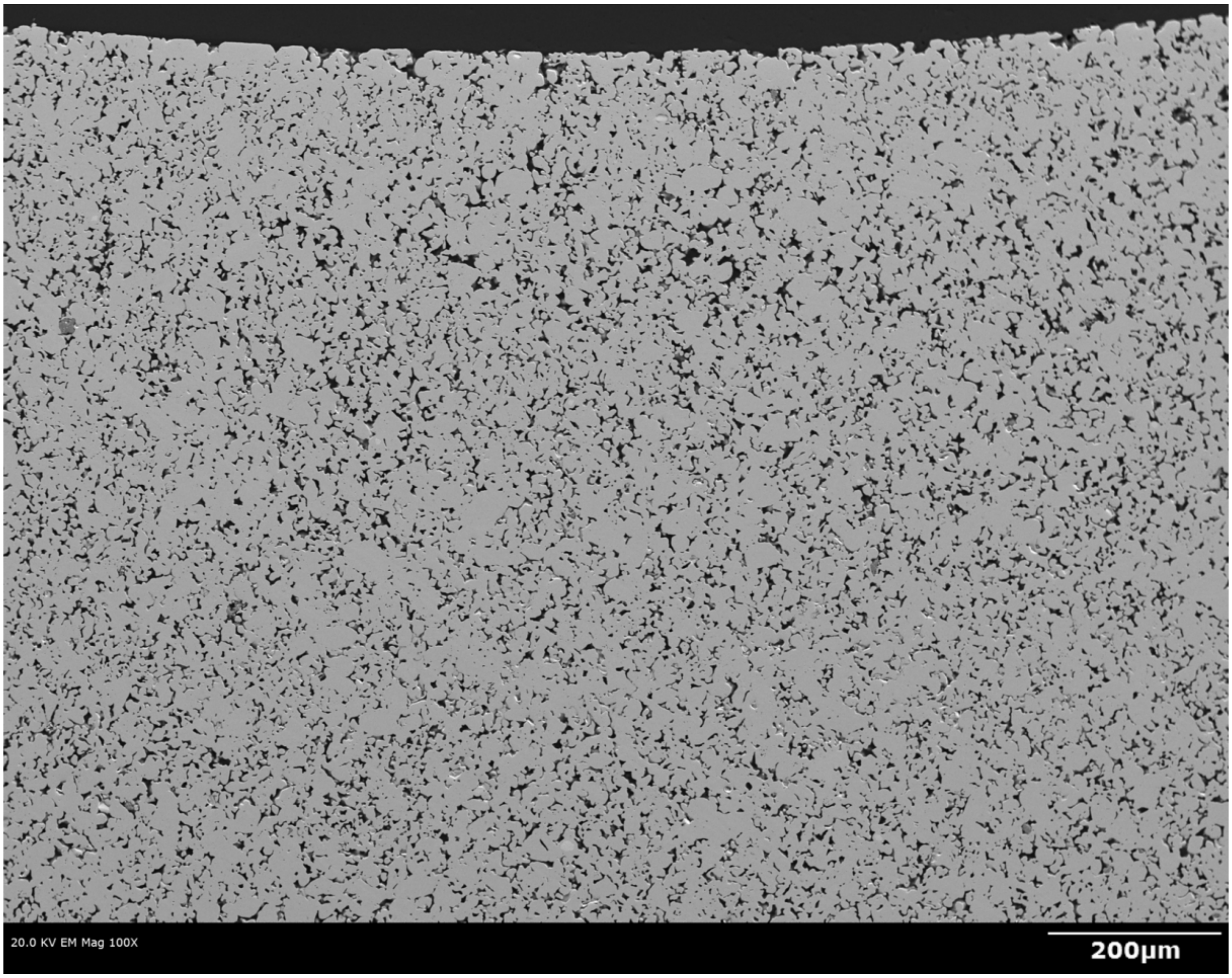

Figure 3.2. SEM cross-section image of a Mott Grade 0.1 sintered stainless steel filter element.

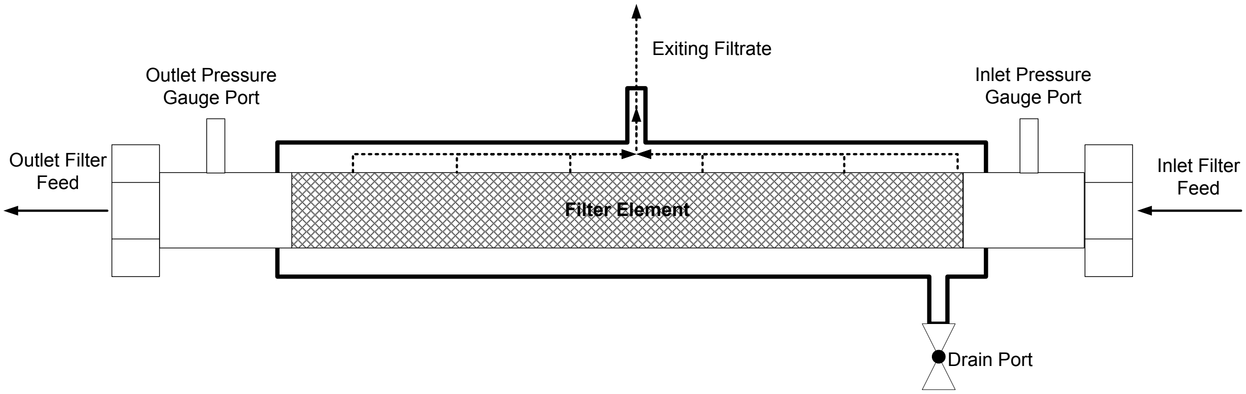

Figure 3.3. Sketch of the filter assembly (not to scale).

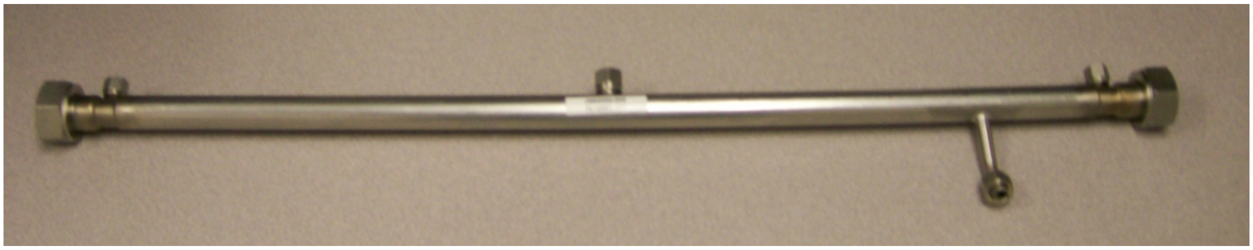

Figure 3.4. Photograph of a 2-foot filter assembly.

3.2.2 Crossflow Filtration Testing System

The filter assembly described in Section 3.2.1 is installed in a larger system capable of 1) delivering a metered, pressurized flow of test suspension to the filter assembly, 2) collecting and metering permeate flow, and 3) delivering metered backflow of cleaning solutions to the filter assembly during cleaning operations. The overall system is shown in Figure 3.5 (simplified process and instrumentation diagram) and in Figure 3.6 (system photograph with an 8-foot-long filter element).

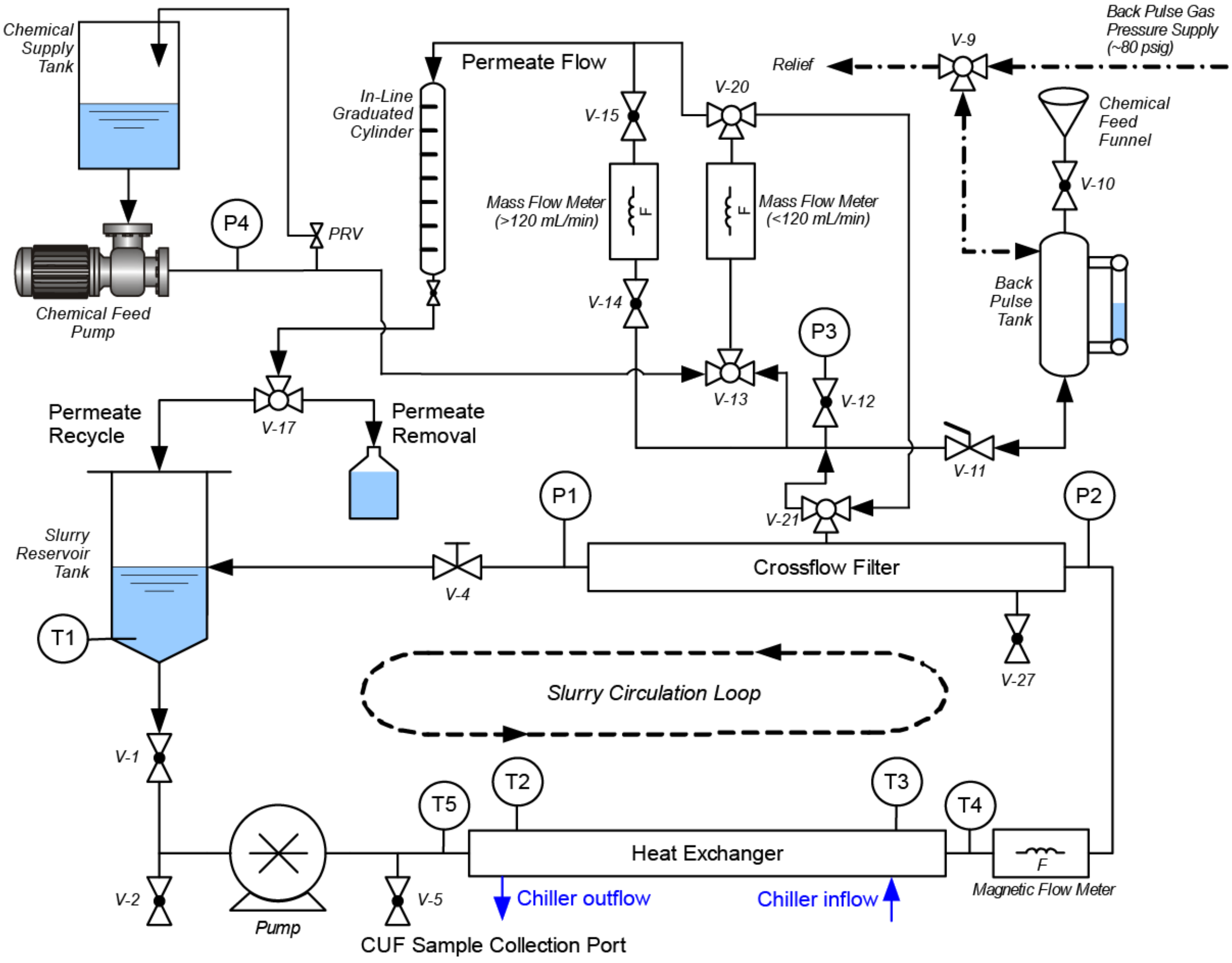

Figure 3.5. Simplified CUF process and instrumentation diagram.

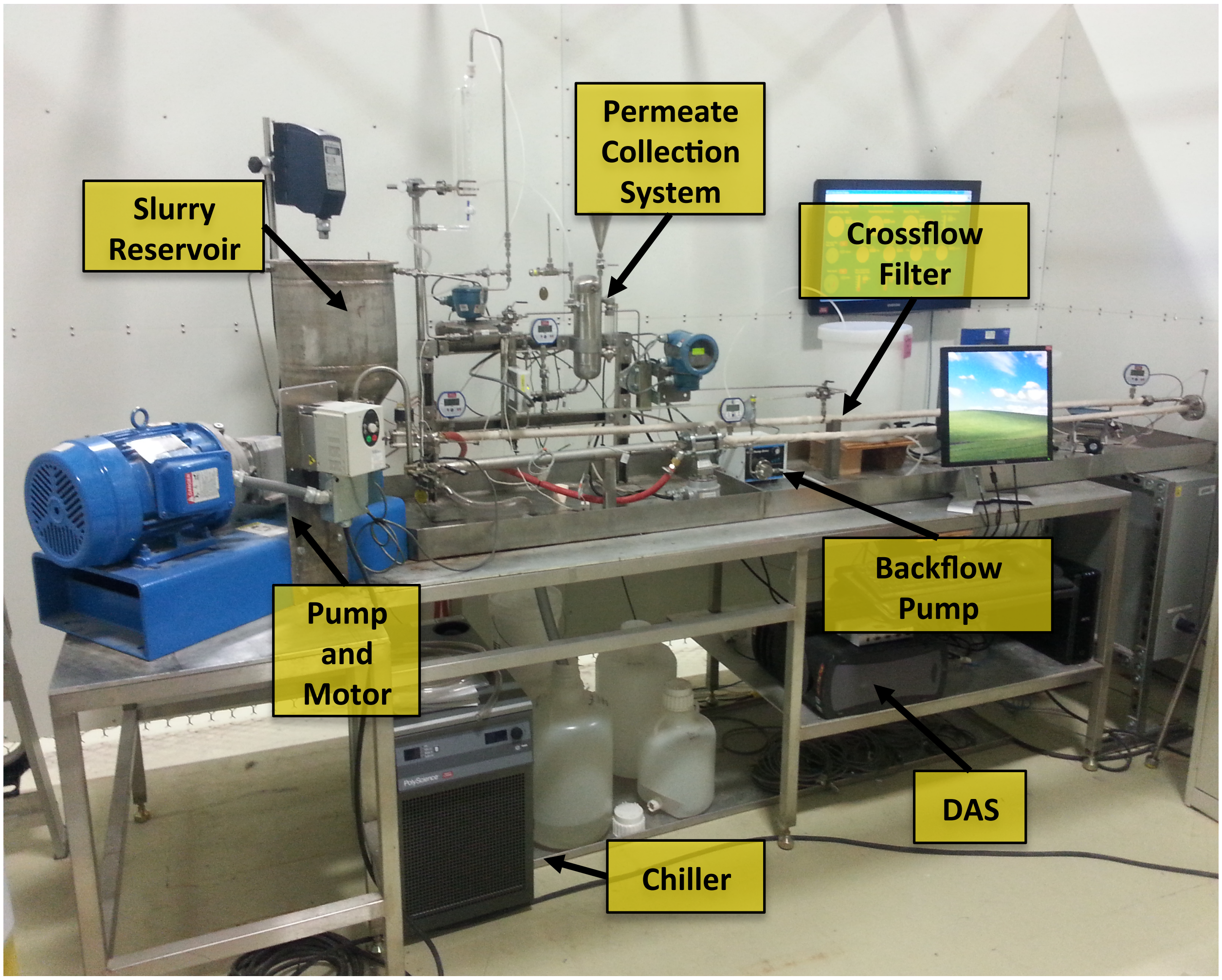

Figure 3.6. Photograph of the CUF system with an 8-foot-long filter assembly installed.

3.2.3 Instrumentation and Data Acquisition System

Most of the sensors on the testing apparatus transmit analog data to an external DAS from National Instruments Corporation (Austin, TX). This system relays analog data to a LabVIEW data collection program operating on a computer desktop system using Windows XP™, Service Pack 2. The software program scales the analog data, simultaneously records the data electronically, and displays it on the computer monitor. Figure 3.7 shows a diagram of the electronic sensors attached to the DAS.

3.3 Simulants

LAWPS CFF testing employed non-radioactive chemical simulants intended to mimic the chemistry of Hanford wastes relevant to LAWPS processing. Two subsets of LAWPS relevant simulants were considered: the LAWPS baseline performance simulants and the LAWPS fouling simulants. The former consists of the 4.0 M, 5.6 M (nominal), 6.0 M, and 8.0 M Na simulants developed by Russell et al. (2017b), while the latter consists of eight unique “fouling” agents selected directly by the CFF testing program and added to a modified version of the 5.6 M (nominal) Na simulant for filtration performance testing. More detailed information regarding both sets of test simulants and their physical properties is provided in Section 4.

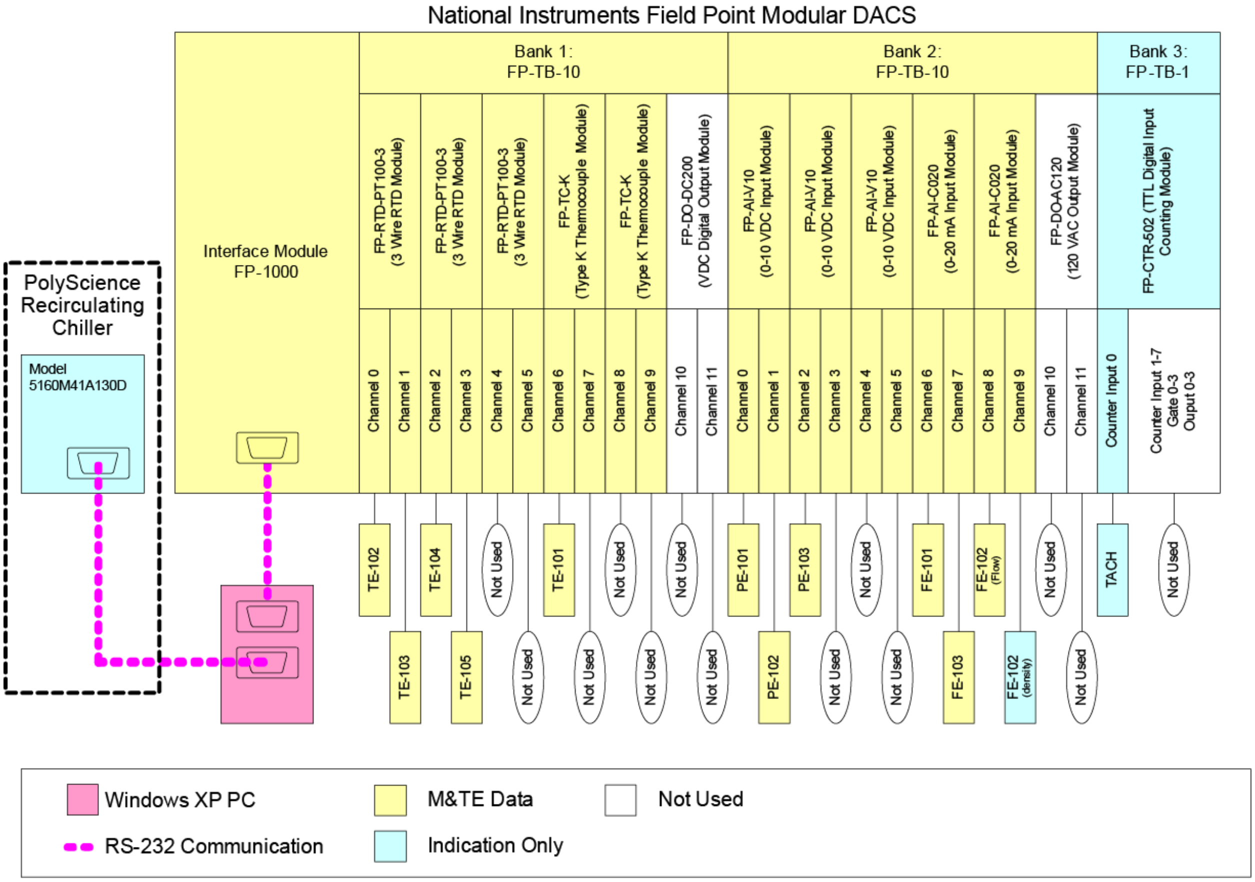

Figure 3.7. Diagram of the CUF DAS.

3.4 Baseline Performance Test Approach

The goal of baseline performance (Series 1) testing was to evaluate the filtration and filter cleaning performance of the four baseline LAWPS simulants. Baseline testing encompassed a set of nine predefined tests that provided a relatively complete sampling of the simulant and test condition parameter space needed to support start-up of ITF operations. Each filtration test explored a unique combination of LAWPS simulant, test temperature, and pressure profile, save for Test 9, which evaluated the reproducibility of filtration data collected during Test 2. In terms of the entire Series 1 test matrix, four simulants, two temperatures (20 and 45 °C), and three TMP profiles were evaluated. The test matrix included tests with one of three test pressure profiles:

- **Constant pressure filtration** (Table 3.1 "Constant" tests) — filtration was effected by use of a constant TMP (see Table 3.1) and was not changed during the test except to maintain the pressure setpoint.

- **Stepped pressure filtration with pressure targets** (Table 3.1 "Stepped TMP" Tests) — filtration was effected by a TMP that was adjusted to the initial target identified in Table 3.1 and then increased by 5 psi at 24-hour intervals.

- **Stepped pressure filtration with flux[1] targets** (Table 3.1 "Stepped Flux" Tests) — filtration was effected by a TMP that was adjusted to reach an initial flux target of 0.03 gpm ft^{-2}. The TMP was subsequently increased (or decreased if appropriate) to return flux to 0.03 gpm ft^{-2} at 24-hour intervals.

Table 3.1 outlines the matrix of tests executed in support of Series 1 testing. Each test employed the CUF test apparatus described in Section 3.2.2. Filter operations used approximately 15 L of test simulant recirculated at an AV of 14.7 ± 0.5 ft s^{-1}. Filtration was effected by application of a constant TMP, a stepped TMP with predefined set-point pressures, or a target step change flux value of 0.030 ± 0.002 gpm ft^{-2}. For the range of LAWPS simulant physical properties and test conditions, it was not always possible to hit both the target TMP and AV. As the AV target was considered secondary to TMP, AV was reduced in cases where there was insufficient pump power to achieve both the target AV and TMP.

The CUF was operated in continuous recycle mode, such that all permeate collected was immediately and continuously returned to the CUF slurry reservoir. In this mode of operation, the concentration of insoluble solids in the test simulant is nominally constant. Any changes in the concentration of the test slurry/suspension that do take place result from 1) initial filling of the filter shell-side permeate collection and return lines and 2) accumulation of solids on the filter and hold-up of solids around fittings and valves. The CUF operating conditions are summarized in Table 3.2

Each filtration test outlined in Table 3.1 was conducted according to a similar set of steps (adjustments were occasionally made during testing and documented in the testing records). For reference, a diagram illustrating these steps is provided in Figure 3.8. The steps are as follows:

Table 3.1. Series 1 test matrix.

Test	Test ID	Simulant	Pressure Profile[(a)]	Test Temp. (°C)
1	LPS-T2S1-NC20-01	5.6 M Na	Constant (20 psi)	20
2	LPS-T2S1-NC45-01	5.6 M Na	Constant (20 psi)	45
3	LPS-T2S1-HC20-01	8.0 M Na	Constant (20 psi	20
4	LPS-T2S1-NS20-01	5.6 M Na	Stepped TMP (10-30 psi)[(b)]	20
5	LPS-T2S1-NS20-02	5.6 M Na	Stepped Flux (0.03 gpm ft^{-2})[(c)]	20
6	LPS-T2S1-HS20-01	8.0 M Na	Stepped Flux (0.03 gpm ft^{-2})[(c,d)]	20
7	LPS-T2S1-4C20-01	4.0 M Na	Constant (20 psi)	20
8	LPS-T2S1-6C20-01	6.0 M Na	Constant (20 psi)	20
9	LPS-T2S1-NC45-02	5.6 M Na	Constant (20 psi)	45

(a) Tests use one of three pressure profile schemes: 1) constant pressure, 2) stepped pressure with fixed pressure targets (i.e., stepped TMP), and 3) stepped pressure with fixed flux targets (i.e., stepped flux).
(b) Pressure is stepped 5 psi after each 24-hour filtration period.
(c) Stepped pressure range to be determined by flux target of 0.03 gpm ft^{-2} after each TMP change if feasible.
(d) Test was not conducted due to results of other Series 1 tests.

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[1] Filter "flux" is defined as the volumetric permeate production rate per unit filter area. The SI units of flux are m s^{-1}; however, filter flux is typically reported in gpm ft^{-2} for Hanford waste-related applications.

Table 3.2. Standard CUF operating conditions for filtration testing.

Parameter	Unit	Value
Filter Manufacturer	n/a	Mott
Filter Grade[(a)]	n/a	0.1
Filter Inner Diameter	inch	0.5
Filter Length	ft	8
Filtration Axial Velocity	$\mathrm{ft\,s^{-1}}$	14.7[(b)]
Filtration Transmembrane Pressure	psi	Varied
Filtration Mode	n/a	Continuous Recycle

(a) The Mott media grade refers to the nominal particle diameter (in micrometers) corresponding to approximately 90% particle removal efficiency in liquid.

(b) The AV target is secondary to TMP. In cases where there is insufficient pump power to achieve both the target AV at the target TMP, AV was reduced.

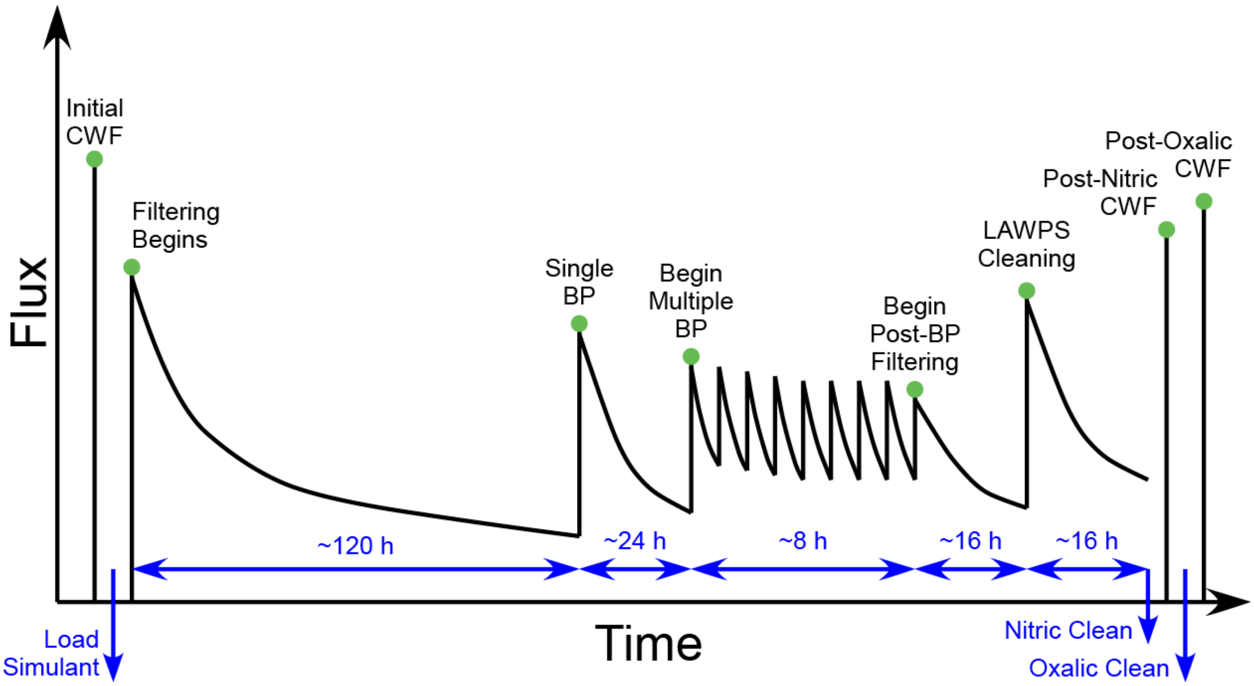

Figure 3.8. Series 1 sequence of test steps with time. The flux response shown is for illustrative purposes only, and the duration of each activity is not scaled. For example, the initial 120-hour filtering period may include "Stepped Pressure Filtration," which would not have the smooth flux response shown here.

1. Clean the filter system thoroughly. This involves chemical cleaning (with nitric and oxalic acids) and disassembly and clean-out of system thermowells, valves, and other locations where insoluble solids hold-up can occur.

2. Measure the pre-test CWF, i.e., Figure 3.8, "Initial CWF".

3. Load the test material into the slurry reservoir, establish target recirculation velocity ($14.7\,\mathrm{ft\,s^{-1}}$), and recirculate for 10 minutes with no applied TMP.

4. Using a TMP of 10 psi, filter sufficient fluid to fill the backpulse chamber and perform a single initial backpulse), i.e., Figure 3.8, "Filtering Begins".

5. Establish filter operating conditions (i.e., set AV to $14.7\,\mathrm{ft\,s^{-1}}$ and set the initial TMP target) and then filter continuously for 120 hours. The purpose of this step is to evaluate LAWPS testing simulant permeate production rate and its decline under varied test conditions and over time-scales relevant to LAWPS operations. During this time, monitor and modify the test conditions as follows:
   (a) Constant pressure filtration (Table 3.1 constant pressure tests) — maintain a single pressure set-point throughout the entire 120-hour period. Do not adjust filter conditions unless a substantial change occurs in AV or TMP.

   (b) Stepped pressure filtration with pressure targets (Table 3.1 stepped TMP tests) — adjust TMP to the initial target identified in Table 3.1 (e.g., 10 psi) and then subsequently increase TMP by 5 psi at 24-hour intervals. Implement pump speed corrections to reach $14.7\,\mathrm{ft\,s^{-1}}$ during each pressure step. Make no adjustments in between pressure set-points.

   (c) Stepped pressure filtration with flux targets (Table 3.1 stepped flux tests) — adjust TMP to reach an initial flux target of $0.03\,\mathrm{gpm\,ft^{-2}}$. Subsequently increase (or decrease if appropriate) the TMP to return flux to $0.03\,\mathrm{gpm\,ft^{-2}}$ at 24-hour intervals. Implement pump speed corrections to maintain $14.7\,\mathrm{ft\,s^{-1}}$ during each pressure step. Between pressure set-points, make no adjustments to correct for drift in the filter flux.

6. Perform a single backpulse and observe the filter response for approximately 24 hours, i.e., Figure 3.8, "Single BP".

7. Perform backpulsed filter operations for 8 hours, i.e., Figure 3.8, "Begin Multiple BP". Here, the filter was operated under the standard, constant-pressure filtering conditions ($14.7\,\mathrm{ft\,s^{-1}}$, 20 psi, and continuous recycle) with a single backpulse performed at 30-minute intervals (for a total of 17 backpulses). The purpose of this step was to provide the LAWPS testing simulant solids sufficient opportunity to foul the filter.

8. After the final backpulse at the 8-hour mark in step 7 (backpulse 17), re-establish filter conditions ($14.7\,\mathrm{ft\,s^{-1}}$, 20 psi, and continuous recycle) and filter continuously for another 16 hours without back-

pulsing, i.e., Figure 3.8, "Begin Post-BP Filtering". The purpose of this step was to collect continuous filtration data on what should be a substantially conditioned (fouled) filter.[1]

9. Partially drain the test simulant.
10. Clean the filter using the LAWPS cleaning protocol (Section 3.6) with no CWF, i.e., Figure 3.8, "LAWPS Cleaning". The filter was reloaded with the test simulant and operated for 16 to 24 hours to measure post-cleaning simulant flux. Note that the cleaning protocol itself had a duration of around 18 hours, which is not shown in Figure 3.8.
11. Clean the filter with nitric acid using the standard nitric acid cleaning protocol (Section 3.6) and retest the CWF, i.e., Figure 3.8, "Post-Nitric CWF".
12. Clean the filter with oxalic acid using the standard oxalic acid cleaning protocol (Section 3.6) and retest the CWF, i.e., Figure 3.8, "Post-Oxalic CWF".

In the test steps described above, a single target TMP (e.g., 20 psi) was established immediately (or at least as quickly as possible) at the start of testing. This approach differs from that used in commercial applications, which gradually raise TMP to 1) limit the initial permeate production rates on relatively clean filters so as not to "overwhelm" the permeate collection system and 2) to slow the initial rate of filter fouling. The approach adopted here was done to limit interaction with the filter system to minimize operator-introduced history effects.

3.5 Filter Fouling Test Approach

The purpose of filter fouling (Series 2) testing was to identify and test "worst-case" fouling solids present in LAWPS-relevant wastes. A review of the nominal- and high-sodium simulant chemistries and of peer-reviewed and historical Hanford waste filtration literature was performed before testing to identify solids that strongly impact filter performance during testing or that could be potentially difficult to clean from the system (and thus pose a risk to the long-term sustainability of operations). From this review, eight fouling components were identified for Series 2 evaluation and CFF testing. Table 3.3 outlines the matrix of Series 2 tests and the selected Series 2 foulants. Each foulant was selected with one or more test goals in mind (as indicated by the "Purpose" column in Table 3.3). The basis for foulant selection and Series 2 goals is discussed more extensively in Section 4.2.

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[1] Repeated backpulsing has been shown to rapidly and effectively condition (foul) the Mott sintered stainless steel filters when filtering Hanford waste simulants (Daniel et al. 2010b, 2011). It is currently postulated that the accelerated fouling associated with backpulse results from repeated disruption of filter cake that protects against particle infiltration into the filter pores. As applied in the current tests, the general function of repeated backpulsing was to hasten irreversible fouling (to better gauge the final extent of irreversible flux loss).

Table 3.3. Series 2 test matrix.

Test	ID	Foulant	Conc.	Temp.	Purpose
1	LPS-T2S2-MS-LO-01	Sodium Oxalate in MSS$^{(a)}$	0.001 wt% UDS$^{(b)}$	20 °C	Evaluate filtration at low UDS content.
2	LPS-T2S2-MS-SP-01	Sodium Phosphate in MSS$^{(a)}$	0.1 wt% UDS$^{(b)}$	45 °C	Evaluate filtration and LAWPS cleaning with known Hanford waste soluble solids and LAWPS cleaning.
3	LPS-T2S2-MS-IO-01	Iron Oxyhydroxide in MSS$^{(a)}$	0.1 wt% UDS$^{(b)}$	20 °C	Evaluate filtration and LAWPS cleaning with known Hanford waste insoluble solids.
4	LPS-T2S2-MS-IP-01	Iron Phosphate in MSS$^{(a)}$	0.1 wt% UDS$^{(b)}$	20 °C	Evaluate filtration and LAWPS cleaning with known Hanford waste insoluble solids.
5	LPS-T2S2-MS-CN-01	Cancrinite in MSS$^{(a)}$	0.1 wt% UDS$^{(b)}$	20 °C	Evaluate filtration and LAWPS cleaning with known Hanford waste insoluble solids.
6	LPS-T2S2-MS-HE-01	HEDTA in MSS$^{(a,c)}$	0.1 wt% TS$^{(e)}$	20 °C	Evaluate filtration and LAWPS cleaning with anticipated Hanford waste soluble organics.
7	LPS-T2S2-NA-RF-01	Degraded sRF Resin in 0.45 M $HNO_3$$^{(d,f)}$	0.1 wt% UDS$^{(b,g)}$	20 °C	Evaluate filtration and LAWPS cleaning with potential LAWPS degradation products.
8	LPS-T2S2-MS-SF-01	Sodium Fluoride in MSS$^{(a,h)}$	6 g L^{-1} TS$^{(e)}$	45 °C	Evaluate filtration and LAWPS cleaning with changing chemistry and precipitation.

(a) MSS — modified nominal (5.6 M) sodium simulant.
(b) UDS — indicates undissolved solids concentration and represents an excess of soluble component above its solubility limit.
(c) HEDTA — N-hydroxyethyl-ethylenediamine-triacetic acid.
(d) sRF — spherical resorcinol-formaldehyde.
(e) TS — indicates a total solids concentration (including both soluble and insoluble) added to simulant suspending phase.
(f) Resin degradation was accomplished before testing by suspending resin in 0.45 M HNO_3 and heating to approximately 50 °C for 24 hours.
(g) Concentration of undegraded resin added to 0.45 M HNO_3 suspending phase.
(h) Solid added to simulant 10 minutes after the start of filtration.

CFF tests evaluating filtration and flux recovery performance for the LAWPS fouling simulants followed a similar test procedure to that used for Series 1 testing. The steps listed below were performed for each test:

1. Clean the filter system thoroughly. This involves chemical cleaning (with nitric and oxalic acids) and disassembly and clean out of system thermowells, valves, and other locations where solids hold-up can occur.

2. Measure the pre-test CWF.

3. Load the test material into the slurry reservoir, establish target recirculation velocity ($14.7 \pm 0.5\,\mathrm{ft\,s^{-1}}$), establish the target operating temperature, and recirculate for 10 minutes with no applied TMP. It should be noted that the simulant added may not initially include the fouling agent. Fouling agents may be added to the slurry reservoir at predetermined intervals over the course of testing. This could include a single addition of fouling agent at the start of testing or could include periodic additions throughout the test series.

4. Using a TMP of 10 psi, filter sufficient fluid to fill the backpulse chamber and perform an initial backpulse.

5. Establish filter operating conditions (i.e., set AV at $14.7 \pm 0.5\,\mathrm{ft\,s^{-1}}$ and TMP to 20 ± 2 psi) and filter continuously for 120 hours. The purpose of this step is to provide baseline flux information regarding the impact of the selected fouling agent relative to Series 1 testing (or another control). During this time, filter conditions will not be adjusted for drift in AV and TMP unless a substantial change occurs in either operating condition and only after discussion with the cognizant scientist and project management. For the purposes of this test matrix, substantial drift is defined as a change in TMP greater than 2 psi or a change in AV greater than $0.5\,\mathrm{ft\,s^{-1}}$.

6. Backpulse the filter and filter for an additional 24 hours at the target TMP and AV.

7. Continue filtering for another 8 hours at set operating conditions (AV at $14.7\,\mathrm{ft\,s^{-1}}$ and TMP to 20 psi), but with a single backpulse of the filter at 30-minute intervals (for a total of 17 backpulses). During this time, no fouling agents are added. The purpose of repeated backpulsing is to drive any fouling agent already added to the simulant deep into the filter element and provide a challenging filter to clean.

8. Backpulse the filter and continue filtering for 12 additional hours. This step will provide baseline flux behavior for a conditioned filter.

9. Drain the test simulant and clean the filter using standard LAWPS cleaning protocol. Following the LAWPS protocol, reload the slurry and filter at target conditions for an additional 16 hours.

10. Clean using the standard nitric and oxalic acid cleaning protocols and retest the CWF after each acid cleaning.

As originally proposed, the Series 2 test steps outlined above included an optional 1- to 2-hour temperature excursion step following the 17 repeated backpulses that was intended to solubilize and re-precipitate soluble fouling agents (e.g., sodium phosphate). Series 1 temperature tests, along with data from the initial 120-

hour test of the “soluble” fouling agents considered under Series 2, suggested the temperature excursion would not be of sufficient duration to provide meaningful results. As such, the temperature excursion step was not executed during Series 2 testing and has been excluded from the steps above.

Several of the fouling simulants proposed for Series 2 testing, while present in actual wastes or waste simulants that exhibit poor filtration performance, had not been tested in isolation, and as such, the exact fouling proclivity was unknown at the start of Series 2 testing. In several cases, the selected fouling agents (e.g., HEDTA) did not lead to filter performance that was substantially below that of the nominal 5.6 M Na LAWPS simulant. Given the limited number of tests available to explore LAWPS fouling, additional fouling agents were added to the test suspension for tests where the fouling agents did not yield significant fouling. Table 3.4 summarizes the fouling test modifications implemented during Series 2 testing. The intent of these modifications was to increase the degree of fouling through increased fouling agent concentration (via direct addition or precipitation) or through synergistic effects with other fouling agents.

Table 3.4. Series 2 test modifications.

Test	Modification	Time Performed	Purpose
2	Dropped temperature to 20 °C to precipitate phosphate solids	After 24 hours elapsed during initial 120-hour period	Attempt to precipitate sodium phosphate in situ during filter operations
2	Added additional 0.1 wt% sodium phosphate	After 96 hours elapsed during initial 120-hour period	Observe if sodium phosphate fouling has a concentration effect
6	Added additional 0.1 wt% HEDTA	After 24 hours elapsed during initial 120-hour period	Observe if additional HEDTA had similar impact to first 0.1 wt% increment
6	Added ∼0.02 wt% Fe_2O_3 from Test 3	After 48 hours elapsed during initial 120-hour period	Observe if HEDTA and insoluble Fe have any chemical interactions
7	Added HEDTA and $Fe(NO_3)_3$ in equal amounts (∼0.05 wt%)	Approximately 4 hours after start of post-LAWPS cleaning operation period	sRF resin had high flux — observe if complexes form with soluble Fe and HEDTA
8	Dropped temperature to 20 °C to induce precipitation	After 48 hours elapsed during initial 120-hour period	Attempt to precipitate fluoride-containing solids in situ during filter operations

3.6 Filter Flux Recovery Operations

The test steps outlined in Section 3.4 invoke several standard filter flux recovery operations. These operations include standard chemical cleaning with nitric and oxalic acids, backpulse operations, and LAWPS backflow cleaning operations. The specific methodology for each of these flux recovery operations is outlined in the sections that follow.

3.6.1 Standard Chemical Cleaning

In all testing, chemical cleaning of the CUF system and filter assembly was done as a matter of routine operation, regardless of the actual degree of fouling, in an attempt to provide a "uniformly" clean filter at the start of each filter test. The testing steps described in the preceding sections invoke cleaning at both the start and end of testing; these cleaning steps are not necessarily unique, and the cleaning operations at the end of one test may be taken credit for at the start of the next test if the CWF is found to be in an acceptable range as defined by historical CUF testing and bubble point data [e.g., see Section 7 of Daniel et al. (2011)]. Acceptance criteria for CWF are stated in the instructions below.

Standard chemical cleaning operations used at the start and end of testing used both nitric and oxalic acids. Standard nitric acid cleaning operations involve the following steps:

1. Add approximately 2 L of 2 M nitric acid solution to the slurry reservoir and recirculate through the slurry loop for 30 minutes.

2. Circulate the acid back and forth across the filter element by filling and discharging the backpulse chamber with the flow meters isolated.

3. Drain the nitric acid solution from the system.

4. Rinse the system with 1 L of deionized water followed by two 2-L rinses with 0.01 M sodium hydroxide solution.

Standard oxalic acid cleaning entails the following:

1. Add approximately 2 L of 0.5 M oxalic acid solution to the slurry reservoir and recirculate through the slurry loop for 30 minutes.

2. Circulate the acid back and forth across the filter element by filling and discharging the backpulse chamber with the flow meters isolated.

3. Drain the oxalic acid solution from the system.

4. Rinse the system with 1 L of deionized water followed by two 2-L rinses with 0.01 M sodium hydroxide solution.

It must be noted that the two standard chemical cleaning operations outlined in these steps are not representative of any LAWPS flux recover/filter cleaning operations nor are they considered as alternate LAWPS cleaning strategies. They are implemented in current testing as pre- and post-test CUF laboratory cleaning regimen.

3.6.2 Backpulsing

Series 1 filter testing employed backpulsing as a routine method for recovering permeate production rates. Backpulse operations involved the following standardized steps:

1. Without changing filter operating conditions (i.e., AV and backpressure), stop permeate recycle (if in continuous recycle) or collection (if dewatering) and open the backpulse chamber to atmosphere. This will cause the backpulse chamber to fill. Continue filling the backpulse chamber until it is full (as marked on the backpulse chamber sight glass), and then isolate the backpulse chamber by closing valves to both atmosphere and filter.

2. Lower AV until the filter inlet pressure is below 10 psi. The backpressure valve should not be adjusted during this process.

3. Pressurize the backpulse chamber to 80 psi with nitrogen.

4. Open the valve separating filter and backpulse chamber. Allow the backpulse chamber to empty until the level falls below the lowest level visible in the sight glass. Once the chamber is empty, close the valve separating filter and backpulse chamber.

5. Return AV to its original operating value and then resume permeate recycle or collection.

3.6.3 LAWPS Cleaning

All Series 1 filter tests employed a cleaning protocol similar to that planned for use in LAWPS. This "LAWPS cleaning" protocol simulates the use of displacement and elution solutions fed through the IX columns and back through the filter (shell side to tube side). Table 3.5 provides the order of chemical delivery and the expected volumes and flow rates of the cleaning protocol, which are based on scaling down from the full-scale process values (provided by WRPS). The steps are as follows:

1. Isolate the slurry reservoir and pump from the rest of the process piping and the filter.

2. Load a cleaning solution chemical supply tank with the appropriate volume (or partial volume) of cleaning or displace solution to flow through the filter.

3. Use a chemical feed pump and the in-line mass flow meter to set the feed rate of the chemical solution to the nominal target values given in Table 3.5. Small variations from the target flow rates are expected (up to ±10%). The solution will pass through the shell side of the filter to the tube side and then drain into a collection vessel.

4. Continue cleaning or displacement solution delivery until the target volume listed in Table 3.5 has been delivered.

5. Repeat steps 2 through 4 until all the cleaning solutions in Table 3.5 have been delivered.

It should be noted that the cleaning volumes listed in Table 3.5 do not account for differences in pipeline and permeate shell volumes between the CUF and LAWPS. CUF permeate side volume between the chemical feed tank used to supply LAWPS solutions to the filter is minimal, and as such, there is minimal lag time between the start of CUF LAWPS cleaning and breakthrough of cleaning solution through the CUF filter. In contrast, full-scale LAWPS cleaning requires displacement of significant pipe and shell side volume before breakthrough of LAWPS cleaning solutions through the LAWPS filter bundles.

3.7 Key Test Steps and Outcomes

The steps outlined in Series 1 filtration and cleaning tests were conducted with the intent of gathering data to, as stated in the SOW provided by WRPS, "understand baseline filter performance, examine the extent of fouling in the filter over time, suggest approaches for mitigation of fouling (should it occur), and confirm the effectiveness of the planned cleaning strategy." A summary of key test steps and the outcomes they provide is outlined below.

Clean Water Flux Measurement: CWF measurement provides a standardized metric against which the cleanliness of the filter and filter system can be measured. When conducted at the start of the test, it will provide confidence that both the filter and CUF system are free of particulate contamination. When conducted before and after the standardized cleaning operations described in Section 3.6, it can provide a measure of how effectively cleaning operations have restored permeate production rates and cleared solids that "foul" the filter.

Continuous Filtration at Constant TMP: Filtration at constant TMP provides baseline information on the magnitude and rate of decline in permeate production rates. Use of constant pressure allows direct comparison to historical Hanford waste filtration data and interpretation by waste filtration models derived in Daniel et al. (2011) and Schonewill et al. (2012, 2015).

Stepped TMP Filtration (with TMP Targets): Stepped TMP filtration (with TMP targets) provides initial process data on the efficacy and fouling rates that will be observed in controlled flux filtration operations planned for full-scale LAWPS. Here, the TMP set-points are predefined and could result in filter fluxes outside the operating range of LAWPS.

Stepped TMP Filtration (with a Filter Flux Target): Stepped TMP filtration (with flux targets) provides relevant process data on the efficacy and fouling rates that will be observed in controlled flux filtration

Table 3.5. LAWPS cleaning protocol chemical solution volumes and delivery flow rates (scaled for implementation in the CUF test apparatus).

LAWPS Step	Solution	Flow Rate ($\mathrm{mL\,min^{-1}}$)	Volume (L)	Duration (hr)
(1) Displace	0.1 M NaOH	114.6	13.2	1.9
(2) Rinse	Process Water	114.6	8.82	1.3
(3) Elution[a]	0.45 M HNO_3	53.5	34.4	10.7
(4) Rinse	Process Water	53.5	11.5	3.6

(a) The nitric acid volume of 34.4 L represents scaled delivery of 50% of planned elution volume. The first 50% (approximately) is planned to be sent directly to waste.

operations planned for full-scale LAWPS. Here, the flux target is 0.03 gpm ft^{-2} and is representative of the upper range of LAWPS flux rates.

Single Backpulse Operations: Single backpulses of the filter serve to 1) clear solids from the surface of the filter that accumulated during establishment of operating conditions (pressure and AV) at the start of testing and 2) gauge the extent of irreversible (with respect to backpulsing) fouling after a fixed period of filtration. Comparison of permeate production rates before and after backpulse provides information on the effectiveness of the backpulse system at clearing pore and surface accumulation.

Repeated Backpulse Operations: Repeated backpulsing of the filter generally accelerates irreversible loss of filter flux (Daniel et al. 2011) while providing process information on the long-term sustainability of filter operations cleaned primarily by backpulsing. The accelerated flux loss is believed to occur as a result of repeated disruption of surface cakes that limit infiltration of particles into filter pores. As applied in the current testing, repeated backpulsing is intended to provide a worst-case fouling condition to evaluate lower permeate production rate bounds and to provide a filter that is challenging with respect to chemical cleaning operations.

Chemical Cleaning Operations: Chemical cleaning operations are conducted with the primary intent of evaluating the efficacy of the given cleaning agent at clearing solids that have accumulated in the pores and on the surface of the filter and that reduced filter performance. Generally, chemical cleaning is done to address fouling that is irreversible with respect to backpulsing.

3.8 Analysis of Filtration Data

The CUF test system includes instrumentation for measuring 1) filter AV, 2) filter TMP, and 3) rate of permeate production and density. Data from CUF instrumentation are handled by the CUF DAS (described in Section 3.2.3) and are posted to a plain text file. The CUF records data at 0.4 Hz and also generates data processed and averaged over 1-minute intervals. Raw (0.4 Hz) and 1-minute average data are posted to two separate plain text files. The output data parameters generated by the CUF DAS for the 1-minute data results are listed in Table 3.6. Raw data files include all the information necessary to re-create the averaged data.

Table 3.6. CUF filtration system measurement instrumentation (with notation).

Parameter	Symbol	Units
Slurry Reservoir Temperature	T	°C
Permeate Pressure	p_p	psig$^{(a)}$
Filter Inlet Pressure	p_i	psig
Filter Outlet Pressure	p_o	psig
Filter Transmembrane Pressure	δp_t	psid$^{(b)}$
Volumetric Slurry Flow	Q_s	gpm
Filter Axial Velocity	u	ft s^{-1}
Permeate Production Rate	Q_p	mL min^{-1}
Permeate Density	ρ	g mL^{-1}

(a) psig — pounds per square inch gauge
(b) psid — pounds per square inch differential

Under certain operating conditions, the raw and averaged data contained a significant number of outliers, and inclusion of these outliers has a significant impact on the ability to analyze. Because these outliers are considered unrepresentative of the actual filtration behavior, the raw 0.4-Hz data were reanalyzed and re-averaged to exclude any outliers from the data set. Exclusion was based on Dixon's Q-Test using an 80% confidence limit (Rorabacher 1991). For each parameter listed in Table 3.6, the Q-Test was applied to each subset of 24 points used to generate the averaged parameter value until 1) the subset of selected data points passed the Q-Test or 2) a maximum of 3 data points had been rejected from that subset of 24 points. Application of the Q-Test removes most (but not all) of the outliers, while preserving process step changes associated with changing conditions (e.g., start-up activities during 0 to 2 hours elapsed time).

After data filtering and re-averaging, the CUF data were further processed to calculate temperature- and TMP-corrected filter flux. First, the permeate production rate Q_p (which is measured directly by the DAS) was converted to "uncorrected" filter flux j using:

$$j = \frac{Q_p}{A} \tag{3.1}$$

where A is the surface area of the filter. The filter area was calculated using from the known length (L =8 ft) and inner diameter (D =0.5 inch) of the crossflow filter element by:

$$A = \pi DL \tag{3.2}$$

Next, the flux j was corrected for any deviations in the slurry reservoir temperature from the nominal operating temperature (T_o) of 20 °C (298.15 K) using:

$$j_c = j \exp\left[-\beta\left(\frac{1}{T} - \frac{1}{T_o}\right)\right] \tag{3.3}$$

Here, j_c is the "temperature-corrected" filter flux and T is the actual temperature of the slurry reservoir. The value for β is experimentally determined and, per Geeting et al. (2003), is 2500 K. Daniel et al. (2009) demonstrate that temperature correction of permeate production rates for high-level waste simulants derives both from changes in permeate viscosity and changes in the structure of the solids that foul the filter. The temperature dependence of permeate viscosity change generally is well-described over small temperature ranges (∼25 °C) using an equation similar in form to 3.3:

$$\mu = \mu_o \exp\left[\lambda\left(\frac{1}{T} - \frac{1}{T_o}\right)\right] \tag{3.4}$$

where μ_o is the reference viscosity at temperature T_o. From Darcy's law, flow through a porous medium should be inversely proportional to viscosity μ, giving λ and β similar functional meanings. However, direct comparison of these two values for a high-level waste simulant containing approximately 5 wt% solids found $\beta > \lambda$ (Daniel et al. 2009). As knowledge of waste viscosity alone is insufficient to temperature-correct flux, temperature corrections in the current report will employ the standard β value of 2500 K. For comparison, the viscosity measurements taken as a function of simulant temperature will be used to derive coefficients for Eq. 3.4.

The filter flux was also corrected for deviations in actual TMP (δp_t), from the TMP set-point ($\delta p_{t,o}$) using

$$j_{p,c} = \left(\frac{\delta p_{t,o}}{\delta p_t}\right) j_c \tag{3.5}$$

Here, $j_{p,c}$ is the "temperature- and TMP-corrected" flux. The measured TMP was calculated by the DAS by subtracting the permeate pressure p_p (as measured on the shell-side of the filter) from the average of the filter inlet pressure p_i and outlet pressure p_o (as measured on the tube-side of the filter). Thus,

$$\delta p_t = \frac{(p_i + p_o)}{2} - p_p \tag{3.6}$$

Equation 3.5 cannot strictly be applied when a compressible cake exists. Filter cakes formed by inorganic solids are generally considered irreversibly compressible such that they compress when TMP is increased but do not expand when TMP is lowered (McCarthy et al. 2002). The same is likely true of filter cakes formed by the undissolved salts present in the LAWPS simulants. The implications of correcting TMP irreversible cakes formed by Hanford waste have been discussed in Daniel et al. (2011). The error associated with TMP-correcting the flux is acknowledged but not evaluated in any meaningful way.

Given that filtration testing will be done at different pressures and with different test fluids, direct comparison of results at equivalent operating conditions was not always possible. When evaluating different simulants, a measure of fouling that is independent of the impacts of viscosity, temperature, and TMP is needed. Both temperature (and consequently viscosity) and TMP vary with time. This measure is only partially achieved through use of Eq. 3.5, as $j_{p,c}$ still includes a measure of viscosity and of the physical configuration of the filter element itself (including terms that lead to the baseline resistance of the filter itself, such as porosity and thickness). To avoid the effect of local fluid and system geometry to the best extent possible, the following normalized measure of filter flux η is proposed:

$$\eta = \frac{j}{j_o} \tag{3.7}$$

Here, the normalizing flux j_o is based on Darcy's law for flow of liquids through porous media:

$$j_o = \frac{\delta p_t}{\mu \Delta_m r_{m,o}} \tag{3.8}$$

where Δ_m is the thickness of the filter and $r_{m,o}$ is the baseline (as-manufactured) unit-length resistance of the filter element. For the Mott Grade 0.1 filter element used in LAWPS CFF testing, $r_{m,o}$ and Δ_m are $1.36 \times 10^{14}\ \mathrm{m}^{-2}$ and 0.0625 inch, respectively (Rubow and Jha 1999, Daniel et al. 2011). As implemented in this report, determination of the normalized flux uses an expression for $\mu(T(t))$ and the time series of δp_t to calculate j_o at each time step. The viscosity is generally computed using Eq. 3.4 with a reference temperature T_o of 25 °C (298.15 K) and the coefficients given in Table 4.4. The one exception is for 0.45 M HNO_3, whose temperature-dependent viscosity is similar enough to that of water to predict using a general correlation for water viscosity proposed by Laliberté (2007) and valid over 0 to 100 °C. The Laliberté (2007) water viscosity μ_w correlation is:

$$\mu_w = \frac{T + 246}{(0.05594T + 5.2842)T + 137.37} \tag{3.9}$$

where T is temperature in °C.

The volume filtered is a quantity that represents actual permeate production over operating time, and is a measure of potential throughput. Volume filtered is calculated directly from the observed permeate flow rate Q_p by integration:

$$V_F = \int_0^t Q_p dt \tag{3.10}$$

In practice, the integration described in Eq. 3.10 was performed numerically using the trapezoidal rule. A dimensionless measure of the volume filtered ν was estimated by normalizing the filtered volume V_F by V_{tank}. This is akin to the number of feed tank volume exchanges that occur over the filtration period. In this document, V_{tank} is selected as a constant and fixed as the nominal feed tank volume used during testing (15 L). Over an operating period of approximately 5 days, ν was typically > 40.

3.9 Analytical Methods

Physical property testing of the LAWPS simulants was limited to measurement of solid PSD, suspending phase viscosity, mineral phase identification, liquid and suspension solid content, and liquid and suspension density. This section provides a high-level summary of the experimental approaches and/or test equipment used to measure these four select physical properties. Measured physical properties of the baseline LAWPS and fouling simulants can be found in Section 4 of this report.

3.9.1 Particle Size Distribution

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 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 is reduced to 0.02 to 600 µm (dependent on material density). Although particle sizes above 600 µm can be observed with the Hydro μP, volume contribution of solids falling above this size limit may not be accurately determined by the instrument. Measurement data were recorded and analyzed using Mastersizer 2000 software, Version 5.6. Table 3.7 provides a summary of basic information regarding the Mastersizer analyzer and accessory used for the current particle size measurements. All particle size distributions measurements were performed in sample-appropriate supernate. Measurement of four LAWPS simulants (discussed in Section 4) used the corresponding LAWPS supernate obtained by filtering a small volume of simulant through a 0.45 µm syringe filter. For measurement of the LAWPS fouling simulants, solids were dispersed in a modified version of the 5.6 M Na LAWPS simulant sodium simulant that had been filtered through a 0.45 µm, with exception of sRF resin measurements, which were done in 0.45 M HNO_3.

Table 3.7. 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	0–2000 rpm (variable)
Ultrasound Power	0–20 W (variable)
Software Version/Date	5.6/1998–2009

3.9.2 Liquid Viscosity

Simulant liquid viscosity was measured using a Haake RS600 rheometer (now sold by Thermo Fischer Scientific, Waltham, MA) with a Z41 concentric cylinder measuring system. Rheometer control and data acquisition are accomplished through a remote computer connection using the RheoWin Pro Job Manager Software, Version 4. Flow curve measurements (i.e.,material stress response τ versus applied shear rate $\dot{\gamma}$) at controlled temperatures ranging from 20 to 45 °C were performed. Each flow curve was measured over an 11-minute period. During the first 5 minutes, the shear rate was gradually increased from zero up to a pre-defined maximum shear rate (typically between 200 and $1000\,\mathrm{s}^{-1}$). Next, the shear rate was held constant at the predefined maximum shear rate for 1 minute. For the final 5 minutes, the shear rate was gradually reduced back to zero. During this time, the resisting torque and rotational rate were continuously monitored and recorded. After measurement, the recorded flow curve data were analyzed with linear least-squares regression analysis to determine best-fit values for the Newtonian viscosity μ. For all liquid samples tested herein, the expected Newtonian stress response, characterized by:

$$\tau = \mu\dot{\gamma} \tag{3.11}$$

was observed.

3.9.3 Mineral Phase Identification

Solid mineral phase identification was accomplished through X-ray diffraction (XRD) using a Bruker D8 Advance diffractometer (Bruker AXS Inc., Madison, WI) with Cu Kα x-rays and a LynxEye™ position-sensitive detector with a collection window of 3° 2θ. Scan parameters were 5–75° 2θ with a step of 0.015° 2θ and a 0.6 s dwell at each step. Bruker AXS DIFFRACplus EVA software (Version 14.0.0.0) was used to identify crystalline phases from the measured XRD patterns. XRD analyses were not performed in accordance with the requirements of the LAWPS Integrated Support Test program QA guidelines, and as such should be considered unqualified and "For Information Only." As employed in the current report, XRD analysis was used only to provide insight into expected mineral phases or phase changes and to confirm synthesis products from previously developed synthesis methods.

Simulant solids for XRD analysis were provided either as solids suspended in a LAWPS supernate or as dry stock solids. To prepare suspensions for XRD analysis, the sample solids were first separated from simulant suspensions using a bench-top micro-centrifuge (~3 mL). The solids recovered from centrifuging were then washed several times with a suitable solvent (typically isopropanol or ethanol). The washed solids were finally suspended in a solvent and added drop-wise to a low background sample holder to form a thin film upon solvent evaporation. Dry powder samples were finely ground and pressed into a disk suitable for XRD analysis.

3.9.4 Solids Content

Analysis of the solid content of test material suspensions and suspending phases involves determination of 1) the total solids content of the suspension (which includes both dissolved and undissolved solids), 2) the suspension undissolved solids, and 3) the suspending phase dissolved solids. Solids content analysis employs an HR83 halogen moisture analyzer from Mettler Toledo. Total solids, x_{TS}, is defined as the mass of dried solids per mass "wet" slurry before drying. Total solids is determined by simply drying an aliquot of mass of the test material in the HR83 moisture analyzer and taking the ratio of dried material and original wet masses, $m_{T,d}$ and m_T, respectively, such that:

$$x_{\mathrm{TS}} = \frac{m_{T,d}}{m_T} \tag{3.12}$$

Dissolved solids content, x_{DS}, is defined as the mass solids dissolved in a unit mass of suspension suspending phase. Dissolved solids content is determined by first separating any undissolved solids from the suspension using a centrifuge and decant operation. A subsample (of mass m_S) of the resultant liquid is then placed in the HR83 moisture analyzer and dried. The dissolved solids content is then determined by calculating the ratio of the final mass of dried sample solids ($m_{S,d}$) to its original wet mass, such that:

$$x_{\mathrm{DS}} = \frac{m_{S,d}}{m_S} \tag{3.13}$$

The undissolved solids content, x_{UDS}, is defined as the mass of undissolved solids per unit mass of suspension. It can be calculated from measured total and dissolved solids contents using the following formula:

$$x_{\mathrm{UDS}} = \frac{x_{\mathrm{TS}} - x_{\mathrm{DS}}}{1 - x_{\mathrm{DS}}} \tag{3.14}$$

All solids content analyses performed in the HR83 moisture analyzer involved heating the sample to 95 °C for 30 minutes to drive off bulk moisture without boiling. Then, the sample is heated to 105 °C to drive off the remaining moisture until the sample reaches a weight stability criterion 5 in the Mettler Toledo user interface settings. Upon achievement of weight stability, the measurement concludes and the moisture analyzer automatically calculates the solids content (either x_{TS} for suspensions or x_{DS} for liquids). The undissolved solids content x_{UDS} requires separate measurement of suspension and suspending phase samples, and must be calculated by the moisture analyzer user.

3.9.5 Density

Test suspension and suspending liquid density were measured using glass pycnometers of standardized volume. For each density measurement, an aliquot of the test material was loaded into a pre-weighed pycnometer to the marked fill level. The total mass of the filled pycnometer was weighed using a 4-place analytical balance. Next, the mass of fluid added to the pycnometer was determined by the difference of the total filled mass and the pycnometer tare. Density was then determined by dividing fluid mass by the standardized pycnometer volume.

4.0 Filtration Simulants

To address the separate needs of LAWPS baseline filter performance (Series 1) tests and LAWPS fouling (Series 2) test, two subsets of simulants were considered. The first were the LAWPS baseline simulants developed and reported in Russell et al. (2017b) and include the 4.0 M, 5.6 M (nominal), 6.0 M, and 8.0 M Na simulants. Although already documented in Russell et al. (2017b), the chemical make-up and physical properties of these four LAWPS simulants are reproduced herein to ensure that the simulant properties are readily available to readers of the current report. In addition to the baseline simulants, eight fouling simulants were developed by adding select fouling agents to a suspending phase that, with one exception, was a modified version of the 5.6 M Na sodium simulant. The basis for selection of these fouling agents, their chemical and physical properties, and the chemistry of the suspending phases used in the fouling tests are discussed in the second half of this report section.

4.1 LAWPS Baseline Performance Simulants

Four unique LAWPS simulants with chemical compositions representative of the waste chemistries and solids loadings of the expected feed streams to the LAWPS facility were developed by Russell et al. (2017b) for CFF Series 1 testing. Development of these four simulants was guided by operating conditions relevant for the LAWPS filters (see Table 4.1) and key simulant attributes requested by WRPS (see Table 4.2). The simulants are differentiated primarily by their total sodium molarity [4.0 M, 5.6 M (nominal), 6.0 M, and 8.0 M Na] and are generated by ordered mixing of soluble salts and, in select cases, insoluble mineral oxides in water. The chemical compounds that make up the baseline simulants are provided in Table 4.3; each simulant make-up recipe listed in Table 4.3 used chemicals or stock solutions that were purchased from Sigma-Aldrich, Inc. (St. Louis, MO), with exception of sodium oxalate ($Na_2C_2O_4$, purchased from Noah Technologies Inc., San Antonio, TX), boehmite (AlOOH, purchased from Nabaltec AG, Schwandorf, Germany), and deionized water [produced by Laboratory purification units (Barnstead Nanopure and equivalent) capable of generating $>$18 MΩ cm water].

The physical properties of the four LAWPS baseline simulants were determined using the methods described in Section 3. A tabular summary of simulant PSD, viscosity, and solids content is provided in Table 4.4. Graphs of PSD and viscosity temperature dependence for the four baseline simulants are provided in Figures 4.2 and 4.1, respectively. Figure 4.2 shows that the LAWPS baseline simulants exhibit a broad range

Table 4.1. Relevant operating conditions for LAWPS filters.

Parameter	**Condition**
Filter	Mott Grade 0.1, 316L sintered stainless steel filter (0.5-inch inner diameter)
Filtrate Production	4 to 17 gpm [Note: This corresponds to a filter flux of 0.0075 to 0.0320 gpm ft^{-2} on full-scale filters (532 ft^2)]
Tube [Axial] Velocity	14.7 ft s^{-1} (superficial velocity in a 0.5-inch filter element)
Solids	0 to 3.3 wt% (0.8 wt% nominal) [Note: The nominal concentration represents a maximum filter solids loading of approximately 0.5 kg ft^{-2}]
Particle Size	0.01 to 210 μm (7.5 μm nominal)

Table 4.2. Key attributes of the LAWPS simulants.

Parameter	**Attribute(s)**
Chemical Species	Sodium oxalate
	Non-radioactive cesium
	Sodium hydroxide
	Sodium nitrate
	Potassium-containing salts
	Phosphate-containing salts
Density	1.0 to 1.35 $\mathrm{g\,mL^{-1}}$
Viscosity	1 to 15 cP
Temperature	20 to 45 °C
Solids Concentration	0 to 3.3 wt%
Solids Particle Size	0.01 to 210 µm

Table 4.3. LAWPS simulant recipes.

Component	Formula Weight ($\mathrm{g\,mol^{-1}}$)	Composition ($\mathrm{g\,kg^{-1}}$)[(a)]			
		4.0 M Na	5.6 M Na	6.0 M Na	8.0 M Na
Primary Supernate Composition					
$Al(NO_3)_3 \cdot 9\,H_2O$	375.13	37.51	49.82	52.17	65.93
NaOH (50% solution, w/w)	40.00	99.92	132.73	138.91	181.61
$CsNO_3$	194.91	0.0122	0.016	0.0169	0.021
KCl	74.55	5.48	7.28	7.63	9.63
NaF	41.99	– –	– –	2.29	– –
Na_2SO_4	142.04	5.65	7.51	7.86	9.94
$NaNO_2$	69.00	42.39	56.30	58.77	74.51
$NaNO_3$	84.99	65.63	87.17	91.39	115.36
$Na_3PO_4 \cdot 12\,H_2O$	380.12	9.89	13.14	13.75	12.16
Na_2CO_3	105.99	29.81	– –	41.41	– –
$Na_2CO_3 \cdot H_2O$	124.00	– –	46.33	– –	59.70
$Na_2C_2O_4$	134.00	1.02	1.36	1.42	1.26
Water, Deionized	18.02	702.69	598.35	584.38	469.87
Post Makeup Additions					
$Na_2C_2O_4$	134.00	– –	8.065	– –	– –
Boehmite (AlOOH)[(b)]	59.99	1.00	– –	– –	– –

(a) Basis is gram component per kilogram of simulant supernate solution.
(b) Nabaltec APRYAL AOH 60 boehmite powder.

of particle sizes that span the lower end of detection for laser diffraction (20 to 200 nm) up to approximately 1 mm. Given the high ionic strength (>1 M) of the suspending phases of the four LAWPS simulants, the precipitated and added simulant solids likely include a significant fraction of particle agglomerates that can break apart during suspension mixing or flow. For this reason, the size measurements shown in Figure 4.2 correspond to "post-sonication" dispersion conditions intended to lead to size attrition representative of that found during CFF and waste processing. Additional particle size measurements for the baseline simulant can be found in Appendix C.

Mott Grade 0.1 sintered stainless steel filter media has an estimated hydrodynamic pore size of 1 to 3 µm. Given this pore size range, all four of the LAWPS simulants contain a fraction of solids that should be able to depth foul the filter. Of the four, the 4.0 M Na simulant has the largest population of fine sub-micrometer particles that could depth foul the filter. Similarly, the 6.0 M Na simulant contains a significant population of particles in the 1 to 10 µm size range that could depth foul the filter. The 5.6 M Na and 8.0 M Na simulants both show similar size distributions, with the primary population of particles falling in the 10 to 100 µm size range and above the pore size of the filter element. On the basis of size distribution alone, it might be concluded that both 4.0 M Na and 6.0 M Na simulants will have the greatest fouling proclivity of the four LAWPS simulants, but the baseline LAWPS simulant filtration testing discussed in Section 5 finds this not to be the case. Rather, both the 5.6 M Na and 8.0 M Na simulants show the greatest fouling proclivity of the four LAWPS simulants. This highlights that particle size measurements are a poor indictor of fouling proclivity [see Daniel et al. (2011)]. Instead, fouling proclivity appears to more strongly correlate with the mineral phase or chemistry of the solid materials.

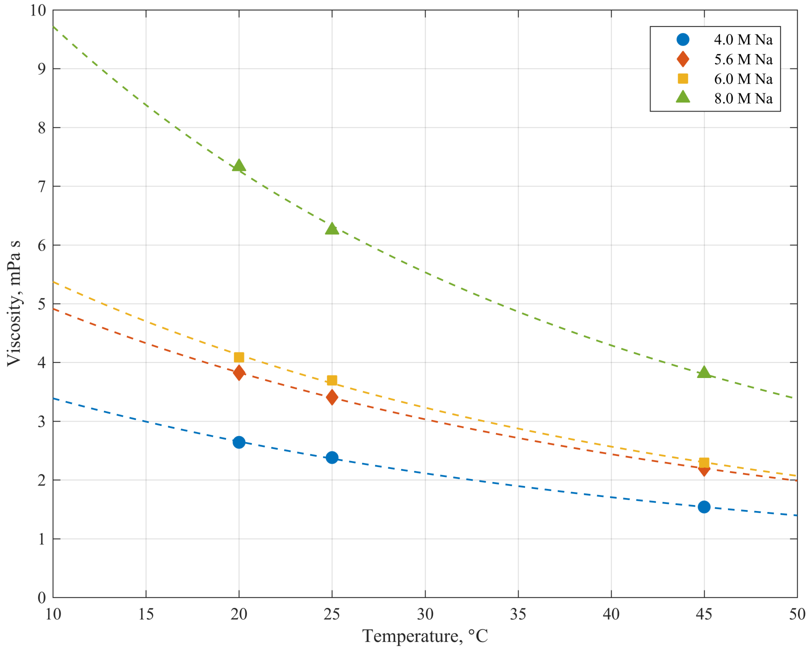

Figure 4.1. Measured viscosity as a function of temperature for the four baseline LAWPS simulants. Dashed lines represent a best-fit of the viscosity data to Eq. 3.4. Values for the Eq. 3.4 coefficients (μ_o and λ) are given in Table 4.4.

Table 4.4. Select physical properties of the four baseline LAWPS simulants.

Parameter	**Unit**	**Simulant**			
		4.0 M Na	5.6 M Na	6.0 M Na	8.0 M Na
		Density, Viscosity, and Solids Content			
Density$^{(a)}$	g mL^{-1}	1.19	1.26	1.27	1.34
Viscosity, 20 °C	mPa s	2.6	3.8	4.1	7.3
Viscosity, 25 °C	mPa s	2.4	3.4	3.7	6.3
Viscosity, 45 °C	mPa s	1.5	2.2	2.3	3.8
μ_o (Eq. 3.4)	mPa s	2.36	3.40	3.65	6.33
λ (Eq. 3.4)	K	2030	2070	2180	2420
Total Solids	wt%	22.7	30.1	31.3	39.1
Dissolved Solids	wt%	22.6	29.8	30.6	38.9
Undissolved Solids	wt%	0.14	0.45	1.1	0.24
		Particle Size Distribution$^{(b)}$			
d_1	µm	0.078	0.885	0.977	1.19
d_5	µm	0.347	3.41	1.67	6.58
d_{10}	µm	0.506	7.85	2.35	12.1
d_{25}	µm	0.770	15.8	5.03	19.6
d_{50}	µm	1.18	26.9	15.1	30.2
d_{75}	µm	1.83	42.1	58.2	45.2
d_{90}	µm	2.74	59.8	120	65.2
d_{95}	µm	3.64	71.8	160	84.1
d_{99}	µm	12.4	94.4	235	369

(a) Density measurement made at laboratory temperature (20 to 25 °C).
(b) PSD percentiles reported herein correspond to post-sonication measurements.

XRD analysis of select LAWPS simulants was performed in support of simulant development activities reported in Russell et al. (2017b). Here, XRD was only performed on simulants without added solids, namely the 6.0 M Na and 8.0 M Na simulants. For the 6.0 M Na simulant, the precipitated solids were determined to be a mixture of sodium aluminum phosphate and sodium fluorophosphate solids. Precipitated solids in the 8.0 M Na simulant were found to be a mixture of sodium oxalate, sodium phosphate, and sodium carbonate. Solids in the remaining simulants were assumed to largely be dominated by the added solids, namely boehmite and sodium oxalate in the 4.0 M Na and 5.6 M Na simulants, respectively. Supplemental XRD analysis of the 5.6 M Na simulant solids was performed to better understand fouling behavior of this simulant at elevated temperature during execution of Series 1 baseline filter performance tests. XRD analysis of the 5.6 M Na simulant solids before and after filter testing at 45 °C confirms the 5.6 M Na simulant solids are indeed dominated by sodium oxalate.

4.2 Fouling Simulants

As discussed above, eight fouling simulants were selected to support the Series 2 test objective to "evaluate various components and precipitates that will cause fouling" in LAWPS prototypic filter operations. A review of filtration literature relevant to Hanford waste treatment applications was performed to develop a basis of recommendation for up to eight select fouling simulants for use in CFF fouling tests.

