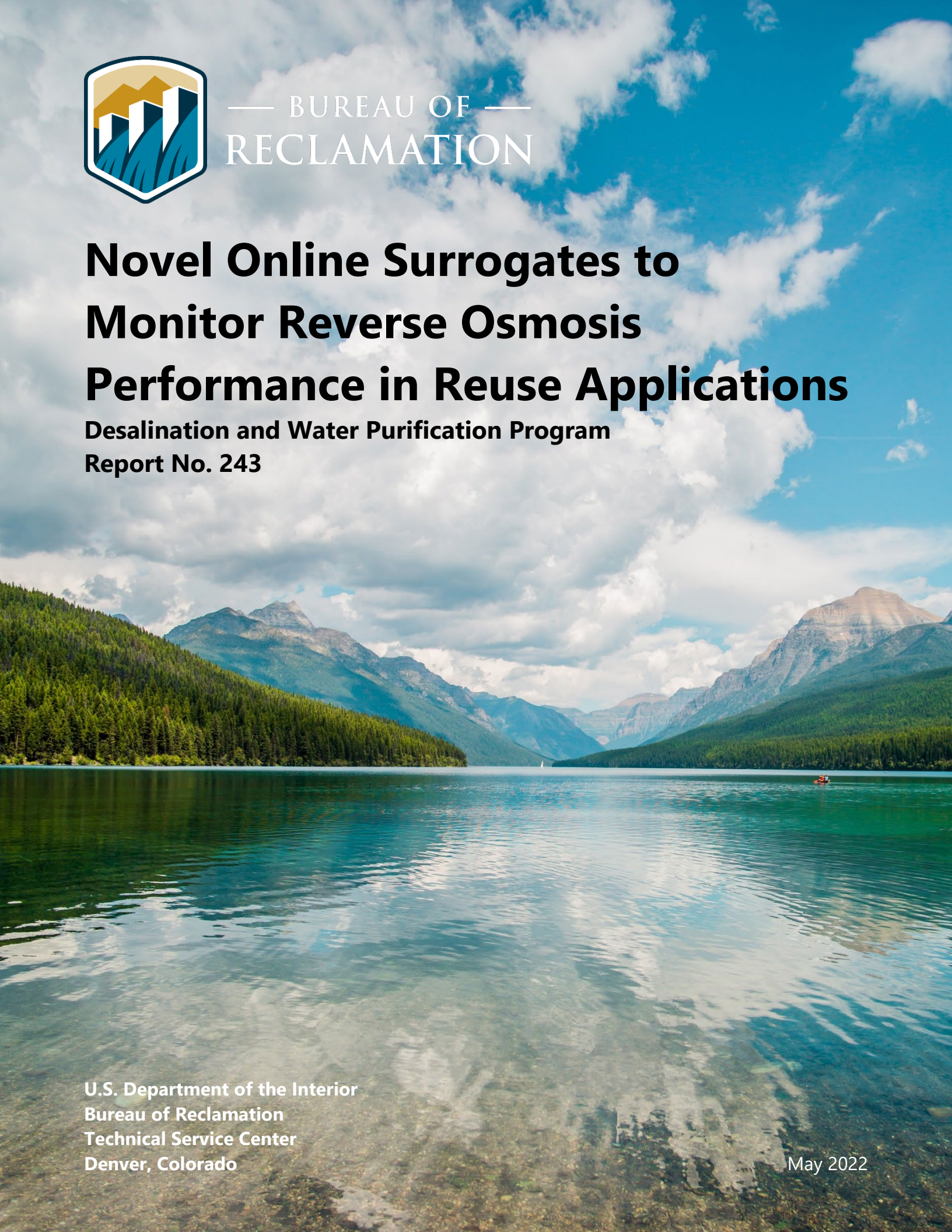

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Novel Online Surrogates to Monitor Reverse Osmosis Performance in Reuse Applications

Desalination and Water Purification Program
Report No. 243

U.S. Department of the Interior
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14. ABSTRACT
This project identified and tested improved surrogates for monitoring reverse osmosis performance for reuse applications that demonstrate greater removal to help bridge the gap between actual performance and the awarded pathogen log removal credits.

15. SUBJECT TERMS
Surrogates, reverse osmosis, log removal, reuse, closed circuit

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Prepared for the Bureau of Reclamation Under Agreement No. R18AC00111

by

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The mission of the Bureau of Reclamation is to manage, develop, and protect water and related resources in an environmentally and economically sound manner in the interest of the American public.

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Acronyms and Abbreviations

Acronym or Abbreviation	Definition
AOP	advanced oxidation process
ATP	adenosine triphosphate
AWP	advanced water purification
AWPF	advanced water purification facility
cATP	cellular adenosine triphosphate
CCD	closed-circuit desalination
CCR	California Code of Regulations
CCRO	closed-ecircuit reverse osmosis
CIIM	continuous indirect integrity monitoring
CIP	clean-in-place
DDW	Division of Drinking Water
DIT	direct integrity test
DOI	Department of the Interior
DPR	direct potable reuse
EC	electrical conductivity
EEA	Eurofins Eaton Analytical
EEM	excitation-emission matrix
EPA	Environmental Protection Agency
fDOM	fluorescent dissolved organic matter
FI	fluorescence index
Free ATP	free adenosine triphosphate
FRI	fluorescence regional integration
GWRS	Groundwater Replenishment System
H_2O_2	hydrogen peroxide
HMI	human machine interface
HPLC	high-performance liquid chromatography
IC	ion chromatography
ICP-MS	inductively coupled plasma-mass spectroscopy
LOD	limit of detection
LRV	log removal value
LT2ESWTR	Long Term 2 Enhanced Surface Water Treatment Rule
MDL	method detection limit
MF	microfiltration
MFGM	Membranes Filtration Guidance Manual
MRL	method reporting limit
MS2	male-specific bacteriophage

Acronym or Abbreviation	Definition
NSF	National Science Foundation
NTA	nanoparticle tracking analyzer
OC San	Orange County Sanitation District
OCWD	Orange County Water District
ORP	oxidation reduction potential
PF	plug flow
PLC	programmable logic controller
Reclamation	Bureau of Reclamation
RO	reverse osmosis
SM	standard methods
TDS	total dissolved solids
TOC	total organic carbon
TMP	transmembrane pressure
UF	ultrafiltration
USEPA	United States Environmental Protection Agency
UV	ultraviolet
UV/H2O2	ultraviolet disinfection with hydrogen peroxide addition
WRF	water reclamation facility
XFR	X-ray fluorescence

Measurements

Acronym or Abbreviation	Definition
°F	degree Fahrenheit
cm	centimeter
Da	dalton
ft^2	square feet
gfd	gallons per square feet per day
gpd	gallons per day
gpm	gallons per minute
mgd	million gallons per day
mg/L	milligrams per liter
nm	nanometer
µg/L	microgram per liter
µm	micrometer

Variables

Symbol or Abbreviation	Definition
C_f	feed concentration of constituent
C_p	filtrate concentration of constituent

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Executive Summary

Reverse osmosis (RO) is a widely accepted treatment technology in reuse scenarios, serving as a physical barrier to pathogens and most dissolved constituents. However, for potable reuse, pathogen removal credit for RO systems is dependent upon proving continuous integrity of the membranes, usually through online monitoring of a surrogate for virus rejection. Traditional surrogates, such as total dissolved solids and total organic carbon, demonstrate 1 to 2 logs of removal credit. These values are much lower than the 6 logs of virus removal demonstrated by RO in past studies. To obtain greater log removal credits, new surrogates are necessary.

This project identified novel naturally occurring surrogates and their online monitoring capabilities to continuously monitor and demonstrate RO integrity for reuse scenarios and to ensure public health is being protected. Part 1 of this project took place at the Orange County Water District (OCWD) Advanced Water Purification Facility (AWPF) in Fountain Valley, California. The AWPF produces high quality recycled water as part of the Groundwater Replenishment System (GWRS), a potable reuse project jointly operated by OCWD and the Orange County Sanitation District (OC San). GWRS is currently the world's largest water reclamation facility for potable reuse and served as a test case representing many other reuse facilities.

At the AWPF, five naturally occurring surrogates, free adenosine triphosphate (free ATP), fluorescence Peak C, sulfate, strontium, and nanoparticles were evaluated as having potential to increase virus removal credits for the RO membrane treatment process by demonstrating RO integrity. Online monitoring was used where online instruments were available. The findings showed that free ATP, fluorescence Peak C, sulfate, and strontium are four possible surrogates that can be used for this purpose which all demonstrated average log removal values (LRVs) that exceed current typical LRVs achieved by use of total organic carbon (TOC) or conductivity. Strontium and sulfate (from grab samples) and free ATP (measured online) are the three naturally occurring surrogates that showed the highest removal by the RO system with average LRVs of 3.28, 2.90, and 3.03 respectively. Online fluorescence Peak C was also noteworthy due to ease of online measurement and low cost; however, it was more conservative, with an average LRV of 2.70. Results for nanoparticle analysis indicated that current instrumentation and related technology are not sensitive enough to detect enough nanoparticles for LRV demonstration purposes and/or the nanoparticle concentration in the RO feedwater and RO permeate is too low.

Part 2 of this project took place at the Padre Dam Municipal Water District (Padre Dam) Advanced Water Purification (AWP) demonstration facility using a closed-circuit reverse osmosis (CCRO) pilot unit operated at 95 percent recovery. The goal of Part 2 was to identify membrane integrity surrogates that demonstrate greater than 2 logs for enhanced pathogen removal credit purposes that still apply for a CCRO system at high recovery; this is in contrast to Part 1, which focused on a conventional RO system. Challenge testing using male-specific bacteriophage (MS2), an accepted surrogate for enteric virus, was also performed as part of Part 2. Strontium rejection was greater than 3.4 logs and provided a conservative estimate of MS2 rejection and was able to accurately track decreases in MS2 rejection for a compromised CCRO

system. Magnesium and sulfate, while well-rejected, were often below the limits of detection by conventional analytical methods. Part 2 showed that the achievable credit for the RO system at 95 percent overall recovery was greater than 3.0 logs. Overall, both sites (Part 1 and Part 2) demonstrated strontium as having the greatest LRV credit potential, while other surrogates were promising and still exceeded TOC or conductivity LRV. As a result of this study, OCWD is pursuing RO LRV credit based on a tiered approach using free ATP, strontium, and sulfate, and Padre Dam will be pursuing a tiered approach using strontium; both agencies will be employing TOC and EC as backup surrogates.

1. Introduction

Reverse osmosis (RO) is a widely accepted treatment technology in reuse scenarios. RO serves as a physical barrier to even the smallest pathogens and most dissolved constituents. Past studies have shown that commercial RO membranes can achieve greater than 6 logs of removal for MS2 bacteriophage, a commonly accepted surrogate for enteric virus (DeCarolis et al. 2006; Adham et al. 1997; Jacangelo et al. 2015). However, there is a discrepancy between actual log removals achieved and the pathogen log removal credit awarded by regulatory agencies. Current potable reuse regulations require ongoing performance monitoring of RO systems to demonstrate membrane integrity and to protect public health. However, real-time monitoring of viruses is not possible with current technologies. Therefore, surrogates are used to confirm removal and demonstrate overall RO system integrity. Traditional surrogates include electrical conductivity (EC), which results in up to 1.5 logs of observed removal (and therefore credits) in brackish water applications including potable reuse, and total organic carbon (TOC), which can result in up to 2 logs of removal credit.

Increasing the log removal credits assigned to the RO system in potable reuse treatment facilities has the following main benefits: (1) increasing confidence, from both the industry and the public, in RO's ability to remove high levels of pathogens; and (2) reducing the burden of pathogen removal credits on the rest of the treatment train, which has significant design implications in terms of operational flexibility, costs, energy use, and footprint.

The principal objective of this project was to identify and test naturally occurring surrogates for monitoring RO performance for reuse that can demonstrate greater removal and, therefore, begin to bridge the gap between actual performance and the awarded pathogen log removal credits. Naturally occurring surrogates are preferable to avoid the complexity and expense of spiking constituents into the feed water to achieve sufficient log removal value (LRV). The project investigated surrogates at two facilities. Part 1 of this Project took place at the Orange County Water District (OCWD) Advanced Water Purification Facility (AWPF) in Fountain Valley, California. The AWPF produces high quality recycled water as part of the Groundwater Replenishment System (GWRS), a potable reuse project jointly operated by OCWD and the Orange County Sanitation District (OC San). GWRS is currently the world's largest water reclamation facility for potable reuse and served as a test case representing many other reuse facilities. Part 2 took place at the Padre Dam Municipal Water District (Padre Dam) Advanced Water Purification (AWP) demonstration facility using a closed-circuit reverse osmosis (CCRO) pilot unit operated at 95 percent overall recovery. Specific objectives of the study were to:

1. Identify the current state of the science on surrogates for pathogen removal through RO;
2. Analyze historical data from the OCWD AWPF to identify the four most promising surrogates;
3. Test each of these novel surrogates for 3 to 6 months at a full-scale reuse facility (OCWD AWPF) to assess their feasibility to replace traditional surrogates; and
4. Demonstrate that novel surrogates are also applicable for a CCRO type system at high RO recovery.

2. Project Background

The following subsections discuss the project needs, describe the overall approach, and provide a summary of the state of the science on surrogates for RO that were evaluated to select four novel surrogates for testing in this study.

2.1. Problem and Needs

One of the key design criteria for potable reuse facilities is the log removal credits that can be assigned to each treatment process. RO has traditionally been and still is under-credited due to the lack of an online, near real-time, or daily grab sample-based monitoring strategy to continuously demonstrate membrane and system integrity at levels close to actual pathogen removals. This under-crediting results in additional infrastructure for treatment processes in order to obtain the total credits required by recycled water facility permits. This is a significant issue for the reuse industry and is getting more attention as the reuse industry begins to regulate and design direct potable reuse (DPR) projects. In California, for example, draft regulations require that DPR facilities must demonstrate at least 20 log reduction for virus (DDW 2021), where only approximately 2 logs are currently obtained by the RO treatment process using EC or TOC.

This project aims to increase the log removal credits awarded to RO in reuse applications by evaluating promising and new online surrogates. For example, the latest increase in log removal credits was 0.5 logs for facilities switching from EC to TOC. The novel surrogates proposed for investigation in this study have the potential to demonstrate 1 to 1.5 additional logs over that which TOC monitoring can provide, which can translate into a major increase in confidence with respect to water quality and cost savings with respect to water treatment. Potable reuse is a rapidly growing practice and is trending towards requirements for higher log removal credits as the physical and psychological connection between wastewater and drinking water becomes closer (i.e., DPR facilities). Additional log removal credits have measurable value in the design and permitting of potable reuse facilities that are extremely protective of public health; with RO being the most common treatment technology used for potable reuse, the impact of increased log removal credits via better and improved monitoring affects all utilities nationwide with interests in pursuing potable reuse.

2.2. Overall Approach

The goal of this study was to identify and test novel surrogates for monitoring RO performance for reuse that can demonstrate greater removal and, therefore, begin to bridge the gap between actual performance and the awarded pathogen log removal credits.

Part 1 of this project focused on the evaluation of online surrogates and indicators for monitoring RO integrity at OCWD's 100 million gallons per day (MGD) AWPF potable reuse

treatment facility in Fountain Valley, CA. Based on a literature review and data query of OCWD's AWPF historical water quality data, four novel surrogates were selected for monitoring and comparison during this study, including free adenosine triphosphate (free ATP), fluorescence Peak C (humic like fluorescent dissolved organic matter (fDOM)), naturally occurring ions sulfate and strontium, and nanoparticles. The selected test surrogates were used to monitor the integrity of one of OCWD's full-scale conventional three-stage 5-MGD RO units equipped with online instruments installed on the feed and permeate side of the RO unit. Grab samples for surrogates without online instrumentation (i.e., sulfate and strontium) were collected using portable autosamplers to sample the feed and permeate. Traditional surrogates (EC and TOC) were measured in parallel.

Part 2 of this project focused on integrity testing for a CCRO system at Padre Dam's AWP demonstration facility. CCRO is a variation on conventional two- or three-stage RO that has potential to increase water recovery by reducing scaling. Testing included three cycle assessments to evaluate the LRVs of different surrogates, including virus challenge test with male-specific bacteriophage (MS2), at different times during a CCRO cycle.

2.3. Current State of Science on Surrogates for RO Integrity Monitoring

This section provides an overview of the current state of the science on surrogates for RO integrity monitoring. This includes an overview of past studies that evaluated surrogates for demonstrating log removal for RO system applications. Additionally, the section includes a brief overview of the regulatory framework focused on current potable reuse regulations and guidance from the Membrane Filtration Guidance Manual (USEPA 2005).

2.3.1. Purpose of Surrogates

Surrogates are used to demonstrate the ability of an RO system to effectively reject pathogens that are of concern for public health. Since RO is a physical barrier that removes contaminants (chemical or microbial) primarily through size exclusion, and the smallest pathogens of concern are viruses, an ideal surrogate must be conservative relative to virus rejection. In addition, an ideal surrogate accurately tracks decreases in virus rejection that may occur if there are compromises or breaches in the RO system. In the context of RO integrity monitoring, a surrogate for virus is not intended to simulate an actual virus (i.e., have the same size, structure, behavior, properties, etc.), but rather to accurately indicate that the RO system is functioning normally (high integrity) and whether any system breach has occurred as indicated by reduced log removal of the surrogate which might in some cases correspond to decreased removal of actual virus.

Figure 1 contrasts the behavior of two theoretical surrogates – poor and good – versus virus rejection with respect to different degrees of compromise. The good surrogate remained conservative to virus rejection during a "no compromise" condition as well as when compromises of different degrees were imposed. In contrast, while the poor surrogate did

remain conservative to virus during the "no compromise" condition, it failed to accurately track the decrease in virus rejection, ultimately overestimating virus rejection itself for a "severe compromise" condition.

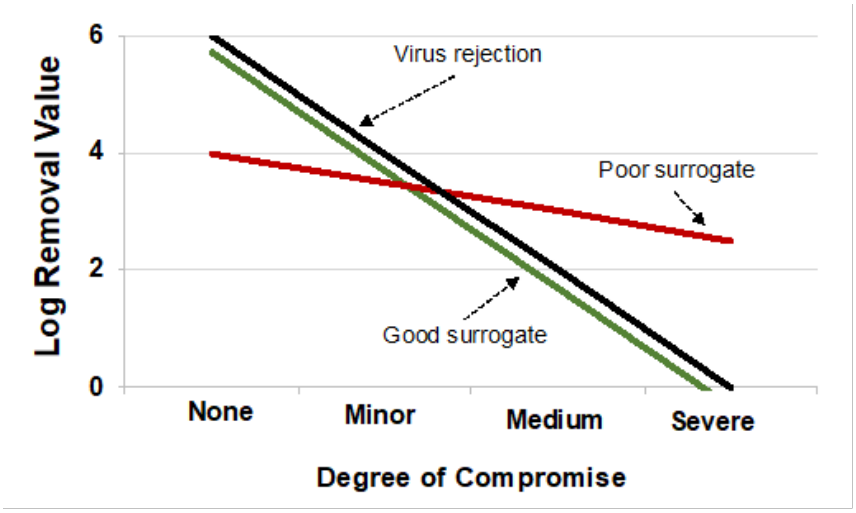


Figure 1. Response of theoretical surrogates to membrane compromises in comparison to virus removal (adapted from Trussell et al. 2017)

In addition to these characteristics, an even better surrogate for RO integrity monitoring demonstrates an overall high degree of log removal, i.e., a high LRV greater than 1.5 (EC) or 2 (TOC) that ideally approximates pathogen rejection yet remains conservative, in order to bridge the gap between actual performance of RO (> ~6 LRV for real virus) and the awarded pathogen log removal credit (currently ~ 2 LRV).

2.3.2. MS2 Rejection through RO

Previous research has demonstrated the ability of RO membranes to effectively reject virus. While not a human pathogen, MS2 (typical size of 25-27 nanometers [nm]) spiked into feed water during challenge testing is the most common method to demonstrate rejection of real virus given its lower cost and safety relative to actual human viruses, its similarities to enteroviruses (Pype et al. 2016a), and its size range relative to the reference pathogen – human enteric viruses (which have a typical size of 28-90 nm; Crittenden et al. 2012). MS2 challenge tests are an accepted form of providing evidence of an RO system's ability to achieve pathogen log reduction reliably and consistently.

These challenge tests are conducted by spiking a known concentration of MS2 to the RO feed water and collecting samples from both the feed and permeate to measure the concentration of MS2 from the sampled locations. Several considerations, such as assay method, presence of feed water disinfectant (e.g., chloramines), phage adsorption, and ensuring sufficient retention time prior to sampling must all be given thought when conducting these labor-intensive challenge tests (Pype et al. 2016b; Trussell et al. 2017; Lozier et al. 2003). MS2 challenge tests are done in lieu of measuring removal of ambient viruses, since there is often not enough naturally occurring virus in the feedwater to measure a permeate value above current detection limits to ascertain meaningful virus removal through RO membranes (Pype et al. 2016a). Table 1 presents a

summary of MS2 LRV results reported in the literature for uncompromised RO membranes. An MS2 LRV of 4 to 5 is common for commercially available RO membranes.

Table 1. Literature summary on MS2 rejection through RO membranes

MS2 Log Removal Value (uncompromised conditions)	Reference
6.7	Madireddi et al. (1997)
3-4.8	Kruithof et al. (2001)
4.0->6	Lozier et al. (2003)
5.4	Mi et al. (2004)
6.2	Steinle-Darling et al. (2015)
4.2->6	Pype et al. (2016a)
4.6->6.2	Antony et al. (2016)
4.6-7.3	Trussell et al. (2017)
5.0-5.4	Vickers (2018)

2.3.3. Verifying Surrogate Performance Through Virus Rejection

As shown in Figure 1, a good surrogate must be conservative to virus during intact conditions. In addition, the figure also indicates that a good surrogate must also accurately track decreases in virus rejection during compromised conditions. Previous research has shown this relationship by sampling surrogates during intact and compromised conditions when MS2 challenge tests are conducted. Jacangelo et al. (2015) evaluated surrogate and MS2 rejection in response to several membrane compromises, including chlorine damage, O-ring breach, and surface scratch. Testing was performed on a pilot-scale RO unit fitted with 4-inch membrane elements. Summarized results are shown in Figure 2. It was found that rejection of the tested surrogates (EC and Rhodamine-WT) showed decreased rejection when most compromises were tested. In contrast, rejection of MS2 was significantly decreased when a physical bulk breach was performed, tested as a cut O-ring in Jacangelo et al. (2015). Other physical breaches, tested as surface scratch and glueline leak, also decreased MS2 rejection.

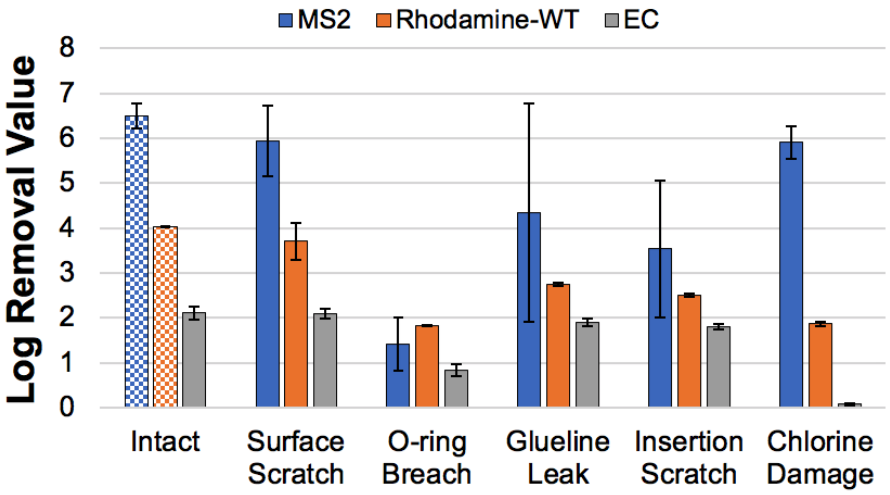


Figure 2. Response of surrogates to membrane compromises (adapted from Jacangelo et al., 2015; error bars represent standard deviation of average removal from two events; checkered bars represent removal limited by detection in permeate sample)

The function of the O-rings is to seal the connection that keeps the feed and permeate streams separate. When an O-ring is compromised, feed water may pass into the permeate stream without passing through the membrane, causing unintentional contamination of the permeate. In contrast, chlorine damage affects the outer polyamide layer of RO membranes, revealing the polysulfone support layer, similar to an ultrafiltration (UF) membrane structure in pore size which is still an effective barrier for virus sized or larger particles (Antony et al. 2016). Indeed, Antony et al. (2016) reported that MS2 rejection by RO was constantly greater than 4 logs even at a decreased EC rejection of 93 percent (Figure 3).

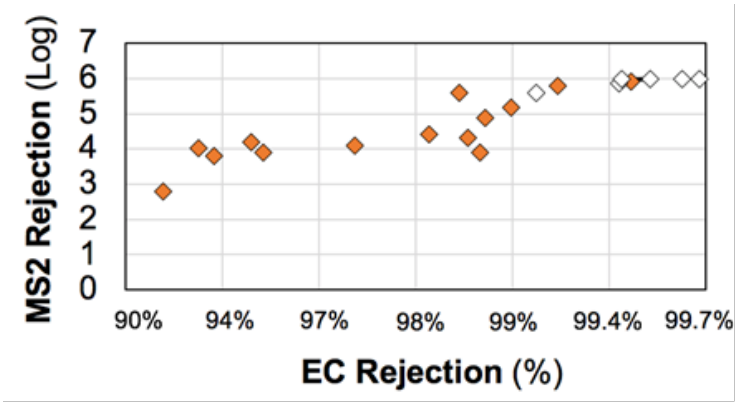


Figure 3. Correlation between EC rejection and MS2 (adapted from Antony et al., 2016; unfilled markers represent removal limited by detection in permeate sample)

Trussell et al. (2017) also evaluated surrogate and MS2 rejection in response to the removal of O-rings for a demonstration-scale 0.5-mgd RO system fitted with 8-inch membrane elements (Figure 4). Overall, past studies have shown that surrogates are sensitive to membrane compromises, particularly those that inflict a concern due to decreased virus rejection. Importantly, the surrogates were shown to be conservative for intact membranes and accurately tracked decrease in virus rejection. In the Trussell et al. (2017) study, O-rings were removed

from different locations (i.e., endcaps and interconnector). Removing O-rings from the feed-side endcap was found to be the most impactful on both surrogate and MS2 rejection. This is likely due to the feed flow being highest at the feed end of an RO vessel. Removing O-rings from other locations did not inflict as much of a compromise to the surrogates as could be expected but did show decreased MS2 rejection beyond what any of the tested surrogates could measure. It is understood that this was because the surrogates were not sufficiently sensitive to capture the decrease in MS2 rejection for those specific compromises. In terms of surrogate performance, the inability to detect the MS2 decrease is considered acceptable since 1) the surrogate remained conservative to MS2 for the conditions tested, and 2) the credit sought for any of the surrogates would be several orders of magnitude lower than the observed decreases in MS2 rejection.

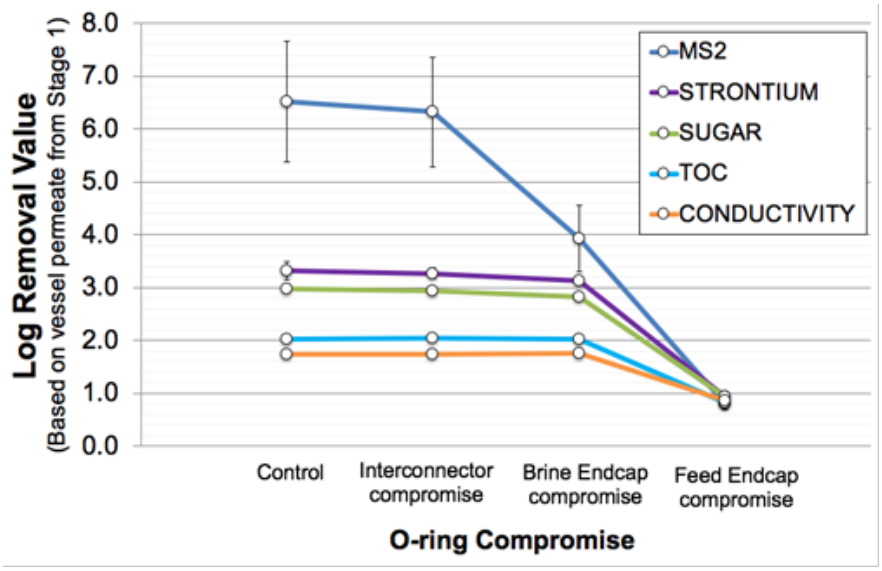


Figure 4. Response of surrogates to O-ring compromises (adapted from Trussell et al., 2017; error bars represent standard deviation of average removal from three events)

2.3.4. Regulatory Framework for Surrogates in Reuse

Per current California indirect potable reuse regulations and draft DPR regulations, the pursuing agency must validate each treatment process and provide evidence of the treatment process's ability to reliably and consistently achieve the log reduction pursued for the process, which can be done using a challenge test approved by the State Board (22 CCR § 60320.208; 22 CCR § 60320.308; DDW, 2021). Specific to the RO treatment process, monitoring should include one form of continuous monitoring, as well as an associated surrogate and/or operational parameter limits and alarm settings that indicate when the integrity has been compromised (22 CCR § 60320.201; 22 CCR § 60320.302). The California Division of Drinking Water (DDW) has approved EC and TOC as acceptable surrogates for pathogen LRV credit for RO systems (22 CCR § 60320.302). Other surrogates, such as strontium, which currently does not have an available online method, have since been proposed (e.g., City of San Diego, 2019 – T22 ER) using a tiered approach that includes EC and TOC as backup surrogates.

Additional regulatory guidance for monitoring the integrity of RO systems is provided in the U.S. Environmental Protection Agency's (EPA) Membrane Filtration Guidance Manual (MFGM) (USEPA 2005). Importantly, the MFGM is expressly directed toward membrane filtration systems seeking to gain Cryptosporidium removal credit that is compliant with the Long Term 2 Enhanced Surface Water Treatment Rule (LT2ESWTR). As such, the MFGM is geared toward membrane systems, including RO, that fall under drinking water regulations. Nonetheless, there are important concepts from the MFGM that are also applicable per current indirect potable reuse regulations to define suitable surrogates. Per the MFGM, a membrane system must demonstrate membrane integrity through two separate tests to receive removal credit for pathogens:

1. Periodic direct integrity testing (DIT); and
2. Continuous indirect integrity monitoring (CIIM).

The EPA Membrane Filtration Guidance Manual defines a DIT as a "physical test applied on each membrane unit and monitored on a daily basis in order to identify and/or isolate integrity breaches" and a CIIM as "monitoring of some aspect of the filtrate water quality […] at a frequency of no less than once every 15 min" such that "a marked decline in filtrate quality may indicate an integrity problem" (USEPA, 2005). In this context, "some aspect" of water quality is interpreted as either a bulk surrogate, such as total dissolved solids (TDS), EC, and TOC, or a molecular marker such as neutral organic and inorganic molecules, and ions.

With the lack of an equivalent for the pressure decay test utilized effectively for low-pressure membrane systems (i.e., micro- and ultra-filtration) and classified as a "physical test," there is currently no such equivalent DIT method for RO. Per the MFGM, the confirmation of integrity using molecular markers can be performed in lieu of a pressure-decay test. As such, regulators have approved online EC and TOC for pathogen LRV credit based on the concept of molecular markers from the MFGM (22 CCR § 60320.302; Bernados 2018). Thus, RO membrane integrity at potable reuse facilities is demonstrated through the monitoring of TOC and/or EC in the feed and permeate.

2.3.5. Summary of Previously Evaluated Surrogates for RO Integrity Monitoring

Several surrogates have been evaluated in other studies for the purpose of RO integrity monitoring. Trussell et al. (2017), Pype et al. (2016b), and Ostarcevic (2018) provide a detailed description of surrogates previously evaluated. For brevity, Table 2 provides a summary of previously evaluated surrogates for RO membrane integrity monitoring. Key differences between the evaluated surrogates are related to cost of implementation, presence of surrogate in feed water (i.e., ambient or spiked), detection limitations, online monitoring capabilities, and proprietary products.

Table 2. Summary of previously evaluated surrogates for RO integrity monitoring (adapted and updated from Trussell et al. 2017)

Surrogate	Ambient or Spiked	Instrumentation and Method	Scale of Previous Testing	Advantages	Drawbacks	LRV Potential	References
EC	Ambient	EC meter (online and/or handheld) SM 2510B	Pilot-, demo-, and full-scale	• Easily implementable • Relatively inexpensive • Fast response • Online monitoring capabilities • Regulatory and industry accepted	• Relatively low LRV • Fluctuates with temperature and membrane condition	Up to 2 logs (1.5-2.0 LRV is typical for reuse applications)	Adham et al. (1998), OCWD (2018), Trussell et al. (2017)
TOC	Ambient	TOC analyzer (online and/or grab samples) SM 5310C	Pilot-, demo-, and full-scale	• Easily implementable • Online monitoring capabilities • Regulatory and industry accepted	• Relatively low LRV • Maintenance of analyzers • High cost of maintenance	Up to 2.5 logs (2.0-2.5 LRV is typical for reuse applications)	Adham et al. (1998), OCWD (2018), Trussell et al. (2017)
Strontium	Ambient	ICP-MS (grab sample) EPA 200.8	Pilot- and demo-scale	• Relatively high LRV • No spiking necessary • Regulatory accepted • Sufficiently sensitive EPA method available	• Rejection affected by membrane aging thus LRV may decrease • Lacks online monitoring capabilities • Dedicated personnel to operate and upkeep instrument • Must have sufficient strontium in feedwater (i.e., plant specific)	Up to 3.5 logs (2.5 – 3.5 LRV is typical for reuse applications)	City of San Diego (2019), Trussell et al. (2017)
Sulfate	Ambient	IC (grab sample) EPA 300.0	Pilot-, demo-, full-scale	• Relatively high LRV • No spiking necessary • Online monitoring capabilities may be available in near future	• LRV limited by detection limit because lacks sufficiently sensitive EPA method • Must have sufficient sulfate in feedwater (i.e., plant specific)	Up to 3.0 logs	Kruithof et al. (2001), Lozier et al. (2016)
Phosphate	Ambient	Phosphate meter (e.g., Hach 5500sc) or off-site analysis	Pilot-, demo-, and full-scale	• Relatively high LRV • No spiking necessary	• LRV limited by detection limit • Must have sufficient phosphate in feedwater (i.e., plant specific)	Up to 3.0 logs	Trussell et al. (2017)

Surrogate	Ambient or Spiked	Instrumentation and Method	Scale of Previous Testing	Advantages	Drawbacks	LRV Potential	References
		EPA 365.1		• Online monitoring capabilities	• May be influenced by membrane scaling		
Fluorescence	Ambient	Fluorometer (e.g., Turner Designs C3 submersible fluorometer)	Pilot- and demo-scale	• Naturally present • Online monitoring capabilities	• Plant specific • No EPA standardized method	Up to 2.7	Pype et al. (2013), Rosario-Ortiz and Korak (2017)
Rhodamine-WT (fluorescent dye)	Spiked	Fluorometer (e.g., Turner Designs C3 submersible fluorometer)	Pilot-, demo-, and full-scale	• High LRV potential • Dye is NSF 60 certified • Online monitoring capabilities • Not proprietary	• Spiking necessary • Dyed concentrate stream • Possible membrane adsorption	Up to 4 logs (3.0-3.5 typical for reuse applications)	Lozier et al. (2003), Jacangelo et al. (2015), Trussell et al. (2017)
3D TRASAR (fluorescent dye)	Spiked	Proprietary fluorometer by Nalco	Pilot- and demo-scale	• High LRV potential • Dye is NSF 60 certified • Online monitoring capabilities • Very low detection limit	• Spiking necessary • Proprietary technology • Potentially too costly for smaller systems	3.4-3.7	Trussell et al. (2017), Steinle-Darling et al. (2015)
Silver nanoparticle	Spiked	ICP-MS (grab sample)	Pilot-scale (UF)	• High LRV potential • Established method for detection	• Spiking necessary • Nanoparticle fabrication process • Costly	Potentially 4 logs	Antony et al. (2014)
Microbial surrogate (e.g., MS2)	Spiked	Microbial assay EPA 1602, Adams 1959 (double layer)	Pilot-, demo-, and full-scale	• High LRV potential • Similar characteristics to enteric virus	• Spiking necessary • Costly • Possible need to divert flows during testing • Labor intensive	Above 6 logs (4.0-5.0 typical for reuse applications)	See Table 1

ICP-MS = inductively coupled plasma-mass spectroscopy; IC = ion chromatography; NSF = National Science Foundation; SM = Standard Methods; EPA = Environmental Protection Agency; TOC = total organic carbon; LRV = log removal value

3. Technical Approach and Methods

3.1. Testing Facilities

The following subsections describe the testing facilities used for this project.

3.1.1. OCWD AWPF

The evaluation of naturally occurring surrogates for monitoring RO performance was performed onsite at OCWD's AWPF in Fountain Valley, California. The AWPF produces high quality recycled water as part of the GWRS, a potable reuse project jointly operated by OCWD and OC San.

GWRS is currently the world's largest water reclamation facility for potable reuse and a recognized industry standard. The GWRS AWPF facility treats secondary-treated wastewater to produce 100 MGD of highly purified water that would otherwise be discharged to the ocean. The treatment train is comprised of microfiltration (MF), RO, ultraviolet disinfection and hydrogen peroxide addition (UV/H2O2) as an advanced oxidation process (UV/AOP), followed by decarbonation and lime stabilization (Figure 5). Chlorine is added before MF to form chloramines, and antiscalant and sulfuric acid are added before RO to control scaling. The AWPF RO process uses three types of membranes, Hydranautics ESPA2-LD, Dupont FilmTec BW30XFRLE, and LG BW400EX in standard 8-inch pressure vessels in different 5-MGD units depending on year of replacement need and bids received. All membranes are thin film polyamide membranes with high flux and high salt rejection. There is a total of 21 RO treatment units at the facility, each with 5-MGD rated capacity, running in parallel to produce the total 100 MGD of RO permeate (one unit is redundant). Each 5-MGD unit features three stages and operates over a range of total recovery from 80 to 85 percent. The finished water is recharged into the local groundwater aquifer (a drinking water source) as opposed to being directly used for drinking water distribution system (i.e., delivery straight to tap, DPR).

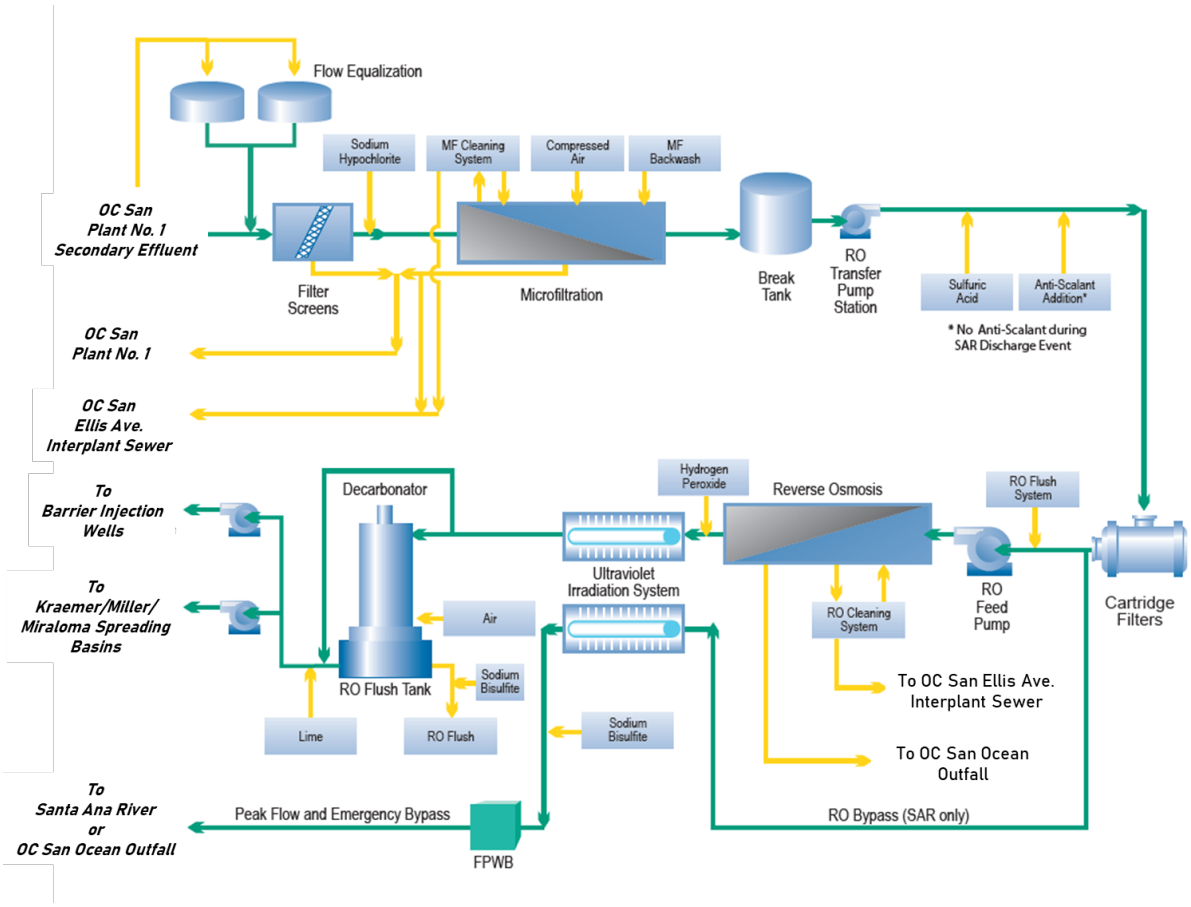


Figure 5. OCWD AWPF GWRS treatment train

The selected test surrogates for this study were primarily monitored on one of the full-scale 5-MGD RO units equipped with the Hydranautics ESPA2-LD RO membranes and operated at 85 percent recovery (Figure 6). For test surrogates with available online instruments (free ATP and fluorescence), the online instrumentation was installed on the feed and permeate sides of the monitored RO unit. Grab samples for surrogates without online instrumentation (strontium and sulfate) were collected using ISCO 3700 portable auto samplers to sample the feed and permeate from the monitored 5-MGD RO unit at preselected time intervals. For nanoparticles, grab samples were collected and analyzed with a bench-top unit, recognizing that an online version of the instrument could potentially be developed should bench data prove promising, based on comments from the manufacturer.

Figure 6. A 5-MGD RO unit at OCWD AWPF equipped with Hydranautics ESPA2-LD membranes

3.1.2. Padre Dam AWP Demonstration Facility

The Padre Dam AWP Demonstration Facility began operation in 2015 to evaluate the performance of advanced treatment unit processes in achieving pathogen control and removal of a suite of regulated and other chemicals of concern to meet California potable reuse requirements. The AWP Demonstration Facility receives a high quality nitrified tertiary effluent from the Ray Stoyer Water Reclamation Facility (WRF). The AWP treatment consists of UF, RO, and UV/AOP. The AWP Demo Facility produces 100,000 gallons per day (gpd) or 70 gallons per minute (gpm) of treated effluent through the RO process with a minimum of 10 gpm treated through UV/AOP.

The AWP Demonstration Facility RO served as the primary RO during testing. The primary RO system consists of skid-mounted equipment (Figure 7) and is designed to produce 100,000 gpd of RO permeate. The system is a conventional two-stage system, operated at 75 percent recovery with a design flux rate of 12 gallons per square foot per day (gfd). During testing, feed water underwent chloramination as well as filtration through the UF process prior to reaching the primary RO. Chloramines were preformed by combining ammonium sulfate (40 percent) and sodium hypochlorite (12.5 percent) in a side-stream of RO permeate. The side stream RO permeate pH was adjusted to approximately 8 pH units using sodium hydroxide (25 percent) prior to injection of the two chemicals. During testing, the chloramine dose was kept between 2 mg/L and 3 mg/L. The demonstration facility contains various monitors to confirm absence of free chlorine, including oxidation reduction potential (ORP) monitors, a free chlorine analyzer, and a free ammonia analyzer. During testing, the first stage of the primary RO consisted of two 8-inch vessels containing six membrane elements each. The second stage is one 8-inch vessel and contained six membrane elements. The operational settings for the primary RO system are summarized in Table 1.

Figure 7. AWP Demonstration Facility primary RO system

Table 3. Primary RO system design criteria

Parameter	Value
Permeate Flow	58 gpm
System Recovery	75%
Feed Flow	77.3 gpm
Concentrate Flow	19.3 gpm
Number of Stages	2
Vessel Array Configuration	2:1
Pressure Vessel Diameter	8 inches
Elements per Pressure Vessel	6
Total Number of Element	18
Membrane Area per Element	400 ft^2
Element Manufacturer	Dow FilmTec
Element Model	BW30XFR
Maximum Average Flux	12 gfd

Sulfuric acid and antiscalant (Avista Vitec 1400) were dosed for pH adjustment and scaling control, respectively. These chemicals were dosed to the feed of the primary RO system using existing chemical injection ports. Sulfuric acid was dosed to achieve a target pH of 6.5 in the primary RO concentrate, and antiscalant (Avista, Vitec 1400) was dosed to achieve a concentration of 2 mg/L in the primary RO feed.

A Desalitech CCRO served as the recovery RO for purposes of testing. A CCRO system uses a recirculation loop that decouples cross-flow velocity from the flow rate through the system.

During a recirculation loop, known as closed-circuit desalination (CCD) mode, feedwater enters the system in the first cycle, producing permeate and recirculating concentrate. As more product water is produced and brine is recirculated, the brine concentration increases. When the system achieves its recovery set point, the concentrate is purged from the system, known as Plug Flow (PF) mode, and the CC begins again. The location of the CCRO system in the process stream relative to the primary RO at the AWP Demonstration Facility is presented in Figure 8, each system being identified by dashed lines. Figure 9 shows example of a containerized CCRO unit used for the study. Permeate of the primary RO and CCRO systems was not physically plumbed to combine the two streams at the time of testing. For the CCRO system, a break feed tank upstream of the CCRO booster pump provided flow equalization due to the semi-batch nature of the CCRO system operation. The operational settings used for the CCRO pilot are provided in Table 4.

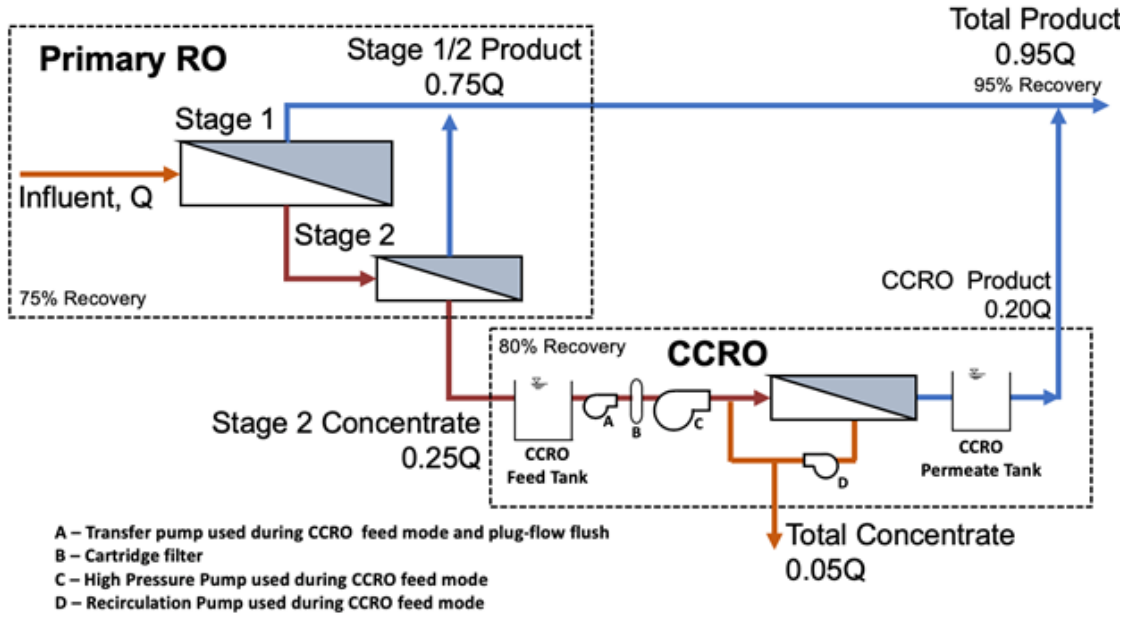


Figure 8 Schematic of the CCRO system relative to primary RO at Padre Dam's Demonstration Facility

Figure 9. Example of containerized CCRO pilot used for study (courtesy of Desalitech)

Table 4. CCRO pilot system design criteria

Parameter	Value
Permeate flow during closed circuit mode	8 gpm
Permeate flow during plug flow mode	≤ 2 gpm
Feed flow during closed circuit mode	8 gpm
Feed flow during plug flow mode	14-25 gpm
Concentrate flow during plug flow mode	12-23 gpm
Concentrate flow during closed circuit mode	0 gpm
Concentrate recirculation flow during closed circuit mode	23-30 gpm
Number of stages	1
Number of pressure vessels	1
Elements per pressure vessel	4 (3 membrane elements + 1 spacer)
Pressure vessel diameter	8 inches
Membrane area per element	400 ft^2
Element manufacturer	Dupont or Hydranautics
Element model	FilmTec BW30XFR-400/34 or ESPA2-LD
Average flux	9 gfd

Note: range represents changes to setpoints as part of troubleshooting activities

3.2. Data Query

A data query of OCWD's historical water quality was performed to identify potential naturally occurring surrogates at OCWD's facility to monitor as part of this study. Specifically, there was interest to monitor ions detected in the permeate above the method reporting limit (MRL) to

ensure confidence in the obtained LRV of the ions. Further, sufficient concentration of the surrogate is necessary in the RO feedwater to achieve greater LRV. The basis of the data query was to screen for and shortlist ions that have shown promise in terms of integrity monitoring as shown in Table 2. The data query of the shortlisted ions is presented in Table 5 with statistics for strontium, phosphate, magnesium, and sulfate.

Table 5. Data query of select naturally occurring ions at OCWD's AWPF

Constituent	Average RO Feed Concentration[a]	Average Calculated LRV[a,b]	N	MRL	Permeate Samples above MRL	Data Range
Strontium	693 ± 97 µg/L	2.79 ± 0.33	6	0.3 µg/L	100%	2017-2018
Sulfate	204 ± 27 mg/L	2.63 ± 0.17	35	0.5 mg/L	20%	2008-2018
Phosphate	0.24 ± 0.36 mg/L as P	1.11 ± 0.36	2	0.01 mg/L as P	0%	2017-2018
Magnesium	25.5 ± 1.6 mg/L	1.71 ± 0.03	63	0.5 mg/L	3%	2008-2020

LRV = log removal value; N = data points; MRL = method reporting limit;
[a] Value reported represents average ± standard deviation
[b] LRV calculated based on paired feed and permeate concentrations. Permeate concentration taken as MRL for events where MRL is reported.

Based on the data query, strontium and sulfate showed the most promise for further monitoring as part of this project. Both constituents showed sufficient feed concentration and with sufficiently sensitive analytical methods to measure permeate concentrations above the MRL. Meeting both of these criteria provides an LRV that is not underestimated by the detection limit. On an average basis, the LRV for strontium and sulfate was 2.79 ± 0.33 and 2.63 ± 0.17, respectively. On the other hand, phosphate and magnesium predominantly had permeate values limited by the MRL, with 0 percent and 3 percent of the permeate samples measured above the MRL, respectively, thus potentially underestimating the LRV for these two surrogates. More sensitive methods (or higher feed concentrations) could potentially allow for a higher LRV for magnesium and phosphate.

3.3. OCWD AWPF Surrogate Testing

Based on the literature review and historical data query, five surrogates were selected for monitoring and comparison during this study. Two of the novel surrogates, free ATP and nanoparticles, have not been previously evaluated. These candidates were identified by OCWD based on prior use of free ATP (via grab samples) for general water quality purposes in the AWPF (e.g., monitoring cartridge filter biogrowth ahead of RO), and on knowledge that an online ATP analyzer measuring both free and cellular ATP had become available just before this study began; OCWD use of online nanoparticle measurement in a 2016-2019 research study focused on MF pre-coagulation (Rajagopalan et.al. 2021).

All selected surrogates are naturally occurring in treated municipal wastewater and in the RO feed water at OCWD. No non-native surrogates were selected since the focus of this study is naturally occurring surrogates to avoid spiking compounds due to cost and operational

complexity of such an approach. Four of the surrogates – free ATP, fluorescence Peak C, strontium, and sulfate – were monitored for 3 to 6 months at one of OCWD's full-scale 5-MGD RO units to assess their feasibility to replace traditional surrogates, i.e., TOC and EC (Table 6). The fifth surrogate, nanoparticles, was tested using a bench-scale nanoparticle analyzer, recognizing that an online version of this instrument could be installed if the bench-scale data was promising. Bench-scale nanoparticle analysis indicated that current instrumentation and technology is not sensitive enough to detect enough nanoparticles for LRV purposes and/or the nanoparticle concentration in the RO feedwater and RO permeate is too low. Therefore, nanoparticles were not monitored for the remainder of the study.

Table 6. Summary of five surrogates monitored at OCWD

Parameter/Surrogate	Free ATP	Fluorescence Peak C	Strontium	Sulfate	Nanoparticles
Sampling Mode	Online	Online and grab samples	Grab samples	Grab Samples	Grab samples
Frequency	30 minutes	Every second (averaged)	Hourly for 24 hours	Hourly for 24 hours	NA
Monitoring Location	(see note)	(see note)	(see note)	(see note)	(see note)
Analyzer/ Method	Hach EZ7300 ATP	Turner Cyclops 7	EPA 200.8	EPA 300.0	Particle-Metrix (ZetaView Multiple Parameters Particle Tracking Analyzer)
Detection limit	0.5 pg/L	0.1 µg/L	0.3 µg/L	0.25 mg/L	10^6 nanoparticles/mL

NA = not applicable
Note: RO feed and RO permeate from same 5-MGD RO unit for all surrogates

The following subsections describe each surrogate and monitoring procedures.

3.3.1. ATP

ATP was evaluated as one of the surrogates at OCWD. ATP is a nucleotide found in all living cells and is therefore useful as a surrogate measure of all active and unculturable microbial cells in a water sample. An ATP measurement provides a better estimate of the total microbial biomass than heterotrophic plate counts, where only a fraction of the total cells can be quantified (Maki et al. 1996; Siebel et al. 2008). The total ATP in a water sample is the sum of cellular ATP (cATP) that is still bound within the living cells plus extra-cellular ATP (free ATP) present in water from dead or lysed cells. ATP's relatively high molecular weight (507 Da) and abundance in the RO feed water made free ATP a potential candidate for use in RO integrity monitoring. Free ATP should be removed by RO membranes which typically reject compounds greater than 150 Da (Ozaki and Li 2002). Some studies have shown that the molecular weight cut-off for certain low-pressure RO membranes, such as the ones used in the AWPF, are closer to 220 Da (Kimura et al. 2003).

For this study, two online ATP-based detection systems using the Hach EZ7300-ATP online analyzer, which measures both cATP and free ATP, were installed onto one of the AWPF's 21 5-MGD RO units. Initially, the two online EZ-ATP analyzers were installed with one on the RO feed and one on RO permeate. Each instrument was calibrated using ATP standard solutions

(Promega Corp). Later, the two EZ-ATP analyzers were replaced with a new dual stream EZ-ATP analyzer that project partner Hach developed for this study; this analyzer was capable of analyzing both RO feed and RO permeate using one instrument. Free ATP measurements were collected every 7 to 30 minutes with a detection limit of 0.5 pg/L.

3.3.2. Fluorescence Peak C (Humic-Like fDOM)

Fluorescence spectroscopy has shown promise as a sensitive and accurate monitoring tool for water quality and process performance, in part because it is a rapid and fairly inexpensive analytical technique that requires no reagents or sample pretreatment. Fluorescence spectroscopy has been identified by the industry as having strong potential for online monitoring of recycled water quality and treatment process performance because it can distinguish between different types of organic carbon and has been shown to be 10 to 1,000 times more sensitive than other commonly used techniques such as UV absorption spectroscopy and high-performance liquid chromatography (HPLC) (Henderson et al. 2009). A previous study by Singh et al. (2012) identified humic-like fDOM (Peak C) in RO permeate as having the greatest potential for evaluating membrane integrity compared to other fDOM fluorescence peaks.

Fluorescing dissolved organic matter (fDOM) is present in wastewater and has a characteristic fluorescence pattern. Fluorescence spectroscopy offers insight into both the quantity and composition of fDOM. The following are key fluorescence signals of fDOM that have been used to distinguish treated wastewater effluents: humic-like (Peak A: λEx/Em = 237-260/400-500 nm; Peak C: λEx/Em = 320-340/410-430 nm and Peak C2: λEx/Em = 370-390/460-480 nm), tyrosine-like (Peak B1: λEx/Em = 225-237/30-321nm and Peak B2: λEx/Em = 248/310 nm) and tryptophan-like (Peak T1: λEx/Em = 275-290/340-360 nm peaks and Peak T2: λEx/Em = 225-237/340-381 nm).

Initially, analysis of RO feed and RO permeate grab samples for humic-like Peak C was performed using an Aqualog benchtop fluorometer from Horiba Scientific (Tokyo, Japan) for an excitation range 240-470 nm and emission range of 280-580 nm. Aqualog supplied software was used to collect fluorescence spectra and processed using a modified fluorescence regional integration (FRI) (Stanford et al. 2011; Gerrity et al. 2011) and the fluorescence index (FI) (McKnight et al., 2001). The excitation-emission matrix (EEM) data was corrected for the Raman Scatter by subtracting emission of the blank and corrected for inner-filter effect (MacDonald et al. 1997). Peak C fluorescence signatures of the RO feed and permeate were extracted and an average LRV of 2.51 was calculated (Figure 10). Following this grab-sample based validation step, where it was confirmed that humic Peak C rejection across the RO membrane could be measured, two Cyclops 7 fluorometers from Turner Designs (San Jose, California) were purchased to monitor online fluorescence in the feed and permeate to determine diurnal fluctuations and long-term variability for LRV monitoring. The Cyclops 7 fluorometers have a Peak C detection limit of 0.5 μg/L.

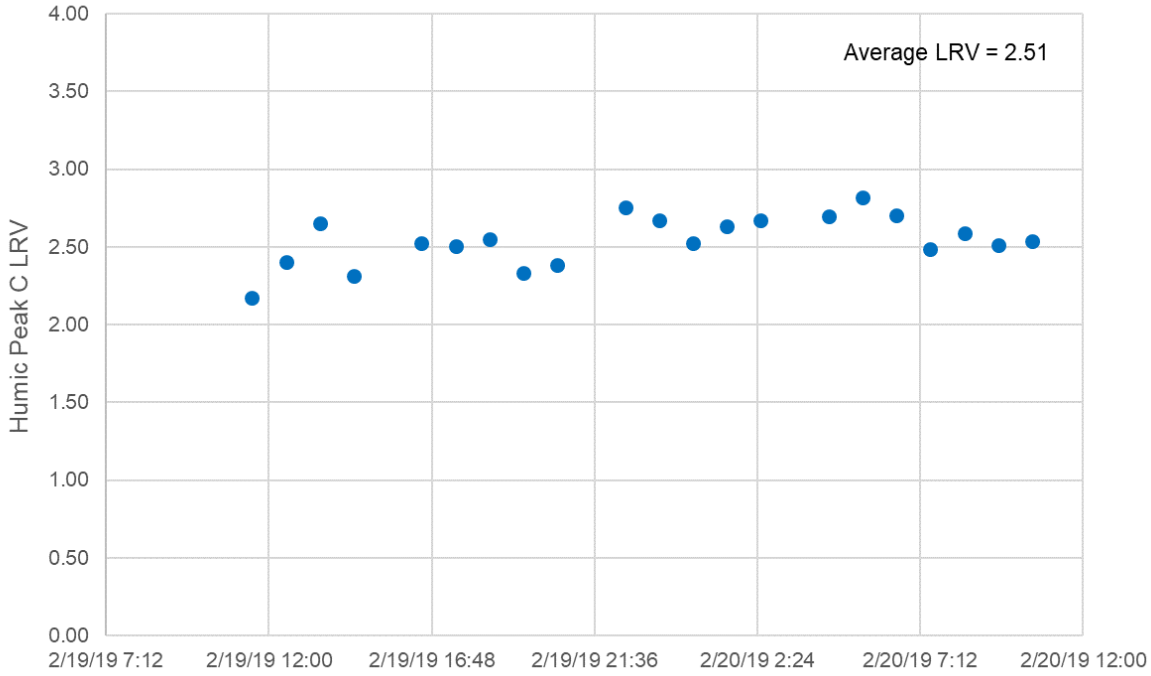


Figure 10. Fluorescence Peak C rejection across a 5-MGD RO unit with ESPA2-LD RO membranes measured using Horiba Scientific Aqualog benchtop fluorometer. Grab samples collected every hour for 24 hours.

3.3.3. Naturally Occurring Ions: Sulfate and Strontium

Similar to ATP, sulfate occurs naturally in treated municipal wastewater and is present at the RO feed water for advanced treatment such that there is no need to "spike" these constituents into the feed. Therefore, sulfate has been previously identified as a potential surrogate for monitoring membrane integrity. A study by Kruithof et al. (2001) observed sulfate reduction from 140 mg/L in the feed to 0.1 mg/L in the permeate, which is an LRV above 3. In the case of OCWD's facility, it is present in AWPF feedwater at 150 to 250 mg/L, which is high enough to quantify significant removal across the RO membranes. Online sulfate measurement can be performed by ion chromatography (IC), but the system is relatively expensive. An alternative method of analysis for monitoring RO integrity is via grab sampling and performing same-day analysis. Even with grab samples, frequency of samples can be high enough to collect adequate data to determine log removal and diurnal fluctuations.

Strontium also occurs naturally in the RO feed water. Its salts are detectable in essentially all drinking waters. In the case of OCWD's facility, it is present in AWPF feedwater at 650 to 750 µg/L, which is high enough to quantify significant removal across the RO membranes. Online strontium analyzers have not yet been validated for this application. Alternatively, like for sulfate, the frequency of grab samples can be high enough to collect adequate data to determine log removal and diurnal fluctuations.

Sulfate and strontium grab samples were collected using ISCO 3700 portable auto samplers. Ion analysis was performed by a subcontracted laboratory (Eurofins Eaton Analytical – Monrovia, California). Sulfate was analyzed using EPA method 300.0 with an MRL of 0.25 mg/L (instrumentation: ion chromatography). Strontium was analyzed using EPA method 200.8 with an MRL of 0.30 µg/L (instrumentation: inductively coupled plasma – mass spectroscopy [ICP-MS]).

Testing for sulfate and strontium focused on five sampling events and took place over a 2-year period. Each sampling event featured multiple, sequential grab samples to understand potential for diurnal variation and to simulate data that would be observed with an online instrument. Four of the sampling events (03/12/2019, 06/12/2019, 02/24/2020, and 09/26/2020) occurred when older ESPA2-LD membranes were still installed in the sampled full-scale RO unit; one sampling event (12/20/2020) was performed after new Dupont FilmTec BW30XFRLE membranes were installed. The membrane change was not driven by this study and was performed as part of normal plant maintenance and operations.

3.3.4. Nanoparticles

The use of nanoparticles as a novel surrogate shows promise in the field of RO integrity monitoring because a nanoparticle analyzer could be used to measure viral sized particles in the RO feed and permeate. Prior work has evaluated use of silver nanoparticles (Antony et al. 2014), but this would require spiking with such particles.

A nanoparticle tracking analyzer (NTA) was evaluated for this study to measure viral sized particles in the RO feed and permeate. The theory of NTA is that particles in suspension are under Brownian motion and the speed of the particle is in reverse proportion to their size. Illuminating the particles with a laser (photon) causes light intensity fluctuations, and the fluctuating rates (Brownian motion) are recorded in a video. The measured change in location within a certain time (t) gives a specific diffusion coefficient (D) for each individual particle. Using the Stoke-Einstein equation, the hydrodynamic particle radius (r) and thus the diameter of each particle is calculated (www.particle-metrix.de). Nanoparticle tracking is a rapidly evolving field and is expected to offer online monitoring capabilities in the near future. Thus, this may allow further evaluation of nanoparticles as a potential surrogate for RO integrity monitoring beyond the initial findings from the present study.

For this study, nanoparticle monitoring was performed using a ZetaView Particle Metrix Analyzer by Particle Metrix (Wildmoos, Germany). RO feed and RO permeate grab samples were collected and analyzed using the Particle Metrix analyzer. Depending on the sample type and measuring mode, the analyzer has a measuring range for particle sizes between 0.015 µm and 5 µm. The analyzer is able to measure both size and concentration of particles.

3.4. Padre Dam AWP Demonstration Facility Surrogate Testing

Testing at the Padre Dam Demonstration Facility focused on four testing activities that took place over a period of approximately six months. The CCRO was operated at 95 percent overall recovery throughout this period. The testing activities were:

1. Execution of MS2 challenge tests to validate pathogen removal through the CCRO.
2. Performance of cycle assessments to evaluate the removal of surrogates and MS2 at different times within the closed-circuit desalination (CCD) cycle.
3. CCRO compromise testing with missing O-rings to evaluate the impact of membrane compromises on MS2 and surrogate rejection.
4. Routine monitoring/sampling of membrane integrity surrogates.

The following subsections describe the procedures that were followed for each testing activity.

3.4.1. Surrogates Monitored

This section describes the surrogates that were sampled and monitored during testing.

3.4.1.1. MS2 Bacteriophage Challenge Tests

MS2 bacteriophage (typical size of 25-27 nanometers [nm]) has generally been accepted as a surrogate for validating removal of enteric viruses by RO because of its similarities (Pype et al. 2016b; USEPA 2005). MS2 bacteriophage challenge tests were performed to benchmark virus rejection for the RO systems. MS2 challenge tests were prepared using a MS2 stock solution (1011 plaque forming units per milliliter [pfu/mL]) supplied by IEH-BioVir Laboratories (Benicia, California). Phage enumeration was performed by IEH-BioVir, primarily using the double agar layer plaque technique per Adams (1959) (limit of detection [LOD] = 1 pfu/mL), with EPA 1602 single agar layer (LOD = 1 pfu/100mL) used as a backup for non-detect double agar layer samples.

3.4.1.2. Naturally Occurring Ions

Using grab samples, several naturally occurring ions in the RO feed water and permeate were routinely sampled as surrogates for integrity monitoring, including phosphate, sulfate, strontium, and magnesium. The methods, MRLs, and method detection limits (MDLs) for the sampled constituents are shown in Table 7. Analysis was performed by Eurofins Eaton Analytical (EEA) (Monrovia, California).

Table 7. Methods for Naturally Occurring Ion and Metals

Constituent	Method	Instrumentation	MRL	MDL
Magnesium	EPA 200.7	ICP-AES	0.1 mg/L	0.003 mg/L
Strontium	EPA 200.8	ICP-MS	0.3 µg/L	0.016 µg/L
Sulfate	EPA 300.0	IC	0.5 mg/L	0.06 mg/L
Orthophosphate	4500P-E/365.1	Automated colorimetry	0.01 mg/L as P	0.007 mg/L as P

3.4.1.3. EC and Total Dissolved Solids

EC was monitored as a baseline surrogate for integrity monitoring. The CCRO pilot had online EC instruments that recorded during testing. Additional grab samples were measured using a handheld Myron L 6PIIFCE Ultrameter. TDS grab samples were also collected and analyzed by EEA using SM2540C/E160.1 method.

3.4.1.4. TOC

TOC was also monitored since it is an already established surrogate for integrity monitoring for various potable reuse facilities in California. Grab samples from the feed and permeate for both RO systems were collected and analyzed by EEA using the SM5310C/E415.3 method (MRL = 0.3 mg/L).

3.4.2. Testing Protocol

This subsection describes the procedures followed for the testing activities.

3.4.2.1. MS2 Challenge Tests

A total of five MS2 bacteriophage challenge tests were performed to benchmark and validate MS2 removal for the CCRO system. The MS2 bacteriophage spiking solution was prepared by diluting the MS2 stock solution (1,011 pfu/mL) supplied by IEH-BioVir Laboratories (Benicia, California) with non-chloraminated UF filtrate from the AWP Demonstration Facility. Non-chloraminated filtrate was used to avoid phage inactivation by residual chloramines. In addition, chloramines were turned off prior to and during MS2 challenge testing. A fresh MS2 phage stock was used for each challenge test. The stocks were used within 2 weeks of their preparation and stored at 4°C prior to being used.

A small batch tank was used to spike MS2 in the primary RO feed to reach a target feed concentration set at approximately 106 pfu/mL for all MS2 challenge tests. At this target feed concentration, it was possible to measure up to 6 log MS2 removal using the double-agar layer and up to 8 log MS2 removal using EPA 1602 (single-agar layer) when used as backup analysis for non-detect double-agar layer samples. MS2 was spiked in the primary RO feed using existing injection port. Considering rejection across the primary RO system, sufficient MS2 reached the CCRO system to measure MS2 removal through it.

For each MS2 spiking event, triplicate samples were collected back-to-back from each sampling location. Four sample locations were used for this study: CCRO tank, CCRO permeate tank, feed-concentrate recirculation line, and CCRO vessel permeate.

The MS2 solution was spiked for at least 90 minutes prior to collecting samples. This ensured steady-state MS2 concentrations throughout CCRO system, including the CCRO feed and permeate tanks. Upon sampling, samples were immediately chilled in a cooler with ice packs and the cooler was overnighted to IEH-BioVir Laboratories where the samples were assayed. MS2 challenge test parameters are shown in Table 8.

Table 8. MS2 challenge test parameters

Parameter	Description
MS2 spike location	Primary RO feed
MS2 solution metric	Non-chloraminated UF filtrate + MS2 stock (10^{11} pfu/mL)
MS2 target feed concentration	10^{6} pfu/mL (spiked)
Sampling Locations (varies per testing activity)	CCRO feed tank CCRO permeate tank CCRO feed-concentrate recirculation line CCRO vessel permeate Note: all samples collected in triplicates
Assay Method	Double-agar layer (Adams 1959) – LOD: 1 pfu/mL Single-agar layer (EPA 1602) – LOD: 1 pfu/100mL (backup)

3.4.2.2. CCRO Cycle Assessments

Three cycle assessment sampling events were performed to capture any differences in surrogate and MS2 removal during the CCD mode. Since the CCRO recirculates the feed-concentrate across the membranes during the CCD mode, the water quality of both the feed-concentrate and permeate produced becomes more concentrated as the CCD cycle progresses. Figure 11 shows the permeate and feed-concentrate samples collected at different times of a closed-circuit cycle, showing the visual progression as the feed-concentrate concentration increases.

Figure 11. CCRO samples from past cycle assessment

During the cycle assessments, paired samples were collected for surrogates and MS2 from the both the feed-concentrate line and the vessel permeate line. Samples were collected at different times during a CCD cycle in a 2L glass beaker and then transferred to designated sample bottles. Table 9 provides the monitored surrogates as well as key parameters for the CCRO cycle assessments.

Table 9. CCRO cycled assessment parameters

Parameter	Description
Number of Cycle Assessments Performed	3
Sample Locations	CCRO feed-concentrate (early, middle, late in CCD cycle) CCRO vessel permeate (early, middle, late in CCD cycle)
Surrogates Monitored during Cycle Assessments	EC (grab), TDS, TOC, MS2 (spiked), ions present in feedwater

3.4.2.3. Compromise Testing

Compromise testing was performed to investigate the removal of surrogates and MS2 during an intentionally created compromised condition. The purpose of this test was to demonstrate that surrogates are able to accurately track any decrease in virus rejection during compromised conditions, using MS2 as a virus surrogate.

Previous RO integrity studies have shown that compromises to O-rings cause the greatest impact on a system's integrity (Jacangelo et al. 2015). The CCRO recirculates feed-concentrate during the closed-circuit cycle, providing higher crossflow velocities than a conventional RO system. As such, it is important to better understand the effects of a damaged/missing O-ring on the integrity of a CCRO system due to its unique operational mode.

To this extent, the compromises for study focused on removal of O-rings. O-rings were removed from permeate interconnectors or vessel endcaps. For the CCRO tested, each interconnector had two O-rings on each end for a total of four O-rings. Vessel endcaps are located at both ends of the pressure vessels and hold the vessel together under operation while providing a final channel for the permeate before entering a manifold containing permeate from other vessels. Each endcap contains two O-rings. Figure 12 shows location of vessel endcaps and interconnectors for the CCRO system tested. Table 10 provides the test parameters for compromise testing and Table 11 provides a description of the compromises performed.

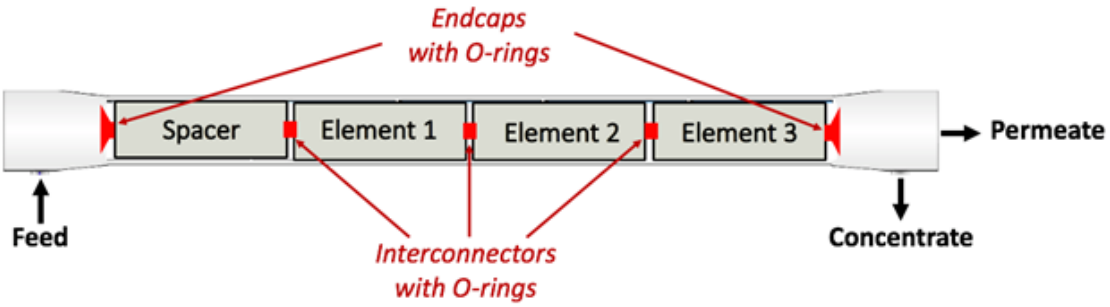


Figure 12. Location of O-ring for tested CCRO system

Table 10. CCRO compromise test parameters

Parameter	Description
Compromise Tests Performed	Three compromise conditions tested in one event
Sample Locations	CCRO feed tank CCRO permeate tank
Surrogates Monitored during Compromise Tests	EC (online), TOC, MS2 (spiked), ions present in feedwater

Table 11. Description of compromised to evaluate surrogate rejection

Compromise Test	Compromise Description
Missing interconnector O-rings	Removal of O-rings (4 total) from a single permeate interconnector between two elements
Missing Concentrate Endcap O-rings	Removal of O-rings (2 total) from endcap located on concentrate side of the pressure vessel
Missing Feed Endcap O-rings	Removal of O-rings (2 total) from endcap located on feed side of the pressure vessel

3.4.2.4. Routine Monitoring and Sampling

Routine monitoring/sampling of multiple surrogates took place over the duration of pilot testing to track changes in rejection offering insights regarding sensitivity of surrogates to membrane aging. Surrogates included EC, TDS, TOC, and other less common surrogates (such as ions present in feed water). CCRO permeate samples were collected from the CCRO permeate tank which received permeate from the system and represented an average sample in terms of water quality from the CCD cycles. Table 12 provides a list of surrogates that were monitored/sampled during testing, along with measurement frequency.

Table 12. Routine monitoring/sampling for RO integrity monitoring

Surrogate	Measurement Frequency	Sampling Locations
EC	Online/weekly grab samples	Primary RO Feed Primary RO Combined Permeate CCRO feed tank CCRO permeate tank
TOC	Online/weekly grab samples	Primary RO Feed Primary RO Combined Permeate CCRO feed tank CCRO permeate tank
Ions present in feedwater, e.g., strontium, magnesium, sulfate, phosphate	Weekly grab samples	Primary RO Feed Primary RO Combined Permeate CCRO feed tank CCRO permeate tank

3.4.2.5. Data Handling

RO system operational data (e.g., pressure, flow, EC, temperature, pH) was monitored for the CCRO system. The CCRO programmable logic controller (PLC) system provided continuous monitoring for a set of parameters. A dedicated software application (SeeQ) was used to store and visualize high frequency data. SeeQ data was exported at user-defined frequency and then transferred to a conventional spreadsheet to perform custom analysis. A data frequency of one hour was selected for data analysis. A screenshot of the SeeQ platform is provided in Figure 13. Analysis used data recorded at the end of each CCD cycle to track long-term membrane performance (e.g., specific flux). Hourly data frequency was chosen for tracking the CCRO process and offered sufficient frequency to track operational deviations and performance trending for the purpose of this study. This allowed normalization of the data to capture changes in performance that are important for tracking RO systems.