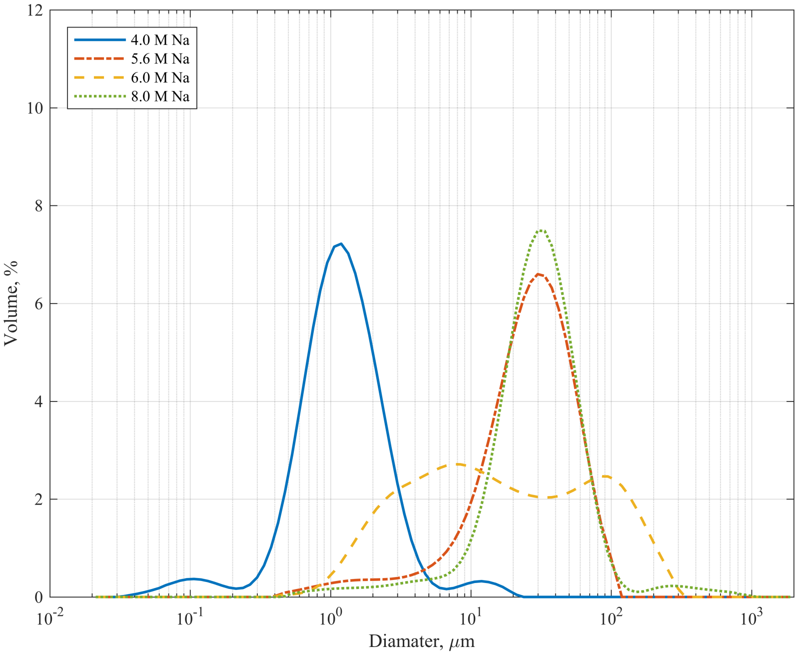

Figure 4.2. Measured PSDs for the four as-prepared LAWPS simulants. All size distribution measurements shown correspond to a "post-sonication" dispersion condition and should generally be representative of those that exist in highly-sheared conditions like CFF.

4.2.1 Review of Fouling Chemistries

In evaluating the waste chemistries that lead to fouling, it is necessary to evaluate the historical filtration literature of relevance to Hanford. To inform selection of fouling simulants, the studies considered must link filtration or filter fouling to mineral phases or salts present in the wastes tested. The number of relevant studies involving both filtration of saltcake wastes and mineral phase determination of saltcake waste solids is greatly limited, and as such, the number of studies included in the literature review is likewise limited. More general and complete literature reviews of Hanford waste filtration have been performed by Johnson and Duignan (2011) and Daniel et al. (2010b)

The filtration performance and mineral phase speciation of Hanford wastes have been historically studied by PNNL using LAWPS prototypic filtration equipment (Brooks et al. 1999, Edwards et al. 2009, Fiskum et al. 2008, 2009, Lumetta et al. 2009, Shimskey et al. 2009a,b, Snow et al. 2009). Several of these studies concern saltcake wastes or ad-mixtures of saltcake wastes and insoluble solids that may be of relevance to fouling in LAWPS filtration:

Brooks et al. (1999): evaluated the filtration performance of AW-101 supernate and its entrained solids using Hanford relevant filter media. Fluxes measured for AW-101 wastes ranged from 0.015 to 0.027 $\text{gpm}\,\text{ft}^{-2}$, depending on the filter operating conditions, after 1 hour of continuous filtration (following a filter backpulse). Although inductively coupled plasma analysis of the AW-101 entrained solids was conducted, it only provided an elemental composition of the waste rather than a salt and/or mineral oxide speciation. As such, the chemistry of the solids yielding the observed filter behavior cannot be directly inferred.

Fiskum et al. (2008): characterized Hanford REDOX sludge and S-saltcake wastes. Although no filtration testing was done, mineral phase analysis of the waste solids by XRD was performed and identified solids phases including gibbsite [$Al(OH)_3$], boehmite [AlOOH], sodium oxalate [$Na_2C_2O_4$], cancrinite [$Na_{7.92}(AlSiO_4)_6(NO_3)_{1.7}(H_2O)_{2.34}$], silicon dioxide [$SiO_2$], sodium uranium oxide [$Na_2U_2O_7$], and chromium oxide [e.g., CrO_3] (along with other various unknown and amorphous components).

Shimskey et al. (2009b): evaluated the filtration performance REDOX sludge, S-Saltcake waste, and their mixtures. Test results suggest that the filter flux of a 4 wt% S-saltcake slurry can be at least as low as approximately 0.005 $\mathrm{gpm\,ft^{-2}}$ at 20 psid at 25 °C after 12 hours of filtration. These results should be approached with caution, as the TMP was varied from 30 to 50 psid in the filtration period before that at 20 psid and may have resulted in increased fouling.

Lumetta et al. (2009): characterized and tested the filtration performance of $BiPO_4$ sludge and saltcake wastes. Mineral phase analysis of $BiPO_4$ saltcake waste insoluble solids by XRD suggests the presence of cancrinite [$Na_{7.92}(AlSiO_4)_6(NO_3)_{1.7}(H_2O)_{2.34}$], urancalcarite [$Ca(UO_2)_3CO_3(OH)(H_2O)_3$], dorfmanite [$Na_2HPO_4(H_2O)_2$], and gibbsite [$Al(OH)_3$]. Spectroscopic analysis of $BiPO_4$ also suggested the presence of amorphous iron phosphate solids [$FePO_4 \cdot nH_2O$]. Because of the limited actual waste solids inventory for both $BiPO_4$ sludge and saltcake, samples for both waste streams needed to be combined to meet the minimum waste volume requirements for the filtration test apparatus used in testing in Lumetta et al. (2009). As such, filtration performance results for $BiPO_4$ waste are available only for mixed sludge and saltcake solids. The results find that filter flux can be as low as 0.01 $\mathrm{gpm\,ft^{-2}}$ after approximately 14 hours of filtration at 25 °C.

To better isolate the fouling proclivity of saltcake waste solids, Daniel et al. (2011) and Schonewill et al. (2012) evaluated the long-term filtration performance of different soluble and insoluble solids representative of salt cake wastes. The insoluble solids considered included gibbsite [$Al(OH)_3$], boehmite [AlOOH], cancrinite [$Na_8[Al_6Si_6O_{24}](NO_3)_2 \cdot 4H_2O$], grimaldiite [hydrothermal CrOOH], and precipitated amorphous CrOOH (Scheele et al. 2009). The soluble components considered were sodium oxalate [$Na_2C_2O_4$] and sodium phosphate [Na_3PO_4]. To test the filtration performance of the saltcake solids, the insoluble solid fouling components were suspended in a 0.05 M $NaNO_3$ matrix, whereas the soluble solid fouling components were suspended in a concentrated electrolyte solution whose chemistry was modeled after Hanford saltcake liquids.

Of the insoluble solids tested in Daniel et al. (2011) and Schonewill et al. (2012), the cancrinite, amorphous FeOOH, grimaldiite, and amorphous CrOOH showed the greatest fouling proclivity, each yielding flux of 0.17, 0.18 , 0.18 , and 0.12 $\mathrm{gpm\,ft^{-2}}$, respectively, after 12 hours of continuous filtration. It should be noted that these fluxes derive from a 0.05 M $NaNO_3$ suspending phase, which has a viscosity of 0.892 mPa s at 25 °C that is nearly equivalent to that of water (0.890 mPa s). For a typical waste supernate viscosity of 3 mPa s, this translates to fluxes that span 0.035 to 0.050 $\mathrm{gpm\,ft^{-2}}$ (although this estimate neglects changes in how the solids structure at the filter surface at the high ionic strengths found in waste supernates). With respect to the other insoluble solids tested, both boehmite and gibbsite yielded fluxes above 0.7 $\mathrm{gpm\,ft^{-2}}$.

Nash et al. (2000) studied removal of ^{90}Sr from an Envelope C AN-107 simulant (with target Na concentration of approximately 7 M) using $Sr(NO)_3$ and metal nitrate additions. This study also evaluates the filterability of the resultant "decontaminated" waste simulant to understand the effect of the precipitates formed by ^{90}Sr removal (primarily strontium and metal hydroxides). Nash et al. (2000) first provide relevant background information regarding "worst-case" fouling simulants. In particular, the authors state:

> [Earlier FY1997] crossflow testing of a "worst case" Envelope C simulant with strontium and ferric precipitation found no filtrate production despite trials with both Mott 0.5 micron and a Graver pre-coated (effective 0.07 micron) filter tubes. The simulant was high in aluminum but also high in organics similar to AN-107 supernate. The test matrices were cancelled because of the difficulties. It was thought that filter fouling was not the problem but that the precipitate slurry formed impermeable cakes.

Nash et al. (2000) also outline and discuss the results related to the impact of decontamination precipitates on filterability of an AN-107 simulant obtained through direct CUF system testing. Of the numerous metal nitrate salts added, the combination of iron (III) and strontium nitrate salt precipitates in the caustic waste simulant was not filterable (confirming earlier tests reported in Nash and Siler (1997). Precipitates formed by addition of iron (III) nitrate alone yielded the lowest measurable flux ($\sim$0.0018 gpm ft^{-2} at $\sim$5 ft s^{-1} and $\sim$55 psi) of all salts tested. However, it is not clear that these decontamination studies, where precipitates are formed through direct addition of metal nitrate salts to caustic waste, are relevant for actual waste filtration processes that do not implement these contamination techniques.

From the discussion in the preceding paragraphs, several distinct fouling categories of interest arise: fouling from existing waste solids that are known bad actors, fouling from precipitated species, and fouling from complexes or organics. With respect to fouling from existing waste solids that are known bad actors, previous Hanford filtration testing identified the mineral phases of concern tested in Daniel et al. (2011) and Schonewill et al. (2012), namely the cancrinite, amorphous FeOOH, grimaldiite, and amorphous CrOOH, boehmite, and gibbsite. Of these phases, cancrinite, amorphous FeOOH, and amorphous CrOOH solids have sufficiently small primary particle sizes (on the order of 20 nm or less) to allow them to remain suspended through prolonged decant operations. For saltcake systems, the two solids of greatest relevance are cancrinite and amorphous FeOOH. The analytical characterizations associated with Fiskum et al. (2008), Shimskey et al. (2009b), and Lumetta et al. (2009) indicate other insoluble solid chemistries whose fouling proclivity has not been directly evaluated in isolation, namely sodium phosphate and iron phosphate.

Nash et al. (2000) highlight the potential of precipitates formed by changing waste chemistry to strongly foul the filter element and reduce filter performance. In Nash et al. (2000), planned waste treatment activities led to precipitation or the formation of complexes that created a significant (and total in some cases) reduction in filter flux. During development of the fouling test matrix, no direct chemical treatments, such as ^{90}Sr decontamination, are planned at LAWPS beyond chemical cleaning of the filters. It should be noted that LAWPS cleaning involves substantial changes in the chemistry of the filter working fluid (see Section 3.6.3). The fouling matrix need not directly consider precipitation under normal LAWPS cleaning conditions, as such testing is already captured in Series 1 and 2 testing.[1] Precipitation resulting from off-normal LAWPS cleaning conditions could be severe, particularly if 0.45 M HNO_3 were mixed directly with Hanford wastes. However, client and stakeholder discussions leading up to the development of the fouling matrix considered the drastic chemistry changes associated with off-normal events in cleaning as avoidable, and as such, of limited relevance to the fouling test matrix. Precipitation under normal filtration operations should be minimal as long as operating conditions (such as the temperature of the filter bank and slurry reservoir) are maintained and any waste mixing operations are done upstream of the filters. Based on the considerations outlined above, the fouling matrix more strongly emphasized study of solids and complexents that exist in

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[1] The impact of hold-up cannot be fully resolved by the current test program because 1) hold-up volumes (such as the permeate shell) are minimized in the CUF test apparatus and 2) simulant can be effectively drained from the system before cleaning.

the waste than on fouling interactions by solids that could potentially precipitate in the filter. During development of the nominal simulant, it was noted that the presence of sodium fluoride (NaF) in the nominal simulant led to the precipitation of sodium fluorophosphate crystals over LAWPS filtration-relevant time scales. This mechanism was proposed as one means of evaluating the impacts of slow precipitation on filtration.

The role organic materials play in filtration of Hanford wastes is poorly understood. Waste organics are typically only reported in terms of total organic carbon. Organic materials selected for simulants are typically chelating agents like EDTA and HEDTA (Delegard 1980) or low-molecular-weight salts such as sodium citrate or sodium format (Golcar et al. 2000). As discussed above, the presence of chelating agents during ^{90}Sr decontamination treatments resulted in significantly reduced flux. During scaled demonstrations of the WTP's filtration operations at the Pretreatment Engineering Platform, it was observed that initial CWFs were greatly reduced ($0.030\,\mathrm{gpm\,ft^{-2}}$ versus the expected $\sim 1.5\,\mathrm{gpm\,ft^{-2}}$) after addition of a commercial blue food dye to improve sensitivity of tank laser level sensors. Bench-scale filter testing of the dyed process water confirmed poor filtration results, and found that the initial permeate produced was colorless and only turned blue after a substantial lag time (Billing et al. 2009). The lag-time associated with the start of filtration and component break-through is of particular interest, as it indicates a molecular sieving capacity associated with the Mott Grade 0.1 filter (despite having a relatively large pore size of 1 to 3 µm). The food dye in question contained several organics, namely propylene glycol ($C_3H_8O_2$), FD&C Blue 1 ($C_{37}H_{34}N_2Na_2O_9S_3$), FD&C Red 40 ($C_{18}H_{14}N_2Na_2O_8S_2$), and propylparaben ($C_{10}H_{12}O_3$), that could have challenged filter performance. The influence of organics on filtration could derive from a number of physical mechanisms, including gel polarization, molecular adsorption onto and desorption from the filter surface, and osmotic effects (Belfort et al. 1994). The latter depends on the permeability of the filter with respect to the given organic; however, any ability of the filter to reject small organics that dissociate into several ions (such as the trisodium form of EDTA) could result in the generation of significant osmotic pressure, which in turn creates additional resistance to permeate flow.

Contact between organic molecules and the filter is generally expected to derive from organics that exist in the waste. However, LAWPS filters are used in conjunction with IX columns loaded with sRF resin. Planned LAWPS cleaning will use the IX elution fluid, a 0.45 M HNO_3 solution, to also clean the filter elements. It is possible that thermal, chemical, or radiolytic degradation of the resin material could shed organic material that will be carried to the filter during elution. Likewise, elution could also carry degraded resin particulate to the filter shell, where it could lodge into the filter pores. Given the reduced filter performance observed with the organics present in food dye (Billing et al. 2009), similar fouling could result from resin particulate and degradation by-products.

The SOW that governs filter fouling tests for the LAWPS integrated support testing program focuses primarily on waste "chemistries" detrimental to long-term filter performance. Although not explicitly stated, waste chemistry is interpreted to also encompass the concentration of insoluble solids fouling the filter. LAWPS waste feeds are expected to follow from waste settle and decant operations, and as such, should have solids inventories that are lower than those that have been previously evaluated in the controlled test studies cited in the preceding paragraphs. In earlier studies [e.g., Daniel et al. (2011)], similar levels of fouling have been observed at particle concentrations of ~ 0.1 vol% and ~ 5 vol%. The only period where solids concentration has an observable effect is during slurry dewatering operations at solids loadings greater than ~ 10 vol% (Daniel et al. 2010a). At sufficient high solids content, Hanford waste filter flux obeys a gel-polarization model such that:

$$j = k \ln\left(\frac{c}{c_g}\right) \tag{4.1}$$

where j is flux, k is constant, c is the volume fraction of solids in the suspension, and c_g is a characteristic limiting gel volume fraction of the suspension (called the gel-polarization volume fraction) that can be estimated through the centrifuged solids concentrations measured during routine physical characterization of the slurry (Peterson et al. 2007). Similar testing at low-solids concentrations has not been performed to establish if solids concentration impacts the overall rate of fouling or the general magnitude of filter flux during operational periods. CWF testing, such as that documented in Section 7 of Daniel et al. (2011) (see Figure 7.4), demonstrates evidence of fouling when working with fluids in which solids are at nominally trace levels. Although not directly related to testing at low-solids content, current filtration testing results suggest that low-solids content could render the filter more susceptible to irreversible fouling. The latter derives from tests where multiple backpulses were performed in rapid succession during filtration of high-level waste simulants. Here, repeated backpulsing resulted in a dramatic acceleration in the loss of filter performance (Peterson et al. 2007, Daniel et al. 2011). The authors of these studies postulated that the acceleration in fouling resulted from loss of a protective cake layer on the filter surface during backpulse events and that the selected backpulse frequency of one pulse per 30 minutes did not allow sufficient time for this protective layer to rebuild.[1] LAWPS stakeholders have postulated that the low waste solids concentrations expected in the LAWPS feed are not sufficient to engender rapid growth of a protective cake layer on LAWPS filters, and could therefore result in accelerated depth fouling of the filters. Given the dearth of well-controlled low-concentration filter tests and the concerns raised above, a low-concentration fouling testing was recommended to address the need to evaluate the possibility of enhanced fouling at LAWPS relevant solids concentrations.

4.2.2 Selection of Fouling Simulants

The limited review of fouling chemistries and fouling behaviors given in Section 4.2.1 provides guidance for the selection of fouling simulants for Series 2 testing. To aid in final selection, the Series 2 test objectives were recast into five simulant selection objectives. In particular, fouling simulants must be selected

1. to evaluate the efficiency of LAWPS cleaning at clearing “difficult-to-remove” solids that have collected on the filter element
2. to determine if there are solid chemistries that would make it difficult to maintain flux within the target production rate [0.0075 to 0.0320 $\mathrm{gpm\,ft^{-2}}$] over a filtration period of 120 hours
3. to observe the impact of reduced solids content on the sustainability of filter operations
4. to resolve the impact of organic molecules on the sustainability of filtration and cleaning operations
5. to evaluate the impact of solids precipitation (or change chemistry) during filtration operations on filter performance.

Table 3.3 presents the final Series 2 test matrix. A breakdown of how each proposed Series 2 fouling simulant satisfies these five objectives outlined above is given in Table 4.5. It should be noted that development of the Series 2 test matrix followed completion of the majority of Series 1 baseline performance tests. The

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[1] Indirect analysis of filter flux suggests, but does not fully support, formation of filter cakes during CFF of Hanford wastes (Daniel et al. 2011, Schonewill et al. 2015). However, post-test destructive imaging of used filter elements does not provide evidence for existence of a coherent filter cake.

results of Series 1 testing helped guide selection of fouling simulants. For this reason, selection justifications given in Table 4.5 sometimes refer to baseline testing results presented in Section 5 of this report.

Table 4.5. Basis for Series 2 fouling simulant selection.

Simulant Selection/Justification	Objectives Targeted
Sodium Oxalate: Baseline testing of the nominal LAWPS simulant (Section 5) has demonstrated that sodium oxalate does not substantially challenge filter operations under normal operating conditions (20 °C). Significant fouling occurred at elevated temperature (45 °C in bench-scale testing), but was attributed to precipitation of solids from the recycled permeate stream. Similar fouling was not reproducible in elevated nominal simulant testing at the ITF. All tests were done at a relatively high undissolved solids content of 0.45 wt%. Given the acceptable baseline filter performance, sodium oxalate was selected for reduced-concentration effect testing, with the final selected concentration being 0.001 wt%.	**3)** Evaluate filtration at low-solids content
Sodium Phosphate: The fouling proclivity of sodium phosphate has been previously demonstrated by Schonewill et al. (2012) and indicates greater fouling potential than sodium oxalate. LAWPS relevant feeds are rich in sodium phosphate, and evaluation of filtration and flux recovery performance with sodium phosphate solids would provide operating confidence of LAWPS filtration operations.	**1)** Evaluate LAWPS cleaning efficacy **2)** Evaluate permeate production rates
Iron Oxyhydroxide: Extensive testing of amorphous iron oxides and oxyhydroxides has demonstrated significant fouling proclivity. Trace amounts of amorphous iron oxides are expected in LAWPS feeds. Standard laboratory cleaning with nitric and oxalic acids removes iron solids from sintered stainless steel filters, but similar removal has not been demonstrated with LAWPS cleaning.	**1)** Evaluate LAWPS cleaning efficacy
Iron Phosphate: Lumetta et al. (2009) identified iron phosphate [$FePO_4$] in the bismuth phosphate sludge and saltcake wastes. Although latter efforts (Peterson et al. 2016) characterized the observed phosphate mineral as a mixed iron-bismuth phosphate compound [$Fe_{3.7}Bi(PO_4)_{4.7}$], study of the fouling proclivity of iron phosphate is of interest as it is a compound observed in bismuth phosphate waste that showed fluxes that could potentially challenge the LAWPS target range [$\sim$0.01 gpm ft^{-2} at 20 psid per Figure 5.9 in Lumetta et al. (2009)] and whose effect on filtration has not been tested in isolation. Furthermore, iron phosphate should nominally react with sodium hydroxide in the waste to form iron oxide compounds: $FePO_4 + 3\,NaOH \longrightarrow Fe(OH)_3 + Na_3PO_4$. This reaction provides opportunity to determine the impact of in-process changes in solids chemistry on filtration.	**1)** Evaluate LAWPS cleaning efficacy **2)** Evaluate permeate production rates **5)** Evaluate precipitation/changing chemistry

Table 4.5. Basis for Series 2 fouling simulant selection (cont'd).

Simulant Selection/Justification	Objectives Targeted
Cancrinite: Filtration testing reported in Daniel et al. (2011) and Schonewill et al. (2012) demonstrates cancrinite exhibits significant fouling proclivity that could challenge LAWPS operations. Trace amounts of cancrinite solids are expected in LAWPS feeds. Standard laboratory cleaning with nitric and oxalic acids removes cancrinite solids from sintered stainless steel filters, but similar removal has not been demonstrated with LAWPS cleaning.	**1)** Evaluate LAWPS cleaning efficacy
HEDTA: Nash et al. (2000) provide evidence suggesting organic complexants play a role in fouling of filters. These complexants, along with several other organics like sodium citrate, are suggested components in a number of Hanford waste relevant simulants (Golcar et al. 2000). Although HEDTA ($C_{10}H_{18}N_2O_7$) was one of many potential organic materials from the referenced studies, it was selected for its role as a waste complexant, for its relatively low molecular weight ($\sim$280 $g\,mol^{-1}$), and because it has four hydroxyl groups that dissociate at high pH. Given both the dissociative behavior and low molecular weight of HEDTA, its addition to the waste simulant should generate additional resistance to filtration through osmotic pressure should the filter retain a portion of the HEDTA. To avoid consumption of free hydroxide, the sodium form of HEDTA was selected for testing.	**4)** Evaluate organic and complexant fouling
Degraded sRF Resin: As discussed on the preceding pages, sRF resin degradation products may contaminate the filter. The sRF resin test was selected to evaluate the impact, if any, of the resin degradation products on filtration (both soluble and particulate products) and the ability of LAWPS cleaning to recover any loss in filter performance. Degradation is effected before testing by contact with 0.45 M HNO_3 for 24 hours. It should be noted that sRF filter contamination in LAWPS will occur on the permeate-side of the filter during filter backflow operations. For simplicity, bench-scale CFF testing introduced the degraded sRF resin beads on the slurry-side of the filter. As such, bench-scale resin fouling results are indicative and not truly representative of sRF fouling that might occur in full-scale LAWPS operations. However, attrition of resin by the CUF pump and increased fouling time (120 hours for baseline testing versus $\sim$18 hours of LAWPS cleaning backflow) are expected to lead to increased resin fouling, and as such, bench-scale testing is assumed conservative with respect to the potential impact of resin-filter contact.	**4)** Evaluate organic and complexant fouling
Sodium Fluoride: Testing sought to evaluate the impact of changing particle chemistry and solid precipitation on filtration. This was accomplished by addition of sodium fluoride to a modified version of the 5.6 M Na simulant. LAWPS simulant development efforts (Russell et al. 2017b) indicate the addition of sodium fluoride will lead to the slow precipitation of sodium fluorophosphate (Na_2PFO_3).	**1)** Evaluate LAWPS cleaning efficacy **2)** Evaluate permeate production rates **5)** Evaluate precipitation/changing chemistry

With one exception, all CFF fouling simulant tests employed a modified version of the 5.6 M baseline LAWPS simulant, termed the modified sodium simulant (MSS), in which the concentration of sodium oxalate was reduced to limit the fraction of undissolved solids not associated with the "foulant" in the final filtration test suspension. In preparing the MSS, the 5.6 M simulant recipe listed in Table 4.3 was modified by reducing the initial amount of sodium oxalate added to the solution by 20 % (from 1.36 $\mathrm{g\,kg^{-1}}$ to 1.09 $\mathrm{g\,kg^{-1}}$) and completely forgoing post-makeup sodium oxalate addition. The only test not using the MSS was the sRF resin evaluation. Here, filtration testing was conducted in 0.45 M HNO_3 to simulate the elution fluid. The make-up recipe and physical properties of the fouling simulant suspending phases are given in Tables 4.6 and 4.7, respectively. The simulant materials listed in Table 4.6 are sourced from the same suppliers as those used for make-up of the four baseline LAWPS simulants. The only new chemical used in baseline simulant make-up is HNO_3, which was purchased as a 70 wt% stock solution from Sigma-Aldrich, Inc. (St. Louis, MO).

Addition of fouling simulants generally targeted an undissolved solids test concentration of 0.1 wt%. Exceptions include:

- low concentration tests with sodium oxalate at 0.001 wt% undissolved solids
- tests with soluble solids, including HEDTA (sodium form) and sodium fluoride at dissolved solids concentrations of 0.1 wt% and 6 $\mathrm{g\,L^{-1}}$, respectively

As originally planned, all fouling tests were to be conducted at 20 °C except for tests with sodium phosphate and sodium fluoride, which targeted 45 °C to drive solubility of the fouling materials.

As discussed in Section 3, several of the fouling simulants proposed for Series 2 testing, while present in actual wastes or waste simulants that exhibit poor filtration performance, had not been tested in isolation. In

Table 4.6. Make-up recipes for the two fouling simulant suspending phases.

Component	Formula Weight ($\mathrm{g\,mol^{-1}}$)	Composition ($\mathrm{g\,kg^{-1}}$)$^{(a)}$	
		MSS	0.45 M HNO_3
Primary Supernate Composition			
$Al(NO_3)_3 \cdot 9H_2O$	375.13	49.82	– –
NaOH (50% solution, w/w)	40.00	132.73	– –
$CsNO_3$	194.91	0.016	– –
KCl	74.55	7.28	– –
Na_2SO_4	142.04	7.51	– –
$NaNO_2$	69.00	56.30	– –
$NaNO_3$	84.99	87.17	– –
$Na_3PO_4 \cdot 12H_2O$	380.12	13.14	– –
$Na_2CO_3 \cdot H_2O$	124.00	46.33	– –
$Na_2C_2O_4$	134.00	1.09	– –
HNO_3 (70% solution, w/w)	63.01	– –	39.75
Water, Deionized	18.02	598.35	960.25

(a) Basis is gram component per kilogram of simulant supernate solution.

Table 4.7. Physical properties of the two fouling simulant suspending phases, MSS and 0.45 M HNO_3. The physical properties of the 5.6 M Na are included for reference. As the two suspending phases used in Series 2 testing do not nominally have any suspended solids, PSD percentiles are not included in the current table (cf. Table 4.4).

Parameter	**Unit**	**Simulant**		
		5.6 M Na	MSS	0.45 M HNO_3
Density[(a)]	$\mathrm{g\,mL^{-1}}$	1.26	1.26	1.01
Viscosity, 20 °C	mPa s	3.8	3.9	1.0
Viscosity, 25 °C	mPa s	3.4	3.4	0.90
Viscosity, 45 °C	mPa s	2.2	2.1	0.63
μ_o (Eq. 3.4)	mPa s	3.40	3.40	– –[(b)]
λ (Eq. 3.4)	K	2070	2290	– –[(b)]
Total Solids	wt%	30.1	29.7	2.80
Dissolved Solids	wt%	29.8	29.7 [(c)]	2.80
Undissolved Solids	wt%	0.45	0 [(c)]	0

(a) Density measurement made at laboratory temperature (20 to 25 °C).
(b) The viscosity of 0.45 M HNO_3 is not nominally different from that of water. Temperature corrections were made using a water viscosity correlation proposed in Laliberté (2007) (see Eq. 3.9).
(c) Trace undissolved solid precipitates were observed in the MSS but were too few to accurately quantify through total and undissolved solids analysis.

cases where the selected fouling agents did not lead to filter performance that was substantially below that of the nominal 5.6 M Na LAWPS simulant, in-process test modifications were implemented to increase fouling either through increased fouling agent concentration (via direct addition or precipitation) or synergistic effects with other fouling agents. These test modifications are summarized in Table 3.4 and will not be reproduced in this section.

The majority of the selected fouling simulants were sourced from existing chemical stocks at the laboratory or from commercial vendors. Sodium oxalate was sourced from existing stock originally purchased from Noah Technologies, Inc. (San Antonio, TX), and is the same material used in make-up of the nominal sodium simulant. Sodium phosphate (as $Na_3PO_4 \cdot 12\,H_2O$), iron phosphate (as $FePO_4 \cdot 4\,H_2O$), and HEDTA (sodium form) came from a mix of existing stock and new stock purchased from Sigma-Aldrich, Inc. (St. Louis, MO). Sodium fluoride was sourced from existing stock (Mallinckrodt Chemical Works, St. Louis, MO). The sRF resin used in testing was Spheromers® RF 380 solid mono-sized resorcinol formaldehyde microbeads (Microbeads AS, Skedsmokorset, Norway). The remaining simulants, iron oxide and cancrinite, were generated through direct synthesis as outlined in the two paragraphs that follow.

Amorphous Iron(III) Oxide (Fe_2O_3): An amorphous iron(III) oxide was synthesized by hydrolysis of a solution of iron(III) nitrate [$Fe(NO_3)_3$] using an adapted version of the iron-rich sludge recipe reported in Appendix A of Russell et al. (2009). Specifically, synthesis was accomplished by

1. dissolving 128.1 g of iron(III) nitrate nonahydrate [$Fe(NO_3)_3 \cdot 9\,H_2O$] in 300 g of water

2. adding sufficient 8 M NaOH solution to increase the solution pH to 10 to 11 (approximately 190 g)

3. mixing the solution for 1 hour

4. adding an additional 8 M NaOH to return the pH to 10 if it is lower than 10 after mixing

This procedure yields approximately 30 g of solids. The resulting suspension was centrifuged and washed with MSS and then added to 0.1 wt% to a larger batch of MSS for testing. The 8 M NaOH solution used in step 2 was prepared by diluting a 50 wt% NaOH stock solution. All chemicals used for Fe_2O_3 synthesis were purchased from Sigma-Aldrich, Inc. (St. Louis, MO). It should be noted that the chemical form of this component has been listed as FeOOH (iron-oxyhydroxide) in previous PNNL simulant development and filtration reports. However, analysis of the material synthesized for the current filtration campaign by XRD indicates the material is amorphous iron oxide.

Cancrinite: A nitrate form of cancrinite was synthesized using the method described in Liu et al. (2005). Synthesis was accomplished by:

1. dissolving 11.44 g sodium silicate ($Na_2SiO_3 \cdot 9\,H_2O$, Fisher Chemicals, Fair Lawn, NJ), 40.76 g sodium nitrate ($NaNO_3$, Sigma-Aldrich, St. Louis, MO), and 8.72 g sodium hydroxide (NaOH, Sigma-Aldrich, St. Louis, MO) in approximately 70 mL of deionized water

2. separately adding 3.28 g of sodium aluminate ($Al_2O_3 \cdot Na_2O$, EM Science, Gibbstown, NJ) in 10 mL of deionized water and collecting the permeate produced by filtering the resultant suspension through a 0.45 μm syringe filter

3. combining the solution and permeate from steps 1 and 2, respectively

4. heating the suspension resulting from step 3 in an oven at 90 °C for 12 to 16 hours

The resulting suspension was cooled and washed several times with MSS and then added to 0.1 wt% to a larger batch of MSS for testing.

For CFF testing, the prepared simulants were generally added to the CFF slurry reservoir (pre-filled with 15 L of MSS) without further modification shortly before or after the start of filtration testing. The one exception to this approach involved sRF resin testing. Planned filtration testing with the sRF resin called for degradation of the resin before testing. Degradation was accomplished by loading a pre-determined mass of resin (sufficient to yield an $\sim$0.1 wt% sRF suspension when added to 15 L of test suspension) into a 200 mL Nalgene bottled filled with approximately 150 mL of 0.45 M HNO_3. The $\sim$150 mL sRF resin was then placed in a laboratory oven and heated at 50 °C for 24 hours. The final treated sRF suspension was then added to a CUF slurry reservoir that had been pre-filled with 15 L of 0.45 M HNO_3.

4.2.3 Fouling Simulant Physical Properties

The physical properties of the eight fouling simulants and the MSS suspending phase were characterized on a limited basis. With exception of HEDTA (which is completely soluble in the MSS suspending phase), the post-test simulant solids samples are shown in Figure 4.3. It should be noted that only the measurements corresponding to the test solids collected at the end of testing are shown to limit the number of results shown and reduce graph clutter. A complete set of measurements is included in Appendix C of this report.

The fouling simulant PSDs in Figure 4.3 show a broad range of PSD, ranging from the lower limit of detection of the laser diffraction analyzer used (20 nm for iron phosphate solids in MSS) up to approximately 800 μm (for sodium phosphate solids). As the suspending phase is a high-ionic-strength, complex electrolyte solution that was not (and likely cannot be easily) modified to allow complete dispersion of the solids materials tested, the PSDs in Figure 4.3 likely correspond to a mix of primary and aggregated particles similar to those in the CFF test system. The PSD measurements indicate that of the solids tested, sodium phosphate crystals and/or agglomerates were the largest, while iron phosphate crystals/agglomerates were the smallest. However, all solids present a substantial volume fraction of particles that spans and falls below the estimated Mott Grade 0.1 filter pore size of 1 to 3 μm. As such, all fouling simulants have the potential to interact with and foul the filter element. That being said, PSD is generally not a meaningful measure of fouling proclivity of solids during CFF [see Daniel et al. (2011)]. Tabular PSD data are shown in Table 4.8.

The temperature dependencies of MSS and 0.45 M HNO_3 viscosities are shown in Figure 4.4. As expected, the MSS viscosity values are virtually equivalent to those of the 5.6 M Na simulant. However, viscosity measurement uncertainty (expected to be on the order of ±0.2 mPa s) leads to slightly different viscosity-temperature correlation parameters, μ_o and λ, upon application of Eq. 3.4. These differences should not lead to significantly different temperature corrections over 20 to 45 °C. The viscosity of 0.45 M HNO_3 is not substantially different than that of water, and as such, Eq. 3.9, which is valid over 0 to 100 °C, is used in lieu of a direct fit of 0.45 M HNO_3 viscosity data.

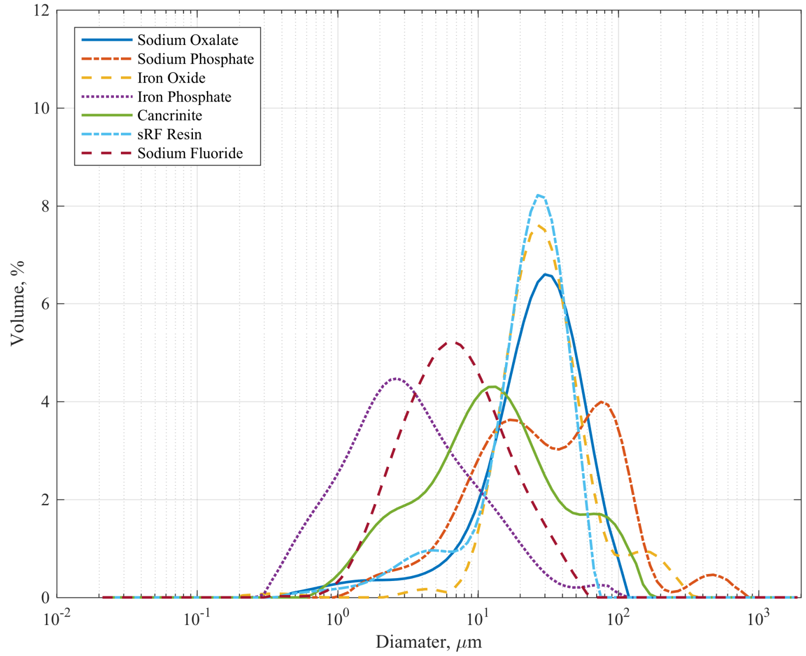

Figure 4.3. Measured PSDs for the fouling solids collected at the end of each Series 2 fouling test. All size distribution measurements shown correspond to a “post-sonication” dispersion condition and should generally be representative of those that exist in highly-sheared conditions like CFF. HEDTA is excluded as it is entirely soluble in the MSS suspending phase. All solid PSDs were measured in their source suspending phase. The suspending phase was MSS for all except the sRF resin, which was suspended in 0.45 M HNO_3.

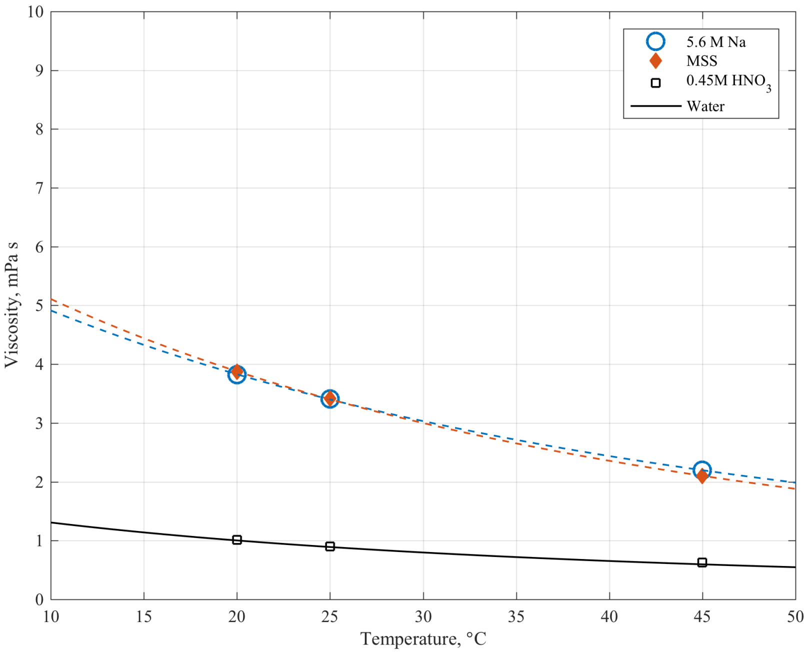

Figure 4.4. Viscosity of the 0.45 M HNO_3 and MSS as a function of temperature. The viscosity of the 5.6 M Na simulant is included for reference, and as expected, is virtually identical to that of the MSS. For the MSS and 5.6 M Na simulant, dashed lines represent a best-fit of the viscosity data to Eq. 3.4. Values for the Eq. 3.4 coefficients are given in Table 4.7. The solid black line corresponds to predictions of water viscosity using Eq. 3.9 and provides a reasonably accurate description for the the viscosity of 0.45 M HNO_3 (black squares).

XRD analysis of the fouling solids was performed to confirm expected mineral phase of stock or synthesized materials and expected mineral phase changes upon addition to simulant. In particular, XRD analysis was aimed primarily at confirming the iron oxide and cancrinite synthesis methods and verifying the expected changes in iron phosphate chemistry upon addition to the MSS suspending phase. XRD analysis of iron oxide solids prepared using the method outlined in Russell et al. (2009) indicated the resultant iron oxide particles were largely amorphous, producing few interpretable peaks on the XRD scan, with primary particle sizes on the order of 1 nm. Likewise, XRD analysis of the solids resulting from cancrinite preparation per Liu et al. (2005) were indeed cancrinite. Evaluation of the stock iron phosphate powder purchased from Sigma-Aldrich, Inc. (St. Louis, MO) confirmed iron phosphate. XRD analysis of the iron phosphate solids after addition to the MSS and extensive CFF testing found no iron phosphate and crystalline solids representative of the MSS simulant. No iron oxide solids were identified, which is characteristic of amorphous iron oxide and consistent with the XRD scan for solids generated using the method of Russell et al. (2009).

Table 4.8. Select PSD percentiles for fouling simulants with insoluble solids. All test samples were collected at the end of each Series 2 fouling test. All size distribution measurements shown correspond to a “post-sonication” dispersion condition and should generally be representative of those that exist in highly-sheared conditions like CFF. HEDTA is excluded as it is entirely soluble in the MSS suspending phase. All solid PSDs were measured in their source suspending phase. The suspending phase was MSS for all except the sRF resin, which was suspended in 0.45 M HNO_3.

Parameter	Unit	Sodium Oxalate	Sodium Phosphate	Iron Oxide	Iron Phosphate	Cancrinite	sRF Resin	Sodium Fluoride
d_1	µm	0.885	1.76	3.69	0.403	1.03	1.05	1.12
d_5	µm	3.41	3.92	10.9	0.604	1.72	3.16	1.75
d_{10}	µm	7.85	6.51	13.6	0.825	2.47	5.90	2.28
d_{25}	µm	15.8	12.8	19.3	1.52	5.63	15.2	3.73
d_{50}	µm	26.9	29.3	28.7	3.01	12.3	24.0	6.71
d_{75}	µm	42.1	70.0	44.1	6.55	26.4	34.3	12.3
d_{90}	µm	59.8	112	74.0	13.8	62.3	44.8	21.4
d_{95}	µm	71.8	147	134	20.9	87.6	51.1	29.0
d_{99}	µm	94.4	520	230	60.5	127	61.0	43.8

5.0 Baseline LAWPS Filtration Results

The filtration behavior of the four LAWPS simulants was tested in accordance with the methods discussed in Section 3 of this report. Each test allowed assessment of the fouling proclivity of the simulant and the effectiveness of flux recovery methods, namely backpulsing and LAWPS cleaning. Figure 5.1 provides an example of a typical CFF test result, showing filter flux measured as a function of time, and highlights the segmented nature of the test. Each test consists of six separate segments:

Segment A: 120 hr of continuous filtration at constant TMP (i.e., 20 psid)

Segment B: 24 hr of continuous filtration at constant TMP following a single backpulse operation

Segment C: backpulsed operations (one backpulse every 30 min for 8 hr) followed by a 16 hr period of continuous filtration at constant TMP

Segment D: LAWPS prototypic cleaning with displacement by 0.1 M NaOH, rinsing with process water, cleaning with 0.45 M HNO_3 (nitric acid), and final rinsing with process water

Segment E: 24 hr of continuous filtration at constant TMP following LAWPS cleaning

Segment F: Post-test CWF measurements and standardized chemical cleaning operations with 2 M HNO_3 (nitric acid) and 0.5 M $H_2C_2O_4$ (oxalic acid) solutions

Evaluation of the filter performance focuses on only a limited portion of data from the segments noted above. First, fouling proclivity will be assessed primarily through Segment A data, as this represents the operational period most relevant to sustained LAWPS filter operations. Although the ultimate extent of fouling can be better assessed through flux data following multiple consecutive backpulses (Segment C), these operations are not typical of LAWPS and serve primarily to render the filter more difficult to clean in the current testing. Next, flux recovery operation performance is evaluated by comparing flux data from Segments B and F to that in Segment A. Finally, the impact of temperature and the pressure profile (constant versus stepped pressure) on filtration performance will be evaluated using select fouling and cleaning performance data. For additional data generated from the Series 1 tests, see Appendix A, which includes average data over the initial and final hour of various operating periods and a plot of the uncorrected flux for the entire test.

5.1 Fouling Proclivity

Filter flux as a function of time is shown for the four baseline LAWPS simulants, namely the 4.0 M, 5.6 M, 6.0 M, and 8.0 M Na simulants, in Figure 5.2 for the initial 120-hour period of filter operation. All four tests shown in Figure 5.2 were conducted at a constant TMP of 20 psi and an AV and temperature of 14.7 $\mathrm{ft\,s^{-1}}$ and 20 °C, respectively. As expected, flux declines over the 120-hour filtration period for all baseline simulants, with the most significant decline occurring during the first 24 hours of filtration. Step-changes in flux like that at approximately 45 hours for the 4.0 M Na simulant result from corresponding step-changes in the TMP to correct for pressure drift and do not result from fouling (as is apparent in the normalized flux result shown in Figure 5.4). In terms of uncorrected flux throughout the majority of 120-hour testing, the filtration performance is best for the 4.0 M Na simulant (0.09 $\mathrm{gpm\,ft^{-2}}$) and worst for the 8.0 M Na simulant (0.025 $\mathrm{gpm\,ft^{-2}}$). An equivalent conclusion is reached by evaluating the total volume of permeate produced during filtration of each of the four simulants (see Figure 5.3). That the 4.0 M and 8.0 M Na simulants fall

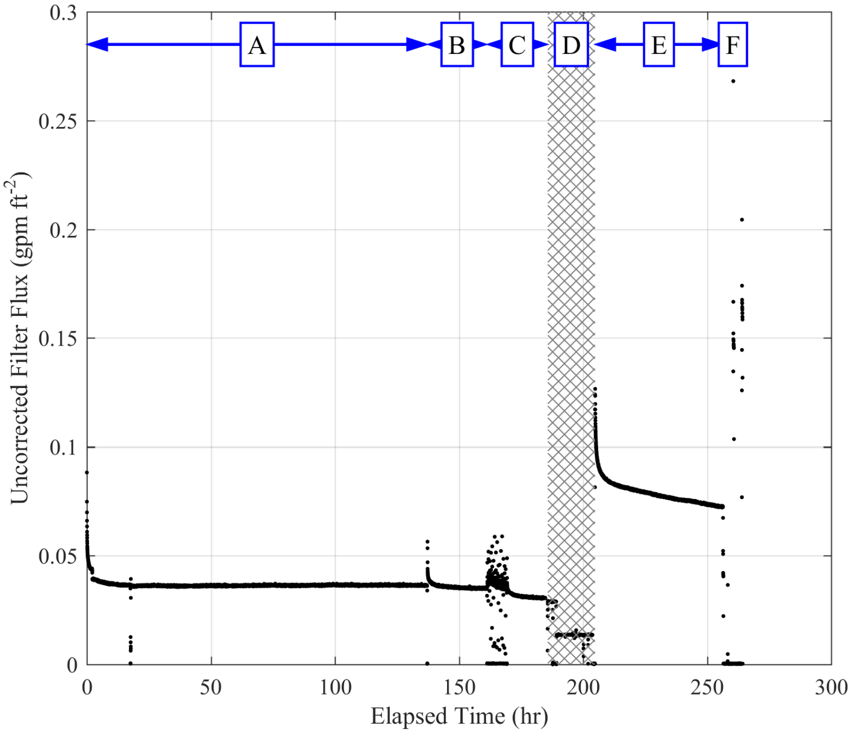

Figure 5.1. Example of a typical CFF test result. The figure shows uncorrected flux *j* as a function of time for Series 1 Test 1 (LPS-T2S1-NC20-01), which is a performance test with a 5.6 M (nominal) Na simulant at a constant TMP of 20 psid and 20 °C. Measured flux in the shaded region corresponds to cleaning backflush flow, and is not a valid measure of filter flux.

at the bounds of performance is reasonable, given the influence of viscosity on the filtration performance based on Darcy's law. Of all the simulants tested, only the flux measured for 8.0 M Na simulant falls within the LAWPS flux target range of 0.0075 to 0.032 gpm ft^{-2} at 20 psi. All other simulants exhibit 120-hour fluxes that fall above the upper bound of 0.032 gpm ft^{-2}. As such, maintaining the target flux ranges at the LAWPS facility should be feasible for wastes that filter similarly to the four baseline LAWPS simulants over operation ranges of up to 120 hours (if not longer). It should be noted that the exact rate of flux decline depends on the applied pressure profile (see Section 5.3); given that fluxes generally fall above 0.032 gpm ft^{-2}, operational pressures lower than 20 psi can be used to achieve fluxes within the range, which will result in slower flux declines over the baseline 120-hour period.

The flux measurements shown in Figure 5.2 are not normalized and include the direct effects to TMP, suspending phase viscosity, temperature, and filter media. Although filter fouling is path-dependent, the flux dependence on operating parameters can be minimized through normalization of flux by Eq. 3.7. Doing so provides a more direct measurement of the fouling proclivity of solids within the simulant. Ideally, normalized flux will range between 0 and 1 and represents the ratio of measured flux to the maximum that can be achieved for the test material. Normalized flux is dimensionless and has no associated units. Figure 5.4 recasts the baseline flux data for the four LAWPS simulants in terms of normalized flux η; in these terms, the 5.6 M Na solids (primarily sodium oxalate) represent the worst foulant of all precipitated and added solids tested in Series 1 at a normalized flux of ∼0.15. That the 8.0 M Na simulant is not the most difficult to filter on a normalized flux basis indicates that the low 8.0 M Na simulant flux derives in

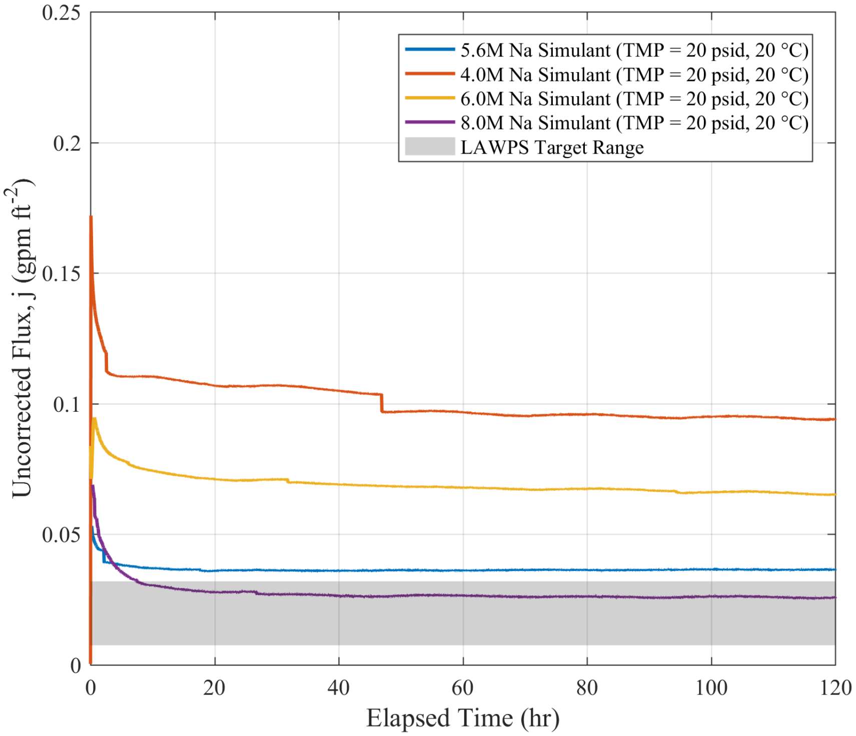

Figure 5.2. Uncorrected flux measured for the four LAWPS simulants during the initial (120-hour) baseline filtration period. All tests were run at nominal conditions (AV, 14.7 ft s^{-1}; TMP, 20 psi; temperature, 20 °C). Note: The shaded gray region indicates target flux range of LAWPS operations (0.0075 to 0.032 gpm ft^{-2}).

part from its high dissolved solids content and high viscosity (relative to the three other simulants). Given the lower level of fouling observed in tests with the 6.0 M and 8.0 M Na simulants, it can be postulated that the fouling performance of the 5.6 M Na simulant results from interactions between the filter and the added sodium oxalate solids. Similar fouling might be expected of the colloidal boehmite (Nabaltec APRYAL AOH 60) added to the 4.0 M Na simulant; however, previous testing (Daniel et al. 2011, Schonewill et al. 2015) has demonstrated that addition of colloidal boehmite does not significantly increase the rate or degree of crossflow filter fouling on the Mott filters used in current testing. It could be postulated that the addition of boehmite reduces the filtration performance of the 4.0 M Na simulant (based on the relative magnitudes of the 6.0 M and 8.0 M Na simulants, neither of which had added solids). However, the impact of solids addition cannot be directly assessed without equivalent tests of the 4.0 M and 5.6 M Na simulants without added boehmite or oxalate solids.

The normalized flux results shown in Figure 5.4 provide a cleaner measure of the rate of filter fouling for Series 1 simulants, as the impact of step changes in TMP and temperature are almost entirely removed. The two simulants without added solids, namely the 6.0 M and 8.0 M Na simulants, show slower rates of flux decline relative to the two other Series 1 simulants, where flux decline is rapid over the first 5 hours of testing. Beyond the rapid decline seen in all simulants during the first 24 hours, there appears to be a slow, persistent decline over the remainder of the baseline 120-hour period for all simulants except the 5.6 M Na simulant. This decline is typical of waste and waste simulant CFF performance [see Daniel et al. (2010b, 2011)]. In contrast, the flux measured for 5.6 M Na simulant appears to reach a steady-state value of ~0.15

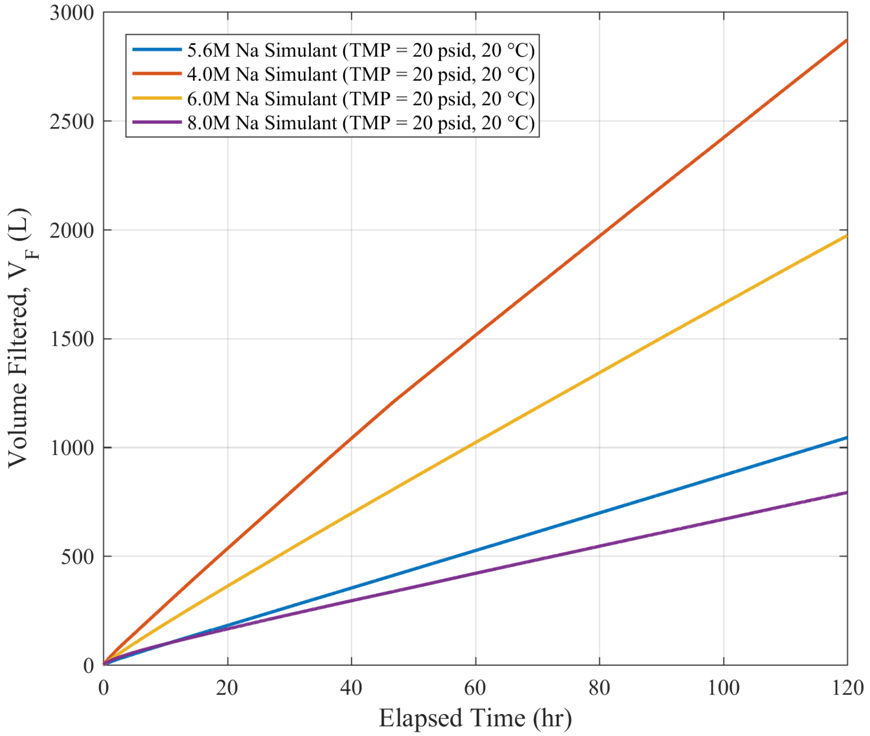

Figure 5.3. Volume of filtration produced as a function of time for the four LAWPS simulants during the initial (120-hour) baseline filtration period.

after 24 hours of filtration. Similar achievement of a steady-state flux has not been observed in previous testing with sodium-oxalate-rich saltcake simulants [cf. Schonewill et al. (2012)]; as such, it is unclear if the current test behavior is representative of the 5.6 M Na itself or from operational history effects during testing of this material. Further evaluation of the 5.6 M Na simulant at reduced solids content, elevated temperature, and different pressures does not provide results that clearly resolve this question. Although anomalous, the flux rate change behavior observed in 5.6 M Na simulant testing at 20 °C and 20 psi does not raise long-term performance concerns given that the un-normalized flux falls well above the target LAWPS operational flux range.

The discussions in the preceding paragraphs concern time-rate data and do not provide a single, easily reference-able measurement of fouling performance. To provide such metrics, the flux (both uncorrected and normalized) was calculated for the initial and final hour of the 120-hour baseline filter performance period. Graphical summaries of these averages are provided in Figures 5.5 and 5.6 for uncorrected and normalized flux, respectively. Likewise, tabular results are summarized in Table 5.1. Tabular and graphical summaries of the initial and final flux averages highlight the general flux magnitudes discussed in the preceding paragraphs, namely that the 8.0 M Na simulant exhibits the lowest flux at the end of the 120-hour period (on an uncorrected flux basis), while the 5.6 M Na simulant exhibits the greatest fouling proclivity (based on normalized flux assessments). The graphical summaries, Figures 5.5 and 5.6, demonstrate the change in flux over the 120-hour period more clearly than that shown in the time series graphs (Figures 5.2 and 5.4). On a normalized flux basis, the 8.0 M Na simulant shows the greatest change in flux, while both the 4.0 M Na and 6.0 M Na simulants show similar, slightly lower changes in flux between the initial and final hour of testing. The 5.6 M Na simulant is unique in that it shows very little difference in normalized

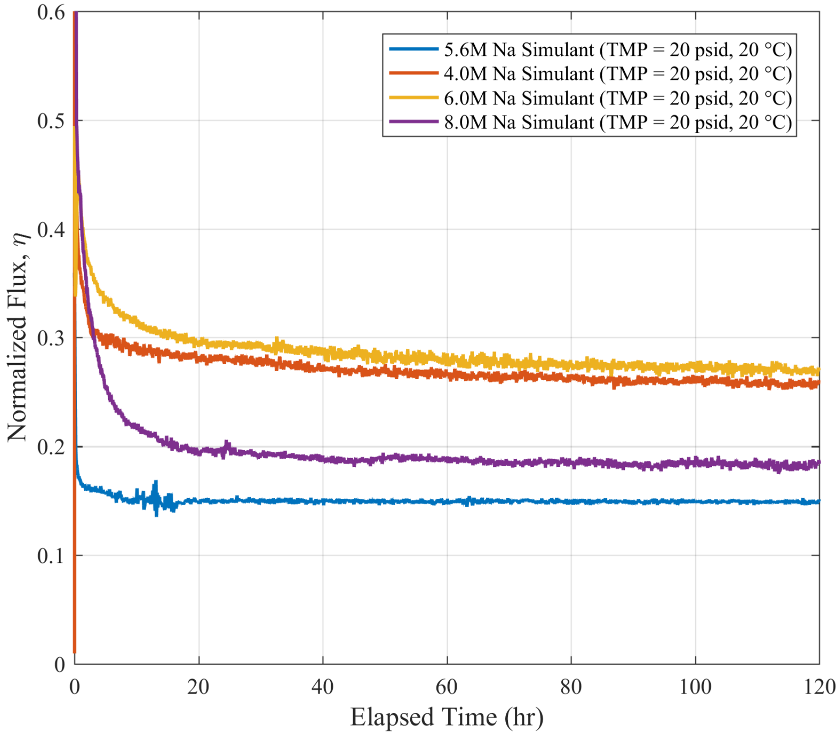

Figure 5.4. Normalized flux η for the four LAWPS simulants during the initial (120-hour) baseline filtration period. Normalization is based on Eq. 3.7.

flux between the initial and final test periods. This behavior results from the rapid decline and approach to steady state flux observed in Figure 5.4. A summary of percent decline in flux is provided in Table 5.2. Overall, the results evidence no clear correlation between initial flux and the final fouling proclivity of the simulant. Indeed, the highest fouling proclivity is observed for the two LAWPS simulants with both the lowest and highest initial hour fluxes (the 5.6 M Na and 8.0 M Na simulants, respectively).

Overall, the fouling proclivity of the four LAWPS simulants is not substantial enough to raise concerns that the LAWPS operational fluxes could not be maintained over a 120-hour period if the filters were clean at the start of testing. Although not tested directly, the results suggest that acceptable performance could continue for filtration periods longer than 120 hours and, as discussed later in this report, could be further

Table 5.1. Uncorrected and normalized flux for the four LAWPS simulants averaged over the initial and final hour of filtration in the 120-hour baseline performance period.

Test	**Description**	Uncorrected Flux j, gpm ft^{-2}		Normalized Flux η	
		Initial Hour	**Final Hour**	**Initial Hour**	**Final Hour**
7	4.0 M Na (20 psi, 20 °C)	0.14(1)	0.0940(2)	0.40(3)	0.258(2)
1	5.6 M Na (20 psi, 20 °C)	0.049(4)	0.036(5)	0.18(1)	0.14(2)
8	6.0 M Na (20 psi, 20 °C)	0.088(8)	0.0652(2)	0.41(4)	0.269(2)
3	8.0 M Na (20 psi, 20 °C)	0.07(2)	0.0257(2)	0.5(1)	0.184(2)

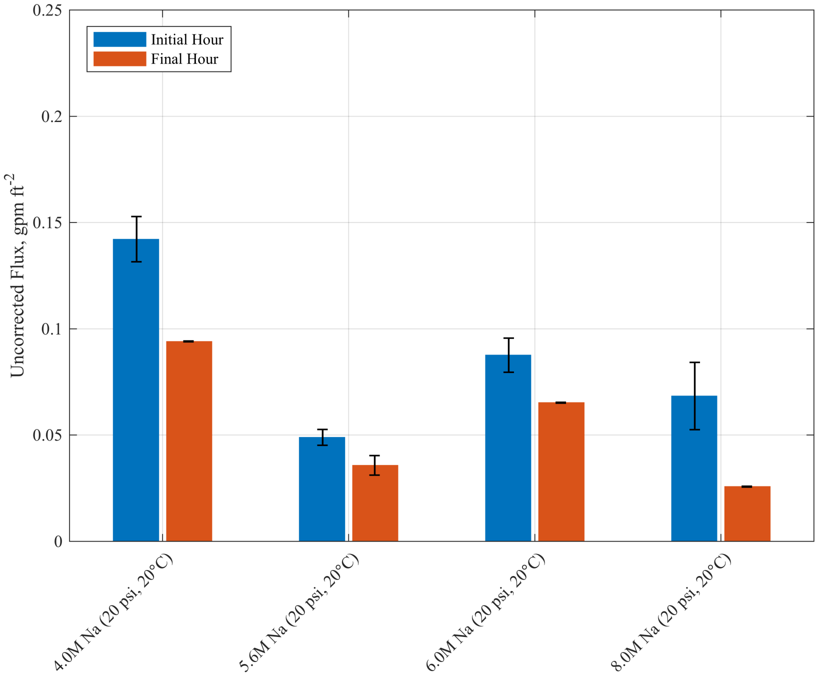

Figure 5.5. Uncorrected flux for the four LAWPS simulants averaged over the initial and final hour of filtration in the 120-hour baseline performance period. Error bars represent one standard deviation.

Table 5.2. Percent decline in uncorrected and normalized fluxes, j and η, respectively, between the initial and final hour of filtration during the 120-hour baseline performance period.

Test	**Description**	**Percent Decline (%)**	
		$j^{(a)}$	$\eta^{(b)}$
7	4.0 M Na (20 psi, 20 °C)	34	36
1	5.6 M Na (20 psi, 20 °C)	27	20
8	6.0 M Na (20 psi, 20 °C)	26	35
3	8.0 M Na (20 psi, 20 °C)	62	63

(a) For the selected operation period, defined as (average j in the first hour − average j in the last hour) divided by average j in the first hour (×100).

(b) For the selected operation period, defined as (average η in the first hour − average η in the last hour) divided by average η in the first hour (×100).

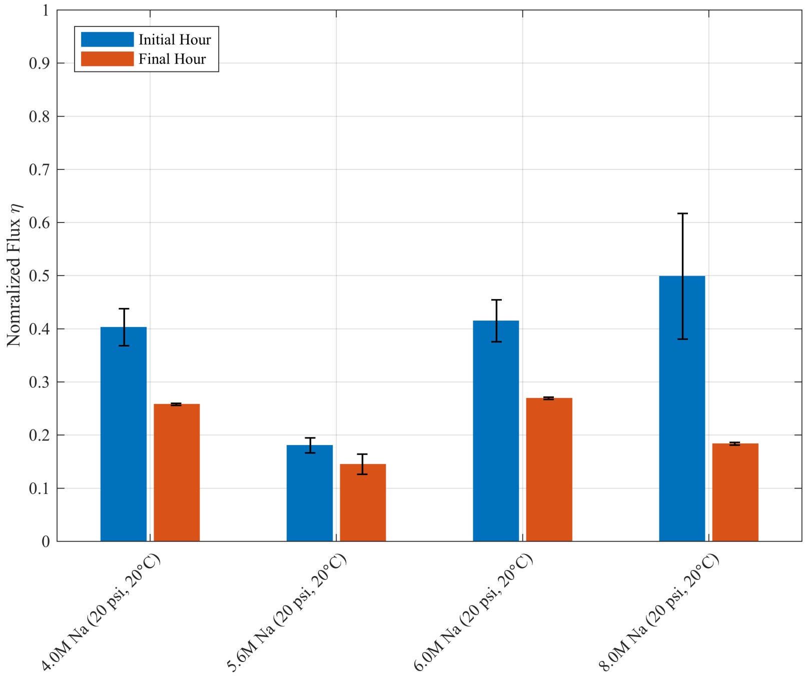

Figure 5.6. Normalized flux for the four LAWPS simulants averaged over the initial and final hour of filtration in the 120-hour baseline performance period. Error bars represent one standard deviation.

extended by operating at lower TMPs or in a constant flux mode. It should be stressed that this conclusion only applies to filtration with materials that filter like the LAWPS simulants; previous testing (Daniel et al. 2011, Schonewill et al. 2012, 2015) indicates that the fouling solids selected for testing, namely sodium oxalate and boehmite, are not the most adverse foulants potentially present in saltcake wastes. Likewise, the morphology of the added solids may not be representative of that in actual wastes such that changes in morphology, caused by dissolution and re-precipitation, could lead to more adverse fouling (see Section 5.3).

5.2 Flux Recovery Performance

The preceding section evaluated Series 1 simulant filter performance over sustained filtration periods with no attempted flux recovery. Although only the 8.0 M Na simulant challenged the LAWPS flux operating range, flux recovery methods may be needed to sustain performance at longer operating times. Two flux recovery strategies were considered: backpulsing and LAWPS chemical cleaning with 0.45 M HNO_3 (see Section 3.6.3 for details). This section evaluates the effectiveness of these strategies at recovering flux, using the flux at the start of the 120-hour baseline filtration period as a reference. The reference flux follows pre-test chemical cleaning with nitric and oxalic acids (see Section 3.6.1) and represents the historical metric for performance on an "acceptably" clean filter. Moreover, the standard laboratory cleaning protocols associated with the flux are demonstrated methods for flux recovery and have, at least for PNNL-related studies, allowed continued use of original filters across the lifetime of their associated test campaigns [e.g., see Daniel et al. (2011)]. For the current report, "effective" flux recovery strategies are those that yield measured fluxes that meet or exceed the reference value measured at the start of testing.

Figure 5.7 shows the uncorrected fluxes measured for the four baseline simulants during the initial hour of filtration that follows the start of testing, the single backpulse, and LAWPS cleaning. Figure 5.8 shows the same result in terms of normalized flux. Both uncorrected and normalized flux show the same trends, namely, that backpulsing effectively restores flux to levels equivalent to the start of testing for the three lower Na molarity simulants and that LAWPS cleaning restores fluxes to levels that exceed those associated with standard chemical cleaning for all four LAWPS baseline simulants. Evaluation of the time rate change in flux following backpulsing (Figure 5.9) shows that the recovered flux behavior is similar to that observed during the start of the test for all simulants except the 8.0 M Na simulant, which shows little to no recovery and an almost immediate decline to a normalized flux of 0.2. In contrast, LAWPS cleaning appears to lead to post-cleaning filter fluxes that are substantially larger than those measured at the start of testing. Indeed, comparison of post-recovery averages and time-rate behavior (Figures 5.8 and 5.9) finds all fluxes increased by approximately 0.1 to 0.2 normalized flux units. Several causes for this fundamental improvement in the post-LAWPS filter behavior can be proposed. The extended duration ($\sim$11 hours) of 0.45 M HNO_3 contact associated with LAWPS cleaning may be more effective at removing intractable solids than the standard laboratory cleaning protocol (which involves contact times of less than 1 hour with 2 M HNO_3). Clean water testing, which follows standard laboratory testing but not LAWPS cleaning, may lead to limited pre-fouling of the filter prior to testing. Another possibility is that testing fundamentally changes the nature of the solids fouling the filter. Prolonged recirculation and shear may change the size of solids and how they interact with the filter. Likewise, solids that preferentially stick to and foul the filter may remain in the test loop during the gravity drain before LAWPS cleaning, and are thus preferentially removed by cleaning and no longer present in the simulant re-introduced to the CFF test apparatus for post-LAWPS cleaning filtration. Regardless of the exact cause, the results indicate LAWPS cleaning effectively restores flux in bench-scale testing for the four LAWPS simulants tested.

The preceding paragraph highlights the poor backpulse recovery for the 8.0 M Na simulant. Although it is unclear why backpulse recovery in the 8.0 M Na simulant was poor, evaluation of the full test profiles in Appendix A demonstrates similar backpulse performance (for both single and repeated backpulses) between the 8.0 M Na and 5.6 M Na simulants. Furthermore, the 8.0 M Na simulant shows relatively significant flux transients at the start of testing and after LAWPS cleaning. In particular, post-LAWPS filtration of the 8.0 M Na simulant shows an two distinct periods of decline: one immediately after LAWPS cleaning and another approximately 8 hours into the post-LAWPS filtration period (see Figure 5.9). The poor recovery during 8.0 M Na simulant backpulsing could suggest that one or more of 8.0 M Na solids precipitated during make-up is a “difficult-to-remove” foulant. As documented in Figure 4.3 of the LAWPS simulant development report (Russell et al. 2017b), XRD finds these solids are composed primarily of sodium oxalate and sodium phosphate, which renders comparison of the 8.0 M Na simulant flux to that of the 5.6 M Na simulant more apt than comparison to those of the 4.0 M Na (primarily boehmite solids) and 6.0 M Na (primarily sodium aluminum phosphate solids) simulants. Indeed, comparison of the time-rate change behavior of the 5.6 M Na and 8.0 M Na simulants shown in Figure 5.9 finds nearly equivalent flux behavior following the single backpulse: an immediate drop in flux over approximately 15 minutes followed by a relatively stable normalized flux of 0.2. It can be postulated that oxalate solids are difficult to remove from the filter via backpulsing and that “acceptable” backpulse performance for the 5.6 M Na simulant results from an anomalously low starting flux (rather than a “good” backpulse recovery). As no replicate baseline 5.6 M Na simulant test was performed, the latter assertion cannot be directly evaluated. However, tests of the 5.6 M Na simulant operated at 20 °C with different pressure profiles (stepped and minimum) appear to confirm “poor” single backpulse performance with the 5.6 M Na simulant (see Section 5.3).

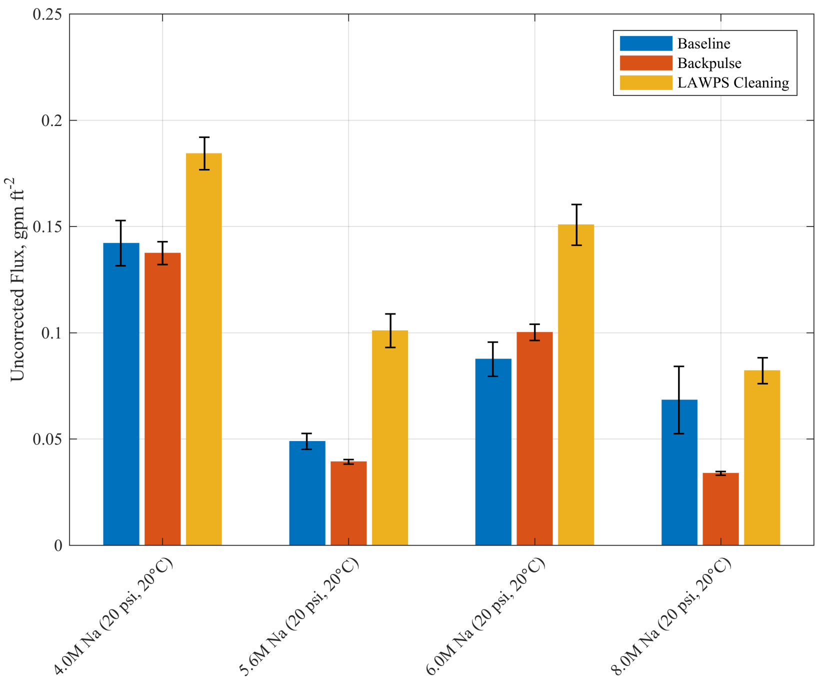

Figure 5.7. Uncorrected flux j for the four LAWPS simulants measured following the start of testing, the single backpulse, and LAWPS cleaning. The error bars represent one standard deviation.

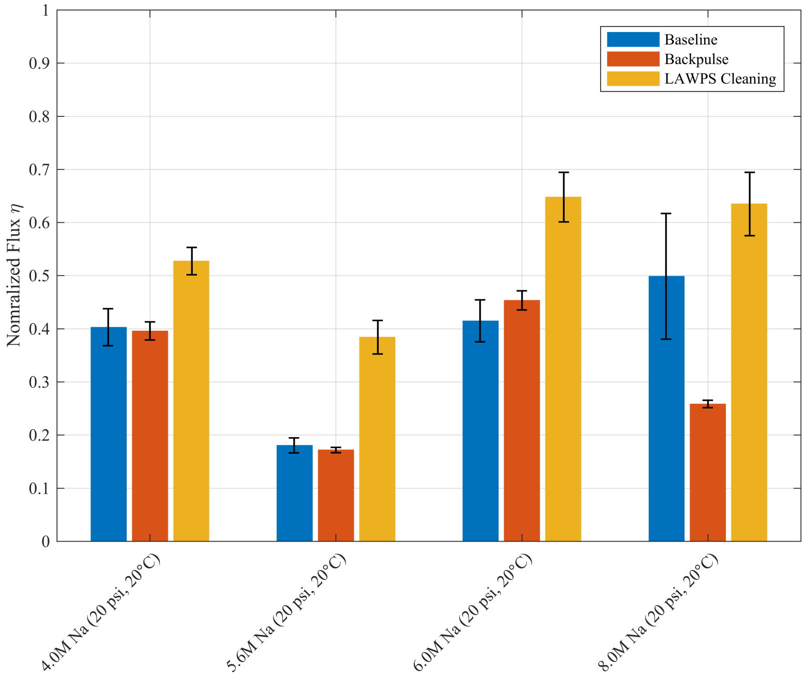

Figure 5.8. Normalized flux η for the four LAWPS simulants measured following the start of testing, the single backpulse, and LAWPS cleaning. The error bars represent one standard deviation.