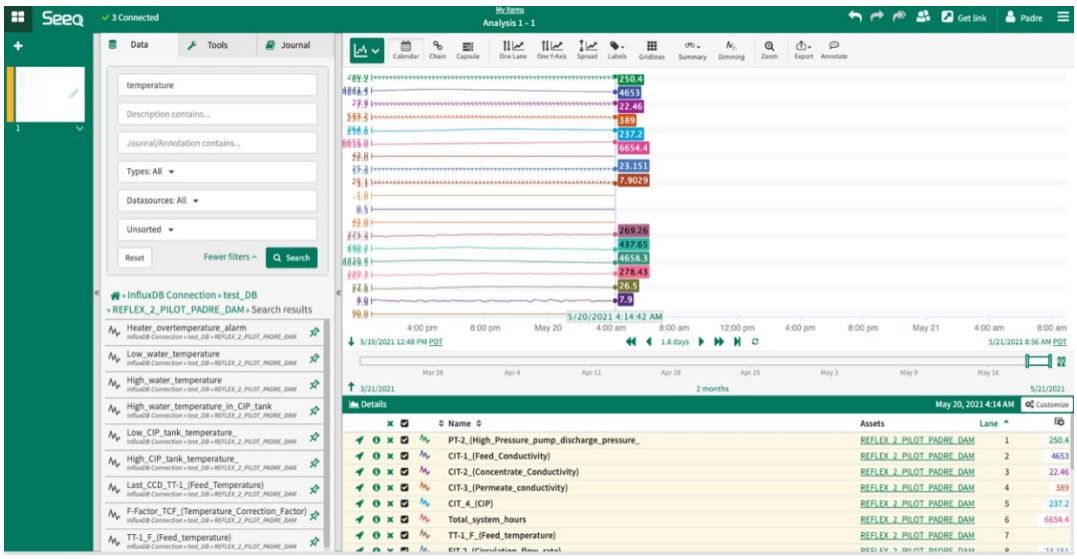


Figure 13. Example of SeeQ platform used for CCRO performance tracking

Performance of the RO systems was monitored using recovery and temperature corrected specific flux. These parameters were calculated using the collected performance data, namely: temperature, EC, flow, and pressure. Salt passage, which is based on salt rejection, was also monitored for the CCRO system. Table 13 provides a summary of operability parameters that were tracked during testing.

Table 13. CCRO system operability tracking during testing

Parameter	Description
Operational data recorded	Pressure, flow, EC, pH, temperature
Recording Frequency	Hourly (custom frequency available)
Data Storage	SeeQ Data Server
Data Handling	SeeQ, Spreadsheet
Performance parameters	Temperature corrected specific flux, recovery, salt passage

3.5. LRV Calculation Methodology

Constituent removal efficiency by the RO process (calculated as an LRV) for each constituent was calculated using Equation 3.7 of the EPA Membrane Filtration Guidance Manual (USEPA 2005):

$$LRV = \log (C_f) - \log (C_p)$$

where:

- LRV = log removal value demonstrated during sampling of constituent;
- Cf = feed concentration of constituent; and
- CP = filtrate concentration of constituent.

For OCWD LRV calculations, it takes approximately 2 minutes for a plug of water to move through a 5-MGD RO unit (RO feed to RO permeate). Thus, RO feed and permeate concentrations were measured 2 minutes apart via an online instrument (or grabs) located at the feed and permeate, and these paired data were used to calculate the LRV for each time point. However, this staggered approach is likely unnecessary given that the feed and permeate concentrations do not change appreciably in such a short time.

It should be noted that any averages of LRVs were taken by averaging the percent removal data (e.g., 90 percent, not 1 log) and then converting the average percent removal value to an LRV. It is a common error to take arithmetic averages of log values and it is not mathematically correct (Schmidt et al. 2020). The error results in overestimation of log removal. When the log values do not vary substantially, the error is less significant.

4. Results and Discussion

4.1. OCWD AWPF Surrogate Testing

This section summarizes the LRV results observed at the OCWD AWPF RO unit for free ATP, fluorescence Peak C, sulfate, and strontium.

4.1.1. ATP

Free ATP measurements were collected every 30 minutes over a 2-month period using the dual stream EZ-ATP analyzer. Instantaneous LRV ranged between 2.60 to 3.30 with an average LRV of 3.03 (Figure 14). Average daily LRV was calculated from the daily average RO feed concentration of free ATP compared to the daily average RO permeate concentration of free ATP (calculated using the same approach currently used by OCWD for DDW compliance for calculating TOC LRV for virus credit). Average daily LRV values ranged between 2.75 to 3.13 (Figure 15). The gaps in data collection shown in these figures were due to instrument scheduled maintenance, and the later gap in 2020 was related to the SARS-CoV-2 pandemic.

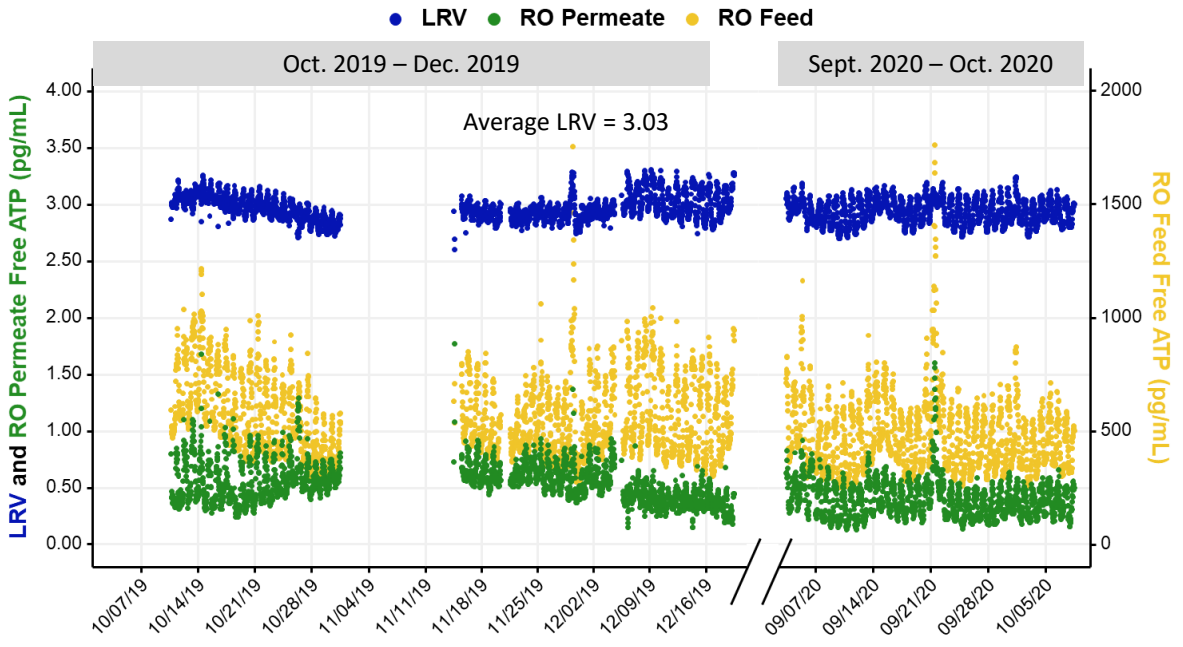


Figure 14. Free ATP rejection as LRV (blue) across a 5-MGD RO unit with ESPA2-LD RO membranes measured using dual stream ATP analyzer. RO feed concentration of free ATP (yellow) and permeate concentration (green) are also shown.

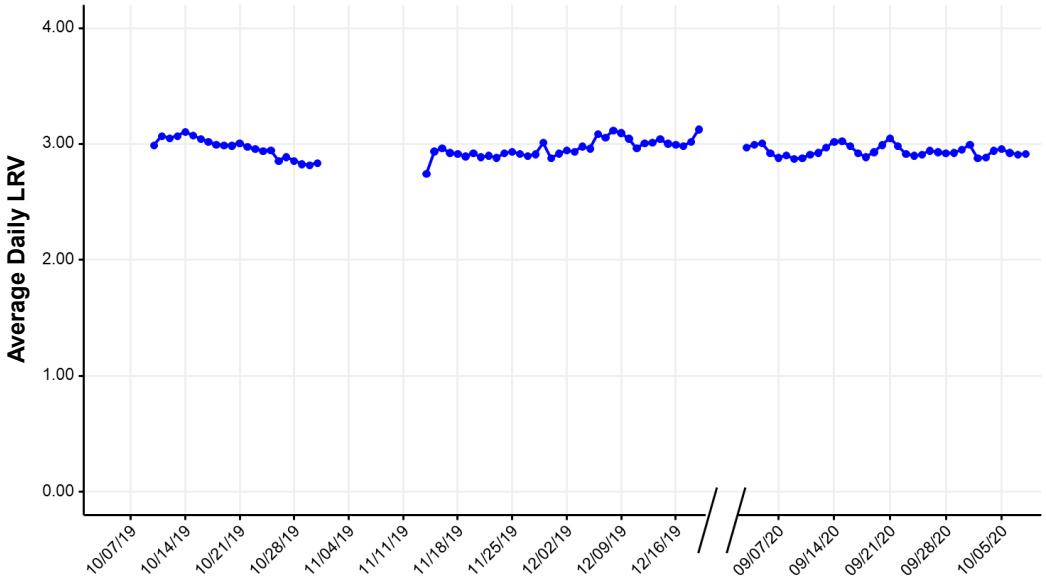


Figure 15. Average daily LRV of free ATP across a 5-MGD RO unit with ESPA2-LD RO membranes

Diurnal variability in the RO feed was observed with higher free ATP concentrations between 11:00 am and 3:00 pm, and the diurnal variation propagated to the RO permeate. The LRV increased as the free ATP increased in the RO feed. The diurnal variation may be the result of varying flows and water quality from the upstream wastewater treatment facility. Even with increased RO feed concentrations, with only few exceptions, the RO permeate free ATP remained ≤1.7 pg/mL. The EZ-ATP analyzer reports values below its reported free ATP detection limit of 0.5 pg/mL. If observed permeate values were below the free ATP detection limit of 0.5 pg/mL, the detection limit value was used to calculated LRV. This is a conservative approach since the true LRV may be greater if lower detection limits were to be used.

4.1.2. Fluorescence Peak C (humic like fDOM)

Peak C fluorescence data was gathered using online fluorometers, measured in the RO feed and permeate to determine diurnal fluctuations and long-term variability for LRV monitoring (Figure 16). Each graphed data point in Figure 16 represents a 15-minute average of 1-second measurements. Online Peak C fluorescence LRV of ESPA2-LD membranes ranged between 2.27 and 3.00 with an average of 2.70. The gap in data collection shown in the figure was due to computer and acquisition software issues. Average daily LRV values ranged between 2.50 and 2.88 (Figure 17), using the same approach currently used by OCWD for DDW compliance for calculating TOC LRV for virus credit. As with free ATP, diurnal variability in the RO feed was observed with higher Peak C concentrations between 11:00 am and 3:00 pm, and the diurnal variation propagated to the RO permeate. The LRV increased as the Peak C increased in the RO feed. The diurnal variation may be the result of varying flows and water quality from the upstream wastewater treatment facility.

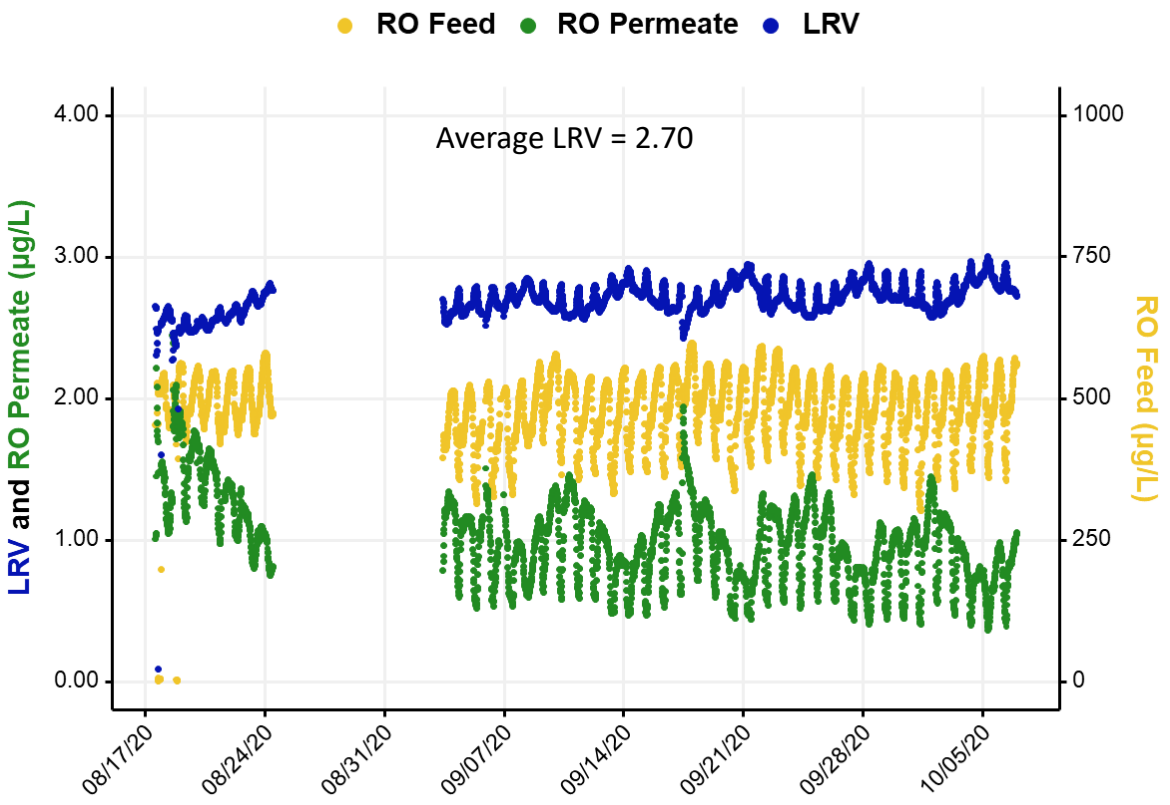


Figure 16. Fluorescence Peak C rejection (blue) across a 5-MGD RO unit with ESPA2-LD RO membranes measured using online C3 fluorometers. RO feed concentration of fluorescence Peak C (yellow) and permeate concentration (green) are also shown.

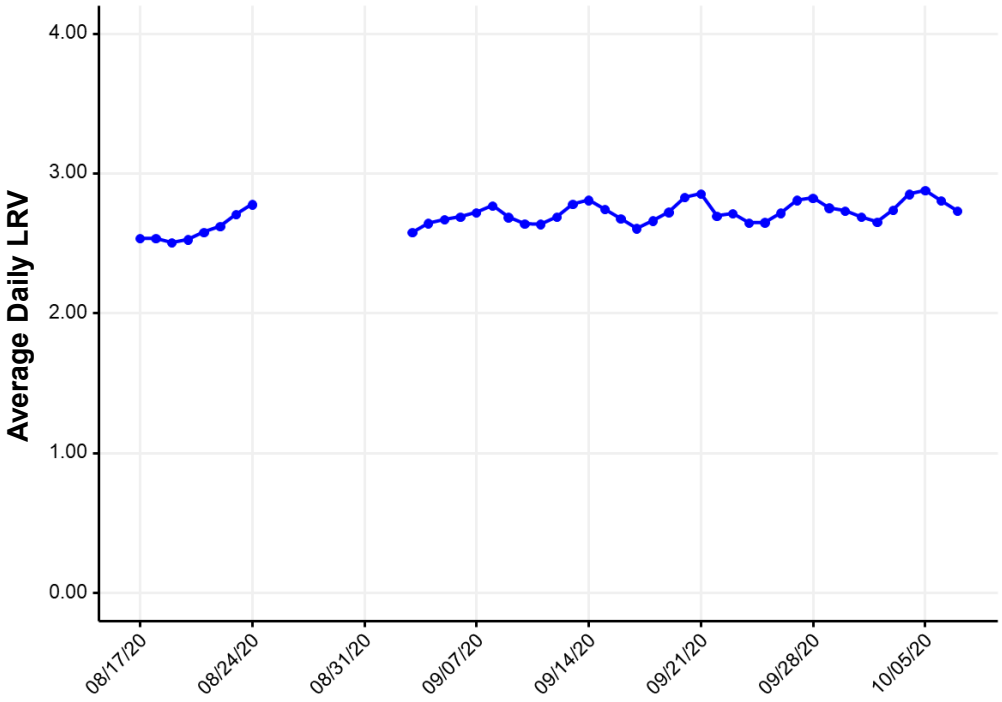


Figure 17. Average daily LRV of Fluorescence Peak C free across a 5-MGD RO unit with ESPA2-LD RO membranes

Online Peak C fluorescence was measured for 24 hours after the new Dupont FilmTec BW30XFRLE membranes were installed. The 24-hour online sampling event was performed in parallel to 24-hour strontium and sulfate sampling events on 12/22/2020 (described below in section 4.1.3). The DuPont membrane Peak C fluorescence LRV ranged between 2.95 and 3.05 with an average of 2.99 (Figure 18); this is slightly higher than that measured previously with ESPA2-LD membranes. On this day, the RO feed diurnal variability was lower between 11:00 am and 3:00 pm, opposite of what was observed previously (Figure 17). As stated above, the change in RO feedwater is the result of varying flows and water quality from the upstream wastewater treatment facility (OC San). Because the diurnal variation propagates to the RO permeate, the overall LRV remained >2.9.

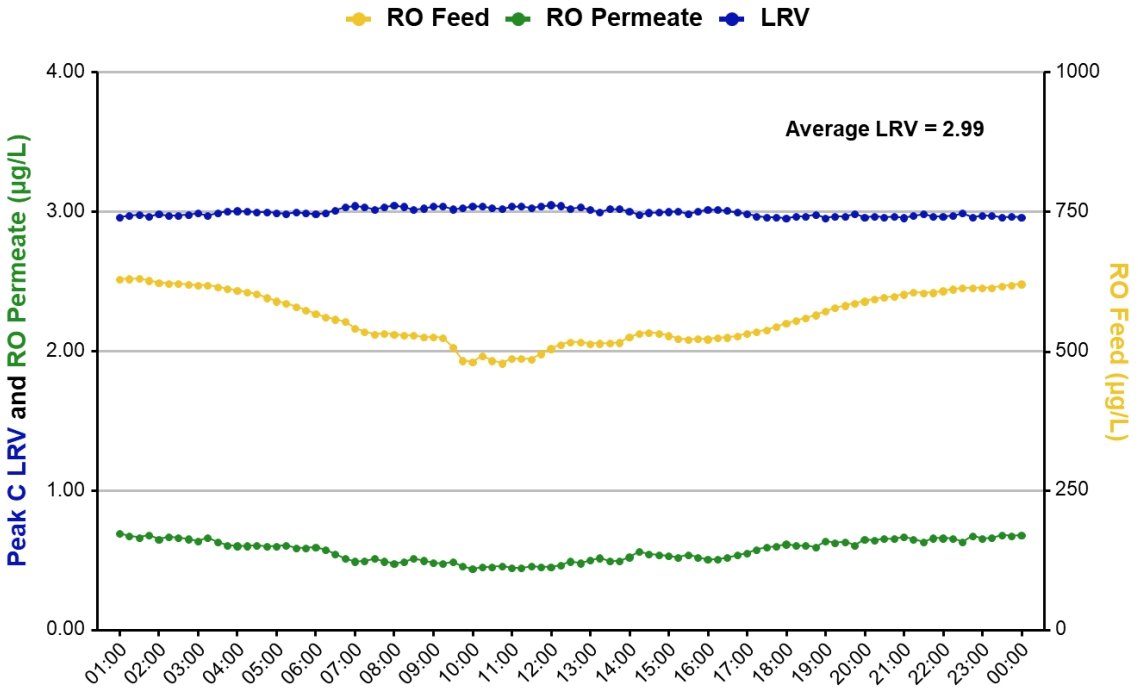
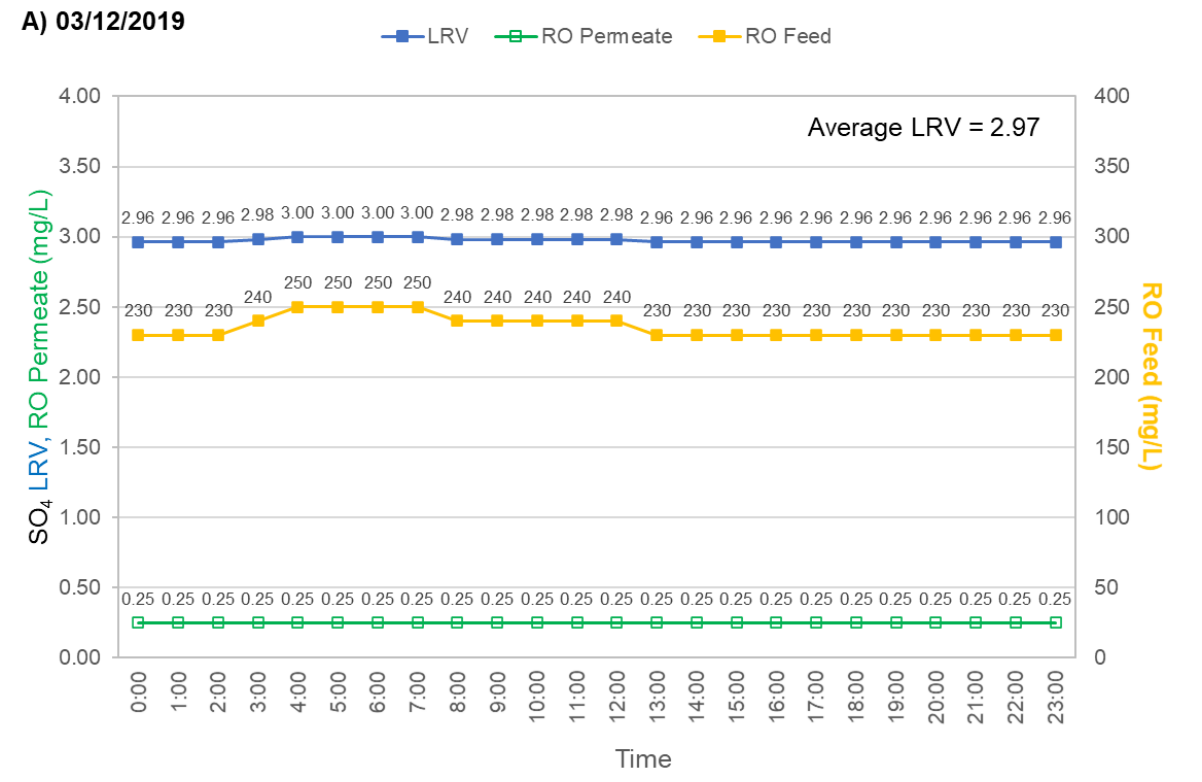


Figure 18. Fluorescence Peak C rejection (blue) across a 5-MGD RO unit with Dupont FilmTec BW30XFRLE membranes. Each graphed data point represents 15-minute average of 1-second measurements. Measurements were collected for 24 hours. RO feed concentration of fluorescence Peak C (yellow) and permeate concentration (green) are also shown.

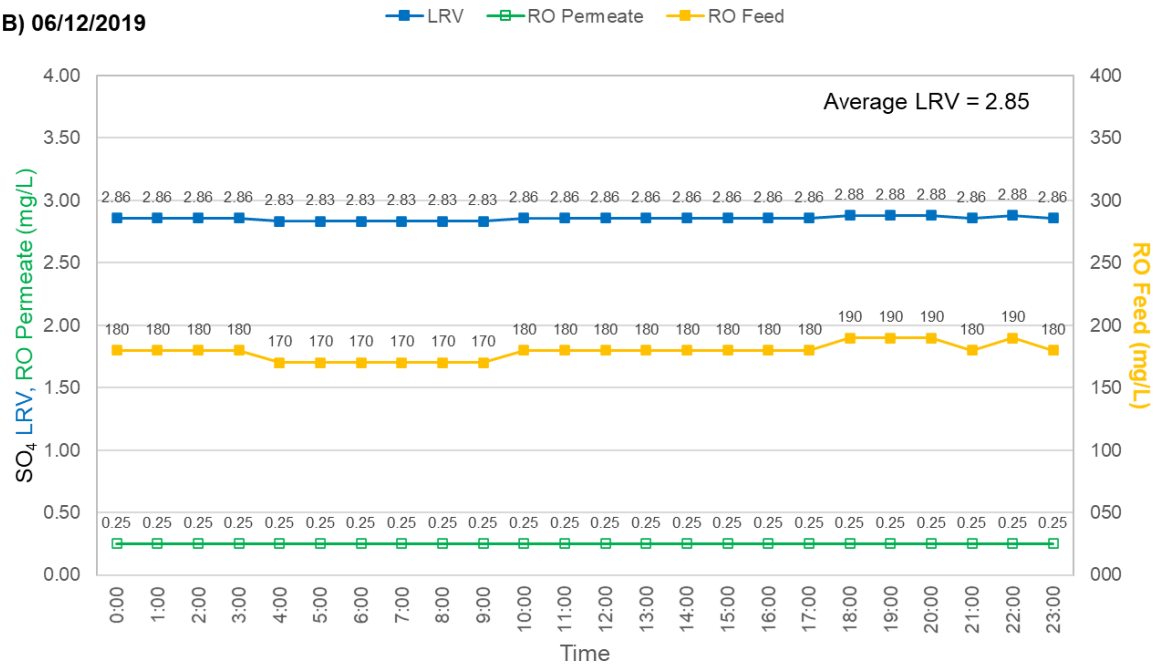
4.1.3. Naturally Occurring Ions: Sulfate and Strontium

Compared to the diurnal variability in LRV observed for free ATP and fluorescence Peak C, both sulfate and strontium LRVs were extremely stable with LRV maintained in a narrow, steady range. Data shown in Figure 19 represents five sulfate sampling events performed on 03/12/2019 (ESPA2-LD), 06/12/2019 (ESPA2-LD), 02/24/2020 (ESPA2-LD), 09/26/2020 (ESPA2-LD), and 12/22/2020 (Dupont FilmTec BW30XFRLE) with grab samples taken every hour for 24 hours. Sulfate showed a slightly lower LRV than strontium depicted in Figure 19. Average daily sulfate LRVs on these different days (sampling events) were 2.97 (Figure 19 A), 2.85 (Figure 19 B), 2.89 (Figure 19 C), 2.92 (D), and 2.94 (Figure 19 E), respectively. The average LRV for all five sampling events was 2.91. There was no apparent difference in LRVs between the ESPA2-LD and DuPont membranes. Beyond this more intensive (hourly) sampling, additional monthly/bimonthly grab samples were collected later from the same 5-MGD RO unit for comparison over time and showed sulfate LRVs eventually improved to >3 (Table 14). RO permeate mostly remained at or below the MRL value of 0.25 mg/L, which was used to calculate LRV when the sample value measured less than 0.25 mg/L. Note that open green squares on Figure 19 represent permeate concentrations less than the MRL for sulfate.

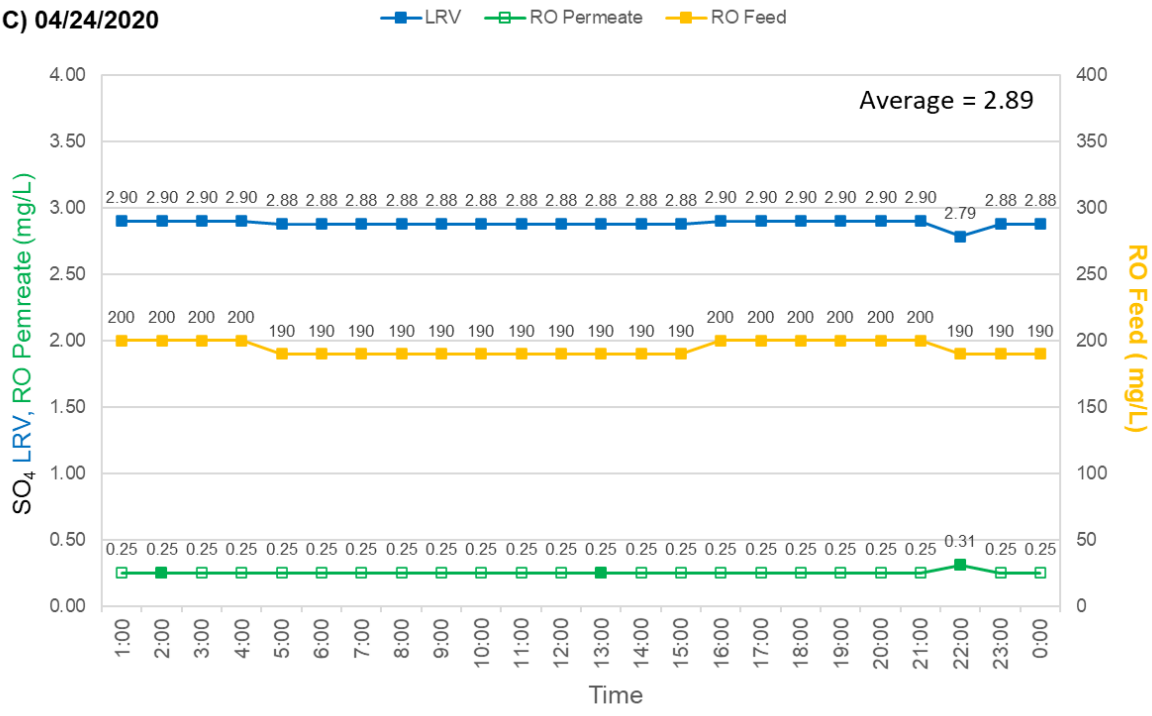
As noted for sulfate, strontium LRV appeared to be very stable and did not exhibit the same diurnal fluctuations as free ATP and fluorescence Peak C. Strontium consistently provided a detectable LRV with average daily LRVs of 3.29 (Figure 20 A), 3.24 (Figure 20 B), 3.24 (Figure 20 C) and 3.14 (Figure 20 D), respectively, for the sampling events corresponding to ESPA2-LD membranes. A lower LRV of 2.86 (Figure 20 E) was observed after the new DuPont membranes were installed in this RO unit. Findings from these sampling events show strontium rejection was more stable with ESPA2-LD membranes than the new Dupont FilmTec BW30XFRLE. It should be noted that newly installed RO membranes may take up to several weeks to acclimate and to stabilize. During the acclimation period, membrane rejection of some constituents may vary but eventually stabilizes to produce a more consistent permeate. Additional monthly/bimonthly grab samples were collected later and data showed LRVs stabilized and improved to >3 (Table 14). The MRL value was used to calculate LRV when the sample value measured less than 0.30 µg/L. Note that open green circles on Figure 20 represent permeate concentrations less than the MRL for strontium.



B) 06/12/2019



C) 04/24/2020



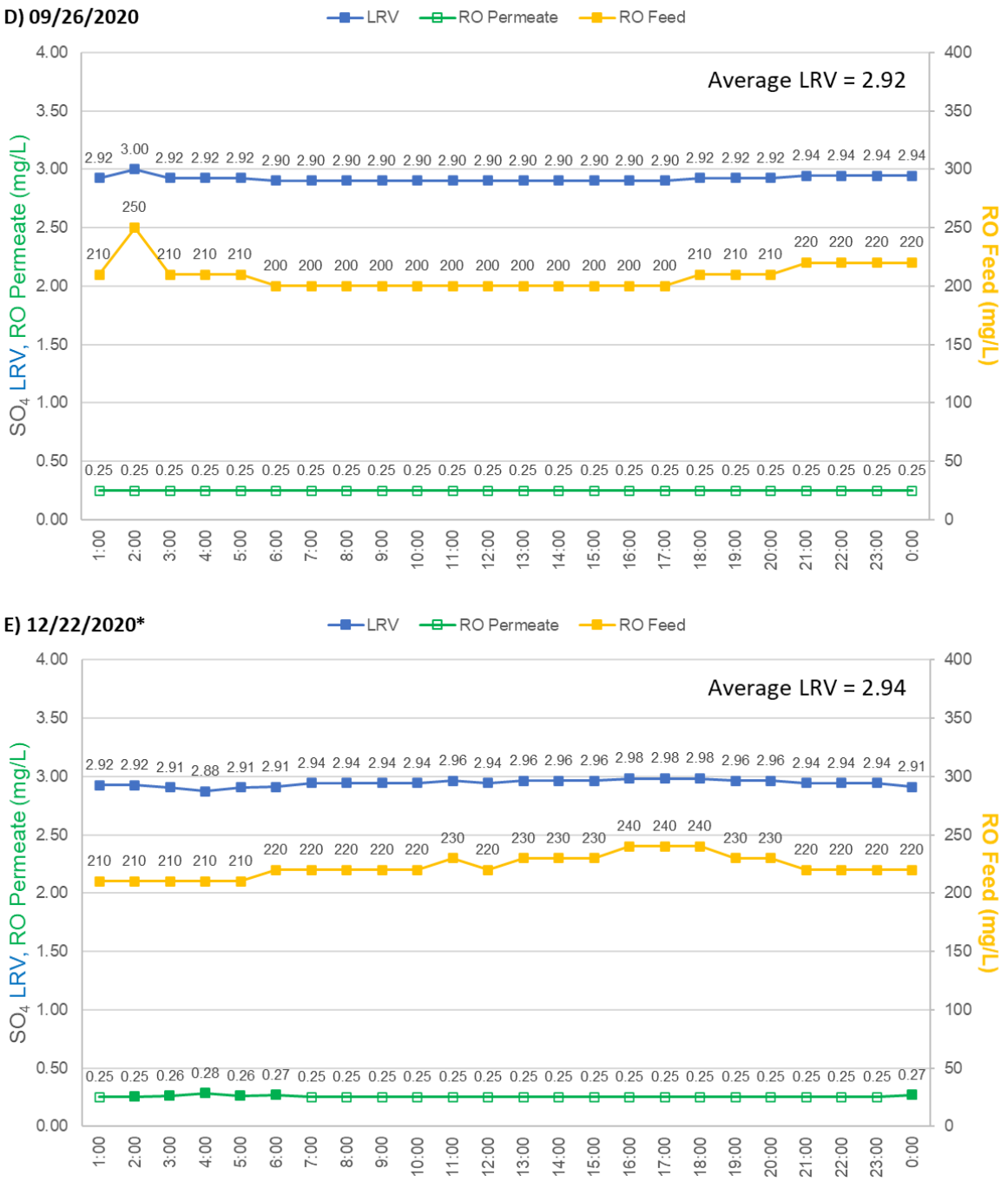
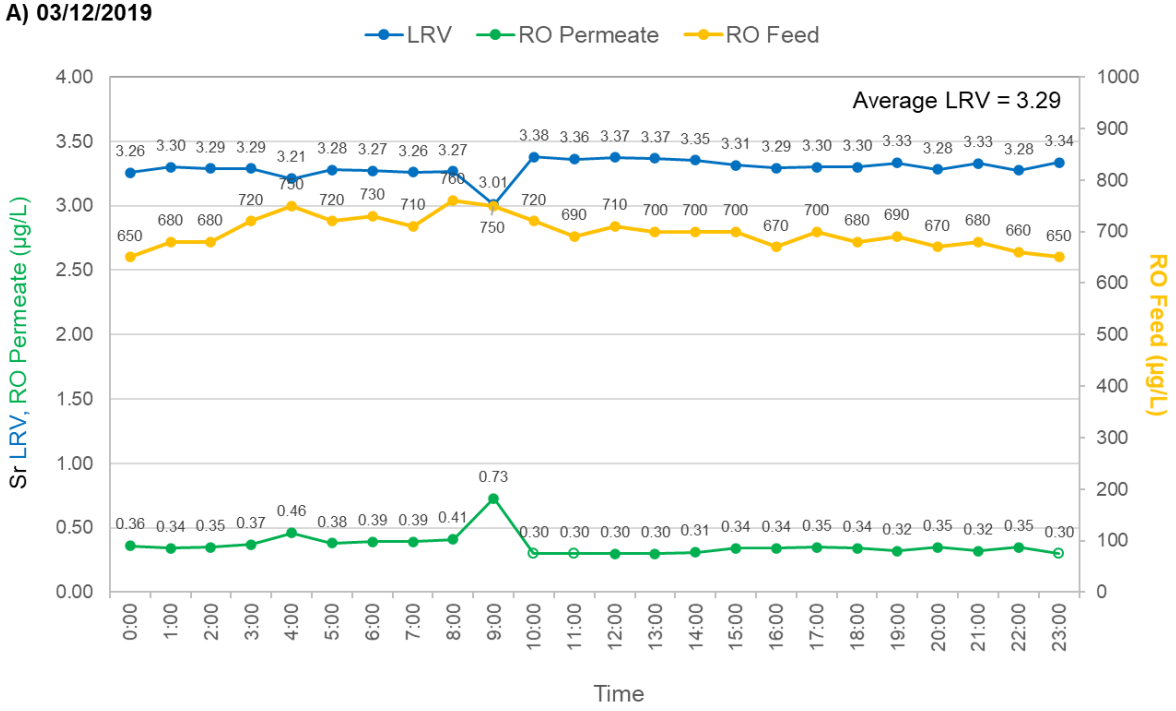
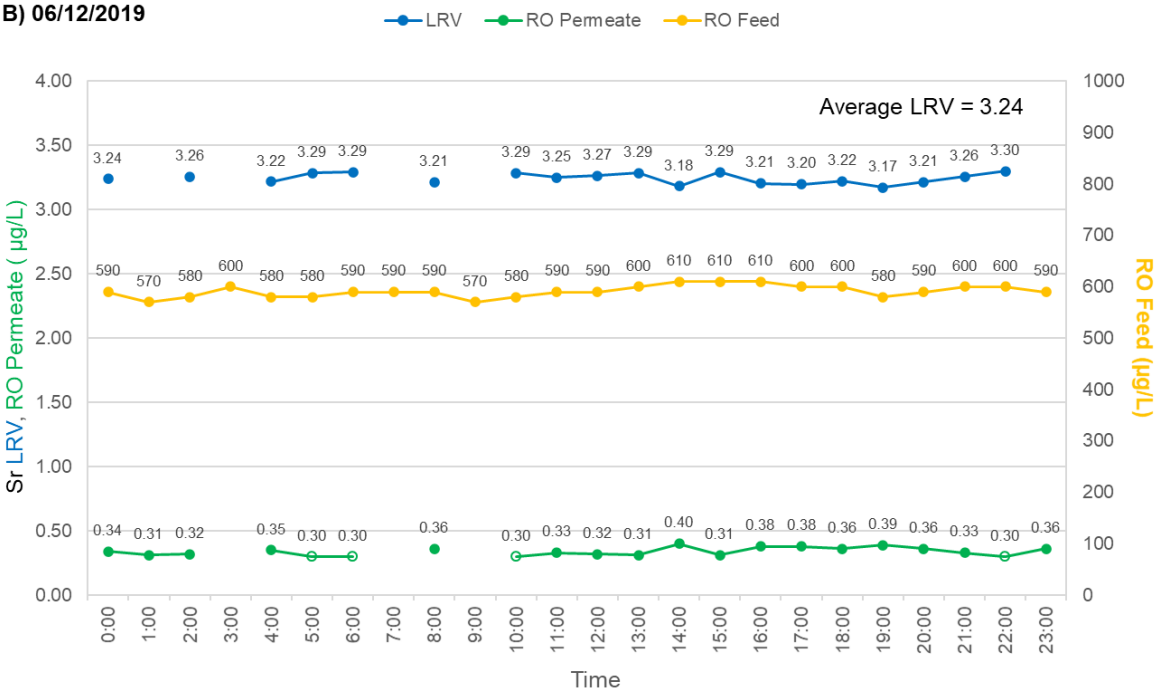


Figure 19. Sulfate rejection (blue) across a 5-MGD RO unit. Charts A through D represent sulfate removal with ESPA2-LD RO membranes. Chart E* represents sulfate removal with Dupont FilmTec BW30XFRLE, installed 10/31/2020. RO feed concentration of sulfate (yellow) and permeate concentration (green) are also shown.

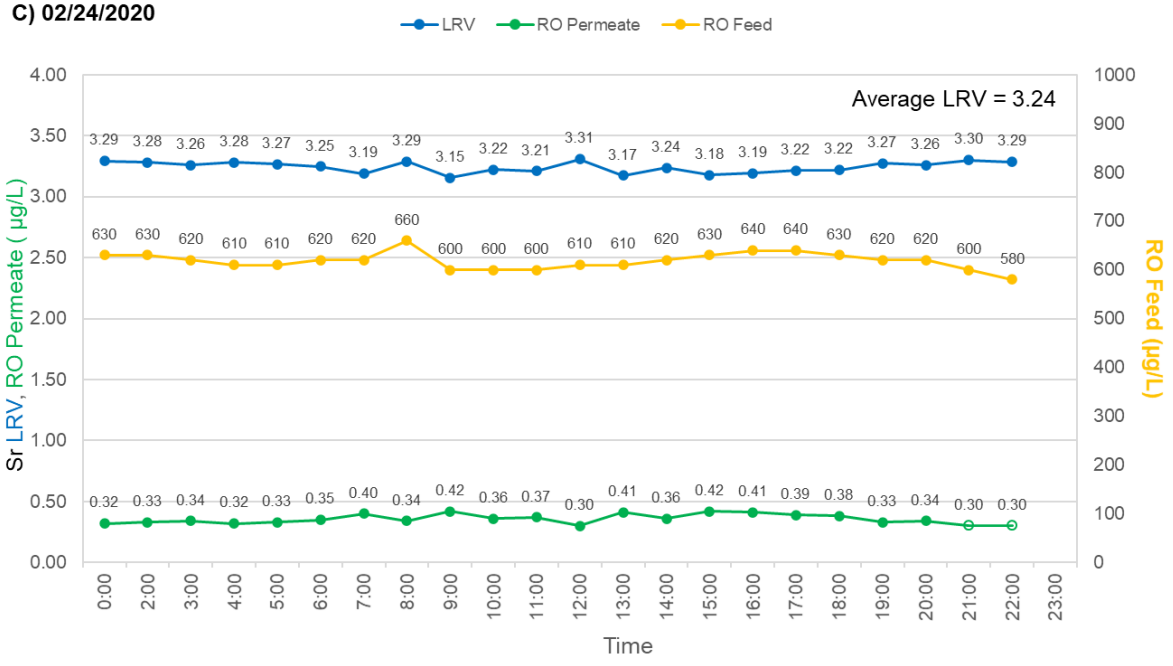
A) 03/12/2019



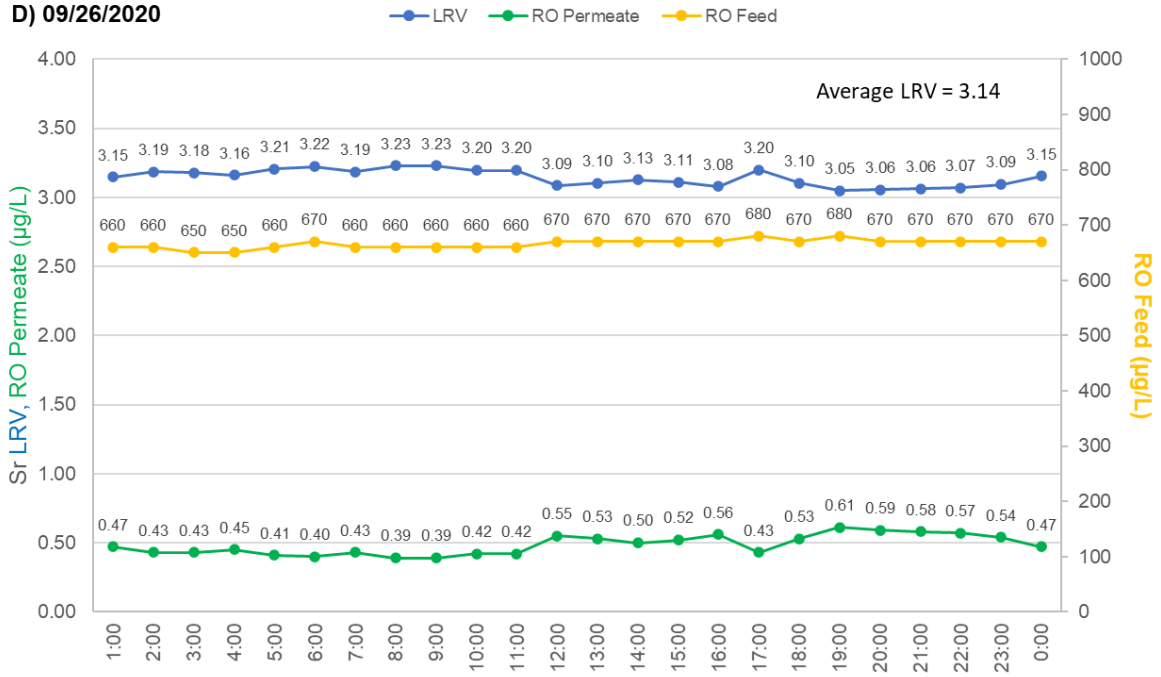
B) 06/12/2019



C) 02/24/2020



D) 09/26/2020



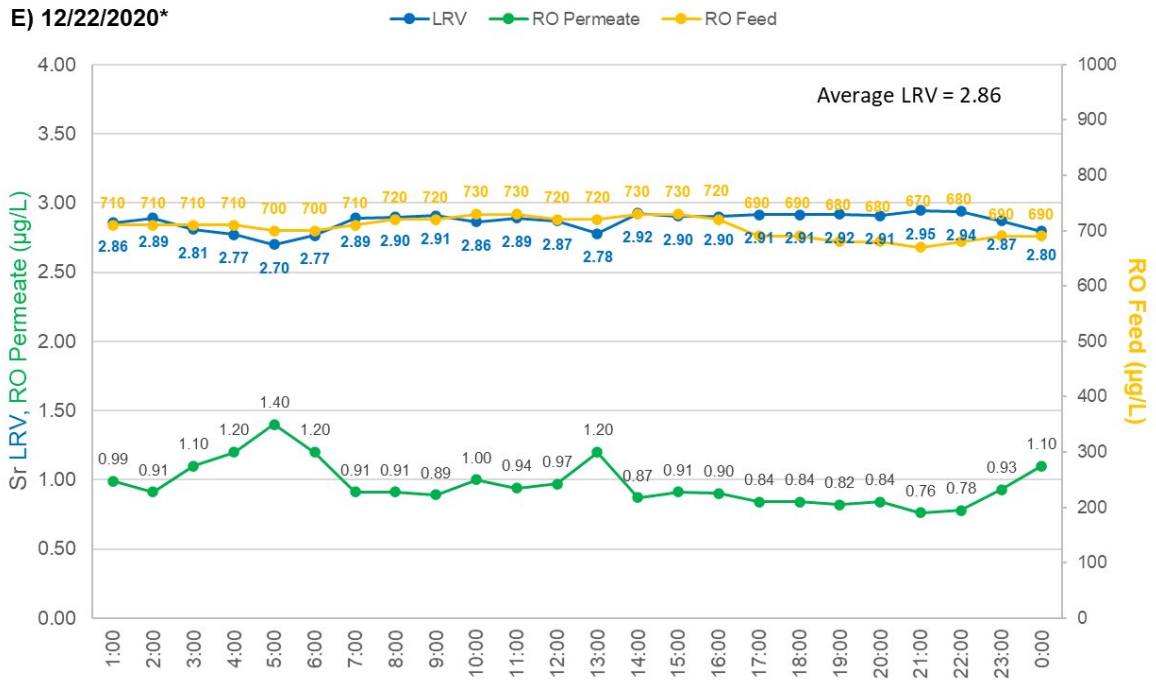


Figure 20. Strontium rejection across a 5-MGD RO unit. Charts A through D represent strontium removal with Hydranautics ESPA2-LD RO membranes. Chart E* represents strontium removal with Dupont FilmTec BW30XFRLE, installed 10/31/2020. RO feed concentration of strontium (yellow) and permeate concentration (green) are also shown.

Table 14. Grab sample LRV measurements for OCWD 5-MGD RO unit after installation of new Dupont FilmTec BW30XFRLE membranes

Date	LRV – Strontium	LRV – Sulfate
12/22/2020	2.86[a]	2.94[a]
1/21/2021	3.14	2.79
3/12/2021	3.08	3.03
4/14/2021	3.08	3.07
5/17/2021	NA	NA
7/13/2021	3.12	3.07

a – average LRV of 24-hr hourly sampling event
NA – not available

To capture LRV differences across the facility, additional strontium and sulfate grab samples were collected on full-scale 5-MGD RO units from the same plant featuring different brands and ages of membranes as well as on bulk permeate from all RO units (combined permeate from 21 RO units that proceeds as a blend to the UV/AOP treatment step). The LRVs for these additional samples are provided in Table 15 and Table 16. They indicate that both the Hydranautics ESPA2-LD, Dupont BW30XFRLE and LG BW 400 ES membranes are capable of providing 3 logs of strontium rejection. All three membrane types are capable of providing ≥2.8 logs of sulfate rejection. The data shows there is some unit-to-unit variability for both strontium and sulfate rejection, but the bulk permeate LRV remains >3 for strontium and >2.8

for sulfate. On a bulk permeate basis, the LRVs for both sulfate and strontium slightly improved for December 2020 when some RO units had membranes replaced.

Table 15. Additional strontium sampling on OCWD AWPF RO units; data shown are strontium log removal values

Membrane	RO Unit	9/26/ 2020[a]	12/22/ 2020[c]	1/21/ 2021	3/12/ 2021	4/14/ 2021	5/17/ 2021	7/13/ 2021
ESPA2-LD/ Dupont FilmTec BW30XFRLE [a]	B01	3.15	2.89	3.14	3.08	3.08	NA	3.12
ESPA2-LD	D03	3.22	3.40	1.94	3.30	3.40	3.40	3.41
DOW XFRLE[d]	G03	2.89	3.10	3.40	3.30	3.40	3.40	3.40
Dupont FilmTec BW30XFRLE	C01	NA	NA	NA	2.93	2.89	2.99	2.99
LG BW 400 ES	A01	NA	NA	NA	3.30	3.45	3.40	3.31
NA	Bulk Permeate[e]	3.15	3.10	3.15	3.33	3.33	3.34	3.27

a – 10/31/2020 ESPA2-LD membranes were replaced with DuPont FilmTec BW30XFRLE
b – ESPA2-LD membranes
c – DuPont FilmTec BW30XFRLE – 12/20/2020 to 07/13/2021
d – older DuPont FilmTec BW30XFRLE membranes – installed May 2015
e – bulk permeate from all RO units at GWRS that are fitted with different membranes
NA – not available

Table 16. Additional sulfate sampling on OCWD AWPF RO units; data shown are sulfate removal values

Membrane	RO Unit	9/26/ 2020	12/22/ 2020	1/21/ 2021	3/12/ 2021	4/14/ 2021	5/17/ 2021	7/13/ 2021
ESPA2-LD/Dupont FilmTec BW30XFRLE	B01[a]	2.92[b]	2.96	2.79	2.82	2.92	NA	2.92
ESPA2-LD	D03	2.92	2.96	2.78	2.82	2.92	2.88	2.92
DOW XFRLE	G03	2.82	2.96	2.92	2.53	2.62	2.88	2.92
Dupont/FilmTec BW30XFRLE	C01	NA	NA	NA	2.82	2.89	2.88	2.92
LG BW 400 ES	A01	NA	NA	NA	2.82	2.92	2.88	2.92
NA	Bulk Permeate[d]	2.92	2.96	2.92	2.82	2.92	2.88	2.92

a – 10/31/2020 ESPA2-LD membranes were replaced with DuPont FilmTec BW30XFRLE
b – ESPA2-LD membranes
c – DuPont FilmTec BW30XFRLE – 12/20/2020 to 07/13/2021
d – bulk permeate from all RO units at GWRS that are fitted with different membranes
NA – not available

4.1.4. Nanoparticles

The nanoparticle analysis did not deliver as expected based on prior research conducted by the research team for OCWD MF process feed/effluent (Rajagopalan et al. 2021), though that prior work was using a different nanoparticle analyzer no longer supported by that particular company. For the present study, data generated by the NTA software was not reproducible and

only background noise was recorded in the RO permeate and often in the RO feed. This could have been caused by either the instrument not being sensitive enough for the application or by the concentration of nanoparticles being too low in the OCWD samples. During testing, the research team worked with Particle Metrix engineers to standardize the sampling method and data analysis but these efforts were not successful. Particle Metrix engineers are working on improvements and updates to their equipment and software which may improve nanoparticle detection for future applications.

4.2. Padre Dam AWP Demonstration Facility Surrogate Testing

This section discusses the Padre Dam AWP Demonstration Facility testing results.

4.2.1. Water Quality

Table 17 summarizes feed water quality from the CCRO system operated at 95 percent overall recovery, having received concentrate from the primary RO system operated at 75 percent recovery. The dataset consists of two feed water quality sampling events, one collected at the beginning of testing (June 2019) and the other at the end (December 2019). Additional data was added to those constituents sampled as part of routine surrogate monitoring (e.g., strontium, magnesium, and orthophosphate).

Table 17. Primary RO and CCRO water quality average data

Parameter	Unit	CCRO Feed – Average	CCRO Feed – STDEV	CCRO Feed – N
Aluminum	µg/L	≤10	--	2
Barium	µg/L	70	10	2
Calcium	mg/L	203	29	2
Iron	mg/L	0.11	0.09	4
Magnesium	mg/L	63	8	25
Potassium	mg/L	59.9	8.1	2
Silica	mg/L	46.2	9.5	8
Sodium	mg/L	561	68	2
TOC	mg/L	27.9	3.0	23
TDS	mg/L	2383	103	23
Chloride	mg/L	764	179	2
Sulfate	mg/L	695	50	25
Manganese	mg/L	0.07	0.04	2
Fluoride	mg/L	2.8	1.0	2
Strontium	mg/L	1.16	0.25	25
Bicarbonate	mg/L	222	65	2
Alkalinity	mg/L as $CaCO_3$	210	14	2
Ammonia	mg/L as N	3.30	0.99	2
Nitrate	mg/L as N	29.9	13.5	23

Parameter	Unit	CCRO Feed – Average	CCRO Feed – STDEV	CCRO Feed – N
Orthophosphate	mg/L as P	0.48	0.30	24
pH	--	6.43	0.24	26

STDEV: standard deviation. TOC: total organic carbon. TDS: total dissolved solids. N: number of data points.

4.2.2. CCRO Operational Performance

Operation of the CCRO pilot began on June 12th, 2019, using new FilmTec BW30XFR membranes provided directly from Dupont. Primary RO and CCRO recoveries were set at 75 percent and 80 percent, respectively, to achieve an overall recovery of 95 percent. At this setting and with these membranes, the CCD cycles had a duration of approximately 11 to 12 minutes, while the plug flow flushes lasted approximately 1.5 minutes each. CCRO data logging using Desalitech's SeeQ portal began on 07/09/2019. Before data logging began, the pilot was checked via remote monitoring as well as on-site checks to verify steady operational performance (i.e., flows, pressure, and controls). Figure 21 shows CCRO operational performance from the start of data logging to the end of testing in terms of specific flux and feed pressure, with key events labeled as dashed lines.

CCRO feed pressure was around 200 psi at the start of data logging. Specific flux at 25ºC was approximately 0.07 gfd/psi at the start of data logging, yet a calibration of the feed EC probe on 07/26/2019 (282 runtime hours) shifted specific flux to approximately 0.05 gfd/psi since feed EC is used to calculate specific flux. Per Figure 21, a general increase in specific flux and decrease feed pressure was observed starting from approximately 300 runtime hours until membranes were replaced on 08/18/2019 (717 runtime hours) due to an unforeseen integrity issue. The integrity issue was captured as sharp rise in permeate EC as measured in the CCRO permeate tank, as shown in Figure 22. In an effort to recommence testing under normal conditions, the CCRO membranes were replaced with tail elements taken from primary RO on 07/31/2019. No other components (e.g., seals, connections, spacer element) were replaced at the time and integrity was restored. Once membranes were replaced, permeate conductivity was restored to approximately 120 μS/cm. In addition, specific flux at 25ºC slightly increased to 0.06 gfd/psi.

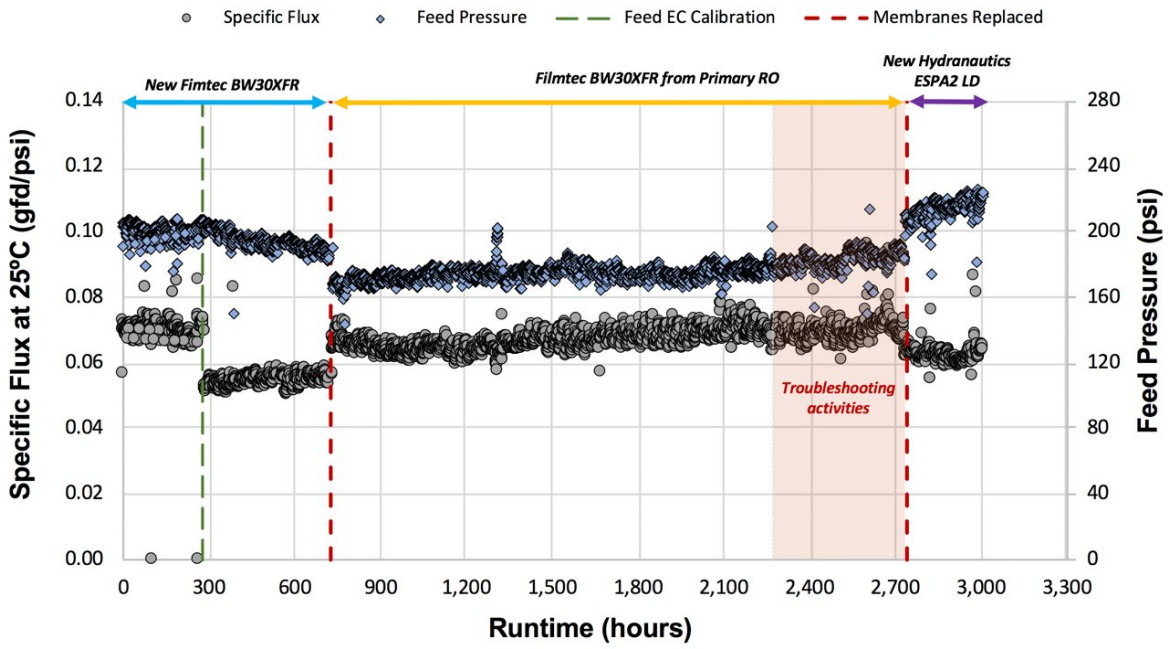


Figure 21. CCRO operational performance during testing

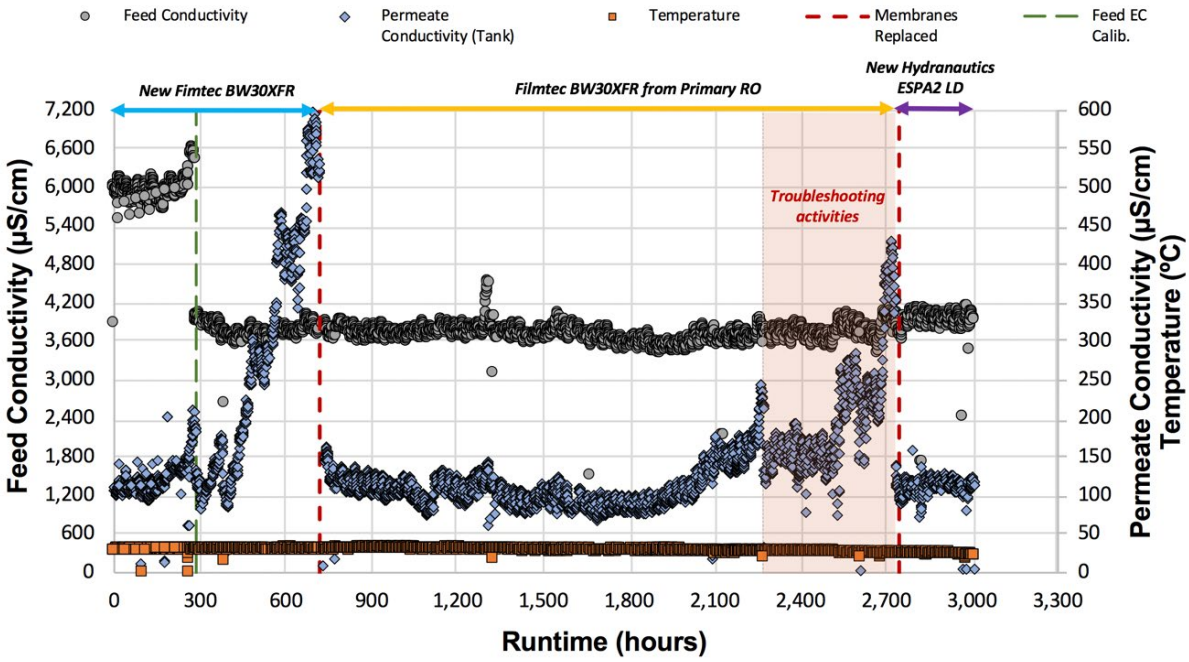


Figure 22. CCRO conductivity monitoring during testing

For the following 1,200 runtime hours (717 to 1,950 runtime hours on Figure 22) membrane integrity was steady with respect to permeate conductivity. Specific flux at 25°C was also relatively stable during this period, with a slight increasing trend throughout. Starting at approximately 1,917 runtime hours (10/23/2019), permeate EC began to increase steadily. After recording a sustained increase in permeate EC to approximately 200 µS/cm, field troubleshooting activities began on 11/8/2019 (2,274 runtime hours) to identify the suspected

integrity issue. The troubleshooting activities had the objective of restoring integrity. Troubleshooting activities included:

- Replacing all seals from all interconnectors and endcaps
- Replacing all interconnectors
- Changing spacer element to a different model
- Adding shims to feed endcap
- Verifying setpoints
- Conducting multiple rounds of vessel probing

Integrity was not restored from these troubleshooting activities and a second CCRO membrane replacement took place on 12/04/2019 (2,735 runtime hours). Results from autopsy showed noticeable dye uptake on the permeate side and dark spots on the feed side (Appendix A). These indicated damage to the membranes.

For the second replacement, new Hydranautics ESPA2-LD membranes provided directly from Hydranautics in Oceanside, California were used. Similar to the first replacement, no other components were changed during this membrane replacement. Following replacement, permeate EC was restored to approximately 120 μS/cm. Testing continued until 12/20/2019 (3,007 runtime hours) when the CCRO pilot was shutdown to conclude testing as scheduled. The Hydranautics membranes did not show any signs of integrity loss during the 272 hours of runtime. This runtime is inferior to that on the previous FilmTec BW30XFR membranes. That said, previous CCRO testing at Padre Dam logged over 4,500 run hours using Hydranautics ESPA2-LD without encountering membrane integrity issues (Idica et al. 2017).

4.2.3. Surrogate performance and MS2 rejection

A reduction in surrogate rejection was observed as permeate EC increased. Figure 23 shows results from routine monitoring in terms of surrogate removal with key events and membrane replacements highlighted as dashed lines.

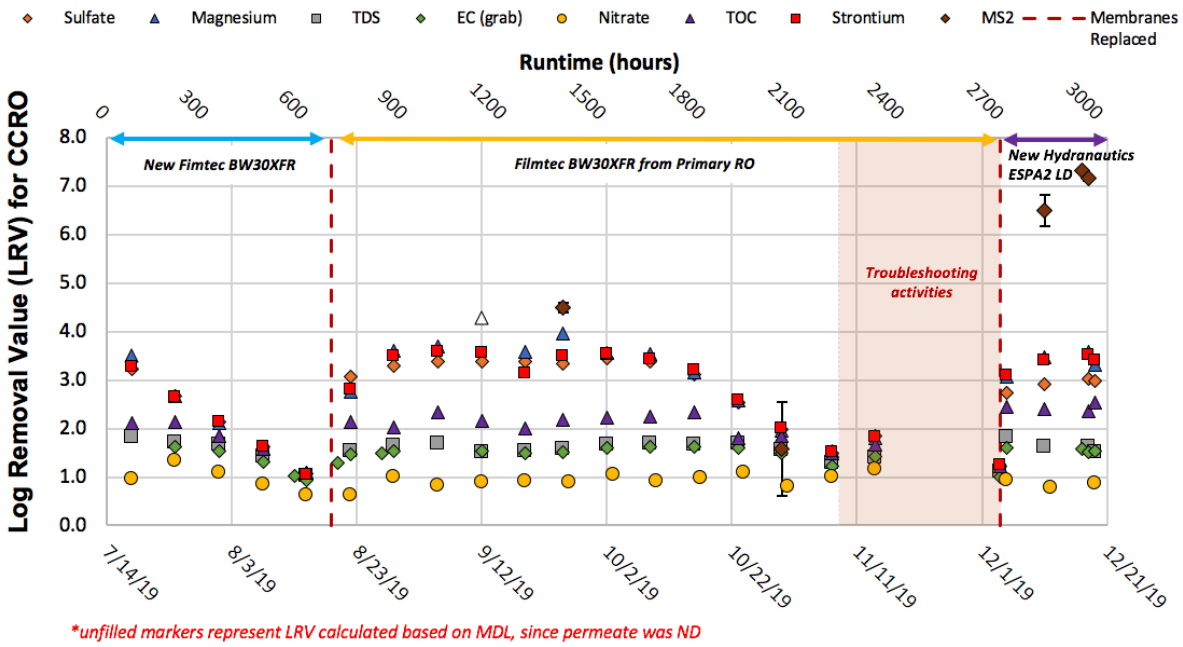


Figure 23. CCRO log removal values of surrogates and MS2

With first set of FilmTec BW30XFR membranes, sampling began on 07/09/2019 when the CCRO pilot had 165 runtime hours from the start of SeeQ data logging. The first sampling event showed a strontium, magnesium, and sulfate rejection above 3 logs, as expected per primary RO surrogate performance. The following four sampling events showed a decreasing trend in removal. The initial drop in rejection was primarily observed for strontium, magnesium, and sulfate, whereas rejection in TOC, TDS, and EC showed relatively stable removal at first. This shows that surrogates rejected to a higher degree (i.e., more sensitive surrogate) were more sensitive to the developing membrane integrity issue. For the last sampling event on the first set of membranes (08/15/2019), rejection of all surrogates converged to approximately 1 log. Following this event, membranes were replaced on 08/19/2019 in efforts to resume testing under normal conditions.

Routine sampling resumed on 08/22/2019 once the second set of FilmTec BW30XFR membranes were installed. Although TOC, TDS, and EC rejection appeared to be fully restored for the first sampling event following membrane replacement, rejection of sulfate, magnesium, and strontium was slightly lower (e.g., 2.8 logs for strontium) than was observed at the start of sampling on first set of membranes (3.3 logs for strontium). For the following seven weekly sampling events (from 08/29/2019 to 10/09/2019), the rejection of sulfate, magnesium, and strontium was above 3 logs which coincides with the 1,200 runtime hours of stable performance in terms of permeate EC (717 to 1,950 runtime hours on Figure 23). Specifically, strontium rejection during this time was 3.47 ± 0.15 logs (N = 7).

Starting 10/16/2019, rejection of strontium, magnesium, and sulfate began to steadily decrease over the next weeks. In comparison to the first set of membranes, the rate of rejection decrease was very similar with the second set of membranes, dropping nearly 0.5 log per week for strontium, for example. Figure 24 provides a comparison of the LRV drop for TDS and

strontium from when an observable decrease in strontium rejection was measured. For both sets of membranes, reduction in strontium followed a strong correlation (R2 > 99 percent) decreasing nearly 0.5 log per week. In contrast, an observable decrease in TDS only became apparent 3 weeks after strontium rejection decreased. This shows how more sensitive surrogates, such as strontium, are suitable to provide an early warning regarding issues with membrane integrity. Sampling events were put on hold during the troubleshooting activities. A last sampling event on these membranes took place on 12/04/2019 and confirmed that integrity was not restored. Similar to the first set of membranes, rejection of all surrogates decreased to approximately 1.0 to 1.2 logs before they were removed.

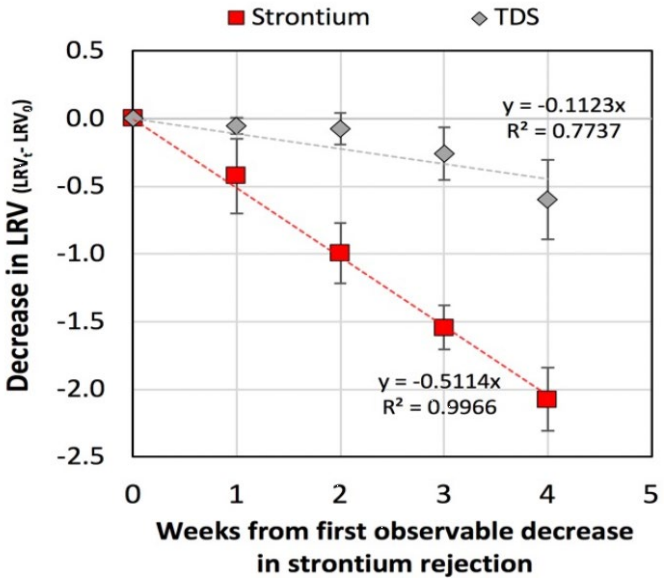


Figure 24. Reduction in CCRO LRV for select surrogates during the development of membrane integrity issues

After the 12/04/2019 sampling event, membranes were replaced with new Hydranautics ESPA2-LD. No other parts aside from the membranes were changed for this replacement as well. A first round of sampling on these new membranes took place the following day (12/05/2019). Figure 23 shows that rejection of surrogates was clearly restored after the third membrane replacement. An additional three sampling events were performed on the ESPA2-LD membranes over the following 2 weeks, showing rejection of strontium in the 3.4-3.5 LRV range. Rejection of magnesium was also in this range, while sulfate was slightly lower at approximately 2.8-3.0 LRV. The CCRO pilot testing was concluded on 12/20/2019, necessitated by the fact that Ray Stoyer WRF underwent planned maintenance activities.

MS2 Challenge Testing:

A total of five challenge test events were performed to calculate MS2 rejection for the CCRO. MS2 rejection shown in Figure 23 shows median removal based on paired feed and permeate samples, with the error bar representing the standard deviation of paired MS2 removals. Table 18 provides the median and standard deviation of the feed and permeate samples for each MS2 event, with associated dates and runtimes.

Table 18. MS2 challenge test results for CCRO

Event Date	CRRO Runtime (hours)	Median Feed MS2 (pfu/mL)	Median Permeate MS2 (pfu/mL)	Median Calculated LRV
9/25/19	1,329	$1.0 \pm 0.1 \times 10^7$	$3.2 \pm 0.7 \times 10^2$	4.5 ± 0.1
10/30/19	2,080	$1.9 \pm 1.1 \times 10^7$	$5.0 \pm 1.9 \times 10^{5,a}$	1.6 ± 1.0[b]
12/11/19	2,839	$1.6 \pm 1.2 \times 10^7$	5.0 ± 3.2	6.5 ± 0.3
12/17/19	2,968	$2.1 \pm 0.1 \times 10^7$	1.0 ± 0.0	7.3 ± 0.0
12/18/19	2,977	$1.5 \pm 0.1 \times 10^7$	1.0 ± 0.0	7.2 ± 0.1

Note: triplicate samples collected and analyzed for feed and permeate sample locations.
Plus–minus (±) represents standard deviation from paired MS2 removals or standard deviation of the MS2 triplicate samples
[a] triplicate results were 5.0×10^5, 3.7×10^6, 1.0×10^5 pfu/mL, such that standard deviation LRV for this event was heavily skewed by one of the three permeate samples
[b] MS2 challenge testing conducted at the time rejection of surrogates was also low suggesting impaired membranes

The first CCRO MS2 challenge event took place on 09/25/2019 (1,329 runtime hours). At this time, the CCRO was providing high rejection of key surrogates (e.g., strontium rejection that day was 3.5 logs). The median MS2 log rejection for this event was 4.5 ± 0.1 LRV, which is higher than the rejection of all other surrogates sampled that day.

The following CCRO MS2 challenge test took place on 10/30/2019 (2,080 runtime hours) as the CCRO was experiencing integrity issues. MS2 rejection during this event was 1.6 ± 1.0 (95 percent confidence interval: [0.1, 2.3]). For this event, one of the three permeate triplicate samples was an order or magnitude higher in phage concentration (pfu/mL) and provided a large uncertainty in terms MS2 LRV for this event. Indeed, the elevated permeate sample was statistically different to the other samples per Student's t-test considering an alpha (α) of 0.05. One important observation from this challenge test was that rejection of TDS was only slightly lower when compared to the first MS2 event. TDS LRV rejection only dropped from 1.58 to 1.57 during the same timeframe, whereas median MS2 LRV dropped from 4.5 ± 0.1 to 1.6 ± 1.0. In contrast, strontium dropped from 3.5 to 2.0 for the same timeframe. This makes strontium a desirable surrogate since it indicates when integrity has been compromised.

Three additional MS2 challenge tests were performed once membranes were replaced to Hydranautics ESPA2-LD. All three challenge events indicated high MS2 rejection with LRVs ranging from 6.3 ± 0.3 to 7.3 ± 0.0 as shown in Table 18. During these events all monitored surrogates were conservative to MS2 rejection.

4.2.4. Cycles Assessments

Three cycle assessments were performed on the CCRO pilot. For these events, paired samples from the CCRO vessel permeate and feed-concentrate line were collected early, middle, and late within the CCD cycle. The CCD cycles had a duration of approximately 11 minutes during the course of this study. The "early" samples were simultaneously collected at approximately 1.5–3.5 minutes from the start of a CCD cycle. The "middle" samples were collected 5.0–7.0 minutes

from the start of a CCD cycle, and the "late" samples were collected 8.5–10.5 minutes from the start of a cycle.

The results for the three cycle assessments are shown in Figure 25. The first cycle assessment took place on 10/30/2019 (2,080 runtime hours) as the membranes were undergoing integrity issues as previously mentioned. As a result, the removal of strontium, magnesium, and sulfate were capped at approximately 2.5 logs for samples collected at different times during the CCD cycle. MS2 rejection was also affected, providing rejection no greater than the surrogates for this event. This shows that the surrogates did not overestimate MS2 removal when membranes were impaired and sampled within the CCD cycle. Of note, rejection of TDS and EC was not as affected as the more sensitive surrogates, making them less ideal to detect impaired membranes. For the 10/30/2019 event, one of the triplicate permeate MS2 samples from the "early" CCD cycle was an order of magnitude higher than the other two samples even though all three samples were taken from same sample recipient (i.e., 2L beaker). This single permeate sample caused the paired LRV standard deviation to be higher than the "middle" and "late" data sets (i.e., 2.4 ± 0.8, 95% confidence interval [0.7, 2.9]). Additionally, further evaluation indicated that this one permeate sample was statistically different than the other two by using the Student's t-test considering an alpha (α) of 0.05.

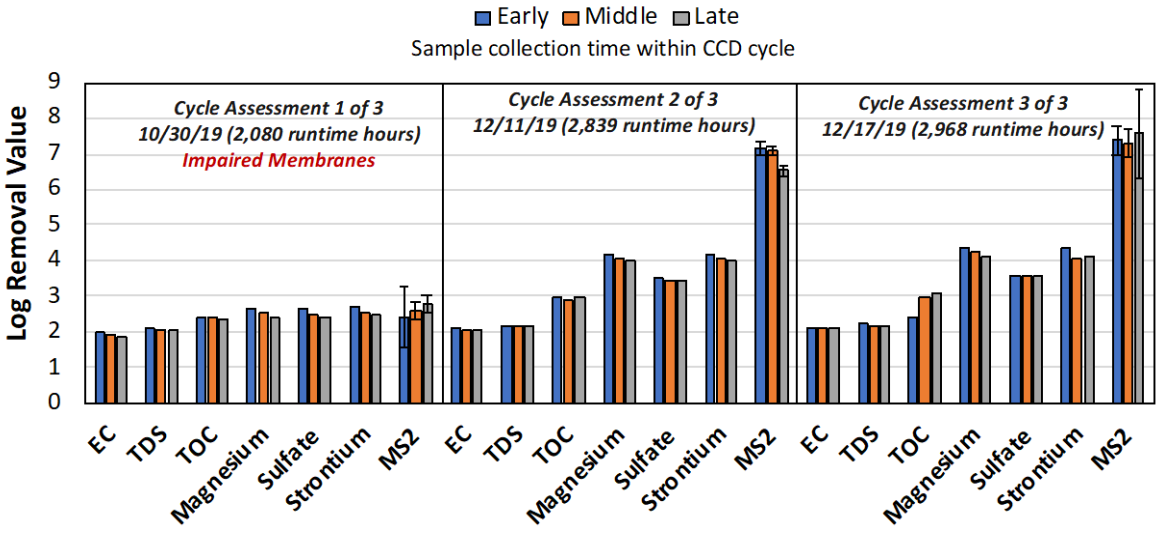


Figure 25. CCRO log removal values of surrogates and MS2 during cycle assessment

Two additional cycle events were performed on the ESPA2-LD membranes, on 12/11/2019 (2,839 runtime hours) and 12/17/2019 (2,968 runtime hours). These two events were performed on membranes representative of intact conditions, as supported by rejection of routinely sampled surrogates and online permeate conductivity. The rejection of surrogates and MS2 remained relatively unchanged at different times within the CCD cycle (i.e., early, middle, and end). Notably, rejection of MS2 was no less than 6.6 logs for samples collected throughout the CCD cycle, such that all surrogates provided a conservative estimate relative to MS2 rejection where highest strontium LRV during the cycle assessments was 4.3 logs. Overall, the cycle assessments served to support three important concepts:

- Rejection of surrogates and MS2 was not reduced as feed concentration increased during CCD cycles
- Surrogates remained conservative to MS2 rejection during the cycle assessment performed on intact membranes
- Reduced rejection was observed for more sensitive surrogates (e.g., strontium) when the cycle assessment was conducted on impaired membranes

4.2.5. Compromise Testing

Compromise testing was conducted on 12/18/2019 (2,977 runtime hours) to evaluate the ability of surrogates to track virus rejection during a compromised condition. A total of three compromise conditions were performed which focused on the removal of O-rings from the permeate interconnector, concentrate endcap, and feed endcap. MS2 was added to the CCRO feed water and allowed CCRO to reach steady state prior to sampling for each compromise condition. In preparation for each compromise test, the system was shut down and pressure vessel was opened to remove O-rings. Figure 26 provides the locations where O-rings were removed during compromise testing. One condition was tested at a time, i.e., they were not all tested at once.