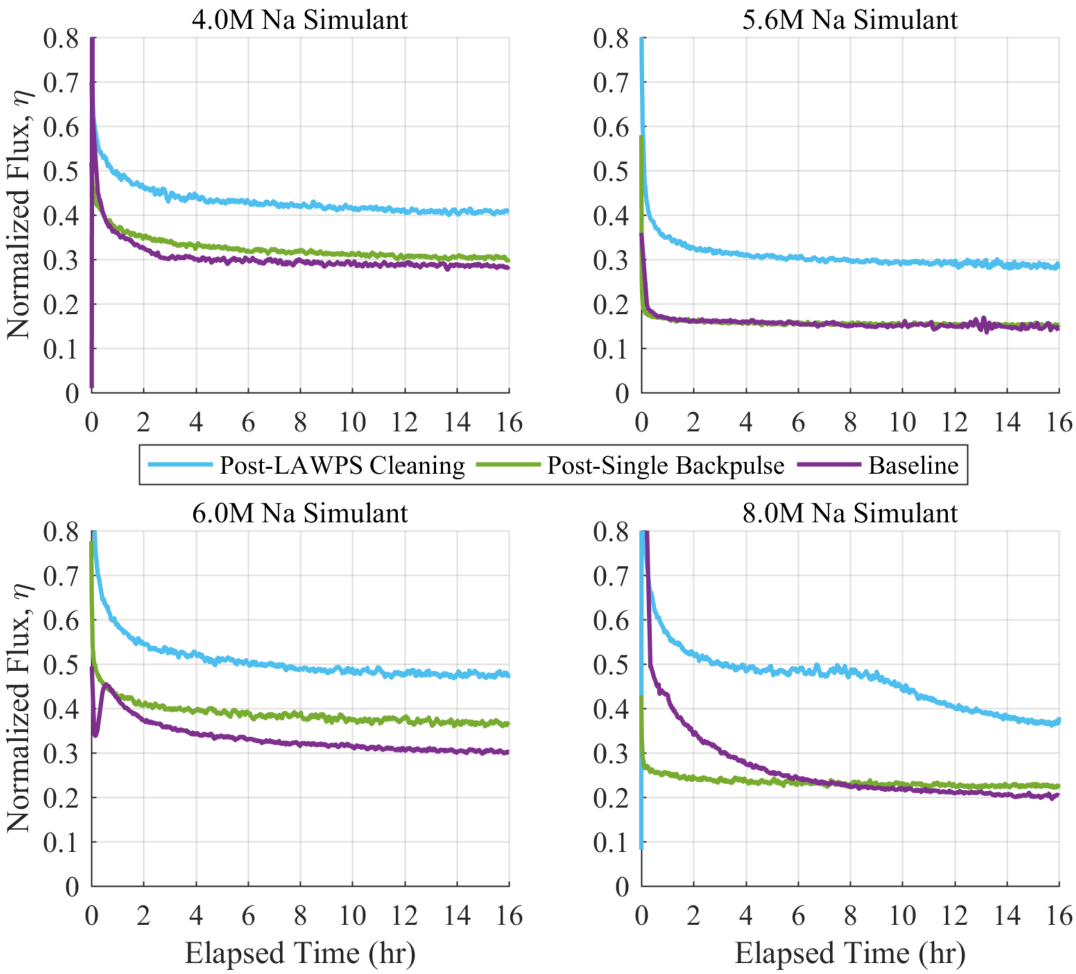

Figure 5.9. Normalized flux η as a function of time following flux recovery for the four LAWPS simulants.

5.3 Temperature and Pressure Profile Dependencies

The baseline testing of the LAWPS simulants described in the preceding sections was done at a target test temperature and TMP of 20 °C and 20 psi, respectively. The range of operating temperatures that bound LAWPS filter operations is approximately 20 to 45 °C. As such, the CFF Series 1 test matrix (Table 3.1) originally included one test evaluating filter performance of the 5.6 M Na simulant at 45 °C; the test matrix was later amended to include a replicate of this test. Furthermore, LAWPS filter operations will target a constant flux by controlling TMP. Under such operating conditions, the reduction in flux caused fouling of the filters by waste solids will be balanced by a corresponding (gradual) increase in the TMP effecting filtration. CFF tests operated at constant pressure do not replicate this functionality, and more so, current CFF hardware does not include controllers that would also direct implementation of constant flux filtration through TMP control. As such, the Series 1 test matrix includes tests intended to indirectly assess the influence that changing TMP has on 5.6 M Na simulant filtration performance. Two TMP functionalities are considered: 1) stepped-pressure and 2) stepped-flux. The stepped-pressure profile implemented fixed 5-psi increases in TMP at 24-hour intervals during the first 120-hour baseline filtration period. The stepped-flux profile implemented step-changes in TMP at 24-hour intervals sufficient to restore filter flux to at least

0.032 gpm ft^{-2} (i.e., the upper LAWPS flux target). Unfortunately, the latter operating mode could not be implemented as planned as the measured 5.6 M Na simulant flux never fell below 0.032 gpm ft^{-2} at the minimum operating TMP of the CFF test apparatus (∼5 psi). As such, stepped-flux filtration of the 5.6 M Na simulant ran at the minimum operating TMP for the duration of the 120-hour baseline filtration period and is termed "Minimum Pressure" filtration rather than the planned "Stepped Flux" filtration. The results of temperature and pressure profile effects on 5.6 M Na simulant filtration performance are discussed in the paragraphs that follow.

5.3.1 The Impact of Temperature

Figure 5.10, Figure 5.11, and Table 5.3 summarize the impact of temperature on the filtration performance of the 5.6 M Na simulant, expressed in terms of average flux during the initial and final hour of the 120-hour baseline period. It is expected that evaluated temperature nominally increases flux through the expected reduction in suspending viscosity (from 3.8 mPa s at 20 °C to 2.2 mPa s at 45 °C for the 5.6 M Na simulant). Assuming Darcy's law holds, a ∼70 % increase in flux should result from increasing temperature from 20 to 45 °C. Secondary improvements may also be realized through 1) dissolution of undissolved solids at elevated temperature (although such improvements cannot be guaranteed as the role of solids content on filtration of LAWPS simulants has not been assessed) and/or 2) reduction in the resistance of solids collected on the filter surface at elevated temperature [as discussed in Daniel et al. (2009)]. In uncorrected flux terms, the expected increases are realized during the initial hour in both initial and repeat performance tests at elevated temperature. However, the initial 45 °C test did not appear to realize the expected degree of improvement. A more significant (and surprising) observation for both initial and repeat elevated temperature tests is the apparent acceleration in the time rate decline of filter flux 90 to 100 hours into the 120-hour baseline filtration period as shown in the normalized flux time graph in Figure 5.12. As observed under nominal operating conditions, backpulsing does not effectively recover lost flux, and the decline continues (for the initial measure) until flux falls to 3% of the maximum flux achievable at the end of the 16-hour period of filtration following the single-backpulse and 0.3% of the maximum flux achievable following repeated backpulsing (see Figure 5.13).

Attempts to understand the radically different fouling behavior of sodium oxalate solids at elevated temperature could not identify any chemical changes in the combined precipitated and added sodium oxalate solids. Indeed, XRD analysis of simulant solids pulled from the final 5.6 M Na simulant suspension processed at elevated temperature still identified the undissolved solids as primarily sodium oxalate. A 7-day heat treatment study evaluated precipitation from the 5.6 M Na simulant supernate in the absence of added sodium oxalate solids. XRD analysis of the heat treated samples identified precipitated sodium aluminosilicate solids as well as several species that could not be identified. However, a similar heat treatment study with added oxalate solids did not evidence similar formation of sodium aluminosilicate or other solids. As

Table 5.3. Uncorrected and normalized flux for the 5.6 M Na simulant at 20 and 45 °C averaged during the initial and final hour of filtration during the 120-hour baseline performance period.

Test	Description	Uncorrected Flux j, gpm ft^{-2}		Normalized Flux η	
		Initial	**Final**	**Initial**	**Final**
1	5.6 M Na (20 psi, 20 °C)	0.049(4)	0.036(5)	0.18(1)	0.14(2)
2	5.6 M Na (20 psi, 45 °C)	0.16(1)	0.0443(5)	0.33(3)	0.093(1)
9	5.6 M Na (20 psi, 45 °C, repeat)	0.21(2)	0.1346(3)	0.7(1)	0.319(6)

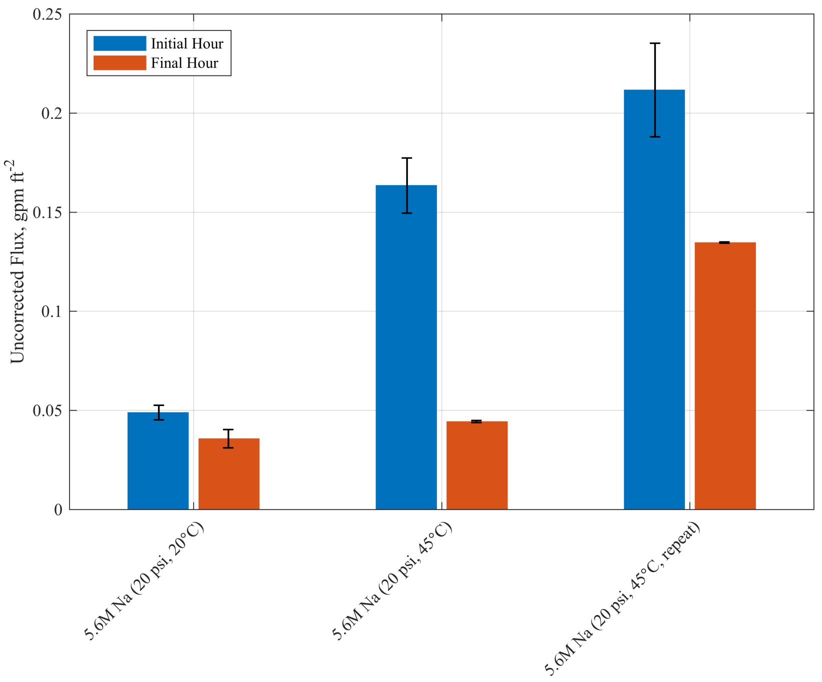

Figure 5.10. Uncorrected flux *j* for the 5.6 M Na simulant at 20 and 45 °C averaged during the initial and final hour of filtration during the 120-hour baseline performance period. The error bars represent one standard deviation.

such, it is unclear if sodium oxalate arrests formation of other solid phases during elevated temperature processing or if the presence of sodium oxalate prevents identification of minor co-precipitates during XRD analysis. Regardless, XRD analysis results described above do not conclusively tie the 45 °C 5.6 M Na simulant fouling behavior to changes in the mineral phase or chemistry of the solids being filtered.

Equivalent elevated temperature tests 5.6 M Na simulant at the ITF suggested that the cause of increased bench-scale CFF fouling at 45 °C may be related to a physical change caused by dissolution and re-precipitation of oxalate solids (Wilson 2017). The key observations supporting this assertion are that Wilson (2017) 1) did not see a similar corresponding reduction in filter performance of the 5.6 M Na simulant at 45 °C in scaled filter testing but 2) did observe the formation of precipitated solids in the test facility filter permeate collection tank. The ITF works with simulant volumes sufficient to allow long-term filtration without the need to recycle permeate. In contrast, bench-scale CFF testing continuously recycles all permeate collected to the slurry reservoir to maintain a reservoir liquid volume of $\sim$15 L. Given the absence of degraded filter performance at elevated temperature in engineering-scale test of the 5.6 M Na simulant, it can be postulated that reduced performance at the bench-scale results from dissolution and re-precipitation of sodium oxalate fines. The dissolution and re-precipitation process is driven by temperature differentials that exist between the recirculation loop, which is temperature-controlled through a heat-exchanger installed at the re-circulation pump outlet, and the permeate metering system, which is not temperature-controlled. As such, the permeate recycled back into the tank can be 1-2 °C lower that the recirculated suspension when testing at elevated temperature. Use of continuous permeate recycle to maintain slurry reservoir volume during testing entrains the re-precipitated solids into the filter re-circulation loop, where they eventually contact the filter and degrade filter performance. Occurrence of the dissolution and re-precipitation process would generally be evidenced by a reduction in particle size of the 5.6 M Na simulant as a function of temperature

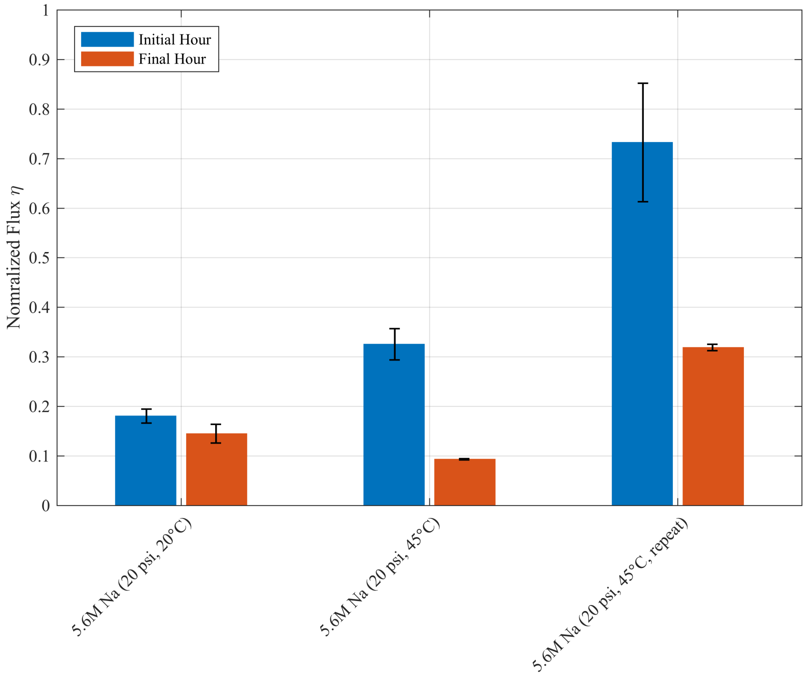

Figure 5.11. Normalized flux η for the 5.6 M Na simulant at 20 and 45 °C averaged during the initial and final hour of filtration during the 120-hour baseline performance period. The error bars represent one standard deviation.

(beyond that incurred by normal attrition under shear during recirculation). To test this postulate, the PSDs of the as-prepared and as-tested (at both 20 to 45 °C) 5.6 M Na simulant was measured. The measured size distributions, shown in Figure 5.14, do indeed evidence an increased population of fine oxalate solids in the 5.6 M Na simulant tested at elevated temperature that cannot be attributed to particle attrition under shear alone.

Given the results discussed in the preceding paragraph, the anomalous fouling behavior observed in bench-scale testing of the 5.6 M Na simulant at 45 °C does not create immediate concerns for the sustainability of filter operations in the LAWPS facility. The physical mechanism for flux decline in bench-scale testing appears strongly tied to the use of continuous permeate recycle to maintain slurry reservoir volume. That being stated, it does not exclude the potential for similar changes in solid particle size for waste species that can engage in dissolution and re-precipitation upstream of LAWPS filter process. However, while the flux decline was repeatable in replicate testing, the overall magnitude of flux in all tests with sodium oxalate (both the standard LAWPS simulants and the fouling simulants as well) was not repeatable. There was significant test-to-test variation in the flux, which is most readily apparent when comparing the initial and replicate test results in Table 5.3, which demonstrate a factor of 2-3 difference in the normalized flux values between the two nominally identical tests with the 5.6 M Na simulant. As such, the current test results indicate sodium oxalate has significant fouling proclivity, but it is unclear what exact combination of operational or chemical conditions drive the worst of fouling behavior associated with this solid.

For the temperature tests, subsequent flux recovery operations (shown in Figure 5.15) found results similar to those for the sodium-oxalate-rich LAWPS simulants (i.e., the 5.6 M Na and 8.0 M Na simulants). In both initial and repeat temperature tests, backpulsing yields only a limited recovery in flux. LAWPS cleaning

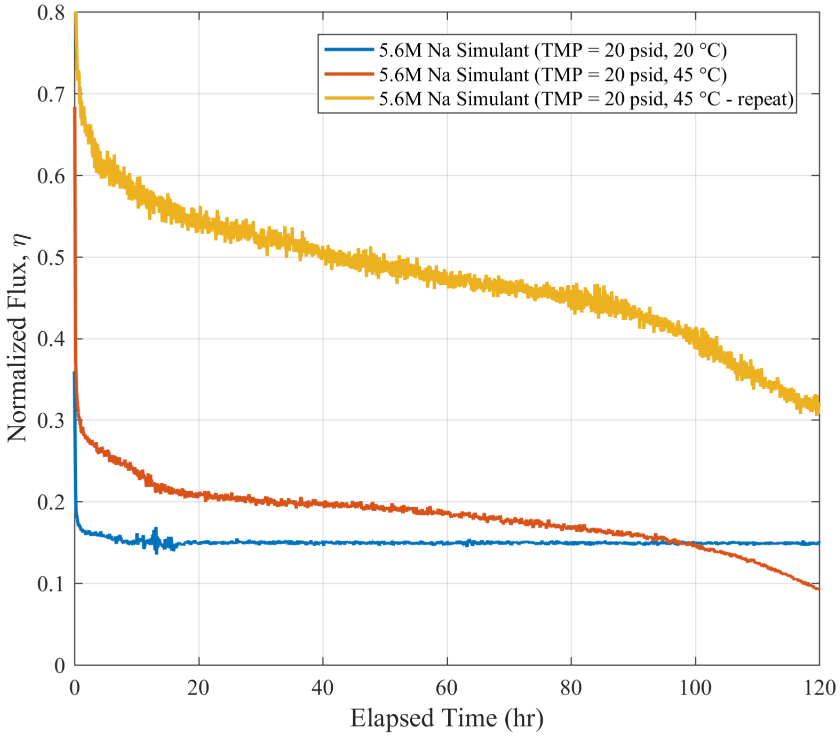

Figure 5.12. Normalized flux η as a function of time for the 5.6 M Na simulant at 20 and 45 °C during the 120-hour baseline period.

leads to complete recovery in flux during the first temperature test, but only a partial recovery in flux in the second test. This reduced performance does not mean that LAWPS cleaning failed during the replicate temperature test, as cleaning greatly increases the flux performance relative to backpulsing. Furthermore, the initial normalized flux measured during replicate testing of the 5.6 M Na simulant at 45 °C is large, not just relative to the initial 5.6 M Na simulant at 45 °C but relative to all other tests with LAWPS simulants except those operated at reduced pressure (cf. Figure 5.17). The tabulated results (provided in Appendix A) show a difference in average TMP between the baseline (14 psi) and LAWPS cleaning (16 psi) filtration periods during the initial hour over which the averages shown in Figure 5.15 are taken. It is expected that this difference in pressure could have led to the slight reduction in post-LAWPS cleaning performance observed in the replicate test, as fouling depends strongly on the TMP history (as discussed in the paragraphs that follow). For this reason, it is concluded that LAWPS cleaning is as effective as the standard chemical cleaning protocol following operation at elevated temperature.

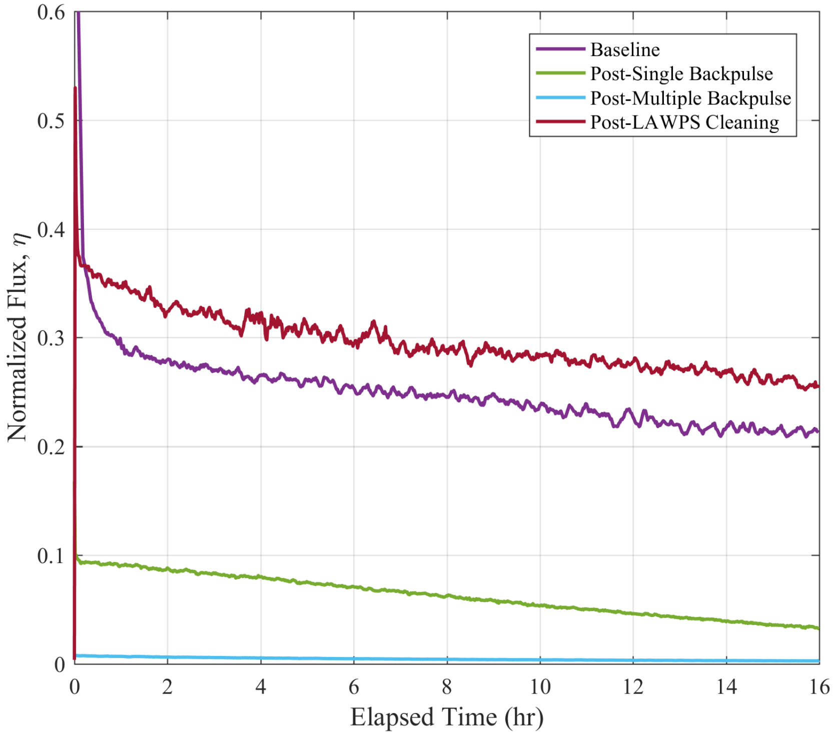

Figure 5.13. Normalized flux η as a function of time for the 5.6 M Na simulant at 45 °C (initial test) for the 16-hour periods following the start of testing, single backpulsing, multiple backpulsing, and LAWPS cleaning.

5.3.2 The Impact of TMP Profile

Darcy's law predicts that permeate flux will be directly proportional to applied TMP. While there are factors, such as the compressibility of accumulated solids or osmotic pressure gradients, that reduce or alter the pressure dependence, it is generally expected that higher pressure will lead to higher fluxes. Uncorrected filter flux as a function of time for the three pressure profiles (constant, stepped pressure, and minimum pressure) evaluated in Series 1 testing are shown in Figure 5.16 and demonstrate the expected behavior: Step increases in pressure lead to step increases in filter flux. However, that the 5.6 M Na simulant operated at minimum and stepped pressures outperforms the test at 20 psi, even during the period of initial operation (where process history effects should be minimum), is unexpected. There is no clear explanation for this behavior, although it is consistent with earlier assertions that the initial flux for 5.6 M Na is anomalously low (see Section 5.2). At longer times, differences in flux caused by the underlying difference in TMP obscure the underlying fouling behavior of the solids. As increased flux drives more solids to the surface, it is expected that operations at higher TMP will lead to increased fouling. Figure 5.17, which shows flux on a normalized basis, clearly demonstrates the increased fouling that occurs with increasing operating pressure. Of the three profiles, the constant 20 psi 5.6 M Na simulant test shows the lowest normalized flux of 0.15 at 120 hours, whereas the test operated at the minimum TMP achievable on the system operated at a normalized flux of 0.5 at 120 hours. The stepped pressure test fell between these two extremes, and exhibited a gradual decline in normalized flux with increasing pressure. Table 5.4 summarizes the fluxes observed during the initial and final hour of pressure profile testing. The pressure profile test results clearly delineate the cost of increasing flux through increased applied pressure — high TMP leads to accelerated fouling of the filter.

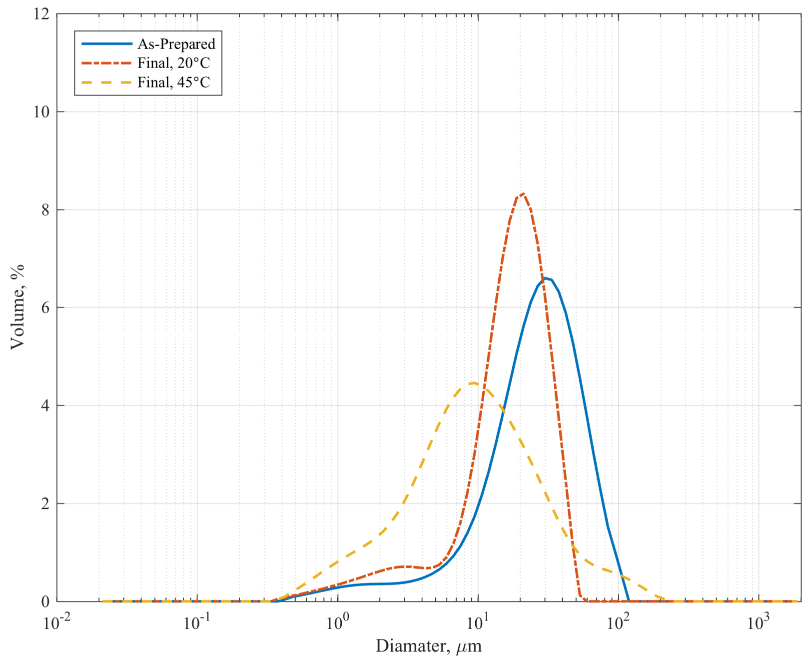

Figure 5.14. PSDs for the as-prepared, as-tested at 20 °C, and as-tested at 45 °C 5.6 M Na simulant.

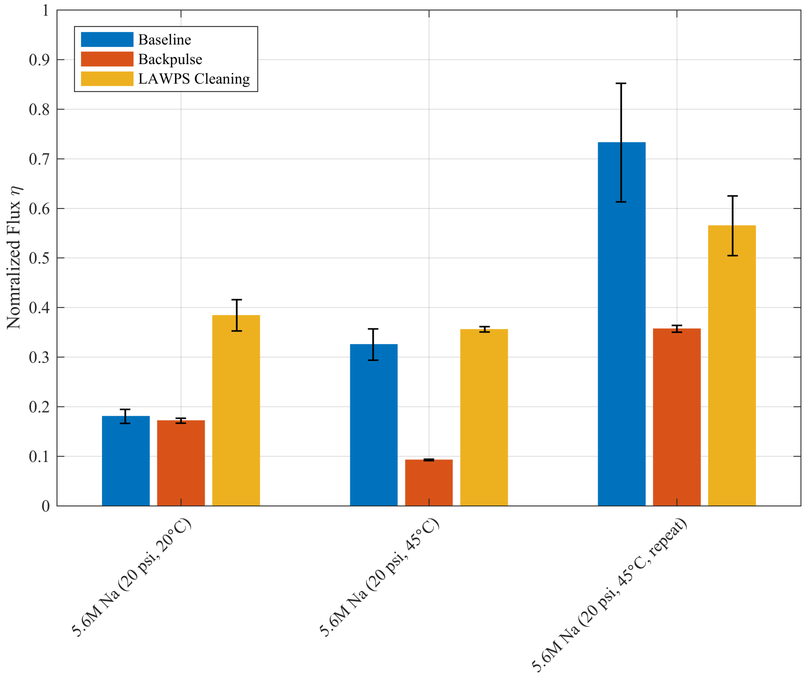

Figure 5.15. Normalized flux η for the 5.6 M Na simulant tested at 20 and 45 °C at the start of testing, the single backpulse, and LAWPS cleaning. The error bars represent one standard deviation.

However, these increases do yield improved filter throughput. As shown in Figure 5.18, the total volume of permeate separated by the stepped pressure profile rapidly exceeds that produced at minimum pressure over LAWPS relevant filter periods, despite the increase in filter fouling associated with increased TMP.

From the observations and discussion above, two important conclusions can be made. First, bench-scale CFF pressure profile testing supports the appropriateness of the constant flux strategy selected for LAWPS filter operations. For wastes that filter like the LAWPS simulants tested herein, use of the lowest TMP needed to meet the LAWPS flux targets will minimize fouling and reduce the frequency of flux recovery operations needed to maintain acceptable filtrate production rates. Second, with respect to bench-scale testing, the pressure profile tests demonstrate that constant-pressure filtration is likely representative or bounding for cases where the measured filter flux falls above or within the LAWPS testing range. It should be noted that all baseline LAWPS simulant and fouling simulant tests reported herein meet this requirement and can be considered relevant assessments of LAWPS performance.

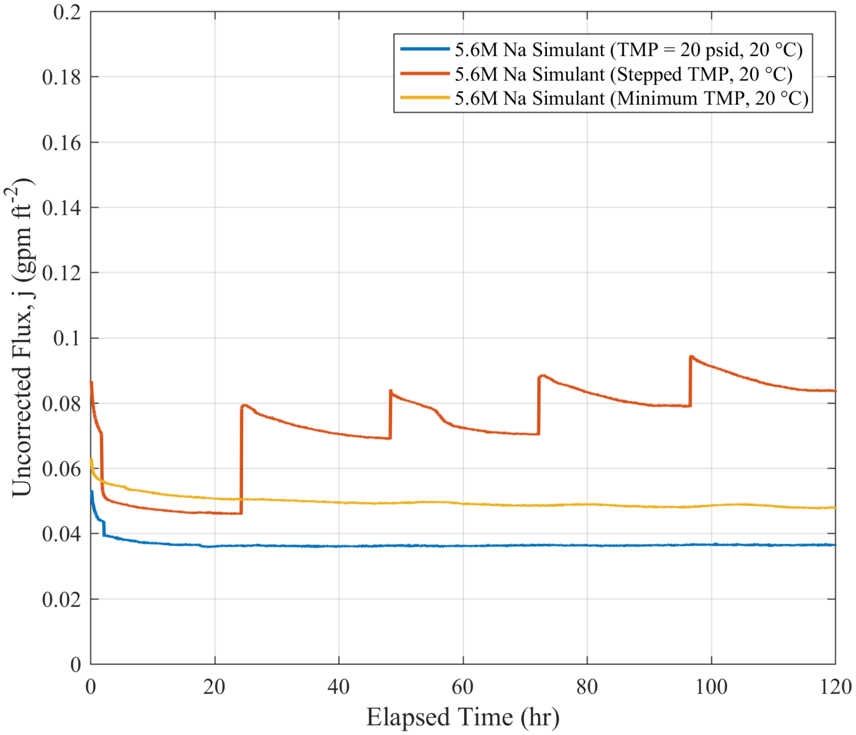

Figure 5.16. Uncorrected flux j for the 5.6 M Na simulant pressure profile tests.

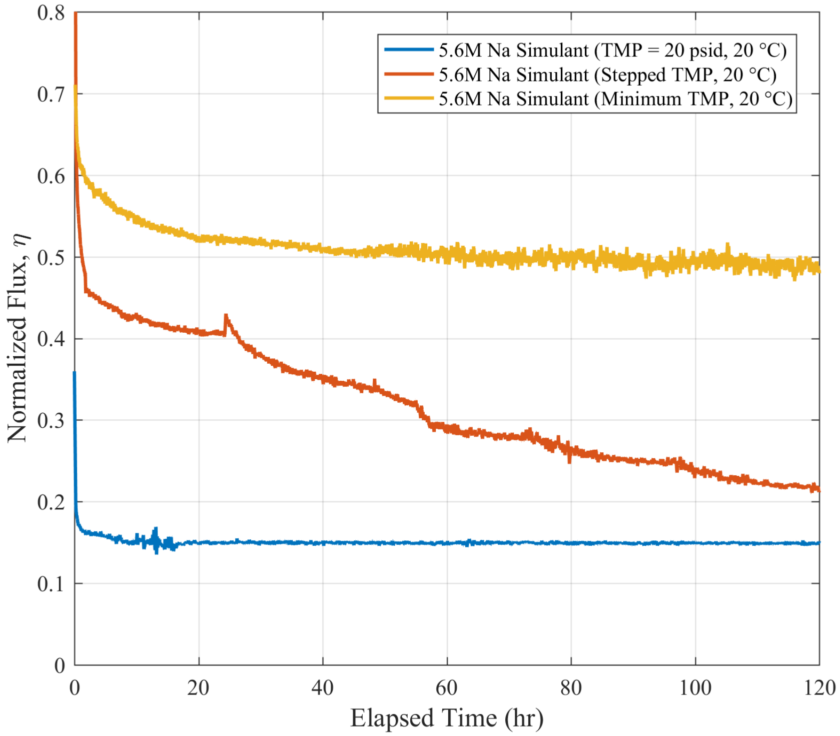

Figure 5.17. Normalized flux η for the 5.6 M Na simulant pressure profile tests.

Table 5.4. Uncorrected and normalized flux for the 5.6 M Na simulant pressure profile testing during the initial and final hour of filtration during the 120-hour baseline performance period.

Test	**Description**	Uncorrected Flux j, gpm ft^{-2}		Normalized Flux η	
		Initial	**Final**	**Initial**	**Final**
1	5.6 M Na (20 psi, 20 °C)	0.049(4)	0.036(5)	0.18(1)	0.14(2)
4	5.6 M Na (Stepped Pressure)	0.079(5)	0.0797(2)	0.58(4)	0.207(2)
5	5.6 M Na (Minimum Pressure)	0.059(2)	0.0479(1)	0.64(2)	0.486(4)

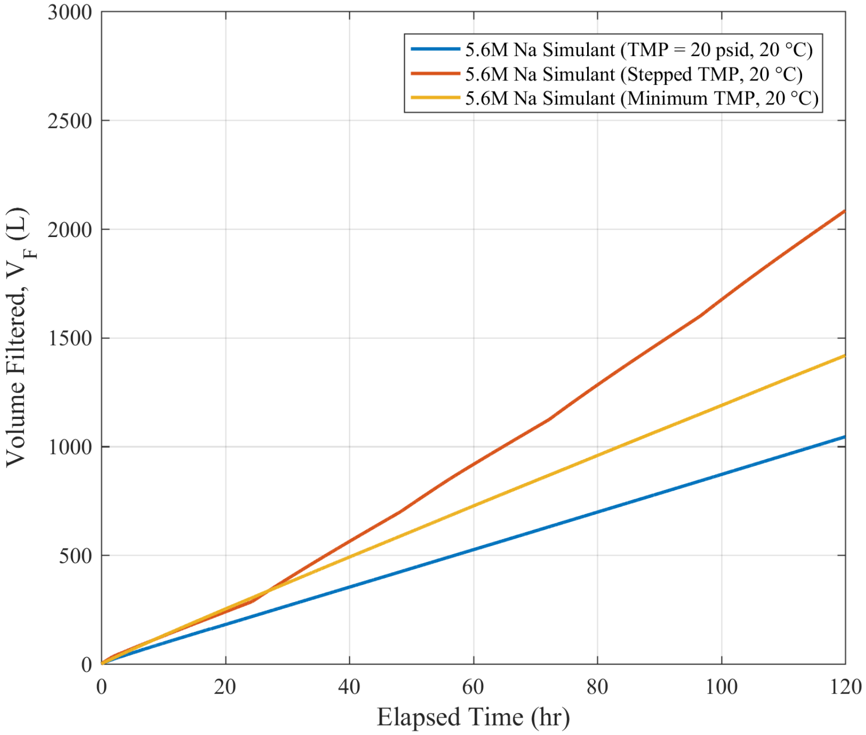

Figure 5.18. Volume of 5.6 M Na simulant filtered during pressure profile testing as a function of filtration time.

5.4 Key Observations

The baseline filter performance of the four LAWPS simulants has been discussed in detail in the preceding sections and provides information useful in assessing scale-up of filtration operations at the ITF and at the full-scale LAWPS facility. Based on the performance of the four LAWPS baseline simulants, the following key observations can be made with respect to the sustainability and optimization of LAWPS CFF filtration operations:

- On a uncorrected flux basis, the 8.0 M Na simulant was the most difficult to filter and was the only simulant that challenged the target LAWPS flux range of 0.0075 to 0.032 $\mathrm{gpm\,ft^{-2}}$. However, the 8.0 M Na simulant is challenging in part due to its high viscosity and dissolved solids content. On a normalized basis, the 5.6 M Na simulant is the most challenging to filter, likely in part due to the addition of sodium oxalate solids to supplement the fines precipitated during simulant preparation. In overall terms, the final fluxes observed during the 120-hour baseline performance period for the 4.0 M, 5.6 M, 6.0 M, and 8.0 M Na simulants represent an estimated 26%, 14%, 27%, and 18%, respectively, of the maximum flux that could be realized on the filter element (see Table 5.1). As such, the 5.6 M Na simulant evidences the worst fouling proclivity of the four baseline simulants tested. As a result, its flux-based filter performance is only marginally better than that of the 8.0 M Na simulant. These results highlight the difficulty in predicting simulant performance based on a single chemical metric, such as sodium molarity. Rather, filter performance must be based on an understanding of the suspending phase chemistry, the fouling proclivity of the suspended solids, and other operational parameters (such as TMP profile). Currently, there is no means of gauging solid component fouling proclivity beyond broad chemical generalizations (such as the identification of sodium oxalate solids as “bad” actors), which hinders predictive assessments of filter performance in the absence direct testing.

- Waste materials that filter like the four baseline LAWPS simulants should not challenge the sustainability of LAWPS filter operations during 120 hours of continuous filtration and longer (given operational strategies that avoid fouling, such as using the lowest TMP possible to achieve the desired target flux).

- Backpulsing is an effective means of flux recovery for at least two of the LAWPS simulants (i.e., the 4.0 M, and 6.0 M Na simulants). However, as both the 5.6 M Na and 8.0 M Na simulants show poor backpulse recovery, the effectiveness appears to depend on the chemistry of the fouling solids and in particular on the presence of solid oxalate solids. Thus, single backpulsing can be an effective means of recovering flux, but may not recover flux efficiently for all waste chemistries. In the latter case, repeated backpulsing can lead to substantial declines in filter flux like that observed in initial 45 °C 5.6 M Na simulant temperature test (with a final flux of 0.1 % of the maximum possible).

- For the test materials evaluated in this report, LAWPS cleaning appears to be an effective means of lost flux recovery. Indeed, LAWPS cleaning protocols were frequently as effective as, if not more effective than, standard laboratory cleaning protocols used to clean the filter before the start of each test and that have enabled previous bench-scale CFF test campaigns conducted at PNNL to re-use the same filter element without any cumulative loss in performance over several months of testing. This conclusion should be approached with some caution, as the current testing evaluated LAWPS cleaning in a highly idealized configuration that allowed efficient removal of the test simulant from the recirculation and permeate collection lines before displacement with 0.1 M NaOH solution and that minimized potential hold-up of test material throughout the system. Hold-up of waste is expected in the full-scale LAWPS CFF system, and contact of the waste residual with cleaning solutions could lead to the formation of precipitates that are stable in the subsequent cleaning solutions and/or that embed in the filter elements, reducing the flux recovery effected by cleaning.

- Use of a single, constant transmembrane pressure of 20 psi for bench-scale testing should yield filter fouling that bounds that realized during constant-flux filtration operations (like that planned for LAWPS operations) as long as the fluxes realized in bench-scale testing meet or exceed the LAWPS flux target of 0.0075 to 0.032 $\mathrm{gpm\,ft^{-2}}$. This requirement is satisfied for the four LAWPS simulants and eight fouling simulants tested in the current report. As such, the filter performance data generated herein are relevant to LAWPS testing.

6.0 Fouling Simulant Filtration Results

After assessment of the filtration behavior of the four LAWPS simulants as described in Section 5, this section presents results for a series of eight tests that were conducted with fouling simulants (also referred to as Series 2 testing). The eight fouling simulants used are described in Section 4.2, and were tested in the manner discussed in Section 3.5. The tests contained the same segments as the Series 1 tests (see the description in Section 5), and a similar focus on selected portions of the data collected is used to discuss the results. Fouling proclivity of each of the eight simulants is assessed using Segment A data, i.e., a nominally 120-hour period of continuous filtration at constant TMP (refer to Figure 5.1). Evaluation of the performance of flux recovery approaches uses flux data from Segments B and F in comparison with Segment A. Other unique or special cases are demonstrated using selected data appropriate to the topic being investigated. For additional data generated from the Series 2 tests, see Appendix B, which includes average data over the initial and final hour of various operating periods and a plot of the uncorrected flux for the entire test.

6.1 Fouling Proclivity

Filter flux as a function of time is shown for the eight fouling LAWPS simulants (see Table 3.3 for the complete list) in Figure 6.1 for the initial 120-hour period of filter operation. The various simulants are indicated in the figure by referring to the primary solid component (and corresponding solids loading) added to the MSS at the start of each test. Since the objective was to create simulants that exhibited fouling behavior that was "worse" than that of the 5.6 M simulant tested in Series 1, in cases where the initial filter flux did not appear to have significant fouling, the simulant was adjusted in an attempt to identify simulant configurations that fouled more significantly. These adjustments are split into two categories:

Solid Component Adjustments: Each test was started with the nominal solids component and loading as shown in Table 3.3. Three tests were adjusted after the initial loading as described below:

- Test 2 (LPS-T2S2-MS-SP-01) — Test 2 contained sodium phosphate solids. The test began at 45 °C and was dropped to 20 °C after approximately 24 hours elapsed to see if additional sodium phosphate solids would precipitate. The filter flux did not decrease significantly with time after this temperature decrease. At approximately 96 hours elapsed time, an additional 0.1 wt% of sodium phosphate solids was added to the slurry reservoir to see if sodium phosphate solids fouling had a substantial concentration effect.

- Test 6 (LPS-T2S2-MS-HE-01) — Test 6 contained HEDTA at an initial loading of 0.1 wt%. The HEDTA was soluble and almost immediately reduced the filter flux, after which it appeared to stabilize with time. After 24 hours had elapsed, an additional 0.1 wt% was introduced to the slurry reservoir to observe if the initial effect was additive. When an additional 24 hours had elapsed, it did not appear to have had the same impact as the initial increment of HEDTA. Approximately 0.02 wt% of insoluble amorphous iron oxide[1] was added to see if HEDTA and insoluble Fe performed any chemical interactions that would produce additional fouling.

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[1] As discussed in Section 4.2.2, XRD identified the iron solid synthesized for testing as amorphous iron oxide. This component has been referred to as iron oxyhydroxide in previous PNNL reports. The current report section uses both names, iron oxyhydroxide and amorphous iron oxide (or simply iron oxide), to refer to this component.

- Test 7 (LPS-T2S2-NA-RF-01) — Test 7 solids components were not adjusted during the initial 120-hour period. However, in the interest of having a complete list of adjustments, equal amounts of HEDTA and $Fe(NO_3)_3$ were added during the post-LAWPS cleaning period to observe if soluble Fe and HEDTA performed any complexant chemistry. This impacts only the post-LAWPS cleaning flux period and is not visible in Figure 6.1.

Operational Adjustments: In general, tests shown in Figure 6.1 were conducted at a constant TMP of 20 psi and an AV and temperature of $14.7\,\mathrm{ft\,s^{-1}}$ and 20 °C. However, in Tests 2 (LPS-S2T2-MS-SP-01) and 8 (LPS-S2T2-MS-SF-01), the temperature was initially established at 45 °C. After a portion of the 120 hours had elapsed, the temperature was reduced to 20 °C and remained there for the duration of the operating period. The drop in temperature was enacted to observe if precipitation of solids would occur in the slurry reservoir that might significantly foul the filter. Test 7 (LPS-S2T2-NA-RF-01) was not operated at a constant TMP of 20 psi because the simulant viscosity was similar to that of water and the resulting permeate production rate would have overwhelmed the permeate collection system used in the testing. Thus, it was operated at a lower constant TMP that represented the minimum backpressure of the system (approximately 6 psi at the outset).

Note that these adjustments are also summarized in Table 3.4.

Figure 6.1 illustrates the separation between two classes of low-solids-loading fouling simulants tested in this study: those that did not foul significantly and those that did. For purposes of discussion, significant fouling is defined as a filter flux that falls below the 5.6 M simulant average flux for the baseline period ($0.0365\,\mathrm{gpm\,ft^{-2}}$). Since the upper bound of the shaded region in Figure 6.1 is equivalent to $0.032\,\mathrm{gpm\,ft^{-2}}$, the shaded area serves as an approximate visual indicator of the fouling agents that fall below the 5.6 M simulant average flux. Fouling agents that were deemed insignificant include sodium phosphate (Test 2), HEDTA (Test 6), sRF resin (Test 7), and sodium fluoride (Test 8). Despite adjustments to those four fouling simulants previously described in this section, significant fouling was not observed over the entire baseline filtration period. Although the HEDTA data does begin to foul rapidly after approximately 48 hours, this corresponds to the addition of a small amount of amorphous iron oxide, which is the same component used in Test 3. The addition of that species to the HEDTA-containing simulant was an attempt to see if any insoluble-Fe/HEDTA chemistry occurred that would adversely affect filter performance; the similarity of the combined insoluble-Fe/HEDTA slurry flux to that of amorphous iron oxide slurry (Test 3) after 120 hours in Figure 6.1 suggests an absence of notable chemical interactions other than the fouling behavior of the iron oxide particles. HEDTA on its own did not lead to significant long-term fouling.

The significant fouling simulants were sodium oxalate (Test 1), amorphous iron oxide (Test 3), iron phosphate (Test 4), and cancrinite (Test 5). The flux of each of these simulants decayed below the 5.6 M simulant average flux within the first 24 hours of operation in the baseline period. There was some variation in the rate of flux decay, but the flux observed at the end of the baseline period for these four simulants was similar, varying between $0.0137\,\mathrm{gpm\,ft^{-2}}$ and $0.0219\,\mathrm{gpm\,ft^{-2}}$. Table 6.1 summarizes the flux measured in the initial and final hour of the baseline period for all eight simulants on both an uncorrected and normalized basis. Note that the significant fouling simulants do not always exhibit the greatest absolute change in flux between the initial and final hour — for example, the sodium phosphate (Test 2) and sodium fluoride (Test 8) tests both decline approximately 60% over the baseline period, which is comparable to the declines observed for significant fouling simulants such as the iron compounds (Tests 3 and 4). In the case of the sodium phosphate and sodium fluoride tests, the primary contributor to the magnitude of the flux decline is known because adjustments were made to those tests in situ during operation. Regardless, those tests still illustrate how characterizing filtration performance should not be limited to one aspect of the measured flux, and it is important to consider several aspects of the flux behavior when assessing fouling: the rate of flux change, the magnitude of the change, and the absolute value of the flux (or a normalized version such as η) itself.

Examining the flux on a normalized basis for the eight fouling simulants allows for comparison across tests despite temporal differences in temperature, viscosity, and TMP. The normalized flux data are shown in Figure 6.2. For example, the sudden decrease in uncorrected flux for Tests 2 (LPS-S2T2-MS-SP-01) and 8 (LPS-S2T2-MS-SF-01) that were due to a temperature change from 45 °C to 20 °C are less apparent with the flux recast as η. Only two sudden flux changes are still resolved, and both are related to adjustments to tests where additional solids were added, e.g., Tests 2 and 6. Figure 6.2 data do not imply that any of the fouling simulants should be re-classified as significant to insignificant fouling agents (or vice versa). However, the data do indicate that the significant fouling simulants generally achieved values of $\eta < 0.1$ within the first 24 hours of operation, whereas insignificant fouling simulants did not decay below an η of 0.2 even after 120 hours. The demarcation in η values between the two foulant classes is consistent with the uncorrected filter flux. One interesting observation is that the degraded sRF resin (Test 7) experienced a decrease in η from 0.7 to approximately 0.2 over the baseline period. This is in contrast to the other three insignificant fouling simulants, which had η that did not change more than 15 % (when considering periods where solid component adjustments were not made to the simulants). The sRF resin had the largest absolute change in η of any simulant, but the final magnitude of η was still more than twice the η values

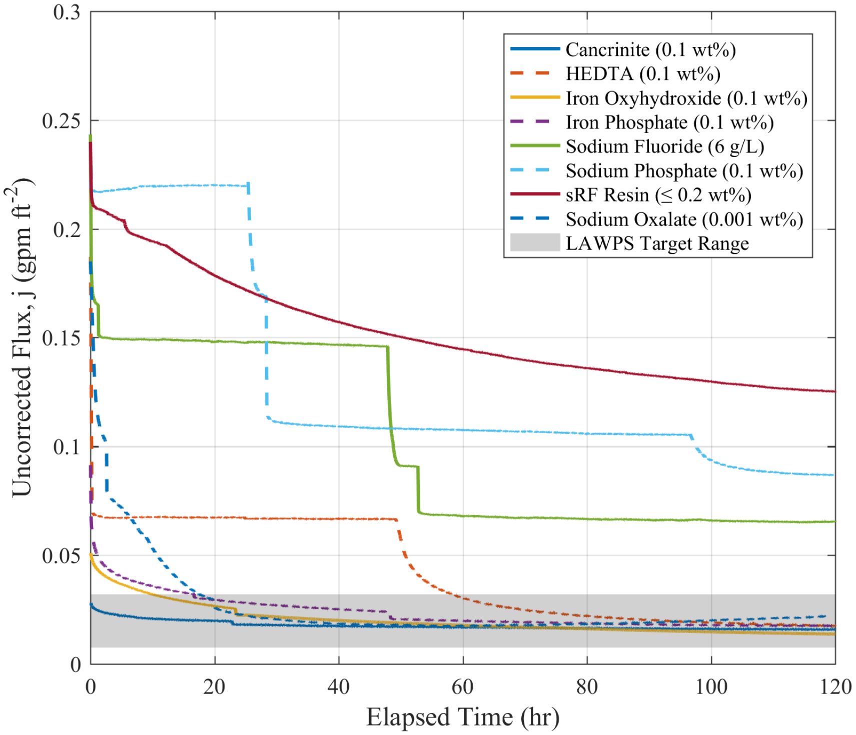

Figure 6.1. Uncorrected flux measured for the eight fouling simulants during the initial (120-hour) baseline filtration period. In general, tests were run at nominal conditions (AV, 14.7 ft s^{-1}; TMP, 20 psi; temperature, 20 °C), with select exceptions noted in the accompanying text. Note: The shaded gray region indicates target flux range of LAWPS operations (0.0075 to 0.032 gpm ft^{-2}).

Table 6.1. Uncorrected and normalized flux for the eight fouling simulants averaged over the initial and final hour of filtration in the 120-hour baseline performance period.

Test[(a)]	**Description**[(b)]	Uncorrected Flux j, $\mathrm{gpm\,ft^{-2}}$		Normalized Flux η	
		Initial Hour	**Final Hour**	**Initial Hour**	**Final Hour**
1	*Sodium Oxalate*	0.15(3)	0.0219(2)	0.5(1)	0.090(1)
2	Sodium Phosphate	0.218(2)	0.0869(2)	0.53(1)	0.367(2)
3	*Iron Oxide*	0.046(2)	0.0137(4)	0.192(8)	0.050(2)
4	*Iron Phosphate*	0.056(5)	0.0175(1)	0.21(2)	0.073(1)
5	*Cancrinite*	0.0258(8)	0.0159(2)	0.104(3)	0.0645(9)
6	HEDTA	0.07(1)	0.0174(8)	0.28(4)	0.058(3)
7	Degraded sRF Resin	0.212(3)	0.1253(1)	0.67(3)	0.226(3)
8	Sodium Fluoride	0.170(6)	0.0654(2)	0.35(2)	0.260(3)

(a) The test number reflects the order in which tests were originally proposed in the Series 2 test matrix and does not reflect the actual order in which they were tested.
(b) Italics denotes component as one of the four down-selected "worst" fouling simulants.

measured for the significant foulants at the same time. Given enough operating time, degraded sRF resin may approach $\eta < 0.1$ just as the four significant fouling simulants did. This is relevant because the η data indicate that degraded sRF resin, if it were present in a CFF system, should be expected to contribute to fouling behavior over long periods of operation. However, the presence of sRF resin on the tube side of the filter in the amount tested is unlikely to occur in the LAWPS CFF system (see the Table 4.5 entry for sRF resin for additional discussion), and the result is considered conservative. It is mentioned to draw attention to the possibility that sRF resin could impact performance over time if it accumulated on the CFF shell-side of the filter.

From the perspective of identifying solid components that are likely to foul in the LAWPS CFF system, the four simulants classified as significant fouling agents are of greater interest. This does not preclude the insignificant fouling agents from contributing to fouling behavior in a particular simulant or waste slurry, but they are not likely to be the dominant component controlling the filtration performance. The presence of a small amount of dominant foulants has been described in past work (Daniel et al. 2011, Schonewill et al. 2012). For the remainder of Section 6, the four significant fouling agents will be referred to as the "worst" fouling simulants for convenience. In Figure 6.3, the normalized flux for the "worst" simulants in the initial and final hour of the baseline period is presented. Based on the data, three of the simulants rapidly reduce the flux even in the first hour of operation, where η is already $\lesssim 0.2$. Cancrinite is a particularly rapid fouling agent; the normalized flux is only 10% of maximum achievable flux in the initial hour ($\eta \sim 0.1$), which is a factor of two lower than either of the iron compounds. The iron compounds both exhibit a comparatively more gradual flux decline but ultimately drive the flux to the lowest η values.

Conversely, the sodium oxalate simulant has an initial hour η that is greater than that for any of the baseline simulants (see Figure 5.6). However, it eventually declines to an η in the final hour that is similar to the other three tests. Compared to the baseline 5.6 M simulant, which contained sodium oxalate at a significantly higher loading (nominally 0.45 wt% versus 0.001 wt%), the sodium oxalate fouling simulant behavior is unexpected. The 5.6 M simulant demonstrated very little fouling when operated at the same conditions (AV, $14.7\,\mathrm{ft\,s^{-1}}$; TMP, 20 psi; temperature, 20 °C). During the prior test (Test 7, LPS-T2S2-NA-RF-01), the permeate and backpulse system were difficult to clean of residual iron and (possibly) degraded sRF resin. Some of this material may have remained on the permeate or shell side of the filter until the sodium oxalate

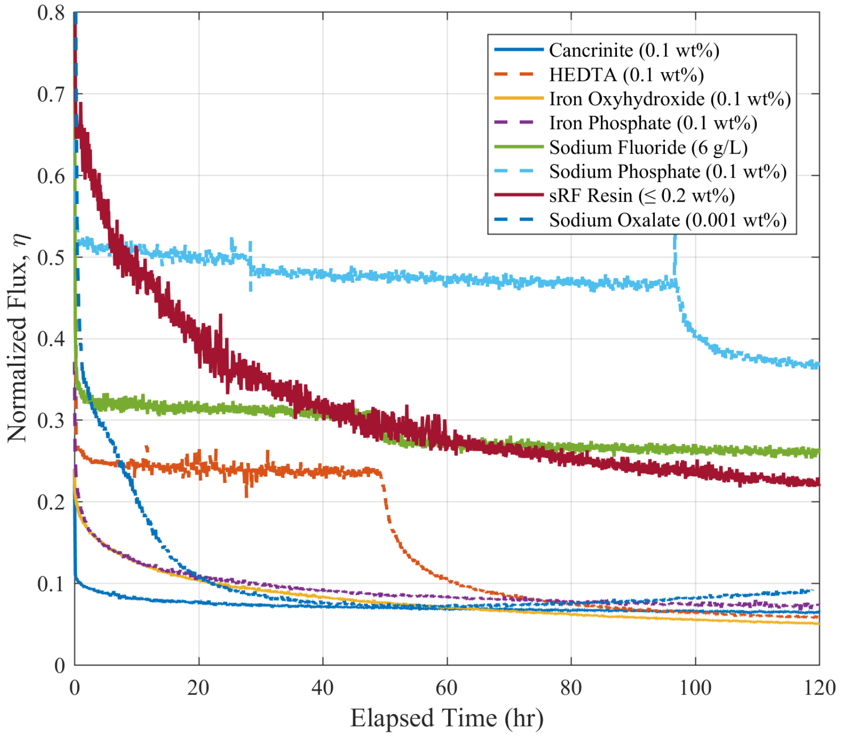

Figure 6.2. Normalized flux η measured for the eight fouling simulants during the initial (120-hour) baseline filtration period. Normalization was performed using Eq. 3.7.

fouling simulant test was conducted, influencing the fouling behavior of the experiment as these contaminants were slowly introduced into the bulk slurry. However, this information is anecdotal and difficult to verify. Lacking any other explanations, the fouling behavior of the sodium oxalate at a very low loading (0.001 wt%) cannot be dismissed as an anomaly and may need to be confirmed with additional testing.

The impact of the "worst" fouling simulants on LAWPS plant operations is best demonstrated by examining the volume filtered over the baseline period of operation. Recall that the volume filtered is calculated using Eq. 3.10. In Figure 6.4, V_F is shown over time compared to the nominal 5.6 M simulant test conducted at the same filtration conditions (the base case). Over 120 hours, the total volume filtered is reduced (with respect to the base case) by approximately a factor of 1.5 to 2 depending on the fouling agent. Nominally, it would be expected that to achieve the same throughput (V_F) when these fouling agents are present, the TMP would need to be increased to approximately twice that of the base case over the operating period. Longer periods of operation (greater than 120 hours) may require even greater increases in TMP depending on the foulant(s) present. For instance,the fluxes of both of the iron-containing simulants were still declining appreciably in the final hour of operation during the tests. CFF of slurries containing similar iron components is likely to experience similar flux decay even past the duration of the baseline period [see, for example Schonewill et al. (2015)] and would be expected to require mitigation, either by increasing TMP, backpulsing, or cleaning to maintain filter performance (target flux).

The data from the "worst" fouling simulants indicate, based on current equipment specifications (Aguilar 2016), that sufficient excess capacity exists in the LAWPS CFF system to achieve target processing rates. None of the fouling tests resulted in a flux that declined below the lower limit of operation for the LAWPS

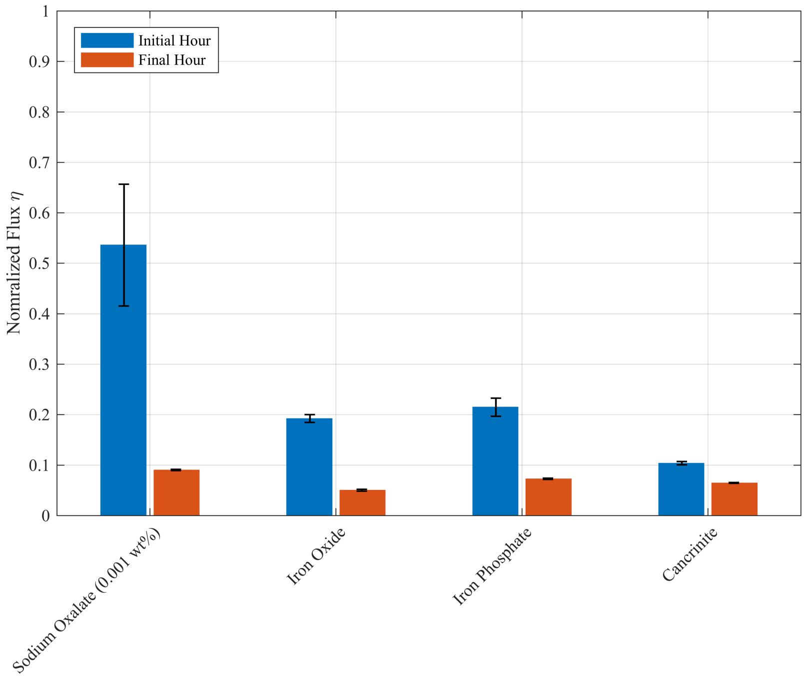

Figure 6.3. Normalized flux η for the four "worst" fouling simulants averaged over the initial and final hour of filtration in the 120-hour baseline filtration period. Error bars represent one standard deviation.

facility, e.g., 0.0075 $\mathrm{gpm\,ft^{-2}}$, while operating at a constant TMP of 20 psi for 120 hours. The CFF system in the recently conducted engineering-scale integrated testing had two filter modules capable of TMPs of 60 psi (Wilson 2017). Therefore, the CFF system should have the capability to process slurries with more severe fouling behavior than the "worst" fouling simulants by increasing TMP with time, assuming that some combination of various solid components present at low concentrations does not cause "extreme" fouling in the filter. Since all Series 2 fouling simulants were tested in isolation from other potential components, the probability of synergistic fouling can not be ruled out when considering worst-case CFF scenarios.

6.2 Flux Recovery Performance

The preceding section evaluated only the fouling behavior of Series 2 simulants over the baseline operating period absent attempts to recover lost flux. One important assessment conducted in the fouling tests was to establish if flux recovery strategies, namely backpulsing and LAWPS chemical cleaning with 0.45 M HNO_3 (see Section 3.6.3 for details), would mitigate the fouling. Despite the observation that even the fouling simulants were still in the LAWPS target range at the end of the baseline period, flux recovery methods may be needed to sustain performance at longer operating times. This section evaluates the effectiveness of these strategies at recovering flux, using the flux at the start of the 120-hour baseline filtration period as a reference. As described in Section 5.2, for purposes of the discussion in this section, "effective" flux recovery strategies are those that yield measured fluxes that meet or exceed the reference value measured at the start of testing.

Table 6.2 presents the percent of η restored in the first hour after the recovery strategy compared to the first hour of operation in the baseline period. By this measure, backpulsing is an inconsistent recovery

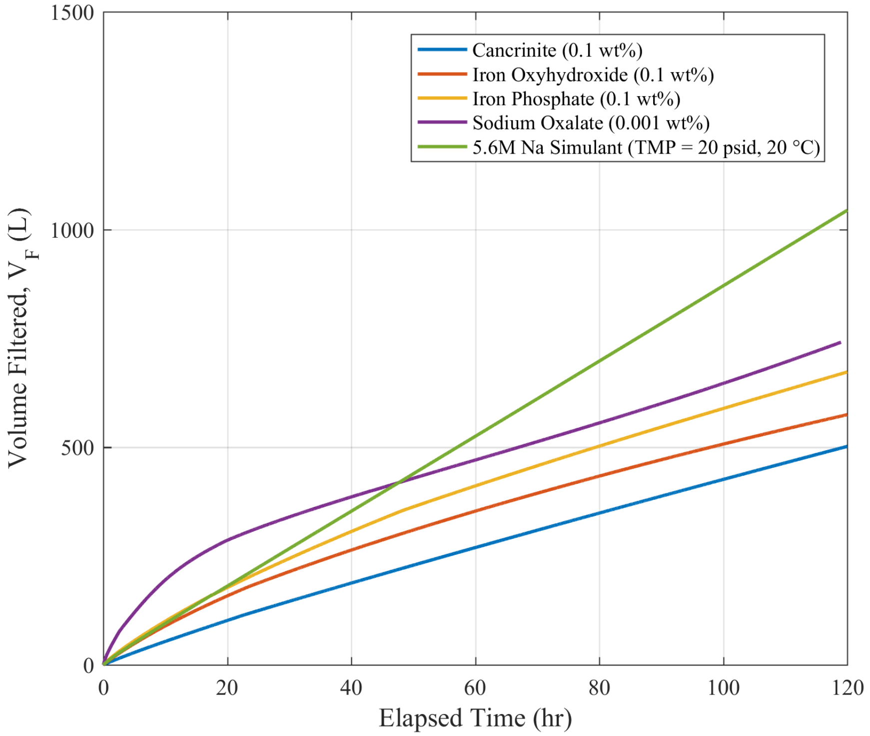

Figure 6.4. Volume of filtration V_F produced as a function of time for the four "worst" fouling simulants during the initial (120-hour) baseline filtration period. For comparison with a baseline test, the V_F measured for the 5.6 M simulant from Series 1, Test 1 is also shown.

technique: More than 75% of the normalized flux is restored in five of the tests, but in three tests it is $< 50\%$. One of those three tests (HEDTA, Test 6) has poor flux recovery primarily because the initial simulant contained only HEDTA, but at the time the single backpulse was applied, a small amount of iron oxide had also been added to the simulant. The initial baseline flux if the iron oxide had been present from the beginning with the HEDTA would have been lower; if this were the case, the post-backpulse flux recovery would have been larger.

The other two tests in question include one of the "worst" foulants (sodium oxalate, Test 1) and a material that was intended to (and appeared to) be gradually degraded during testing (sRF resin, Test 7). As mentioned previously, the sodium oxalate test was suspected of being contaminated by material from the test that preceded it, which was the sRF resin test. If this suspicion is accurate, degraded resin may have been fouled deep into the filter or even crossed through to the shell side. Backpulsing would not be expected to dislodge firmly bound particulate or force particulate to the tube side from the shell side. Assuming this material contaminated the sodium oxalate test, the same problem would have persisted in that test as well, especially if a sufficient amount of particulate was still on the filter shell side. On the other hand, that speculation may not be correct, in which case there are scenarios where backpulsing mitigates fouling much less effectively than others.

Table 6.2 also contains the flux recovery following LAWPS cleaning. The cleaning restores the flux completely for the majority of the simulants ($\eta > 90\%$) except for the HEDTA and sRF resin tests. The HEDTA test percent recovery value is impacted by the addition of iron oxide solids partway through the test, as dis-

Table 6.2. Recovery in normalized flux for the eight fouling simulants following a single backpulse and LAWPS cleaning.

Test	Description	Percent Recovery[(a)] (%)	
		Backpulse	LAWPS Cleaning
1	Sodium Oxalate (0.001 wt%)	32	172
2	Sodium Phosphate	80	107
3	Iron Oxide	77	94
4	Iron Phosphate	91	91
5	Cancrinite	106	159
6	HEDTA (with Iron Oxide)	46	55
7	Degraded sRF Resin	34	26
8	Sodium Fluoride	83	155

(a) Defined as average η in the first hour of the given operating period divided by average η in the first hour of the initial 120-hr filtration period ($\times 100$).

cussed before. The sRF resin test is an interesting case because the resin was suspended in 0.45 M HNO_3 during the testing, so the LAWPS acid cleaning used a fluid indistinguishable from the suspending phase of the simulant. Thus, it would not be expected to dissolve or clean any additional sRF resin particles from the filter if they were present. Assuming the other cleaning steps did not have a significant impact, then it seems very likely that sRF resin solids remained lodged in the filter pores during cleaning. Effectively, the filter was "pre-fouled" and the resultant post-LAWPS cleaning period flux was low, with a correspondingly poor flux recovery.

Aside from the two exceptions noted, the LAWPS cleaning approach was consistently successful in restoring the initial flux. In some instances, the flux after LAWPS cleaning was even greater than it was at the start of the baseline period. The apparent robustness of the cleaning method is notable since some of the fouling agents selected for testing have not always been readily cleaned via standard laboratory cleaning approaches in similar historical work. Figure 6.5 presents the values of uncorrected flux for the baseline, post-backpulse, and post-LAWPS cleaning filtration periods for all the fouling simulant tests.

It is instructive to consider how the backpulsing and cleaning strategies performed for the "worst" foulants, since the objective of using a mitigation approach is to combat the performance decline caused by filter fouling. Figure 6.6 contains the normalized flux averages for the "worst" foulants during the initial hour of the three relevant operating periods. The data clearly show how much flux change occurred during the baseline period for the sodium oxalate test compared to the other three tests; from this perspective, backpulsing does not grade as very effective for the sodium oxalate simulant versus the other three fouling simulants. Note, however, that the values of η for each simulant after backpulsing are very similar: approximately between $0.1 < \eta < 0.2$. Backpulsing, if performed at an appropriate fixed frequency, would likely be able to temporarily return the normalized flux to at least a value of 0.1 based on these data (assuming these "worst" fouling agents are bounding performers with respect to potential waste slurries to be filtered in LAWPS). Recall that other testing contradicts this statement (see Section 5.3), and some of the simulants that did not foul as significantly also exhibited some reduced flux recovery with backpulsing. Hence, the test data demonstrated that a backpulse is a useful approach for flux decline mitigation, but was not a panacea and would be best paired with another flux recovery strategy.

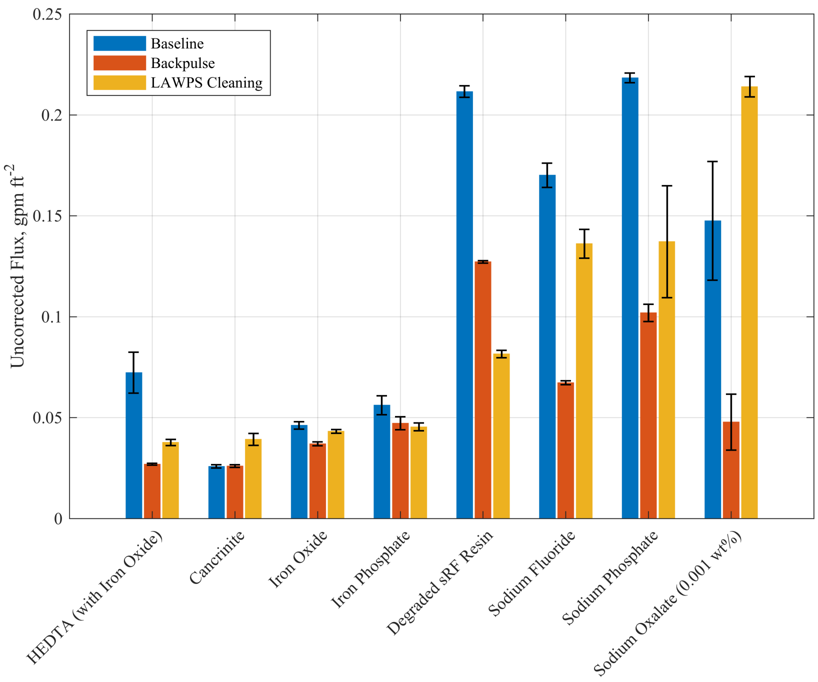

Figure 6.5. Uncorrected flux j for all eight fouling simulants measured following the start of testing, the single backpulse, and LAWPS cleaning. The error bars represent one standard deviation.

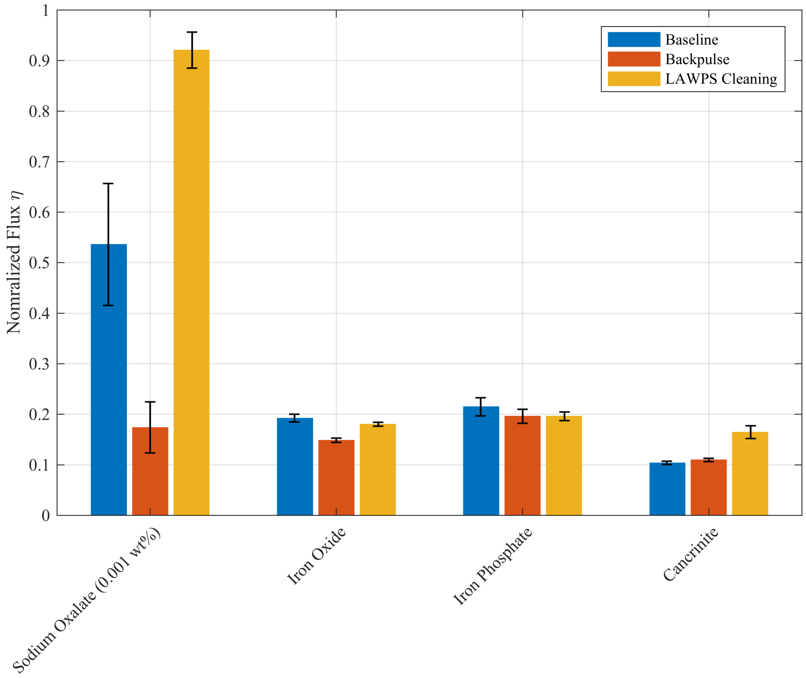

Figure 6.6. Normalized flux η for the "worst" fouling simulants measured following the start of testing, the single backpulse, and LAWPS cleaning. The error bars represent one standard deviation.

None of the four "worst" fouling simulants irreversibly fouled the filter to a degree that the LAWPS cleaning regimen could not restore flux to the original performance level. LAWPS cleaning appeared to be an effective alternative to traditional laboratory cleaning approaches; its effectiveness was probably derived from the relatively long contact time between the filter media and the 0.45 M HNO_3. The sodium oxalate case is once again an exception — the normalized flux was greatly increased compared to even the initial flux, with an $\eta > 0.9$. The unusual flux recovery for that test was further evidence that perhaps the sodium oxalate simulant had been contaminated, and only when a thorough cleaning approach (as the LAWPS cleaning cycle has been demonstrated to be) was applied did the simulant perform as it should have originally.

6.3 Key Observations

This section discussed the eight fouling simulants CFF tests conducted as part of Series 2 testing. The fouling simulant tests described in this section provided some insight on potentially troublesome solid components in LAWPS feeds that may challenge the CFF system, as well as informed how LAWPS operations might be impacted in the presence of hard-to-filter materials. From the information that was collected, the following key observations can be made:

- Four of the eight fouling simulants exhibited significant fouling over the 120-hour baseline operating period when compared to the filter flux obtained with the nominal 5.6 M simulant. These four "worst" fouling simulants contained 0.1 wt% undissolved solids, whereas the 5.6 M simulant had a higher loading of sodium oxalate solids (~ 0.45 wt%). The particle size of the tested solid compo-

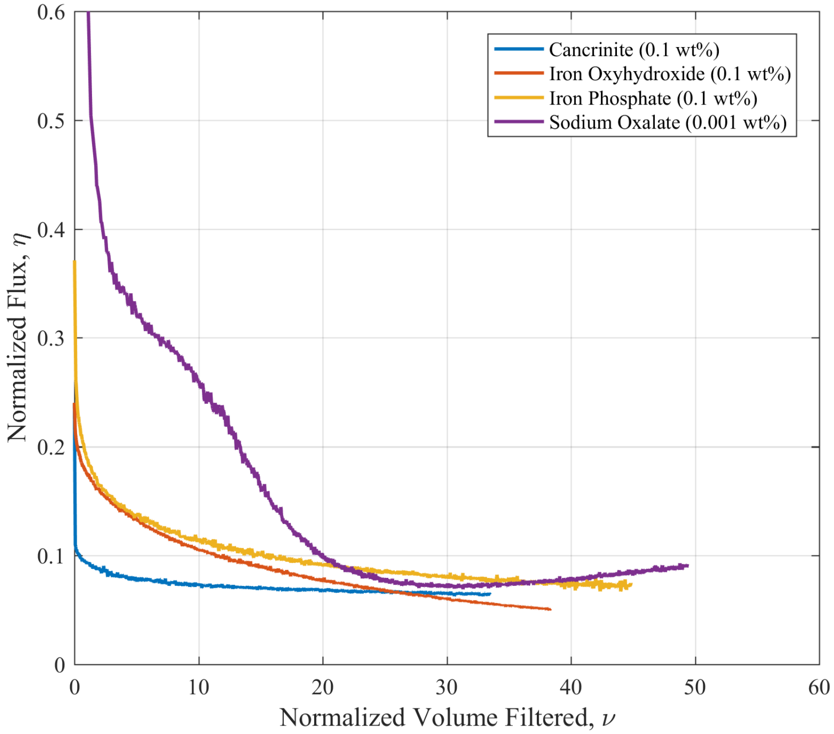

Figure 6.7. Normalized flux η versus normalized volume ν for the "worst" fouling simulants during the baseline filtration period.

nents (refer to Figure 4.3 or Appendix C) was not the sole factor in predicting which materials would foul more than others; the performance of the 4.0 M simulant (see Section 5.1) provided similar evidence that “small” solids are not always challenging to the filter. In general, the “worst” fouling agents had solids that were amorphous and were difficult to identify (once in solution) by XRD analysis.

- The measured uncorrected fluxes of the “worst” fouling simulants after 120-hours were similar in magnitude, but this masks some of the differences in flux behavior with time. Figure 6.7 shows the “worst” simulants on a normalized flux (η) and volume (ν) basis to better illustrate these differences. There is a clear separation in short-term flux decline between cancrinite (rapid), the insoluble iron components (modest), and sodium oxalate (slow). Due to the initially fast flux decay, cancrinite is the worst-case foulant with respect to throughput over the baseline period. This is reflected in the cancrinite η achieving the lowest value of ν compared to the other simulants. Amorphous iron oxide is the fouling agent still exhibiting the largest change in flux with time, i.e., the greatest slope, after 120 hours. If the trend were to continue, the amorphous iron oxide would be the the most adverse to longer-term performance and throughput. The sodium oxalate has several unusual features, including a flux recovery that occurred lacking any known cause starting at around $\nu = 25$.

- Given the observations on the differences between the transient behavior of the “worst” fouling simulants, the insoluble solid components recommended as the most challenging materials to be encountered in the LAWPS CFF system are cancrinite and amorphous iron oxide (with iron phosphate as a suitable alternative due to its similar performance). The sodium oxalate data collected at low concentration were unusual and further study is recommended before sodium oxalate is identified as a challenging material.

- Even though significant foulants were identified, the most significant fouling simulants tested were still within the performance limits (0.0075 to 0.032 $\text{gpm}\,\text{ft}^{-2}$) of the LAWPS facility after 120 hours of operation at a constant TMP of 20 psi. Consequently, waste material that filters like the fouling simulants tested herein would be expected to meet CFF production targets in the LAWPS facility. It is also likely that operation for longer than 120 hours would remain within the capability of the LAWPS CFF system, especially given the ability to access larger TMP over time and possibly conduct periodic backpulses.