Figure 26. Compromise conditions tested on CCRO

Results for CCRO compromise testing are shown in Figure 27. For the control condition, rejection of surrogates was conservative to MS2. For every compromise performed rejection of surrogates as well as MS2 dropped to approximately 0.2–0.4 logs. These results suggest two important findings:

1. CCRO integrity is highly sensitive to the removal of O-rings from the locations tested (interconnectors and endcaps).
2. All surrogates accurately tracked the reduction of MS2 rejection when the O-rings were removed, showing their ability to indicate when membrane integrity has been compromised.

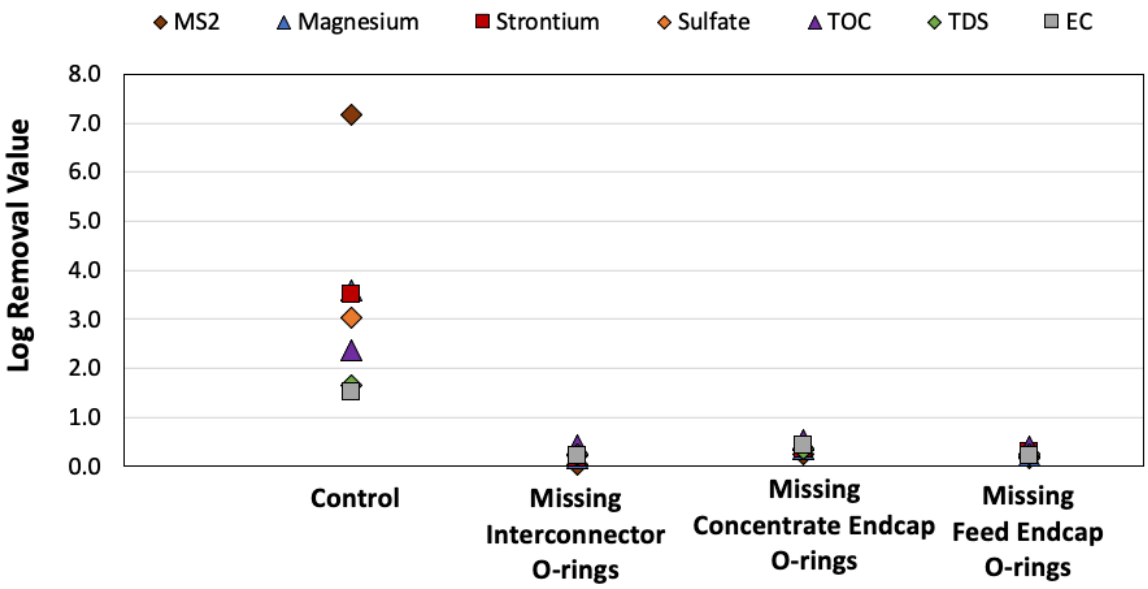


Figure 27. Rejection of surrogates and MS2 during CCRO compromise testing

5. Conclusions

The goal of testing was to evaluate naturally occurring candidate surrogates that have potential to increase virus removal credits for the RO membrane treatment process. If a new surrogate is implemented, a project can continue monitoring online TOC as supplemental organics removal monitoring and to serve as a back-up for virus removal credit in the event that, for any reason, the new surrogate(s) were not available. The findings from OCWD testing showed that free ATP, fluorescence Peak C, sulfate and strontium are four possible surrogates that can be used for this purpose which all demonstrated average LRVs that exceed current typical LRVs achieved by use of TOC or EC. Strontium, sulfate and free ATP are naturally occurring surrogates that showed the highest removal by the RO system with average LRVs of 3.29, 2.97, and 3.03, respectively, with strontium and sulfate determined from grab sampling and free ATP measured online. However, online strontium and sulfate analysis technologies (separate instruments) have recently become available and thus could be considered if preferable over grab samples. Online fluorescence Peak C was also noteworthy but more conservative with an average LRV of 2.70.

In the case of online free ATP, the minimum observed LRV was 2.60 such that it was always at least 0.5 log above the current TOC-based LRV credit for OCWD AWPF RO of ~2.0 LRV. Irrespective of the minimum daily value, it is expected that a plant could base credit on the average daily LRV (for free ATP or other online surrogates) as is currently permitted for OCWD for TOC-based virus LRV credit. For free ATP, the observed average daily LRV ranged from 2.75 to 3.13, representing a minimum ~0.75 log increase over current use of TOC at ~2.0 LRV.

Online fluorescence features much less expensive instrumentation that is easier to calibrate and maintain compared to ATP (or online sulfate or strontium). The minimum observed LRV in this study was 2.27, while the average daily LRV ranged between 2.50 and 2.88, indicating that for OCWD this surrogate may not have much advantage over current use of TOC at ~2.0 LRV (i.e., approximately 0.5 log credit increase). On the other hand, OCWD leadership has noted that even a 0.5 log increase in credits for any unit process is meaningful and may be worth pursuing if available despite added cost or complexity. Further, other facilities may exhibit higher RO feed fluorescence, leading to perhaps more potential for this surrogate, as with any surrogate.

With respect to naturally occurring ions, LRVs for sulfate and strontium were very stable and maintained in a narrow, steady range compared to the higher observed variability in LRV for ATP and fluorescence. Strontium shows the greatest LRV for potential virus credits but would require a permanent program of frequent grab sampling or validation of an online analyzer. Sulfate is the next-highest LRV and would also require a permanent program of frequent grab sampling or validation of an online analyzer. Assuming online strontium or sulfate instrumentation performance is acceptable, the data from this study featuring high-frequency (hourly) strontium and sulfate grab samples can be considered as a simulation of the potential online data. The very stable concentrations (and LRVs) over the sampling period suggest that frequency of online sampling for strontium or sulfate could be minimized, i.e., daily or every few

hours depending on regulatory requirements, which could reduce instrument maintenance and operating costs.

Based on this study, in their recent Title 22 Engineering Report related to permitting the GWRS Final Expansion, OCWD proposed to use on-line ATP, sulfate, or strontium as the primary surrogates in a tiered approach for performance indicators for virus LRV credit by the RO process for the GWRS and GWRS Final Expansion. The implementation of the monitoring programs is still being finalized, but either free ATP or sulfate or strontium analyzers are proposed to be installed on a permanent basis. On-line analyzers installed on the common headers (bulk) of the RO feed and RO permeate streams are proposed to measure free ATP, sulfate, or strontium concentrations continuously and track RO performance.

In contrast to the conventional three-stage RO system (85 percent recovery) tested at OCWD AWPF, the testing at Padre Dam served as an opportunity to evaluate RO performance surrogates for an alternative CCRO system at 95 percent recovery, and for intact and compromised RO membrane conditions. Strontium concentration was high enough in the CCRO feed to detect it in the CCRO permeate. During intact conditions, strontium LRV was greater than 3.4 (slightly greater than average for OCWD AWPF RO unit, which was 3.3), whereas MS2 LRV was between 4.5 and 7.2. One of the compromises was planned and two were unplanned. The planned integrity compromise event was conducted by removing O-rings to which all surrogates responded, including MS2. The other two unplanned compromises gave an opportunity to track the response of surrogates for a relatively slow developing compromise. More sensitive surrogates, such as strontium, were successful in capturing the compromise well in advance when compared to more traditional surrogates (e.g., EC and TOC). Based on this study, Padre will be pursuing a tiered approach to demonstrate RO integrity using strontium as the primary surrogate with TOC and EC as backup surrogates.

5.1. Recommended for Future Research

Future research of surrogates should consider measuring additional data for 1) different seasons, since variation in influent quality (concentration of surrogate) can influence LRV, and 2) different RO membrane models to confirm results over a broader range of conditions. Although multiple seasons were sampled in the present study for OCWD, the California climate is more temperate. For CCRO testing, membrane model should be confirmed with membrane manufacturer to ensure it is suitable for the CCRO process.

Validation of online measurement for strontium and sulfate should be pursued for potable reuse applications, as these technologies have recently become available. For example, the Xact 920 continuous water analyzer from Cooper Environmental uses non-destructive X-ray fluorescence (XRF) for elemental analysis of aqueous solutions including strontium, and the 850 Professional IC from Metrohm is coupled with an extension module for liquid handling for online capabilities including sulfate. Although grab sampling can also be suitable, online analysis may be preferable.

More optimization of nanoparticle analysis is desirable to ascertain whether such a method could be suitable for RO LRV credit towards virus or for other water quality monitoring purposes.

Depending on the technology, the detection limit for size for these technologies is approaching the smallest viruses, e.g., MS coliphage, which is promising. It remains to be seen whether the detection limit in terms of concentration can reach low enough levels for an RO application, and there are questions around how to calculate LRV (i.e., particle counts would be available for a range of multiple particle sizes in a single measurement).

Overall, while increasing the LRV credit by up to ~0.5 to almost 1.5 logs over current use of TOC (~2.0 LRV) is a significant improvement as was achieved in this study, future research may identify other surrogates that can approach a removal value closer to the ~6 log LRV expected for viruses by RO treatment. However, this seems to be a challenging task simply due to the relatively high quality of the RO feed water (which is generally pre-treated micro- or ultrafiltered effluent) such that no single molecular marker or bulk parameter would be expected to occur at a concentration that is orders of magnitude (i.e., 6 logs) above its current detection limit in the RO permeate, as necessary for a large log removal value to be continuously observed. That said, there continue to be method improvements such as lower detection limits. This will help to demonstrate higher LRV for constituents highly rejected by RO.

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Appendix A

Autopsy of Padre CCRO membranes

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Avista TECHNOLOGIES
a Kurita company

Membrane Autopsy Report

Completed for:

Trussell Technologies, Inc.

Padre Dam

Serial Number T4048676

Lead Element

02/20/2020 WO#120219-3

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Table of Contents

Executive Summary

Background

Trussell Technologies provided three reverse osmosis (RO) elements to Avista Technologies for analysis. Elements Serial Number (SN) T4048676 was removed from the lead position, element SN T4048771 was removed from the middle position and element SN T4048772 was removed from the tail position. The following is a summary of the results pertaining to element SN T4048676 (lead).

Initial Element Testing

Element SN T4048676 produced normal flow, normal rejection (99.4%) and a differential pressure of 6 psi during initial wet testing. The element passed integrity testing suggesting the absence of damage to the internal components of the spiral wound element.

External Inspection

The external components (fiberglass casing, brine seal, anti-telescoping devices (ATDs), permeate tube) were in good mechanical condition.

Internal Inspection

The scroll ends were in good mechanical condition and free of debris. Tan-colored foulant was noted in primarily the feed spacer contact points. Additionally, random spots were observed across the membrane on all membrane leaves. Foulant was observed on the feed spacer material but it was free of blockages. The membrane backing contained brown colored foulant that corresponded to the spots observed on the membrane surface.

Foulant Analysis

The wet foulant density was measured at 0.05 mg/cm^2. The organic content was 78% indicating the bulk of the foulant was organic. Microbiological analysis identified bioslime, algae, yeast and bacteria and aerobic bacteria counts were 1000 CFU/cm^2 after 72 hour incubation period. Fourier Transform Infrared (FT-IR) spectroscopy confirmed the presence of organics by detecting bands associated with bioslime, microorganisms and lipids (hydrophobic fatty acids).

Energy Dispersive Spectroscopy (EDS) detected low concentrations of silicon and aluminum on the membrane. Chromatic Elemental Imaging^{SM} (CEI^{SM}) identified patches of organic across the membrane surface with clay deposits embedded in and above the organics.

Based on the analysis the bulk of the foulant consisted of organics.

Flat Sheet Performance and Cleaning Study

Flat sheet samples harvested from the full element produced normal permeability and normal salt passage during baseline cell testing. Cleaning the flat sheet with RoClean P112 (2% by weight, 2 hrs heated cleaning solution) removed the bulk of the foulant and increased water passage by 10%.

Flat Sheet Damage

Fujiwara testing was negative for the presence of halogen (e.g. chlorine) oxidation. However, dye testing revealed dye passage within the spots observed on the membrane surface. The damaged areas were very circular in shape indicative of a reaction between a metal and an oxidizer. Further analysis with EDS and CEI showed iron in the damaged areas. Based on these observations the most likely cause for the damage observed is a reaction between metals and an oxidizer.

Initial Element Test

Element Weight

All elements are weighed prior to autopsy as weight is often indicative of the degree of fouling. New eight-inch elements weigh approximately 30 to 35 pounds.

SN T4048676 weighed 32 pounds.

Full Element Wet Test

Test results were normalized to the manufacturer's published test conditions.

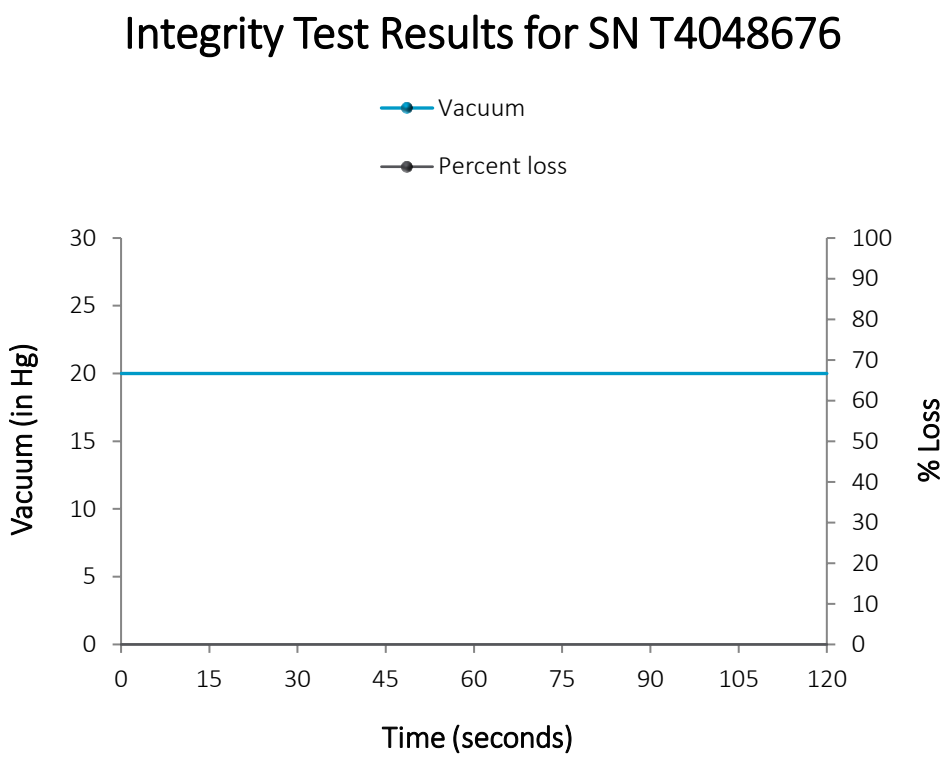
Filmtec BW30XFR-400/34	Flow (gpm)	Rejection (%)	Pressure Drop (psi)
SN T4048676	7.89	99.4	6
Manufacturer's Specifications	6.79 to 9.19	99.4 to 99.7	≤15

Element wet testing

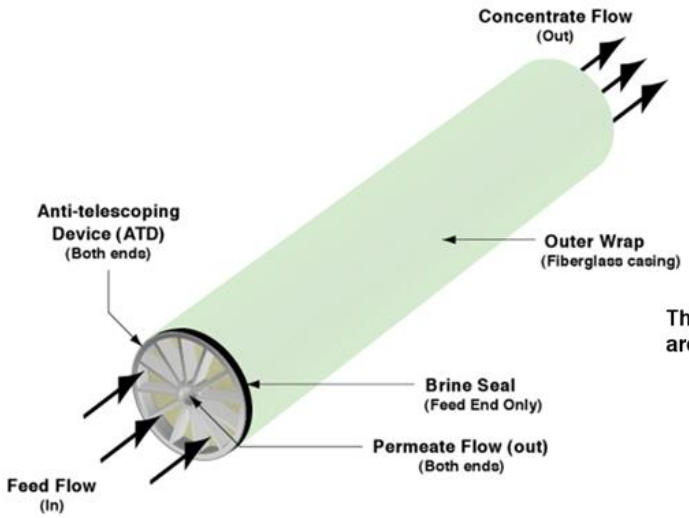
Integrity Test

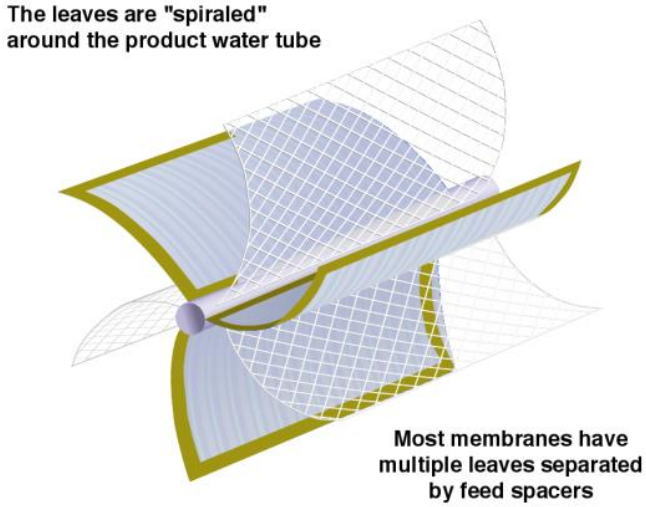
Integrity testing is performed to identify mechanical damage to the internal components of the spiral wound element. In this test, a vacuum of approximately 20 inches Mercury (in. Hg) is applied to the permeate side of the membrane and the membrane is then sealed. The vacuum is monitored for a duration of 120 seconds. Any loss of vacuum indicates the presence of damage; however, losses of over 35% of the vacuum within the 120 second period suggests severe physical damage.

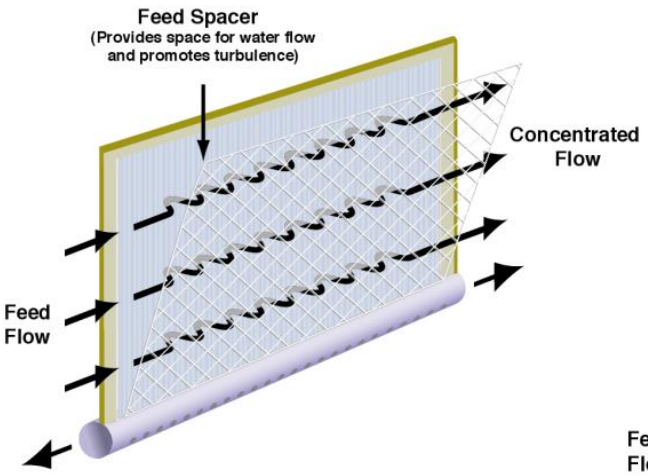
The element passed integrity testing.

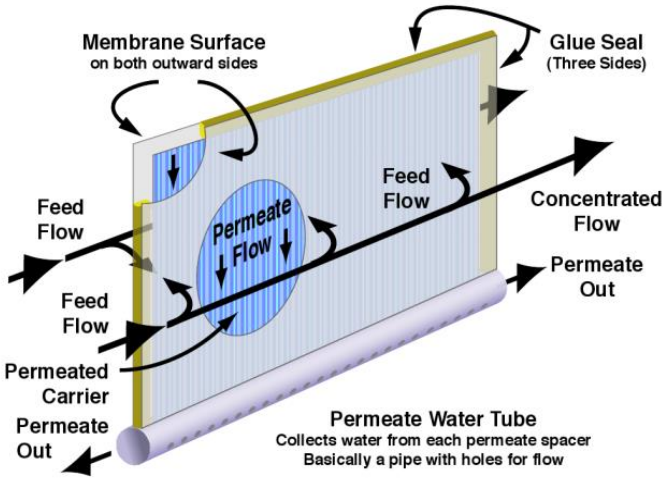


Membrane Construction Diagrams







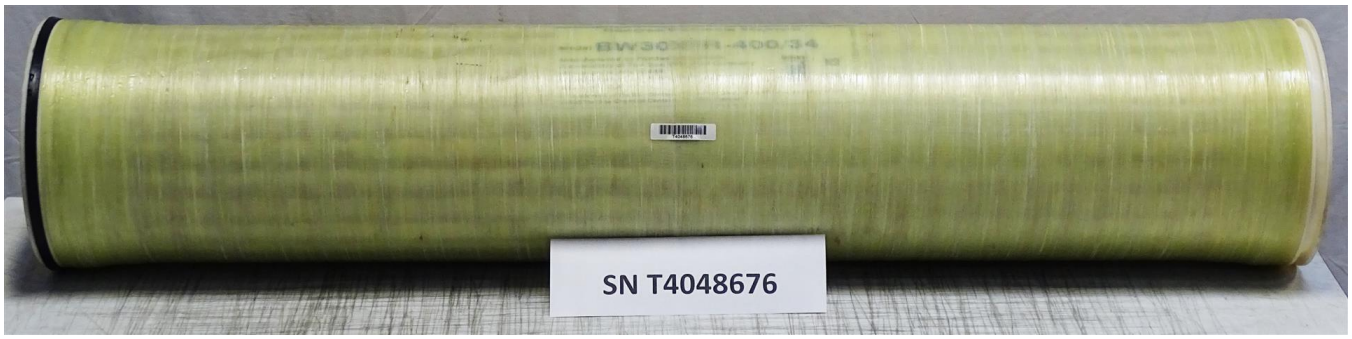


External Inspection

Fiberglass Casing

The purpose of the fiberglass casing is to ensure that the various membrane components are held in their correct position for optimum performance. Damage to the casing can be an indication of rough handling or damage from excessive differential pressure across the element from heavy fouling.

The fiberglass casing was in good mechanical condition.



Fiberglass casing of SN T4048676

Brine Seal

The brine seal is used to seal against the inside diameter of the pressure vessels and the outside diameter of the membrane to ensure that all the feed water passes through the element. Feed water passing on the exterior of the element can result in higher pressures, which can cause cracking of the fiberglass casing.

The brine seal was in good mechanical condition.

Permeate Tube

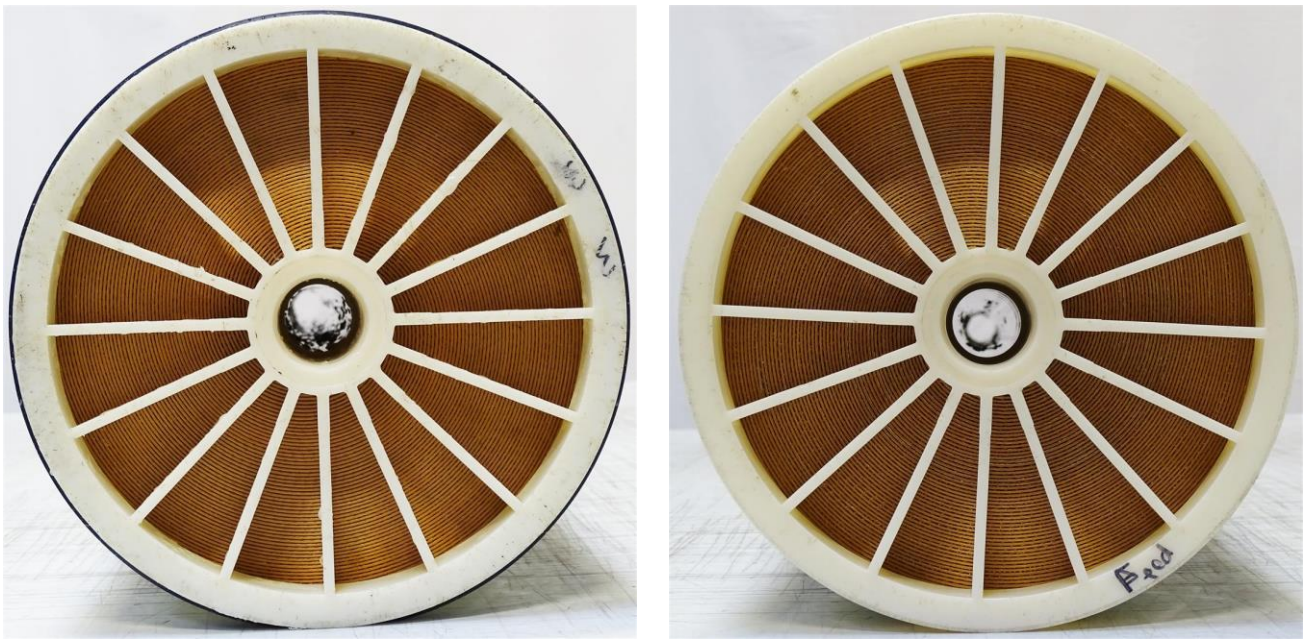
The permeate tube is a pipe that is located at the center of the element. It contains lines of holes and is bonded to each membrane leaf, allowing permeate water to travel from the leaves into the permeate tube to be collected. Damage to the ends of the permeate tube can lead to o-ring failures, causing bypass of feed or concentrate water into the permeate stream. Cracking of the permeate tube can also result in permeate contamination.

The permeate tube was in good mechanical condition.

Anti-Telescoping Devices (ATDs)

The function of the ATDs is to stabilize the components of the element. This helps to prevent shifting of the internal mechanical components under pressure, also known as telescoping. Telescoping may still occur if pressures exceed the manufacturer's specifications.

The ATDs were in good mechanical condition.



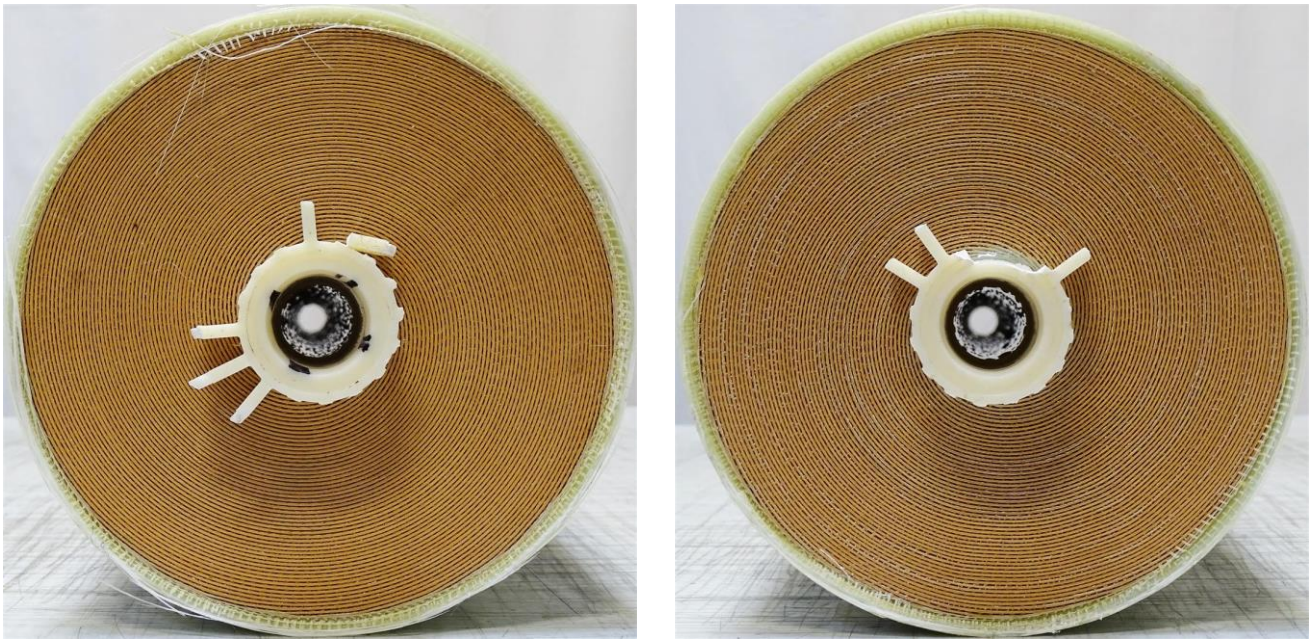
Feed (left) and concentrate (right) ATDs of SN T4048676

Internal Inspection

Scroll Ends

The ends of the element are called scroll ends. They are examined for the presence of foulant debris and mechanical damage (e.g. gapping, feed spacer extrusion). The presence of foulant on the scroll ends can cause elevated delta pressures while gapping and feed spacer extrusion indicate uneven hydraulics (high flow/low flow regions). In addition, each scroll end is examined for telescoping, the gradual axial shift of the membrane leaves from the outer diameter of the element towards the permeate tube. Telescoping is often caused by the development of high differential pressure (greater than the manufacturer's specification) across the element or when pressure is applied too quickly, causing a water hammer effect.

The scroll ends were in good mechanical condition.



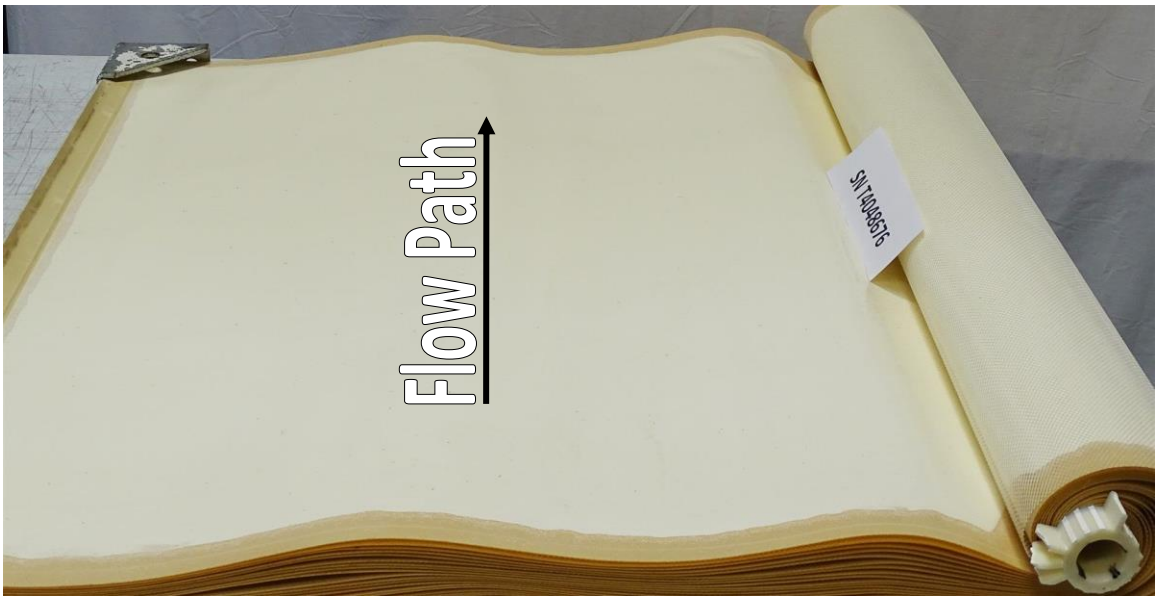
Feed (left) and concentrate (right) scroll ends of SN T4048676

Membrane Surface

New membrane surfaces are uniform and shiny. Foulant can often be detected through visual examination; however, membrane appearance can be misleading as some foulants are not visible. The presence of foulant on the membrane surface can cause elevated delta pressure, loss in flow and damage if the foulant is abrasive. Additionally, the membrane surface is inspected for damage such as delamination. Delamination is the lifting of the thin-film membrane from the support layer and often occurs due to a positive pressure on the permeate side of the element.

Tan-colored foulant was noted in primarily the feed spacer contact points. Additionally, random spots were observed across the membrane on all membrane leaves.

Exposed membrane surface of SN T4048676



Exposed membrane surface from feed end of SN T4048676

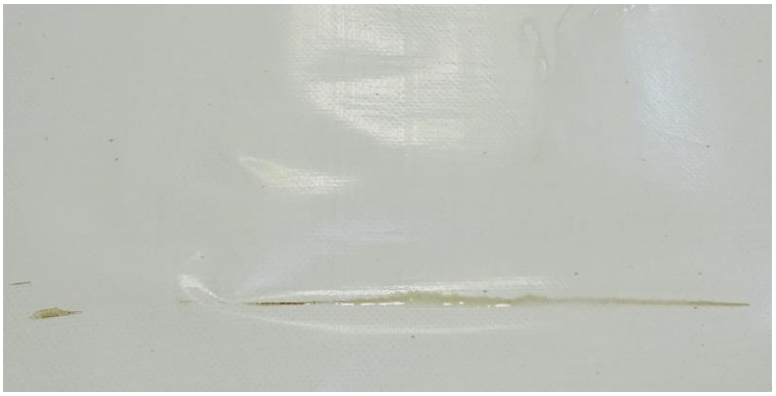


Image of foulant scraped on the membrane surface

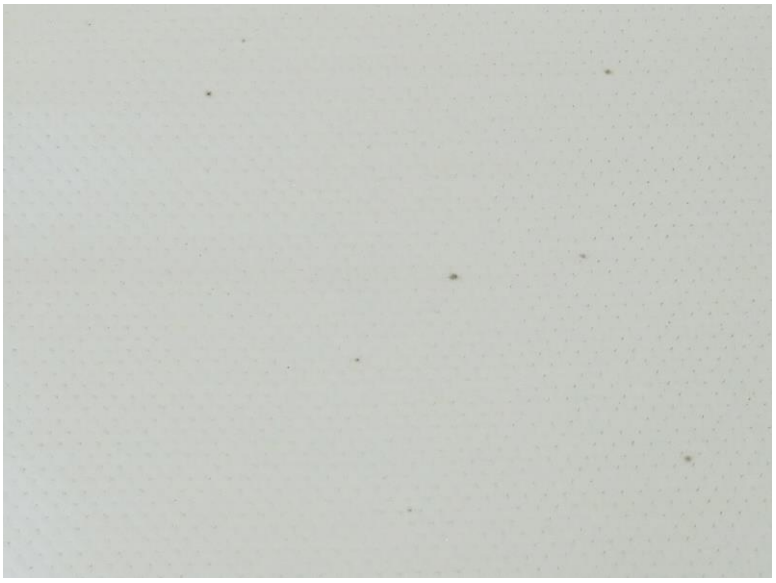
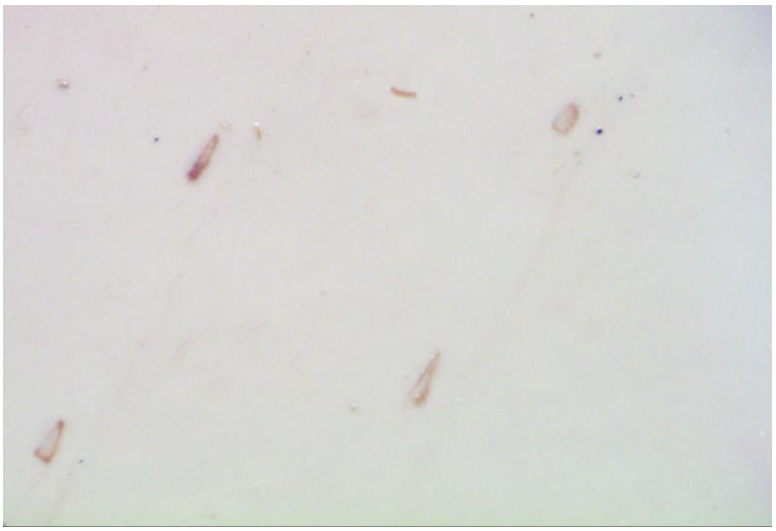
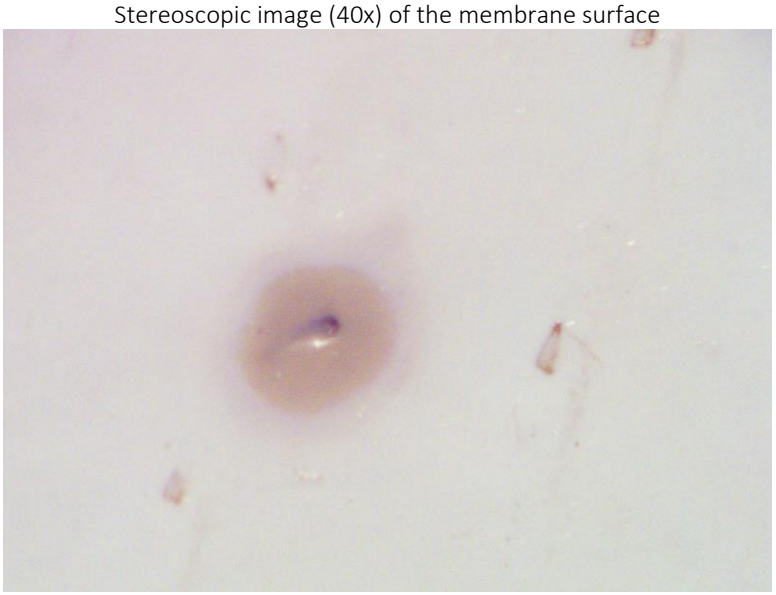


Image of spots observed on the membrane surface



Stereoscopic image (40x) of the membrane surface

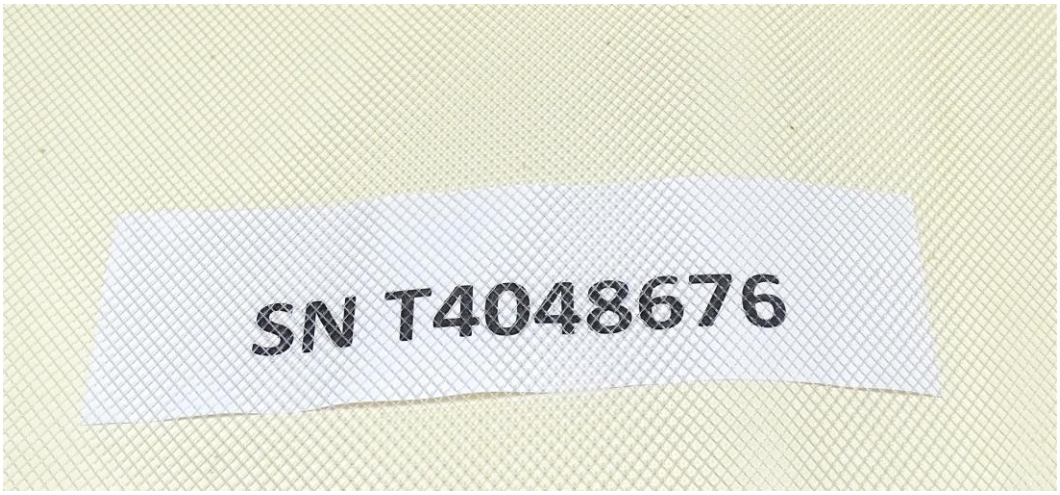


Stereoscopic image (40x) of a spot on the membrane surface

Feed Spacers

The feed spacer is a plastic net material designed to separate the membrane leaves, forming a flow path, and to promote turbulence within the feed water channels. Foulant blocking the feed channels causes more resistance for the feed water flowing through the element and results in higher than normal delta pressures.

Although some of the foulant was observed on the feed spacer they were in good mechanical condition and free of blockages.



Feed spacer of SN T4048676

Glue Lines

Membrane leaves are glued on three sides to separate the feed and permeate streams. The glue lines are inspected for specific damage, including glue flaps and pouching. Glue flaps refer to excess inactive membrane material located closest to the ends of the element. Flaps found on the feed end of the element can flare during operation, blocking the feed channels on the scroll end, potentially causing increased differential pressure. Pouching of the glue line, which is often a result of delamination, allows feed water to pass through the inactive membrane at the glue line, contaminating the permeate stream.

The glue lines were in good mechanical condition.

Permeate Carriers and Membrane Backing

The permeate carriers provide a path for permeate water to flow towards the permeate tube, which minimizes permeate-side pressure losses. New permeate carriers and membrane backing are uniform in color. Foulant found on the permeate side of the membrane leaves indicates contamination of the permeate stream.

Tan-colored spots were observed on the membrane backing and permeate carriers.

Membrane backing of SN T4048676

Foulant Analysis

Acid Testing

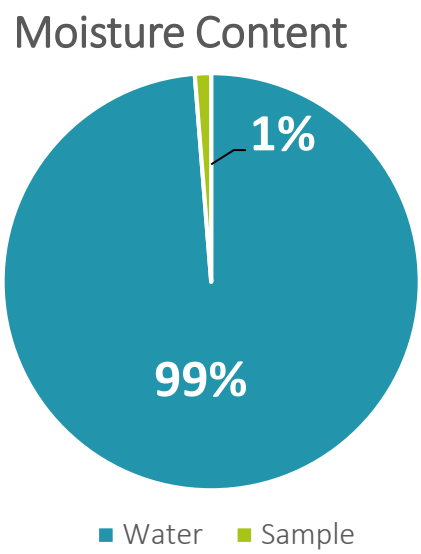
Acid testing is used to determine the presence of carbonates and metals on the membrane surface. In this test, several drops of dilute hydrochloric acid (HCl) were placed on the foulant surfaces. Effervescing indicates the presence of carbonates while a color change is associated with the presence of metals.

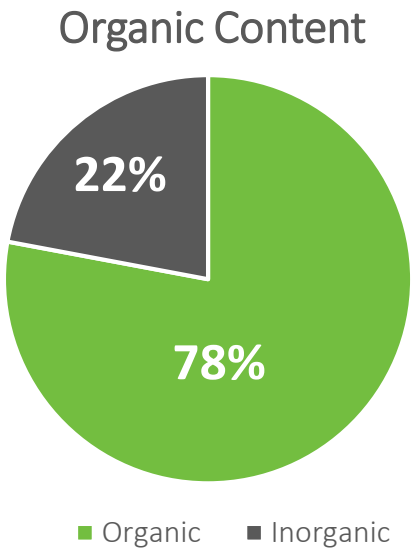
Acid testing was negative for the presence of carbonates and metals.

Foulant Density Measurement and Composition Testing

A sample collected from a known area of the membrane surface is weighed before and after drying to determine the foulant density (reported as dry foulant density – mg/cm^2) and moisture content of the sample. Different types of foulant materials exhibit higher moisture contents. Relative water concentrations greater than 95% indicate an extremely hydrated, biological material. Alternatively, scales (crystalline material) typically contain very little moisture. The organic content of the dehydrated material is then measured through loss on ignition (LOI) testing. If the organic content of the total solids is greater than 65%, it is considered primarily organic.

The wet foulant density was measured at 0.05 mg/cm^2. The moisture and organic contents are shown in the graph below.

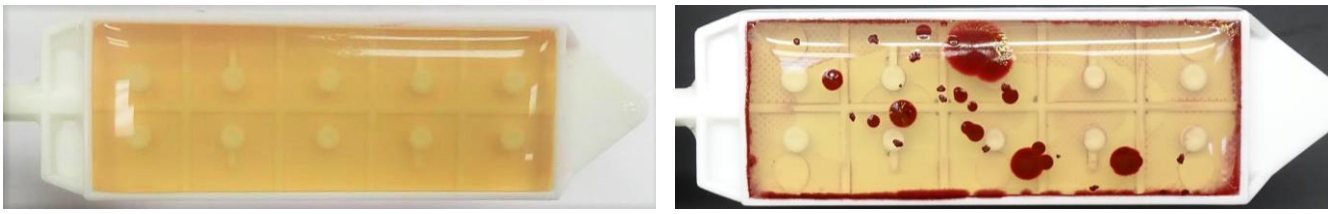




Biological Activity Testing

Dip slides for aerobic bacteria are exposed to the foulant material on the membrane surface. The slides are incubated for 72 hours and inspected for biological growth. Greater colony density, measured in colony-forming units (CFU)/cm^2, indicates a more biologically active sample.

The aerobic slide showed approximately 1000 CFU/cm^2 after the 72 hour incubation period.

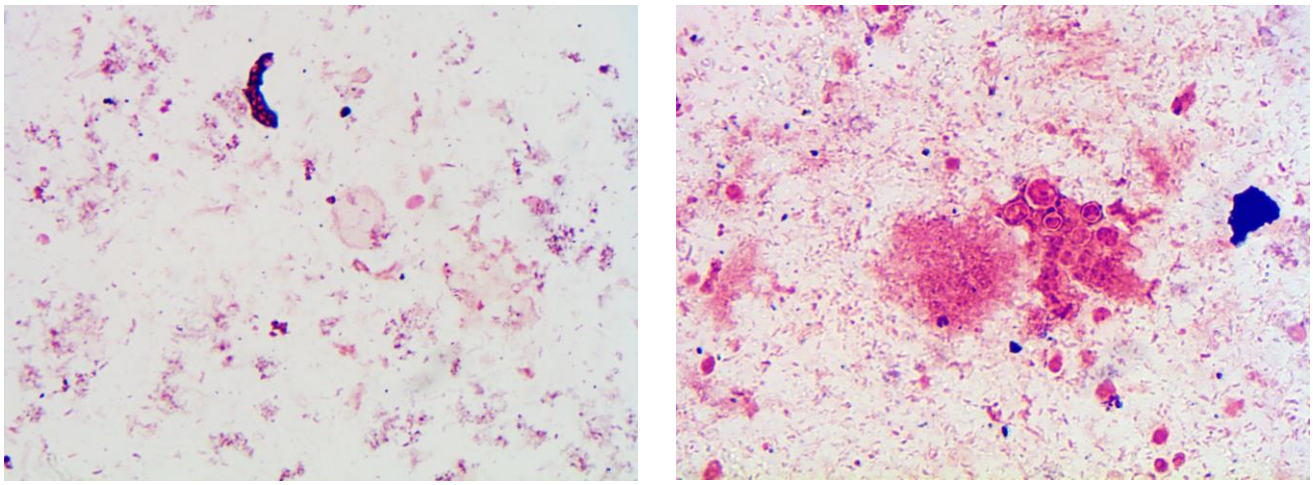


Aerobic bacteria slide before incubation (left) and after incubation (right)

Microbiological Analysis

This analysis is performed to identify microbiological components of the foulant removed from the membrane surface. Foulant samples are stained and examined with a light microscope at 1000x using an oil immersion lens. Gram positive bacteria are stained purple while Gram negative bacteria are stained pink.

Microbiological analysis performed on foulant scraped from the membrane surface identified bioslime, algae, yeast and bacteria.

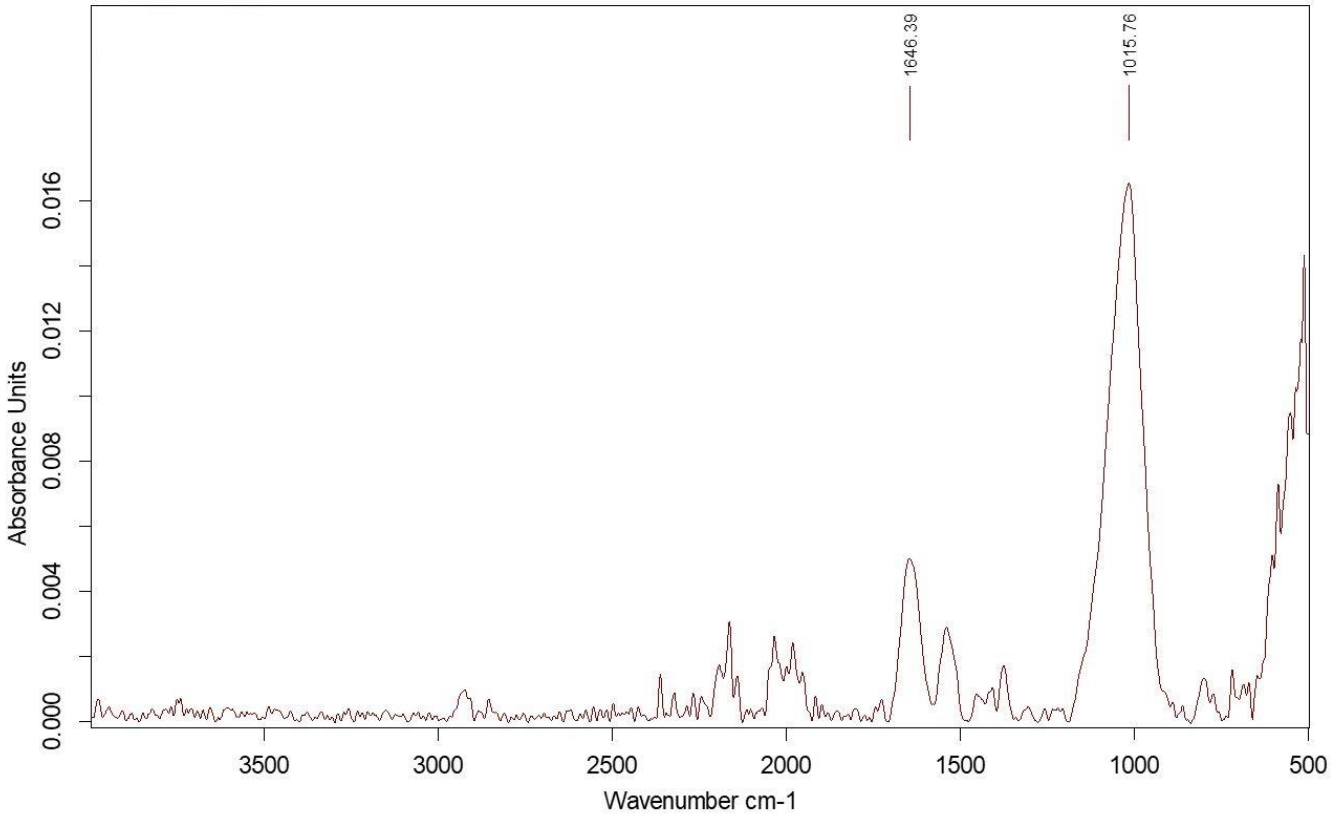


Light microscope images (1000x) of foulant scraped from SN T4048676

Fourier Transform Infrared Spectroscopy Analysis

Fourier Transform Infrared Spectroscopy (FT-IR) is an analytical technique used to identify functional groups (specific groups of atoms or bonds within molecules). Infrared radiation passes through a sample, with some of the radiation absorbed and some transmitted. A measurement and interpretation of this data produces a spectrum which can then be compared and matched to the known spectra for functional groups based on the wavenumber at which bands appear and their respective shapes (e.g. sharp, broad, strong, weak).

FT-IR spectroscopy performed on foulant scraped from the membrane surface displayed a strong, sharp peak at approximately 1000 cm^{-1} which is associated with organics (e.g. carbohydrates) and silicon-oxygen (Si-O) bond stretching (e.g. silica/clays). The double peak between 1650 and 1500 cm^{-1} is associated with amino acids (i.e. proteins) from microorganisms. The weaker bands between 2200 cm^{-1} and 2000 cm^{-1} are contributed by lipids (hydrophobic fatty acids).



FT-IR spectral image of foulant removed from the membrane surface of SN T4048676

Energy Dispersive Spectroscopy (EDS) Analysis

Energy Dispersive Spectroscopy analysis is used to determine the relative concentration of elements present in a sample. EDS analysis is performed on a dry membrane sample. The element sulfur is at least in part associated with the membrane support material (polysulfone) rather than a foulant layer. Avista's analysis of new membranes typically detects between 5.00 and 7.00 weight percentage. Relative concentrations below 5.00 percent indicate the presence of a foulant layer masking the membrane surface.

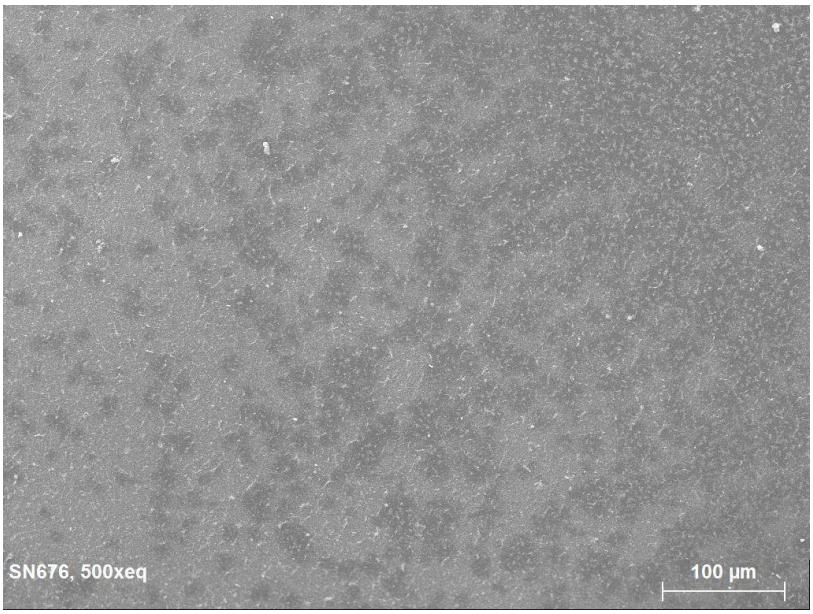
EDS analysis identified only detected trace amounts (<0.50 wt%) of aluminum and silicon as the inorganic elements present in the foulant layer. Although the carbon weight percent is associated with the membrane materials, a portion may be contributed by foreign organic material.

Elements	SN T4048676 Weight Percent
Carbon	77.13
Oxygen	16.07
Sulfur	6.42
Aluminum	0.12
Silicon	0.11

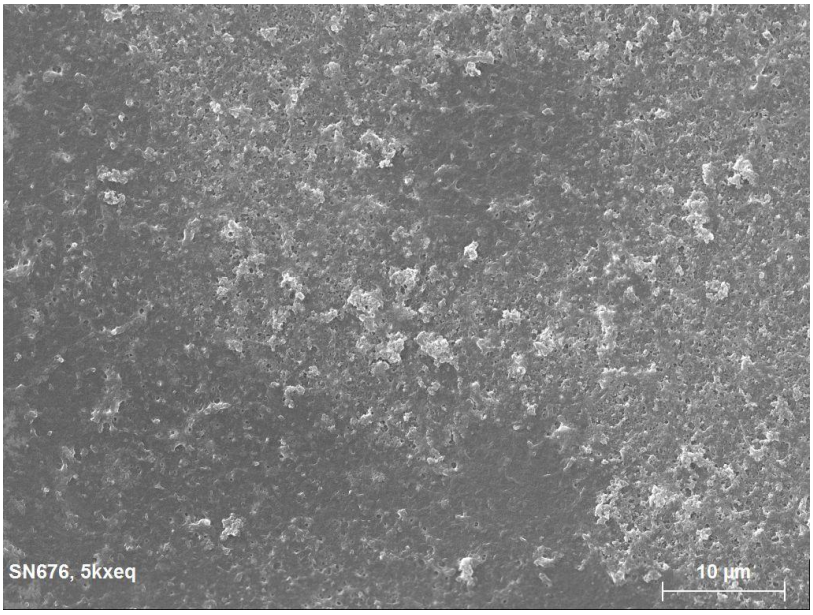
Scanning Electron Microscope (SEM) Imaging

SEM imaging is performed on the membrane surface to observe the topography of the foulant material. Foulant morphology can be an indicator of the type of foulant.

SEM images displayed patches of smooth foulant material across on the membrane surface. Particles were observed randomly across the membrane and embedded in the smooth organics.



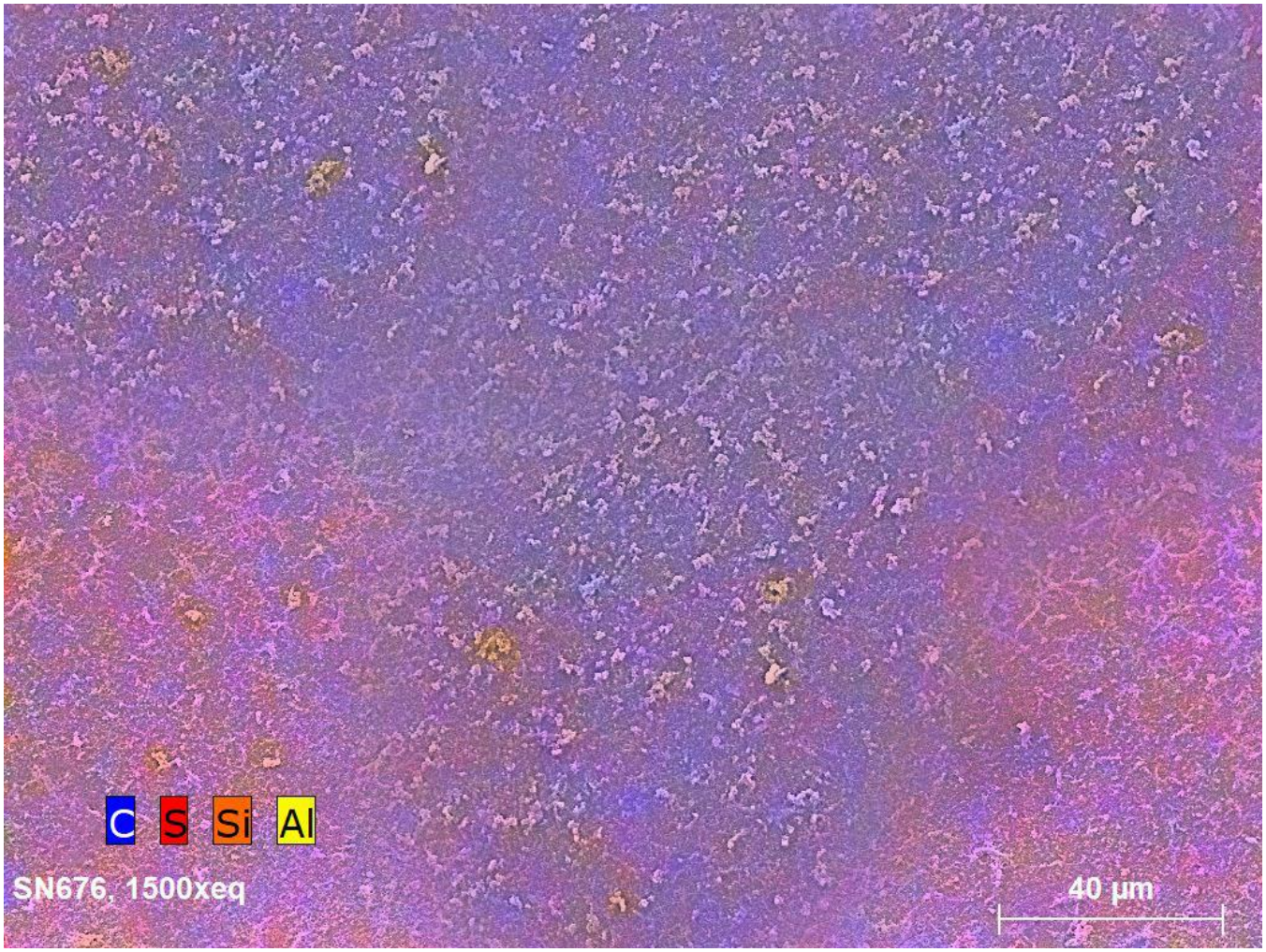
SEM image (150x) of the membrane surface of SN T4048676



Close-up SEM image (5000x) of the membrane surface of SN T4048676

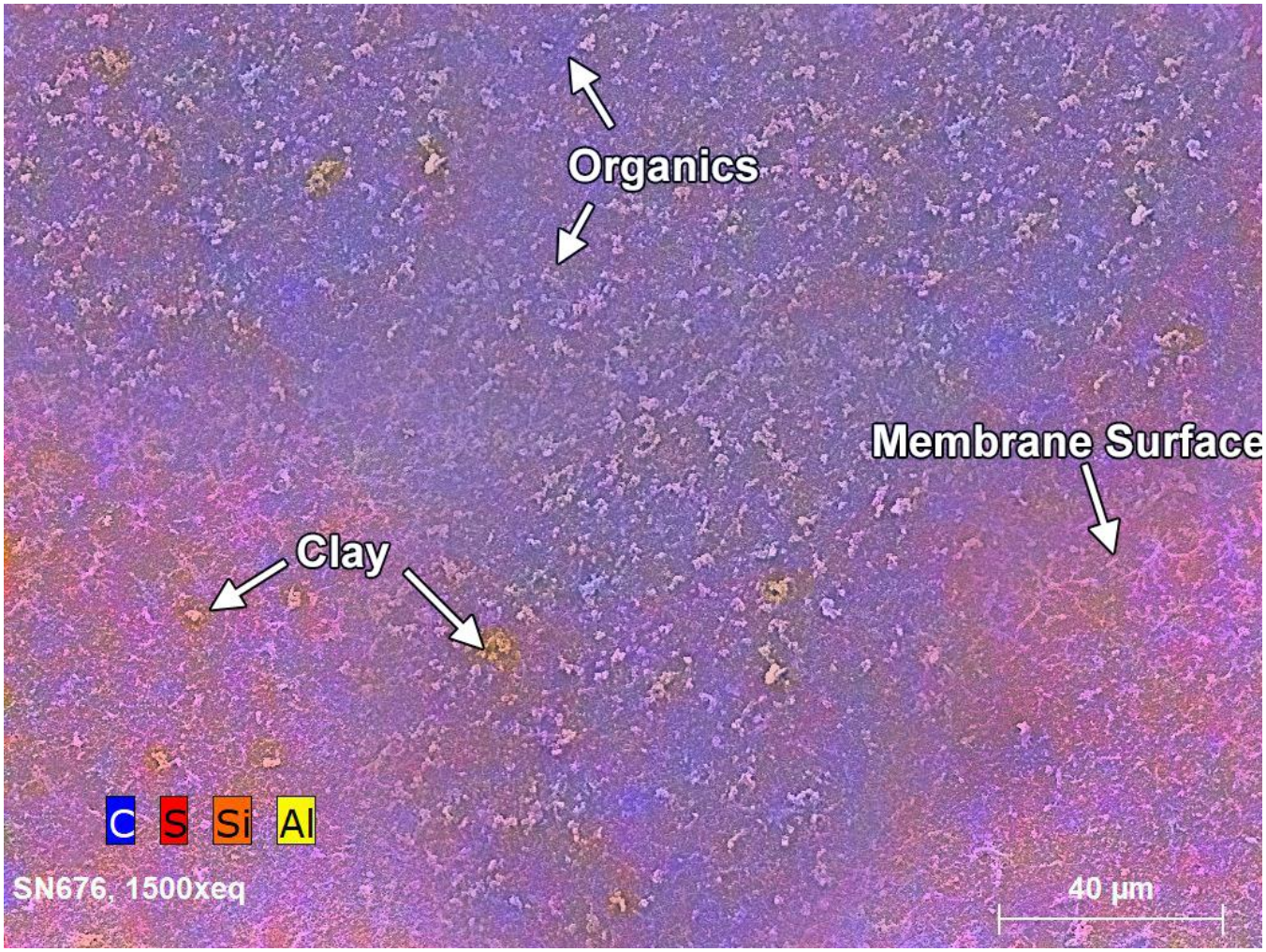
Chromatic Elemental Imaging℠ (CEI℠)

CEI is a high-resolution imaging technique used to determine the spatial distribution of elements in a foulant sample. Each element is assigned a color (shown in a legend on the bottom left corner of the CEI image) and the colors correspond to the location of the elements in the sample. An element's color intensity is associated with its concentration in the sample (i.e. elements present with higher relative concentrations are displayed with greater color intensity in the image). Additionally, a blending of colors signifies a compound (material composed of more than one element such as calcium carbonate).



CEI image (1500x) of the membrane surface

CEI[SM] showed organic foulant, denoted by a high carbon content (dark blue) in patches on the membrane surface. Clay (yellow-range) particles were visible within and above the organics. The membrane surface, represented by sulfur (red), was visible in areas where the organic layer was relatively thin.



CEI image (1500x) of the membrane surface with labels

Flat Sheet Performance and Cleaning Study

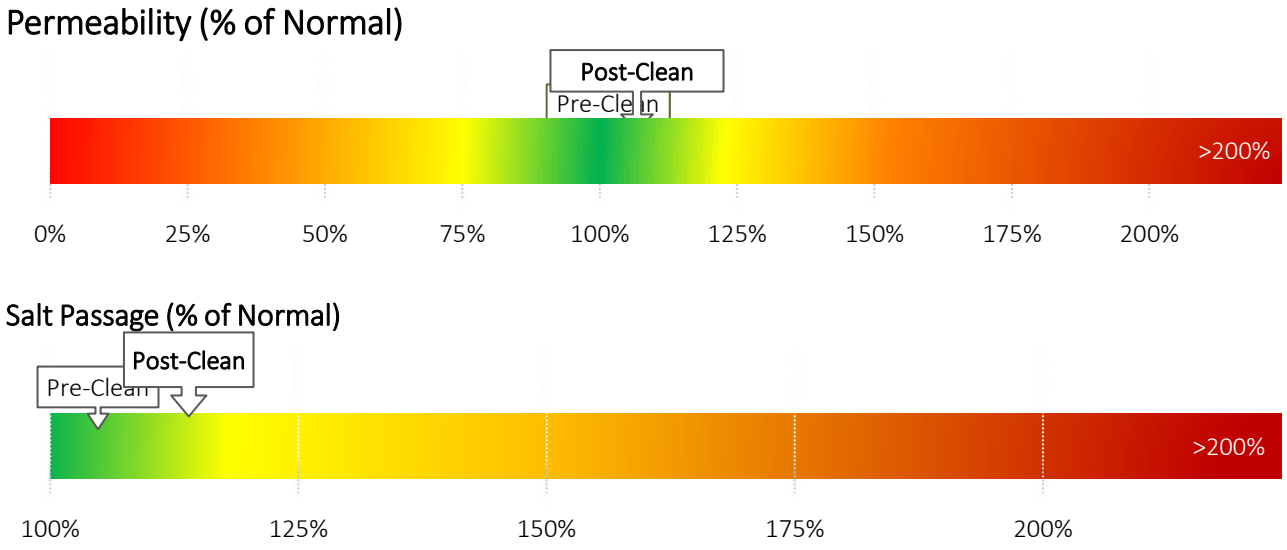
To evaluate flat sheet performance, membrane samples harvested from the full element are tested for permeability and salt passage. The raw flow and conductivity measurements from the test are used to calculate the permeability and salt passage constants, which are independent of pressure, temperature and salt content of the feed stream. The permeability constant is measured in cm/s/atm and the salt passage constant in cm/s. Discrepancies between the flat sheet and full element performance can indicate the presence of mechanical damage.

The flat sheet samples are then cleaned with various Avista chemicals to determine the most effective cleaner combinations and contact times. Cleaner efficacy is based on overall improvement in permeability and salt passage constant as well as visual foulant removal.

Flat sheet samples harvested from the full element produced normal permeability and 116% of normal salt passage during baseline cell testing. Flat sheet samples were cleaned with RoClean P111 (2% by weight in RO/DI water and heated to approximately 35 degrees Celsius and circulated) for 2 hours which removed the visual foulant and increased flow by approximately 10%; however, salt passage remained high before and after cleaning.

SN T4048676	Permeability Constant	Salt Passage Constant
Baseline	1.35E-04 Normal	7.37E-06 Normal
Post-Clean	1.49E-04 Normal	9.54E-06 Normal
Manufacturer's Specifications	1.10 to 1.37E-04 Normal Range	5.67 to 9.75E-06 Normal Range

Note: testing conducted with dechlorinated city water from San Marcos, CA



Testing for Flat Sheet Damage

Fujiwara Testing

Fujiwara testing is a qualitative analysis which determines if a polyamide (PA) thin-film membrane has been exposed to an oxidizing halogen, such as chlorine, bromine, or iodine. A color change does not occur if the membranes has not been exposed to halogens. Common symptoms of halogen oxidation include increased flow and loss in permeate quality.

Fujiwara testing was negative for the presence of halogens (e.g. chlorine) in the membrane structure.

Example of a negative Fujiwara color change

Dye Test

Cleaned flat sheet samples were exposed to dye in a cell test apparatus at 100 psi for 15 minutes. Physically and/or chemically damaged membranes will absorb the dye on the membrane surface. Dye penetration through the membrane backing indicates severe physical and/or chemical damage.

Dye passed through the membrane in the areas where the spots were identified indicating severe damage in these areas.

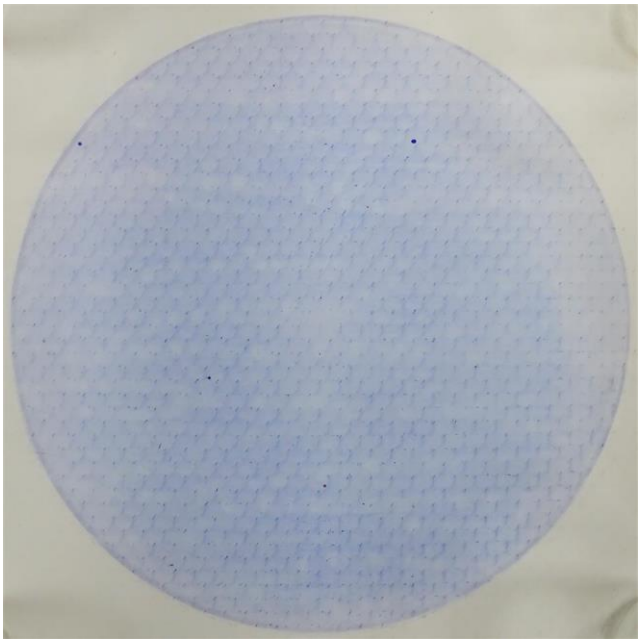
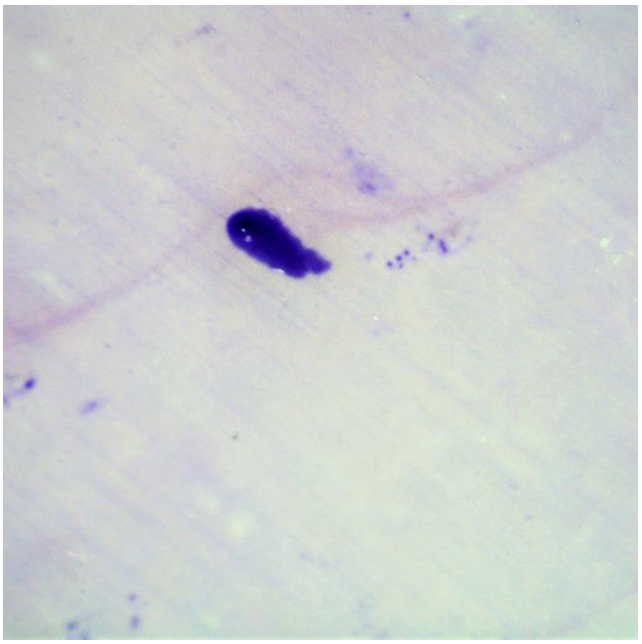


Image of dye uptake on the membrane surface

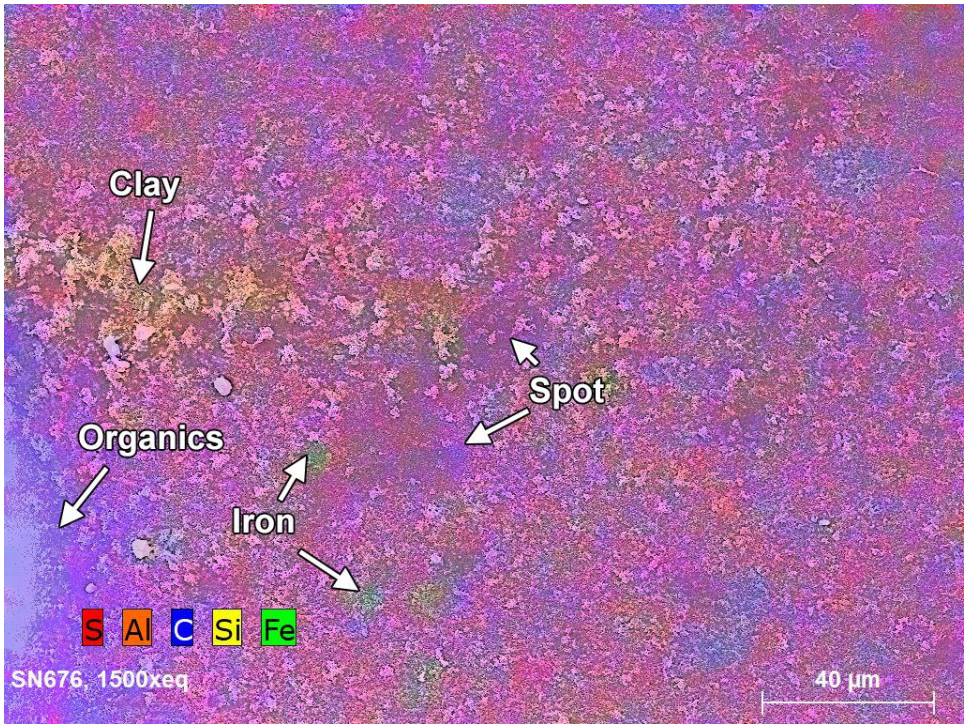


Stereoscopic image (20x) of dye uptake on the tan-colored spots of the membrane surface

Spot Analysis

EDS analysis and CEI were performed on one of the visual spots observed across the membrane. Both techniques detected iron in the areas where the spots were observed which was not in present in areas away from the spots.

Elements	SN T4048676 Away from Spot Weight Percent	SN T4048772 At Spot Weight Percent
Carbon	77.13	73.06
Oxygen	16.07	20.07
Sulfur	6.42	6.54
Aluminum	0.12	0.09
Silicon	0.11	0.07
Iron	ND	0.17



CEI image (1500x) of the tan-colored spot on the membrane surface with labels

Certification by Laboratory

Report Number	Report Content	Element Serial Number	Report Date
WO#120219-3	Standard Spiral Autopsy	T4048676 T4048771 T4048772	February 20th, 2020

We the undersigned being the technical specialists in membrane autopsy and related testing procedures and protocol for Avista Technologies certify to the best of our knowledge and belief that the tests listed in this report have been conducted following Avista's standard testing practices and that the results are accurate and complete.

By signing this certificate neither the laboratory employees nor their employer makes any warranty, expressed or implied, concerning the cleaning study results.

Date: 02/20/2020

Signed:

Megan Lee
Laboratory Services Manager

Jaime La Cuesta
Laboratory Services Chemist

Avista TECHNOLOGIES
a Kurita company

Membrane Autopsy Report

Completed for:

Trussell Technologies, Inc.

Padre Dam

Serial Number T4048771

Middle Element

02/20/2020 WO#120219-3

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Table of Contents

Executive Summary

Background

Trussell Technologies provided three reverse osmosis (RO) elements to Avista Technologies for analysis. Elements Serial Number (SN) T4048676 was removed from the lead position, element SN T4048771 was removed from the middle position and element SN T4048772 was removed from the tail position. The following is a summary of the results pertaining to element SN T4048771 (Middle).

Initial Element Testing

Element SN T4048771 produced normal flow, lower than normal rejection (99.3%) and a differential pressure of 6 psi during initial wet testing. The element passed integrity testing suggesting the absence of damage to the internal components of the spiral wound element.

External Inspection

The external components (fiberglass casing, brine seal, anti-telescoping devices (ATDs), permeate tube) were in good mechanical condition.

Internal Inspection

The scroll ends were in good mechanical condition and free of debris. The membrane surface contained a small amount of brown colored foulant which was found primarily in the feed spacer contact points. Random spots were observed across the membrane on all membrane leaves. Foulant was observed on the feed spacer material but it was free of blockages. The membrane backing contained brown colored foulant that corresponded to the spots observed on the membrane surface.

Foulant Analysis

The wet foulant density was measured at 0.02 mg/cm^2. The organic content was 76% suggesting the bulk of the foulant was organic. Microbiological analysis identified bioslime, algae, yeast and bacteria and aerobic bacteria counts were 100 CFU/cm^2 after 72 hour incubation period. Fourier Transform Infrared (FT-IR) spectroscopy confirmed the presence of organics by detecting bands associated with bioslime, microorganisms and lipids (hydrophobic fatty acids). Energy Dispersive Spectroscopy (EDS) detected low concentrations of silicon and aluminum on the membrane. Chromatic Elemental Imaging^{SM} (CEI^{SM}) identified patches of organic across the membrane surface with clay deposits embedded in and above the organics

Based on the analysis the bulk of the foulant consisted of organics.

Flat Sheet Performance and Cleaning Study

Flat sheet samples harvested from the full element produced normal permeability and 116% of normal salt passage during baseline cell testing. Cleaning the flat sheet with RoClean P112 (2% by weight, 2 hrs heated cleaning solution) removed the bulk of the foulant and increased water passage by 12% Unfortunately, salt passage was high before and after cleaning due to preexisting physical damage of the membrane.

Flat Sheet Damage

Fujiwara testing was negative for the presence of halogen (e.g. chlorine) oxidation. However, dye testing revealed dye passage within the spots observed on the membrane surface. The damaged areas were very circular in shape indicative of a reaction between a metal and an oxidizer. Further analysis with EDS and CEI showed iron in the damaged areas. Based on these observations the most likely cause for the damage observed is a reaction between metals and an oxidizer.

Initial Element Test

Element Weight

All elements are weighed prior to autopsy as weight is often indicative of the degree of fouling. New eight-inch elements weigh approximately 30 to 35 pounds.

SN T4048771 weighed 32 pounds.