- The effect of undissolved solids concentration was not examined in the Series 2 testing. The majority of the tests were conducted at a fixed solids loading of 0.1 wt%, and demonstrated that fouling can occur at this relatively low undissolved solids concentration. This observation is also supported by recent waste testing with AP-105 supernate described in Russell et al. (2017a). In LAWPS operations, undissolved solids concentrations up to 3.3 wt% are permitted, and this may increase the rate or extent of fouling. The expectation is that flux decline at higher solids concentration is more likely to be due to the development of a filter cake and thus could likely be disrupted by periodic backpulsing.

- Backpulsing was a recovery tool with varying degrees of effectiveness, ranging from approximately 30% to 100% flux recovery depending on the simulant being tested. The presence of sodium oxalate and/or iron-containing insoluble solids generally reduced the effectiveness of backpulsing. Operational variations may improve flux recovery — for example, the backpulse could be coupled with a reduction in TMP in order to limit the fouling rate immediately after the backpulse is completed.

- LAWPS cleaning was effective at restoring the filter flux to its initial performance level across the breadth of fouling simulants tested, and was routinely more effective than standard laboratory cleaning protocols. This is similar to the observation based on testing described in Section 5. The lone exception was Test 7 (LPS-T2S2-NA-RF-01): It is uncertain how effective the protocol would be at dissolving degraded sRF resin if any were present in the LAWPS system. Note that though the volumes of cleaning solutions delivered in the current testing were scaled to the prototypic full-scale LAWPS conditions, the delivery was performed in an idealized configuration that is likely to be more efficient than the LAWPS facility. The bench-scale CFF system lacked the hold-up volume of the full-scale system and drained the slurry lines of residual material prior to the start of cleaning.

7.0 Conclusions and Recommendations

This report described CFF testing of four LAWPS simulants and eight fouling simulants to support engineering evaluations of the sustainability of LAWPS filter operations and associated scaled demonstration tests conducted at the ITF. Bench-scale testing resulted in several key observations relevant to LAWPS filter operations:

- The sustainability of CFF operations depends on feed chemistry and filter operating parameters, including but not limited to the suspending phase viscosity (with higher viscosities yielding lower performance), the fouling proclivity of the waste solids (which appears strongly dependent on solid phase chemistry), and the pressure profile used to control flux (where the most sustainable filter operations are those that use the minimum pressure possible to effect the desired flux). However, filter performance cannot be readily predicted from a single chemical or operational metric, such as the particle size of insoluble solids or the total sodium molarity.

- Waste materials that filter like the four baseline LAWPS simulants should not challenge the sustainability of LAWPS filter operations during 120 hours of continuous filtration and longer (given operational strategies that avoid fouling, such as using the lowest TMP possible to achieve the desired target flux). Similarly, waste materials like any of the eight fouling simulants, which included some materials that were more challenging to filter than the baseline simulants, should also be within the capabilities of LAWPS filter operations. It should be noted that the four baseline LAWPS simulants all exhibit relative high fluxes relative to actual waste [see Russell et al. (2017a)], and as such, are not expected to be as challenging to filter as actual waste feeds. The fouling simulants exhibit fouling behaviors similar to that of actual wastes [cf. Edwards et al. (2009), Fiskum et al. (2008), Fiskum et al. (2009), Lumetta et al. (2009), Shimskey et al. (2009a), Shimskey et al. (2009b), and Snow et al. (2009)].

- Backpusling can be an effective means of flux recovery; however, its effectiveness appears to depend strongly on the chemistry of the fouling solids. In the current testing, mediocre to poor recovery was observed for simulants containing sodium oxalate and amorphous iron oxide solids, and repeated, frequent backpulsing generally accelerated loss of filter performance.

- For the test materials evaluated in this report, LAWPS cleaning appears to be an effective means of lost flux recovery. Indeed, LAWPS cleaning protocols were frequently as effective as, if not more effective than, standard laboratory cleaning protocols used to clean the filter before the start of each test and that have enabled previous bench-scale CFF test campaigns conducted at PNNL to re-use the same filter element without any cumulative loss in performance over several months of testing. This conclusion should be approached with some caution, as the studies evaluated LAWPS cleaning in a highly idealized configuration that allowed efficient removal of the test simulant from the recirculation and permeate collection lines before displacement with 0.1 M NaOH solution and that minimized potential hold-up of test material throughout the system. Hold-up of waste is expected in the full-scale LAWPS CFF system, and contact of the waste residual with cleaning solutions could lead to the formation of precipitates that are stable in the subsequent cleaning solutions and/or that embed in the filter elements, reducing the flux recovery effected by cleaning. Likewise, the CUF and full-scale LAWPS filtration and cleaning systems operate at vastly different scales, and how well the LAWPS cleaning protocol scales must be evaluated before final conclusions on the effectiveness of LAWPS cleaning can be made.

- Use of a single constant transmembrane of 20 psi for bench-scale testing should yield filter fouling that bounds the permeate production rate realized during constant flux filtration operations (like that planned for LAWPS operations) so long as the fluxes realized in bench-scale testing meet or exceed the LAWPS flux target of 0.0075 to 0.032 gpm ft^{-2}. This requirement is satisfied for the four LAWPS simulants and eight fouling simulants tested in the current report. As such, the filter performance data generated herein are relevant to LAWPS testing.

Given the underlying complexity of Hanford wastes (and chemical simulants developed to replicate wastes), the potential range of operating parameters, and significant processing time required to evaluate the filtration performance of a single combination of simulant and operating parameters, baseline (Series 1) and fouling (Series 2) filter tests left several open issues whose resolution could improve understanding of the potential operating challenges and their associated mitigation strategies for LAWPS filtration. These issues and their potential impacts are discussed below, along with recommended additional engineering evaluations or follow-on testing (as applicable). Issues recommended for resolution and future CFF testing include:

- The current testing identified a highly variant fouling proclivity for simulants rich in sodium oxalate solids. While the test results clearly indicate limited flux recovery for filters fouled by oxalate-rich simulants, the magnitude of flux varied significantly for even nominally equivalent tests (such as the initial and repeat tests of 5.6 M Na simulant filtration performance at 45 °C). Uncertainty with respect to sodium oxalate's fouling proclivity is further increased by the anomalous filtration results observed during low undissolved solids (0.001 wt%) fouling tests with sodium oxalate (as discussed in Section 6.1 of this report). Increased confidence in the sustainability of LAWPS filter operations, which may frequently handle sodium-oxalate-rich waste feeds, may be gained by isolating the origin of the variance observed in sodium oxalate testing. For this reason, follow-on testing that seeks to better understand test-to-test filter performance reproducibility for the 5.6 M Na simulant as a function of process variables and simulant batch preparation is recommended.

- In order to control the number of test variables explored in this study and focus on relatively low solids concentration, which is the expectation for the LAWPS facility, the effect of solids concentration was not addressed. However, CFF operation allows for solids concentrations up to 3.3 wt%. Some of the most challenging fouling agents may have a more adverse effect at solids loadings > 0.1 wt%, the level at which they were tested. Testing the cancrinite and amorphous iron oxide solids at higher solids concentrations is recommended to address this uncertainty.

- The vast majority of simulants tested in this study had a single solid phase component (or targeted a single solid phase). This was done to isolate the behavior of individual solids that may be found in potential LAWPS waste feeds. Mixtures of solid components are highly probable and expected in the LAWPS feeds. Past work has shown that only a few components typically "control" the filtration behavior, but it is possible that a mixture of solids exists in potential waste feeds that is more challenging with respect to filtration performance than a single solid component. Testing one or two more complex solids mixtures would reduce the uncertainty of unexpected extreme synergistic fouling occurring during LAWPS processing.

- The LAWPS facility physical properties limits permit a suspending phase with a viscosity as high as 15 mPa s [refer to Russell et al. (2017b)]. Of all simulants tested in the current report, the most viscous simulant (i.e., the 8.0 M simulant) still has a viscosity that is approximately a factor of two lower than the upper LAWPS limit of 15 mPa s. Moreover, the 8.0 M simulant yielded the lowest uncorrected filter flux of all simulants tested. The filter performance of a 15 mPa s simulant with similar fouling proclivity to that of the 8.0 M simulant would likely show even lower filter performance that could challenge LAWPS flux operational targets. Testing a simulant with the upper LAWPS limit viscosity would provide additional confidence in the ability of the LAWPS CFF system to maintain production rate targets. In fact, a waste simulant with a viscosity targeting that upper limit was recently developed at PNNL and would be a good candidate for high-viscosity CFF testing.[1]

- The impact of residual waste (i.e., waste hold-up) on LAWPS cleaning efficacy was not addressed in the current testing. The PNNL CFF test system is designed to allow near-complete removal of test material from the recirculation and permeate collection lines. Hold-up of residual waste is expected in the full-scale LAWPS system and could lead to precipitation of solids during cleaning that limit the effectiveness of the LAWPS cleaning protocol. To resolve this issue, direct evaluations of hold-up on LAWPS cleaning effectiveness should be performed through additional bench-scale testing, scaled LAWPS testing at the ITF, or some combination of both.

- Off-normal events or failure of isolation valves during waste processing could also lead to inadvertent contact between waste and cleaning solutions (e.g., 0.45 M HNO_3), leading to the formation of precipitates that could degrade filter performance. Bench-scale CFF testing to date has not evaluated the consequence of direct waste and cleaning solution contact. Bench-scale testing of the fouling consequence of direct mixing of a LAWPS simulant and 0.45 M HNO_3 is recommended to better understand the consequence of such mixing on the filter performance and the ability to recover from such an off-normal event.

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Appendix A

LAWPS Filtration Simulants CFF Performance Data

Appendix A – LAWPS Filtration Simulants CFF Performance Data

This appendix provides a detailed summary of filter performance data collected during Series 1 crossflow filtration (CFF) testing. Table A.1 lists the Series 1 test matrix. All tests were performed except Test 6 [a stepped flux test with the 8.0 M (high) Na simulant at 20 °C]. Test data are provided in both graphical and tabular forms. The graphical summaries have been marked with the specific Series 1 test periods as follows:

Segment A: 120 hr of continuous filtration at constant transmembrane pressure (TMP) (i.e., 20 psid)

Segment B: 24 hr of continuous filtration at constant TMP following a single backpulse operation

Segment C: backpulsed operations (one backpulse every 30 min for 8 hr) followed by a 16 hr period of continuous filtration at constant TMP

Segment D: Low-Activity Waste Pretreatment System (LAWPS) prototypic[1] cleaning with displacement by 0.1 M NaOH, rinsing with process water, cleaning with 0.45 M HNO_3 (nitric acid), and final rinsing with process water

Segment E: 24 hr of continuous filtration at constant TMP following LAWPS cleaning

Segment F: Post-test clean water flux measurements and standardized chemical cleaning operations with 2 M HNO_3 (nitric acid) and 0.5 M $H_2C_2O_4$ (oxalic acid) solutions

Tabular results consider the filter performance and operational conditions during the 120-hour period of continuous filtration (Segment A), after the single backpulse (Segment B), and after LAWPS cleaning (Segment E). To provide a gross measure of flux reduction from fouling of the filter element with solids, the filter performance is provided for both the initial and final hour of filtration in the given filtration period. Flux is presented as uncorrected flux (j), temperature-corrected flux (j_c), and TMP- and temperature-corrected flux ($j_{p,c}$). All uncertainties reported in the tabular results are standard deviations and are provided in reduced form [e.g., 1.005 ± 0.001 is reported as 1.005(1)].

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[1] Although the graphical results in the overview section show measurable flux during the LAWPS cleaning segment, these flux measurements should be ignored as they correspond to the rate of cleaning solution delivery to the filter rather than any true permeate flux.

Table A.1. Series 1 test matrix.

Test	Test ID	Simulant	Pressure Profile[(a)]	Test Temp. (°C)
1	LPS-T2S1-NC20-01	5.6 M Na	Constant (20 psi)	20
2	LPS-T2S1-NC45-01	5.6 M Na	Constant (20 psi)	45
3	LPS-T2S1-HC20-01	8.0 M Na	Constant (20 psi	20
4	LPS-T2S1-NS20-01	5.6 M Na	Stepped TMP (10-30 psi)[(b)]	20
5	LPS-T2S1-NS20-02	5.6 M Na	Stepped Flux (0.03 gpm ft^{-2})[(c)]	20
6	LPS-T2S1-HS20-01	8.0 M Na	Stepped Flux (0.03 gpm ft^{-2})[(c,d)]	20
7	LPS-T2S1-4C20-01	4.0 M Na	Constant (20 psi)	20
8	LPS-T2S1-6C20-01	6.0 M Na	Constant (20 psi)	20
9	LPS-T2S1-NC45-02	5.6 M Na	Constant (20 psi)	45

(a) Tests use one of three pressure profile schemes: 1) constant pressure, 2) stepped pressure with fixed pressure targets (i.e., stepped TMP), and 3) stepped pressure with fixed flux targets (i.e., stepped flux).

(b) Pressure is stepped 5 psi after each 24-hour filtration period.

(c) Stepped pressure range to be determined by flux target of 0.03 gpm ft^{-2} after each TMP change if feasible.

(d) Test was not conducted due to results of other Series 1 tests.

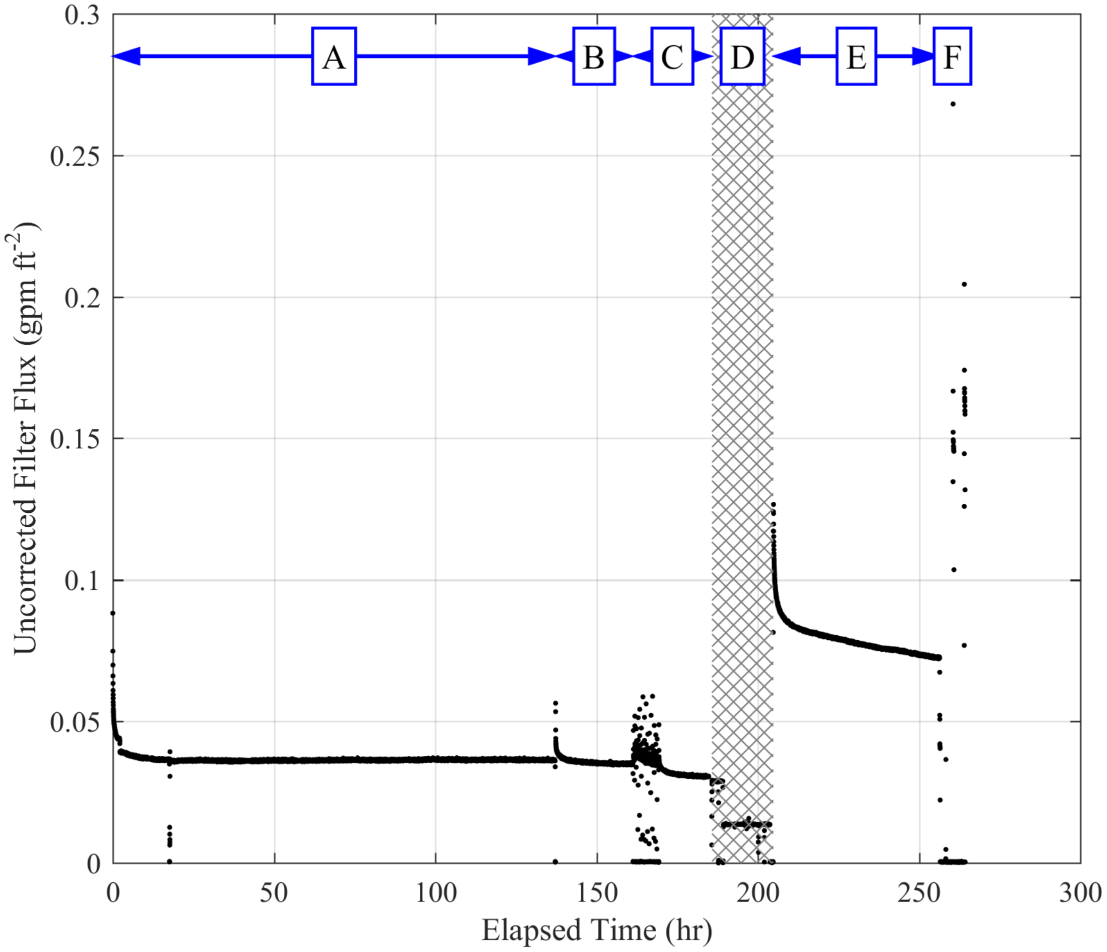


Figure A.1. Test overview for Series 1 Test 1 (LPS-T2S1-NC20-01): a performance test with a 5.6 M (nominal) Na simulant at a constant transmembrane pressure of 20 psid and 20 °C.

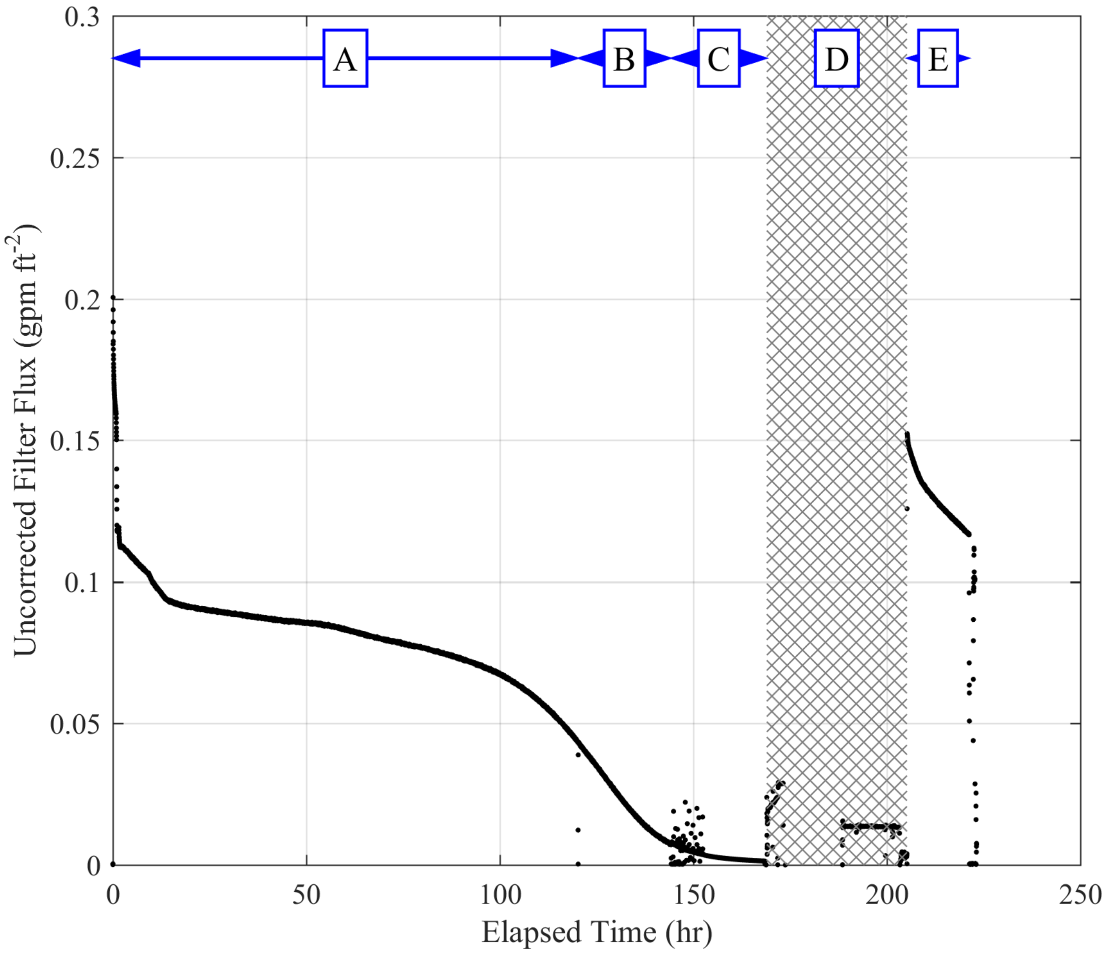


Figure A.2. Test overview for Series 1 Test 2 (LPS-T2S1-NC45-01): a performance test with a 5.6 M (nominal) Na simulant at a constant transmembrane pressure of 20 psid and 45 °C (initial test).

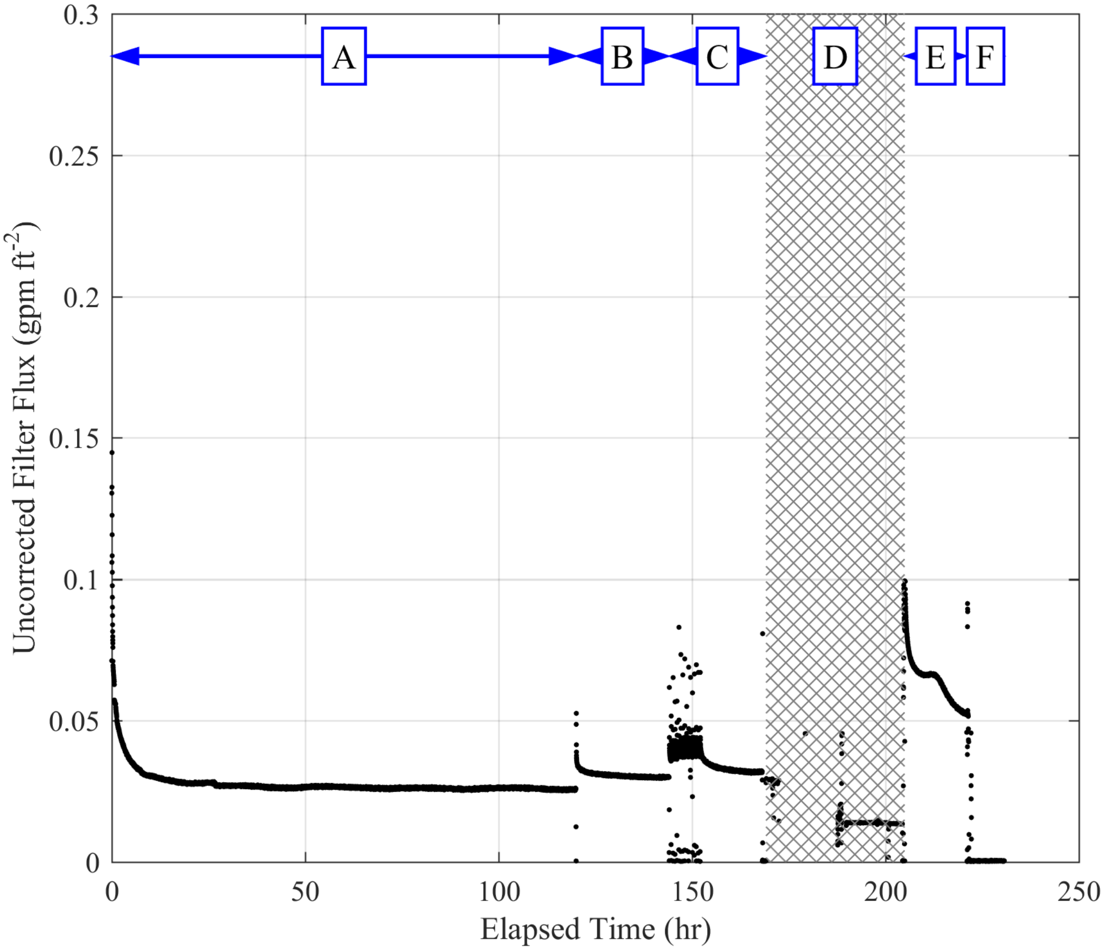


Figure A.3. Test overview for Series 1 Test 3 (LPS-T2S1-HC20-01): a performance test with a 8.0 M (high) Na simulant at a constant transmembrane pressure of 20 psid and 20 °C.

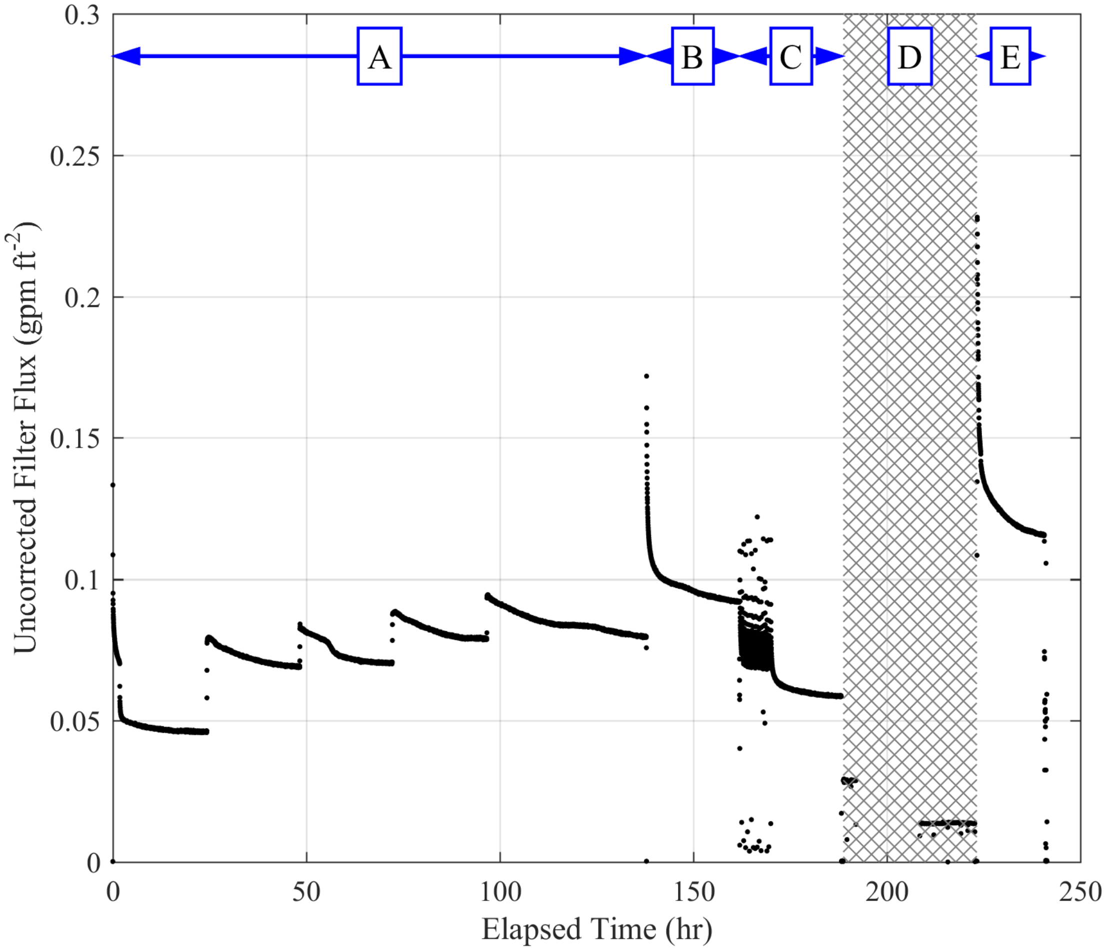


Figure A.4. Test overview for Series 1 Test 4 (LPS-T2S1-NS20-01): a performance test with a 5.6 M (nominal) Na simulant with a stepped transmembrane pressure (10 -30 psid) at 20 °C.

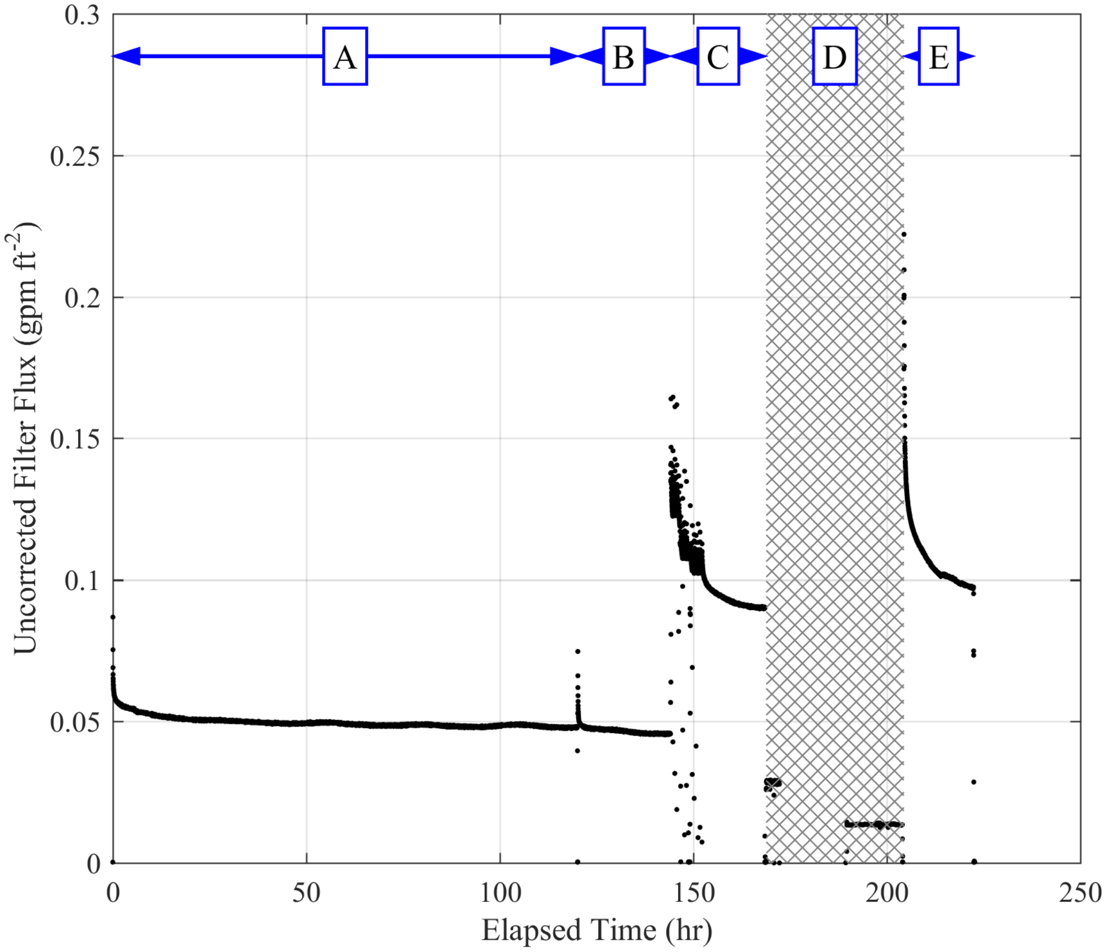


Figure A.5. Test overview for Series 1 Test 5 (LPS-T2S1-NS20-02): a performance test with a 5.6 M (nominal) Na simulant with a stepped flux (0.03 gpm ft^{-2}) at 20 °C.

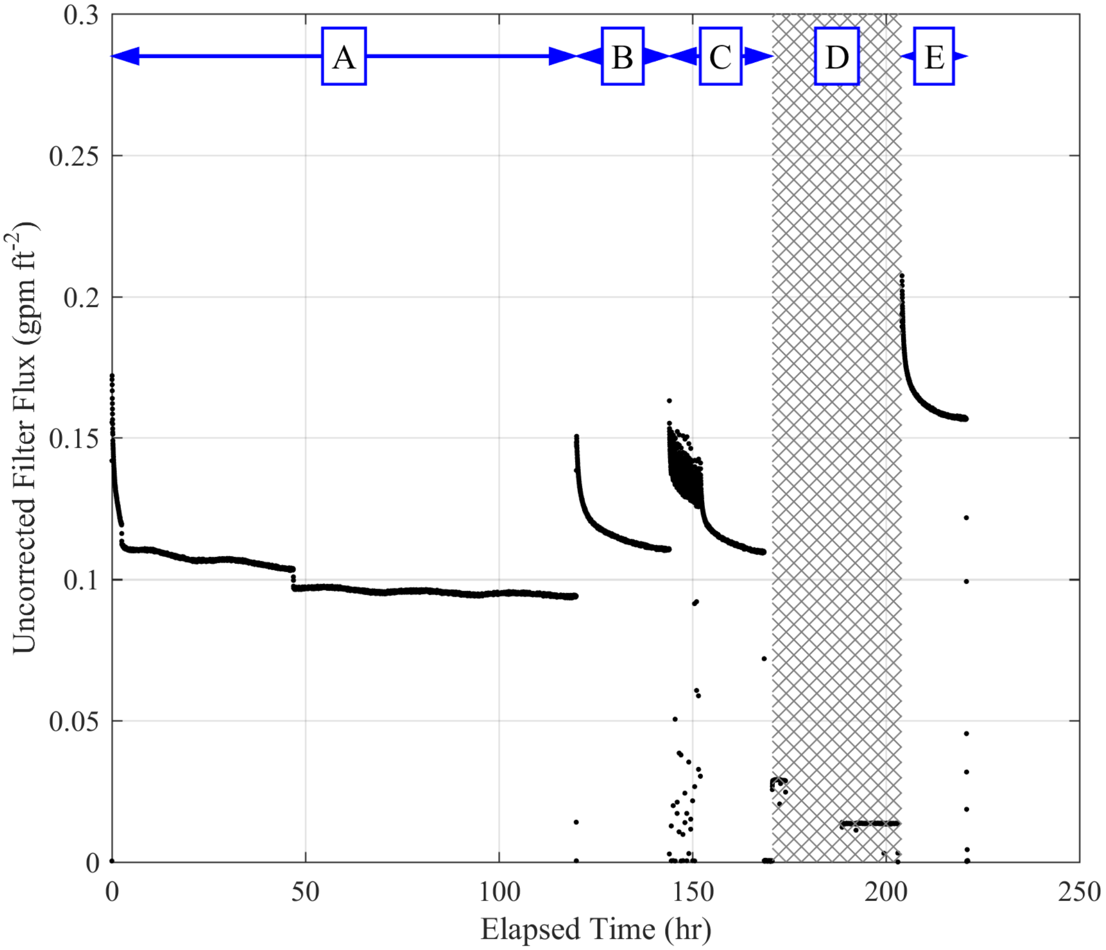


Figure A.6. Test overview for Series 1 Test 7 (LPS-T2S1-4C20-01): a performance test with a 4.0 M Na simulant at a constant transmembrane pressure of 20 psid and 20 °C.

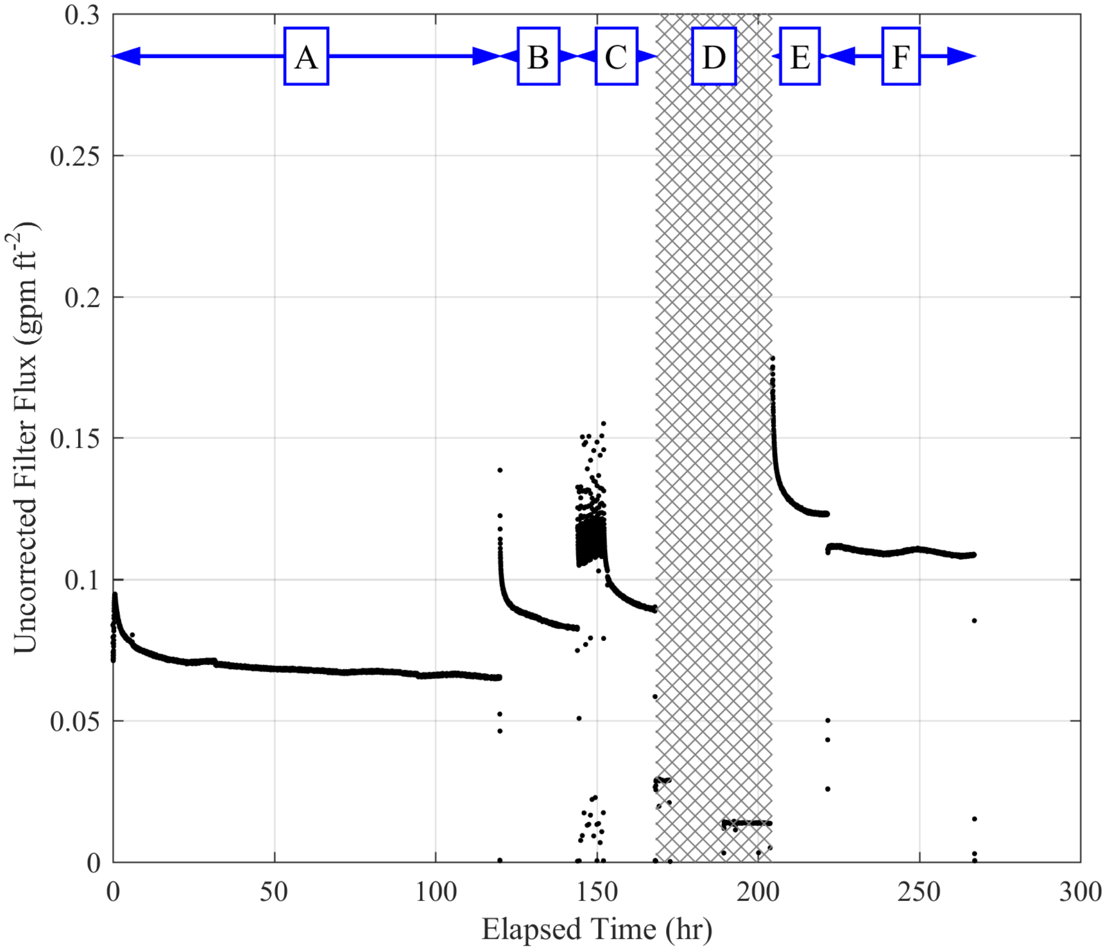


Figure A.7. Test overview for Series 1 Test 8 (LPS-T2S1-6C20-01): a performance test with a 6.0 M Na simulant at a constant transmembrane pressure of 20 psid and 20 °C.

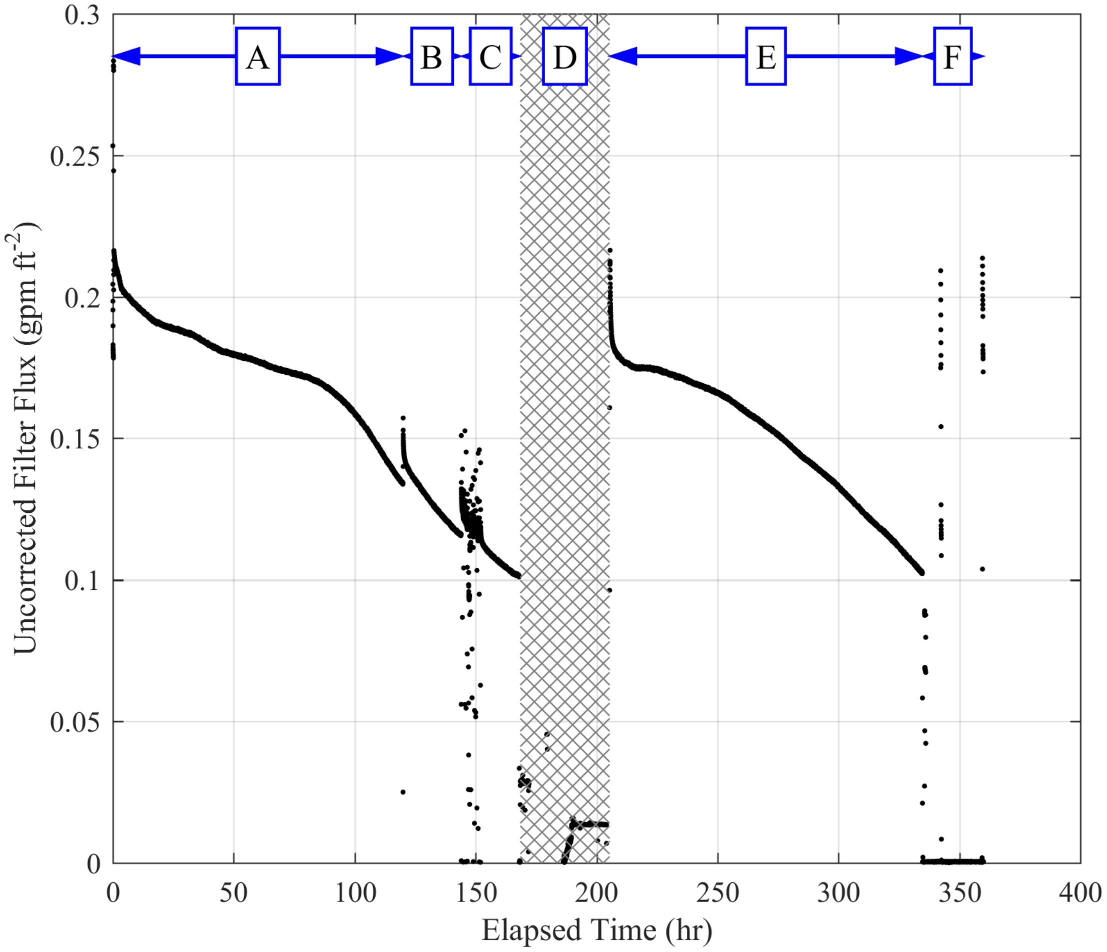


Figure A.8. Test overview for Series 1 Test 9 (LPS-T2S1-NC45-02): a performance test with a 5.6 M (nominal) Na simulant at a constant transmembrane pressure of 20 psid and 45 °C (replicate test).

Table A.2. Operational summary for Series 1 Test 1 (LPS-T2S1-NC20-01): a performance test with a 5.6 M (nominal) Na simulant at a constant transmembrane pressure of 20 psid and 20 °C.

Parameter	**Symbol**	**Unit**	**Initial Hour$^{(a)}$**	**Final Hour$^{(b)}$**
Initial 120 hr Filtration Period				
Inlet Pressure	p_i	psig	28.7(3)	27.6(3)
Outlet Pressure	p_o	psig	18.7(2)	16.98(7)
Permeate Pressure	p_f	psig	2.5(1)	2.1(3)
TMP	δp_t	psid	21.2(3)	20.2(4)
Axial Velocity	u	$\mathrm{ft\,s^{-1}}$	14.48(2)	14.71(2)
Slurry Temperature	T	°C	21.7(5)	19.71(1)
Flux (Uncorrected)	j	$\mathrm{gpm\,ft^{-2}}$	0.049(4)	0.036(5)
Flux (Corrected)	j_c	$\mathrm{gpm\,ft^{-2}}$	0.054(4)	0.042(5)
Flux (Corrected)	$j_{p,c}$	$\mathrm{gpm\,ft^{-2}}$	0.051(4)	0.041(5)
Post-Single Backpulse Filtration Period				
Inlet Pressure	p_i	psig	26.3(2)	26.4(3)
Outlet Pressure	p_o	psig	15.74(7)	15.76(7)
Permeate Pressure	p_f	psig	2.25(3)	2.13(2)
TMP	δp_t	psid	18.8(1)	18.9(2)
Axial Velocity	u	$\mathrm{ft\,s^{-1}}$	14.81(1)	14.79(2)
Slurry Temperature	T	°C	19.6962	19.73(3)
Flux (Uncorrected)	j	$\mathrm{gpm\,ft^{-2}}$	0.039(1)	0.0350(2)
Flux (Corrected)	j_c	$\mathrm{gpm\,ft^{-2}}$	0.046(1)	0.0407(3)
Flux (Corrected)	$j_{p,c}$	$\mathrm{gpm\,ft^{-2}}$	0.049(1)	0.0430(5)
Post-Multiple Backpulse Filtration Period				
Inlet Pressure	p_i	psig	26.7(3)	27.0(2)
Outlet Pressure	p_o	psig	16.22(7)	16.44(7)
Permeate Pressure	p_f	psig	2.04(2)	2.01(1)
TMP	δp_t	psid	19.4(2)	19.7(1)
Axial Velocity	u	$\mathrm{ft\,s^{-1}}$	14.76(1)	14.76(1)
Slurry Temperature	T	°C	20.14(9)	19.71(2)
Flux (Uncorrected)	j	$\mathrm{gpm\,ft^{-2}}$	0.0339(7)	0.0306
Flux (Corrected)	j_c	$\mathrm{gpm\,ft^{-2}}$	0.0390(9)	0.0356(1)
Flux (Corrected)	$j_{p,c}$	$\mathrm{gpm\,ft^{-2}}$	0.040(1)	0.0360(2)
Post-LAWPS Cleaning Filtration Period				
Inlet Pressure	p_i	psig	30.9(4)	31.1(7)
Outlet Pressure	p_o	psig	21.1(1)	21.5(1)
Permeate Pressure	p_f	psig	4.6(4)	3.34(1)
TMP	δp_t	psid	21.4(6)	22.9(3)
Axial Velocity	u	$\mathrm{ft\,s^{-1}}$	14.49(2)	14.45(1)
Slurry Temperature	T	°C	20.0(2)	20.24(4)
Flux (Uncorrected)	j	$\mathrm{gpm\,ft^{-2}}$	0.101(8)	0.0725(2)
Flux (Corrected)	j_c	$\mathrm{gpm\,ft^{-2}}$	0.12(1)	0.0831(2)
Flux (Corrected)	$j_{p,c}$	$\mathrm{gpm\,ft^{-2}}$	0.11(1)	0.072(1)

(a) Measurements averaged over the initial hour of the given filtration period.
(b) Measurements averaged over the final hour of the given filtration period.

Table A.3. Operational summary for Series 1 Test 2 (LPS-T2S1-NC45-01): a performance test with a 5.6 M (nominal) Na simulant at a constant transmembrane pressure of 20 psid and 45 °C (initial test).

Parameter	**Symbol**	**Unit**	**Initial Hour(a)**	**Final Hour(b)**
Initial 120 hr Filtration Period				
Inlet Pressure	p_i	psig	33.0(9)	28.9(4)
Outlet Pressure	p_o	psig	25(1)	19.98(6)
Permeate Pressure	p_f	psig	6.3(7)	2.067(8)
TMP	δp_t	psid	22.6(8)	22.4(2)
Axial Velocity	u	$\mathrm{ft\,s^{-1}}$	14.54(8)	14.66(1)
Slurry Temperature	T	°C	47(1)	44.69(4)
Flux (Uncorrected)	j	$\mathrm{gpm\,ft^{-2}}$	0.16(1)	0.0443(5)
Flux (Corrected)	j_c	$\mathrm{gpm\,ft^{-2}}$	0.092(9)	0.0264(3)
Flux (Corrected)	$j_{p,c}$	$\mathrm{gpm\,ft^{-2}}$	0.08(1)	0.0236(3)
Post-Single Backpulse Filtration Period				
Inlet Pressure	p_i	psig	28.1(3)	28.2(4)
Outlet Pressure	p_o	psig	18.99(6)	19.24(7)
Permeate Pressure	p_f	psig	1.99(2)	1.30(2)
TMP	δp_t	psid	21.6(2)	22.4(2)
Axial Velocity	u	$\mathrm{ft\,s^{-1}}$	14.74(1)	14.71(1)
Slurry Temperature	T	°C	44.57(7)	45.13(5)
Flux (Uncorrected)	j	$\mathrm{gpm\,ft^{-2}}$	0.0423(6)	0.0078(3)
Flux (Corrected)	j_c	$\mathrm{gpm\,ft^{-2}}$	0.0252(4)	0.0046(2)
Flux (Corrected)	$j_{p,c}$	$\mathrm{gpm\,ft^{-2}}$	0.0234(3)	0.0041(1)
Post-Multiple Backpulse Filtration Period				
Inlet Pressure	p_i	psig	28.5(5)	28.8(5)
Outlet Pressure	p_o	psig	19.27(9)	19.53(6)
Permeate Pressure	p_f	psig	1.85(2)	1.47(2)
TMP	δp_t	psid	22.1(3)	22.7(2)
Axial Velocity	u	$\mathrm{ft\,s^{-1}}$	14.73(1)	14.71(1)
Slurry Temperature	T	°C	45.52(8)	45.42(9)
Flux (Uncorrected)	j	$\mathrm{gpm\,ft^{-2}}$	0.0034(1)	0.0014
Flux (Corrected)	j_c	$\mathrm{gpm\,ft^{-2}}$	0.002	0.0008
Flux (Corrected)	$j_{p,c}$	$\mathrm{gpm\,ft^{-2}}$	0.0018	0.0007
Post-LAWPS Cleaning Filtration Period				
Inlet Pressure	p_i	psig	29.6(2)	30.1(5)
Outlet Pressure	p_o	psig	20.71(7)	20.99(8)
Permeate Pressure	p_f	psig	5.7(1)	4.32(2)
TMP	δp_t	psid	19.5(2)	21.2(3)
Axial Velocity	u	$\mathrm{ft\,s^{-1}}$	14.62(1)	14.57(1)
Slurry Temperature	T	°C	44.7(2)	44.97(9)
Flux (Uncorrected)	j	$\mathrm{gpm\,ft^{-2}}$	0.148(2)	0.1174(4)
Flux (Corrected)	j_c	$\mathrm{gpm\,ft^{-2}}$	0.088(2)	0.0694(3)
Flux (Corrected)	$j_{p,c}$	$\mathrm{gpm\,ft^{-2}}$	0.090(2)	0.065(1)

(a) Measurements averaged over the initial hour of the given filtration period.
(b) Measurements averaged over the final hour of the given filtration period.

Table A.4. Operational summary for Series 1 Test 3 (LPS-T2S1-HC20-01): a performance test with a 8.0 M (high) Na simulant at a constant transmembrane pressure of 20 psid and 20 °C.

Parameter	**Symbol**	**Unit**	**Initial Hour**[(a)]	**Final Hour**[(b)]
Initial 120 hr Filtration Period				
Inlet Pressure	p_i	psig	32(2)	29.5(5)
Outlet Pressure	p_o	psig	19(2)	17.09(3)
Permeate Pressure	p_f	psig	5(1)	2.326(6)
TMP	δp_t	psid	20(1)	21.0(2)
Axial Velocity	u	$\mathrm{ft\,s^{-1}}$	14.7(1)	14.63(2)
Slurry Temperature	T	°C	21.13(8)	21.11(3)
Flux (Uncorrected)	j	$\mathrm{gpm\,ft^{-2}}$	0.07(2)	0.0257(2)
Flux (Corrected)	j_c	$\mathrm{gpm\,ft^{-2}}$	0.08(2)	0.0287(2)
Flux (Corrected)	$j_{p,c}$	$\mathrm{gpm\,ft^{-2}}$	0.07(2)	0.0274(4)
Post-Single Backpulse Filtration Period				
Inlet Pressure	p_i	psig	28.7(4)	29.0(2)
Outlet Pressure	p_o	psig	16.05(4)	16.09(5)
Permeate Pressure	p_f	psig	2.76(4)	2.54(1)
TMP	δp_t	psid	19.6(2)	20.0(1)
Axial Velocity	u	$\mathrm{ft\,s^{-1}}$	14.67(1)	14.67(1)
Slurry Temperature	T	°C	21.12(4)	21.3(1)
Flux (Uncorrected)	j	$\mathrm{gpm\,ft^{-2}}$	0.0338(8)	0.030(1)
Flux (Corrected)	j_c	$\mathrm{gpm\,ft^{-2}}$	0.038(1)	0.0333(2)
Flux (Corrected)	$j_{p,c}$	$\mathrm{gpm\,ft^{-2}}$	0.039(1)	0.0334(2)
Post-Multiple Backpulse Filtration Period				
Inlet Pressure	p_i	psig	28(1)	28.3(5)
Outlet Pressure	p_o	psig	15.38(5)	15.66(6)
Permeate Pressure	p_f	psig	2.85(7)	2.61(1)
TMP	δp_t	psid	18.6(5)	19.4(3)
Axial Velocity	u	$\mathrm{ft\,s^{-1}}$	14.67(1)	14.70(2)
Slurry Temperature	T	°C	22.211(3)	21.48(2)
Flux (Uncorrected)	j	$\mathrm{gpm\,ft^{-2}}$	0.037(1)	0.0319(2)
Flux (Corrected)	j_c	$\mathrm{gpm\,ft^{-2}}$	0.041(1)	0.0353(2)
Flux (Corrected)	$j_{p,c}$	$\mathrm{gpm\,ft^{-2}}$	0.044(2)	0.0364(5)
Post-LAWPS Cleaning Filtration Period				
Inlet Pressure	p_i	psig	30.1(9)	31.3(2)
Outlet Pressure	p_o	psig	18(1)	18.80(5)
Permeate Pressure	p_f	psig	5.2(3)	3.57(2)
TMP	δp_t	psid	19(1)	21.5(1)
Axial Velocity	u	$\mathrm{ft\,s^{-1}}$	14.50(6)	14.54(2)
Slurry Temperature	T	°C	22.1(4)	21.13(1)
Flux (Uncorrected)	j	$\mathrm{gpm\,ft^{-2}}$	0.082(6)	0.0527(3)
Flux (Corrected)	j_c	$\mathrm{gpm\,ft^{-2}}$	0.089(6)	0.0589(3)
Flux (Corrected)	$j_{p,c}$	$\mathrm{gpm\,ft^{-2}}$	0.10(1)	0.0549(4)

(a) Measurements averaged over the initial hour of the given filtration period.
(b) Measurements averaged over the final hour of the given filtration period.

Table A.5. Operational summary for Series 1 Test 4 (LPS-T2S1-NS20-01): a performance test with a 5.6 M (nominal) Na simulant with a stepped transmembrane pressure (10 -30 psid) at 20 °C.

Parameter	Symbol	Unit	Initial Hour[(a)]	Final Hour[(b)]
Initial 120 hr Filtration Period				
Inlet Pressure	p_i	psig	19.8(3)	39.3(5)
Outlet Pressure	p_o	psig	9.7(3)	29.94(7)
Permeate Pressure	p_f	psig	3.6(2)	3.63(1)
TMP	δp_t	psid	11.1(5)	31.0(3)
Axial Velocity	u	$\mathrm{ft\,s^{-1}}$	14.60(2)	14.36(2)
Slurry Temperature	T	°C	20.0(1)	20.47(3)
Flux (Uncorrected)	j	$\mathrm{gpm\,ft^{-2}}$	0.079(5)	0.0797(2)
Flux (Corrected)	j_c	$\mathrm{gpm\,ft^{-2}}$	0.091(5)	0.0908(2)
Flux (Corrected)	$j_{p,c}$	$\mathrm{gpm\,ft^{-2}}$	0.16(2)	0.0586(5)
Post-Single Backpulse Filtration Period				
Inlet Pressure	p_i	psig	41.8(4)	41.7(3)
Outlet Pressure	p_o	psig	32.27(7)	32.71(6)
Permeate Pressure	p_f	psig	5.3(4)	4.12(2)
TMP	δp_t	psid	31.7(6)	33.1(2)
Axial Velocity	u	$\mathrm{ft\,s^{-1}}$	14.18(1)	14.14(2)
Slurry Temperature	T	°C	20.6(1)	20.61(5)
Flux (Uncorrected)	j	$\mathrm{gpm\,ft^{-2}}$	0.118(9)	0.0921(2)
Flux (Corrected)	j_c	$\mathrm{gpm\,ft^{-2}}$	0.13(1)	0.1044(2)
Flux (Corrected)	$j_{p,c}$	$\mathrm{gpm\,ft^{-2}}$	0.084(8)	0.0632(4)
Post-Multiple Backpulse Filtration Period				
Inlet Pressure	p_i	psig	27.3(5)	27.8(5)
Outlet Pressure	p_o	psig	17.15(6)	17.32(4)
Permeate Pressure	p_f	psig	3.2(1)	2.83(1)
TMP	δp_t	psid	19.0(3)	19.7(2)
Axial Velocity	u	$\mathrm{ft\,s^{-1}}$	14.79(1)	14.80(1)
Slurry Temperature	T	°C	20.03(3)	19.68(1)
Flux (Uncorrected)	j	$\mathrm{gpm\,ft^{-2}}$	0.068(3)	0.0587(1)
Flux (Corrected)	j_c	$\mathrm{gpm\,ft^{-2}}$	0.079(4)	0.0683(2)
Flux (Corrected)	$j_{p,c}$	$\mathrm{gpm\,ft^{-2}}$	0.083(5)	0.0693(8)
Post-LAWPS Cleaning Filtration Period				
Inlet Pressure	p_i	psig	33(1)	32.5(5)
Outlet Pressure	p_o	psig	24(1)	21.99(5)
Permeate Pressure	p_f	psig	8(1)	5.07(2)
TMP	δp_t	psid	21(1)	22.1(2)
Axial Velocity	u	$\mathrm{ft\,s^{-1}}$	14.60(8)	14.80(1)
Slurry Temperature	T	°C	22.8(6)	21.80(9)
Flux (Uncorrected)	j	$\mathrm{gpm\,ft^{-2}}$	0.17(2)	0.1158(2)
Flux (Corrected)	j_c	$\mathrm{gpm\,ft^{-2}}$	0.18(2)	0.1268(4)
Flux (Corrected)	$j_{p,c}$	$\mathrm{gpm\,ft^{-2}}$	0.17(3)	0.115(1)

(a) Measurements averaged over the initial hour of the given filtration period.
(b) Measurements averaged over the final hour of the given filtration period.

Table A.6. Operational summary for Series 1 Test 5 (LPS-T2S1-NS20-02): a performance test with a 5.6 M (nominal) Na simulant with a stepped flux (0.03 $\mathrm{gpm\,ft^{-2}}$) at 20 °C.

Parameter	**Symbol**	**Unit**	**Initial Hour**[a]	**Final Hour**[b]
Initial 120 hr Filtration Period				
Inlet Pressure	p_i	psig	15.56(7)	16.0(1)
Outlet Pressure	p_o	psig	5.11(2)	5.18(2)
Permeate Pressure	p_f	psig	2.89(8)	2.52(1)
TMP	δp_t	psid	7.4(1)	8.05(7)
Axial Velocity	u	$\mathrm{ft\,s^{-1}}$	14.68(2)	14.81(1)
Slurry Temperature	T	°C	20.1(2)	19.8(1)
Flux (Uncorrected)	j	$\mathrm{gpm\,ft^{-2}}$	0.059(2)	0.0479(1)
Flux (Corrected)	j_c	$\mathrm{gpm\,ft^{-2}}$	0.068(3)	0.0555(2)
Flux (Corrected)	$j_{p,c}$	$\mathrm{gpm\,ft^{-2}}$	0.18(1)	0.138(1)
Post-Single Backpulse Filtration Period				
Inlet Pressure	p_i	psig	15.6(2)	15.6(2)
Outlet Pressure	p_o	psig	5.09(2)	5.11(3)
Permeate Pressure	p_f	psig	2.58(8)	2.44(2)
TMP	δp_t	psid	7.8(1)	7.93(9)
Axial Velocity	u	$\mathrm{ft\,s^{-1}}$	14.699(8)	14.71(1)
Slurry Temperature	T	°C	19.9(1)	19.41(6)
Flux (Uncorrected)	j	$\mathrm{gpm\,ft^{-2}}$	0.050(2)	0.0456(2)
Flux (Corrected)	j_c	$\mathrm{gpm\,ft^{-2}}$	0.058(3)	0.0536(2)
Flux (Corrected)	$j_{p,c}$	$\mathrm{gpm\,ft^{-2}}$	0.149(8)	0.135(2)
Post-Multiple Backpulse Filtration Period				
Inlet Pressure	p_i	psig	31.3(3)	32.1(2)
Outlet Pressure	p_o	psig	21.11(6)	21.91(4)
Permeate Pressure	p_f	psig	4.72(2)	4.31(2)
TMP	δp_t	psid	21.5(1)	22.7(1)
Axial Velocity	u	$\mathrm{ft\,s^{-1}}$	14.50(1)	14.48(2)
Slurry Temperature	T	°C	21.18(8)	20.13(9)
Flux (Uncorrected)	j	$\mathrm{gpm\,ft^{-2}}$	0.101(2)	0.0902(2)
Flux (Corrected)	j_c	$\mathrm{gpm\,ft^{-2}}$	0.113(2)	0.1037(4)
Flux (Corrected)	$j_{p,c}$	$\mathrm{gpm\,ft^{-2}}$	0.105(2)	0.0915(5)
Post-LAWPS Cleaning Filtration Period				
Inlet Pressure	p_i	psig	30.0(7)	30.1(3)
Outlet Pressure	p_o	psig	20.0(7)	19.82(4)
Permeate Pressure	p_f	psig	6.3(7)	4.31(1)
TMP	δp_t	psid	18.7(4)	20.6(1)
Axial Velocity	u	$\mathrm{ft\,s^{-1}}$	14.61(6)	14.72(1)
Slurry Temperature	T	°C	20.7(4)	20.48(3)
Flux (Uncorrected)	j	$\mathrm{gpm\,ft^{-2}}$	0.14(1)	0.0974(2)
Flux (Corrected)	j_c	$\mathrm{gpm\,ft^{-2}}$	0.16(1)	0.1108(3)
Flux (Corrected)	$j_{p,c}$	$\mathrm{gpm\,ft^{-2}}$	0.17(2)	0.1073(8)

(a) Measurements averaged over the initial hour of the given filtration period.
(b) Measurements averaged over the final hour of the given filtration period.

Table A.7. Operational summary for Series 1 Test 7 (LPS-T2S1-4C20-01): a performance test with a 4.0 M Na simulant at a constant transmembrane pressure of 20 psid and 20 °C.

Parameter	Symbol	Unit	Initial Hour[(a)]	Final Hour[(b)]
Initial 120 hr Filtration Period				
Inlet Pressure	p_i	psig	29.2(5)	28.8(3)
Outlet Pressure	p_o	psig	20.9(2)	20.10(6)
Permeate Pressure	p_f	psig	5.4(5)	3.528(9)
TMP	δp_t	psid	19.7(8)	20.9(2)
Axial Velocity	u	$\mathrm{ft\,s^{-1}}$	14.29(2)	14.65(2)
Slurry Temperature	T	°C	20.5(2)	19.35(2)
Flux (Uncorrected)	j	$\mathrm{gpm\,ft^{-2}}$	0.14(1)	0.0940(2)
Flux (Corrected)	j_c	$\mathrm{gpm\,ft^{-2}}$	0.16(1)	0.1105(2)
Flux (Corrected)	$j_{p,c}$	$\mathrm{gpm\,ft^{-2}}$	0.17(2)	0.1057(8)
Post-Single Backpulse Filtration Period				
Inlet Pressure	p_i	psig	29.3(4)	29.3(3)
Outlet Pressure	p_o	psig	20.73(5)	20.83(5)
Permeate Pressure	p_f	psig	5.2(2)	4.11(2)
TMP	δp_t	psid	19.8(4)	21.0(1)
Axial Velocity	u	$\mathrm{ft\,s^{-1}}$	14.60(1)	14.58(1)
Slurry Temperature	T	°C	19.6(1)	19.72(2)
Flux (Uncorrected)	j	$\mathrm{gpm\,ft^{-2}}$	0.137(5)	0.1106(1)
Flux (Corrected)	j_c	$\mathrm{gpm\,ft^{-2}}$	0.161(7)	0.1286(2)
Flux (Corrected)	$j_{p,c}$	$\mathrm{gpm\,ft^{-2}}$	0.16(1)	0.1227(9)
Post-Multiple Backpulse Filtration Period				
Inlet Pressure	p_i	psig	29.3(4)	29.4(5)
Outlet Pressure	p_o	psig	20.57(4)	21.0(5)
Permeate Pressure	p_f	psig	4.7(1)	4.07(1)
TMP	δp_t	psid	20.2(3)	21.1(2)
Axial Velocity	u	$\mathrm{ft\,s^{-1}}$	14.60(1)	14.59(1)
Slurry Temperature	T	°C	20.11(4)	19.337(3)
Flux (Uncorrected)	j	$\mathrm{gpm\,ft^{-2}}$	0.125(3)	0.1097(2)
Flux (Corrected)	j_c	$\mathrm{gpm\,ft^{-2}}$	0.144(4)	0.1290(2)
Flux (Corrected)	$j_{p,c}$	$\mathrm{gpm\,ft^{-2}}$	0.142(6)	0.122(1)
Post-LAWPS Cleaning Filtration Period				
Inlet Pressure	p_i	psig	31.4(3)	32.0(3)
Outlet Pressure	p_o	psig	23.2(1)	23.84(4)
Permeate Pressure	p_f	psig	7.6(5)	6.01(2)
TMP	δp_t	psid	19.8(5)	21.9(1)
Axial Velocity	u	$\mathrm{ft\,s^{-1}}$	14.27(1)	14.31(2)
Slurry Temperature	T	°C	19.9(1)	19.88(7)
Flux (Uncorrected)	j	$\mathrm{gpm\,ft^{-2}}$	0.184(8)	0.1568(2)
Flux (Corrected)	j_c	$\mathrm{gpm\,ft^{-2}}$	0.213(8)	0.1815(5)
Flux (Corrected)	$j_{p,c}$	$\mathrm{gpm\,ft^{-2}}$	0.22(1)	0.166(1)

(a) Measurements averaged over the initial hour of the given filtration period.
(b) Measurements averaged over the final hour of the given filtration period.

Table A.8. Operational summary for Series 1 Test 8 (LPS-T2S1-6C20-01): a performance test with a 6.0 M Na simulant at a constant transmembrane pressure of 20 psid and 20 °C.

Parameter	Symbol	Unit	Initial Hour(a)	Final Hour(b)
Initial 120 hr Filtration Period				
Inlet Pressure	p_i	psig	28.34(9)	30.2(3)
Outlet Pressure	p_o	psig	17.72(9)	19.66(5)
Permeate Pressure	p_f	psig	4.3(3)	3.32(1)
TMP	δp_t	psid	18.7(3)	21.6(2)
Axial Velocity	u	$\mathrm{ft\,s^{-1}}$	14.49(2)	14.58(2)
Slurry Temperature	T	°C	19.7(7)	19.36(2)
Flux (Uncorrected)	j	$\mathrm{gpm\,ft^{-2}}$	0.088(8)	0.0652(2)
Flux (Corrected)	j_c	$\mathrm{gpm\,ft^{-2}}$	0.102(8)	0.0766(2)
Flux (Corrected)	$j_{p,c}$	$\mathrm{gpm\,ft^{-2}}$	0.11(1)	0.0708(5)
Post-Single Backpulse Filtration Period				
Inlet Pressure	p_i	psig	29.9(2)	30.3(6)
Outlet Pressure	p_o	psig	19.41(8)	19.56(7)
Permeate Pressure	p_f	psig	4.9(2)	4.07(1)
TMP	δp_t	psid	19.7(2)	20.9(3)
Axial Velocity	u	$\mathrm{ft\,s^{-1}}$	14.63(2)	14.61(2)
Slurry Temperature	T	°C	19.340(8)	19.32(1)
Flux (Uncorrected)	j	$\mathrm{gpm\,ft^{-2}}$	0.10(4)	0.0828(2)
Flux (Corrected)	j_c	$\mathrm{gpm\,ft^{-2}}$	0.118(4)	0.0974(2)
Flux (Corrected)	$j_{p,c}$	$\mathrm{gpm\,ft^{-2}}$	0.119(6)	0.093(1)
Post-Multiple Backpulse Filtration Period				
Inlet Pressure	p_i	psig	29.2(5)	29.7(6)
Outlet Pressure	p_o	psig	18.93(9)	18.8(1)
Permeate Pressure	p_f	psig	5.3(4)	4.40(2)
TMP	δp_t	psid	18.8(5)	19.8(3)
Axial Velocity	u	$\mathrm{ft\,s^{-1}}$	14.64(1)	14.66(1)
Slurry Temperature	T	°C	20.37(4)	19.57(4)
Flux (Uncorrected)	j	$\mathrm{gpm\,ft^{-2}}$	0.110(7)	0.0893(2)
Flux (Corrected)	j_c	$\mathrm{gpm\,ft^{-2}}$	0.126(8)	0.1043(2)
Flux (Corrected)	$j_{p,c}$	$\mathrm{gpm\,ft^{-2}}$	0.13(1)	0.105(2)
Post-LAWPS Cleaning Filtration Period				
Inlet Pressure	p_i	psig	32.6(3)	33.5(4)
Outlet Pressure	p_o	psig	22.9(2)	23.72(6)
Permeate Pressure	p_f	psig	7.3(5)	5.83(1)
TMP	δp_t	psid	20.4(7)	22.8(2)
Axial Velocity	u	$\mathrm{ft\,s^{-1}}$	14.15(2)	14.20(2)
Slurry Temperature	T	°C	20.08(2)	20.06(2)
Flux (Uncorrected)	j	$\mathrm{gpm\,ft^{-2}}$	0.15(1)	0.1231(2)
Flux (Corrected)	j_c	$\mathrm{gpm\,ft^{-2}}$	0.17(1)	0.1418(2)
Flux (Corrected)	$j_{p,c}$	$\mathrm{gpm\,ft^{-2}}$	0.17(2)	0.124(1)

(a) Measurements averaged over the initial hour of the given filtration period.
(b) Measurements averaged over the final hour of the given filtration period.

Table A.9. Operational summary for Series 1 Test 9 (LPS-T2S1-NC45-02): a performance test with a 5.6 M (nominal) Na simulant at a constant transmembrane pressure of 20 psid and 45 °C (replicate test).

Parameter	**Symbol**	**Unit**	**Initial Hour**[(a)]	**Final Hour**[(b)]
		Initial 120 hr Filtration Period		
Inlet Pressure	p_i	psig	27(3)	28.6(8)
Outlet Pressure	p_o	psig	19(3)	20.77(7)
Permeate Pressure	p_f	psig	9(2)	4.92(3)
TMP	δp_t	psid	14(2)	19.8(4)
Axial Velocity	u	$\mathrm{ft\,s^{-1}}$	14.2(4)	14.36(2)
Slurry Temperature	T	°C	44.8(5)	44.852(5)
Flux (Uncorrected)	j	$\mathrm{gpm\,ft^{-2}}$	0.21(2)	0.1346(3)
Flux (Corrected)	j_c	$\mathrm{gpm\,ft^{-2}}$	0.13(1)	0.0798(2)
Flux (Corrected)	$j_{p,c}$	$\mathrm{gpm\,ft^{-2}}$	0.19(2)	0.081(2)
		Post-Single Backpulse Filtration Period		
Inlet Pressure	p_i	psig	28.3(5)	29.2(5)
Outlet Pressure	p_o	psig	19.99(5)	20.37(5)
Permeate Pressure	p_f	psig	5.3(1)	4.17(2)
TMP	δp_t	psid	18.8(2)	20.6(2)
Axial Velocity	u	$\mathrm{ft\,s^{-1}}$	14.46(3)	14.45(1)
Slurry Temperature	T	°C	44.87(5)	44.65(8)
Flux (Uncorrected)	j	$\mathrm{gpm\,ft^{-2}}$	0.144(2)	0.1162(3)
Flux (Corrected)	j_c	$\mathrm{gpm\,ft^{-2}}$	0.085(1)	0.0692(3)
Flux (Corrected)	$j_{p,c}$	$\mathrm{gpm\,ft^{-2}}$	0.090(2)	0.0671(8)
		Post-Multiple Backpulse Filtration Period		
Inlet Pressure	p_i	psig	28.4(6)	28.6(5)
Outlet Pressure	p_o	psig	19.73(7)	20.03(7)
Permeate Pressure	p_f	psig	4.06(5)	3.65(2)
TMP	δp_t	psid	20.0(3)	20.6(3)
Axial Velocity	u	$\mathrm{ft\,s^{-1}}$	14.49(2)	14.49(2)
Slurry Temperature	T	°C	45.19(6)	44.87(2)
Flux (Uncorrected)	j	$\mathrm{gpm\,ft^{-2}}$	0.114(1)	0.1017(2)
Flux (Corrected)	j_c	$\mathrm{gpm\,ft^{-2}}$	0.0668(8)	0.0602(1)
Flux (Corrected)	$j_{p,c}$	$\mathrm{gpm\,ft^{-2}}$	0.067(1)	0.0583(7)
		Post-LAWPS Cleaning Filtration Period		
Inlet Pressure	p_i	psig	28(1)	30.7(4)
Outlet Pressure	p_o	psig	20(1)	22.09(5)
Permeate Pressure	p_f	psig	7.9(6)	3.82(2)
TMP	δp_t	psid	16(2)	22.6(2)
Axial Velocity	u	$\mathrm{ft\,s^{-1}}$	14.46(7)	14.42(2)
Slurry Temperature	T	°C	44.7(3)	45.33(6)
Flux (Uncorrected)	j	$\mathrm{gpm\,ft^{-2}}$	0.193(9)	0.1030(3)
Flux (Corrected)	j_c	$\mathrm{gpm\,ft^{-2}}$	0.115(6)	0.0603(2)
Flux (Corrected)	$j_{p,c}$	$\mathrm{gpm\,ft^{-2}}$	0.15(3)	0.0534(5)

(a) Measurements averaged over the initial hour of the given filtration period.
(b) Measurements averaged over the final hour of the given filtration period.

Appendix B

LAWPS Fouling Simulants CFF Performance Data

Appendix B – LAWPS Fouling Simulants CFF Performance Data

This appendix provides a detailed summary of filter performance data collected during Series 2 crossflow filtration (CFF) testing. Table B.1 lists the Series 2 test matrix. Test data are presented in the same graphical and tabular formats described in Appendix A of this report.

Table B.1. Series 2 test matrix.

Test	ID	Foulant	Conc.	Temp.	Purpose
1	LPS-T2S2-MS-LO-01	Sodium Oxalate in MSS(a)	0.001 wt% UDS(b)	20 °C	Evaluate filtration at low UDS content.
2	LPS-T2S2-MS-SP-01	Sodium Phosphate in MSS(a)	0.1 wt% UDS(b)	45 °C	Evaluate filtration and LAWPS cleaning with known Hanford waste soluble solids and LAWPS cleaning.
3	LPS-T2S2-MS-IO-01	Iron Oxyhydroxide in MSS(a)	0.1 wt% UDS(b)	20 °C	Evaluate filtration and LAWPS cleaning with known Hanford waste insoluble solids.
4	LPS-T2S2-MS-IP-01	Iron Phosphate in MSS(a)	0.1 wt% UDS(b)	20 °C	Evaluate filtration and LAWPS cleaning with known Hanford waste insoluble solids.
5	LPS-T2S2-MS-CN-01	Cancrinite in MSS(a)	0.1 wt% UDS(b)	20 °C	Evaluate filtration and LAWPS cleaning with known Hanford waste insoluble solids.
6	LPS-T2S2-MS-HE-01	HEDTA in MSS(a,c)	0.1 wt% TS(e)	20 °C	Evaluate filtration and LAWPS cleaning with anticipated Hanford waste soluble organics.
7	LPS-T2S2-NA-RF-01	Degraded sRF Resin in 0.45 M HNO$_3$(d,f)	0.1 wt% UDS(b,g)	20 °C	Evaluate filtration and LAWPS cleaning with potential LAWPS degradation products.
8	LPS-T2S2-MS-SF-01	Sodium Fluoride in MSS(a,h)	6 g L^{-1} TS(e)	45 °C	Evaluate filtration and LAWPS cleaning with changing chemistry and precipitation.