Full Element Wet Test

Test results were normalized to the manufacturer's published test conditions.

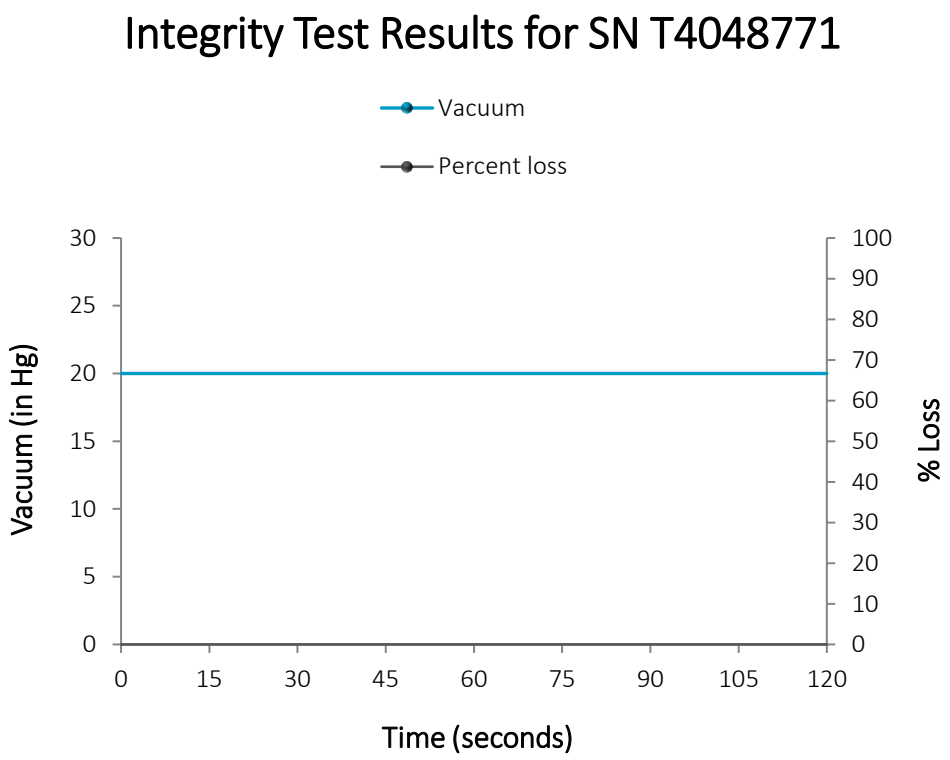
Filmtec BW30XFR-400/34	Flow (gpm)	Rejection (%)	Pressure Drop (psi)
SN T4048771	7.22	99.3	6
Manufacturer's Specifications	6.79 to 9.19	99.4 to 99.7	≤15

Element wet testing

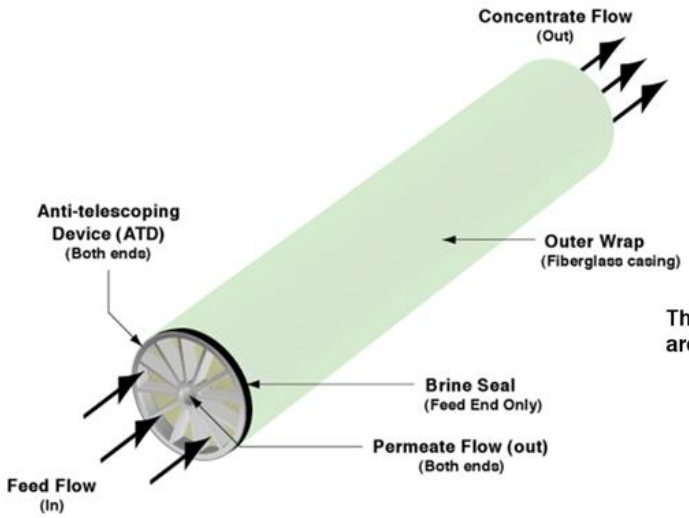
Integrity Test

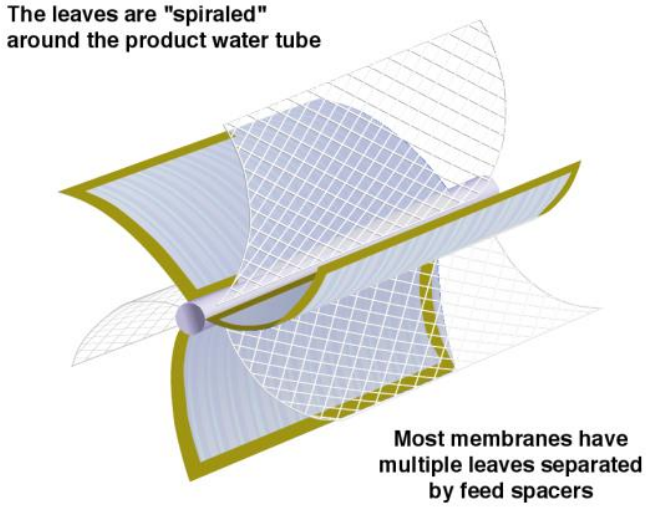
Integrity testing is performed to identify mechanical damage to the internal components of the spiral wound element. In this test, a vacuum of approximately 20 inches Mercury (in. Hg) is applied to the permeate side of the membrane and the membrane is then sealed. The vacuum is monitored for a duration of 120 seconds. Any loss of vacuum indicates the presence of damage; however, losses of over 35% of the vacuum within the 120 second period suggests severe physical damage.

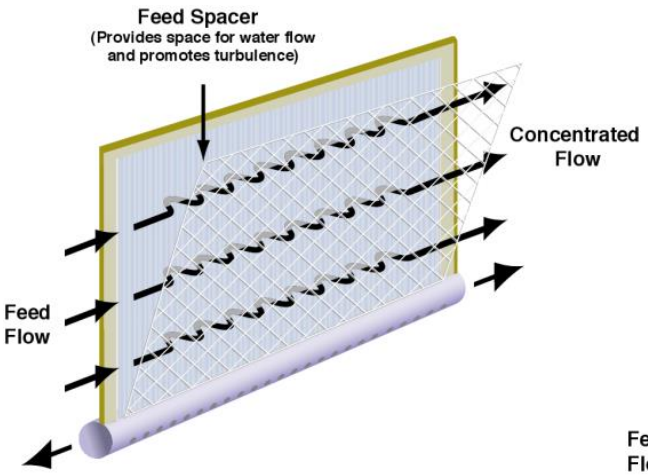
The element passed integrity testing.

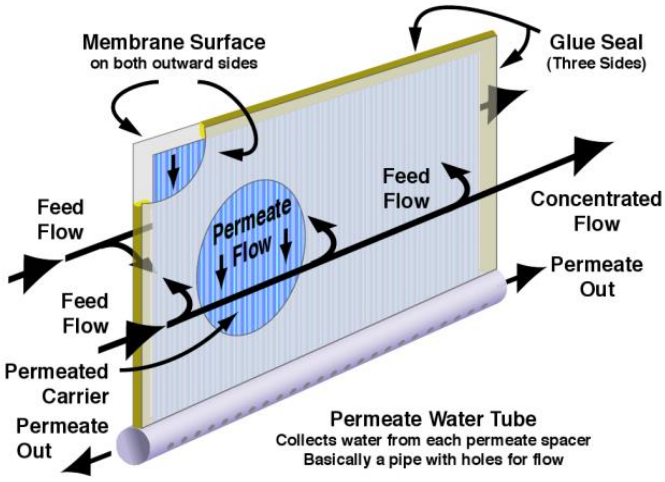


Membrane Construction Diagrams









External Inspection

Fiberglass Casing

The purpose of the fiberglass casing is to ensure that the various membrane components are held in their correct position for optimum performance. Damage to the casing can be an indication of rough handling or damage from excessive differential pressure across the element from heavy fouling.

The fiberglass casing was in good mechanical condition.

Fiberglass casing of SN T4048771

Brine Seal

The brine seal is used to seal against the inside diameter of the pressure vessels and the outside diameter of the membrane to ensure that all the feed water passes through the element. Feed water passing on the exterior of the element can result in higher pressures, which can cause cracking of the fiberglass casing.

The brine seal was in good mechanical condition.

Permeate Tube

The permeate tube is a pipe that is located at the center of the element. It contains lines of holes and is bonded to each membrane leaf, allowing permeate water to travel from the leaves into the permeate tube to be collected. Damage to the ends of the permeate tube can lead to o-ring failures, causing bypass of feed or concentrate water into the permeate stream. Cracking of the permeate tube can also result in permeate contamination.

The permeate tube was in good mechanical condition.

Anti-Telescoping Devices (ATDs)

The function of the ATDs is to stabilize the components of the element. This helps to prevent shifting of the internal mechanical components under pressure, also known as telescoping. Telescoping may still occur if pressures exceed the manufacturer's specifications.

The ATDs were in good mechanical condition.

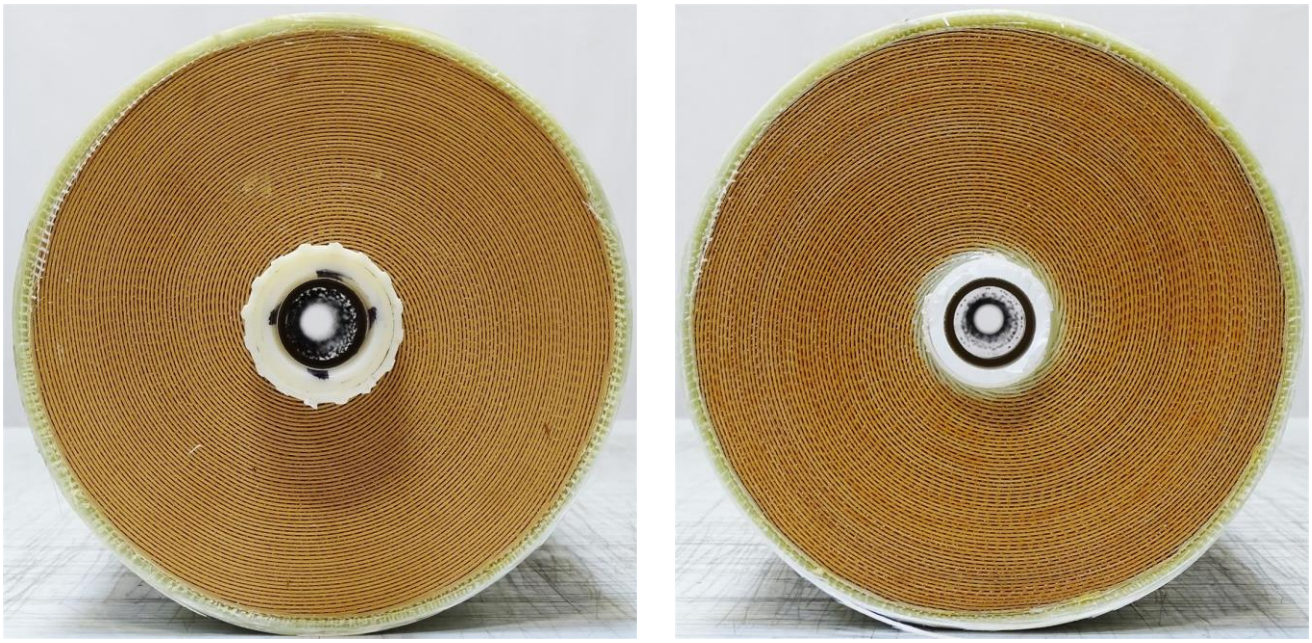
Feed (left) and concentrate (right) ATDs of SN T4048771

Internal Inspection

Scroll Ends

The ends of the element are called scroll ends. They are examined for the presence of foulant debris and mechanical damage (e.g. gapping, feed spacer extrusion). The presence of foulant on the scroll ends can cause elevated delta pressures while gapping and feed spacer extrusion indicate uneven hydraulics (high flow/low flow regions). In addition, each scroll end is examined for telescoping, the gradual axial shift of the membrane leaves from the outer diameter of the element towards the permeate tube. Telescoping is often caused by the development of high differential pressure (greater than the manufacturer's specification) across the element or when pressure is applied too quickly, causing a water hammer effect.

The scroll ends were in good mechanical condition.

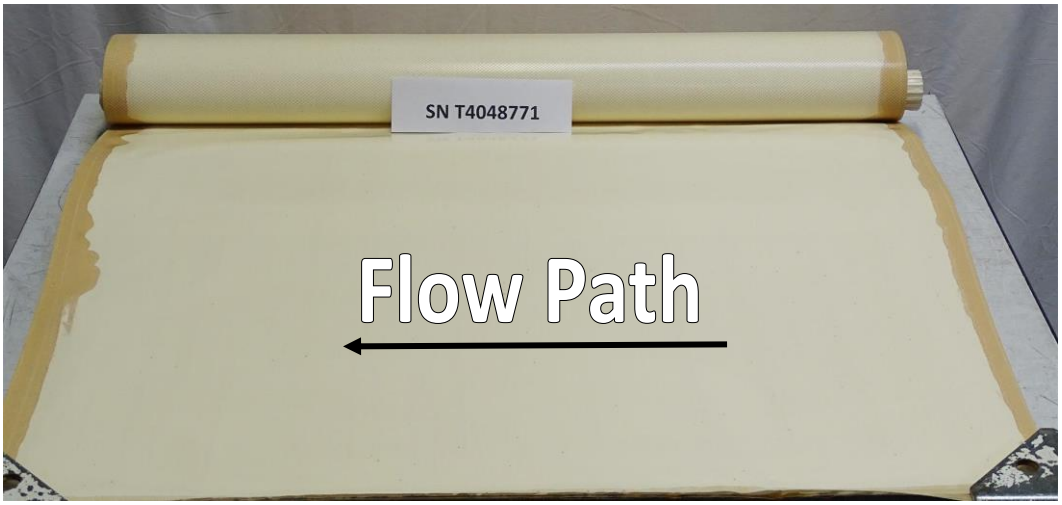


Feed (left) and concentrate (right) scroll ends of SN T4048771

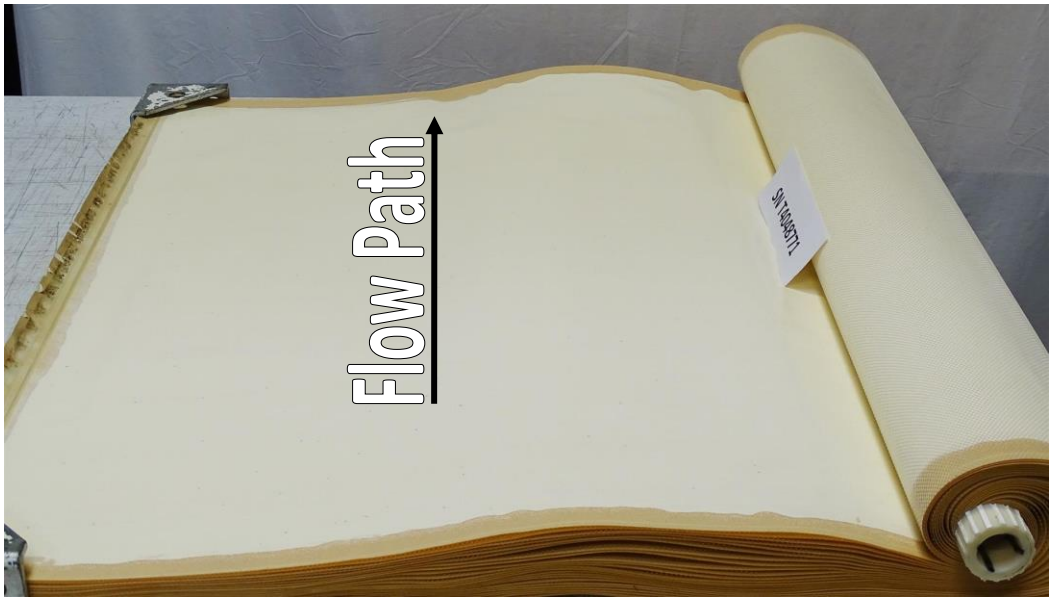
Membrane Surface

New membrane surfaces are uniform and shiny. Foulant can often be detected through visual examination; however, membrane appearance can be misleading as some foulants are not visible. The presence of foulant on the membrane surface can cause elevated delta pressure, loss in flow and damage if the foulant is abrasive. Additionally, the membrane surface is inspected for damage such as delamination. Delamination is the lifting of the thin-film membrane from the support layer and often occurs due to a positive pressure on the permeate side of the element.

The membrane surface contained a small amount of brown colored foulant which was found primarily in the feed spacer contact points. Random spots were observed across the membrane on all membrane leaves.



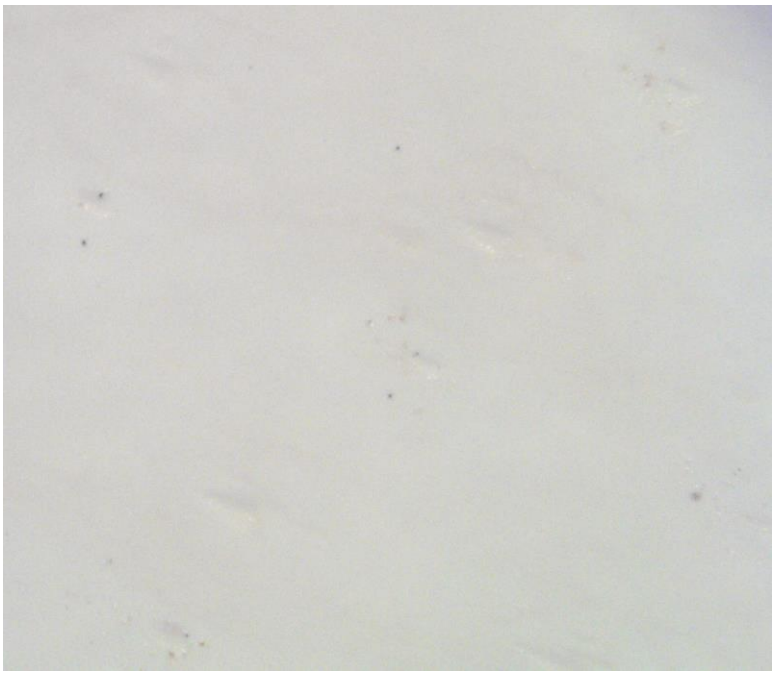
Exposed membrane surface of SN T4048771



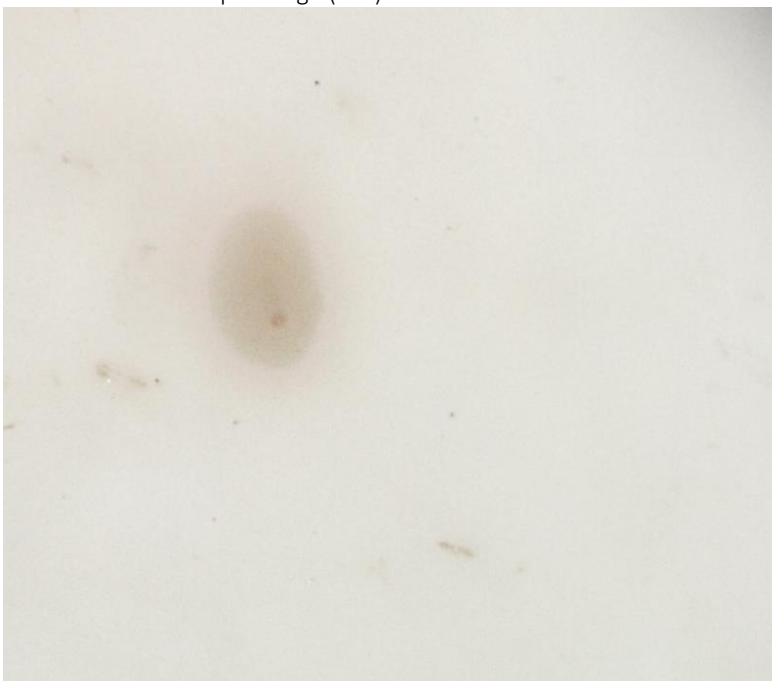
Exposed membrane surface from feed end of SN T4048771

Image of foulant scraped on the membrane surface

Image of spots observed on the membrane surface



Stereoscopic image (40x) of the membrane surface

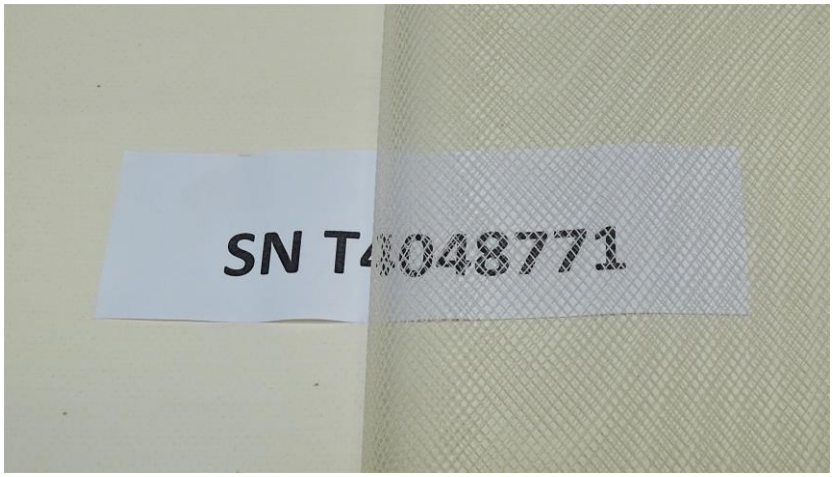


Stereoscopic image (40x) of a spot on the membrane surface

Feed Spacers

The feed spacer is a plastic net material designed to separate the membrane leaves, forming a flow path, and to promote turbulence within the feed water channels. Foulant blocking the feed channels causes more resistance for the feed water flowing through the element and results in higher than normal delta pressures.

Although some of the foulant was observed on the feed spacer they were in good mechanical condition and free of blockages.



Feed spacer of SN T4048771

Glue Lines

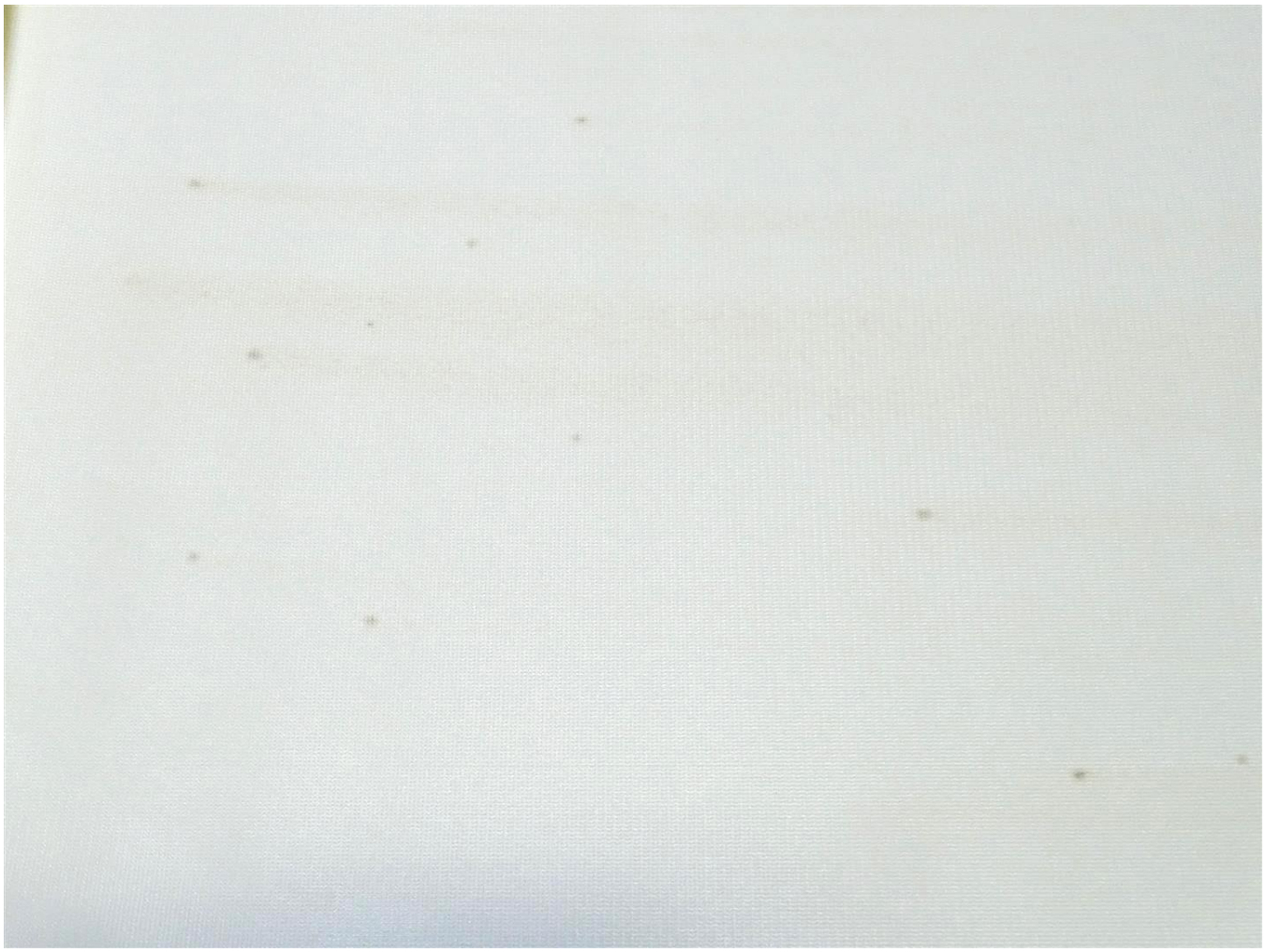
Membrane leaves are glued on three sides to separate the feed and permeate streams. The glue lines are inspected for specific damage, including glue flaps and pouching. Glue flaps refer to excess inactive membrane material located closest to the ends of the element. Flaps found on the feed end of the element can flare during operation, blocking the feed channels on the scroll end, potentially causing increased differential pressure. Pouching of the glue line, which is often a result of delamination, allows feed water to pass through the inactive membrane at the glue line, contaminating the permeate stream.

The glue lines were in good mechanical condition.

Permeate Carriers and Membrane Backing

The permeate carriers provide a path for permeate water to flow towards the permeate tube, which minimizes permeate-side pressure losses. New permeate carriers and membrane backing are uniform in color. Foulant found on the permeate side of the membrane leaves indicates contamination of the permeate stream.

Tan-colored spots were observed on the membrane backing and permeate carriers.



Membrane backing of SN T4048771

Foulant Analysis

Acid Testing

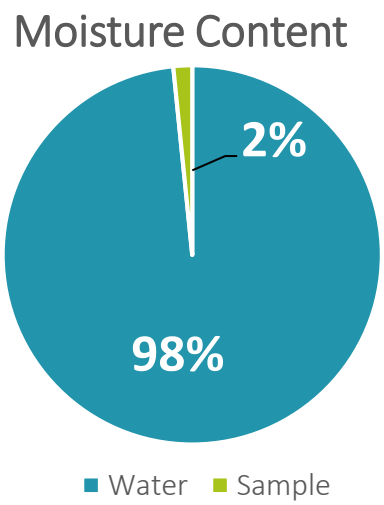
Acid testing is used to determine the presence of carbonates and metals on the membrane surface. In this test, several drops of dilute hydrochloric acid (HCl) were placed on the foulant surfaces. Effervescing indicates the presence of carbonates while a color change is associated with the presence of metals.

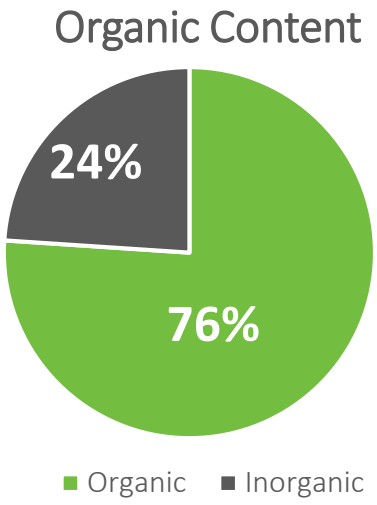
Acid testing was negative for the presence of carbonates and metals.

Foulant Density Measurement and Composition Testing

A sample collected from a known area of the membrane surface is weighed before and after drying to determine the foulant density (reported as dry foulant density – mg/cm^2) and moisture content of the sample. Different types of foulant materials exhibit higher moisture contents. Relative water concentrations greater than 95% indicate an extremely hydrated, biological material. Alternatively, scales (crystalline material) typically contain very little moisture. The organic content of the dehydrated material is then measured through loss on ignition (LOI) testing. If the organic content of the total solids is greater than 65%, it is considered primarily organic.

The wet foulant density was measured at 0.02 mg/cm^2. The moisture and organic contents are shown in the graphs below.

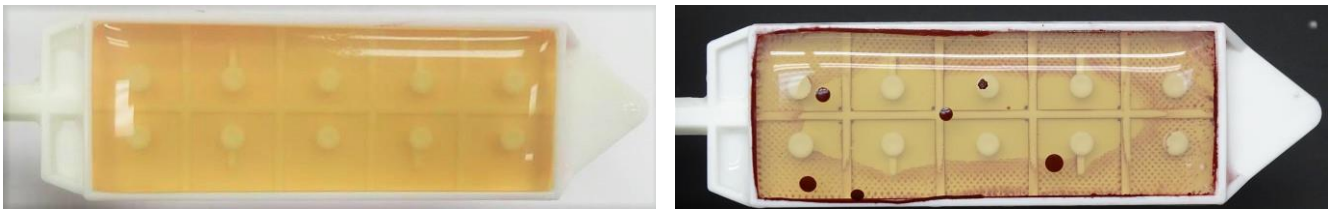




Biological Activity Testing

Dip slides for aerobic bacteria are exposed to the foulant material on the membrane surface. The slides are incubated for 72 hours and inspected for biological growth. Greater colony density, measured in colony-forming units (CFU)/cm^2, indicates a more biologically active sample.

The aerobic slide showed approximately 100 CFU/cm^2 after the 72 hour incubation period.

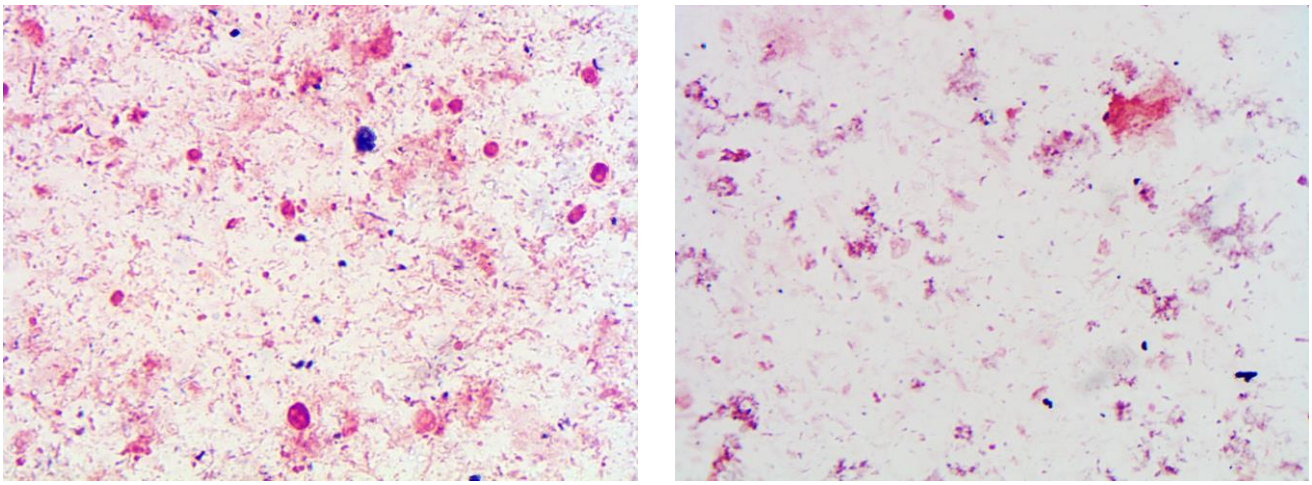


Aerobic bacteria slide before incubation (left) and after incubation (right)

Microbiological Analysis

This analysis is performed to identify microbiological components of the foulant removed from the membrane surface. Foulant samples are stained and examined with a light microscope at 1000x using an oil immersion lens. Gram positive bacteria are stained purple while Gram negative bacteria are stained pink.

Microbiological analysis performed on foulant scraped from the membrane surface identified bioslime, algae, yeast and bacteria.

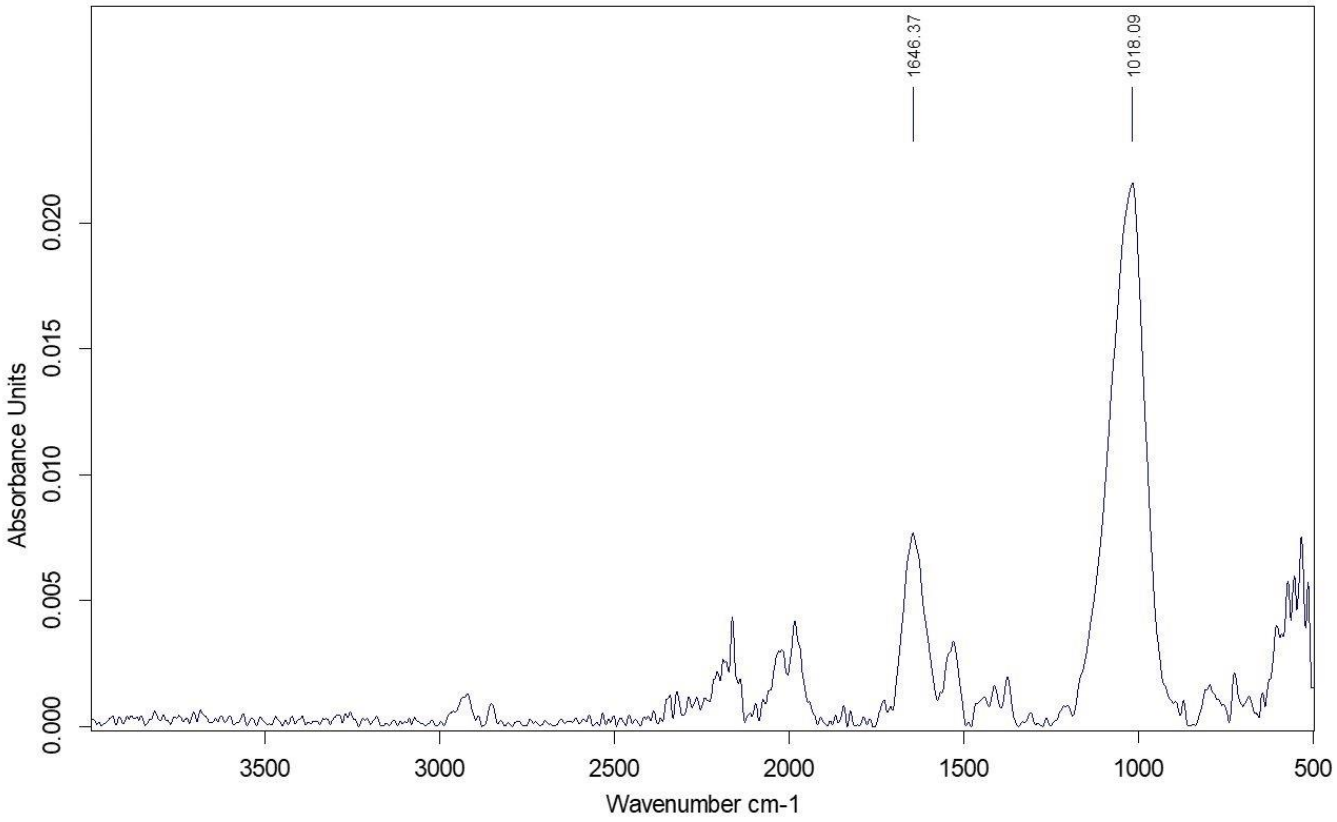


Light microscope images (1000x) of foulant scraped from SN T4048771

Fourier Transform Infrared Spectroscopy Analysis

Fourier Transform Infrared Spectroscopy (FT-IR) is an analytical technique used to identify functional groups (specific groups of atoms or bonds within molecules). Infrared radiation passes through a sample, with some of the radiation absorbed and some transmitted. A measurement and interpretation of this data produces a spectrum which can then be compared and matched to the known spectra for functional groups based on the wavenumber at which bands appear and their respective shapes (e.g. sharp, broad, strong, weak).

FT-IR spectroscopy performed on foulant scraped from the membrane surface displayed a strong, sharp peak at approximately 1000 cm^{-1} which is associated with carbohydrates and Si-O bond stretching. The double peak between 1650 and 1500 cm^{-1} is associated with amino acids (i.e. proteins) from microorganisms. The weaker bands between 2200 cm^{-1} and 2000 cm^{-1} are contributed by lipids (hydrophobic fatty acids).



FT-IR spectral image of foulant removed from the membrane surface of SN T4048771

Energy Dispersive Spectroscopy (EDS) Analysis

Energy Dispersive Spectroscopy analysis is used to determine the relative concentration of elements present in a sample. EDS analysis is performed on a dry membrane sample. The element sulfur is at least in part associated with the membrane support material (polysulfone) rather than a foulant layer. Avista's analysis of new membranes typically detects between 5.00 and 7.00 weight percentage. Relative concentrations below 5.00 percent indicate the presence of a foulant layer masking the membrane surface.

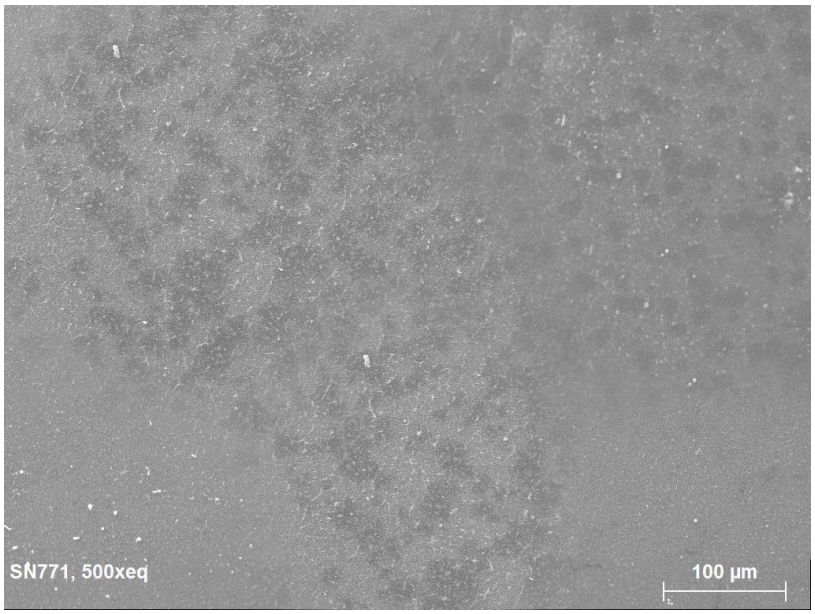
EDS analysis only detected trace amounts (<0.50 wt%) of silicon and aluminum as the inorganic elements present in the foulant layer. The sulfur weight percent (6.49 wt%) was within normal values suggesting the foulant layer was thin.