(a) MSS — modified nominal (5.6 M) sodium simulant.
(b) UDS — indicates undissolved solids concentration and represents an excess of soluble component above its solubility limit.
(c) HEDTA — N-hydroxyethyl-ethylenediamine-triacetic acid.
(d) sRF — spherical resorcinol-formaldehyde.
(e) TS — indicates a total solids concentration (including both soluble and insoluble) added to simulant suspending phase.
(f) Resin degradation was accomplished before testing by suspending resin in 0.45 M HNO$_3$ and heating to approximately 50 °C for 24 hours.
(g) Concentration of undegraded resin added to 0.45 M HNO$_3$ suspending phase.
(h) Solid added to simulant 10 minutes after the start of filtration.

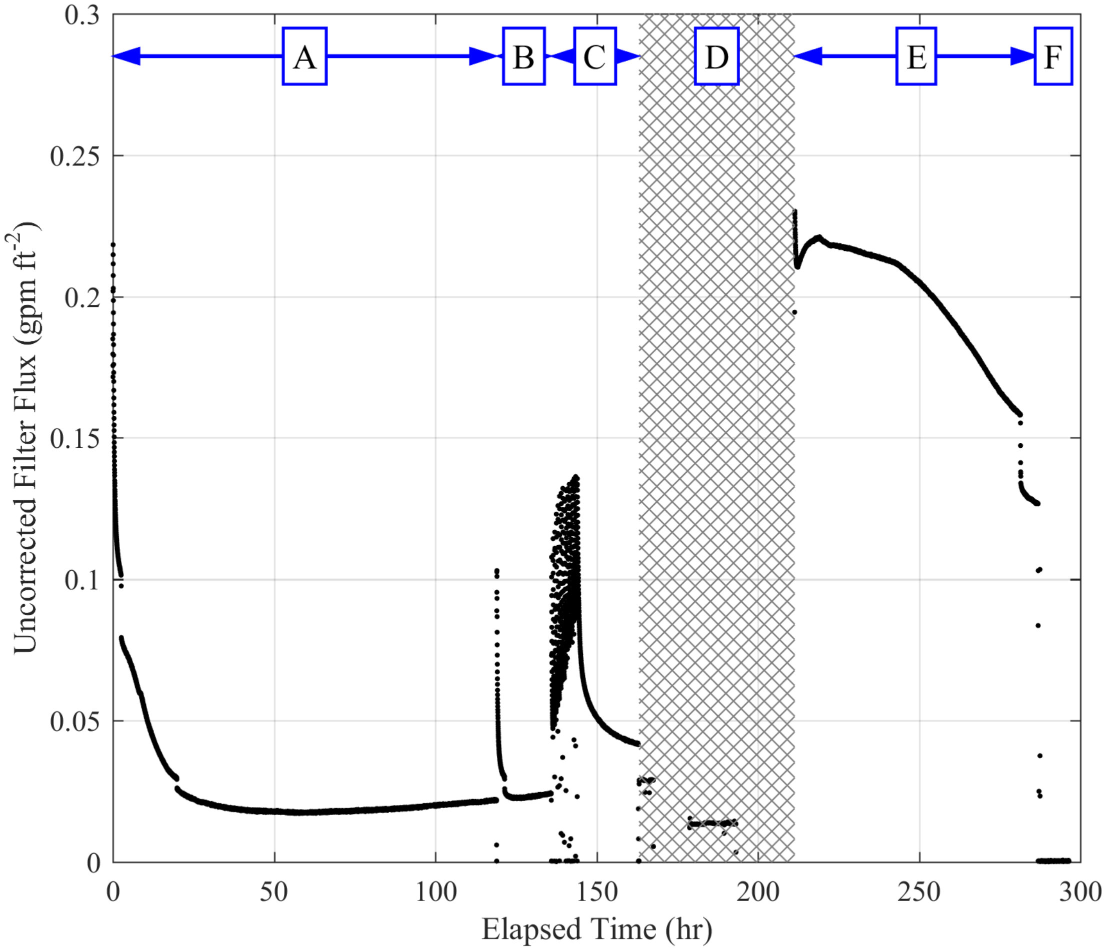


Figure B.1. Test overview for Series 2 Test 1 (LPS-T2S2-MS-LO-01): a fouling test with 0.001 wt% sodium oxalate ($Na_2C_2O_4$) in a modified sodium simulant at 20 °C.

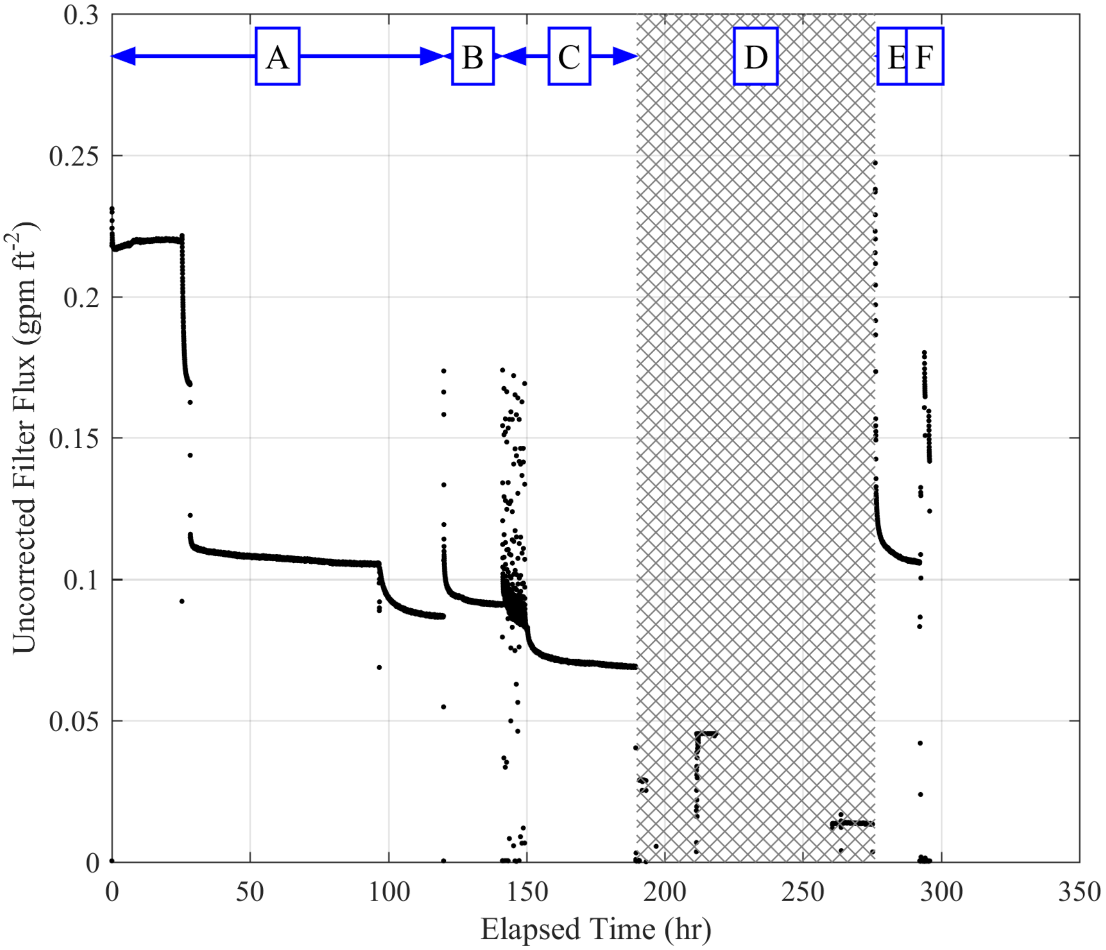


Figure B.2. Test overview for Series 2 Test 2 (LPS-T2S2-MS-SP-01): a fouling test with 0.1 wt% sodium phosphate (Na_3PO_4) in a modified sodium simulant at 45 °C.

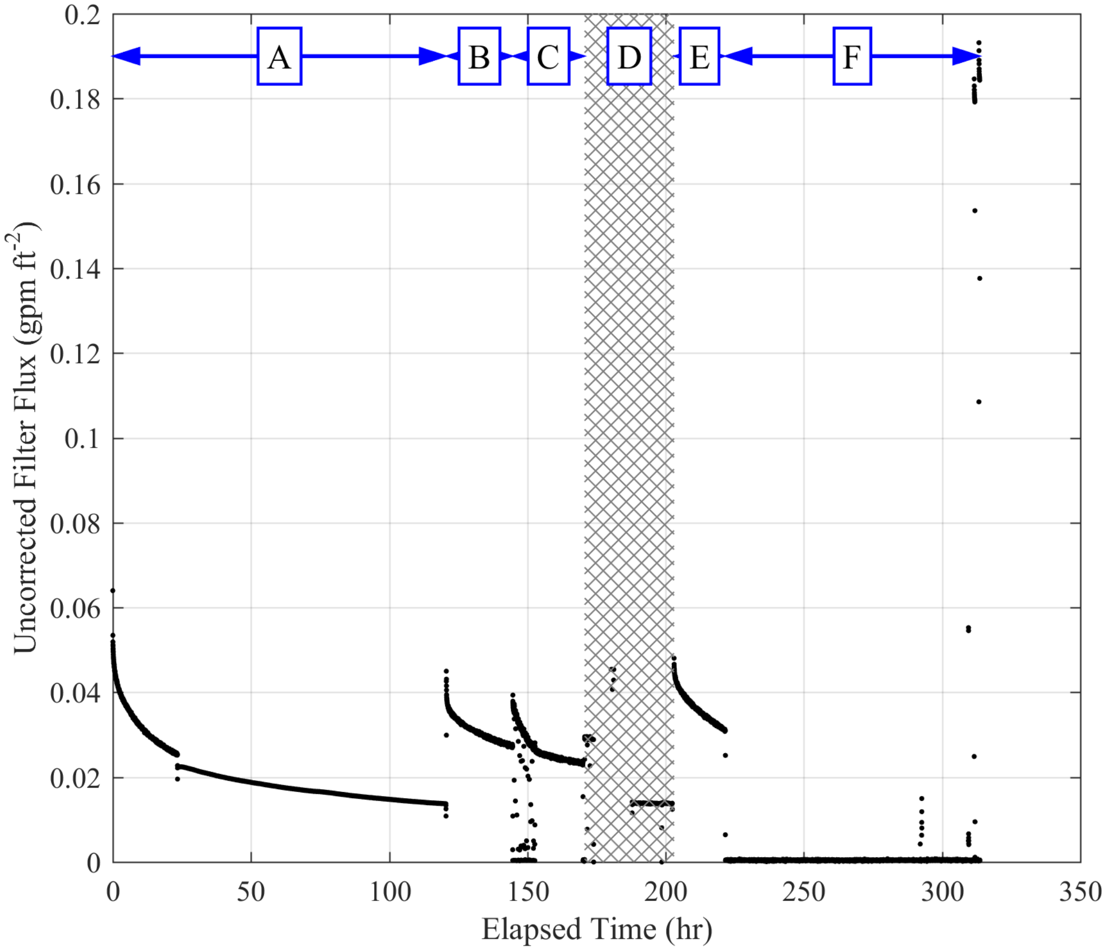


Figure B.3. Test overview for Series 2 Test 3 (LPS-T2S2-MS-IO-01): a fouling test with 0.1 wt% iron oxide (Fe_2O_3) in a modified sodium simulant at 20 °C.

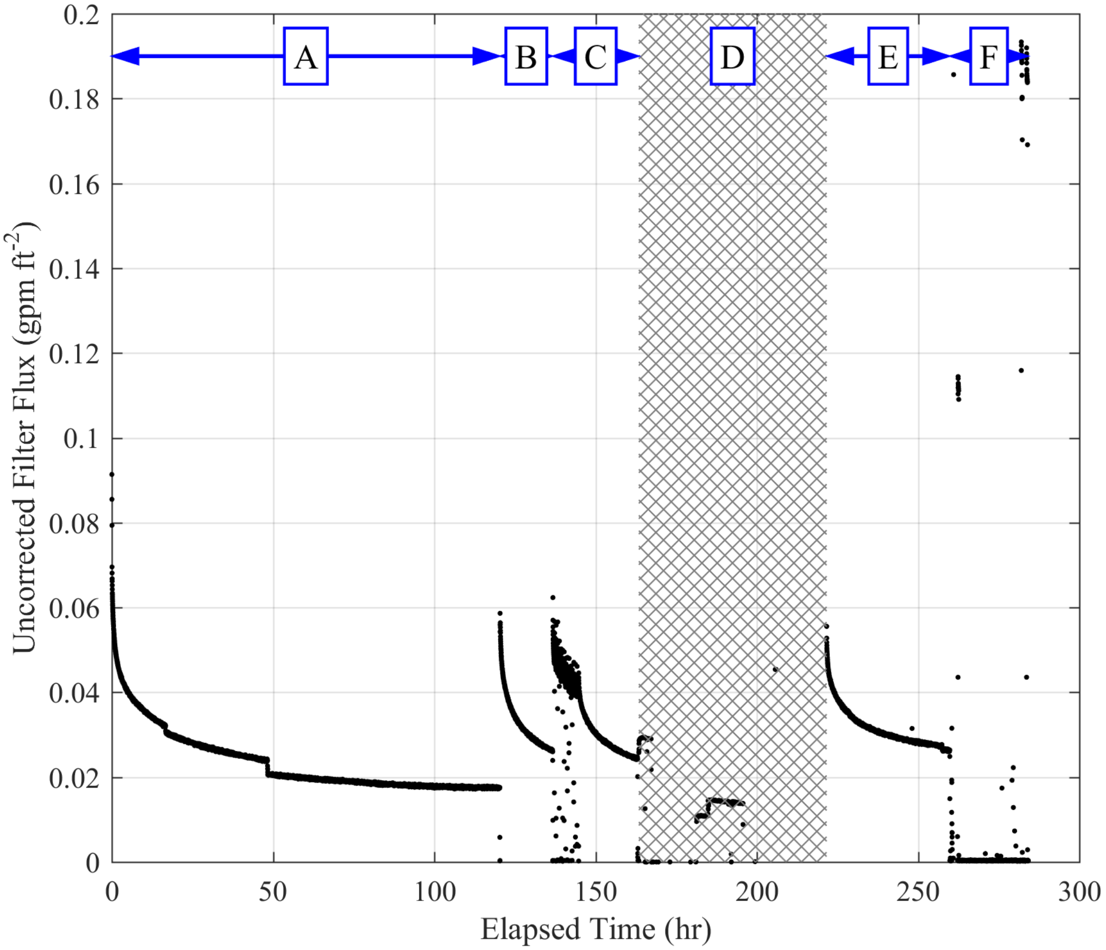


Figure B.4. Test overview for Series 2 Test 4 (LPS-T2S2-MS-IP-01): a fouling test with 0.1 wt% iron phosphate ($FePO_4$) in a modified sodium simulant at 20 °C.

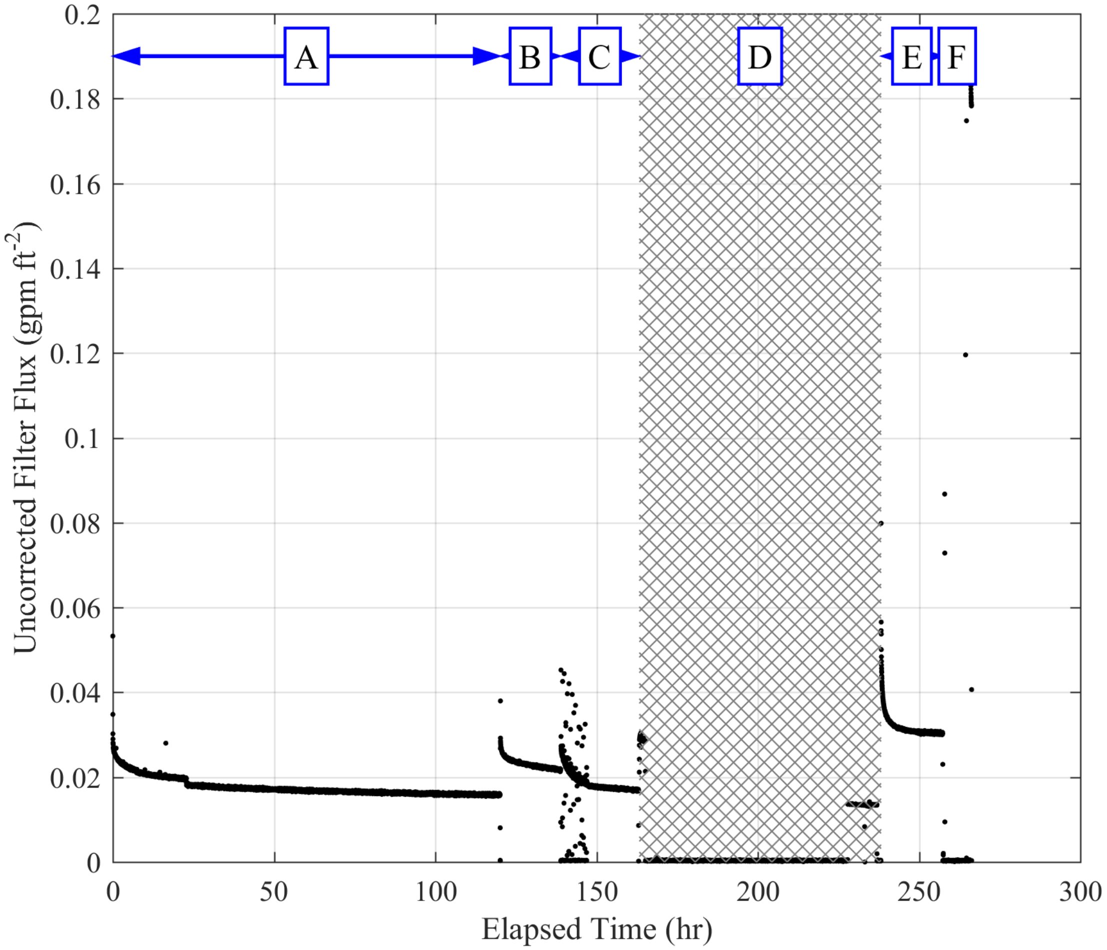


Figure B.5. Test overview for Series 2 Test 5 (LPS-T2S2-MS-CN-01): a fouling test with 0.1 wt% cancrinite ($Na_8[Al_6Si_6O_{24}](NO_3)_2 \cdot 4H_2O$) in a modified sodium simulant at 20 °C.

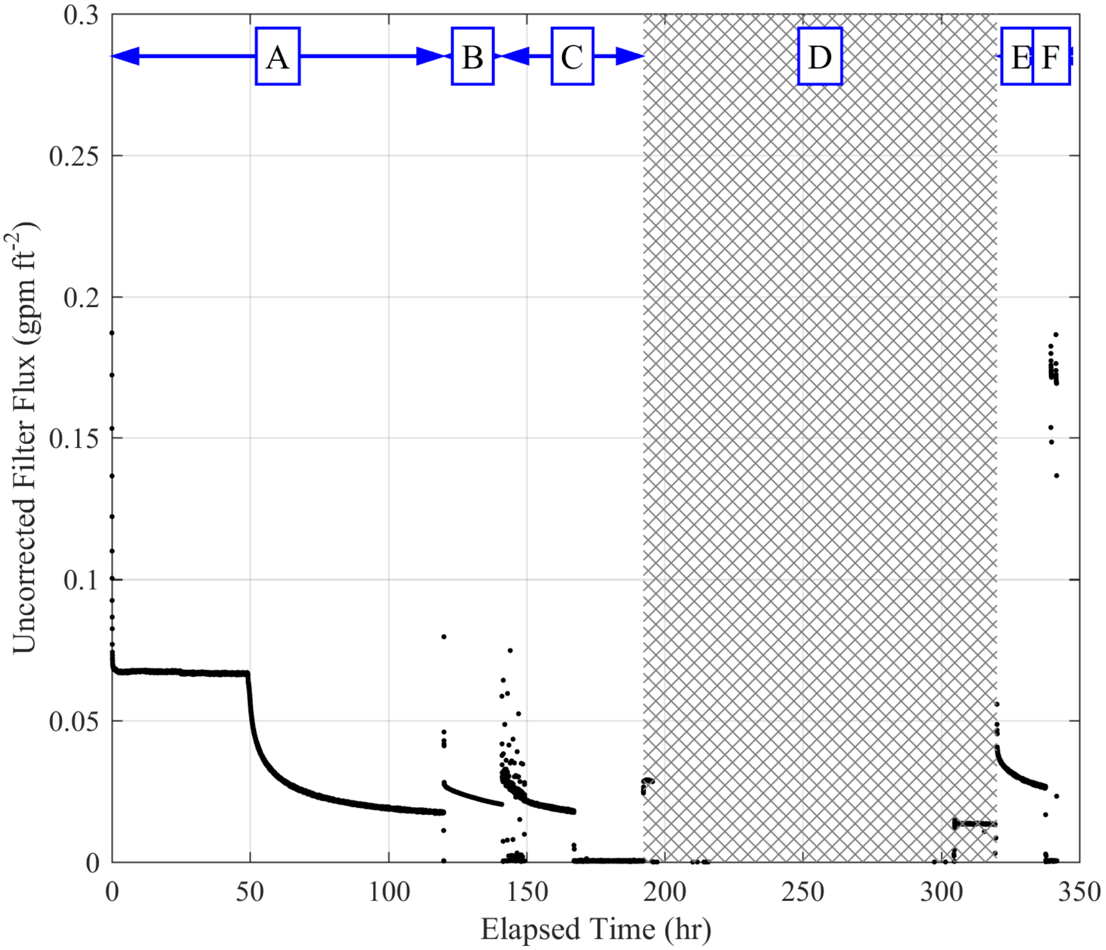


Figure B.6. Test overview for Series 2 Test 6 (LPS-T2S2-MS-HE-01): a fouling test with 0.1 wt% HEDTA (N-hydroxyethyl-ethylenediamine-triacetic acid) in a modified sodium simulant at 20 °C.

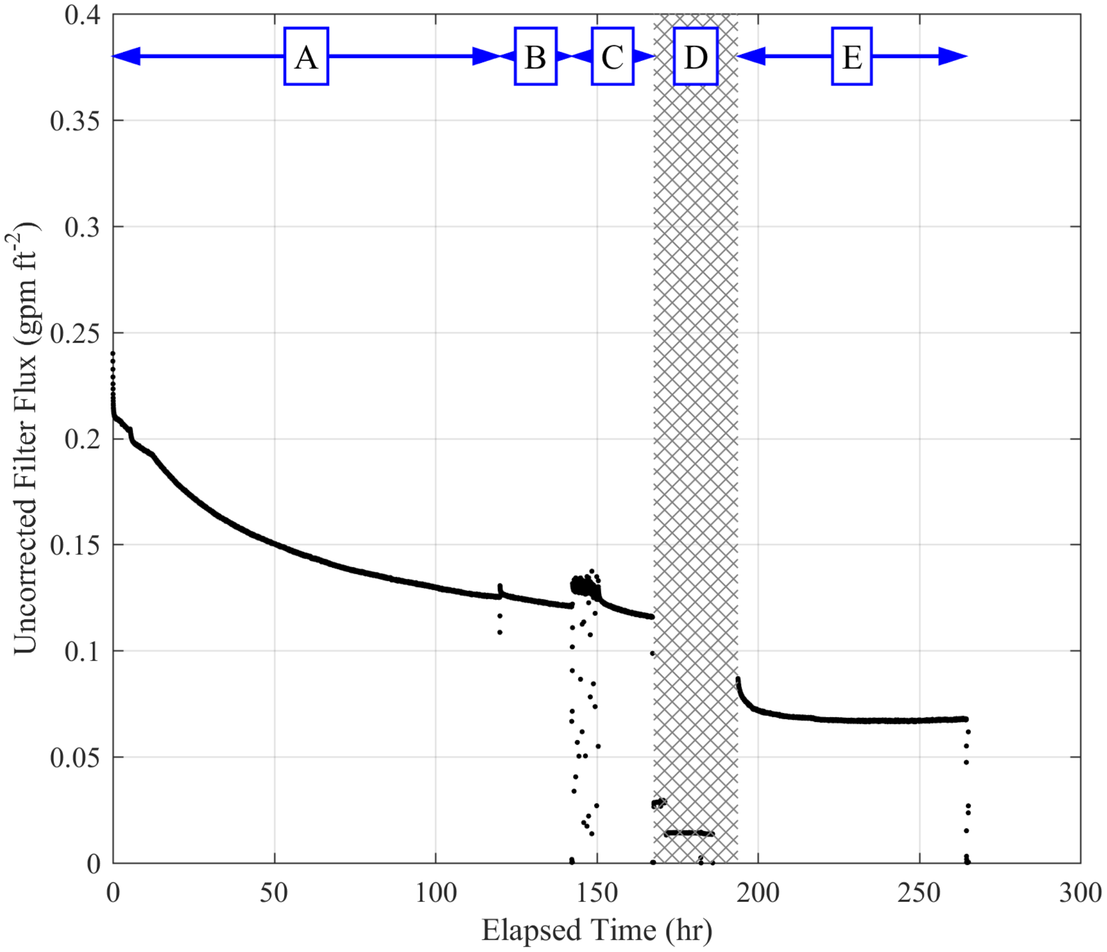


Figure B.7. Test overview for Series 2 Test 7 (LPS-T2S2-NA-RF-01): a fouling test with 0.1 wt% degraded sRF resin in 0.45 M HNO_3 at 20 °C.

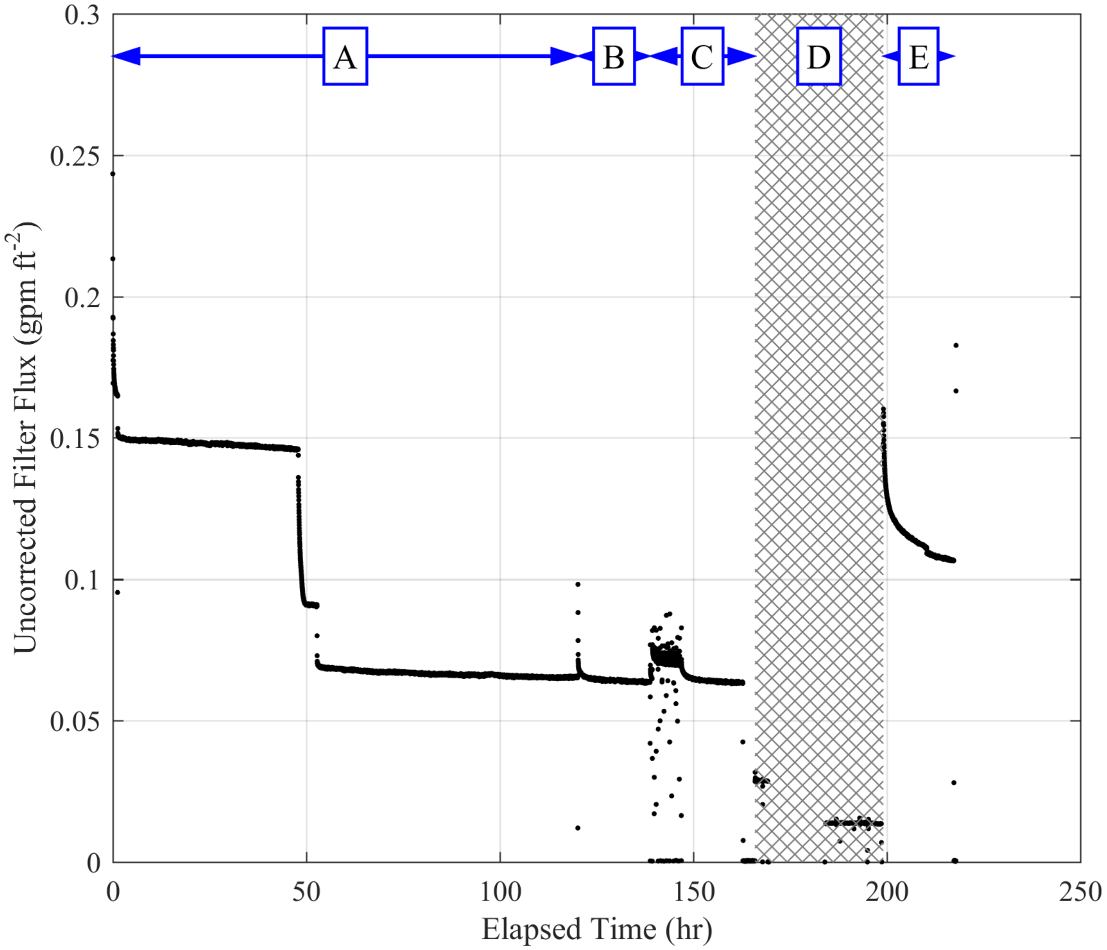


Figure B.8. Test overview for Series 2 Test 8 (LPS-T2S2-MS-SF-01): a fouling test with 6 g L^{-1} sodium fluoride (NaF) in modified sodium simulant at 45 °C.

Table B.2. Operational summary for Series 2 Test 1 (LPS-T2S2-MS-LO-01): a fouling test with 0.001 wt% sodium oxalate ($Na_2C_2O_4$) in a modified sodium simulant at 20 °C.

Parameter	**Symbol**	**Unit**	**Initial Hour**[(a)]	**Final Hour**[(b)]
Initial 120 hr Filtration Period				
Inlet Pressure	p_i	psig	34(1)	27.4(4)
Outlet Pressure	p_o	psig	24.7(4)	16.81(5)
Permeate Pressure	p_f	psig	7(2)	1.94(2)
TMP	δp_t	psid	22(2)	20.2(2)
Axial Velocity	u	$\mathrm{ft\,s^{-1}}$	14.60(2)	14.71(1)
Slurry Temperature	T	°C	20.8(3)	19.696(2)
Flux (Uncorrected)	j	$\mathrm{gpm\,ft^{-2}}$	0.15(3)	0.0219(2)
Flux (Corrected)	j_c	$\mathrm{gpm\,ft^{-2}}$	0.17(4)	0.0254(2)
Flux (Corrected)	$j_{p,c}$	$\mathrm{gpm\,ft^{-2}}$	0.16(5)	0.0252(3)
Post-Single Backpulse Filtration Period				
Inlet Pressure	p_i	psig	30.7(7)	27.0(4)
Outlet Pressure	p_o	psig	20.29(8)	16.65(5)
Permeate Pressure	p_f	psig	2.8(5)	2.05(1)
TMP	δp_t	psid	22.7(7)	19.8(2)
Axial Velocity	u	$\mathrm{ft\,s^{-1}}$	14.60(2)	14.61(2)
Slurry Temperature	T	°C	19.9(1)	19.26(3)
Flux (Uncorrected)	j	$\mathrm{gpm\,ft^{-2}}$	0.05(1)	0.0242(1)
Flux (Corrected)	j_c	$\mathrm{gpm\,ft^{-2}}$	0.06(2)	0.0285(1)
Flux (Corrected)	$j_{p,c}$	$\mathrm{gpm\,ft^{-2}}$	0.05(2)	0.0288(3)
Post-Multiple Backpulse Filtration Period				
Inlet Pressure	p_i	psig	25.7(3)	26.3(4)
Outlet Pressure	p_o	psig	15.18(7)	15.44(5)
Permeate Pressure	p_f	psig	4.5(7)	2.557(7)
TMP	δp_t	psid	15.9(8)	18.3(2)
Axial Velocity	u	$\mathrm{ft\,s^{-1}}$	14.76(2)	14.74(1)
Slurry Temperature	T	°C	19.6(1)	19.341(8)
Flux (Uncorrected)	j	$\mathrm{gpm\,ft^{-2}}$	0.09(2)	0.0419(2)
Flux (Corrected)	j_c	$\mathrm{gpm\,ft^{-2}}$	0.11(2)	0.0493(2)
Flux (Corrected)	$j_{p,c}$	$\mathrm{gpm\,ft^{-2}}$	0.14(3)	0.0538(7)
Post-LAWPS Cleaning Filtration Period				
Inlet Pressure	p_i	psig	35.1(6)	31.8(6)
Outlet Pressure	p_o	psig	25.09(6)	21.82(6)
Permeate Pressure	p_f	psig	11.4(4)	6.32(1)
TMP	δp_t	psid	18.8(6)	20.5(3)
Axial Velocity	u	$\mathrm{ft\,s^{-1}}$	14.64(2)	14.59(1)
Slurry Temperature	T	°C	20.8(4)	19.47(9)
Flux (Uncorrected)	j	$\mathrm{gpm\,ft^{-2}}$	0.214(5)	0.1270(2)
Flux (Corrected)	j_c	$\mathrm{gpm\,ft^{-2}}$	0.241(8)	0.1488(5)
Flux (Corrected)	$j_{p,c}$	$\mathrm{gpm\,ft^{-2}}$	0.26(2)	0.145(2)

(a) Measurements averaged over the initial hour of the given filtration period.
(b) Measurements averaged over the final hour of the given filtration period.

Table B.3. Operational summary for Series 2 Test 2 (LPS-T2S2-MS-SP-01): a fouling test with 0.1 wt% sodium phosphate (Na_3PO_4) in a modified sodium simulant at 45 °C.

Parameter	**Symbol**	**Unit**	**Initial Hour**[(a)]	**Final Hour**[(b)]
Initial 120 hr Filtration Period				
Inlet Pressure	p_i	psig	32.2(4)	29.3(2)
Outlet Pressure	p_o	psig	24.13(8)	19.15(3)
Permeate Pressure	p_f	psig	9.6(2)	4.15(2)
TMP	δp_t	psid	18.6(3)	20.1(1)
Axial Velocity	u	$\mathrm{ft\,s^{-1}}$	14.17(2)	14.61(1)
Slurry Temperature	T	°C	44.5(4)	18.95(3)
Flux (Uncorrected)	j	$\mathrm{gpm\,ft^{-2}}$	0.218(2)	0.0869(2)
Flux (Corrected)	j_c	$\mathrm{gpm\,ft^{-2}}$	0.131(3)	0.1034(2)
Flux (Corrected)	$j_{p,c}$	$\mathrm{gpm\,ft^{-2}}$	0.141(5)	0.1029(7)
Post-Single Backpulse Filtration Period				
Inlet Pressure	p_i	psig	30.3(2)	30.0(2)
Outlet Pressure	p_o	psig	19.96(5)	19.92(4)
Permeate Pressure	p_f	psig	4.9(2)	4.34(1)
TMP	δp_t	psid	20.3(1)	20.6(1)
Axial Velocity	u	$\mathrm{ft\,s^{-1}}$	14.61(1)	14.62(1)
Slurry Temperature	T	°C	18.99(7)	19.02(2)
Flux (Uncorrected)	j	$\mathrm{gpm\,ft^{-2}}$	0.102(4)	0.0911(1)
Flux (Corrected)	j_c	$\mathrm{gpm\,ft^{-2}}$	0.121(5)	0.1081(2)
Flux (Corrected)	$j_{p,c}$	$\mathrm{gpm\,ft^{-2}}$	0.119(6)	0.1048(6)
Post-Multiple Backpulse Filtration Period				
Inlet Pressure	p_i	psig	29.0(3)	29.2(5)
Outlet Pressure	p_o	psig	18.82(7)	19.14(3)
Permeate Pressure	p_f	psig	3.99(8)	3.35(1)
TMP	δp_t	psid	19.9(1)	20.8(3)
Axial Velocity	u	$\mathrm{ft\,s^{-1}}$	14.58(2)	14.57(1)
Slurry Temperature	T	°C	19.70(1)	19.337(3)
Flux (Uncorrected)	j	$\mathrm{gpm\,ft^{-2}}$	0.084(2)	0.0691(1)
Flux (Corrected)	j_c	$\mathrm{gpm\,ft^{-2}}$	0.097(2)	0.0813(1)
Flux (Corrected)	$j_{p,c}$	$\mathrm{gpm\,ft^{-2}}$	0.098(3)	0.078(1)
Post-LAWPS Cleaning Filtration Period				
Inlet Pressure	p_i	psig	31(2)	29.8(5)
Outlet Pressure	p_o	psig	21(2)	19.79(7)
Permeate Pressure	p_f	psig	6(2)	4.90(2)
TMP	δp_t	psid	19.8(9)	19.9(2)
Axial Velocity	u	$\mathrm{ft\,s^{-1}}$	14.5(2)	14.76(2)
Slurry Temperature	T	°C	20.2(2)	20.02(3)
Flux (Uncorrected)	j	$\mathrm{gpm\,ft^{-2}}$	0.14(3)	0.1060(2)
Flux (Corrected)	j_c	$\mathrm{gpm\,ft^{-2}}$	0.16(3)	0.1223(2)
Flux (Corrected)	$j_{p,c}$	$\mathrm{gpm\,ft^{-2}}$	0.16(3)	0.123(1)

(a) Measurements averaged over the initial hour of the given filtration period.
(b) Measurements averaged over the final hour of the given filtration period.

Table B.4. Operational summary for Series 2 Test 3 (LPS-T2S2-MS-IO-01): a fouling test with 0.1 wt% iron oxide (Fe_2O_3) in a modified sodium simulant at 20 °C.

Parameter	Symbol	Unit	Initial Hour[(a)]	Final Hour[(b)]
Initial 120 hr Filtration Period				
Inlet Pressure	p_i	psig	27.7(1)	32.5(7)
Outlet Pressure	p_o	psig	17.41(4)	21.4(5)
Permeate Pressure	p_f	psig	2.46(8)	4.7(6)
TMP	δp_t	psid	20.12(7)	22.3(5)
Axial Velocity	u	$\mathrm{ft\,s^{-1}}$	14.61(2)	14.66(5)
Slurry Temperature	T	°C	19.4(2)	20.414(5)
Flux (Uncorrected)	j	$\mathrm{gpm\,ft^{-2}}$	0.046(2)	0.0137(4)
Flux (Corrected)	j_c	$\mathrm{gpm\,ft^{-2}}$	0.054(3)	0.0156(5)
Flux (Corrected)	$j_{p,c}$	$\mathrm{gpm\,ft^{-2}}$	0.054(3)	0.0140(4)
Post-Single Backpulse Filtration Period				
Inlet Pressure	p_i	psig	27.9(6)	28.2(8)
Outlet Pressure	p_o	psig	17.6(1)	17.58(7)
Permeate Pressure	p_f	psig	2.14(4)	1.85(1)
TMP	δp_t	psid	20.6(3)	21.1(4)
Axial Velocity	u	$\mathrm{ft\,s^{-1}}$	14.87(2)	14.88(2)
Slurry Temperature	T	°C	19.9(1)	19.70(1)
Flux (Uncorrected)	j	$\mathrm{gpm\,ft^{-2}}$	0.0370(9)	0.0274(2)
Flux (Corrected)	j_c	$\mathrm{gpm\,ft^{-2}}$	0.043(1)	0.0319(2)
Flux (Corrected)	$j_{p,c}$	$\mathrm{gpm\,ft^{-2}}$	0.041(1)	0.0303(6)
Post-Multiple Backpulse Filtration Period				
Inlet Pressure	p_i	psig	27.8(6)	27.9(5)
Outlet Pressure	p_o	psig	17.21(6)	17.32(8)
Permeate Pressure	p_f	psig	1.80(1)	1.70(2)
TMP	δp_t	psid	20.7(3)	20.9(2)
Axial Velocity	u	$\mathrm{ft\,s^{-1}}$	14.88(2)	14.86(2)
Slurry Temperature	T	°C	19.49(8)	19.51(4)
Flux (Uncorrected)	j	$\mathrm{gpm\,ft^{-2}}$	0.0261(2)	0.0234(2)
Flux (Corrected)	j_c	$\mathrm{gpm\,ft^{-2}}$	0.0306(3)	0.0274(2)
Flux (Corrected)	$j_{p,c}$	$\mathrm{gpm\,ft^{-2}}$	0.0296(6)	0.0262(4)
Post-LAWPS Cleaning Filtration Period				
Inlet Pressure	p_i	psig	27.6(2)	27.5(4)
Outlet Pressure	p_o	psig	17.25(5)	17.11(6)
Permeate Pressure	p_f	psig	2.32(2)	1.97(1)
TMP	δp_t	psid	20.1(1)	20.3(2)
Axial Velocity	u	$\mathrm{ft\,s^{-1}}$	14.70(2)	14.81(2)
Slurry Temperature	T	°C	19.35(2)	19.337(3)
Flux (Uncorrected)	j	$\mathrm{gpm\,ft^{-2}}$	0.0431(9)	0.0313(1)
Flux (Corrected)	j_c	$\mathrm{gpm\,ft^{-2}}$	0.051(1)	0.0369(2)
Flux (Corrected)	$j_{p,c}$	$\mathrm{gpm\,ft^{-2}}$	0.051(1)	0.0363(4)

(a) Measurements averaged over the initial hour of the given filtration period.
(b) Measurements averaged over the final hour of the given filtration period.

Table B.5. Operational summary for Series 2 Test 4 (LPS-T2S2-MS-IP-01): a fouling test with 0.1 wt% iron phosphate ($FePO_4$) in a modified sodium simulant at 20 °C.

Parameter	Symbol	Unit	Initial Hour[(a)]	Final Hour[(b)]
Initial 120 hr Filtration Period				
Inlet Pressure	p_i	psig	29.9(3)	27.8(7)
Outlet Pressure	p_o	psig	19.26(9)	16.5(1)
Permeate Pressure	p_f	psig	3.1(2)	1.86(1)
TMP	δp_t	psid	21.5(2)	20.3(3)
Axial Velocity	u	$\mathrm{ft\,s^{-1}}$	14.77(1)	14.82(1)
Slurry Temperature	T	°C	20.19(4)	19.28(3)
Flux (Uncorrected)	j	$\mathrm{gpm\,ft^{-2}}$	0.056(5)	0.0175(1)
Flux (Corrected)	j_c	$\mathrm{gpm\,ft^{-2}}$	0.064(5)	0.0206(1)
Flux (Corrected)	$j_{p,c}$	$\mathrm{gpm\,ft^{-2}}$	0.060(5)	0.0203(3)
Post-Single Backpulse Filtration Period				
Inlet Pressure	p_i	psig	28.4(8)	29(1)
Outlet Pressure	p_o	psig	17.9(1)	18.1(1)
Permeate Pressure	p_f	psig	2.8(1)	2.11(1)
TMP	δp_t	psid	20.3(4)	21.6(5)
Axial Velocity	u	$\mathrm{ft\,s^{-1}}$	14.69(1)	14.64(2)
Slurry Temperature	T	°C	19.14(7)	19.337(3)
Flux (Uncorrected)	j	$\mathrm{gpm\,ft^{-2}}$	0.047(3)	0.0264(2)
Flux (Corrected)	j_c	$\mathrm{gpm\,ft^{-2}}$	0.056(4)	0.0311(2)
Flux (Corrected)	$j_{p,c}$	$\mathrm{gpm\,ft^{-2}}$	0.055(4)	0.0287(6)
Post-Multiple Backpulse Filtration Period				
Inlet Pressure	p_i	psig	28.0(5)	28.8(4)
Outlet Pressure	p_o	psig	17.86(8)	18.01(7)
Permeate Pressure	p_f	psig	2.50(5)	2.065(7)
TMP	δp_t	psid	20.4(3)	21.4(2)
Axial Velocity	u	$\mathrm{ft\,s^{-1}}$	14.62(1)	14.63(1)
Slurry Temperature	T	°C	19.337(2)	19.338(4)
Flux (Uncorrected)	j	$\mathrm{gpm\,ft^{-2}}$	0.038(1)	0.0245(2)
Flux (Corrected)	j_c	$\mathrm{gpm\,ft^{-2}}$	0.045(2)	0.0288(2)
Flux (Corrected)	$j_{p,c}$	$\mathrm{gpm\,ft^{-2}}$	0.044(2)	0.0270(3)
Post-LAWPS Cleaning Filtration Period				
Inlet Pressure	p_i	psig	27.2(3)	26.6(4)
Outlet Pressure	p_o	psig	16.8(1)	15.80(8)
Permeate Pressure	p_f	psig	2.70(8)	2.083(8)
TMP	δp_t	psid	19.3(1)	19.1(2)
Axial Velocity	u	$\mathrm{ft\,s^{-1}}$	14.79(2)	14.81(2)
Slurry Temperature	T	°C	19.695(3)	19.48(6)
Flux (Uncorrected)	j	$\mathrm{gpm\,ft^{-2}}$	0.045(2)	0.0263(1)
Flux (Corrected)	j_c	$\mathrm{gpm\,ft^{-2}}$	0.053(2)	0.0308(2)
Flux (Corrected)	$j_{p,c}$	$\mathrm{gpm\,ft^{-2}}$	0.055(3)	0.0323(5)

(a) Measurements averaged over the initial hour of the given filtration period.
(b) Measurements averaged over the final hour of the given filtration period.

Table B.6. Operational summary for Series 2 Test 5 (LPS-T2S2-MS-CN-01): a fouling test with 0.1 wt% cancrinite ($Na_8[Al_6Si_6O_{24}](NO_3)_2 \cdot 4H_2O$) in a modified sodium simulant at 20 °C.

Parameter	**Symbol**	**Unit**	**Initial Hour**[(a)]	**Final Hour**[(b)]
Initial 120 hr Filtration Period				
Inlet Pressure	p_i	psig	27.9(2)	27.9(5)
Outlet Pressure	p_o	psig	17.38(8)	17.11(5)
Permeate Pressure	p_f	psig	2.09(2)	1.828(7)
TMP	δp_t	psid	20.5(1)	20.7(2)
Axial Velocity	u	$\mathrm{ft\,s^{-1}}$	14.82(2)	14.68(2)
Slurry Temperature	T	°C	19.95(7)	19.33(1)
Flux (Uncorrected)	j	$\mathrm{gpm\,ft^{-2}}$	0.0258(8)	0.0159(2)
Flux (Corrected)	j_c	$\mathrm{gpm\,ft^{-2}}$	0.030(1)	0.0187(2)
Flux (Corrected)	$j_{p,c}$	$\mathrm{gpm\,ft^{-2}}$	0.029(1)	0.0181(3)
Post-Single Backpulse Filtration Period				
Inlet Pressure	p_i	psig	27.7(6)	27.3(6)
Outlet Pressure	p_o	psig	16.7(1)	16.6(2)
Permeate Pressure	p_f	psig	2.11(2)	2.02(2)
TMP	δp_t	psid	20.1(3)	20.0(3)
Axial Velocity	u	$\mathrm{ft\,s^{-1}}$	14.76(1)	14.76(1)
Slurry Temperature	T	°C	19.10(6)	18.99(2)
Flux (Uncorrected)	j	$\mathrm{gpm\,ft^{-2}}$	0.0260(7)	0.0218(1)
Flux (Corrected)	j_c	$\mathrm{gpm\,ft^{-2}}$	0.0308(8)	0.0259(2)
Flux (Corrected)	$j_{p,c}$	$\mathrm{gpm\,ft^{-2}}$	0.0307(8)	0.0259(4)
Post-Multiple Backpulse Filtration Period				
Inlet Pressure	p_i	psig	27.1(3)	27.4(3)
Outlet Pressure	p_o	psig	16.42(7)	16.56(8)
Permeate Pressure	p_f	psig	1.89(2)	1.84(1)
TMP	δp_t	psid	19.9(2)	20.1(1)
Axial Velocity	u	$\mathrm{ft\,s^{-1}}$	14.76(2)	14.77(1)
Slurry Temperature	T	°C	19.2(2)	19.21(5)
Flux (Uncorrected)	j	$\mathrm{gpm\,ft^{-2}}$	0.0181(3)	0.0170(1)
Flux (Corrected)	j_c	$\mathrm{gpm\,ft^{-2}}$	0.0214(4)	0.0201(2)
Flux (Corrected)	$j_{p,c}$	$\mathrm{gpm\,ft^{-2}}$	0.0215(4)	0.020(2)
Post-LAWPS Cleaning Filtration Period				
Inlet Pressure	p_i	psig	27.3(7)	27.4(8)
Outlet Pressure	p_o	psig	16.9(1)	16.58(8)
Permeate Pressure	p_f	psig	2.4(1)	2.12(1)
TMP	δp_t	psid	19.7(3)	19.9(4)
Axial Velocity	u	$\mathrm{ft\,s^{-1}}$	14.64(2)	14.84(2)
Slurry Temperature	T	°C	19.94(7)	19.6962
Flux (Uncorrected)	j	$\mathrm{gpm\,ft^{-2}}$	0.039(3)	0.0303(2)
Flux (Corrected)	j_c	$\mathrm{gpm\,ft^{-2}}$	0.045(3)	0.0353(2)
Flux (Corrected)	$j_{p,c}$	$\mathrm{gpm\,ft^{-2}}$	0.046(3)	0.0355(8)

(a) Measurements averaged over the initial hour of the given filtration period.
(b) Measurements averaged over the final hour of the given filtration period.

Table B.7. Operational summary for Series 2 Test 6 (LPS-T2S2-MS-HE-01): a fouling test with 0.1 wt% HEDTA (N-hydroxyethyl-ethylenediamine-triacetic acid) in a modified sodium simulant at 20 °C.

Parameter	**Symbol**	**Unit**	**Initial Hour(a)**	**Final Hour(b)**
Initial 120 hr Filtration Period				
Inlet Pressure	p_i	psig	29.9(3)	31.7(5)
Outlet Pressure	p_o	psig	19.6(3)	20.71(5)
Permeate Pressure	p_f	psig	3.5(5)	1.61(7)
TMP	δp_t	psid	21.2(3)	24.6(3)
Axial Velocity	u	ft s^{-1}	14.67(3)	14.69(2)
Slurry Temperature	T	°C	19.9(4)	20.44(2)
Flux (Uncorrected)	j	gpm ft^{-2}	0.07(1)	0.0174(8)
Flux (Corrected)	j_c	gpm ft^{-2}	0.08(1)	0.020(1)
Flux (Corrected)	$j_{p,c}$	gpm ft^{-2}	0.08(1)	0.0161(8)
Post-Single Backpulse Filtration Period				
Inlet Pressure	p_i	psig	32.1(7)	32.8(4)
Outlet Pressure	p_o	psig	21.8(2)	21.99(5)
Permeate Pressure	p_f	psig	10.1(3)	7.49(3)
TMP	δp_t	psid	16.8(4)	19.9(2)
Axial Velocity	u	ft s^{-1}	14.64(3)	14.58(1)
Slurry Temperature	T	°C	20.41(4)	20.415(6)
Flux (Uncorrected)	j	gpm ft^{-2}	0.0269(5)	0.0205
Flux (Corrected)	j_c	gpm ft^{-2}	0.0307(6)	0.0234
Flux (Corrected)	$j_{p,c}$	gpm ft^{-2}	0.036(1)	0.0235(3)
Post-Multiple Backpulse Filtration Period				
Inlet Pressure	p_i	psig	27.2(3)	27.3(3)
Outlet Pressure	p_o	psig	16.50(5)	16.58(4)
Permeate Pressure	p_f	psig	1.75(2)	1.62(1)
TMP	δp_t	psid	20.1(1)	20.3(1)
Axial Velocity	u	ft s^{-1}	14.70(1)	14.69(2)
Slurry Temperature	T	°C	19.59(9)	19.53(7)
Flux (Uncorrected)	j	gpm ft^{-2}	0.0217(3)	0.0180(2)
Flux (Corrected)	j_c	gpm ft^{-2}	0.0254(5)	0.0210(2)
Flux (Corrected)	$j_{p,c}$	gpm ft^{-2}	0.0253(4)	0.0207(3)
Post-LAWPS Cleaning Filtration Period				
Inlet Pressure	p_i	psig	27.4(4)	27.3(5)
Outlet Pressure	p_o	psig	17.0(3)	16.61(6)
Permeate Pressure	p_f	psig	2.15(6)	1.819(8)
TMP	δp_t	psid	20.0(3)	20.1(2)
Axial Velocity	u	ft s^{-1}	14.71(3)	14.94(2)
Slurry Temperature	T	°C	20.25(6)	20.04(1)
Flux (Uncorrected)	j	gpm ft^{-2}	0.038(2)	0.0266(2)
Flux (Corrected)	j_c	gpm ft^{-2}	0.043(2)	0.0307(3)
Flux (Corrected)	$j_{p,c}$	gpm ft^{-2}	0.043(1)	0.0305(4)

(a) Measurements averaged over the initial hour of the given filtration period.
(b) Measurements averaged over the final hour of the given filtration period.

Table B.8. Operational summary for Series 2 Test 7 (LPS-T2S2-NA-RF-01): a fouling test with 0.1 wt% degraded sRF resin in 0.45 M HNO_3 at 20 °C.

Parameter	Symbol	Unit	Initial Hour[(a)]	Final Hour[(b)]
Initial 120 hr Filtration Period				
Inlet Pressure	p_i	psig	16.8(6)	18.8(3)
Outlet Pressure	p_o	psig	10.57(3)	12.29(4)
Permeate Pressure	p_f	psig	7.0(2)	3.56(1)
TMP	δp_t	psid	6.7(3)	12.0(2)
Axial Velocity	u	$\mathrm{ft\,s^{-1}}$	14.62(2)	14.52(1)
Slurry Temperature	T	°C	20.1(4)	19.697(4)
Flux (Uncorrected)	j	$\mathrm{gpm\,ft^{-2}}$	0.212(3)	0.1253(1)
Flux (Corrected)	j_c	$\mathrm{gpm\,ft^{-2}}$	0.243(6)	0.1459(1)
Flux (Corrected)	$j_{p,c}$	$\mathrm{gpm\,ft^{-2}}$	0.73(4)	0.243(3)
Post-Single Backpulse Filtration Period				
Inlet Pressure	p_i	psig	18.7(3)	18.7(4)
Outlet Pressure	p_o	psig	12.21(3)	12.42(4)
Permeate Pressure	p_f	psig	3.61(2)	3.39(2)
TMP	δp_t	psid	11.9(2)	12.2(2)
Axial Velocity	u	$\mathrm{ft\,s^{-1}}$	14.52(1)	14.51(2)
Slurry Temperature	T	°C	19.697(3)	19.71(2)
Flux (Uncorrected)	j	$\mathrm{gpm\,ft^{-2}}$	0.1271(6)	0.1210(2)
Flux (Corrected)	j_c	$\mathrm{gpm\,ft^{-2}}$	0.1480(8)	0.1408(2)
Flux (Corrected)	$j_{p,c}$	$\mathrm{gpm\,ft^{-2}}$	0.249(3)	0.231(4)
Post-Multiple Backpulse Filtration Period				
Inlet Pressure	p_i	psig	19.7(3)	19.7(3)
Outlet Pressure	p_o	psig	13.31(5)	13.42(5)
Permeate Pressure	p_f	psig	3.49(4)	3.23(2)
TMP	δp_t	psid	13.0(2)	13.3(2)
Axial Velocity	u	$\mathrm{ft\,s^{-1}}$	14.41(1)	14.39(1)
Slurry Temperature	T	°C	20.0555	19.71(2)
Flux (Uncorrected)	j	$\mathrm{gpm\,ft^{-2}}$	0.125(1)	0.1160(1)
Flux (Corrected)	j_c	$\mathrm{gpm\,ft^{-2}}$	0.143(1)	0.1350(2)
Flux (Corrected)	$j_{p,c}$	$\mathrm{gpm\,ft^{-2}}$	0.220(4)	0.202(2)
Post-LAWPS Cleaning Filtration Period				
Inlet Pressure	p_i	psig	15.6(7)	13.4(8)
Outlet Pressure	p_o	psig	8.9(2)	6.42(8)
Permeate Pressure	p_f	psig	2.35(4)	2.065(9)
TMP	δp_t	psid	9.9(4)	7.8(4)
Axial Velocity	u	$\mathrm{ft\,s^{-1}}$	14.78(2)	15.07(1)
Slurry Temperature	T	°C	19.698(6)	19.41(3)
Flux (Uncorrected)	j	$\mathrm{gpm\,ft^{-2}}$	0.081(2)	0.0678(1)
Flux (Corrected)	j_c	$\mathrm{gpm\,ft^{-2}}$	0.095(2)	0.0795(2)
Flux (Corrected)	$j_{p,c}$	$\mathrm{gpm\,ft^{-2}}$	0.191(6)	0.20(1)

(a) Measurements averaged over the initial hour of the given filtration period.
(b) Measurements averaged over the final hour of the given filtration period.

Table B.9. Operational summary for Series 2 Test 8 (LPS-T2S2-MS-SF-01): a fouling test with 6 $\mathrm{g\,L^{-1}}$ sodium fluoride (NaF) in modified sodium simulant at 45 °C.

Parameter	**Symbol**	**Unit**	**Initial Hour(a)**	**Final Hour(b)**
Initial 120 hr Filtration Period				
Inlet Pressure	p_i	psig	32.2(7)	29.6(4)
Outlet Pressure	p_o	psig	24.0(2)	18.28(5)
Permeate Pressure	p_f	psig	6.8(3)	3.301(8)
TMP	δp_t	psid	21.3(6)	20.6(2)
Axial Velocity	u	$\mathrm{ft\,s^{-1}}$	14.37(2)	14.89(2)
Slurry Temperature	T	°C	45.4(2)	20.21(8)
Flux (Uncorrected)	j	$\mathrm{gpm\,ft^{-2}}$	0.170(6)	0.0654(2)
Flux (Corrected)	j_c	$\mathrm{gpm\,ft^{-2}}$	0.099(4)	0.0750(2)
Flux (Corrected)	$j_{p,c}$	$\mathrm{gpm\,ft^{-2}}$	0.093(7)	0.0728(6)
Post-Single Backpulse Filtration Period				
Inlet Pressure	p_i	psig	28(1)	28.1(7)
Outlet Pressure	p_o	psig	16.76(9)	17.0(6)
Permeate Pressure	p_f	psig	3.38(5)	3.28(1)
TMP	δp_t	psid	18.9(5)	19.3(3)
Axial Velocity	u	$\mathrm{ft\,s^{-1}}$	15.01(2)	14.99(1)
Slurry Temperature	T	°C	20.09(2)	19.72(2)
Flux (Uncorrected)	j	$\mathrm{gpm\,ft^{-2}}$	0.0672(9)	0.0637(1)
Flux (Corrected)	j_c	$\mathrm{gpm\,ft^{-2}}$	0.077(1)	0.0741(1)
Flux (Corrected)	$j_{p,c}$	$\mathrm{gpm\,ft^{-2}}$	0.082(3)	0.077(1)
Post-Multiple Backpulse Filtration Period				
Inlet Pressure	p_i	psig	28.0(4)	28.3(5)
Outlet Pressure	p_o	psig	17.23(5)	17.29(6)
Permeate Pressure	p_f	psig	3.41(5)	3.282(9)
TMP	δp_t	psid	19.2(2)	19.5(2)
Axial Velocity	u	$\mathrm{ft\,s^{-1}}$	14.68(1)	14.68(1)
Slurry Temperature	T	°C	19.65(6)	19.48(5)
Flux (Uncorrected)	j	$\mathrm{gpm\,ft^{-2}}$	0.0671(8)	0.0635(1)
Flux (Corrected)	j_c	$\mathrm{gpm\,ft^{-2}}$	0.078(1)	0.0744(2)
Flux (Corrected)	$j_{p,c}$	$\mathrm{gpm\,ft^{-2}}$	0.082(2)	0.0763(9)
Post-LAWPS Cleaning Filtration Period				
Inlet Pressure	p_i	psig	32.2(8)	32(1)
Outlet Pressure	p_o	psig	21.99(9)	21.6(1)
Permeate Pressure	p_f	psig	6.6(4)	5.03(1)
TMP	δp_t	psid	20.5(6)	21.8(5)
Axial Velocity	u	$\mathrm{ft\,s^{-1}}$	14.58(2)	14.68(1)
Slurry Temperature	T	°C	20.0(3)	20.88(7)
Flux (Uncorrected)	j	$\mathrm{gpm\,ft^{-2}}$	0.136(7)	0.1068(1)
Flux (Corrected)	j_c	$\mathrm{gpm\,ft^{-2}}$	0.16(1)	0.1201(3)
Flux (Corrected)	$j_{p,c}$	$\mathrm{gpm\,ft^{-2}}$	0.15(1)	0.110(3)

(a) Measurements averaged over the initial hour of the given filtration period.
(b) Measurements averaged over the final hour of the given filtration period.

Appendix C

Particle Size Distribution Measurement Data

Appendix C – Particle Size Distribution Measurement Data

The particle size distributions (PSDs) of the baseline Low-Activity Waste Pretreatment System (LAWPS) and LAWPS fouling simulants were measured in accordance with operating procedure OP-WTPSP-003, Rev. 2, *Size Analysis Using Malvern MS2000*, using a Malvern Mastersizer 2000 equipped with a Hydro μP dispersion unit. Particle size data were collected and analyzed using Malvern Mastersizer 2000 software (Version 5.6). For each measurement, a subsample of the simulant solids was suspended in a dispersing medium representative of the chemical conditions used in testing (typically LAWPS simulant supernate) by direct addition to the Hydro μP dispersion unit. A dispersion unit flow speed of 3000 rpm was used to suspend the sample; an in-line sonicator was used to evaluate the influence of sonication on the size distribution of suspended solids. The PSD measurement protocol is outlined in Table C.1, and results in a set of pre-sonication, during-sonication, and post-sonication averages for each sample. The sample naming convention is "[ID]-[*N*].[*M*]", where ID represents a PSD-sample-specific identifier (see Tables C.2 and C.3), *N* is the replicate number (1 for the primary measurement, 2 for the first replicate, 3 for the second replicate, and so on), and *M* is denotes the measurement type (1 – pre-sonciation, 2 – during-sonication, and 3 – post-sonication). The active measurement of PSD is conducted at a scattered light sampling rate of 1000 Hz, with a 20-second period of red light measurement followed immediately by a 20-second period of blue light measurement. Three separate periods of blue and red light measurement (taking a total of 120 seconds) make up each average PSD measurement reported herein. In the two appendix sub-sections that follow, the PSD sample matrix for the LAWPS baseline simulants and fouling simulants are presented, along with the summary PSD measurement outputs provided by the Mastersizer 2000 software.

Table C.1. Measuring protocol applied for determination of sample PSD.

Step	Operation	Sonicator	Duration [s]
1	Recirculate dispersion	Off	60
2	Measure pre-sonicated PSD	Off	120
3	Recirculate dispersion	On (100% power)	60
4	Measure during-sonication PSD	On (100% power)	120
5	Recirculate dispersion	Off	60
6	Measure post-sonication PSD	Off	120

C.1 LAWPS Baseline Simulants

The size distribution associated with undissolved solids for each of the four LAWPS simulants was measured. Table C.2 provides the PSD sample matrix. All samples were measured in their corresponding supernate.

Table C.2. List of LAWPS baseline simulant sample IDs and descriptions.

ID	Description	Suspending Phase
8 M Na PSD	8.0 M Na (As-Prepared Solids)	8.0 M Na Supernate
6 M Na PSD	6.0 M Na (As-Prepared Solids)	6.0 M Na Supernate
5.6 M Na PSD	5.6 M Na (As-Prepared Solids)	5.6 M Na Supernate
4 M Na PSD	4.0 M Na (As-Prepared Solids)	4.0 M Na Supernate

MASTERSIZER 2000

Result Analysis Report

Sample Name:
8 M Na PSD-1.1 - Average

SOP Name:

Measured:
Thursday, September 29, 2016 3:26:17 PM

Sample Source & type:

Measured by:
D3M966

Analysed:
Thursday, September 29, 2016 3:26:18 PM

Sample bulk lot ref:

Result Source:
Averaged

Particle Name:
Sodium Oxalate

Accessory Name:
Hydro 2000μP (A)

Analysis model:
General purpose

Sensitivity:
Normal

Particle RI:
1.480

Absorption:
1

Size range:
0.020 to 2000.000 um

Obscuration:
13.05 %

Dispersant Name:
Water

Dispersant RI:
1.330

Weighted Residual:
0.218 %

Result Emulation:
Off

Concentration:
0.0664 %Vol

Span :
6.794

Uniformity:
2.22

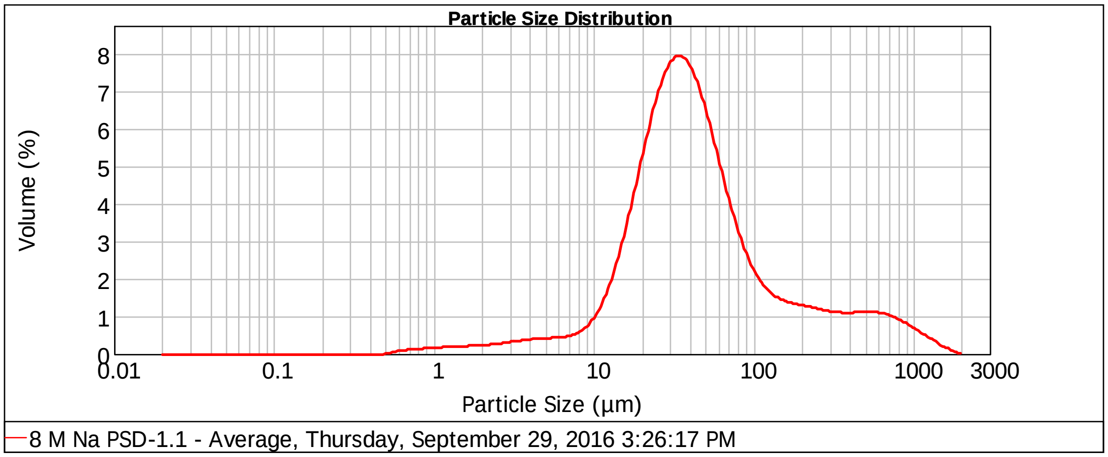
Result units:
Volume

Specific Surface Area:
0.123 m²/g

Surface Weighted Mean D[3,2]:
20.845 um

Vol. Weighted Mean D[4,3]:
110.611 um

d(0.1): 14.566 um **d(0.5): 39.520 um** **d(0.9): 283.079 um**



Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %
0.020	0.00	0.142	0.00	1.002	0.13	7.096	0.39	50.238	4.59	355.656	0.83
0.022	0.00	0.159	0.00	1.125	0.14	7.962	0.47	56.368	3.97	399.052	0.83
0.025	0.00	0.178	0.00	1.262	0.15	8.934	0.62	63.246	3.34	447.744	0.84
0.028	0.00	0.200	0.00	1.416	0.15	10.024	0.85	70.963	2.76	502.377	0.84
0.032	0.00	0.224	0.00	1.589	0.16	11.247	1.19	79.621	2.26	563.677	0.83
0.036	0.00	0.252	0.00	1.783	0.17	12.619	1.64	89.337	1.85	632.456	0.80
0.040	0.00	0.283	0.00	2.000	0.18	14.159	2.21	100.237	1.54	709.627	0.74
0.045	0.00	0.317	0.00	2.244	0.20	15.887	2.85	112.468	1.32	796.214	0.66
0.050	0.00	0.356	0.00	2.518	0.22	17.825	3.57	126.191	1.18	893.367	0.57
0.056	0.00	0.399	0.00	2.825	0.24	20.000	4.27	141.589	1.09	1002.374	0.47
0.063	0.00	0.448	0.00	3.170	0.26	22.440	4.93	158.866	1.03	1124.683	0.37
0.071	0.00	0.502	0.02	3.557	0.28	25.179	5.46	178.250	0.99	1261.915	0.28
0.080	0.00	0.564	0.06	3.991	0.30	28.251	5.82	200.000	0.96	1415.892	0.16
0.089	0.00	0.632	0.08	4.477	0.31	31.698	5.97	224.404	0.93	1588.656	0.09
0.100	0.00	0.710	0.09	5.024	0.32	35.566	5.90	251.785	0.89	1782.502	0.02
0.112	0.00	0.796	0.11	5.637	0.33	39.905	5.62	282.508	0.86	2000.000	
0.126	0.00	0.893	0.12	6.325	0.35	44.774	5.17	316.979	0.84		
0.142		1.002		7.096		50.238		355.656			

Operator notes:

MASTERSIZER 2000

Result Analysis Report

Sample Name:
8 M Na PSD-1.2 - Average

SOP Name:

Measured:
Thursday, September 29, 2016 3:27:58 PM

Sample Source & type:

Measured by:
D3M966

Analysed:
Thursday, September 29, 2016 3:28:00 PM

Sample bulk lot ref:

Result Source:
Averaged

Particle Name:
Sodium Oxalate

Accessory Name:
Hydro 2000µP (A)

Analysis model:
General purpose

Sensitivity:
Normal

Particle RI:
1.480

Absorption:
1

Size range:
0.020 to 2000.000 um

Obscuration:
12.82 %

Dispersant Name:
Water

Dispersant RI:
1.330

Weighted Residual:
0.209 %

Result Emulation:
Off

Concentration:
0.0531 %Vol

Span :
2.261

Uniformity:
1.42

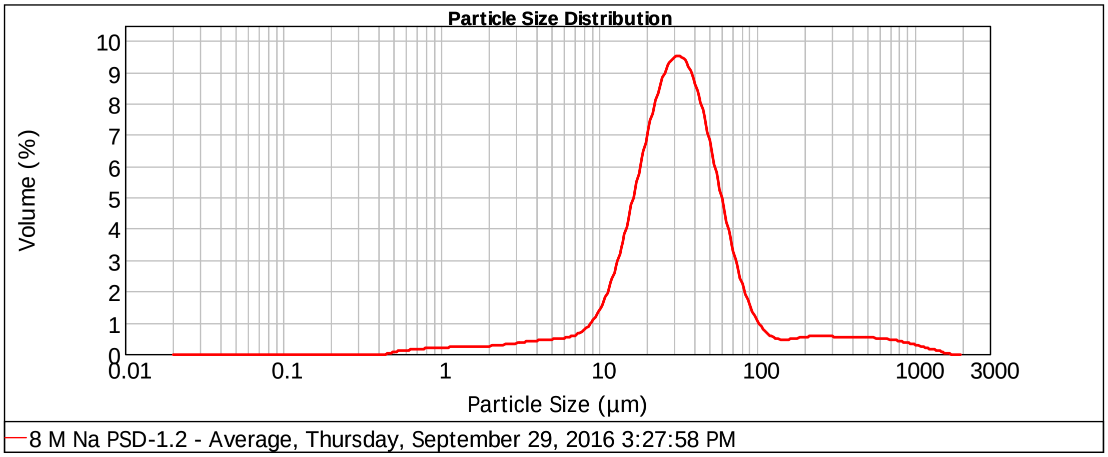
Result units:
Volume

Specific Surface Area:
0.15 m²/g

Surface Weighted Mean D[3,2]:
17.073 um

Vol. Weighted Mean D[4,3]:
64.629 um

d(0.1): **12.655** **um** **d(0.5):** **31.832** **um** **d(0.9):** **84.633** **um**



Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %
0.020	0.00	0.142	0.00	1.002	0.16	7.096	0.50	50.238	4.70	355.656	0.40
0.022	0.00	0.159	0.00	1.125	0.16	7.962	0.64	56.368	3.79	399.052	0.39
0.025	0.00	0.178	0.00	1.262	0.17	8.934	0.87	63.246	2.90	447.744	0.39
0.028	0.00	0.200	0.00	1.416	0.17	10.024	1.22	70.963	2.13	502.377	0.39
0.032	0.00	0.224	0.00	1.589	0.18	11.247	1.69	79.621	1.49	563.677	0.38
0.036	0.00	0.252	0.00	1.783	0.18	12.619	2.31	89.337	1.00	632.456	0.36
0.040	0.00	0.283	0.00	2.000	0.20	14.159	3.05	100.237	0.67	709.627	0.33
0.045	0.00	0.317	0.00	2.244	0.21	15.887	3.87	112.468	0.47	796.214	0.29
0.050	0.00	0.356	0.00	2.518	0.23	17.825	4.74	126.191	0.37	893.367	0.24
0.056	0.00	0.399	0.00	2.825	0.26	20.000	5.57	141.589	0.34	1002.374	0.19
0.063	0.00	0.448	0.01	3.170	0.28	22.440	6.30	158.866	0.36	1124.683	0.14
0.071	0.00	0.502	0.07	3.557	0.31	25.179	6.83	178.250	0.39	1261.915	0.10
0.080	0.00	0.564	0.09	3.991	0.33	28.251	7.13	200.000	0.42	1415.892	0.05
0.089	0.00	0.632	0.11	4.477	0.34	31.698	7.14	224.404	0.43	1588.656	0.00
0.100	0.00	0.710	0.12	5.024	0.36	35.566	6.86	251.785	0.43	1782.502	0.00
0.112	0.00	0.796	0.14	5.637	0.38	39.905	6.32	282.508	0.42	2000.000	
0.126	0.00	0.893	0.15	6.325	0.42	44.774	5.58	316.979	0.41		
0.142		1.002		7.096		50.238		355.656			

Operator notes:

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INSTRUMENTS

MASTERSIZER 2000

Result Analysis Report

Sample Name:
8 M Na PSD-1.3 - Average

SOP Name:

Measured:
Thursday, September 29, 2016 3:29:26 PM

Sample Source & type:

Measured by:
D3M966

Analysed:
Thursday, September 29, 2016 3:29:27 PM

Sample bulk lot ref:

Result Source:
Averaged

Particle Name:
Sodium Oxalate

Accessory Name:
Hydro 2000µP (A)

Analysis model:
General purpose

Sensitivity:
Normal

Particle RI:
1.480

Absorption:
1

Size range:
0.020 to 2000.000 um

Obscuration:
12.67 %

Dispersant Name:
Water

Dispersant RI:
1.330

Weighted Residual:
0.193 %

Result Emulation:
Off

Concentration:
0.0492 %Vol

Span :
1.761

Uniformity:
0.78

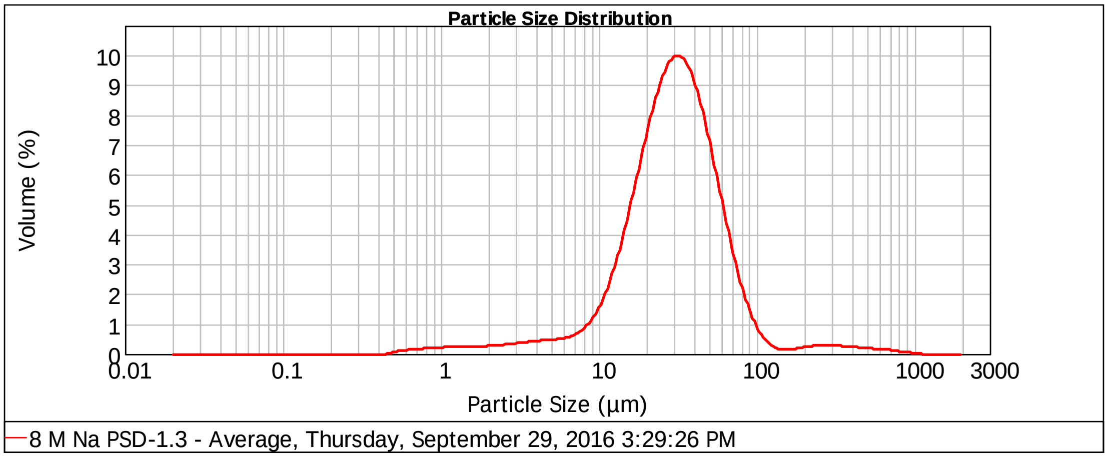
Result units:
Volume

Specific Surface Area:
0.16 m²/g

Surface Weighted Mean D[3,2]:
16.036 um

Vol. Weighted Mean D[4,3]:
42.066 um

d(0.1): 12.068 um **d(0.5): 30.190 um** **d(0.9): 65.240 um**



Size (µm)	Volume In %
0.020	0.00
0.022	0.00
0.025	0.00
0.028	0.00
0.032	0.00
0.036	0.00
0.040	0.00
0.045	0.00
0.050	0.00
0.056	0.00
0.063	0.00
0.071	0.00
0.080	0.00
0.089	0.00
0.100	0.00
0.112	0.00
0.126	0.00
0.142	

Size (µm)	Volume In %
0.142	0.00
0.159	0.00
0.178	0.00
0.200	0.00
0.224	0.00
0.252	0.00
0.283	0.00
0.317	0.00
0.356	0.00
0.399	0.00
0.448	0.02
0.502	0.07
0.564	0.09
0.632	0.12
0.710	0.13
0.796	0.15
0.893	0.16
1.002	

Size (µm)	Volume In %
1.002	0.17
1.125	0.18
1.262	0.18
1.416	0.19
1.589	0.19
1.783	0.20
2.000	0.21
2.244	0.23
2.518	0.24
2.825	0.27
3.170	0.29
3.557	0.31
3.991	0.33
4.477	0.35
5.024	0.37
5.637	0.40
6.325	0.45
7.096	

Size (µm)	Volume In %
7.096	0.55
7.962	0.72
8.934	0.98
10.024	1.37
11.247	1.88
12.619	2.54
14.159	3.32
15.887	4.18
17.825	5.08
20.000	5.92
22.440	6.66
25.179	7.20
28.251	7.49
31.698	7.48
35.566	7.18
39.905	6.60
44.774	5.83
50.238	

Size (µm)	Volume In %
50.238	4.90
56.368	3.94
63.246	2.99
70.963	2.15
79.621	1.44
89.337	0.90
100.237	0.51
112.468	0.27
126.191	0.15
141.589	0.10
158.866	0.11
178.250	0.15
200.000	0.19
224.404	0.21
251.785	0.23
282.508	0.22
316.979	0.21
355.656	

Size (µm)	Volume In %
355.656	0.19
399.052	0.17
447.744	0.15
502.377	0.14
563.677	0.12
632.456	0.11
709.627	0.09
796.214	0.05
893.367	0.03
1002.374	0.01
1124.683	0.00
1261.915	0.00
1415.892	0.00
1588.656	0.00
1782.502	0.00
2000.000	

Operator notes:

MASTERSIZER 2000

Result Analysis Report

Sample Name:
6 M Na PSD-1.1 - Average

SOP Name:

Measured:
Thursday, September 29, 2016 3:49:08 PM

Sample Source & type:

Measured by:
D3M966

Analysed:
Thursday, September 29, 2016 3:49:09 PM

Sample bulk lot ref:

Result Source:
Averaged

Particle Name:
Sodium Oxalate

Accessory Name:
Hydro 2000μP (A)

Analysis model:
General purpose

Sensitivity:
Normal

Particle RI:
1.480

Absorption:
1

Size range:
0.020 to 2000.000 um

Obscuration:
12.48 %

Dispersant Name:
Water

Dispersant RI:
1.330

Weighted Residual:
0.220 %

Result Emulation:
Off

Concentration:
0.0588 %Vol

Span :
2.303

Uniformity:
0.689

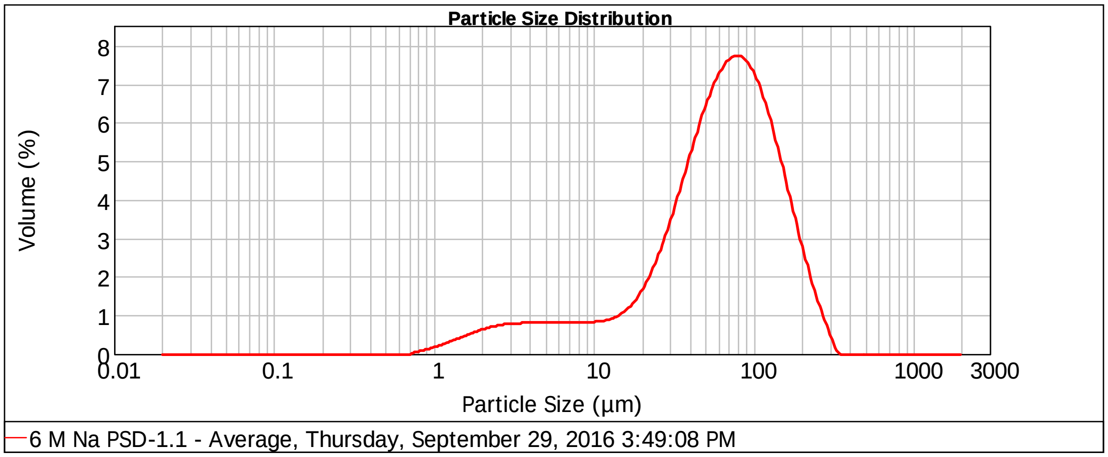
Result units:
Volume

Specific Surface Area:
0.132 m²/g

Surface Weighted Mean D[3,2]:
19.433 um

Vol. Weighted Mean D[4,3]:
77.111 um

d(0.1): 9.434 um **d(0.5): 64.766 um** **d(0.9): 158.583 um**



Size (μm)	Volume In %
0.020	0.00
0.022	0.00
0.025	0.00
0.028	0.00
0.032	0.00
0.036	0.00
0.040	0.00
0.045	0.00
0.050	0.00
0.056	0.00
0.063	0.00
0.071	0.00
0.080	0.00
0.089	0.00
0.100	0.00
0.112	0.00
0.126	0.00
0.142	

Size (μm)	Volume In %
0.142	0.00
0.159	0.00
0.178	0.00
0.200	0.00
0.224	0.00
0.252	0.00
0.283	0.00
0.317	0.00
0.356	0.00
0.399	0.00
0.448	0.00
0.502	0.00
0.564	0.00
0.632	0.00
0.710	0.02
0.796	0.07
0.893	0.11
1.002	

Size (μm)	Volume In %
1.002	0.15
1.125	0.21
1.262	0.27
1.416	0.33
1.589	0.39
1.783	0.45
2.000	0.50
2.244	0.54
2.518	0.57
2.825	0.59
3.170	0.60
3.557	0.60
3.991	0.61
4.477	0.61
5.024	0.61
5.637	0.62
6.325	0.62
7.096	

Size (μm)	Volume In %
7.096	0.62
7.962	0.62
8.934	0.62
10.024	0.63
11.247	0.66
12.619	0.70
14.159	0.79
15.887	0.92
17.825	1.12
20.000	1.38
22.440	1.71
25.179	2.11
28.251	2.57
31.698	3.07
35.566	3.60
39.905	4.13
44.774	4.62
50.238	

Size (μm)	Volume In %
50.238	5.07
56.368	5.43
63.246	5.69
70.963	5.81
79.621	5.80
89.337	5.63
100.237	5.33
112.468	4.88
126.191	4.34
141.589	3.73
158.866	3.09
178.250	2.44
200.000	1.85
224.404	1.30
251.785	0.83
282.508	0.42
316.979	0.02
355.656	

Size (μm)	Volume In %
355.656	0.00
399.052	0.00
447.744	0.00
502.377	0.00
563.677	0.00
632.456	0.00
709.627	0.00
796.214	0.00
893.367	0.00
1002.374	0.00
1124.683	0.00
1261.915	0.00
1415.892	0.00
1588.656	0.00
1782.502	0.00
2000.000	

Operator notes:

MASTERSIZER 2000

Result Analysis Report

Sample Name:
6 M Na PSD-1.2 - Average

SOP Name:

Measured:
Thursday, September 29, 2016 3:50:45 PM

Sample Source & type:

Measured by:
D3M966

Analysed:
Thursday, September 29, 2016 3:50:47 PM

Sample bulk lot ref:

Result Source:
Averaged

Particle Name:
Sodium Oxalate

Accessory Name:
Hydro 2000μP (A)

Analysis model:
General purpose

Sensitivity:
Normal

Particle RI:
1.480

Absorption:
1

Size range:
0.020 to 2000.000 um

Obscuration:
8.96 %

Dispersant Name:
Water

Dispersant RI:
1.330

Weighted Residual:
0.450 %

Result Emulation:
Off

Concentration:
0.0174 %Vol

Span :
3.892

Uniformity:
1.22

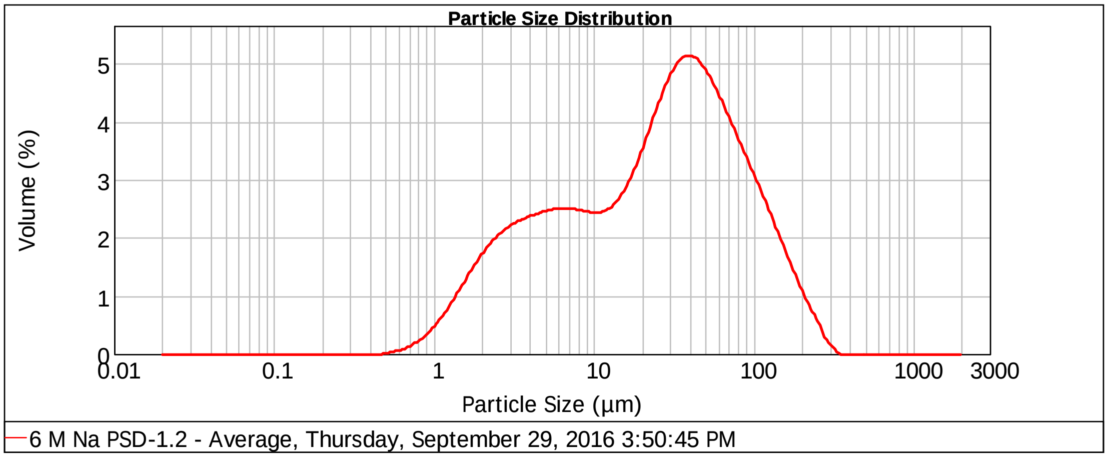
Result units:
Volume

Specific Surface Area:
0.312 m²/g

Surface Weighted Mean D[3,2]:
8.229 um

Vol. Weighted Mean D[4,3]:
42.491 um

d(0.1): 2.815 um **d(0.5): 26.660 um** **d(0.9): 106.578 um**



Size (μm)	Volume In %
0.020	0.00
0.022	0.00
0.025	0.00
0.028	0.00
0.032	0.00
0.036	0.00
0.040	0.00
0.045	0.00
0.050	0.00
0.056	0.00
0.063	0.00
0.071	0.00
0.080	0.00
0.089	0.00
0.100	0.00
0.112	0.00
0.126	0.00
0.142	

Size (μm)	Volume In %
0.142	0.00
0.159	0.00
0.178	0.00
0.200	0.00
0.224	0.00
0.252	0.00
0.283	0.00
0.317	0.00
0.356	0.00
0.399	0.00
0.448	0.00
0.502	0.02
0.564	0.04
0.632	0.07
0.710	0.13
0.796	0.20
0.893	0.29
1.002	

Size (μm)	Volume In %
1.002	0.41
1.125	0.56
1.262	0.72
1.416	0.89
1.589	1.06
1.783	1.22
2.000	1.36
2.244	1.49
2.518	1.58
2.825	1.66
3.170	1.72
3.557	1.76
3.991	1.80
4.477	1.84
5.024	1.87
5.637	1.88
6.325	1.89
7.096	

Size (μm)	Volume In %
7.096	1.88
7.962	1.86
8.934	1.84
10.024	1.83
11.247	1.86
12.619	1.94
14.159	2.07
15.887	2.26
17.825	2.50
20.000	2.78
22.440	3.08
25.179	3.37
28.251	3.61
31.698	3.78
35.566	3.86
39.905	3.85
44.774	3.76
50.238	

Size (μm)	Volume In %
50.238	3.59
56.368	3.39
63.246	3.16
70.963	2.92
79.621	2.69
89.337	2.45
100.237	2.22
112.468	1.97
126.191	1.72
141.589	1.46
158.866	1.20
178.250	0.95
200.000	0.71
224.404	0.52
251.785	0.31
282.508	0.14
316.979	0.02
355.656	

Size (μm)	Volume In %
355.656	0.00
399.052	0.00
447.744	0.00
502.377	0.00
563.677	0.00
632.456	0.00
709.627	0.00
796.214	0.00
893.367	0.00
1002.374	0.00
1124.683	0.00
1261.915	0.00
1415.892	0.00
1588.656	0.00
1782.502	0.00
2000.000	

Operator notes:

MALVERN INSTRUMENTS

MASTERSIZER 2000

Result Analysis Report

Sample Name:
6 M Na PSD-1.3 - Average

SOP Name:

Measured:
Thursday, September 29, 2016 3:52:25 PM

Sample Source & type:

Measured by:
D3M966

Analysed:
Thursday, September 29, 2016 3:52:27 PM

Sample bulk lot ref:

Result Source:
Averaged

Particle Name:
Sodium Oxalate

Accessory Name:
Hydro 2000µP (A)

Analysis model:
General purpose

Sensitivity:
Normal

Particle RI:
1.480

Absorption:
1

Size range:
0.020 to 2000.000 um

Obscuration:
7.46 %

Dispersant Name:
Water

Dispersant RI:
1.330

Weighted Residual:
0.612 %

Result Emulation:
Off

Concentration:
0.0110 %Vol

Span :
7.774

Uniformity:
2.31

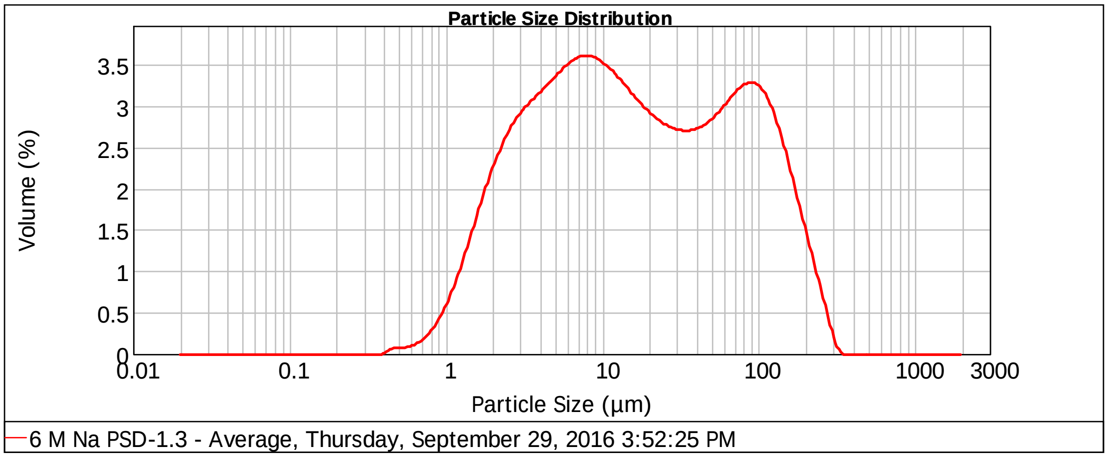
Result units:
Volume

Specific Surface Area:
0.397 m²/g

Surface Weighted Mean D[3,2]:
6.460 um

Vol. Weighted Mean D[4,3]:
40.892 um

d(0.1): **2.353** **um**   **d(0.5):** **15.111** **um**   **d(0.9):** **119.830** **um**



Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %
0.020	0.00	0.142	0.00	1.002	0.53	7.096	2.71	50.238	2.17	355.656	0.00
0.022	0.00	0.159	0.00	1.125	0.71	7.962	2.71	56.368	2.26	399.052	0.00
0.025	0.00	0.178	0.00	1.262	0.93	8.934	2.68	63.246	2.34	447.744	0.00
0.028	0.00	0.200	0.00	1.416	1.16	10.024	2.63	70.963	2.42	502.377	0.00
0.032	0.00	0.224	0.00	1.589	1.39	11.247	2.56	79.621	2.47	563.677	0.00
0.036	0.00	0.252	0.00	1.783	1.60	12.619	2.48	89.337	2.46	632.456	0.00
0.040	0.00	0.283	0.00	2.000	1.80	14.159	2.39	100.237	2.40	709.627	0.00
0.045	0.00	0.317	0.00	2.244	1.96	15.887	2.31	112.468	2.27	796.214	0.00
0.050	0.00	0.356	0.00	2.518	2.09	17.825	2.23	126.191	2.07	893.367	0.00
0.056	0.00	0.399	0.02	2.825	2.19	20.000	2.17	141.589	1.82	1002.374	0.00
0.063	0.00	0.448	0.05	3.170	2.27	22.440	2.11	158.866	1.55	1124.683	0.00
0.071	0.00	0.502	0.05	3.557	2.35	25.179	2.07	178.250	1.26	1261.915	0.00
0.080	0.00	0.564	0.07	3.991	2.42	28.251	2.04	200.000	0.98	1415.892	0.00
0.089	0.00	0.632	0.11	4.477	2.49	31.698	2.03	224.404	0.71	1588.656	0.00
0.100	0.00	0.710	0.16	5.024	2.57	35.566	2.04	251.785	0.44	1782.502	0.00
0.112	0.00	0.796	0.25	5.637	2.63	39.905	2.06	282.508	0.17	2000.000	
0.126	0.00	0.893	0.37	6.325	2.69	44.774	2.11	316.979	0.01		
0.142		1.002		7.096		50.238		355.656			

Operator notes:

Result Analysis Report

Sample Name:
5.6 M Na PSD-1.1 - Average

SOP Name:

Measured:
Thursday, September 29, 2016 4:08:27 PM

Sample Source & type:

Measured by:
D3M966

Analysed:
Thursday, September 29, 2016 4:08:28 PM

Sample bulk lot ref:

Result Source:
Averaged

Particle Name:
Sodium Oxalate

Accessory Name:
Hydro 2000µP (A)

Analysis model:
General purpose

Sensitivity:
Normal

Particle RI:
1.480

Absorption:
1

Size range:
0.020 to 2000.000 um

Obscuration:
17.69 %

Dispersant Name:
Water

Dispersant RI:
1.330

Weighted Residual:
0.184 %

Result Emulation:
Off

Concentration:
0.0607 %Vol

Span :
1.920

Uniformity:
0.661

Result units:
Volume

Specific Surface Area:
0.184 m²/g

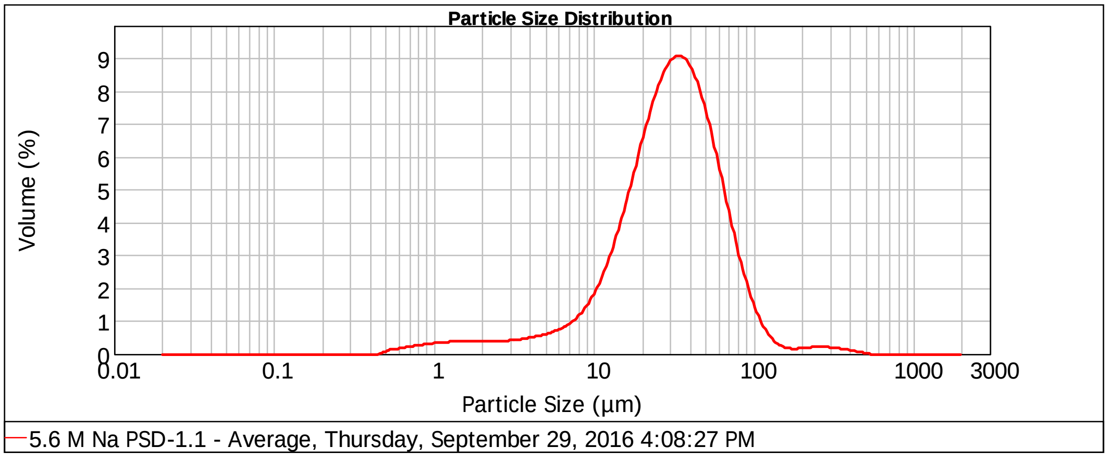
Surface Weighted Mean D[3,2]:
13.927 um

Vol. Weighted Mean D[4,3]:
38.230 um

d(0.1): **10.024** **um**

d(0.5): **30.875** **um**

d(0.9): **69.298** **um**



Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %
0.020	0.00	0.142	0.00	1.002	0.26	7.096	0.77	50.238	5.21	355.656	0.10
0.022	0.00	0.159	0.00	1.125	0.27	7.962	0.96	56.368	4.42	399.052	0.07
0.025	0.00	0.178	0.00	1.262	0.28	8.934	1.21	63.246	3.58	447.744	0.04
0.028	0.00	0.200	0.00	1.416	0.29	10.024	1.55	70.963	2.77	502.377	0.00
0.032	0.00	0.224	0.00	1.589	0.29	11.247	1.98	79.621	2.03	563.677	0.00
0.036	0.00	0.252	0.00	1.783	0.29	12.619	2.51	89.337	1.40	632.456	0.00
0.040	0.00	0.283	0.00	2.000	0.29	14.159	3.12	100.237	0.91	709.627	0.00
0.045	0.00	0.317	0.00	2.244	0.29	15.887	3.80	112.468	0.54	796.214	0.00
0.050	0.00	0.356	0.00	2.518	0.30	17.825	4.51	126.191	0.31	893.367	0.00
0.056	0.00	0.399	0.00	2.825	0.31	20.000	5.20	141.589	0.18	1002.374	0.00
0.063	0.00	0.448	0.02	3.170	0.33	22.440	5.84	158.866	0.12	1124.683	0.00
0.071	0.00	0.502	0.09	3.557	0.36	25.179	6.35	178.250	0.12	1261.915	0.00
0.080	0.00	0.564	0.12	3.991	0.39	28.251	6.69	200.000	0.14	1415.892	0.00
0.089	0.00	0.632	0.16	4.477	0.43	31.698	6.83	224.404	0.16	1588.656	0.00
0.100	0.00	0.710	0.19	5.024	0.48	35.566	6.74	251.785	0.16	1782.502	0.00
0.112	0.00	0.796	0.21	5.637	0.55	39.905	6.42	282.508	0.15	2000.000	
0.126	0.00	0.893	0.24	6.325	0.64	44.774	5.90	316.979	0.13		
0.142		1.002		7.096		50.238		355.656			

Operator notes:

MASTERSIZER 2000

Result Analysis Report

Sample Name:
5.6 M Na PSD-1.2 - Average

SOP Name:

Measured:
Thursday, September 29, 2016 4:10:14 PM

Sample Source & type:

Measured by:
D3M966

Analysed:
Thursday, September 29, 2016 4:10:16 PM

Sample bulk lot ref:

Result Source:
Averaged

Particle Name:
Sodium Oxalate

Accessory Name:
Hydro 2000µP (A)

Analysis model:
General purpose

Sensitivity:
Normal

Particle RI:
1.480

Absorption:
1

Size range:
0.020 to 2000.000 um

Obscuration:
18.04 %

Dispersant Name:
Water

Dispersant RI:
1.330

Weighted Residual:
0.154 %

Result Emulation:
Off

Concentration:
0.0518 %Vol

Span :
1.934

Uniformity:
0.593

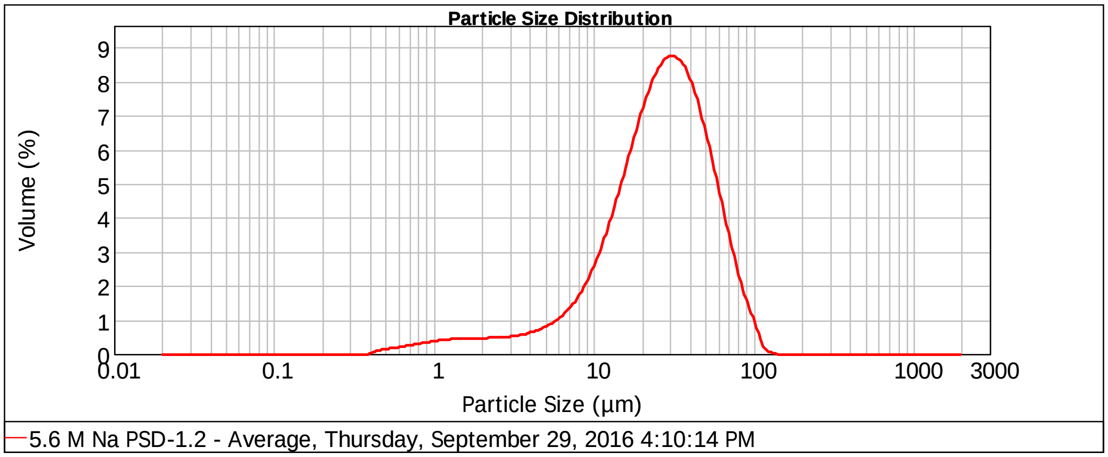
Result units:
Volume

Specific Surface Area:
0.219 m²/g

Surface Weighted Mean D[3,2]:
11.691 um

Vol. Weighted Mean D[4,3]:
30.957 um

d(0.1): 7.843 um **d(0.5): 26.833 um** **d(0.9): 59.730 um**



Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %
0.020	0.00	0.142	0.00	1.002	0.29	7.096	1.14	50.238	4.53	355.656	0.00
0.022	0.00	0.159	0.00	1.125	0.32	7.962	1.41	56.368	3.75	399.052	0.00
0.025	0.00	0.178	0.00	1.262	0.33	8.934	1.74	63.246	2.95	447.744	0.00
0.028	0.00	0.200	0.00	1.416	0.34	10.024	2.16	70.963	2.19	502.377	0.00
0.032	0.00	0.224	0.00	1.589	0.35	11.247	2.65	79.621	1.51	563.677	0.00
0.036	0.00	0.252	0.00	1.783	0.35	12.619	3.22	89.337	0.99	632.456	0.00
0.040	0.00	0.283	0.00	2.000	0.35	14.159	3.83	100.237	0.51	709.627	0.00
0.045	0.00	0.317	0.00	2.244	0.36	15.887	4.46	112.468	0.08	796.214	0.00
0.050	0.00	0.356	0.00	2.518	0.37	17.825	5.08	126.191	0.01	893.367	0.00
0.056	0.00	0.399	0.04	2.825	0.38	20.000	5.64	141.589	0.00	1002.374	0.00
0.063	0.00	0.448	0.09	3.170	0.41	22.440	6.12	158.866	0.00	1124.683	0.00
0.071	0.00	0.502	0.12	3.557	0.45	25.179	6.44	178.250	0.00	1261.915	0.00
0.080	0.00	0.564	0.15	3.991	0.50	28.251	6.59	200.000	0.00	1415.892	0.00
0.089	0.00	0.632	0.18	4.477	0.57	31.698	6.55	224.404	0.00	1588.656	0.00
0.100	0.00	0.710	0.21	5.024	0.66	35.566	6.30	251.785	0.00	1782.502	0.00
0.112	0.00	0.796	0.24	5.637	0.77	39.905	5.86	282.508	0.00	2000.000	
0.126	0.00	0.893	0.27	6.325	0.93	44.774	5.26	316.979	0.00		
0.142		1.002		7.096		50.238		355.656			

Operator notes:

MASTERSIZER 2000

Result Analysis Report

Sample Name:
5.6 M Na PSD-1.3 - Average

SOP Name:

Measured:
Thursday, September 29, 2016 4:11:41 PM

Sample Source & type:

Measured by:
D3M966

Analysed:
Thursday, September 29, 2016 4:11:43 PM

Sample bulk lot ref:

Result Source:
Averaged

Particle Name:
Sodium Oxalate

Accessory Name:
Hydro 2000μP (A)

Analysis model:
General purpose

Sensitivity:
Normal

Particle RI:
1.480

Absorption:
1

Size range:
0.020 to 2000.000 um

Obscuration:
17.79 %

Dispersant Name:
Water

Dispersant RI:
1.330

Weighted Residual:
0.173 %

Result Emulation:
Off

Concentration:
0.0511 %Vol

Span :
1.930

Uniformity:
0.591

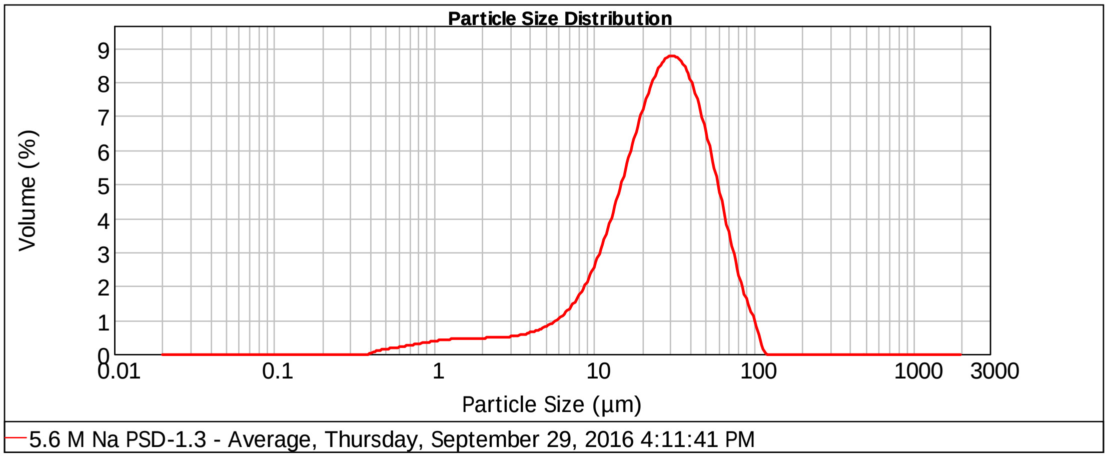
Result units:
Volume

Specific Surface Area:
0.219 m²/g

Surface Weighted Mean D[3,2]:
11.713 um

Vol. Weighted Mean D[4,3]:
30.971 um

d(0.1): 7.849 um **d(0.5): 26.902 um** **d(0.9): 59.767 um**



Size (μm)	Volume In %
0.020	0.00
0.022	0.00
0.025	0.00
0.028	0.00
0.032	0.00
0.036	0.00
0.040	0.00
0.045	0.00
0.050	0.00
0.056	0.00
0.063	0.00
0.071	0.00
0.080	0.00
0.089	0.00
0.100	0.00
0.112	0.00
0.126	0.00
0.142	

Size (μm)	Volume In %
0.142	0.00
0.159	0.00
0.178	0.00
0.200	0.00
0.224	0.00
0.252	0.00
0.283	0.00
0.317	0.00
0.356	0.00
0.399	0.04
0.448	0.09
0.502	0.12
0.564	0.15
0.632	0.18
0.710	0.21
0.796	0.24
0.893	0.27
1.002	

Size (μm)	Volume In %
1.002	0.29
1.125	0.32
1.262	0.33
1.416	0.34
1.589	0.35
1.783	0.35
2.000	0.36
2.244	0.36
2.518	0.37
2.825	0.38
3.170	0.41
3.557	0.45
3.991	0.50
4.477	0.57
5.024	0.65
5.637	0.77
6.325	0.93
7.096	

Size (μm)	Volume In %
7.096	1.13
7.962	1.40
8.934	1.73
10.024	2.15
11.247	2.64
12.619	3.20
14.159	3.81
15.887	4.44
17.825	5.07
20.000	5.63
22.440	6.11
25.179	6.44
28.251	6.60
31.698	6.56
35.566	6.32
39.905	5.88
44.774	5.29
50.238	

Size (μm)	Volume In %
50.238	4.56
56.368	3.78
63.246	2.97
70.963	2.22
79.621	1.52
89.337	1.02
100.237	0.52
112.468	0.00
126.191	0.00
141.589	0.00
158.866	0.00
178.250	0.00
200.000	0.00
224.404	0.00
251.785	0.00
282.508	0.00
316.979	0.00
355.656	

Size (μm)	Volume In %
355.656	0.00
399.052	0.00
447.744	0.00
502.377	0.00
563.677	0.00
632.456	0.00
709.627	0.00
796.214	0.00
893.367	0.00
1002.374	0.00
1124.683	0.00
1261.915	0.00
1415.892	0.00
1588.656	0.00
1782.502	0.00
2000.000	

Operator notes:

Result Analysis Report

Sample Name:
4 M Na PSD-2.1-Average

SOP Name:

Measured:
Thursday, September 29, 2016 4:40:08 PM

Sample Source & type:

Measured by:
D3M966

Analysed:
Friday, September 30, 2016 5:39:19 PM

Sample bulk lot ref:

Result Source:
Averaged

Particle Name:
Alumina

Accessory Name:
Hydro 2000µP (A)

Analysis model:
General purpose

Sensitivity:
Normal

Particle RI:
1.780

Absorption:
0.01

Size range:
0.020 to 2000.000 um

Obscuration:
11.42 %

Dispersant Name:
Water

Dispersant RI:
1.330

Weighted Residual:
0.893 %

Result Emulation:
Off

Concentration:
0.0032 %Vol

Span :
1.202

Uniformity:
0.376

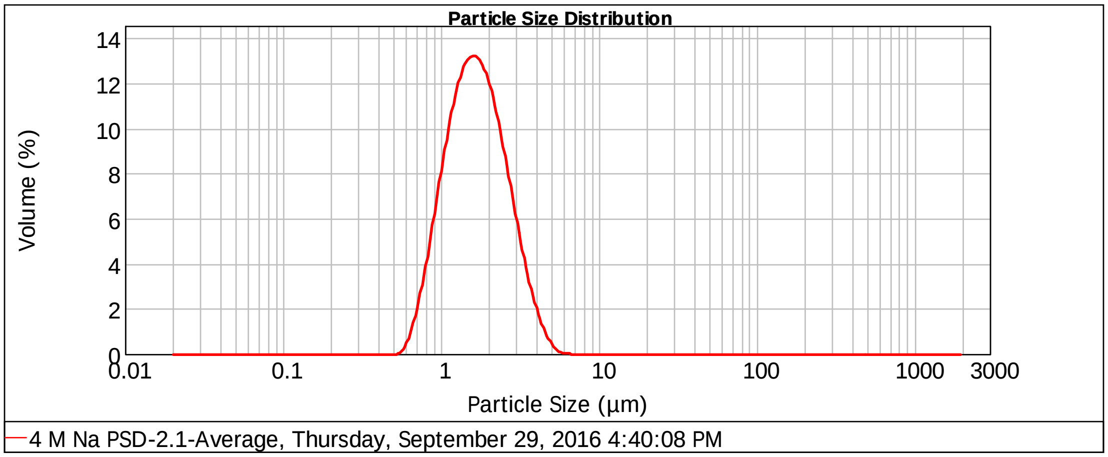
Result units:
Volume

Specific Surface Area:
3.93 m²/g

Surface Weighted Mean D[3,2]:
1.528 um

Vol. Weighted Mean D[4,3]:
1.830 um

d(0.1): 0.959 um **d(0.5): 1.654 um** **d(0.9): 2.947 um**



Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %
0.020	0.00	0.142	0.00	1.002	6.85	7.096	0.00	50.238	0.00	355.656	0.00
0.022	0.00	0.159	0.00	1.125	8.26	7.962	0.00	56.368	0.00	399.052	0.00
0.025	0.00	0.178	0.00	1.262	9.30	8.934	0.00	63.246	0.00	447.744	0.00
0.028	0.00	0.200	0.00	1.416	9.86	10.024	0.00	70.963	0.00	502.377	0.00
0.032	0.00	0.224	0.00	1.589	9.93	11.247	0.00	79.621	0.00	563.677	0.00
0.036	0.00	0.252	0.00	1.783	9.52	12.619	0.00	89.337	0.00	632.456	0.00
0.040	0.00	0.283	0.00	2.000	8.67	14.159	0.00	100.237	0.00	709.627	0.00
0.045	0.00	0.317	0.00	2.244	7.47	15.887	0.00	112.468	0.00	796.214	0.00
0.050	0.00	0.356	0.00	2.518	6.06	17.825	0.00	126.191	0.00	893.367	0.00
0.056	0.00	0.399	0.00	2.825	4.59	20.000	0.00	141.589	0.00	1002.374	0.00
0.063	0.00	0.448	0.00	3.170	3.24	22.440	0.00	158.866	0.00	1124.683	0.00
0.071	0.00	0.502	0.00	3.557	2.07	25.179	0.00	178.250	0.00	1261.915	0.00
0.080	0.00	0.564	0.27	3.991	1.18	28.251	0.00	200.000	0.00	1415.892	0.00
0.089	0.00	0.632	1.01	4.477	0.57	31.698	0.00	224.404	0.00	1588.656	0.00
0.100	0.00	0.710	2.13	5.024	0.17	35.566	0.00	251.785	0.00	1782.502	0.00
0.112	0.00	0.796	3.59	5.637	0.03	39.905	0.00	282.508	0.00	2000.000	
0.126	0.00	0.893	5.22	6.325	0.00	44.774	0.00	316.979	0.00		
0.142		1.002		7.096		50.238		355.656			

Operator notes: *Average of 3 measurements from CUF 2016-Final .mea*

MASTERSIZER 2000

Result Analysis Report

Sample Name:
4 M Na PSD-2.2-Average

SOP Name:

Measured:
Thursday, September 29, 2016 4:42:06 PM

Sample Source & type:

Measured by:
D3M966

Analysed:
Friday, September 30, 2016 5:39:20 PM

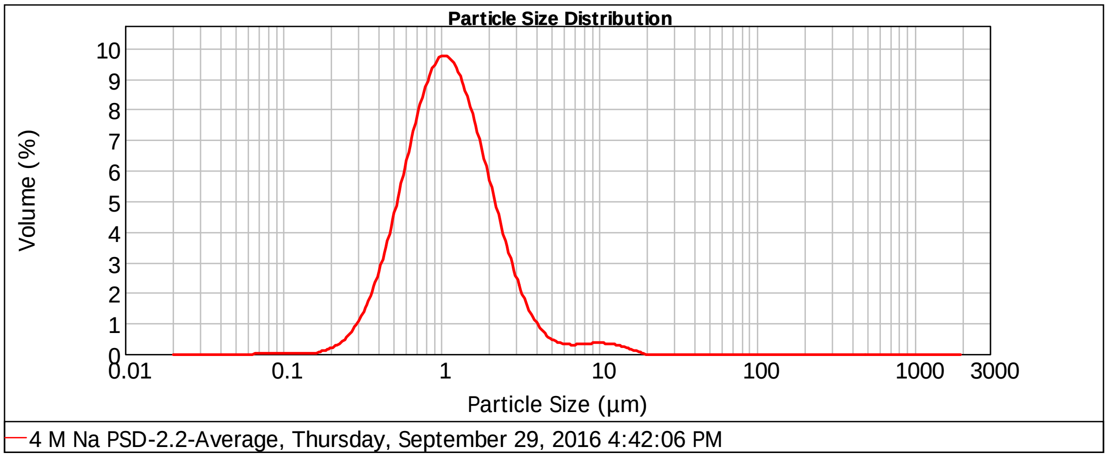
Sample bulk lot ref:

Result Source:
Averaged

Particle Name: Alumina	**Accessory Name:** Hydro 2000µP (A)	**Analysis model:** General purpose	**Sensitivity:** Normal
Particle RI: 1.780	**Absorption:** 0.01	**Size range:** 0.020 to 2000.000 um	**Obscuration:** 10.28 %
Dispersant Name: Water	**Dispersant RI:** 1.330	**Weighted Residual:** 0.991 %	**Result Emulation:** Off

Concentration: 0.0019 %Vol	**Span :** 1.882	**Uniformity:** 0.705	**Result units:** Volume
Specific Surface Area: 6.6 m²/g	**Surface Weighted Mean D[3,2]:** 0.909 um	**Vol. Weighted Mean D[4,3]:** 1.485 um	

d(0.1): 0.499 um **d(0.5): 1.095 um** **d(0.9): 2.560 um**



Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %
0.020	0.00	0.142	0.02	1.002	7.35	7.096	0.24	50.238	0.00	355.656	0.00
0.022	0.00	0.159	0.04	1.125	7.20	7.962	0.25	56.368	0.00	399.052	0.00
0.025	0.00	0.178	0.11	1.262	6.81	8.934	0.27	63.246	0.00	447.744	0.00
0.028	0.00	0.200	0.18	1.416	6.24	10.024	0.27	70.963	0.00	502.377	0.00
0.032	0.00	0.224	0.31	1.589	5.55	11.247	0.25	79.621	0.00	563.677	0.00
0.036	0.00	0.252	0.51	1.783	4.80	12.619	0.21	89.337	0.00	632.456	0.00
0.040	0.00	0.283	0.78	2.000	4.03	14.159	0.15	100.237	0.00	709.627	0.00
0.045	0.00	0.317	1.16	2.244	3.26	15.887	0.09	112.468	0.00	796.214	0.00
0.050	0.00	0.356	1.66	2.518	2.55	17.825	0.01	126.191	0.00	893.367	0.00
0.056	0.00	0.399	2.26	2.825	1.91	20.000	0.00	141.589	0.00	1002.374	0.00
0.063	0.01	0.448	2.98	3.170	1.38	22.440	0.00	158.866	0.00	1124.683	0.00
0.071	0.03	0.502	3.78	3.557	0.96	25.179	0.00	178.250	0.00	1261.915	0.00
0.080	0.03	0.564	4.64	3.991	0.65	28.251	0.00	200.000	0.00	1415.892	0.00
0.089	0.03	0.632	5.47	4.477	0.44	31.698	0.00	224.404	0.00	1588.656	0.00
0.100	0.03	0.710	6.23	5.024	0.31	35.566	0.00	251.785	0.00	1782.502	0.00
0.112	0.03	0.796	6.83	5.637	0.25	39.905	0.00	282.508	0.00	2000.000	
0.126	0.02	0.893	7.22	6.325	0.23	44.774	0.00	316.979	0.00		
0.142		1.002		7.096		50.238		355.656			

Operator notes: *Average of 3 measurements from CUF 2016-Final .mea*

Result Analysis Report

Sample Name:
4 M Na PSD-2.3-Average

SOP Name:

Measured:
Thursday, September 29, 2016 4:43:31 PM

Sample Source & type:

Measured by:
D3M966

Analysed:
Friday, September 30, 2016 5:39:20 PM

Sample bulk lot ref:

Result Source:
Averaged

Particle Name:
Alumina

Accessory Name:
Hydro 2000µP (A)

Analysis model:
General purpose

Sensitivity:
Normal

Particle RI:
1.780

Absorption:
0.01

Size range:
0.020 to 2000.000 um

Obscuration:
10.31 %

Dispersant Name:
Water

Dispersant RI:
1.330

Weighted Residual:
0.989 %

Result Emulation:
Off

Concentration:
0.0021 %Vol

Span :
1.895

Uniformity:
0.753

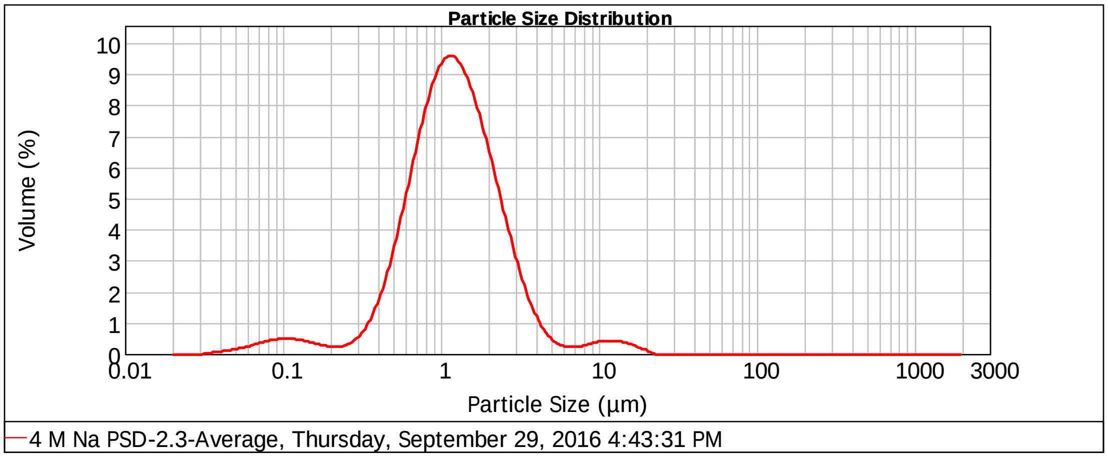
Result units:
Volume

Specific Surface Area:
8.05 m²/g

Surface Weighted Mean D[3,2]:
0.745 um

Vol. Weighted Mean D[4,3]:
1.625 um

d(0.1): 0.506 um   **d(0.5): 1.181 um**   **d(0.9): 2.743 um**



Size (µm)	Volume In %
0.020	0.00
0.022	0.00
0.025	0.00
0.028	0.00
0.032	0.02
0.036	0.04
0.040	0.07
0.045	0.10
0.050	0.14
0.056	0.18
0.063	0.24
0.071	0.29
0.080	0.33
0.089	0.36
0.100	0.37
0.112	0.36
0.126	0.33
0.142	

Size (µm)	Volume In %
0.142	0.28
0.159	0.23
0.178	0.19
0.200	0.17
0.224	0.18
0.252	0.25
0.283	0.40
0.317	0.64
0.356	1.01
0.399	1.52
0.448	2.16
0.502	2.91
0.564	3.77
0.632	4.65
0.710	5.51
0.796	6.26
0.893	6.83
1.002	

Size (µm)	Volume In %
1.002	7.16
1.125	7.22
1.262	7.02
1.416	6.61
1.589	6.04
1.783	5.37
2.000	4.62
2.244	3.83
2.518	3.05
2.825	2.33
3.170	1.69
3.557	1.16
3.991	0.76
4.477	0.47
5.024	0.28
5.637	0.19
6.325	0.16
7.096	

Size (µm)	Volume In %
7.096	0.18
7.962	0.23
8.934	0.28
10.024	0.31
11.247	0.32
12.619	0.30
14.159	0.27
15.887	0.21
17.825	0.12
20.000	0.04
22.440	0.00
25.179	0.00
28.251	0.00
31.698	0.00
35.566	0.00
39.905	0.00
44.774	0.00
50.238	

Size (µm)	Volume In %
50.238	0.00
56.368	0.00
63.246	0.00
70.963	0.00
79.621	0.00
89.337	0.00
100.237	0.00
112.468	0.00
126.191	0.00
141.589	0.00
158.866	0.00
178.250	0.00
200.000	0.00
224.404	0.00
251.785	0.00
282.508	0.00
316.979	0.00
355.656	

Size (µm)	Volume In %
355.656	0.00
399.052	0.00
447.744	0.00
502.377	0.00
563.677	0.00
632.456	0.00
709.627	0.00
796.214	0.00
893.367	0.00
1002.374	0.00
1124.683	0.00
1261.915	0.00
1415.892	0.00
1588.656	0.00
1782.502	0.00
2000.000	

Operator notes: *Average of 3 measurements from CUF 2016-Final .mea*

C.2 LAWPS Fouling Simulants

The size distribution of select solid samples associated with LAWPS Series 2 fouling tests and listed in Table C.3 were measured. The majority of samples corresponded to solids collected at the end of LAWPS CFF fouling tests (denoted as being collected “After CFF Testing”). Other samples were collected shortly after addition of the test simulant to the CFF test apparatus slurry reservoir but before extensive CFF testing (i.e., “Before CFF Testing”). In the case of iron phosphate solids, one PSD measurement evaluates the stock iron phosphate powder provided by Sigma-Aldrich. The suspending phases used to dilute/suspend the test PSD samples are listed in Table C.3. In addition to the fouling simulants, Series 2 PSD measurements also captured select Series 1 samples associated with 5.6 M Na simulant sodium oxalate solids after testing at 20 °C and 45 °C.

Table C.3. List of LAWPS fouling simulant sample IDs and descriptions.

ID	Description	Suspending Phase
S2-Test3-001-iron oxide	Iron Oxide (Before CFF Testing)	Modified Sodium Simulant
S2-Test3-Post Test Grab Sample-IO	Iron Oxide (After CFF Testing)	Modified Sodium Simulant
S2-Test3-Post Test Grab Sample-IP	Iron Phosphate (After CFF Testing)	Modified Sodium Simulant
S2-Test 5-001-CN	Cancrinite (Before CFF Testing)	Modified Sodium Simulant
S2-Test5-Post Test Grab Sample-CN	Cancrinite (After CFF Testing)	Modified Sodium Simulant
S2-Test8-Post Test Grab Sample-SF-01	Sodium Fluoride (Before CFF Testing)	Modified Sodium Simulant
S1-Test 1-Post Test Grab Sample-NC20-01	5.6 M Na Simulant (After CFF Testing)	5.6 M Na Simulant Supernate
S1-Test 2-Post Test Grab Sample-NC45-01	5.6 M Na Simulant (After CFF Testing at 45 °C)	5.6 M Na Simulant Supernate
S2-Test 2-Post Test Grab Sample-NA-FR-01	sRF Resin (with 0.1 wt% dissolved HEDTA and $Fe(NO_3)_3$)	Water
S2-Test2-Post Test Grab Sample-SP-01	Sodium Phosphate (After CFF Testing)	Modified Sodium Simulant
Iron (III) Phosphate Tetrahydrate	Iron Phosphate (Stock Powder)	Water

MALVERN INSTRUMENTS

MASTERSIZER 2000

Result Analysis Report

Sample Name:
S2-Test3-001-iron oxide-1.1 - Average

SOP Name:

Measured:
Friday, August 25, 2017 8:27:29 PM

Sample Source & type:

Measured by:
D3M966

Analysed:
Friday, August 25, 2017 8:27:31 PM

Sample bulk lot ref:

Result Source:
Averaged

Particle Name:
Iron III Oxide 2.98

Accessory Name:
Hydro 2000μP (A)

Analysis model:
General purpose

Sensitivity:
Normal

Particle RI:
2.980

Absorption:
1

Size range:
0.020 to 2000.000 um

Obscuration:
17.94 %

Dispersant Name:
Water

Dispersant RI:
1.330

Weighted Residual:
0.621 %

Result Emulation:
Off

Concentration:
0.0245 %Vol

Span :
11.665

Uniformity:
3.7

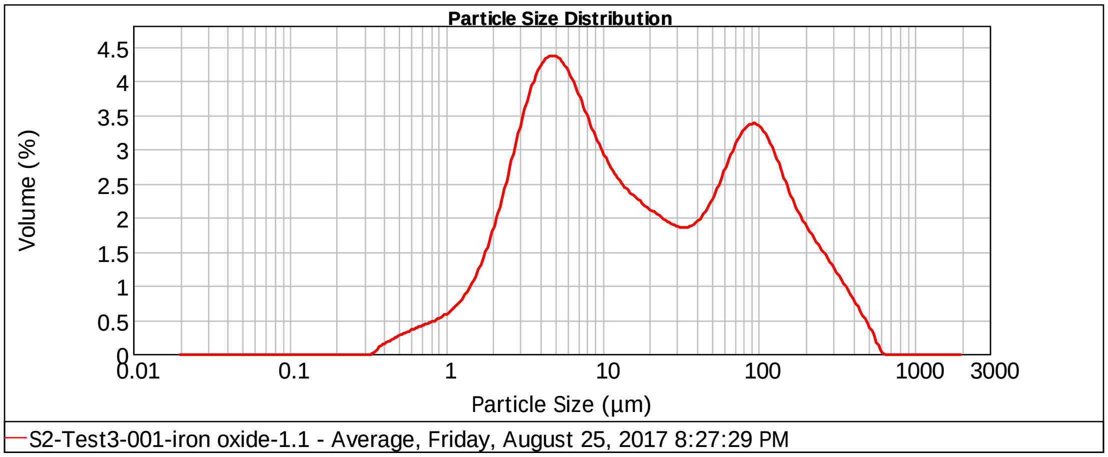
Result units:
Volume

Specific Surface Area:
1.02 m²/g

Surface Weighted Mean D[3,2]:
5.902 um

Vol. Weighted Mean D[4,3]:
56.586 um

d(0.1): 2.469 um **d(0.5): 13.852 um** **d(0.9): 164.058 um**



Size (μm)	Volume In %
0.020	0.00
0.022	0.00
0.025	0.00
0.028	0.00
0.032	0.00
0.036	0.00
0.040	0.00
0.045	0.00
0.050	0.00
0.056	0.00
0.063	0.00
0.071	0.00
0.080	0.00
0.089	0.00
0.100	0.00
0.112	0.00
0.126	0.00
0.142	

Size (μm)	Volume In %
0.142	0.00
0.159	0.00
0.178	0.00
0.200	0.00
0.224	0.00
0.252	0.00
0.283	0.00
0.317	0.00
0.356	0.08
0.399	0.13
0.448	0.18
0.502	0.22
0.564	0.26
0.632	0.30
0.710	0.33
0.796	0.37
0.893	0.41
1.002	

Size (μm)	Volume In %
1.002	0.47
1.125	0.55
1.262	0.66
1.416	0.80
1.589	0.99
1.783	1.23
2.000	1.51
2.244	1.83
2.518	2.17
2.825	2.52
3.170	2.82
3.557	3.07
3.991	3.23
4.477	3.29
5.024	3.25
5.637	3.13
6.325	2.95
7.096	

Size (μm)	Volume In %
7.096	2.74
7.962	2.52
8.934	2.32
10.024	2.14
11.247	1.99
12.619	1.87
14.159	1.78
15.887	1.70
17.825	1.63
20.000	1.57
22.440	1.50
25.179	1.45
28.251	1.41
31.698	1.39
35.566	1.42
39.905	1.49
44.774	1.62
50.238	

Size (μm)	Volume In %
50.238	1.79
56.368	2.00
63.246	2.21
70.963	2.39
79.621	2.51
89.337	2.54
100.237	2.47
112.468	2.32
126.191	2.11
141.589	1.88
158.866	1.67
178.250	1.49
200.000	1.34
224.404	1.21
251.785	1.09
282.508	0.96
316.979	0.83
355.656	

Size (μm)	Volume In %
355.656	0.69
399.052	0.54
447.744	0.39
502.377	0.24
563.677	0.04
632.456	0.00
709.627	0.00
796.214	0.00
893.367	0.00
1002.374	0.00
1124.683	0.00
1261.915	0.00
1415.892	0.00
1588.656	0.00
1782.502	0.00
2000.000	

Operator notes: *Pre test slurry, suspended in LPIST-NS-B06 (modified nominal simulant, filtered)*

MASTERSIZER 2000

Result Analysis Report

Sample Name:
S2-Test3-001-iron oxide-1.2 - Average

SOP Name:

Measured:
Friday, August 25, 2017 8:29:42 PM

Sample Source & type:

Measured by:
D3M966

Analysed:
Friday, August 25, 2017 8:29:44 PM

Sample bulk lot ref:

Result Source:
Averaged

Particle Name:
Iron III Oxide 2.98

Accessory Name:
Hydro 2000μP (A)

Analysis model:
General purpose

Sensitivity:
Normal

Particle RI:
2.980

Absorption:
1

Size range:
0.020 to 2000.000 um

Obscuration:
19.43 %

Dispersant Name:
Water

Dispersant RI:
1.330

Weighted Residual:
0.630 %

Result Emulation:
Off

Concentration:
0.0223 %Vol

Span :
8.938

Uniformity:
2.89

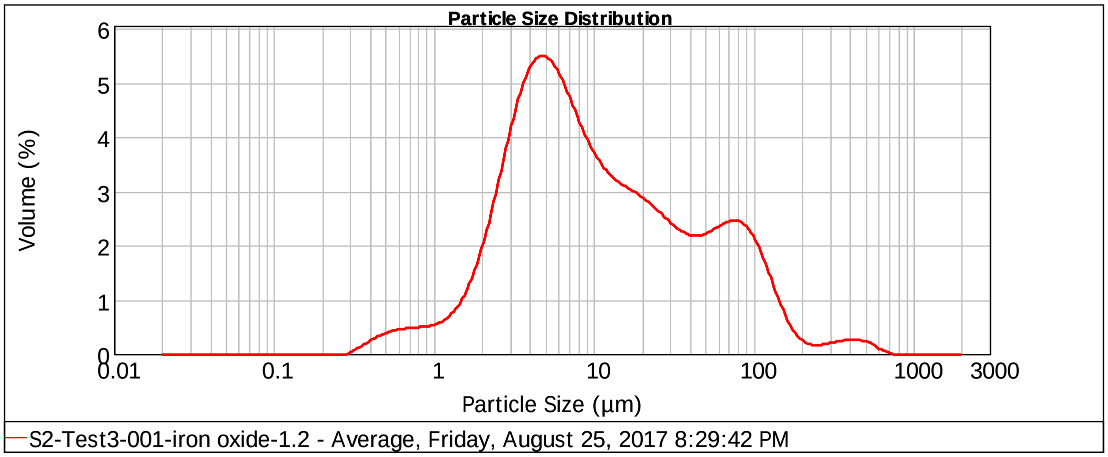
Result units:
Volume

Specific Surface Area:
1.22 m²/g

Surface Weighted Mean D[3,2]:
4.936 um

Vol. Weighted Mean D[4,3]:
29.414 um

d(0.1): **2.368** **um** **d(0.5):** **8.708** **um** **d(0.9):** **80.206** **um**



Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %
0.020	0.00	0.142	0.00	1.002	0.42	7.096	3.41	50.238	1.69	355.656	0.19
0.022	0.00	0.159	0.00	1.125	0.48	7.962	3.14	56.368	1.76	399.052	0.20
0.025	0.00	0.178	0.00	1.262	0.59	8.934	2.90	63.246	1.82	447.744	0.18
0.028	0.00	0.200	0.00	1.416	0.75	10.024	2.70	70.963	1.86	502.377	0.16
0.032	0.00	0.224	0.00	1.589	0.99	11.247	2.55	79.621	1.82	563.677	0.08
0.036	0.00	0.252	0.00	1.783	1.29	12.619	2.44	89.337	1.71	632.456	0.04
0.040	0.00	0.283	0.01	2.000	1.68	14.159	2.35	100.237	1.51	709.627	0.00
0.045	0.00	0.317	0.09	2.244	2.12	15.887	2.29	112.468	1.24	796.214	0.00
0.050	0.00	0.356	0.15	2.518	2.60	17.825	2.21	126.191	0.94	893.367	0.00
0.056	0.00	0.399	0.22	2.825	3.08	20.000	2.13	141.589	0.65	1002.374	0.00
0.063	0.00	0.448	0.27	3.170	3.51	22.440	2.04	158.866	0.41	1124.683	0.00
0.071	0.00	0.502	0.31	3.557	3.85	25.179	1.94	178.250	0.24	1261.915	0.00
0.080	0.00	0.564	0.34	3.991	4.07	28.251	1.83	200.000	0.15	1415.892	0.00
0.089	0.00	0.632	0.35	4.477	4.14	31.698	1.74	224.404	0.13	1588.656	0.00
0.100	0.00	0.710	0.37	5.024	4.08	35.566	1.67	251.785	0.13	1782.502	0.00
0.112	0.00	0.796	0.37	5.637	3.92	39.905	1.64	282.508	0.15	2000.000	
0.126	0.00	0.893	0.39	6.325	3.68	44.774	1.65	316.979	0.17		
0.142		1.002		7.096		50.238		355.656			

Operator notes: *Pre test slurry, suspended in LPIST-NS-B06 (modified nominal simulant, filtered)*

MASTERSIZER 2000

Result Analysis Report

Sample Name:
S2-Test3-001-iron oxide-1.3 - Average

SOP Name:

Measured:
Friday, August 25, 2017 8:31:55 PM

Sample Source & type:

Measured by:
D3M966

Analysed:
Friday, August 25, 2017 8:31:56 PM

Sample bulk lot ref:

Result Source:
Averaged

Particle Name:
Iron III Oxide 2.98

Accessory Name:
Hydro 2000μP (A)

Analysis model:
General purpose

Sensitivity:
Normal

Particle RI:
2.980

Absorption:
1

Size range:
0.020 to 2000.000 um

Obscuration:
21.05 %

Dispersant Name:
Water

Dispersant RI:
1.330

Weighted Residual:
0.640 %

Result Emulation:
Off

Concentration:
0.0221 %Vol

Span :
7.298

Uniformity:
2.06

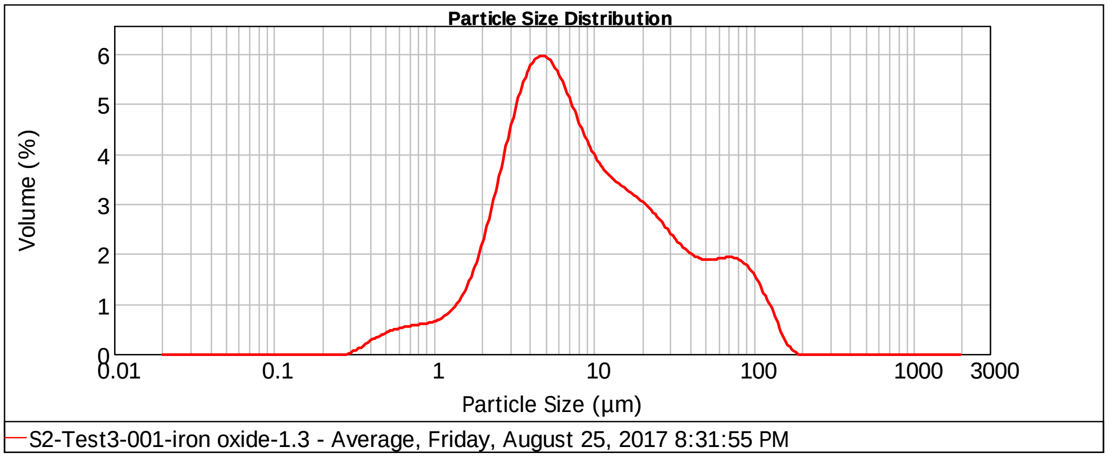
Result units:
Volume

Specific Surface Area:
1.34 m²/g

Surface Weighted Mean D[3,2]:
4.490 um

Vol. Weighted Mean D[4,3]:
19.153 um

d(0.1): 2.202 um **d(0.5): 7.436 um** **d(0.9): 56.465 um**



Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %
0.020	0.00	0.142	0.00	1.002	0.51	7.096	3.68	50.238	1.42	355.656	0.00
0.022	0.00	0.159	0.00	1.125	0.58	7.962	3.38	56.368	1.43	399.052	0.00
0.025	0.00	0.178	0.00	1.262	0.70	8.934	3.12	63.246	1.45	447.744	0.00
0.028	0.00	0.200	0.00	1.416	0.87	10.024	2.90	70.963	1.45	502.377	0.00
0.032	0.00	0.224	0.00	1.589	1.12	11.247	2.74	79.621	1.40	563.677	0.00
0.036	0.00	0.252	0.00	1.783	1.45	12.619	2.62	89.337	1.29	632.456	0.00
0.040	0.00	0.283	0.01	2.000	1.86	14.159	2.52	100.237	1.10	709.627	0.00
0.045	0.00	0.317	0.08	2.244	2.34	15.887	2.44	112.468	0.86	796.214	0.00
0.050	0.00	0.356	0.15	2.518	2.85	17.825	2.35	126.191	0.63	893.367	0.00
0.056	0.00	0.399	0.23	2.825	3.37	20.000	2.25	141.589	0.29	1002.374	0.00
0.063	0.00	0.448	0.29	3.170	3.83	22.440	2.12	158.866	0.12	1124.683	0.00
0.071	0.00	0.502	0.34	3.557	4.20	25.179	1.98	178.250	0.00	1261.915	0.00
0.080	0.00	0.564	0.38	3.991	4.42	28.251	1.83	200.000	0.00	1415.892	0.00
0.089	0.00	0.632	0.41	4.477	4.49	31.698	1.69	224.404	0.00	1588.656	0.00
0.100	0.00	0.710	0.43	5.024	4.42	35.566	1.57	251.785	0.00	1782.502	0.00
0.112	0.00	0.796	0.45	5.637	4.24	39.905	1.48	282.508	0.00	2000.000	
0.126	0.00	0.893	0.47	6.325	3.97	44.774	1.43	316.979	0.00		
0.142		1.002		7.096		50.238		355.656			

Operator notes: *Pre test slurry, suspended in LPIST-NS-B06 (modified nominal simulant, filtered)*

MALVERN INSTRUMENTS

MASTERSIZER 2000

Result Analysis Report

Sample Name:
S2-Test3-Post Test Grab Sample-IO-2.1 -

SOP Name:

Measured:
Thursday, September 14, 2017 5:00:31 PM

Sample Source & type:

Measured by:
D3M966

Analysed:
Thursday, September 14, 2017 5:00:33 PM

Sample bulk lot ref:

Result Source:
Averaged

Particle Name:
Iron III Oxide 2.98

Accessory Name:
Hydro 2000μP (A)

Analysis model:
General purpose

Sensitivity:
Normal

Particle RI:
2.980

Absorption:
1

Size range:
0.020 to 2000.000 um

Obscuration:
9.12 %

Dispersant Name:
Water

Dispersant RI:
1.330

Weighted Residual:
0.529 %

Result Emulation:
Off

Concentration:
0.0448 %Vol

Span :
1.332

Uniformity:
0.417

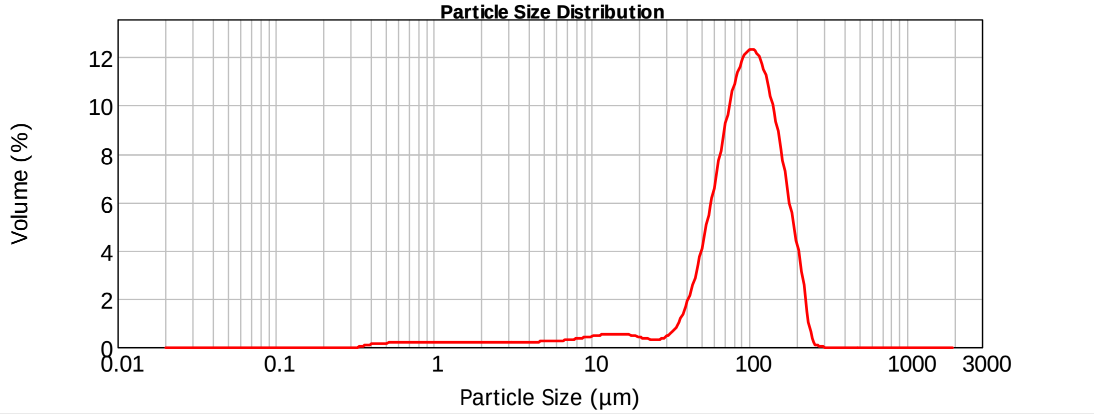
Result units:
Volume

Specific Surface Area:
0.264 m²/g

Surface Weighted Mean D[3,2]:
22.697 um

Vol. Weighted Mean D[4,3]:
100.222 um

d(0.1): 41.264 um **d(0.5): 95.914 um** **d(0.9): 169.021 um**



S2-Test3-Post Test Grab Sample-IO-2.1 - Average, Thursday, September 14, 2017 5:00:31 PM

Size (μm)	Volume In %
0.020	0.00
0.022	0.00
0.025	0.00
0.028	0.00
0.032	0.00
0.036	0.00
0.040	0.00
0.045	0.00
0.050	0.00
0.056	0.00
0.063	0.00
0.071	0.00
0.080	0.00
0.089	0.00
0.100	0.00
0.112	0.00
0.126	0.00
0.142	

Size (μm)	Volume In %
0.142	0.00
0.159	0.00
0.178	0.00
0.200	0.00
0.224	0.00
0.252	0.00
0.283	0.00
0.317	0.00
0.356	0.07
0.399	0.09
0.448	0.11
0.502	0.13
0.564	0.14
0.632	0.15
0.710	0.16
0.796	0.16
0.893	0.16
1.002	

Size (μm)	Volume In %
1.002	0.16
1.125	0.16
1.262	0.15
1.416	0.15
1.589	0.15
1.783	0.15
2.000	0.15
2.244	0.16
2.518	0.16
2.825	0.16
3.170	0.16
3.557	0.16
3.991	0.16
4.477	0.17
5.024	0.17
5.637	0.19
6.325	0.21
7.096	

Size (μm)	Volume In %
7.096	0.24
7.962	0.27
8.934	0.31
10.024	0.35
11.247	0.39
12.619	0.41
14.159	0.41
15.887	0.39
17.825	0.35
20.000	0.29
22.440	0.24
25.179	0.23
28.251	0.31
31.698	0.53
35.566	0.95
39.905	1.60
44.774	2.49
50.238	

Size (μm)	Volume In %
50.238	3.62
56.368	4.89
63.246	6.24
70.963	7.48
79.621	8.49
89.337	9.12
100.237	9.29
112.468	8.94
126.191	8.13
141.589	6.94
158.866	5.53
178.250	4.03
200.000	2.71
224.404	0.83
251.785	0.07
282.508	0.00
316.979	0.00
355.656	

Size (μm)	Volume In %
355.656	0.00
399.052	0.00
447.744	0.00
502.377	0.00
563.677	0.00
632.456	0.00
709.627	0.00
796.214	0.00
893.367	0.00
1002.374	0.00
1124.683	0.00
1261.915	0.00
1415.892	0.00
1588.656	0.00
1782.502	0.00
2000.000	

Operator notes: *LPIST-NS-B05 Post test grab sample*

MALVERN INSTRUMENTS

MASTERSIZER 2000

Result Analysis Report

Sample Name:
S2-Test3-Post Test Grab Sample-IO-2.2 -

SOP Name:

Measured:
Thursday, September 14, 2017 5:02:31 PM

Sample Source & type:

Measured by:
D3M966

Analysed:
Thursday, September 14, 2017 5:02:32 PM

Sample bulk lot ref:

Result Source:
Averaged

Particle Name:
Iron III Oxide 2.98

Accessory Name:
Hydro 2000μP (A)

Analysis model:
General purpose

Sensitivity:
Normal

Particle RI:
2.980

Absorption:
1

Size range:
0.020 to 2000.000 um

Obscuration:
19.07 %

Dispersant Name:
Water

Dispersant RI:
1.330

Weighted Residual:
0.862 %

Result Emulation:
Off

Concentration:
0.0138 %Vol

Span :
2.022

Uniformity:
0.677

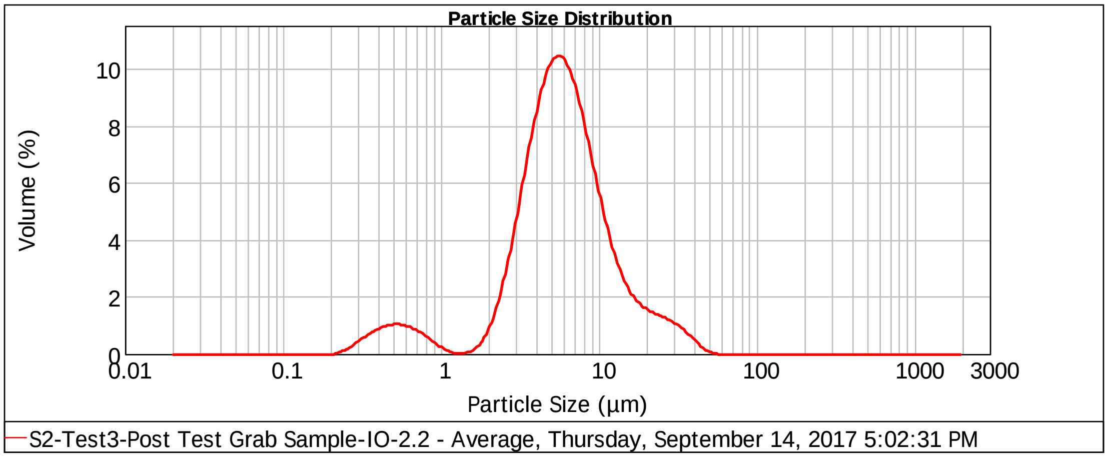
Result units:
Volume

Specific Surface Area:
1.84 m²/g

Surface Weighted Mean D[3,2]:
3.267 um

Vol. Weighted Mean D[4,3]:
7.534 um

d(0.1): 2.550 um **d(0.5): 5.778 um** **d(0.9): 14.232 um**



Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %
0.020	0.00	0.142	0.00	1.002	0.11	7.096	6.69	50.238	0.02	355.656	0.00
0.022	0.00	0.159	0.00	1.125	0.03	7.962	5.77	56.368	0.00	399.052	0.00
0.025	0.00	0.178	0.00	1.262	0.02	8.934	4.79	63.246	0.00	447.744	0.00
0.028	0.00	0.200	0.00	1.416	0.04	10.024	3.82	70.963	0.00	502.377	0.00
0.032	0.00	0.224	0.08	1.589	0.15	11.247	2.99	79.621	0.00	563.677	0.00
0.036	0.00	0.252	0.17	1.783	0.42	12.619	2.32	89.337	0.00	632.456	0.00
0.040	0.00	0.283	0.34	2.000	0.88	14.159	1.82	100.237	0.00	709.627	0.00
0.045	0.00	0.317	0.47	2.244	1.55	15.887	1.48	112.468	0.00	796.214	0.00
0.050	0.00	0.356	0.60	2.518	2.44	17.825	1.26	126.191	0.00	893.367	0.00
0.056	0.00	0.399	0.70	2.825	3.51	20.000	1.13	141.589	0.00	1002.374	0.00
0.063	0.00	0.448	0.77	3.170	4.65	22.440	1.03	158.866	0.00	1124.683	0.00
0.071	0.00	0.502	0.78	3.557	5.80	25.179	0.94	178.250	0.00	1261.915	0.00
0.080	0.00	0.564	0.75	3.991	6.78	28.251	0.83	200.000	0.00	1415.892	0.00
0.089	0.00	0.632	0.68	4.477	7.50	31.698	0.68	224.404	0.00	1588.656	0.00
0.100	0.00	0.710	0.56	5.024	7.86	35.566	0.50	251.785	0.00	1782.502	0.00
0.112	0.00	0.796	0.41	5.637	7.82	39.905	0.30	282.508	0.00	2000.000	
0.126	0.00	0.893	0.26	6.325	7.41	44.774	0.09	316.979	0.00		
0.142		1.002		7.096		50.238		355.656			

Operator notes: *LPIST-NS-B05 Post test grab sample*

MALVERN INSTRUMENTS

MASTERSIZER 2000

Result Analysis Report

Sample Name:
S2-Test3-Post Test Grab Sample-IO-2.3 -

SOP Name:

Measured:
Thursday, September 14, 2017 5:04:09 PM

Sample Source & type:

Measured by:
D3M966

Analysed:
Thursday, September 14, 2017 5:04:10 PM

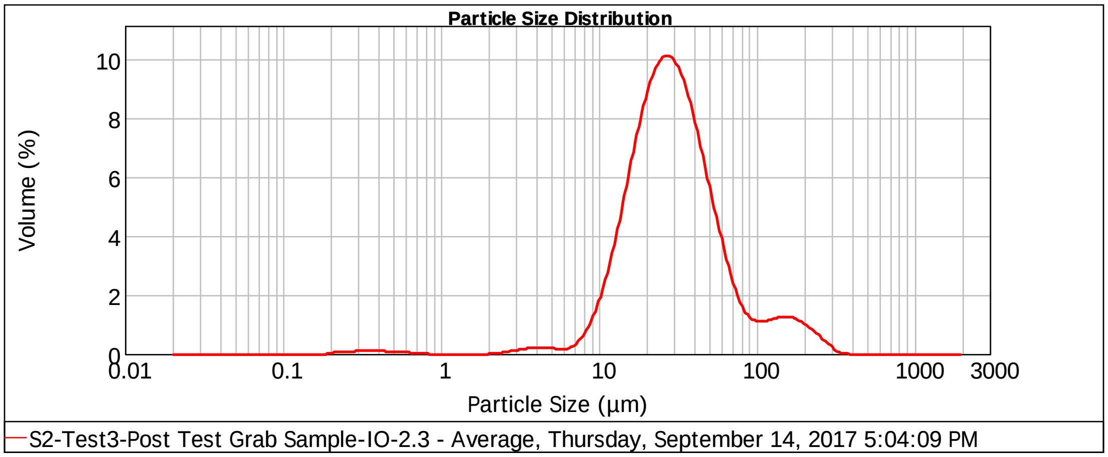
Sample bulk lot ref:

Result Source:
Averaged

Particle Name:	**Accessory Name:**	**Analysis model:**	**Sensitivity:**
Iron III Oxide 2.98	Hydro 2000μP (A)	General purpose	Normal
Particle RI:	**Absorption:**	**Size range:**	**Obscuration:**
2.980	1	0.020 to 2000.000 um	32.47 %
Dispersant Name:	**Dispersant RI:**	**Weighted Residual:**	**Result Emulation:**
Water	1.330	0.582 %	Off

Concentration:	**Span :**	**Uniformity:**	**Result units:**
0.1797 %Vol	2.104	0.777	Volume
Specific Surface Area:	**Surface Weighted Mean D[3,2]:**	**Vol. Weighted Mean D[4,3]:**	
0.356 m²/g	16.874 um	41.085 um	

d(0.1): **13.621** **um** **d(0.5):** **28.717** **um** **d(0.9):** **74.045** **um**



Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %
0.020	0.00	0.142	0.00	1.002	0.00	7.096	0.34	50.238	3.82	355.656	0.00
0.022	0.00	0.159	0.00	1.125	0.00	7.962	0.62	56.368	2.97	399.052	0.00
0.025	0.00	0.178	0.00	1.262	0.00	8.934	1.05	63.246	2.17	447.744	0.00
0.028	0.00	0.200	0.04	1.416	0.00	10.024	1.66	70.963	1.52	502.377	0.00
0.032	0.00	0.224	0.05	1.589	0.00	11.247	2.39	79.621	1.08	563.677	0.00
0.036	0.00	0.252	0.06	1.783	0.00	12.619	3.29	89.337	0.87	632.456	0.00
0.040	0.00	0.283	0.07	2.000	0.00	14.159	4.30	100.237	0.83	709.627	0.00
0.045	0.00	0.317	0.08	2.244	0.03	15.887	5.28	112.468	0.86	796.214	0.00
0.050	0.00	0.356	0.08	2.518	0.06	17.825	6.19	126.191	0.92	893.367	0.00
0.056	0.00	0.399	0.07	2.825	0.09	20.000	6.92	141.589	0.95	1002.374	0.00
0.063	0.00	0.448	0.06	3.170	0.13	22.440	7.41	158.866	0.93	1124.683	0.00
0.071	0.00	0.502	0.05	3.557	0.15	25.179	7.61	178.250	0.84	1261.915	0.00
0.080	0.00	0.564	0.04	3.991	0.17	28.251	7.50	200.000	0.70	1415.892	0.00
0.089	0.00	0.632	0.03	4.477	0.17	31.698	7.11	224.404	0.54	1588.656	0.00
0.100	0.00	0.710	0.01	5.024	0.13	35.566	6.48	251.785	0.36	1782.502	0.00
0.112	0.00	0.796	0.00	5.637	0.11	39.905	5.66	282.508	0.18	2000.000	
0.126	0.00	0.893	0.00	6.325	0.17	44.774	4.74	316.979	0.04		
0.142		1.002		7.096		50.238		355.656			

Operator notes: *LPIST-NS-B05 Post test grab sample*

MASTERSIZER 2000

Result Analysis Report

Sample Name:
S2-Test3-Post Test Grab Sample-IP-1.1 -

SOP Name:

Measured:
Friday, September 15, 2017 8:31:07 AM

Sample Source & type:

Measured by:
D3M966

Analysed:
Friday, September 15, 2017 8:31:09 AM

Sample bulk lot ref:

Result Source:
Averaged

Particle Name:
Iron III Oxide 2.98

Accessory Name:
Hydro 2000μP (A)

Analysis model:
General purpose

Sensitivity:
Normal

Particle RI:
2.980

Absorption:
1

Size range:
0.020 to 2000.000 um

Obscuration:
14.51 %

Dispersant Name:
Water

Dispersant RI:
1.330

Weighted Residual:
0.722 %

Result Emulation:
Off

Concentration:
0.0073 %Vol

Span :
19.107

Uniformity:
5.95

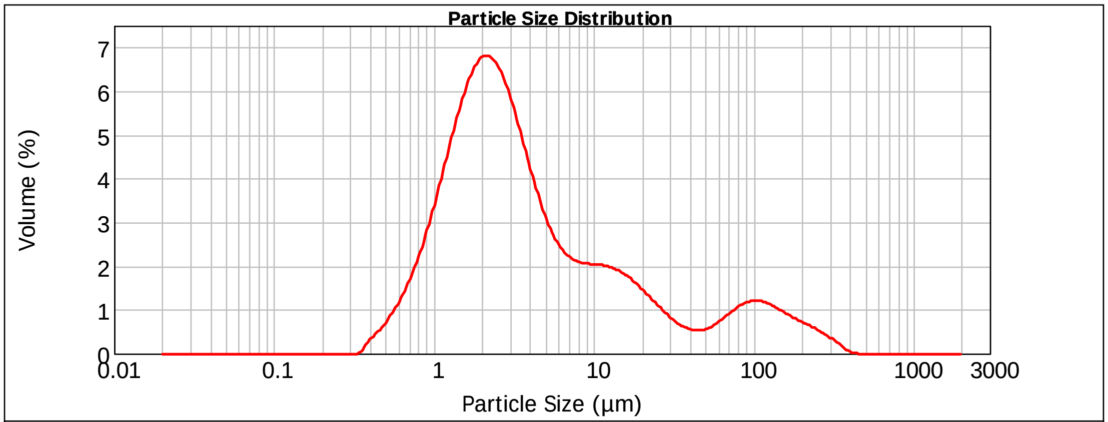
Result units:
Volume

Specific Surface Area:
2.59 m²/g

Surface Weighted Mean D[3,2]:
2.314 um

Vol. Weighted Mean D[4,3]:
19.268 um

d(0.1): 1.043 um **d(0.5): 2.955 um** **d(0.9): 57.513 um**



S2-Test3-Post Test Grab Sample-IP-1.1 - Average, Friday, September 15, 2017 8:31:07 AM

Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %
0.020	0.00	0.142	0.00	1.002	2.79	7.096	1.61	50.238	0.45	355.656	0.06
0.022	0.00	0.159	0.00	1.125	3.33	7.962	1.56	56.368	0.53	399.052	0.01
0.025	0.00	0.178	0.00	1.262	3.87	8.934	1.54	63.246	0.63	447.744	0.00
0.028	0.00	0.200	0.00	1.416	4.36	10.024	1.53	70.963	0.74	502.377	0.00
0.032	0.00	0.224	0.00	1.589	4.76	11.247	1.51	79.621	0.84	563.677	0.00
0.036	0.00	0.252	0.00	1.783	5.03	12.619	1.47	89.337	0.90	632.456	0.00
0.040	0.00	0.283	0.00	2.000	5.13	14.159	1.40	100.237	0.91	709.627	0.00
0.045	0.00	0.317	0.00	2.244	5.05	15.887	1.30	112.468	0.88	796.214	0.00
0.050	0.00	0.356	0.15	2.518	4.81	17.825	1.18	126.191	0.82	893.367	0.00
0.056	0.00	0.399	0.31	2.825	4.43	20.000	1.04	141.589	0.73	1002.374	0.00
0.063	0.00	0.448	0.45	3.170	3.97	22.440	0.90	158.866	0.65	1124.683	0.00
0.071	0.00	0.502	0.64	3.557	3.45	25.179	0.76	178.250	0.58	1261.915	0.00
0.080	0.00	0.564	0.86	3.991	2.96	28.251	0.63	200.000	0.51	1415.892	0.00
0.089	0.00	0.632	1.12	4.477	2.51	31.698	0.52	224.404	0.44	1588.656	0.00
0.100	0.00	0.710	1.45	5.024	2.15	35.566	0.44	251.785	0.36	1782.502	0.00
0.112	0.00	0.796	1.83	5.637	1.89	39.905	0.40	282.508	0.28	2000.000	
0.126	0.00	0.893	2.28	6.325	1.71	44.774	0.40	316.979	0.19		
0.142		1.002		7.096		50.238		355.656			

Operator notes: *LPIST-NS-B08 Post test grab sample, LPIST-NS-B06 carrier fluid*

Result Analysis Report

Sample Name:
S2-Test3-Post Test Grab Sample-IP-1.2 -

SOP Name:

Measured:
Friday, September 15, 2017 8:32:59 AM

Sample Source & type:

Measured by:
D3M966

Analysed:
Friday, September 15, 2017 8:33:00 AM

Sample bulk lot ref:

Result Source:
Averaged

Particle Name:
Iron III Oxide 2.98

Accessory Name:
Hydro 2000μP (A)

Analysis model:
General purpose

Sensitivity:
Normal

Particle RI:
2.980

Absorption:
1

Size range:
0.020 to 2000.000 um

Obscuration:
11.34 %

Dispersant Name:
Water

Dispersant RI:
1.330

Weighted Residual:
1.162 %

Result Emulation:
Off

Concentration:
0.0049 %Vol

Span :
4.672

Uniformity:
1.72

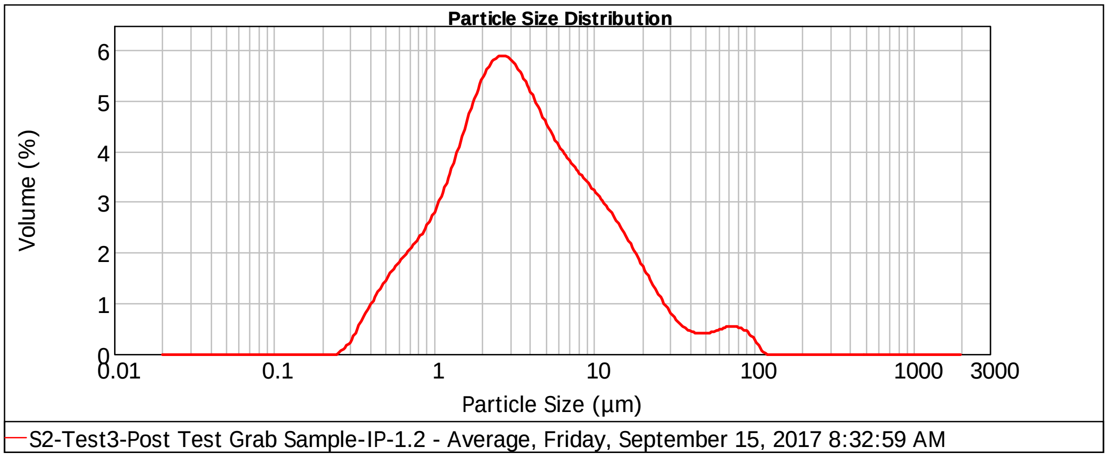
Result units:
Volume

Specific Surface Area:
2.88 m²/g

Surface Weighted Mean D[3,2]:
2.084 um

Vol. Weighted Mean D[4,3]:
7.440 um

d(0.1): 0.865 um **d(0.5): 3.328 um** **d(0.9): 16.411 um**



Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %
0.020	0.00	0.142	0.00	1.002	2.22	7.096	2.78	50.238	0.31	355.656	0.00
0.022	0.00	0.159	0.00	1.125	2.51	7.962	2.64	56.368	0.35	399.052	0.00
0.025	0.00	0.178	0.00	1.262	2.85	8.934	2.50	63.246	0.39	447.744	0.00
0.028	0.00	0.200	0.00	1.416	3.22	10.024	2.36	70.963	0.41	502.377	0.00
0.032	0.00	0.224	0.00	1.589	3.59	11.247	2.20	79.621	0.38	563.677	0.00
0.036	0.00	0.252	0.05	1.783	3.93	12.619	2.04	89.337	0.30	632.456	0.00
0.040	0.00	0.283	0.16	2.000	4.20	14.159	1.85	100.237	0.14	709.627	0.00
0.045	0.00	0.317	0.41	2.244	4.37	15.887	1.65	112.468	0.00	796.214	0.00
0.050	0.00	0.356	0.61	2.518	4.43	17.825	1.43	126.191	0.00	893.367	0.00
0.056	0.00	0.399	0.82	2.825	4.38	20.000	1.22	141.589	0.00	1002.374	0.00
0.063	0.00	0.448	1.02	3.170	4.24	22.440	1.00	158.866	0.00	1124.683	0.00
0.071	0.00	0.502	1.19	3.557	4.03	25.179	0.80	178.250	0.00	1261.915	0.00
0.080	0.00	0.564	1.35	3.991	3.79	28.251	0.62	200.000	0.00	1415.892	0.00
0.089	0.00	0.632	1.49	4.477	3.54	31.698	0.48	224.404	0.00	1588.656	0.00
0.100	0.00	0.710	1.63	5.024	3.31	35.566	0.38	251.785	0.00	1782.502	0.00
0.112	0.00	0.796	1.79	5.637	3.11	39.905	0.32	282.508	0.00	2000.000	
0.126	0.00	0.893	1.98	6.325	2.94	44.774	0.30	316.979	0.00		
0.142		1.002		7.096		50.238		355.656			

Operator notes: *LPIST-NS-B08 Post test grab sample, LPIST-NS-B06 carrier fluid*

MALVERN INSTRUMENTS

MASTERSIZER 2000

Result Analysis Report

Sample Name:
S2-Test3-Post Test Grab Sample-IP-1.3 -

SOP Name:

Measured:
Friday, September 15, 2017 8:34:26 AM

Sample Source & type:

Measured by:
D3M966

Analysed:
Friday, September 15, 2017 8:34:27 AM

Sample bulk lot ref:

Result Source:
Averaged

Particle Name:
Iron III Oxide 2.98

Accessory Name:
Hydro 2000μP (A)

Analysis model:
General purpose

Sensitivity:
Normal

Particle RI:
2.980

Absorption:
1

Size range:
0.020 to 2000.000 um

Obscuration:
11.80 %

Dispersant Name:
Water

Dispersant RI:
1.330

Weighted Residual:
1.095 %

Result Emulation:
Off

Concentration:
0.0049 %Vol

Span :
4.306

Uniformity:
1.53

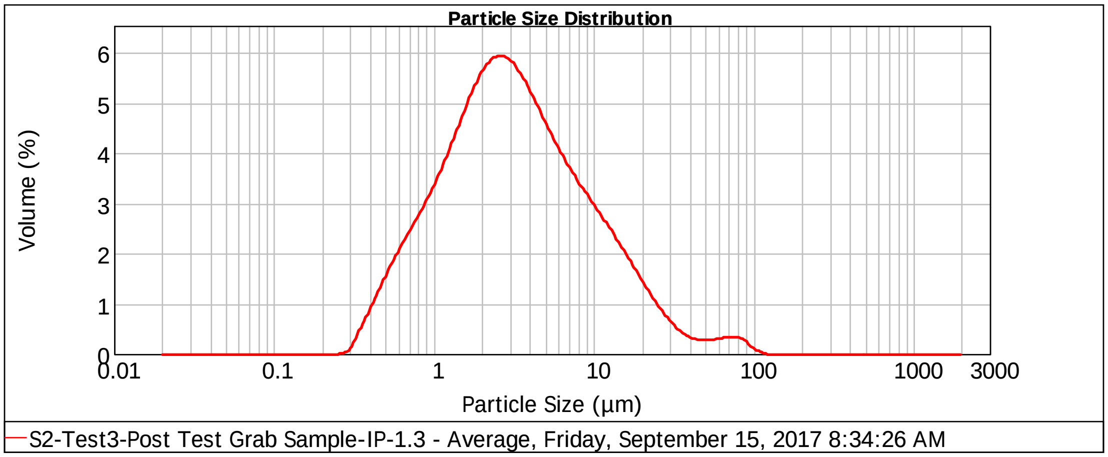
Result units:
Volume

Specific Surface Area:
3.05 m²/g

Surface Weighted Mean D[3,2]:
1.965 um

Vol. Weighted Mean D[4,3]:
6.191 um

d(0.1): 0.825 um **d(0.5): 3.011 um** **d(0.9): 13.789 um**



Size (μm)	Volume In %
0.020	0.00
0.022	0.00
0.025	0.00
0.028	0.00
0.032	0.00
0.036	0.00
0.040	0.00
0.045	0.00
0.050	0.00
0.056	0.00
0.063	0.00
0.071	0.00
0.080	0.00
0.089	0.00
0.100	0.00
0.112	0.00
0.126	0.00
0.142	

Size (μm)	Volume In %
0.142	0.00
0.159	0.00
0.178	0.00
0.200	0.00
0.224	0.00
0.252	0.01
0.283	0.07
0.317	0.32
0.356	0.55
0.399	0.80
0.448	1.06
0.502	1.30
0.564	1.54
0.632	1.75
0.710	1.96
0.796	2.17
0.893	2.40
1.002	

Size (μm)	Volume In %
1.002	2.66
1.125	2.94
1.262	3.24
1.416	3.55
1.589	3.86
1.783	4.12
2.000	4.33
2.244	4.44
2.518	4.47
2.825	4.41
3.170	4.27
3.557	4.06
3.991	3.83
4.477	3.57
5.024	3.33
5.637	3.09
6.325	2.88
7.096	

Size (μm)	Volume In %
7.096	2.68
7.962	2.50
8.934	2.32
10.024	2.15
11.247	1.97
12.619	1.79
14.159	1.60
15.887	1.40
17.825	1.20
20.000	1.01
22.440	0.82
25.179	0.65
28.251	0.50
31.698	0.38
35.566	0.28
39.905	0.23
44.774	0.20
50.238	

Size (μm)	Volume In %
50.238	0.20
56.368	0.22
63.246	0.25
70.963	0.25
79.621	0.24
89.337	0.13
100.237	0.05
112.468	0.00
126.191	0.00
141.589	0.00
158.866	0.00
178.250	0.00
200.000	0.00
224.404	0.00
251.785	0.00
282.508	0.00
316.979	0.00
355.656	

Size (μm)	Volume In %
355.656	0.00
399.052	0.00
447.744	0.00
502.377	0.00
563.677	0.00
632.456	0.00
709.627	0.00
796.214	0.00
893.367	0.00
1002.374	0.00
1124.683	0.00
1261.915	0.00
1415.892	0.00
1588.656	0.00
1782.502	0.00
2000.000	

Operator notes: *LPIST-NS-B08 Post test grab sample, LPIST-NS-B06 carrier fluid*

MASTERSIZER 2000

Result Analysis Report

Sample Name:
S2-Test 5-001-CN-1.1-Average

SOP Name:

Measured:
Friday, September 15, 2017 9:30:18 AM

Sample Source & type:

Measured by:
D3M966

Analysed:
Thursday, September 21, 2017 2:57:57 PM

Sample bulk lot ref:

Result Source:
Averaged

Particle Name:
Iron III Oxide 2.98

Accessory Name:
Hydro 2000μP (A)

Analysis model:
General purpose

Sensitivity:
Normal

Particle RI:
2.980

Absorption:
1

Size range:
0.020 to 2000.000 um

Obscuration:
10.61 %

Dispersant Name:
Water

Dispersant RI:
1.330

Weighted Residual:
0.729 %

Result Emulation:
Off

Concentration:
0.0113 %Vol

Span :
25.430

Uniformity:
6.75

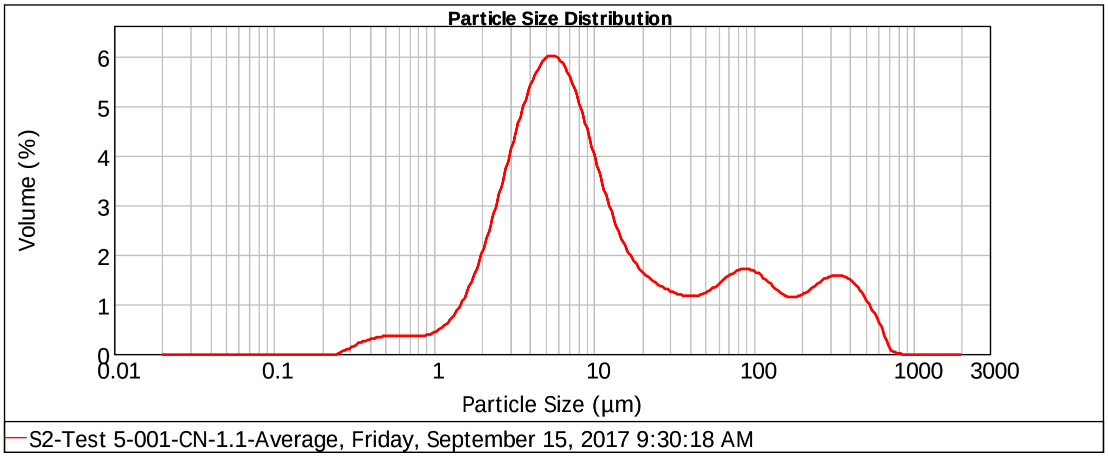
Result units:
Volume

Specific Surface Area:
1.23 m²/g

Surface Weighted Mean D[3,2]:
4.863 um

Vol. Weighted Mean D[4,3]:
57.610 um

d(0.1): 2.382 um **d(0.5): 7.921 um** **d(0.9): 203.802 um**



Size (μm)	Volume In %
0.020	0.00
0.022	0.00
0.025	0.00
0.028	0.00
0.032	0.00
0.036	0.00
0.040	0.00
0.045	0.00
0.050	0.00
0.056	0.00
0.063	0.00
0.071	0.00
0.080	0.00
0.089	0.00
0.100	0.00
0.112	0.00
0.126	0.00
0.142	

Size (μm)	Volume In %
0.142	0.00
0.159	0.00
0.178	0.00
0.200	0.00
0.224	0.00
0.252	0.03
0.283	0.09
0.317	0.16
0.356	0.20
0.399	0.24
0.448	0.26
0.502	0.27
0.564	0.28
0.632	0.27
0.710	0.27
0.796	0.27
0.893	0.30
1.002	

Size (μm)	Volume In %
1.002	0.35
1.125	0.45
1.262	0.59
1.416	0.78
1.589	1.04
1.783	1.36
2.000	1.73
2.244	2.14
2.518	2.59
2.825	3.05
3.170	3.49
3.557	3.89
3.991	4.21
4.477	4.44
5.024	4.54
5.637	4.50
6.325	4.33
7.096	

Size (μm)	Volume In %
7.096	4.05
7.962	3.68
8.934	3.26
10.024	2.82
11.247	2.41
12.619	2.05
14.159	1.74
15.887	1.51
17.825	1.33
20.000	1.20
22.440	1.10
25.179	1.02
28.251	0.96
31.698	0.91
35.566	0.88
39.905	0.88
44.774	0.91
50.238	

Size (μm)	Volume In %
50.238	0.97
56.368	1.05
63.246	1.15
70.963	1.24
79.621	1.29
89.337	1.29
100.237	1.23
112.468	1.13
126.191	1.01
141.589	0.91
158.866	0.86
178.250	0.87
200.000	0.94
224.404	1.03
251.785	1.11
282.508	1.18
316.979	1.20
355.656	

Size (μm)	Volume In %
355.656	1.17
399.052	1.08
447.744	0.93
502.377	0.73
563.677	0.53
632.456	0.22
709.627	0.03
796.214	0.00
893.367	0.00
1002.374	0.00
1124.683	0.00
1261.915	0.00
1415.892	0.00
1588.656	0.00
1782.502	0.00
2000.000	

Operator notes: *Average of 3 measurements from CUF-2017.mea*

File name: CUF-2017-Final.mea
Record Number: 172
9/28/2017 5:48:23 PM

MASTERSIZER 2000

Result Analysis Report

Sample Name:
S2-Test5-001-CN-1.2 - Average

SOP Name:

Measured:
Friday, September 15, 2017 9:32:44 AM

Sample Source & type:

Measured by:
D3M966

Analysed:
Friday, September 15, 2017 9:32:46 AM

Sample bulk lot ref:

Result Source:
Averaged

Particle Name:
Aluminium silicate

Accessory Name:
Hydro 2000μP (A)

Analysis model:
General purpose

Sensitivity:
Normal

Particle RI:
1.650

Absorption:
0.1

Size range:
0.020 to 2000.000 um

Obscuration:
7.61 %

Dispersant Name:
Water

Dispersant RI:
1.330

Weighted Residual:
1.365 %

Result Emulation:
Off

Concentration:
0.0073 %Vol

Span :
7.468

Uniformity:
3.58

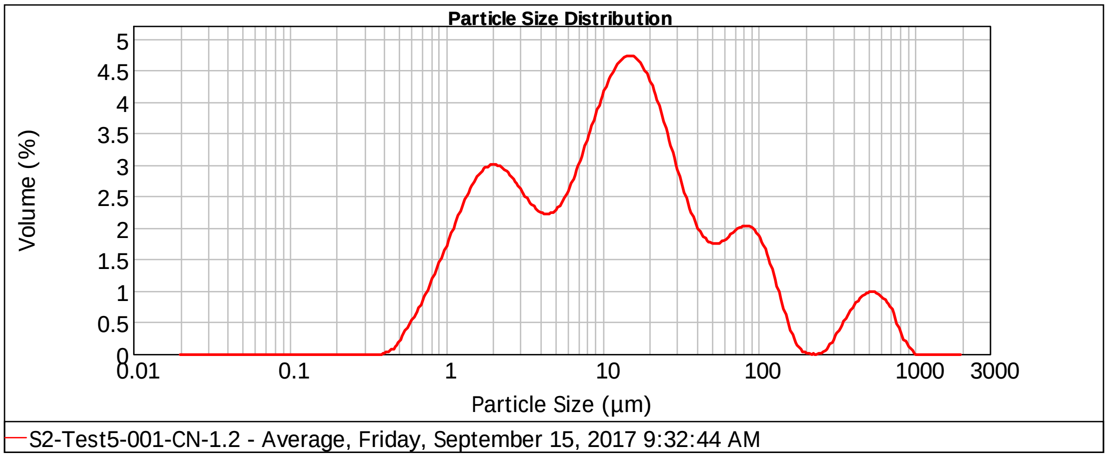
Result units:
Volume

Specific Surface Area:
1.36 m²/g

Surface Weighted Mean D[3,2]:
4.399 um

Vol. Weighted Mean D[4,3]:
47.713 um

d(0.1): **1.500** **um** **d(0.5):** **12.048** **um** **d(0.9):** **91.471** **um**



Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %
0.020	0.00	0.142	0.00	1.002	1.40	7.096	2.40	50.238	1.31	355.656	0.48
0.022	0.00	0.159	0.00	1.125	1.64	7.962	2.68	56.368	1.35	399.052	0.62
0.025	0.00	0.178	0.00	1.262	1.86	8.934	2.96	63.246	1.43	447.744	0.71
0.028	0.00	0.200	0.00	1.416	2.04	10.024	3.20	70.963	1.50	502.377	0.75
0.032	0.00	0.224	0.00	1.589	2.18	11.247	3.40	79.621	1.53	563.677	0.70
0.036	0.00	0.252	0.00	1.783	2.25	12.619	3.52	89.337	1.47	632.456	0.61
0.040	0.00	0.283	0.00	2.000	2.25	14.159	3.56	100.237	1.32	709.627	0.42
0.045	0.00	0.317	0.00	2.244	2.20	15.887	3.51	112.468	1.06	796.214	0.18
0.050	0.00	0.356	0.00	2.518	2.10	17.825	3.37	126.191	0.76	893.367	0.04
0.056	0.00	0.399	0.02	2.825	1.97	20.000	3.16	141.589	0.44	1002.374	0.00
0.063	0.00	0.448	0.08	3.170	1.84	22.440	2.88	158.866	0.19	1124.683	0.00
0.071	0.00	0.502	0.24	3.557	1.73	25.179	2.56	178.250	0.04	1261.915	0.00
0.080	0.00	0.564	0.38	3.991	1.67	28.251	2.23	200.000	0.00	1415.892	0.00
0.089	0.00	0.632	0.54	4.477	1.68	31.698	1.92	224.404	0.00	1588.656	0.00
0.100	0.00	0.710	0.73	5.024	1.77	35.566	1.65	251.785	0.04	1782.502	0.00
0.112	0.00	0.796	0.94	5.637	1.93	39.905	1.46	282.508	0.16	2000.000	
0.126	0.00	0.893	1.17	6.325	2.14	44.774	1.34	316.979	0.32		
0.142		1.002		7.096		50.238		355.656			

Operator notes: *LPIST-NS-B06 carrier fluid, CN pre test sample*

MASTERSIZER 2000

Result Analysis Report

Sample Name:
S2-Test5-001-CN-1.3 - Average

SOP Name:

Measured:
Friday, September 15, 2017 9:34:16 AM

Sample Source & type:

Measured by:
D3M966

Analysed:
Friday, September 15, 2017 9:34:17 AM

Sample bulk lot ref:

Result Source:
Averaged

Particle Name:
Aluminium silicate

Accessory Name:
Hydro 2000μP (A)

Analysis model:
General purpose

Sensitivity:
Normal

Particle RI:
1.650

Absorption:
0.1

Size range:
0.020 to 2000.000 um

Obscuration:
7.31 %

Dispersant Name:
Water

Dispersant RI:
1.330

Weighted Residual:
1.506 %

Result Emulation:
Off

Concentration:
0.0060 %Vol

Span :
4.373

Uniformity:
1.43

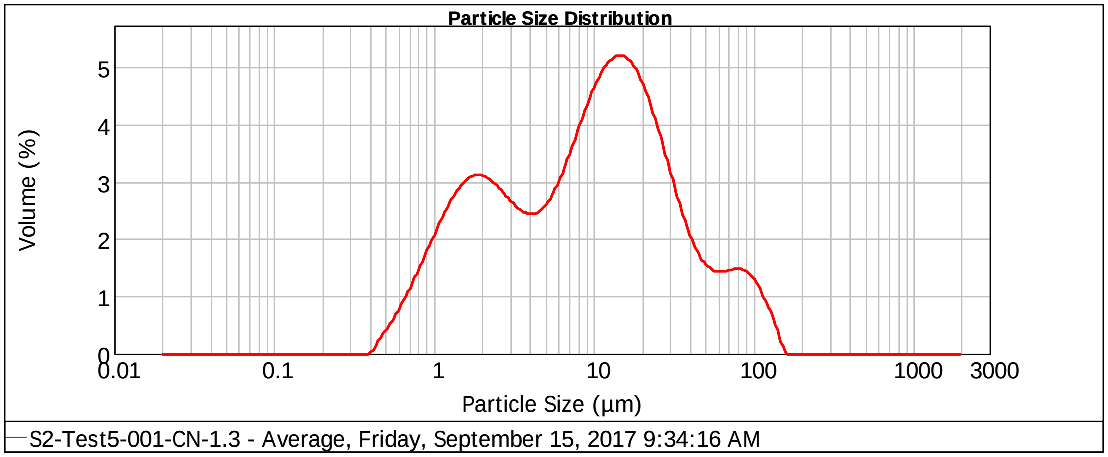
Result units:
Volume

Specific Surface Area:
1.59 m²/g

Surface Weighted Mean D[3,2]:
3.769 um

Vol. Weighted Mean D[4,3]:
18.023 um

d(0.1): 1.322 um **d(0.5): 9.916 um** **d(0.9): 44.690 um**



Size (μm)	Volume In %
0.020	0.00
0.022	0.00
0.025	0.00
0.028	0.00
0.032	0.00
0.036	0.00
0.040	0.00
0.045	0.00
0.050	0.00
0.056	0.00
0.063	0.00
0.071	0.00
0.080	0.00
0.089	0.00
0.100	0.00
0.112	0.00
0.126	0.00
0.142	

Size (μm)	Volume In %
0.142	0.00
0.159	0.00
0.178	0.00
0.200	0.00
0.224	0.00
0.252	0.00
0.283	0.00
0.317	0.00
0.356	0.00
0.399	0.06
0.448	0.25
0.502	0.38
0.564	0.56
0.632	0.76
0.710	0.97
0.796	1.20
0.893	1.44
1.002	

Size (μm)	Volume In %
1.002	1.67
1.125	1.89
1.262	2.08
1.416	2.22
1.589	2.32
1.783	2.35
2.000	2.32
2.244	2.24
2.518	2.14
2.825	2.01
3.170	1.91
3.557	1.84
3.991	1.83
4.477	1.90
5.024	2.03
5.637	2.24
6.325	2.50
7.096	

Size (μm)	Volume In %
7.096	2.78
7.962	3.08
8.934	3.36
10.024	3.60
11.247	3.78
12.619	3.89
14.159	3.91
15.887	3.84
17.825	3.69
20.000	3.45
22.440	3.14
25.179	2.79
28.251	2.41
31.698	2.04
35.566	1.71
39.905	1.44
44.774	1.24
50.238	

Size (μm)	Volume In %
50.238	1.12
56.368	1.08
63.246	1.09
70.963	1.11
79.621	1.11
89.337	1.05
100.237	0.91
112.468	0.69
126.191	0.47
141.589	0.12
158.866	0.00
178.250	0.00
200.000	0.00
224.404	0.00
251.785	0.00
282.508	0.00
316.979	0.00
355.656	

Size (μm)	Volume In %
355.656	0.00
399.052	0.00
447.744	0.00
502.377	0.00
563.677	0.00
632.456	0.00
709.627	0.00
796.214	0.00
893.367	0.00
1002.374	0.00
1124.683	0.00
1261.915	0.00
1415.892	0.00
1588.656	0.00
1782.502	0.00
2000.000	

Operator notes: *LPIST-NS-B06 carrier fluid, CN pre test sample*

MALVERN
INSTRUMENTS

MASTERSIZER 2000

Result Analysis Report

Sample Name:
S2-Test5-Post Test Grab Sample-CN-1.1 -

SOP Name:

Measured:
Friday, September 15, 2017 10:05:24 AM

Sample Source & type:

Measured by:
D3M966

Analysed:
Friday, September 15, 2017 10:05:25 AM

Sample bulk lot ref:

Result Source:
Averaged

Particle Name:
Aluminium silicate

Accessory Name:
Hydro 2000μP (A)

Analysis model:
General purpose

Sensitivity:
Normal

Particle RI:
1.650

Absorption:
0.1

Size range:
0.020 to 2000.000 um

Obscuration:
16.82 %

Dispersant Name:
Water

Dispersant RI:
1.330

Weighted Residual:
1.095 %

Result Emulation:
Off

Concentration:
0.0378 %Vol

Span :
6.283

Uniformity:
2

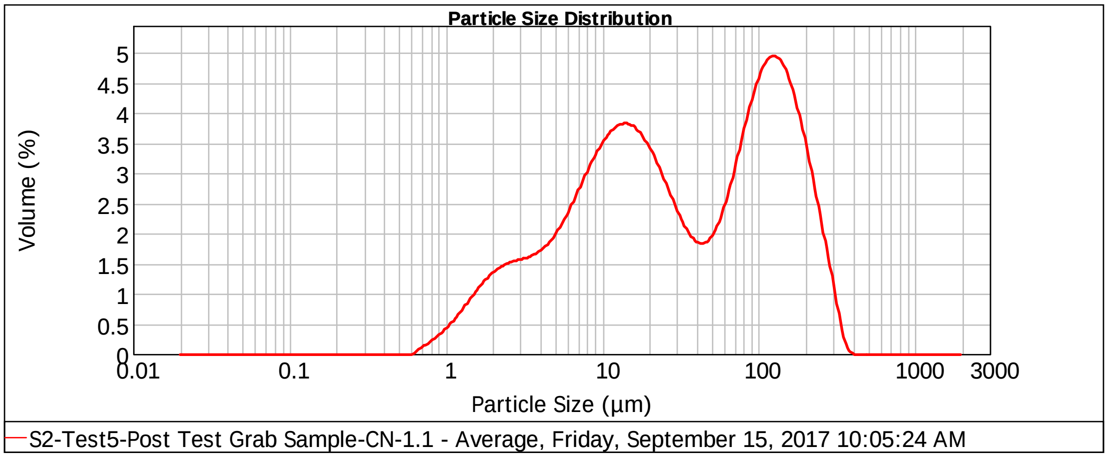
Result units:
Volume

Specific Surface Area:
0.624 m²/g

Surface Weighted Mean D[3,2]:
9.616 um

Vol. Weighted Mean D[4,3]:
65.878 um

d(0.1): **3.426** **um** **d(0.5):** **27.661** **um** **d(0.9):** **177.220** **um**



Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %
0.020	0.00	0.142	0.00	1.002	0.37	7.096	2.17	50.238	1.56	355.656	0.04
0.022	0.00	0.159	0.00	1.125	0.49	7.962	2.38	56.368	1.81	399.052	0.00
0.025	0.00	0.178	0.00	1.262	0.61	8.934	2.56	63.246	2.15	447.744	0.00
0.028	0.00	0.200	0.00	1.416	0.74	10.024	2.72	70.963	2.55	502.377	0.00
0.032	0.00	0.224	0.00	1.589	0.86	11.247	2.82	79.621	2.96	563.677	0.00
0.036	0.00	0.252	0.00	1.783	0.97	12.619	2.88	89.337	3.32	632.456	0.00
0.040	0.00	0.283	0.00	2.000	1.05	14.159	2.87	100.237	3.59	709.627	0.00
0.045	0.00	0.317	0.00	2.244	1.11	15.887	2.80	112.468	3.72	796.214	0.00
0.050	0.00	0.356	0.00	2.518	1.15	17.825	2.67	126.191	3.70	893.367	0.00
0.056	0.00	0.399	0.00	2.825	1.18	20.000	2.49	141.589	3.54	1002.374	0.00
0.063	0.00	0.448	0.00	3.170	1.21	22.440	2.27	158.866	3.25	1124.683	0.00
0.071	0.00	0.502	0.00	3.557	1.25	25.179	2.04	178.250	2.86	1261.915	0.00
0.080	0.00	0.564	0.00	3.991	1.32	28.251	1.80	200.000	2.40	1415.892	0.00
0.089	0.00	0.632	0.05	4.477	1.43	31.698	1.60	224.404	1.90	1588.656	0.00
0.100	0.00	0.710	0.12	5.024	1.57	35.566	1.45	251.785	1.40	1782.502	0.00
0.112	0.00	0.796	0.20	5.637	1.75	39.905	1.38	282.508	0.90	2000.000	
0.126	0.00	0.893	0.27	6.325	1.96	44.774	1.41	316.979	0.36		
0.142		1.002		7.096		50.238		355.656			

Operator notes: *LPIST-NS-B06 carrier fluid, Sample taken from LPIST-NS-B09*

MASTERSIZER 2000

Result Analysis Report

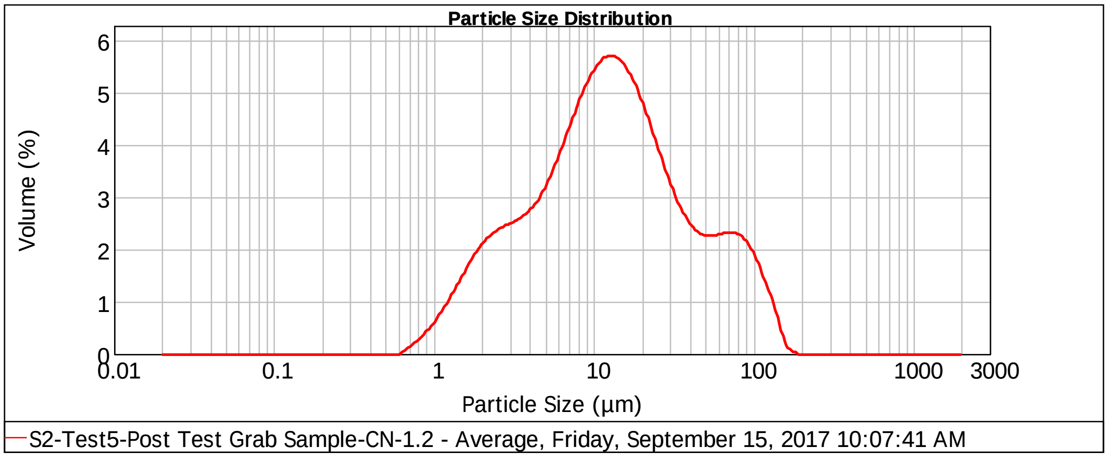
Sample Name: S2-Test5-Post Test Grab Sample-CN-1.2 -	**SOP Name:**	**Measured:** Friday, September 15, 2017 10:07:41 AM
Sample Source & type:	**Measured by:** D3M966	**Analysed:** Friday, September 15, 2017 10:07:42 AM
Sample bulk lot ref:	**Result Source:** Averaged	

Particle Name: Aluminium silicate	**Accessory Name:** Hydro 2000µP (A)	**Analysis model:** General purpose	**Sensitivity:** Normal
Particle RI: 1.650	**Absorption:** 0.1	**Size range:** 0.020 to 2000.000 um	**Obscuration:** 14.90 %
Dispersant Name: Water	**Dispersant RI:** 1.330	**Weighted Residual:** 1.658 %	**Result Emulation:** Off

---

Concentration: 0.0223 %Vol	**Span :** 4.946	**Uniformity:** 1.38	**Result units:** Volume
Specific Surface Area: 0.924 m²/g	**Surface Weighted Mean D[3,2]:** 6.493 um	**Vol. Weighted Mean D[4,3]:** 23.146 um	

d(0.1): **2.466** **um** **d(0.5):** **12.446** **um** **d(0.9):** **64.024** **um**



Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %
0.020	0.00	0.142	0.00	1.002	0.53	7.096	3.44	50.238	1.70	355.656	0.00
0.022	0.00	0.159	0.00	1.125	0.71	7.962	3.74	56.368	1.71	399.052	0.00
0.025	0.00	0.178	0.00	1.262	0.91	8.934	3.98	63.246	1.74	447.744	0.00
0.028	0.00	0.200	0.00	1.416	1.12	10.024	4.17	70.963	1.75	502.377	0.00
0.032	0.00	0.224	0.00	1.589	1.32	11.247	4.27	79.621	1.69	563.677	0.00
0.036	0.00	0.252	0.00	1.783	1.50	12.619	4.28	89.337	1.55	632.456	0.00
0.040	0.00	0.283	0.00	2.000	1.64	14.159	4.20	100.237	1.33	709.627	0.00
0.045	0.00	0.317	0.00	2.244	1.75	15.887	4.03	112.468	1.02	796.214	0.00
0.050	0.00	0.356	0.00	2.518	1.82	17.825	3.79	126.191	0.74	893.367	0.00
0.056	0.00	0.399	0.00	2.825	1.88	20.000	3.48	141.589	0.29	1002.374	0.00
0.063	0.00	0.448	0.00	3.170	1.93	22.440	3.14	158.866	0.08	1124.683	0.00
0.071	0.00	0.502	0.00	3.557	2.01	25.179	2.80	178.250	0.00	1261.915	0.00
0.080	0.00	0.564	0.00	3.991	2.13	28.251	2.47	200.000	0.00	1415.892	0.00
0.089	0.00	0.632	0.06	4.477	2.31	31.698	2.19	224.404	0.00	1588.656	0.00
0.100	0.00	0.710	0.14	5.024	2.54	35.566	1.96	251.785	0.00	1782.502	0.00
0.112	0.00	0.796	0.26	5.637	2.82	39.905	1.81	282.508	0.00	2000.000	
0.126	0.00	0.893	0.38	6.325	3.13	44.774	1.72	316.979	0.00		
0.142		1.002		7.096		50.238		355.656			

Operator notes: *LPIST-NS-B06 carrier fluid, Sample taken from LPIST-NS-B09*

MASTERSIZER 2000

Result Analysis Report

Sample Name: S2-Test5-Post Test Grab Sample-CN-1.3 -	**SOP Name:**	**Measured:** Friday, September 15, 2017 10:09:31 AM
Sample Source & type:	**Measured by:** D3M966	**Analysed:** Friday, September 15, 2017 10:09:33 AM
Sample bulk lot ref:	**Result Source:** Averaged	

Particle Name: Aluminium silicate	**Accessory Name:** Hydro 2000μP (A)	**Analysis model:** General purpose	**Sensitivity:** Normal
Particle RI: 1.650	**Absorption:** 0.1	**Size range:** 0.020 to 2000.000 um	**Obscuration:** 14.57 %
Dispersant Name: Water	**Dispersant RI:** 1.330	**Weighted Residual:** 1.614 %	**Result Emulation:** Off

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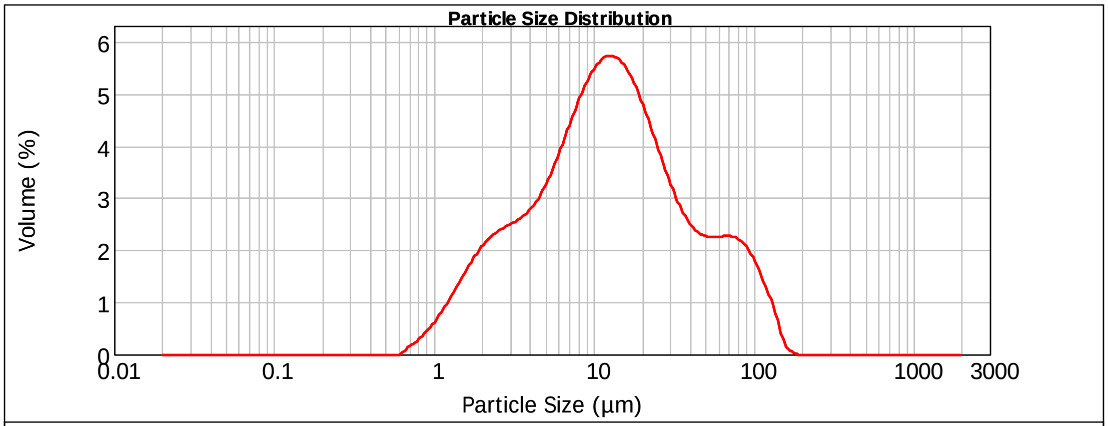
Concentration: 0.0217 %Vol	**Span :** 4.846	**Uniformity:** 1.36	**Result units:** Volume
Specific Surface Area: 0.928 m²/g	**Surface Weighted Mean D[3,2]:** 6.464 um	**Vol. Weighted Mean D[4,3]:** 22.754 um	

d(0.1): **2.471** **um** **d(0.5):** **12.337** **um** **d(0.9):** **62.252** **um**



S2-Test5-Post Test Grab Sample-CN-1.3 - Average, Friday, September 15, 2017 10:09:31 AM

Size (μm)	Volume In %
0.020	0.00
0.022	0.00
0.025	0.00
0.028	0.00
0.032	0.00
0.036	0.00
0.040	0.00
0.045	0.00
0.050	0.00
0.056	0.00
0.063	0.00
0.071	0.00
0.080	0.00
0.089	0.00
0.100	0.00
0.112	0.00
0.126	0.00
0.142	

Size (μm)	Volume In %
0.142	0.00
0.159	0.00
0.178	0.00
0.200	0.00
0.224	0.00
0.252	0.00
0.283	0.00
0.317	0.00
0.356	0.00
0.399	0.00
0.448	0.00
0.502	0.00
0.564	0.00
0.632	0.07
0.710	0.16
0.796	0.27
0.893	0.39
1.002	

Size (μm)	Volume In %
1.002	0.53
1.125	0.71
1.262	0.90
1.416	1.11
1.589	1.31
1.783	1.48
2.000	1.63
2.244	1.74
2.518	1.81
2.825	1.87
3.170	1.94
3.557	2.03
3.991	2.16
4.477	2.34
5.024	2.58
5.637	2.86
6.325	3.18
7.096	

Size (μm)	Volume In %
7.096	3.49
7.962	3.78
8.934	4.02
10.024	4.20
11.247	4.30
12.619	4.30
14.159	4.21
15.887	4.04
17.825	3.79
20.000	3.49
22.440	3.15
25.179	2.81
28.251	2.49
31.698	2.20
35.566	1.98
39.905	1.82
44.774	1.73
50.238	

Size (μm)	Volume In %
50.238	1.69
56.368	1.69
63.246	1.71
70.963	1.70
79.621	1.63
89.337	1.49
100.237	1.26
112.468	0.97
126.191	0.69
141.589	0.27
158.866	0.06
178.250	0.00
200.000	0.00
224.404	0.00
251.785	0.00
282.508	0.00
316.979	0.00
355.656	

Size (μm)	Volume In %
355.656	0.00
399.052	0.00
447.744	0.00
502.377	0.00
563.677	0.00
632.456	0.00
709.627	0.00
796.214	0.00
893.367	0.00
1002.374	0.00
1124.683	0.00
1261.915	0.00
1415.892	0.00
1588.656	0.00
1782.502	0.00
2000.000	

Operator notes: *LPIST-NS-B06 carrier fluid, Sample taken from LPIST-NS-B09*

MALVERN INSTRUMENTS

MASTERSIZER 2000

Result Analysis Report

Sample Name:
S2-Test8-Post Test Grab Sample-SF-01-1.1

SOP Name:

Measured:
Friday, September 15, 2017 10:54:48 AM

Sample Source & type:

Measured by:
D3M966

Analysed:
Friday, September 15, 2017 10:54:50 AM

Sample bulk lot ref:

Result Source:
Averaged

Particle Name:
Sodium Fluoride

Accessory Name:
Hydro 2000µP (A)

Analysis model:
General purpose

Sensitivity:
Normal

Particle RI:
1.320

Absorption:
1

Size range:
0.020 to 2000.000 um

Obscuration:
9.39 %

Dispersant Name:
Water

Dispersant RI:
1.330

Weighted Residual:
0.461 %

Result Emulation:
Off

Concentration:
0.0128 %Vol

Span :
3.991

Uniformity:
1.27

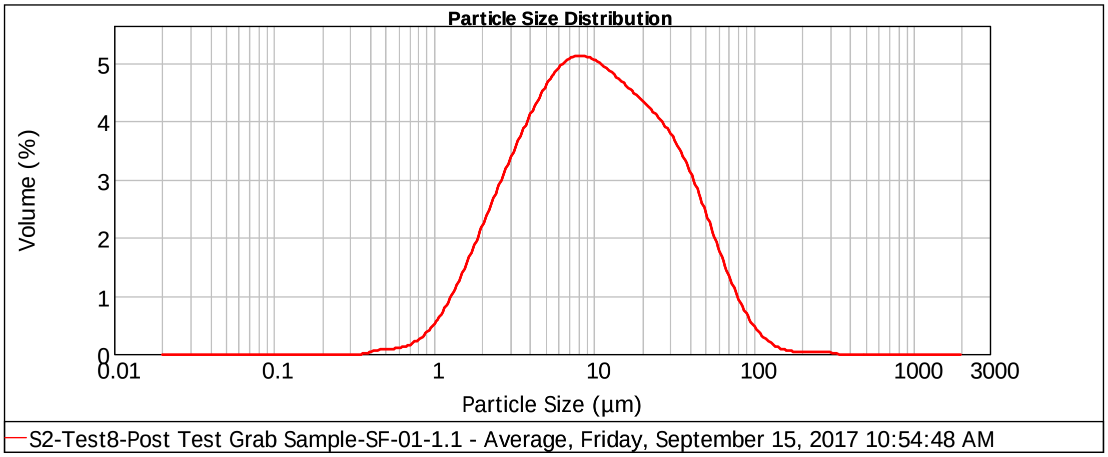
Result units:
Volume

Specific Surface Area:
1.04 m²/g

Surface Weighted Mean D[3,2]:
5.792 um

Vol. Weighted Mean D[4,3]:
17.569 um

d(0.1): 2.449 um **d(0.5): 9.990 um** **d(0.9): 42.319 um**



Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %
0.020	0.00	0.142	0.00	1.002	0.45	7.096	3.84	50.238	1.68	355.656	0.00
0.022	0.00	0.159	0.00	1.125	0.61	7.962	3.85	56.368	1.39	399.052	0.00
0.025	0.00	0.178	0.00	1.262	0.81	8.934	3.83	63.246	1.11	447.744	0.00
0.028	0.00	0.200	0.00	1.416	1.03	10.024	3.77	70.963	0.86	502.377	0.00
0.032	0.00	0.224	0.00	1.589	1.27	11.247	3.70	79.621	0.63	563.677	0.00
0.036	0.00	0.252	0.00	1.783	1.52	12.619	3.61	89.337	0.45	632.456	0.00
0.040	0.00	0.283	0.00	2.000	1.77	14.159	3.52	100.237	0.30	709.627	0.00
0.045	0.00	0.317	0.00	2.244	2.02	15.887	3.42	112.468	0.19	796.214	0.00
0.050	0.00	0.356	0.01	2.518	2.27	17.825	3.33	126.191	0.12	893.367	0.00
0.056	0.00	0.399	0.04	2.825	2.51	20.000	3.23	141.589	0.06	1002.374	0.00
0.063	0.00	0.448	0.06	3.170	2.74	22.440	3.12	158.866	0.04	1124.683	0.00
0.071	0.00	0.502	0.06	3.557	2.97	25.179	3.00	178.250	0.03	1261.915	0.00
0.080	0.00	0.564	0.07	3.991	3.18	28.251	2.86	200.000	0.03	1415.892	0.00
0.089	0.00	0.632	0.10	4.477	3.38	31.698	2.69	224.404	0.03	1588.656	0.00
0.100	0.00	0.710	0.15	5.024	3.55	35.566	2.48	251.785	0.03	1782.502	0.00
0.112	0.00	0.796	0.22	5.637	3.69	39.905	2.24	282.508	0.02	2000.000	
0.126	0.00	0.893	0.32	6.325	3.79	44.774	1.97	316.979	0.00		
0.142		1.002		7.096		50.238		355.656			

Operator notes: *LPIST-NS-B06 carrier fluid, Sample taken from LPIST-NS-B06*

MASTERSIZER 2000

Result Analysis Report

Sample Name: S2-Test8-Post Test Grab Sample-SF-01-1.2	**SOP Name:**	**Measured:** Friday, September 15, 2017 10:56:56 AM
Sample Source & type:	**Measured by:** D3M966	**Analysed:** Friday, September 15, 2017 10:56:57 AM
Sample bulk lot ref:	**Result Source:** Averaged	

Particle Name: Sodium Fluoride	**Accessory Name:** Hydro 2000μP (A)	**Analysis model:** General purpose	**Sensitivity:** Normal
Particle RI: 1.320	**Absorption:** 1	**Size range:** 0.020 to 2000.000 um	**Obscuration:** 8.50 %
Dispersant Name: Water	**Dispersant RI:** 1.330	**Weighted Residual:** 0.924 %	**Result Emulation:** Off

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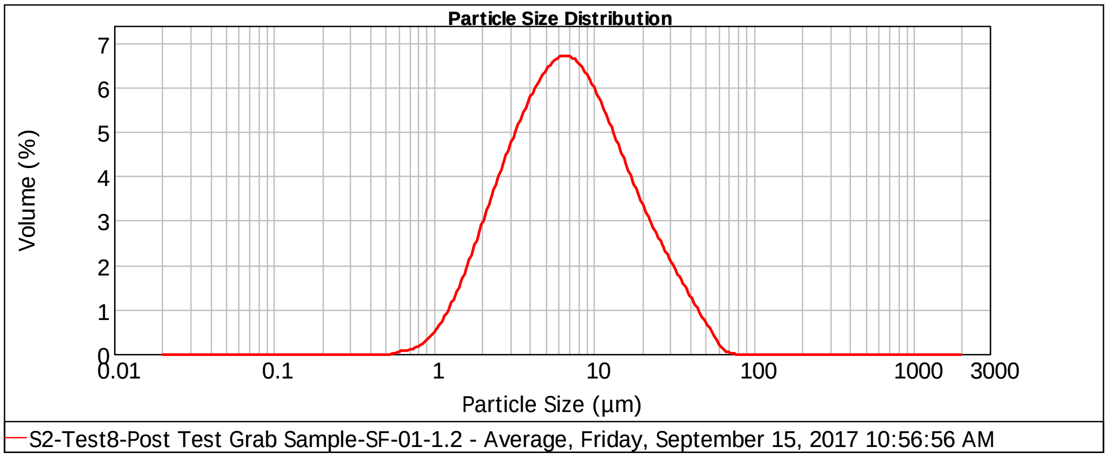
Concentration: 0.0096 %Vol	**Span :** 2.990	**Uniformity:** 0.922	**Result units:** Volume
Specific Surface Area: 1.23 m²/g	**Surface Weighted Mean D[3,2]:** 4.860 um	**Vol. Weighted Mean D[4,3]:** 10.127 um	

d(0.1): **2.246** **um** **d(0.5):** **6.839** **um** **d(0.9):** **22.697** **um**



Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %
0.020	0.00	0.142	0.00	1.002	0.45	7.096	4.99	50.238	0.43	355.656	0.00
0.022	0.00	0.159	0.00	1.125	0.67	7.962	4.85	56.368	0.19	399.052	0.00
0.025	0.00	0.178	0.00	1.262	0.94	8.934	4.64	63.246	0.05	447.744	0.00
0.028	0.00	0.200	0.00	1.416	1.26	10.024	4.37	70.963	0.00	502.377	0.00
0.032	0.00	0.224	0.00	1.589	1.62	11.247	4.06	79.621	0.00	563.677	0.00
0.036	0.00	0.252	0.00	1.783	2.00	12.619	3.72	89.337	0.00	632.456	0.00
0.040	0.00	0.283	0.00	2.000	2.39	14.159	3.38	100.237	0.00	709.627	0.00
0.045	0.00	0.317	0.00	2.244	2.78	15.887	3.04	112.468	0.00	796.214	0.00
0.050	0.00	0.356	0.00	2.518	3.16	17.825	2.71	126.191	0.00	893.367	0.00
0.056	0.00	0.399	0.00	2.825	3.53	20.000	2.41	141.589	0.00	1002.374	0.00
0.063	0.00	0.448	0.00	3.170	3.87	22.440	2.12	158.866	0.00	1124.683	0.00
0.071	0.00	0.502	0.00	3.557	4.18	25.179	1.85	178.250	0.00	1261.915	0.00
0.080	0.00	0.564	0.03	3.991	4.46	28.251	1.60	200.000	0.00	1415.892	0.00
0.089	0.00	0.632	0.06	4.477	4.71	31.698	1.35	224.404	0.00	1588.656	0.00
0.100	0.00	0.710	0.09	5.024	4.89	35.566	1.11	251.785	0.00	1782.502	0.00
0.112	0.00	0.796	0.17	5.637	5.01	39.905	0.87	282.508	0.00	2000.000	
0.126	0.00	0.893	0.29	6.325	5.05	44.774	0.64	316.979	0.00		
0.142		1.002		7.096		50.238		355.656			

Operator notes: *LPIST-NS-B06 carrier fluid, Sample taken from LPIST-NS-B06*

MASTERSIZER 2000

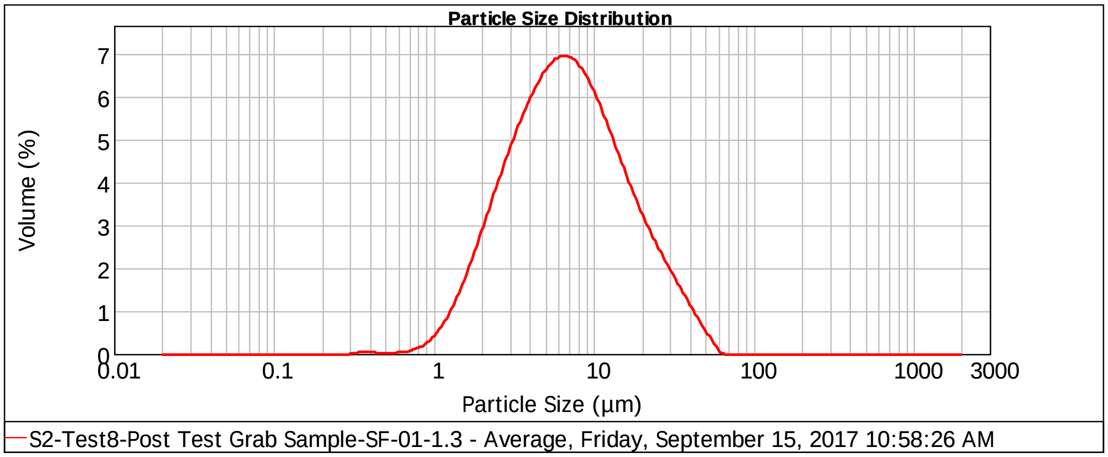
Result Analysis Report

Sample Name: S2-Test8-Post Test Grab Sample-SF-01-1.3	**SOP Name:**	**Measured:** Friday, September 15, 2017 10:58:26 AM
Sample Source & type:	**Measured by:** D3M966	**Analysed:** Friday, September 15, 2017 10:58:28 AM
Sample bulk lot ref:	**Result Source:** Averaged	

Particle Name: Sodium Fluoride	**Accessory Name:** Hydro 2000μP (A)	**Analysis model:** General purpose	**Sensitivity:** Normal
Particle RI: 1.320	**Absorption:** 1	**Size range:** 0.020 to 2000.000 um	**Obscuration:** 8.04 %
Dispersant Name: Water	**Dispersant RI:** 1.330	**Weighted Residual:** 1.133 %	**Result Emulation:** Off

Concentration: 0.0090 %Vol	**Span :** 2.851	**Uniformity:** 0.88	**Result units:** Volume
Specific Surface Area: 1.24 m²/g	**Surface Weighted Mean D[3,2]:** 4.819 um	**Vol. Weighted Mean D[4,3]:** 9.713 um	

d(0.1): 2.282 um **d(0.5): 6.712 um** **d(0.9): 21.419 um**



Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %
0.020	0.00	0.142	0.00	1.002	0.40	7.096	5.15	50.238	0.29	355.656	0.00
0.022	0.00	0.159	0.00	1.125	0.60	7.962	4.99	56.368	0.08	399.052	0.00
0.025	0.00	0.178	0.00	1.262	0.87	8.934	4.74	63.246	0.00	447.744	0.00
0.028	0.00	0.200	0.00	1.416	1.19	10.024	4.43	70.963	0.00	502.377	0.00
0.032	0.00	0.224	0.00	1.589	1.57	11.247	4.09	79.621	0.00	563.677	0.00
0.036	0.00	0.252	0.00	1.783	1.97	12.619	3.72	89.337	0.00	632.456	0.00
0.040	0.00	0.283	0.00	2.000	2.38	14.159	3.34	100.237	0.00	709.627	0.00
0.045	0.00	0.317	0.03	2.244	2.80	15.887	2.97	112.468	0.00	796.214	0.00
0.050	0.00	0.356	0.03	2.518	3.21	17.825	2.63	126.191	0.00	893.367	0.00
0.056	0.00	0.399	0.02	2.825	3.61	20.000	2.31	141.589	0.00	1002.374	0.00
0.063	0.00	0.448	0.02	3.170	3.97	22.440	2.01	158.866	0.00	1124.683	0.00
0.071	0.00	0.502	0.02	3.557	4.32	25.179	1.74	178.250	0.00	1261.915	0.00
0.080	0.00	0.564	0.03	3.991	4.62	28.251	1.49	200.000	0.00	1415.892	0.00
0.089	0.00	0.632	0.04	4.477	4.88	31.698	1.24	224.404	0.00	1588.656	0.00
0.100	0.00	0.710	0.08	5.024	5.07	35.566	0.99	251.785	0.00	1782.502	0.00
0.112	0.00	0.796	0.14	5.637	5.19	39.905	0.75	282.508	0.00	2000.000	
0.126	0.00	0.893	0.24	6.325	5.22	44.774	0.51	316.979	0.00		
0.142		1.002		7.096		50.238		355.656			

Operator notes: *LPIST-NS-B06 carrier fluid, Sample taken from LPIST-NS-B06*

MASTERSIZER 2000

Result Analysis Report

Sample Name:
S1-Test 1-Post Test Grab Sample-NC20-01-1.1 - Average,

SOP Name:

Measured:
Friday, September 15, 2017 2:05:01 PM

Sample Source & type:

Measured by:
D3M966

Analysed:
Friday, September 15, 2017 2:05:02 PM

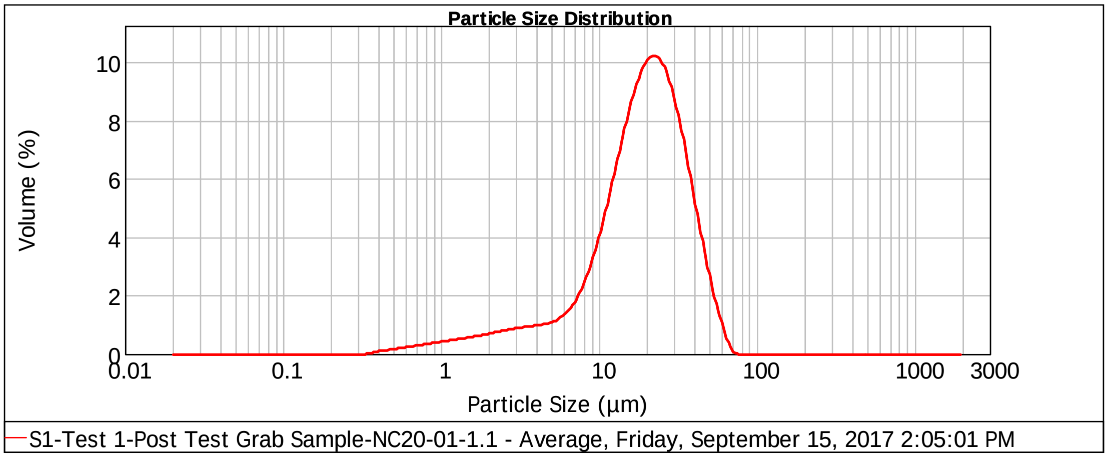
Sample bulk lot ref:

Result Source:
Averaged

Particle Name: Sodium Oxalate	**Accessory Name:** Hydro 2000µP (A)	**Analysis model:** General purpose	**Sensitivity:** Normal
Particle RI: 1.480	**Absorption:** 1	**Size range:** 0.020 to 2000.000 um	**Obscuration:** 11.98 %
Dispersant Name: Water	**Dispersant RI:** 1.330	**Weighted Residual:** 0.336 %	**Result Emulation:** Off

Concentration: 0.0260 %Vol	**Span :** 1.695	**Uniformity:** 0.508	**Result units:** Volume
Specific Surface Area: 0.28 m²/g	**Surface Weighted Mean D[3,2]:** 9.170 um	**Vol. Weighted Mean D[4,3]:** 21.243 um	

d(0.1): **5.483** **um** **d(0.5):** **19.548** **um** **d(0.9):** **38.624** **um**



Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %
0.020	0.00	0.142	0.00	1.002	0.33	7.096	1.54	50.238	1.60	355.656	0.00
0.022	0.00	0.159	0.00	1.125	0.36	7.962	2.02	56.368	0.84	399.052	0.00
0.025	0.00	0.178	0.00	1.262	0.39	8.934	2.63	63.246	0.25	447.744	0.00
0.028	0.00	0.200	0.00	1.416	0.42	10.024	3.38	70.963	0.00	502.377	0.00
0.032	0.00	0.224	0.00	1.589	0.46	11.247	4.23	79.621	0.00	563.677	0.00
0.036	0.00	0.252	0.00	1.783	0.50	12.619	5.13	89.337	0.00	632.456	0.00
0.040	0.00	0.283	0.00	2.000	0.54	14.159	6.01	100.237	0.00	709.627	0.00
0.045	0.00	0.317	0.00	2.244	0.58	15.887	6.78	112.468	0.00	796.214	0.00
0.050	0.00	0.356	0.05	2.518	0.62	17.825	7.36	126.191	0.00	893.367	0.00
0.056	0.00	0.399	0.08	2.825	0.66	20.000	7.66	141.589	0.00	1002.374	0.00
0.063	0.00	0.448	0.11	3.170	0.69	22.440	7.65	158.866	0.00	1124.683	0.00
0.071	0.00	0.502	0.14	3.557	0.72	25.179	7.31	178.250	0.00	1261.915	0.00
0.080	0.00	0.564	0.17	3.991	0.74	28.251	6.65	200.000	0.00	1415.892	0.00
0.089	0.00	0.632	0.20	4.477	0.78	31.698	5.75	224.404	0.00	1588.656	0.00
0.100	0.00	0.710	0.24	5.024	0.86	35.566	4.70	251.785	0.00	1782.502	0.00
0.112	0.00	0.796	0.27	5.637	0.99	39.905	3.58	282.508	0.00	2000.000	
0.126	0.00	0.893	0.30	6.325	1.21	44.774	2.52	316.979	0.00		
0.142		1.002		7.096		50.238		355.656			

Operator notes: *LPIST-NS-B01 filtered carrier fluid, Sample taken from LPIST-NS-B01*

MALVERN INSTRUMENTS

MASTERSIZER 2000

Result Analysis Report

Sample Name:
S1-Test 1-Post Test Grab Sample-NC20-01-1.2 - Average

SOP Name:

Measured:
Friday, September 15, 2017 2:06:44 PM

Sample Source & type:

Measured by:
D3M966

Analysed:
Friday, September 15, 2017 2:06:46 PM

Sample bulk lot ref:

Result Source:
Averaged

Particle Name:
Sodium Oxalate

Accessory Name:
Hydro 2000μP (A)

Analysis model:
General purpose

Sensitivity:
Normal

Particle RI:
1.480

Absorption:
1

Size range:
0.020 to 2000.000 um

Obscuration:
10.09 %

Dispersant Name:
Water

Dispersant RI:
1.330

Weighted Residual:
0.355 %

Result Emulation:
Off

Concentration:
0.0205 %Vol

Span :
1.583

Uniformity:
0.471

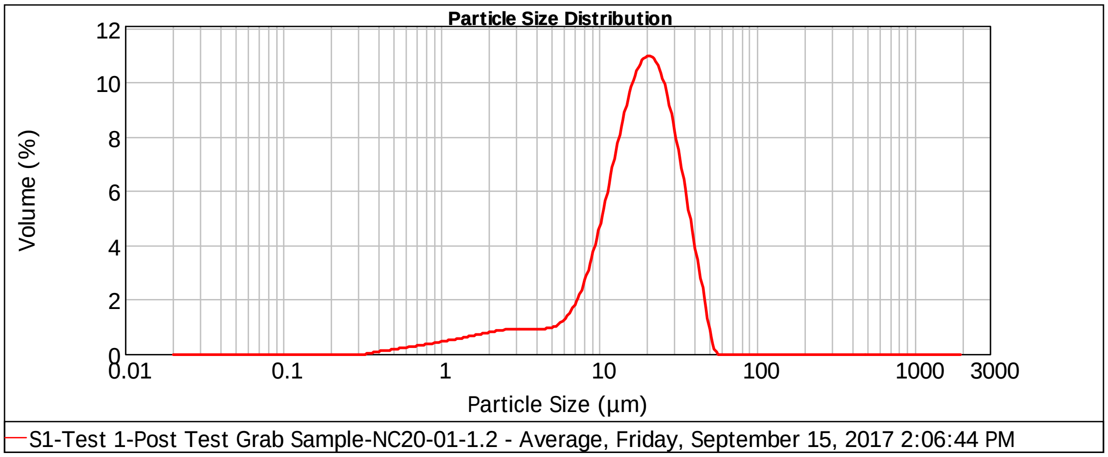
Result units:
Volume

Specific Surface Area:
0.295 m²/g

Surface Weighted Mean D[3,2]:
8.684 um

Vol. Weighted Mean D[4,3]:
19.159 um

d(0.1): 5.169 um **d(0.5): 18.116 um** **d(0.9): 33.856 um**



Size (μm)	Volume In %
0.020	0.00
0.022	0.00
0.025	0.00
0.028	0.00
0.032	0.00
0.036	0.00
0.040	0.00
0.045	0.00
0.050	0.00
0.056	0.00
0.063	0.00
0.071	0.00
0.080	0.00
0.089	0.00
0.100	0.00
0.112	0.00
0.126	0.00
0.142	

Size (μm)	Volume In %
0.142	0.00
0.159	0.00
0.178	0.00
0.200	0.00
0.224	0.00
0.252	0.00
0.283	0.00
0.317	0.00
0.356	0.05
0.399	0.08
0.448	0.11
0.502	0.14
0.564	0.18
0.632	0.21
0.710	0.24
0.796	0.28
0.893	0.32
1.002	

Size (μm)	Volume In %
1.002	0.36
1.125	0.39
1.262	0.44
1.416	0.48
1.589	0.53
1.783	0.57
2.000	0.61
2.244	0.65
2.518	0.68
2.825	0.70
3.170	0.70
3.557	0.70
3.991	0.69
4.477	0.71
5.024	0.77
5.637	0.92
6.325	1.20
7.096	

Size (μm)	Volume In %
7.096	1.62
7.962	2.22
8.934	2.98
10.024	3.91
11.247	4.92
12.619	5.95
14.159	6.90
15.887	7.67
17.825	8.15
20.000	8.26
22.440	7.98
25.179	7.32
28.251	6.33
31.698	5.12
35.566	3.84
39.905	2.59
44.774	1.36
50.238	

Size (μm)	Volume In %
50.238	0.17
56.368	0.00
63.246	0.00
70.963	0.00
79.621	0.00
89.337	0.00
100.237	0.00
112.468	0.00
126.191	0.00
141.589	0.00
158.866	0.00
178.250	0.00
200.000	0.00
224.404	0.00
251.785	0.00
282.508	0.00
316.979	0.00
355.656	

Size (μm)	Volume In %
355.656	0.00
399.052	0.00
447.744	0.00
502.377	0.00
563.677	0.00
632.456	0.00
709.627	0.00
796.214	0.00
893.367	0.00
1002.374	0.00
1124.683	0.00
1261.915	0.00
1415.892	0.00
1588.656	0.00
1782.502	0.00
2000.000	

Operator notes: *LPIST-NS-B01 filtered carrier fluid, Sample taken from LPIST-NS-B01*

MASTERSIZER 2000

Result Analysis Report

Sample Name:
S1-Test 1-Post Test Grab Sample-NC20-01-1.3 - Average

SOP Name:

Measured:
Friday, September 15, 2017 2:08:15 PM

Sample Source & type:

Measured by:
D3M966

Analysed:
Friday, September 15, 2017 2:08:17 PM

Sample bulk lot ref:

Result Source:
Averaged

Particle Name:
Sodium Oxalate

Accessory Name:
Hydro 2000μP (A)

Analysis model:
General purpose

Sensitivity:
Normal

Particle RI:
1.480

Absorption:
1

Size range:
0.020 to 2000.000 um

Obscuration:
9.09 %

Dispersant Name:
Water

Dispersant RI:
1.330

Weighted Residual:
0.403 %

Result Emulation:
Off

Concentration:
0.0182 %Vol

Span :
1.577

Uniformity:
0.467

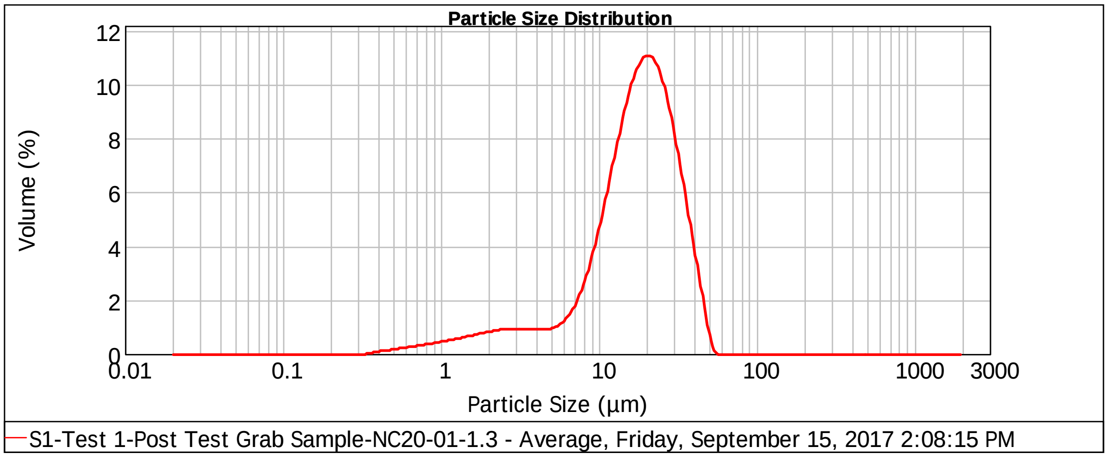
Result units:
Volume

Specific Surface Area:
0.298 m²/g

Surface Weighted Mean D[3,2]:
8.605 um

Vol. Weighted Mean D[4,3]:
18.959 um

d(0.1): 5.072 um **d(0.5): 17.968 um** **d(0.9): 33.402 um**



Size (μm)	Volume In %
0.020	0.00
0.022	0.00
0.025	0.00
0.028	0.00
0.032	0.00
0.036	0.00
0.040	0.00
0.045	0.00
0.050	0.00
0.056	0.00
0.063	0.00
0.071	0.00
0.080	0.00
0.089	0.00
0.100	0.00
0.112	0.00
0.126	0.00
0.142	

Size (μm)	Volume In %
0.142	0.00
0.159	0.00
0.178	0.00
0.200	0.00
0.224	0.00
0.252	0.00
0.283	0.00
0.317	0.00
0.356	0.05
0.399	0.08
0.448	0.11
0.502	0.14
0.564	0.17
0.632	0.21
0.710	0.24
0.796	0.28
0.893	0.32
1.002	

Size (μm)	Volume In %
1.002	0.36
1.125	0.40
1.262	0.45
1.416	0.50
1.589	0.55
1.783	0.59
2.000	0.64
2.244	0.67
2.518	0.70
2.825	0.71
3.170	0.70
3.557	0.69
3.991	0.68
4.477	0.69
5.024	0.75
5.637	0.90
6.325	1.18
7.096	

Size (μm)	Volume In %
7.096	1.61
7.962	2.23
8.934	3.02
10.024	3.97
11.247	5.00
12.619	6.05
14.159	7.01
15.887	7.77
17.825	8.24
20.000	8.32
22.440	8.00
25.179	7.29
28.251	6.26
31.698	5.02
35.566	3.72
39.905	2.44
44.774	1.14
50.238	

Size (μm)	Volume In %
50.238	0.12
56.368	0.00
63.246	0.00
70.963	0.00
79.621	0.00
89.337	0.00
100.237	0.00
112.468	0.00
126.191	0.00
141.589	0.00
158.866	0.00
178.250	0.00
200.000	0.00
224.404	0.00
251.785	0.00
282.508	0.00
316.979	0.00
355.656	

Size (μm)	Volume In %
355.656	0.00
399.052	0.00
447.744	0.00
502.377	0.00
563.677	0.00
632.456	0.00
709.627	0.00
796.214	0.00
893.367	0.00
1002.374	0.00
1124.683	0.00
1261.915	0.00
1415.892	0.00
1588.656	0.00
1782.502	0.00
2000.000	

Operator notes: *LPIST-NS-B01 filtered carrier fluid, Sample taken from LPIST-NS-B01*

MALVERN
INSTRUMENTS

MASTERSIZER 2000

S1-Test 2-Post Test Grab Sample-NC45-01-1.1 - Average

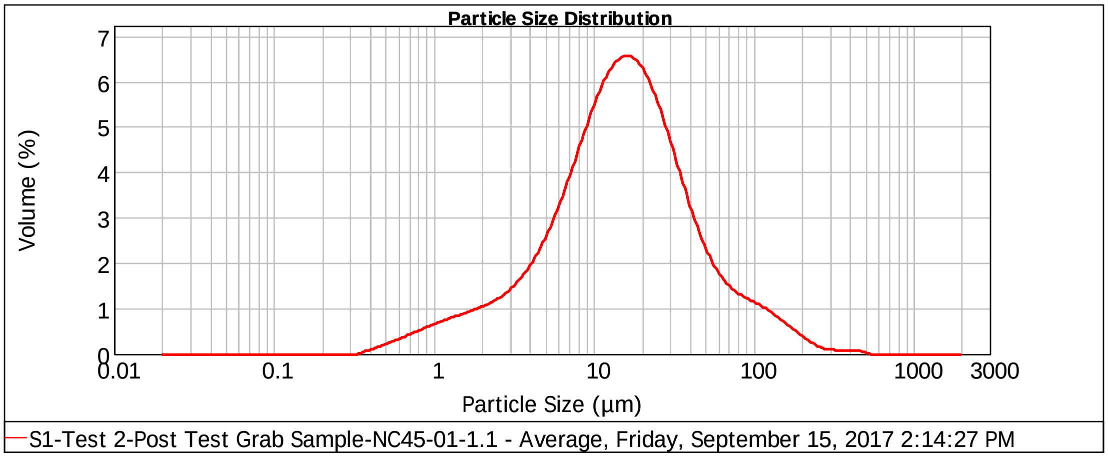
Result Analysis Report

Sample Name:	**SOP Name:**	**Measured:** Friday, September 15, 2017 2:14:27 PM
Sample Source & type:	**Measured by:** D3M966	**Analysed:** Friday, September 15, 2017 2:14:28 PM
Sample bulk lot ref:	**Result Source:** Averaged	

Particle Name: Sodium Oxalate	**Accessory Name:** Hydro 2000μP (A)	**Analysis model:** General purpose	**Sensitivity:** Normal
Particle RI: 1.480	**Absorption:** 1	**Size range:** 0.020 to 2000.000 um	**Obscuration:** 11.81 %
Dispersant Name: Water	**Dispersant RI:** 1.330	**Weighted Residual:** 0.347 %	**Result Emulation:** Off

Concentration: 0.0192 %Vol	**Span :** 3.499	**Uniformity:** 1.23	**Result units:** Volume
Specific Surface Area: 0.371 m²/g	**Surface Weighted Mean D[3,2]:** 6.910 um	**Vol. Weighted Mean D[4,3]:** 26.107 um	

d(0.1): 3.244 um **d(0.5): 15.082 um** **d(0.9): 56.009 um**



Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %
0.020	0.00	0.142	0.00	1.002	0.52	7.096	3.16	50.238	1.60	355.656	0.05
0.022	0.00	0.159	0.00	1.125	0.57	7.962	3.55	56.368	1.36	399.052	0.05
0.025	0.00	0.178	0.00	1.262	0.62	8.934	3.93	63.246	1.18	447.744	0.04
0.028	0.00	0.200	0.00	1.416	0.66	10.024	4.29	70.963	1.06	502.377	0.00
0.032	0.00	0.224	0.00	1.589	0.71	11.247	4.58	79.621	0.97	563.677	0.00
0.036	0.00	0.252	0.00	1.783	0.75	12.619	4.80	89.337	0.90	632.456	0.00
0.040	0.00	0.283	0.00	2.000	0.81	14.159	4.92	100.237	0.83	709.627	0.00
0.045	0.00	0.317	0.00	2.244	0.88	15.887	4.93	112.468	0.75	796.214	0.00
0.050	0.00	0.356	0.05	2.518	0.96	17.825	4.84	126.191	0.67	893.367	0.00
0.056	0.00	0.399	0.08	2.825	1.07	20.000	4.64	141.589	0.57	1002.374	0.00
0.063	0.00	0.448	0.14	3.170	1.20	22.440	4.34	158.866	0.46	1124.683	0.00
0.071	0.00	0.502	0.19	3.557	1.37	25.179	3.98	178.250	0.36	1261.915	0.00
0.080	0.00	0.564	0.24	3.991	1.57	28.251	3.56	200.000	0.26	1415.892	0.00
0.089	0.00	0.632	0.30	4.477	1.81	31.698	3.12	224.404	0.17	1588.656	0.00
0.100	0.00	0.710	0.35	5.024	2.09	35.566	2.68	251.785	0.10	1782.502	0.00
0.112	0.00	0.796	0.41	5.637	2.41	39.905	2.27	282.508	0.08	2000.000	
0.126	0.00	0.893	0.47	6.325	2.77	44.774	1.91	316.979	0.06		
0.142		1.002		7.096		50.238		355.656			

Operator notes: *LPIST-NS-B01 filtered carrier fluid, Sample taken from LPIST-NS-B02*

MASTERSIZER 2000

Result Analysis Report

Sample Name:
S1-Test 2-Post Test Grab Sample-NC45-01-1.2 - Average

SOP Name:

Measured:
Friday, September 15, 2017 2:16:38 PM

Sample Source & type:

Measured by:
D3M966

Analysed:
Friday, September 15, 2017 2:16:39 PM

Sample bulk lot ref:

Result Source:
Averaged

Particle Name:
Sodium Oxalate

Accessory Name:
Hydro 2000μP (A)

Analysis model:
General purpose

Sensitivity:
Normal

Particle RI:
1.480

Absorption:
1

Size range:
0.020 to 2000.000 um

Obscuration:
10.24 %

Dispersant Name:
Water

Dispersant RI:
1.330

Weighted Residual:
0.312 %

Result Emulation:
Off

Concentration:
0.0123 %Vol

Span :
3.446

Uniformity:
1.23

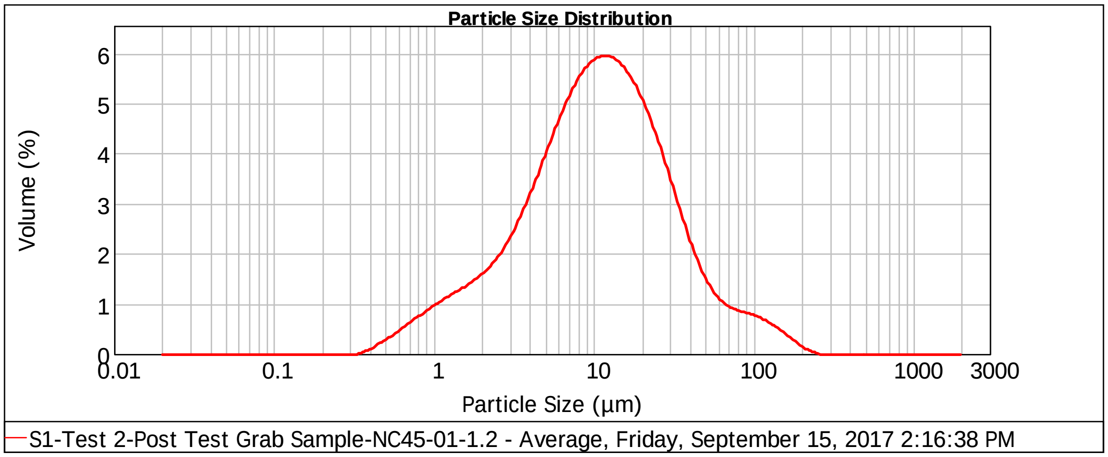
Result units:
Volume

Specific Surface Area:
0.492 m²/g

Surface Weighted Mean D[3,2]:
5.213 um

Vol. Weighted Mean D[4,3]:
18.369 um

d(0.1): **2.222** **um** **d(0.5):** **10.697** **um** **d(0.9):** **39.083** **um**



Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %
0.020	0.00	0.142	0.00	1.002	0.77	7.096	4.02	50.238	1.02	355.656	0.00
0.022	0.00	0.159	0.00	1.125	0.84	7.962	4.23	56.368	0.85	399.052	0.00
0.025	0.00	0.178	0.00	1.262	0.91	8.934	4.37	63.246	0.74	447.744	0.00
0.028	0.00	0.200	0.00	1.416	0.98	10.024	4.46	70.963	0.68	502.377	0.00
0.032	0.00	0.224	0.00	1.589	1.06	11.247	4.48	79.621	0.64	563.677	0.00
0.036	0.00	0.252	0.00	1.783	1.15	12.619	4.44	89.337	0.61	632.456	0.00
0.040	0.00	0.283	0.00	2.000	1.25	14.159	4.34	100.237	0.56	709.627	0.00
0.045	0.00	0.317	0.00	2.244	1.38	15.887	4.18	112.468	0.50	796.214	0.00
0.050	0.00	0.356	0.05	2.518	1.55	17.825	3.97	126.191	0.43	893.367	0.00
0.056	0.00	0.399	0.09	2.825	1.75	20.000	3.70	141.589	0.34	1002.374	0.00
0.063	0.00	0.448	0.18	3.170	1.98	22.440	3.39	158.866	0.24	1124.683	0.00
0.071	0.00	0.502	0.25	3.557	2.25	25.179	3.04	178.250	0.16	1261.915	0.00
0.080	0.00	0.564	0.34	3.991	2.55	28.251	2.66	200.000	0.07	1415.892	0.00
0.089	0.00	0.632	0.43	4.477	2.86	31.698	2.27	224.404	0.03	1588.656	0.00
0.100	0.00	0.710	0.51	5.024	3.18	35.566	1.90	251.785	0.00	1782.502	0.00
0.112	0.00	0.796	0.60	5.637	3.49	39.905	1.55	282.508	0.00	2000.000	
0.126	0.00	0.893	0.69	6.325	3.77	44.774	1.26	316.979	0.00		
0.142		1.002		7.096		50.238		355.656			

Operator notes: *LPIST-NS-B01 filtered carrier fluid, Sample taken from LPIST-NS-B02*

MASTERSIZER 2000

Result Analysis Report

Sample Name:
S1-Test 2-Post Test Grab Sample-NC45-01-1.3 - Average

SOP Name:

Measured:
Friday, September 15, 2017 2:18:12 PM

Sample Source & type:

Measured by:
D3M966

Analysed:
Friday, September 15, 2017 2:18:13 PM

Sample bulk lot ref:

Result Source:
Averaged

Particle Name:
Sodium Oxalate

Accessory Name:
Hydro 2000μP (A)

Analysis model:
General purpose

Sensitivity:
Normal

Particle RI:
1.480

Absorption:
1

Size range:
0.020 to 2000.000 um

Obscuration:
9.46 %

Dispersant Name:
Water

Dispersant RI:
1.330

Weighted Residual:
0.442 %

Result Emulation:
Off

Concentration:
0.0104 %Vol

Span :
3.728

Uniformity:
1.3

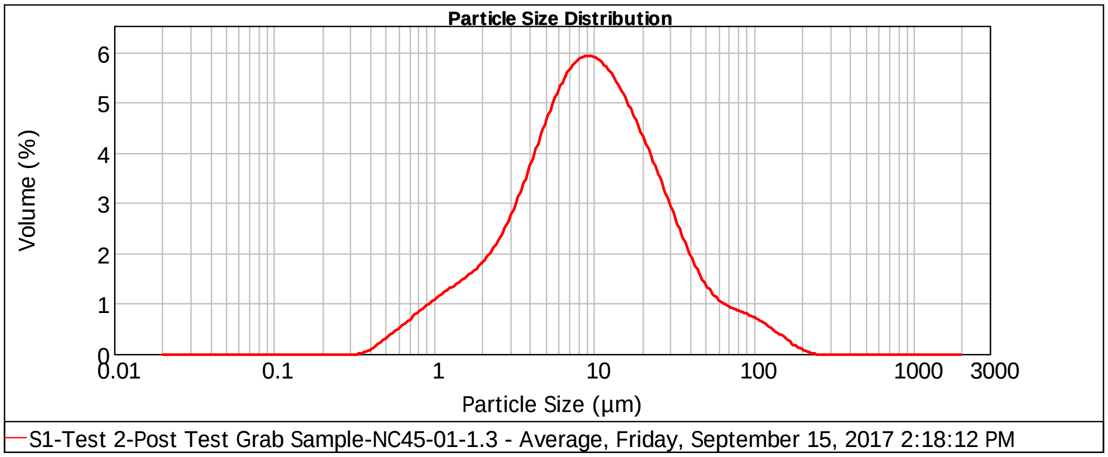
Result units:
Volume

Specific Surface Area:
0.536 m²/g

Surface Weighted Mean D[3,2]:
4.780 um

Vol. Weighted Mean D[4,3]:
16.711 um

d(0.1): **2.020** **um** **d(0.5):** **9.285** **um** **d(0.9):** **36.633** **um**



Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %
0.020	0.00	0.142	0.00	1.002	0.85	7.096	4.34	50.238	0.95	355.656	0.00
0.022	0.00	0.159	0.00	1.125	0.94	7.962	4.44	56.368	0.82	399.052	0.00
0.025	0.00	0.178	0.00	1.262	1.02	8.934	4.46	63.246	0.73	447.744	0.00
0.028	0.00	0.200	0.00	1.416	1.11	10.024	4.41	70.963	0.67	502.377	0.00
0.032	0.00	0.224	0.00	1.589	1.20	11.247	4.29	79.621	0.63	563.677	0.00
0.036	0.00	0.252	0.00	1.783	1.30	12.619	4.13	89.337	0.58	632.456	0.00
0.040	0.00	0.283	0.00	2.000	1.43	14.159	3.92	100.237	0.52	709.627	0.00
0.045	0.00	0.317	0.00	2.244	1.59	15.887	3.69	112.468	0.45	796.214	0.00
0.050	0.00	0.356	0.04	2.518	1.79	17.825	3.43	126.191	0.36	893.367	0.00
0.056	0.00	0.399	0.09	2.825	2.04	20.000	3.15	141.589	0.28	1002.374	0.00
0.063	0.00	0.448	0.20	3.170	2.32	22.440	2.86	158.866	0.18	1124.683	0.00
0.071	0.00	0.502	0.28	3.557	2.64	25.179	2.55	178.250	0.10	1261.915	0.00
0.080	0.00	0.564	0.37	3.991	2.98	28.251	2.24	200.000	0.05	1415.892	0.00
0.089	0.00	0.632	0.47	4.477	3.32	31.698	1.93	224.404	0.00	1588.656	0.00
0.100	0.00	0.710	0.57	5.024	3.64	35.566	1.63	251.785	0.00	1782.502	0.00
0.112	0.00	0.796	0.67	5.637	3.93	39.905	1.36	282.508	0.00	2000.000	
0.126	0.00	0.893	0.76	6.325	4.17	44.774	1.13	316.979	0.00		
0.142		1.002		7.096		50.238		355.656			

Operator notes: *LPIST-NS-B01 filtered carrier fluid, Sample taken from LPIST-NS-B02*

MASTERSIZER 2000

Result Analysis Report

Sample Name:
S2-Test 2-Post Test Grab Sample-NA-FR-01-1.1 - Average

SOP Name:

Measured:
Friday, September 15, 2017 2:43:21 PM

Sample Source & type:

Measured by:
D3M966

Analysed:
Friday, September 15, 2017 2:43:23 PM

Sample bulk lot ref:

Result Source:
Averaged

Particle Name:
Resin

Accessory Name:
Hydro 2000μP (A)

Analysis model:
General purpose

Sensitivity:
Normal

Particle RI:
1.600

Absorption:
1

Size range:
0.020 to 2000.000 um

Obscuration:
10.06 %

Dispersant Name:
Water

Dispersant RI:
1.330

Weighted Residual:
0.827 %

Result Emulation:
Off

Concentration:
0.0422 %Vol

Span :
1.591

Uniformity:
0.467

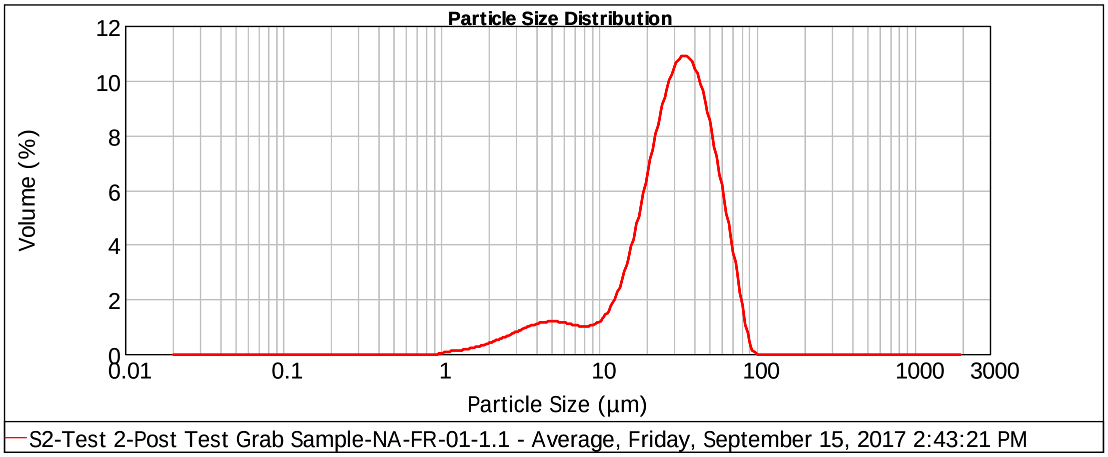
Result units:
Volume

Specific Surface Area:
0.348 m²/g

Surface Weighted Mean D[3,2]:
17.235 um

Vol. Weighted Mean D[4,3]:
32.915 um

d(0.1): 8.961 um **d(0.5): 30.966 um** **d(0.9): 58.215 um**



Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %
0.020	0.00	0.142	0.00	1.002	0.04	7.096	0.78	50.238	5.90	355.656	0.00
0.022	0.00	0.159	0.00	1.125	0.08	7.962	0.76	56.368	4.71	399.052	0.00
0.025	0.00	0.178	0.00	1.262	0.11	8.934	0.82	63.246	3.47	447.744	0.00
0.028	0.00	0.200	0.00	1.416	0.14	10.024	0.98	70.963	2.28	502.377	0.00
0.032	0.00	0.224	0.00	1.589	0.19	11.247	1.29	79.621	0.91	563.677	0.00
0.036	0.00	0.252	0.00	1.783	0.25	12.619	1.77	89.337	0.08	632.456	0.00
0.040	0.00	0.283	0.00	2.000	0.32	14.159	2.45	100.237	0.00	709.627	0.00
0.045	0.00	0.317	0.00	2.244	0.41	15.887	3.29	112.468	0.00	796.214	0.00
0.050	0.00	0.356	0.00	2.518	0.50	17.825	4.28	126.191	0.00	893.367	0.00
0.056	0.00	0.399	0.00	2.825	0.60	20.000	5.32	141.589	0.00	1002.374	0.00
0.063	0.00	0.448	0.00	3.170	0.70	22.440	6.35	158.866	0.00	1124.683	0.00
0.071	0.00	0.502	0.00	3.557	0.79	25.179	7.23	178.250	0.00	1261.915	0.00
0.080	0.00	0.564	0.00	3.991	0.85	28.251	7.88	200.000	0.00	1415.892	0.00
0.089	0.00	0.632	0.00	4.477	0.89	31.698	8.20	224.404	0.00	1588.656	0.00
0.100	0.00	0.710	0.00	5.024	0.89	35.566	8.15	251.785	0.00	1782.502	0.00
0.112	0.00	0.796	0.00	5.637	0.86	39.905	7.72	282.508	0.00	2000.000	
0.126	0.00	0.893	0.00	6.325	0.82	44.774	6.95	316.979	0.00		
0.142		1.002		7.096		50.238		355.656			

Operator notes: *tap water carrier fluid, Solids taken from Poast S2, Test 7 Simulant (3/14/2017) contains sRF, Fe, HEDTA*

MALVERN INSTRUMENTS

MASTERSIZER 2000

Result Analysis Report

Sample Name:
S2-Test 7-Post Test Grab Sample-NA-FR-01-1.2

SOP Name:

Measured:
Friday, September 15, 2017 2:45:47 PM

Sample Source & type:

Measured by:
D3M966

Analysed:
Friday, September 15, 2017 2:45:48 PM

Sample bulk lot ref:

Result Source:
Averaged

Particle Name:
Resin

Accessory Name:
Hydro 2000μP (A)

Analysis model:
General purpose

Sensitivity:
Normal

Particle RI:
1.600

Absorption:
1

Size range:
0.020 to 2000.000 um

Obscuration:
10.56 %

Dispersant Name:
Water

Dispersant RI:
1.330

Weighted Residual:
1.653 %

Result Emulation:
Off

Concentration:
0.0327 %Vol

Span :
1.571

Uniformity:
0.462

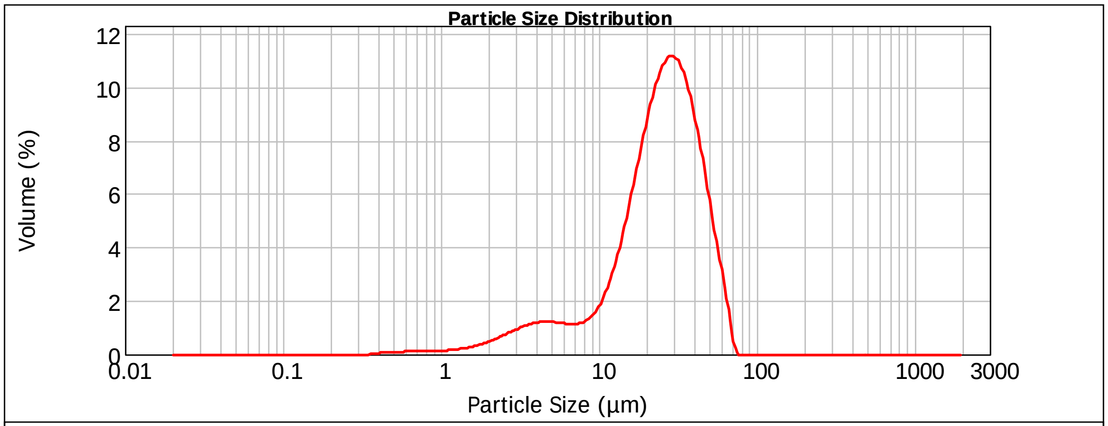
Result units:
Volume

Specific Surface Area:
0.464 m²/g

Surface Weighted Mean D[3,2]:
12.932 um

Vol. Weighted Mean D[4,3]:
26.808 um

d(0.1): 7.043 um **d(0.5): 25.471 um** **d(0.9): 47.067 um**



S2-Test 7-Post Test Grab Sample-NA-FR-01-1.2 - Average, Friday, September 15, 2017 2:45:47 PM

Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %
0.020	0.00	0.142	0.00	1.002	0.11	7.096	0.86	50.238	3.69	355.656	0.00
0.022	0.00	0.159	0.00	1.125	0.13	7.962	0.96	56.368	2.46	399.052	0.00
0.025	0.00	0.178	0.00	1.262	0.15	8.934	1.17	63.246	1.13	447.744	0.00
0.028	0.00	0.200	0.00	1.416	0.19	10.024	1.56	70.963	0.02	502.377	0.00
0.032	0.00	0.224	0.00	1.589	0.25	11.247	2.13	79.621	0.00	563.677	0.00
0.036	0.00	0.252	0.00	1.783	0.32	12.619	2.90	89.337	0.00	632.456	0.00
0.040	0.00	0.283	0.00	2.000	0.40	14.159	3.84	100.237	0.00	709.627	0.00
0.045	0.00	0.317	0.00	2.244	0.49	15.887	4.90	112.468	0.00	796.214	0.00
0.050	0.00	0.356	0.01	2.518	0.60	17.825	6.00	126.191	0.00	893.367	0.00
0.056	0.00	0.399	0.04	2.825	0.70	20.000	7.00	141.589	0.00	1002.374	0.00
0.063	0.00	0.448	0.06	3.170	0.79	22.440	7.81	158.866	0.00	1124.683	0.00
0.071	0.00	0.502	0.07	3.557	0.86	25.179	8.30	178.250	0.00	1261.915	0.00
0.080	0.00	0.564	0.08	3.991	0.91	28.251	8.40	200.000	0.00	1415.892	0.00
0.089	0.00	0.632	0.08	4.477	0.92	31.698	8.08	224.404	0.00	1588.656	0.00
0.100	0.00	0.710	0.08	5.024	0.91	35.566	7.36	251.785	0.00	1782.502	0.00
0.112	0.00	0.796	0.09	5.637	0.87	39.905	6.31	282.508	0.00	2000.000	
0.126	0.00	0.893	0.10	6.325	0.85	44.774	5.05	316.979	0.00		
0.142		1.002		7.096		50.238		355.656			

Operator notes: *tap water carrier fluid, Solids taken from Poast S2, Test 7 Simulant (3/14/2017) contains sRF, Fe, HEDTA*

MALVERN INSTRUMENTS

MASTERSIZER 2000

Result Analysis Report

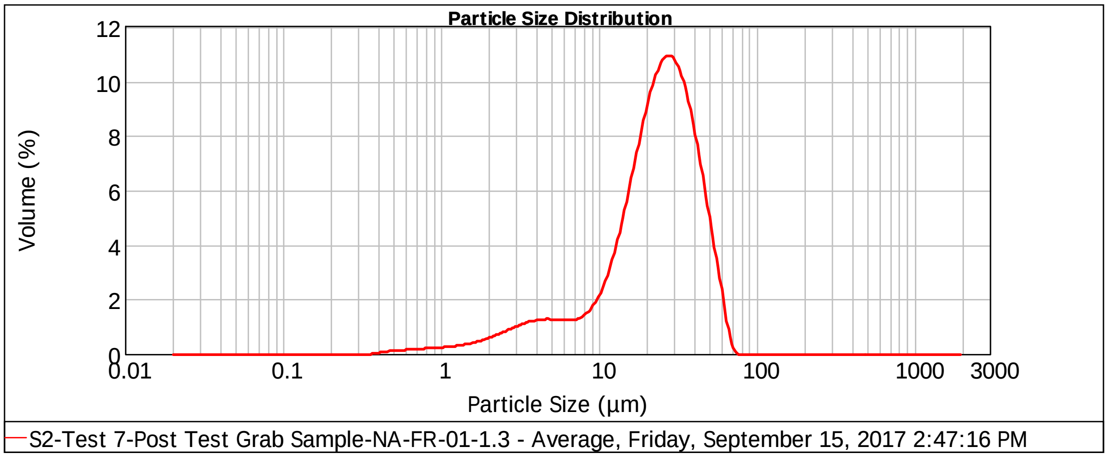
Sample Name: S2-Test 7-Post Test Grab Sample-NA-FR-01-1.3-Average	**SOP Name:**	**Measured:** Friday, September 15, 2017 2:47:16 PM
Sample Source & type:	**Measured by:** D3M966	**Analysed:** Friday, September 15, 2017 2:47:18 PM
Sample bulk lot ref:	**Result Source:** Averaged	

Particle Name: Resin	**Accessory Name:** Hydro 2000μP (A)	**Analysis model:** General purpose	**Sensitivity:** Normal
Particle RI: 1.600	**Absorption:** 1	**Size range:** 0.020 to 2000.000 um	**Obscuration:** 11.07 %
Dispersant Name: Water	**Dispersant RI:** 1.330	**Weighted Residual:** 1.769 %	**Result Emulation:** Off

---

Concentration: 0.0297 %Vol	**Span :** 1.620	**Uniformity:** 0.475	**Result units:** Volume
Specific Surface Area: 0.53 m²/g	**Surface Weighted Mean D[3,2]:** 11.325 um	**Vol. Weighted Mean D[4,3]:** 25.229 um	

d(0.1): **5.897** **um** **d(0.5):** **24.006** **um** **d(0.9):** **44.775** **um**



S2-Test 7-Post Test Grab Sample-NA-FR-01-1.3 - Average, Friday, September 15, 2017 2:47:16 PM

Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %
0.020	0.00	0.142	0.00	1.002	0.19	7.096	0.98	50.238	3.15	355.656	0.00
0.022	0.00	0.159	0.00	1.125	0.21	7.962	1.12	56.368	1.83	399.052	0.00
0.025	0.00	0.178	0.00	1.262	0.24	8.934	1.39	63.246	0.56	447.744	0.00
0.028	0.00	0.200	0.00	1.416	0.28	10.024	1.83	70.963	0.01	502.377	0.00
0.032	0.00	0.224	0.00	1.589	0.33	11.247	2.45	79.621	0.00	563.677	0.00
0.036	0.00	0.252	0.00	1.783	0.40	12.619	3.25	89.337	0.00	632.456	0.00
0.040	0.00	0.283	0.00	2.000	0.47	14.159	4.21	100.237	0.00	709.627	0.00
0.045	0.00	0.317	0.00	2.244	0.56	15.887	5.25	112.468	0.00	796.214	0.00
0.050	0.00	0.356	0.01	2.518	0.66	17.825	6.29	126.191	0.00	893.367	0.00
0.056	0.00	0.399	0.05	2.825	0.75	20.000	7.19	141.589	0.00	1002.374	0.00
0.063	0.00	0.448	0.08	3.170	0.84	22.440	7.88	158.866	0.00	1124.683	0.00
0.071	0.00	0.502	0.09	3.557	0.91	25.179	8.22	178.250	0.00	1261.915	0.00
0.080	0.00	0.564	0.11	3.991	0.95	28.251	8.16	200.000	0.00	1415.892	0.00
0.089	0.00	0.632	0.13	4.477	0.96	31.698	7.70	224.404	0.00	1588.656	0.00
0.100	0.00	0.710	0.14	5.024	0.95	35.566	6.86	251.785	0.00	1782.502	0.00
0.112	0.00	0.796	0.15	5.637	0.94	39.905	5.73	282.508	0.00	2000.000	
0.126	0.00	0.893	0.17	6.325	0.93	44.774	4.45	316.979	0.00		
0.142		1.002		7.096		50.238		355.656			

Operator notes: *tap water carrier fluid, Solids taken from Poast S2, Test 7 Simulant (3/14/2017) contains sRF, Fe, HEDTA*

Result Analysis Report

Sample Name:
S2-Test2-Post Test Grab Sample-SP-01-1.1-Average

SOP Name:

Measured:
Friday, September 15, 2017 10:33:42 AM

Sample Source & type:

Measured by:
D3M966

Analysed:
Thursday, September 21, 2017 4:13:51 PM

Sample bulk lot ref:

Result Source:
Averaged

Particle Name:
Sodium Metaphosphate

Accessory Name:
Hydro 2000μP (A)

Analysis model:
General purpose

Sensitivity:
Normal

Particle RI:
1.600

Absorption:
0.001

Size range:
0.020 to 2000.000 um

Obscuration:
11.97 %

Dispersant Name:
Water

Dispersant RI:
1.330

Weighted Residual:
1.704 %

Result Emulation:
Off

Concentration:
0.0441 %Vol

Span :
5.516

Uniformity:
1.58

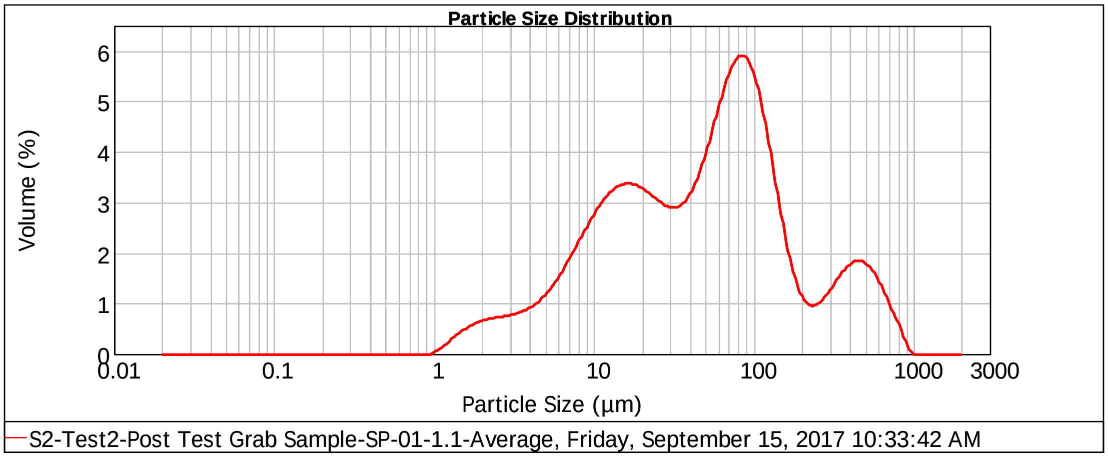
Result units:
Volume

Specific Surface Area:
0.359 m²/g

Surface Weighted Mean D[3,2]:
16.710 um

Vol. Weighted Mean D[4,3]:
102.668 um

d(0.1): 7.035 um **d(0.5): 52.485 um** **d(0.9): 296.519 um**



Size (μm)	Volume In %
0.020	0.00
0.022	0.00
0.025	0.00
0.028	0.00
0.032	0.00
0.036	0.00
0.040	0.00
0.045	0.00
0.050	0.00
0.056	0.00
0.063	0.00
0.071	0.00
0.080	0.00
0.089	0.00
0.100	0.00
0.112	0.00
0.126	0.00
0.142	

Size (μm)	Volume In %
0.142	0.00
0.159	0.00
0.178	0.00
0.200	0.00
0.224	0.00
0.252	0.00
0.283	0.00
0.317	0.00
0.356	0.00
0.399	0.00
0.448	0.00
0.502	0.00
0.564	0.00
0.632	0.00
0.710	0.00
0.796	0.00
0.893	0.00
1.002	

Size (μm)	Volume In %
1.002	0.06
1.125	0.14
1.262	0.26
1.416	0.34
1.589	0.41
1.783	0.47
2.000	0.51
2.244	0.54
2.518	0.55
2.825	0.58
3.170	0.61
3.557	0.66
3.991	0.73
4.477	0.83
5.024	0.96
5.637	1.13
6.325	1.33
7.096	

Size (μm)	Volume In %
7.096	1.54
7.962	1.76
8.934	1.98
10.024	2.17
11.247	2.33
12.619	2.45
14.159	2.52
15.887	2.54
17.825	2.50
20.000	2.43
22.440	2.33
25.179	2.24
28.251	2.18
31.698	2.19
35.566	2.28
39.905	2.49
44.774	2.79
50.238	

Size (μm)	Volume In %
50.238	3.19
56.368	3.62
63.246	4.03
70.963	4.33
79.621	4.44
89.337	4.33
100.237	3.98
112.468	3.41
126.191	2.73
141.589	2.03
158.866	1.43
178.250	1.01
200.000	0.77
224.404	0.71
251.785	0.79
282.508	0.94
316.979	1.12
355.656	

Size (μm)	Volume In %
355.656	1.28
399.052	1.38
447.744	1.38
502.377	1.29
563.677	1.12
632.456	0.87
709.627	0.60
796.214	0.35
893.367	0.05
1002.374	0.00
1124.683	0.00
1261.915	0.00
1415.892	0.00
1588.656	0.00
1782.502	0.00
2000.000	

Operator notes: *Average of 3 measurements from CUF-2017.mea*

MALVERN INSTRUMENTS

MASTERSIZER 2000

Result Analysis Report

Sample Name:
S2-Test2-Post Test Grab Sample-SP-01-1.2-Average

SOP Name:

Measured:
Friday, September 15, 2017 10:35:55 AM

Sample Source & type:

Measured by:
D3M966

Analysed:
Thursday, September 21, 2017 4:13:53 PM

Sample bulk lot ref:

Result Source:
Averaged

Particle Name:
Sodium Metaphosphate

Accessory Name:
Hydro 2000µP (A)

Analysis model:
General purpose

Sensitivity:
Normal

Particle RI:
1.600

Absorption:
0.001

Size range:
0.020 to 2000.000 um

Obscuration:
12.81 %

Dispersant Name:
Water

Dispersant RI:
1.330

Weighted Residual:
1.689 %

Result Emulation:
Off

Concentration:
0.0398 %Vol

Span :
3.957

Uniformity:
1.75

Result units:
Volume

Specific Surface Area:
0.428 m²/g

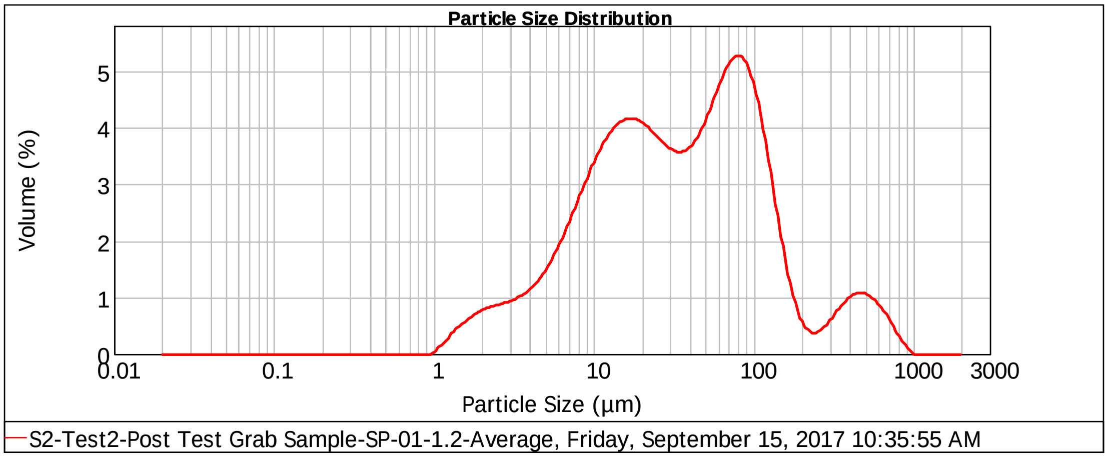
Surface Weighted Mean D[3,2]:
14.013 um

Vol. Weighted Mean D[4,3]:
73.808 um

d(0.1): 5.957 um

d(0.5): 34.017 um

d(0.9): 140.566 um



Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %
0.020	0.00	0.142	0.00	1.002	0.08	7.096	1.91	50.238	3.25	355.656	0.71
0.022	0.00	0.159	0.00	1.125	0.17	7.962	2.18	56.368	3.53	399.052	0.79
0.025	0.00	0.178	0.00	1.262	0.31	8.934	2.43	63.246	3.78	447.744	0.82
0.028	0.00	0.200	0.00	1.416	0.40	10.024	2.66	70.963	3.95	502.377	0.78
0.032	0.00	0.224	0.00	1.589	0.49	11.247	2.86	79.621	3.95	563.677	0.67
0.036	0.00	0.252	0.00	1.783	0.56	12.619	3.01	89.337	3.77	632.456	0.53
0.040	0.00	0.283	0.00	2.000	0.60	14.159	3.10	100.237	3.38	709.627	0.36
0.045	0.00	0.317	0.00	2.244	0.64	15.887	3.13	112.468	2.81	796.214	0.17
0.050	0.00	0.356	0.00	2.518	0.67	17.825	3.11	126.191	2.17	893.367	0.04
0.056	0.00	0.399	0.00	2.825	0.70	20.000	3.04	141.589	1.51	1002.374	0.00
0.063	0.00	0.448	0.00	3.170	0.75	22.440	2.94	158.866	0.97	1124.683	0.00
0.071	0.00	0.502	0.00	3.557	0.82	25.179	2.83	178.250	0.57	1261.915	0.00
0.080	0.00	0.564	0.00	3.991	0.91	28.251	2.73	200.000	0.35	1415.892	0.00
0.089	0.00	0.632	0.00	4.477	1.04	31.698	2.68	224.404	0.28	1588.656	0.00
0.100	0.00	0.710	0.00	5.024	1.21	35.566	2.70	251.785	0.32	1782.502	0.00
0.112	0.00	0.796	0.00	5.637	1.42	39.905	2.81	282.508	0.44	2000.000	
0.126	0.00	0.893	0.00	6.325	1.66	44.774	2.99	316.979	0.58		
0.142		1.002		7.096		50.238		355.656			

Operator notes: *Average of 3 measurements from CUF-2017.mea*

MASTERSIZER 2000

Result Analysis Report

Sample Name:
S2-Test2-Post Test Grab Sample-SP-01-1.3-Average

SOP Name:

Measured:
Friday, September 15, 2017 10:37:39 AM

Sample Source & type:

Measured by:
D3M966

Analysed:
Thursday, September 21, 2017 4:13:54 PM

Sample bulk lot ref:

Result Source:
Averaged

Particle Name:
Sodium Metaphosphate

Accessory Name:
Hydro 2000μP (A)

Analysis model:
General purpose

Sensitivity:
Normal

Particle RI:
1.600

Absorption:
0.001

Size range:
0.020 to 2000.000 um

Obscuration:
12.61 %

Dispersant Name:
Water

Dispersant RI:
1.330

Weighted Residual:
1.571 %

Result Emulation:
Off

Concentration:
0.0415 %Vol

Span :
3.581

Uniformity:
1.45

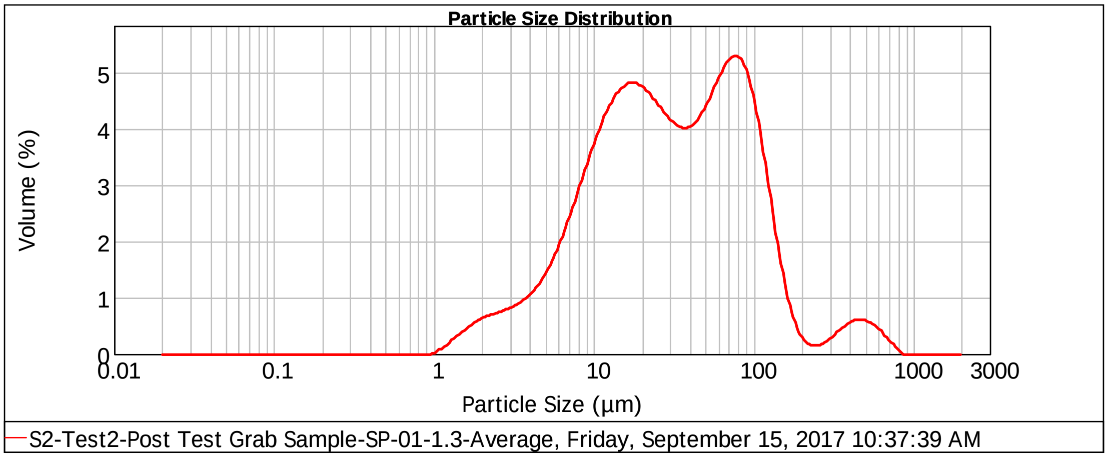
Result units:
Volume

Specific Surface Area:
0.414 m²/g

Surface Weighted Mean D[3,2]:
14.500 um

Vol. Weighted Mean D[4,3]:
56.020 um

d(0.1): 6.513 um **d(0.5): 29.326 um** **d(0.9): 111.536 um**



Size (μm)	Volume In %
0.020	0.00
0.022	0.00
0.025	0.00
0.028	0.00
0.032	0.00
0.036	0.00
0.040	0.00
0.045	0.00
0.050	0.00
0.056	0.00
0.063	0.00
0.071	0.00
0.080	0.00
0.089	0.00
0.100	0.00
0.112	0.00
0.126	0.00
0.142	

Size (μm)	Volume In %
0.142	0.00
0.159	0.00
0.178	0.00
0.200	0.00
0.224	0.00
0.252	0.00
0.283	0.00
0.317	0.00
0.356	0.00
0.399	0.00
0.448	0.00
0.502	0.00
0.564	0.00
0.632	0.00
0.710	0.00
0.796	0.00
0.893	0.00
1.002	

Size (μm)	Volume In %
1.002	0.04
1.125	0.10
1.262	0.22
1.416	0.30
1.589	0.38
1.783	0.45
2.000	0.50
2.244	0.54
2.518	0.57
2.825	0.61
3.170	0.67
3.557	0.75
3.991	0.85
4.477	1.00
5.024	1.19
5.637	1.43
6.325	1.71
7.096	

Size (μm)	Volume In %
7.096	2.01
7.962	2.34
8.934	2.66
10.024	2.96
11.247	3.23
12.619	3.43
14.159	3.57
15.887	3.63
17.825	3.61
20.000	3.54
22.440	3.41
25.179	3.27
28.251	3.14
31.698	3.05
35.566	3.02
39.905	3.08
44.774	3.22
50.238	

Size (μm)	Volume In %
50.238	3.43
56.368	3.67
63.246	3.88
70.963	3.99
79.621	3.94
89.337	3.66
100.237	3.17
112.468	2.52
126.191	1.82
141.589	1.16
158.866	0.65
178.250	0.32
200.000	0.16
224.404	0.11
251.785	0.13
282.508	0.20
316.979	0.30
355.656	

Size (μm)	Volume In %
355.656	0.38
399.052	0.45
447.744	0.46
502.377	0.42
563.677	0.35
632.456	0.22
709.627	0.13
796.214	0.01
893.367	0.00
1002.374	0.00
1124.683	0.00
1261.915	0.00
1415.892	0.00
1588.656	0.00
1782.502	0.00
2000.000	

Operator notes: *Average of 3 measurements from CUF-2017.mea*

MASTERSIZER 2000

Result Analysis Report

Sample Name:
Iron (III) Phosphate Tetrahydrate-1.1 -

SOP Name:

Measured:
Thursday, June 01, 2017 9:04:38 AM

Sample Source & type:

Measured by:
D3M966

Analysed:
Thursday, June 01, 2017 9:04:39 AM

Sample bulk lot ref:

Result Source:
Averaged

Particle Name:
Iron

Accessory Name:
Hydro 2000G (A)

Analysis model:
General purpose (spherical)

Sensitivity:
Normal

Particle RI:
2.860

Absorption:
1

Size range:
0.020 to 2000.000 um

Obscuration:
18.05 %

Dispersant Name:
Water

Dispersant RI:
1.330

Weighted Residual:
0.167 %

Result Emulation:
Off

Concentration:
0.0421 %Vol

Span :
3.111

Uniformity:
1.12

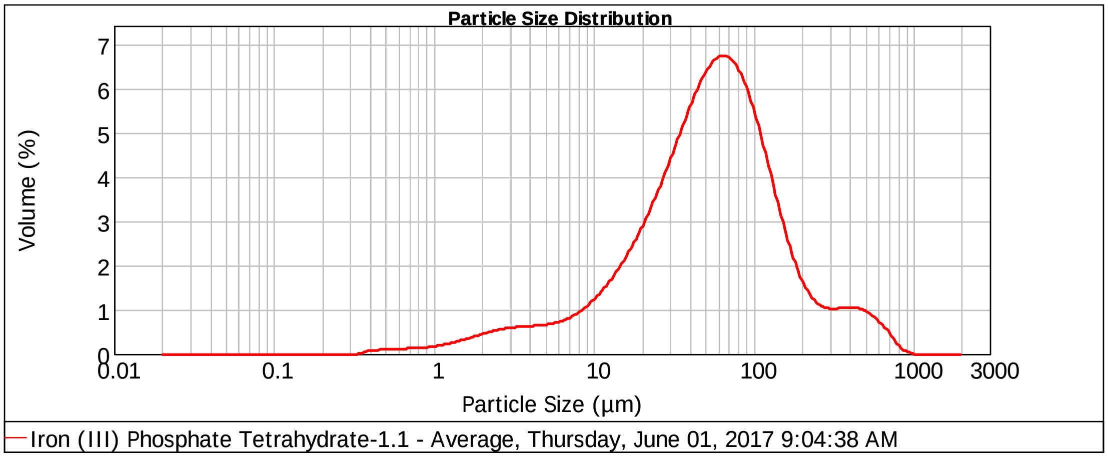
Result units:
Volume

Specific Surface Area:
0.35 m²/g

Surface Weighted Mean D[3,2]:
17.167 um

Vol. Weighted Mean D[4,3]:
88.253 um

d(0.1): 10.630 um **d(0.5): 54.940 um** **d(0.9): 181.567 um**



Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %
0.020	0.00	0.142	0.00	1.002	0.14	7.096	0.66	50.238	4.91	355.656	0.79
0.022	0.00	0.159	0.00	1.125	0.16	7.962	0.75	56.368	5.05	399.052	0.79
0.025	0.00	0.178	0.00	1.262	0.20	8.934	0.86	63.246	5.08	447.744	0.76
0.028	0.00	0.200	0.00	1.416	0.24	10.024	1.00	70.963	4.98	502.377	0.69
0.032	0.00	0.224	0.00	1.589	0.28	11.247	1.16	79.621	4.75	563.677	0.57
0.036	0.00	0.252	0.00	1.783	0.32	12.619	1.34	89.337	4.39	632.456	0.44
0.040	0.00	0.283	0.00	2.000	0.36	14.159	1.54	100.237	3.94	709.627	0.26
0.045	0.00	0.317	0.00	2.244	0.39	15.887	1.77	112.468	3.41	796.214	0.08
0.050	0.00	0.356	0.04	2.518	0.42	17.825	2.02	126.191	2.87	893.367	0.02
0.056	0.00	0.399	0.06	2.825	0.45	20.000	2.30	141.589	2.32	1002.374	0.00
0.063	0.00	0.448	0.07	3.170	0.46	22.440	2.60	158.866	1.84	1124.683	0.00
0.071	0.00	0.502	0.08	3.557	0.47	25.179	2.93	178.250	1.43	1261.915	0.00
0.080	0.00	0.564	0.09	3.991	0.48	28.251	3.29	200.000	1.13	1415.892	0.00
0.089	0.00	0.632	0.09	4.477	0.49	31.698	3.66	224.404	0.92	1588.656	0.00
0.100	0.00	0.710	0.10	5.024	0.51	35.566	4.03	251.785	0.81	1782.502	0.00
0.112	0.00	0.796	0.11	5.637	0.54	39.905	4.37	282.508	0.77	2000.000	
0.126	0.00	0.893	0.12	6.325	0.59	44.774	4.68	316.979	0.77		
0.142		1.002		7.096		50.238		355.656			

Operator notes: *Dispersed solid material in water first then added to Hydro G*

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INSTRUMENTS

MASTERSIZER 2000

Result Analysis Report

Sample Name:
Iron (III) Phosphate Tetrahydrate-1.2 -

SOP Name:

Measured:
Thursday, June 01, 2017 9:06:16 AM

Sample Source & type:

Measured by:
D3M966

Analysed:
Thursday, June 01, 2017 9:06:17 AM

Sample bulk lot ref:

Result Source:
Averaged

Particle Name:
Iron

Accessory Name:
Hydro 2000G (A)

Analysis model:
General purpose (spherical)

Sensitivity:
Normal

Particle RI:
2.860

Absorption:
1

Size range:
0.020 to 2000.000 um

Obscuration:
26.16 %

Dispersant Name:
Water

Dispersant RI:
1.330

Weighted Residual:
0.217 %

Result Emulation:
Off

Concentration:
0.0349 %Vol

Span :
2.472

Uniformity:
0.841

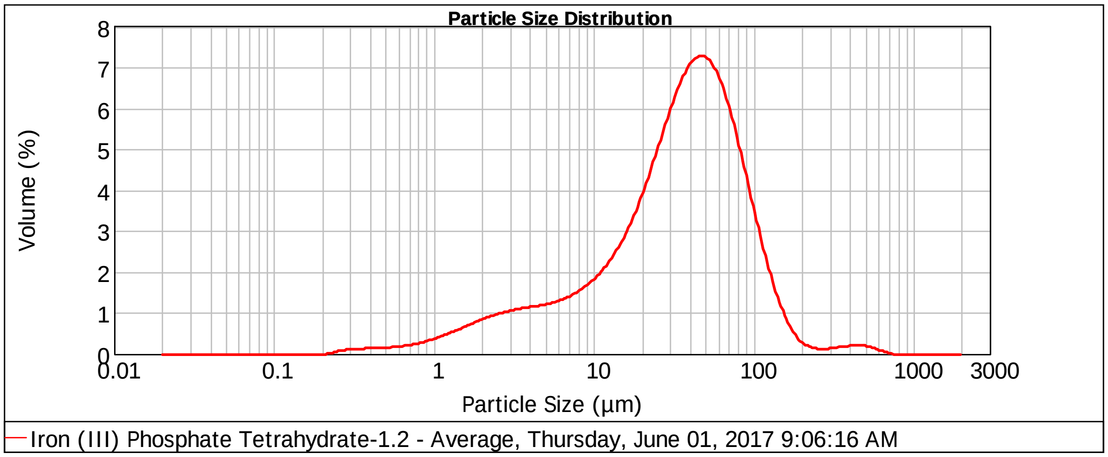
Result units:
Volume

Specific Surface Area:
0.619 m²/g

Surface Weighted Mean D[3,2]:
9.695 um

Vol. Weighted Mean D[4,3]:
47.130 um

d(0.1): 4.588 um **d(0.5): 36.090 um** **d(0.9): 93.795 um**



Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %
0.020	0.00	0.142	0.00	1.002	0.30	7.096	1.10	50.238	5.40	355.656	0.14
0.022	0.00	0.159	0.00	1.125	0.36	7.962	1.20	56.368	5.15	399.052	0.16
0.025	0.00	0.178	0.00	1.262	0.42	8.934	1.31	63.246	4.75	447.744	0.15
0.028	0.00	0.200	0.00	1.416	0.48	10.024	1.45	70.963	4.23	502.377	0.14
0.032	0.00	0.224	0.03	1.589	0.54	11.247	1.62	79.621	3.64	563.677	0.08
0.036	0.00	0.252	0.06	1.783	0.60	12.619	1.82	89.337	3.00	632.456	0.04
0.040	0.00	0.283	0.08	2.000	0.66	14.159	2.07	100.237	2.37	709.627	0.00
0.045	0.00	0.317	0.09	2.244	0.71	15.887	2.37	112.468	1.78	796.214	0.00
0.050	0.00	0.356	0.10	2.518	0.76	17.825	2.72	126.191	1.27	893.367	0.00
0.056	0.00	0.399	0.11	2.825	0.80	20.000	3.12	141.589	0.84	1002.374	0.00
0.063	0.00	0.448	0.11	3.170	0.83	22.440	3.56	158.866	0.52	1124.683	0.00
0.071	0.00	0.502	0.12	3.557	0.85	25.179	4.02	178.250	0.28	1261.915	0.00
0.080	0.00	0.564	0.13	3.991	0.87	28.251	4.47	200.000	0.17	1415.892	0.00
0.089	0.00	0.632	0.15	4.477	0.90	31.698	4.88	224.404	0.11	1588.656	0.00
0.100	0.00	0.710	0.18	5.024	0.93	35.566	5.21	251.785	0.09	1782.502	0.00
0.112	0.00	0.796	0.21	5.637	0.97	39.905	5.42	282.508	0.10	2000.000	
0.126	0.00	0.893	0.25	6.325	1.03	44.774	5.49	316.979	0.12		
0.142		1.002		7.096		50.238		355.656			

Operator notes: *Dispersed solid material in water first then added to Hydro G*

MASTERSIZER 2000

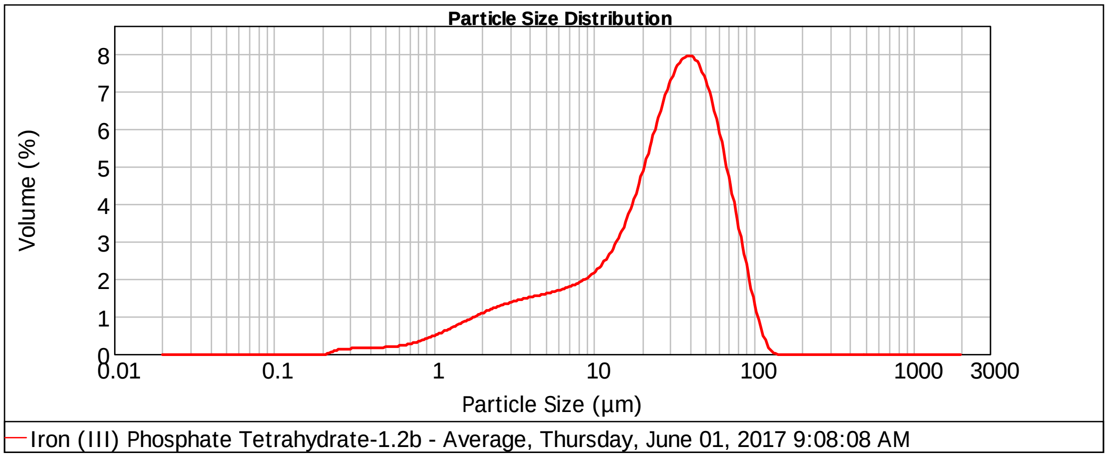
Result Analysis Report

Sample Name: Iron (III) Phosphate Tetrahydrate-1.2b -	**SOP Name:**	**Measured:** Thursday, June 01, 2017 9:08:08 AM
Sample Source & type:	**Measured by:** D3M966	**Analysed:** Thursday, June 01, 2017 9:08:09 AM
Sample bulk lot ref:	**Result Source:** Averaged	

Particle Name: Iron	**Accessory Name:** Hydro 2000G (A)	**Analysis model:** General purpose (spherical)	**Sensitivity:** Normal
Particle RI: 2.860	**Absorption:** 1	**Size range:** 0.020 to 2000.000 um	**Obscuration:** 17.71 %
Dispersant Name: Water	**Dispersant RI:** 1.330	**Weighted Residual:** 0.251 %	**Result Emulation:** Off

Concentration: 0.0178 %Vol	**Span :** 2.211	**Uniformity:** 0.682	**Result units:** Volume
Specific Surface Area: 0.771 m²/g	**Surface Weighted Mean D[3,2]:** 7.786 um	**Vol. Weighted Mean D[4,3]:** 32.111 um	

d(0.1): **3.397** **um** **d(0.5):** **28.385** **um** **d(0.9):** **66.150** **um**



Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %
0.020	0.00	0.142	0.00	1.002	0.40	7.096	1.38	50.238	5.22	355.656	0.00
0.022	0.00	0.159	0.00	1.125	0.47	7.962	1.46	56.368	4.60	399.052	0.00
0.025	0.00	0.178	0.00	1.262	0.55	8.934	1.57	63.246	3.84	447.744	0.00
0.028	0.00	0.200	0.00	1.416	0.62	10.024	1.71	70.963	3.04	502.377	0.00
0.032	0.00	0.224	0.06	1.589	0.70	11.247	1.90	79.621	2.25	563.677	0.00
0.036	0.00	0.252	0.09	1.783	0.78	12.619	2.15	89.337	1.46	632.456	0.00
0.040	0.00	0.283	0.11	2.000	0.85	14.159	2.47	100.237	0.74	709.627	0.00
0.045	0.00	0.317	0.11	2.244	0.92	15.887	2.86	112.468	0.25	796.214	0.00
0.050	0.00	0.356	0.12	2.518	0.98	17.825	3.34	126.191	0.02	893.367	0.00
0.056	0.00	0.399	0.12	2.825	1.03	20.000	3.86	141.589	0.00	1002.374	0.00
0.063	0.00	0.448	0.13	3.170	1.08	22.440	4.42	158.866	0.00	1124.683	0.00
0.071	0.00	0.502	0.14	3.557	1.12	25.179	4.96	178.250	0.00	1261.915	0.00
0.080	0.00	0.564	0.16	3.991	1.15	28.251	5.44	200.000	0.00	1415.892	0.00
0.089	0.00	0.632	0.19	4.477	1.19	31.698	5.79	224.404	0.00	1588.656	0.00
0.100	0.00	0.710	0.22	5.024	1.23	35.566	5.96	251.785	0.00	1782.502	0.00
0.112	0.00	0.796	0.27	5.637	1.27	39.905	5.93	282.508	0.00	2000.000	
0.126	0.00	0.893	0.33	6.325	1.32	44.774	5.69	316.979	0.00		
0.142		1.002		7.096		50.238		355.656			

Operator notes: *Dispersed solid material in water first then added to Hydro G*

MALVERN
INSTRUMENTS

MASTERSIZER 2000

Result Analysis Report

Sample Name:
Iron (III) Phosphate Tetrahydrate-1.2c -

SOP Name:

Measured:
Thursday, June 01, 2017 9:09:56 AM

Sample Source & type:

Measured by:
D3M966

Analysed:
Thursday, June 01, 2017 9:09:58 AM

Sample bulk lot ref:

Result Source:
Averaged

Particle Name:
Iron

Accessory Name:
Hydro 2000G (A)

Analysis model:
General purpose (spherical)

Sensitivity:
Normal

Particle RI:
2.860

Absorption:
1

Size range:
0.020 to 2000.000 um

Obscuration:
19.23 %

Dispersant Name:
Water

Dispersant RI:
1.330

Weighted Residual:
0.335 %

Result Emulation:
Off

Concentration:
0.0168 %Vol

Span :
2.158

Uniformity:
0.667

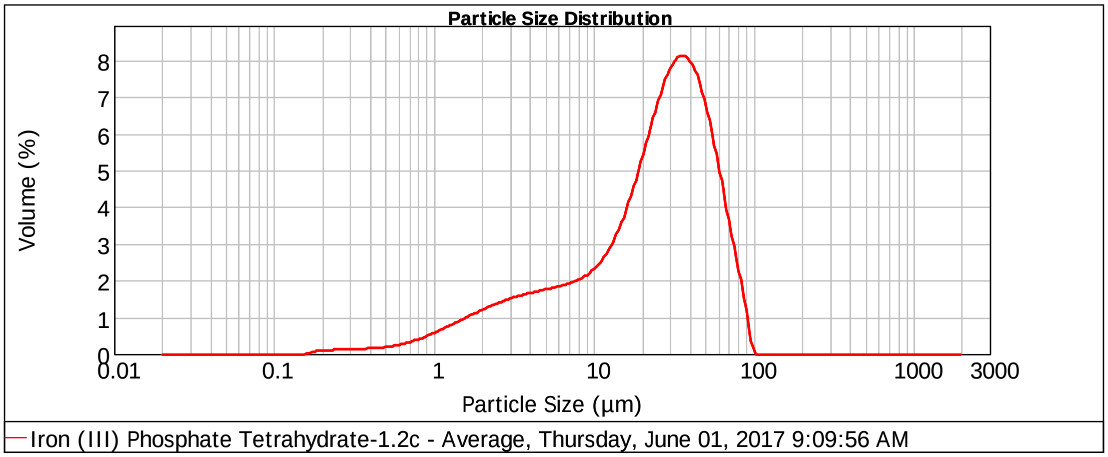
Result units:
Volume

Specific Surface Area:
0.881 m²/g

Surface Weighted Mean D[3,2]:
6.808 um

Vol. Weighted Mean D[4,3]:
28.306 um

d(0.1): **2.989** **um**   **d(0.5):** **25.513** **um**   **d(0.9):** **58.048** **um**



Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %
0.020	0.00	0.142	0.00	1.002	0.47	7.096	1.48	50.238	4.75	355.656	0.00
0.022	0.00	0.159	0.02	1.125	0.54	7.962	1.56	56.368	3.96	399.052	0.00
0.025	0.00	0.178	0.06	1.262	0.63	8.934	1.67	63.246	3.08	447.744	0.00
0.028	0.00	0.200	0.07	1.416	0.71	10.024	1.82	70.963	2.21	502.377	0.00
0.032	0.00	0.224	0.08	1.589	0.79	11.247	2.04	79.621	1.44	563.677	0.00
0.036	0.00	0.252	0.09	1.783	0.87	12.619	2.33	89.337	0.40	632.456	0.00
0.040	0.00	0.283	0.10	2.000	0.94	14.159	2.71	100.237	0.00	709.627	0.00
0.045	0.00	0.317	0.10	2.244	1.01	15.887	3.17	112.468	0.00	796.214	0.00
0.050	0.00	0.356	0.11	2.518	1.07	17.825	3.72	126.191	0.00	893.367	0.00
0.056	0.00	0.399	0.12	2.825	1.13	20.000	4.30	141.589	0.00	1002.374	0.00
0.063	0.00	0.448	0.13	3.170	1.18	22.440	4.89	158.866	0.00	1124.683	0.00
0.071	0.00	0.502	0.15	3.557	1.23	25.179	5.42	178.250	0.00	1261.915	0.00
0.080	0.00	0.564	0.18	3.991	1.27	28.251	5.84	200.000	0.00	1415.892	0.00
0.089	0.00	0.632	0.22	4.477	1.31	31.698	6.08	224.404	0.00	1588.656	0.00
0.100	0.00	0.710	0.27	5.024	1.34	35.566	6.10	251.785	0.00	1782.502	0.00
0.112	0.00	0.796	0.33	5.637	1.38	39.905	5.88	282.508	0.00	2000.000	
0.126	0.00	0.893	0.39	6.325	1.43	44.774	5.42	316.979	0.00		
0.142		1.002		7.096		50.238		355.656			

Operator notes: *Dispersed solid imaterial n water first then added to Hydro G, diluted sample and measured again-added water*

Result Analysis Report

Sample Name:
Iron (III) Phosphate Tetrahydrate-1.3 -

SOP Name:

Measured:
Thursday, June 01, 2017 9:11:25 AM

Sample Source & type:

Measured by:
D3M966

Analysed:
Thursday, June 01, 2017 9:11:26 AM

Sample bulk lot ref:

Result Source:
Averaged

Particle Name:
Iron

Accessory Name:
Hydro 2000G (A)

Analysis model:
General purpose (spherical)

Sensitivity:
Normal

Particle RI:
2.860

Absorption:
1

Size range:
0.020 to 2000.000 um

Obscuration:
19.81 %

Dispersant Name:
Water

Dispersant RI:
1.330

Weighted Residual:
0.385 %

Result Emulation:
Off

Concentration:
0.0168 %Vol

Span :
2.153

Uniformity:
0.668

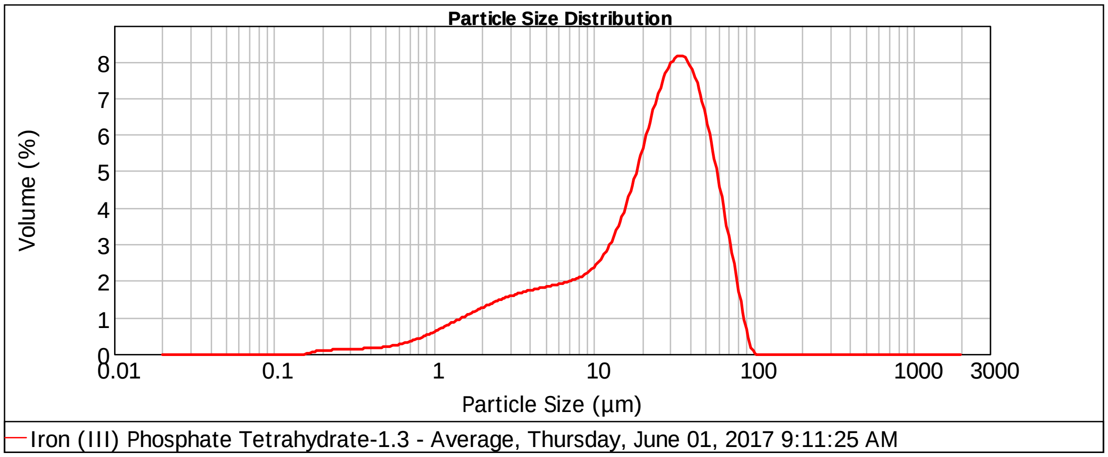
Result units:
Volume

Specific Surface Area:
0.913 m²/g

Surface Weighted Mean D[3,2]:
6.573 um

Vol. Weighted Mean D[4,3]:
27.073 um

d(0.1): 2.853 um **d(0.5): 24.457 um** **d(0.9): 55.516 um**



Size (µm)	Volume In %
0.020	0.00
0.022	0.00
0.025	0.00
0.028	0.00
0.032	0.00
0.036	0.00
0.040	0.00
0.045	0.00
0.050	0.00
0.056	0.00
0.063	0.00
0.071	0.00
0.080	0.00
0.089	0.00
0.100	0.00
0.112	0.00
0.126	0.00
0.142	

Size (µm)	Volume In %
0.142	0.00
0.159	0.02
0.178	0.06
0.200	0.07
0.224	0.08
0.252	0.09
0.283	0.10
0.317	0.11
0.356	0.11
0.399	0.12
0.448	0.14
0.502	0.16
0.564	0.19
0.632	0.23
0.710	0.28
0.796	0.34
0.893	0.41
1.002	

Size (µm)	Volume In %
1.002	0.49
1.125	0.57
1.262	0.66
1.416	0.74
1.589	0.83
1.783	0.91
2.000	0.99
2.244	1.06
2.518	1.13
2.825	1.19
3.170	1.24
3.557	1.29
3.991	1.33
4.477	1.36
5.024	1.40
5.637	1.43
6.325	1.47
7.096	

Size (µm)	Volume In %
7.096	1.52
7.962	1.60
8.934	1.71
10.024	1.87
11.247	2.10
12.619	2.41
14.159	2.81
15.887	3.30
17.825	3.87
20.000	4.46
22.440	5.06
25.179	5.57
28.251	5.95
31.698	6.13
35.566	6.08
39.905	5.77
44.774	5.23
50.238	

Size (µm)	Volume In %
50.238	4.49
56.368	3.65
63.246	2.76
70.963	1.88
79.621	0.97
89.337	0.19
100.237	0.00
112.468	0.00
126.191	0.00
141.589	0.00
158.866	0.00
178.250	0.00
200.000	0.00
224.404	0.00
251.785	0.00
282.508	0.00
316.979	0.00
355.656	

Size (µm)	Volume In %
355.656	0.00
399.052	0.00
447.744	0.00
502.377	0.00
563.677	0.00
632.456	0.00
709.627	0.00
796.214	0.00
893.367	0.00
1002.374	0.00
1124.683	0.00
1261.915	0.00
1415.892	0.00
1588.656	0.00
1782.502	0.00
2000.000	

Operator notes: *Dispersed solid imaterial n water first then added to Hydro G, diluted sample and measured again-added water*

Distribution

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[1] http://www.pnl.gov/main/publications/

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