Elements	SN T4048771 Weight Percent
Carbon	77.81
Oxygen	15.59
Sulfur	6.57
Silicon	0.06
Aluminum	0.05

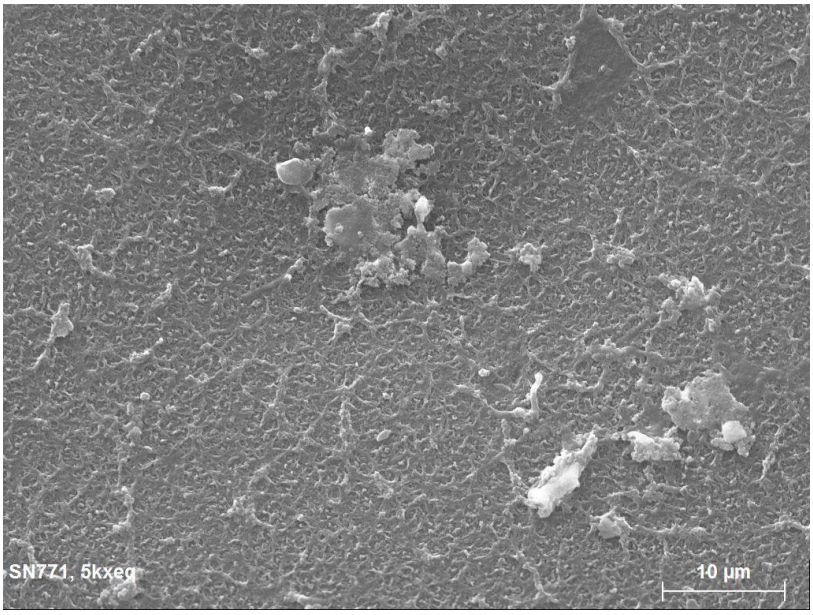
Scanning Electron Microscope (SEM) Imaging

SEM imaging is performed on the membrane surface to observe the topography of the foulant material. Foulant morphology can be an indicator of the type of foulant.

SEM images displayed patches of smooth foulant material across the membrane surface with colloidal material above and embedded in it.



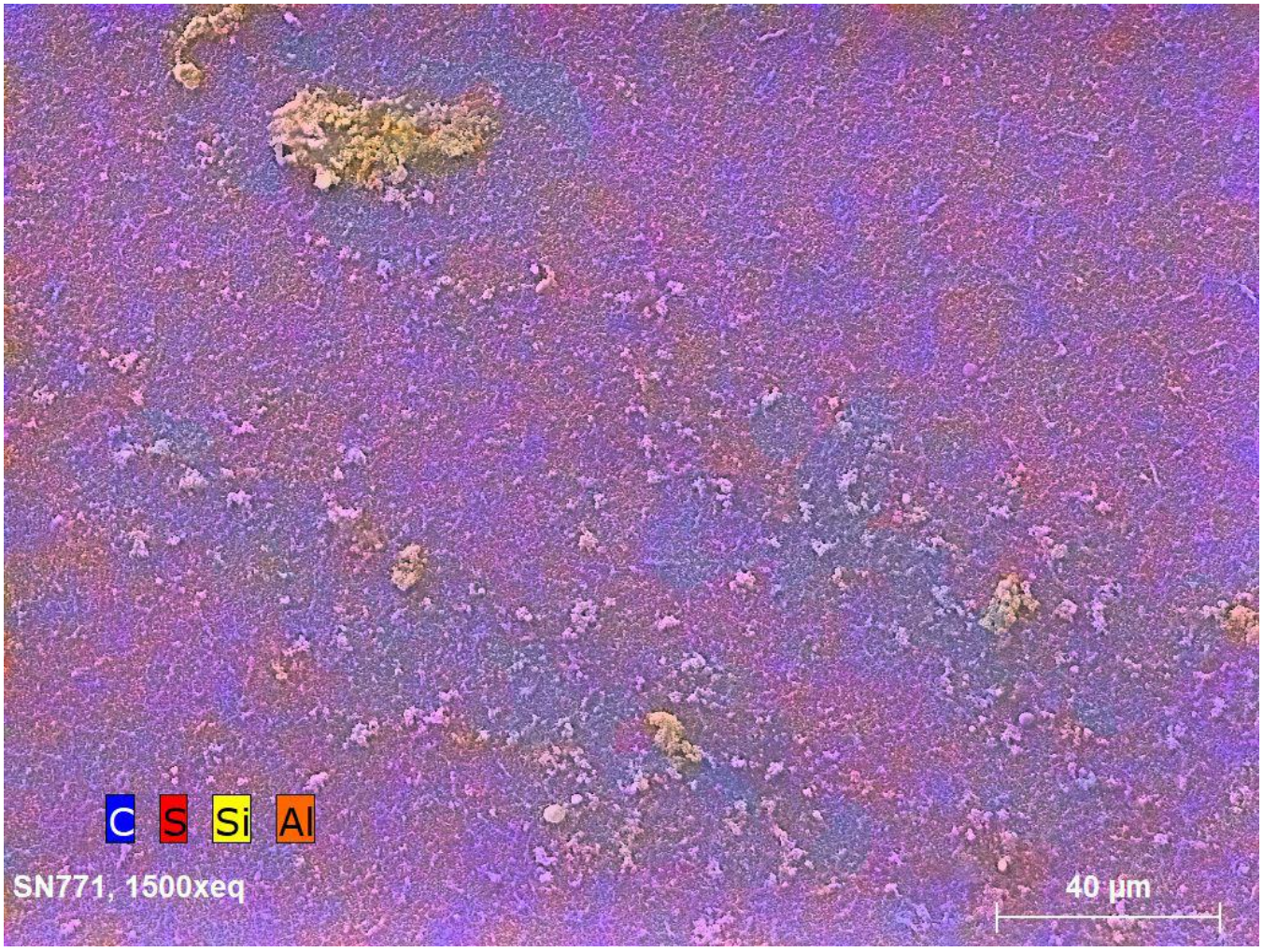
SEM image (150x) of the membrane surface of SN T4048771



Close-up SEM image (5000x) of the membrane surface of SN T4048771

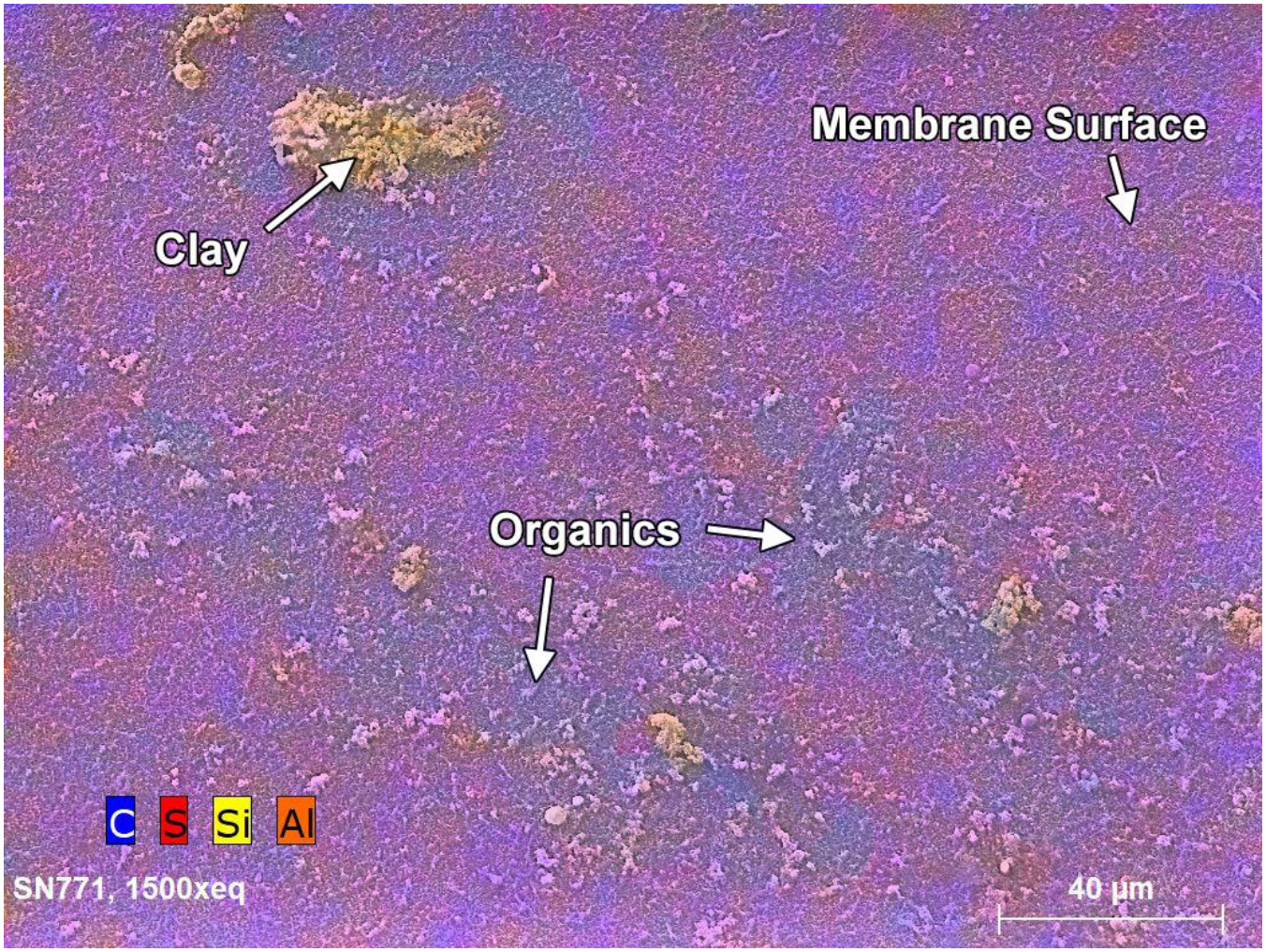
Chromatic Elemental Imaging℠ (CEI℠)

CEI is a high-resolution imaging technique used to determine the spatial distribution of elements in a foulant sample. Each element is assigned a color (shown in a legend on the bottom left corner of the CEI image) and the colors correspond to the location of the elements in the sample. An element's color intensity is associated with its concentration in the sample (i.e. elements present with higher relative concentrations are displayed with greater color intensity in the image). Additionally, a blending of colors signifies a compound (material composed of more than one element such as calcium carbonate).



CEI image (1500x) of the membrane surface

CEI[SM] identified smooth organic patches, noted by a high carbon content (dark blue) across the membrane. Clay particles (yellow-orange) were observed above and embedded in the organic material. The membrane surface, represented by sulfur (red), was visible in the areas the organic foulant was relatively thin.



CEI image (1500x) of the membrane surface with labels

Flat Sheet Performance and Cleaning Study

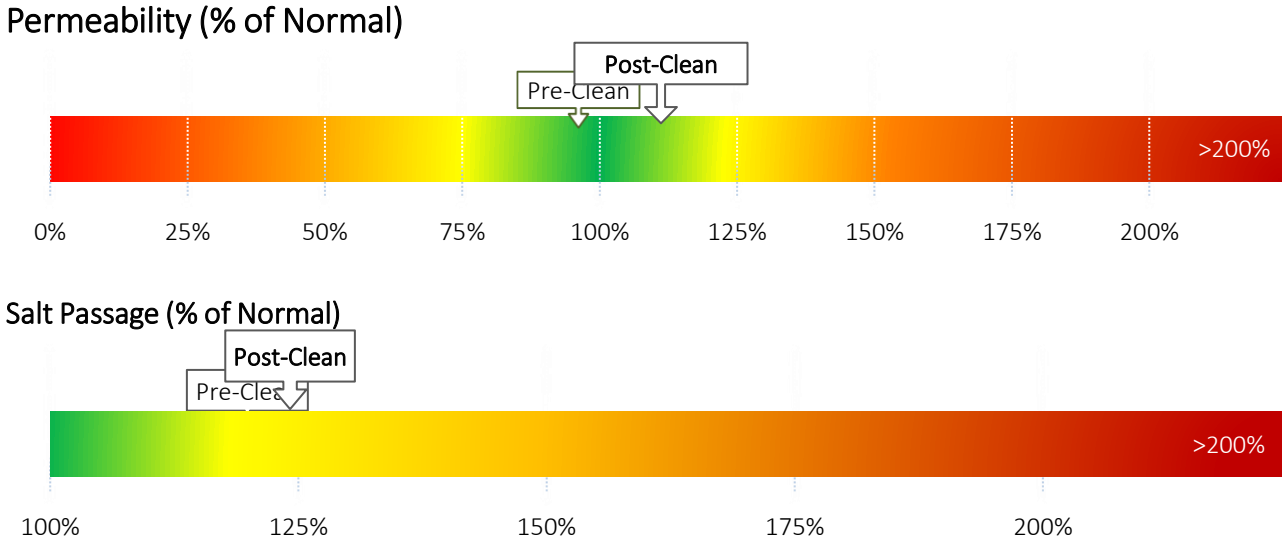
To evaluate flat sheet performance, membrane samples harvested from the full element are tested for permeability and salt passage. The raw flow and conductivity measurements from the test are used to calculate the permeability and salt passage constants, which are independent of pressure, temperature and salt content of the feed stream. The permeability constant is measured in cm/s/atm and the salt passage constant in cm/s. Discrepancies between the flat sheet and full element performance can indicate the presence of mechanical damage.

The flat sheet samples are then cleaned with various Avista chemicals to determine the most effective cleaner combinations and contact times. Cleaner efficacy is based on overall improvement in permeability and salt passage constant as well as visual foulant removal.

Flat sheet samples harvested from the full element produced normal permeability and 116% of normal salt passage during baseline cell testing. Flat sheet samples were cleaned with RoClean P111 (2% by weight in RO/DI water and heated to approximately 35 degrees Celsius and circulated) for 2 hours which removed the visual foulant and increased flow by approximately 12%; however, salt passage remained high before and after cleaning.

SN T4048771	Permeability Constant	Salt Passage Constant
Pre-Clean	1.28E-04 Normal	11.31E-06 116% of Normal
Post-Clean	1.44E-04 Normal	11.99E-06 123% of Normal
Manufacturer's Specifications	1.10 to 1.37E-04 Normal Range	5.67 to 9.75E-06 Normal Range

Note: testing conducted with dechlorinated city water from San Marcos, CA



Testing for Flat Sheet Damage

Fujiwara Testing

Fujiwara testing is a qualitative analysis which determines if a polyamide (PA) thin-film membrane has been exposed to an oxidizing halogen, such as chlorine, bromine, or iodine. A color change does not occur if the membranes has not been exposed to halogens. Common symptoms of halogen oxidation include increased flow and loss in permeate quality.

Fujiwara testing was negative for the presence of halogens (e.g. chlorine) in the membrane structure.

Example of a negative Fujiwara color change

Dye Test

Cleaned flat sheet samples were exposed to dye in a cell test apparatus at 100 psi for 15 minutes. Physically and/or chemically damaged membranes will absorb the dye on the membrane surface. Dye penetration through the membrane backing indicates severe physical and/or chemical damage.

Dye passed through the membrane in the areas where the spots were identified indicating severe damage in these areas.

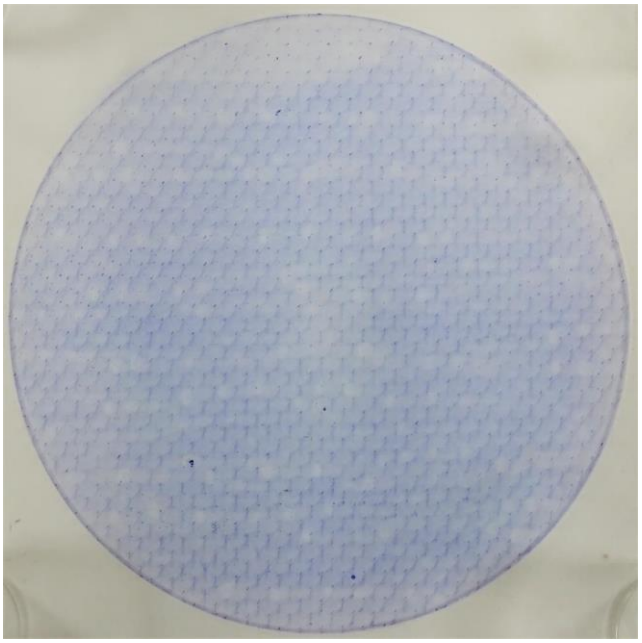
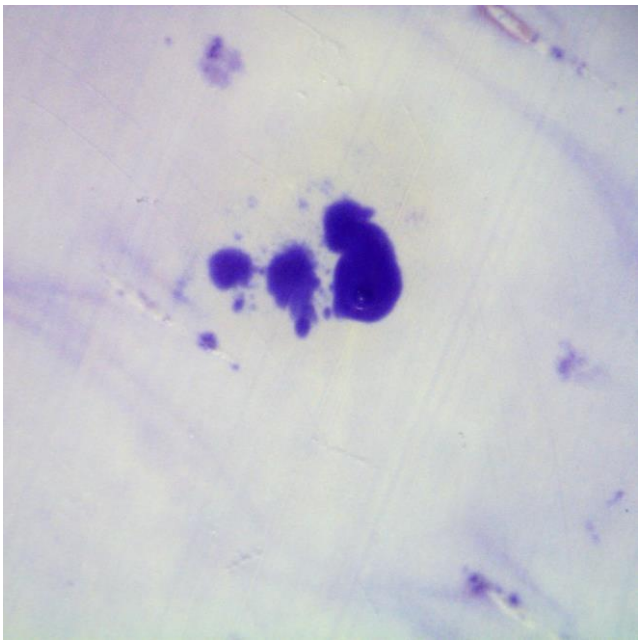


Image of dye uptake on the membrane surface

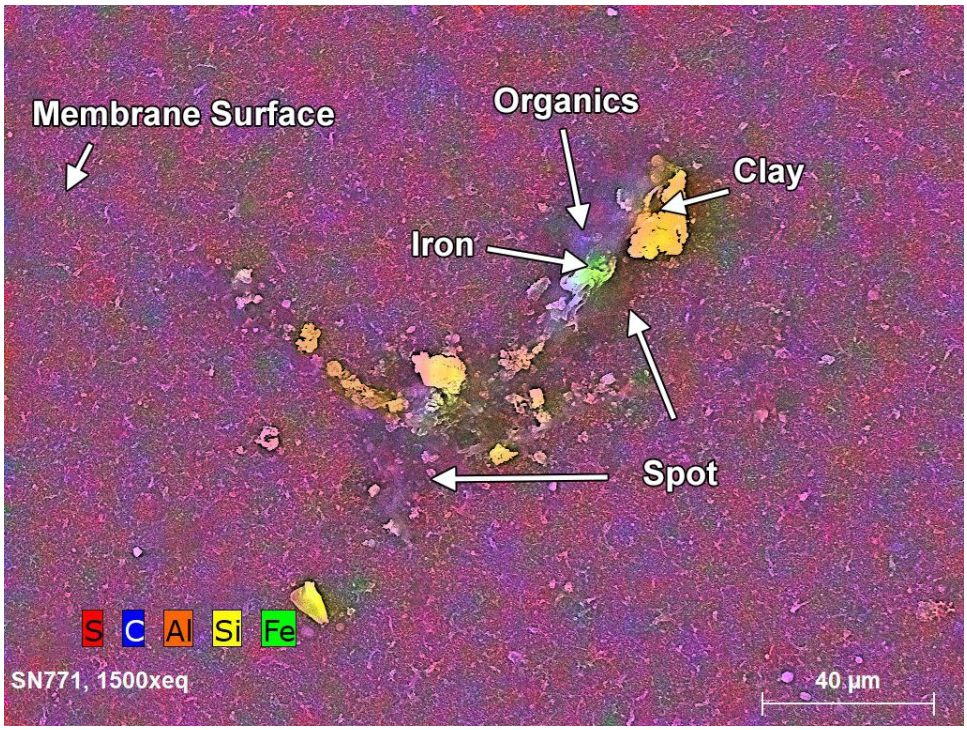


Stereoscopic image (20x) of dye uptake on the tan-colored spots of the membrane surface

Spot Analysis

EDS analysis and CEI detected iron in the areas where the spots were observed which was not in present in areas away from the spots.

Elements	SN T4048771 Away from Spot Weight Percent	SN T4048771 At Spot Weight Percent
Carbon	77.81	72.37
Oxygen	15.59	20.59
Sulfur	6.57	6.52
Silicon	0.06	0.26
Aluminum	0.05	0.15
Iron	ND	0.11



CEI image (1500x) of a spot observed on the membrane surface

Certification by Laboratory

Report Number	Report Content	Element Serial Number	Report Date
WO#120219-3	Standard Spiral Autopsy	T4048676 **T4048771** T4048772	February 20th, 2020

We the undersigned being the technical specialists in membrane autopsy and related testing procedures and protocol for Avista Technologies certify to the best of our knowledge and belief that the tests listed in this report have been conducted following Avista's standard testing practices and that the results are accurate and complete.

By signing this certificate neither the laboratory employees nor their employer makes any warranty, expressed or implied, concerning the cleaning study results.

Date: 02/20/2020

Signed:

Megan Lee
Laboratory Services Manager

Jaime La Cuesta
Laboratory Services Chemist

Avista TECHNOLOGIES
a Kurita company

Membrane Autopsy Report

Completed for:

Trussell Technologies, Inc.
Padre Dam
Serial Number T4048772
Tail Element

01/20/2020 WO#120219-3

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Table of Contents

Executive Summary

Background

Trussell Technologies provided three reverse osmosis (RO) elements to Avista Technologies for analysis. Elements Serial Number (SN) T4048676 was removed from the lead position, element SN T4048771 was removed from the middle position and element SN T4048772 was removed from the tail position. The following is a summary of the results pertaining to element SN T4048772 (Tail).

Initial Element Testing

Element SN T4048772 produced normal flow, lower than normal rejection (99.2%) and a differential pressure of 5 psi during initial wet testing. The element passed integrity testing suggesting the absence of damage to the internal components of the spiral wound element.

External Inspection

The external components (fiberglass casing, brine seal, anti-telescoping devices (ATDs), permeate tube) were in good mechanical condition.

Internal Inspection

The scroll ends were in good mechanical condition and free of debris. The membrane surface contained a small amount of brown colored foulant which was found primarily in the feed spacer contact points. Random spots were observed across the membrane on all membrane leaves. Foulant was observed on the feed spacer material but it was free of blockages. The membrane backing contained brown colored foulant that corresponded to the spots observed on the membrane surface.

Foulant Analysis

The wet foulant density was measured at 0.01 mg/cm^2. The organic content was 72% indicating the bulk of the foulant was organic. Microbiological analysis identified bioslime, algae, yeast and bacteria and aerobic bacteria counts were 40 CFU/cm^2 after 72 hour incubation period. Fourier Transform Infrared (FT-IR) spectroscopy confirmed the presence of organics by detecting bands associated with bioslime, microorganisms and lipids (hydrophobic fatty acids). Energy Dispersive Spectroscopy (EDS) detected low concentrations of silicon and aluminum on the membrane. Chromatic Elemental Imaging^{SM} (CEI^{SM}) identified patches of organic across the membrane surface with clay deposits embedded in and above the organics

Based on the analysis the bulk of the foulant consisted of organics.

Flat Sheet Performance and Cleaning Study

Flat sheet samples harvested from the full element produced normal permeability and 128% of normal salt passage during baseline cell testing. Cleaning the flat sheet with RoClean P112 (2% by weight, 2 hrs heated cleaning solution) removed the bulk of the foulant and increased water passage by 11% Unfortunately, salt passage was high before and after cleaning due to preexisting physical damage of the membrane.

Flat Sheet Damage

Fujiwara testing was negative for the presence of halogen (e.g. chlorine) oxidation. However, dye testing revealed dye passage within the spots observed on the membrane surface. The damaged areas were very circular in shape indicative of a reaction between a metal and an oxidizer. Further analysis with EDS and CEI showed iron in the damaged areas. Based on these observations the most likely cause for the damage observed is a reaction between metals and an oxidizer.

Initial Element Test

Element Weight

All elements are weighed prior to autopsy as weight is often indicative of the degree of fouling. New eight-inch elements weigh approximately 30 to 35 pounds.

SN T4048772 weighed 32 pounds.

Full Element Wet Test

Test results were normalized to the manufacturer's published test conditions.

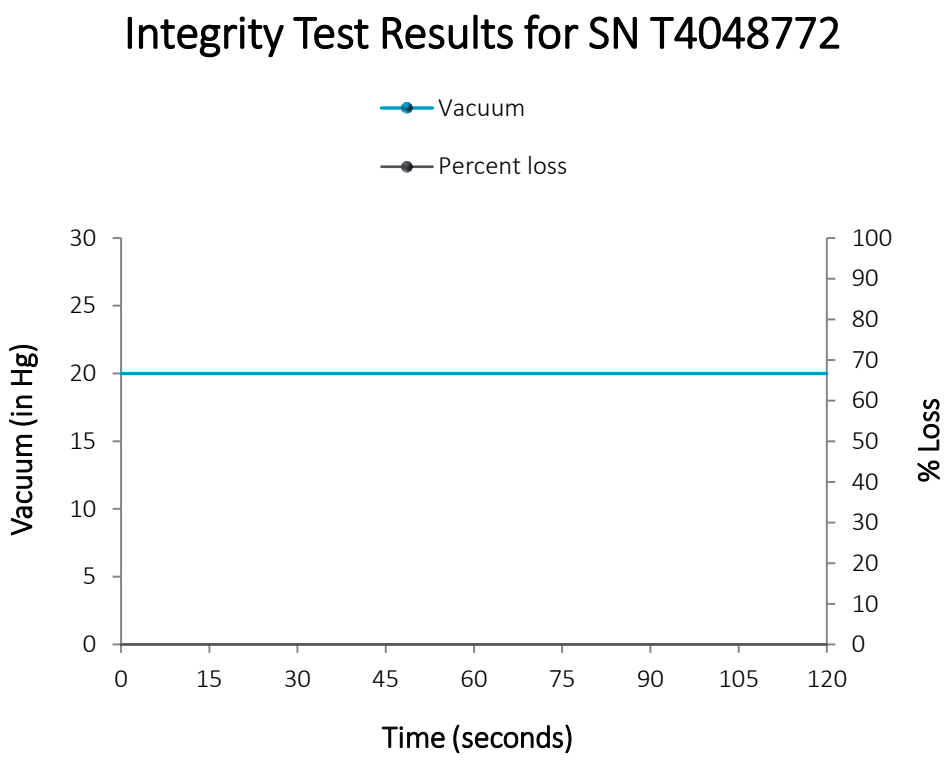
Filmtec BW30XFR-400/34	Flow (gpm)	Rejection (%)	Pressure Drop (psi)
SN T4048772	7.51	99.2	5
Manufacturer's Specifications	6.79 to 9.19	99.4 to 99.7	≤15

Element wet testing

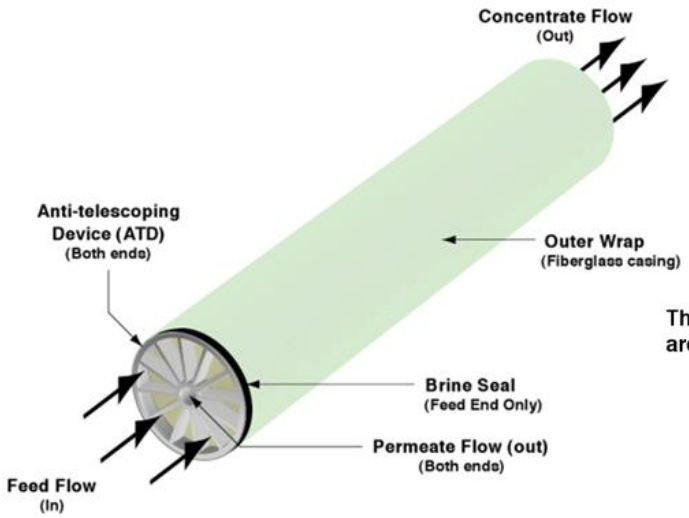
Integrity Test

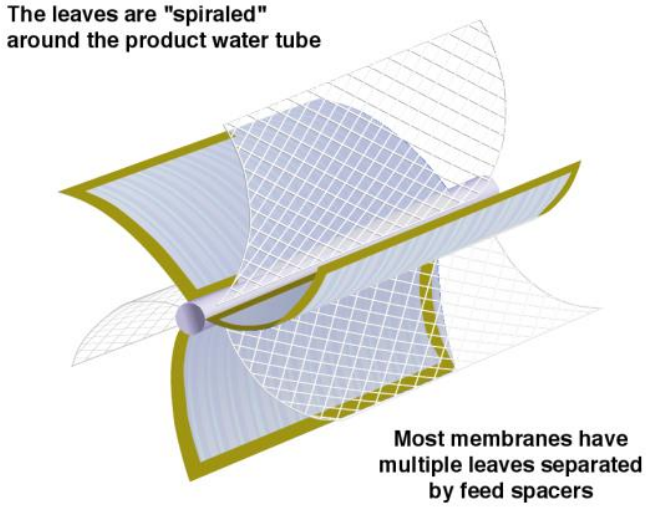
Integrity testing is performed to identify mechanical damage to the internal components of the spiral wound element. In this test, a vacuum of approximately 20 inches Mercury (in. Hg) is applied to the permeate side of the membrane and the membrane is then sealed. The vacuum is monitored for a duration of 120 seconds. Any loss of vacuum indicates the presence of damage; however, losses of over 35% of the vacuum within the 120 second period suggests severe physical damage.

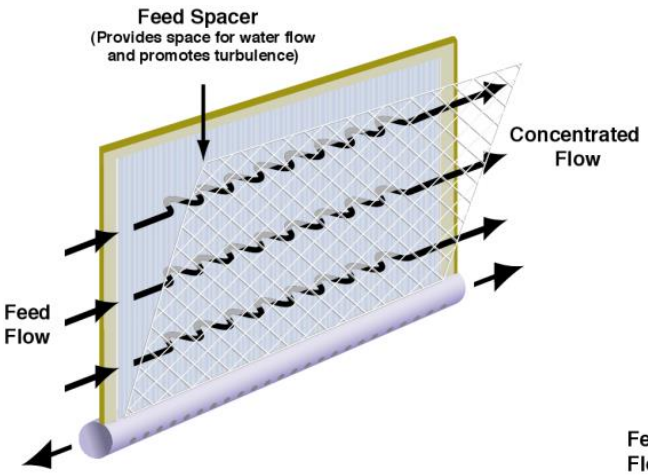
The element passed integrity testing.

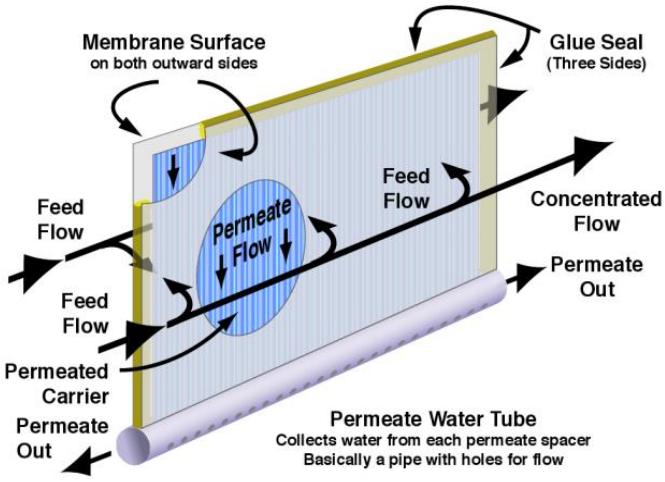


Membrane Construction Diagrams









External Inspection

Fiberglass Casing

The purpose of the fiberglass casing is to ensure that the various membrane components are held in their correct position for optimum performance. Damage to the casing can be an indication of rough handling or damage from excessive differential pressure across the element from heavy fouling.

The fiberglass casing was in good mechanical condition.

Fiberglass casing of SN T4048772

Brine Seal

The brine seal is used to seal against the inside diameter of the pressure vessels and the outside diameter of the membrane to ensure that all the feed water passes through the element. Feed water passing on the exterior of the element can result in higher pressures, which can cause cracking of the fiberglass casing.

The brine seal was in good mechanical condition.

Permeate Tube

The permeate tube is a pipe that is located at the center of the element. It contains lines of holes and is bonded to each membrane leaf, allowing permeate water to travel from the leaves into the permeate tube to be collected. Damage to the ends of the permeate tube can lead to o-ring failures, causing bypass of feed or concentrate water into the permeate stream. Cracking of the permeate tube can also result in permeate contamination.

The permeate tube was in good mechanical condition.

Anti-Telescoping Devices (ATDs)

The function of the ATDs is to stabilize the components of the element. This helps to prevent shifting of the internal mechanical components under pressure, also known as telescoping. Telescoping may still occur if pressures exceed the manufacturer's specifications.

The ATDs were in good mechanical condition.

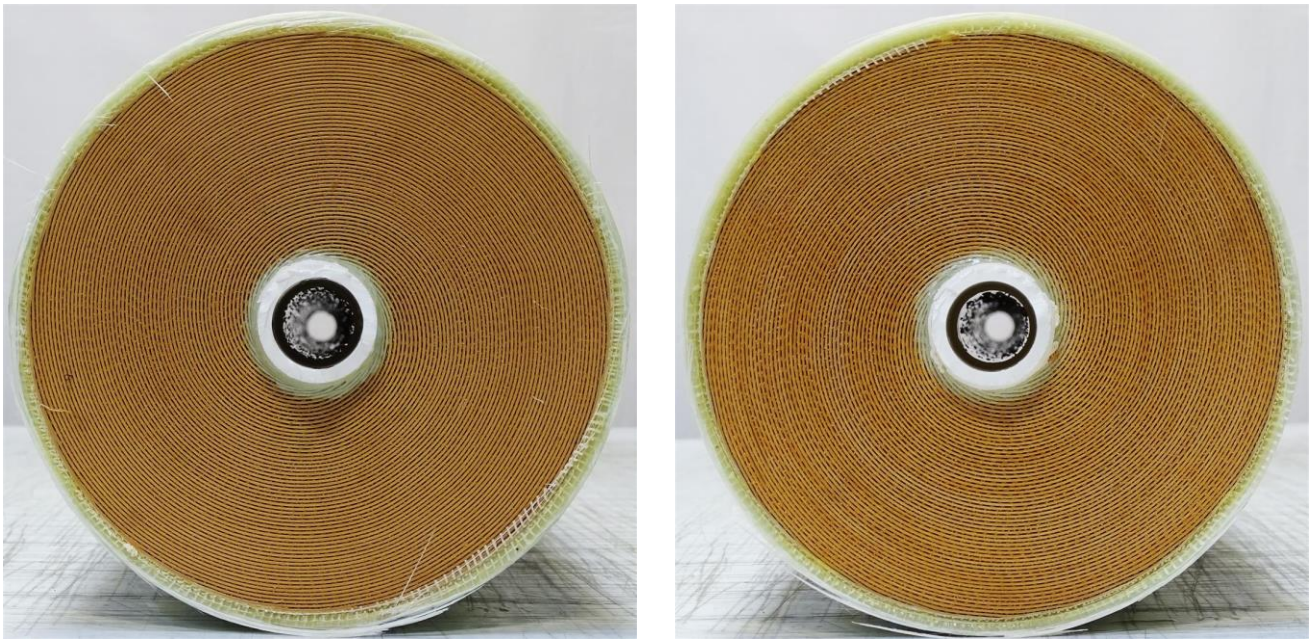
Feed (left) and concentrate (right) ATDs of SN T4048772

Internal Inspection

Scroll Ends

The ends of the element are called scroll ends. They are examined for the presence of foulant debris and mechanical damage (e.g. gapping, feed spacer extrusion). The presence of foulant on the scroll ends can cause elevated delta pressures while gapping and feed spacer extrusion indicate uneven hydraulics (high flow/low flow regions). In addition, each scroll end is examined for telescoping, the gradual axial shift of the membrane leaves from the outer diameter of the element towards the permeate tube. Telescoping is often caused by the development of high differential pressure (greater than the manufacturer's specification) across the element or when pressure is applied too quickly, causing a water hammer effect.

The scroll ends were in good mechanical condition.



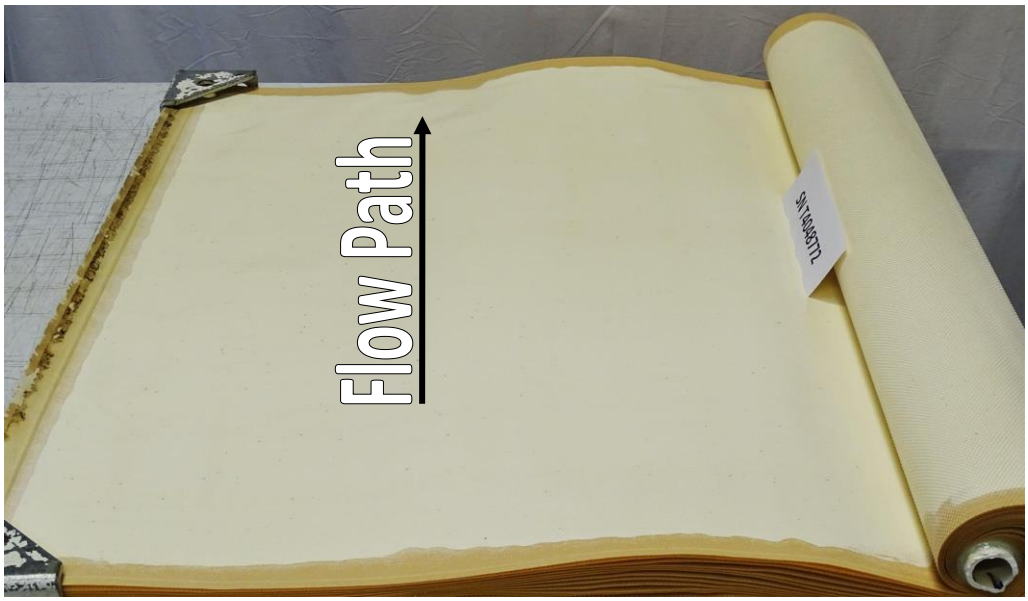
Feed (left) and concentrate (right) scroll ends of SN T4048772

Membrane Surface

New membrane surfaces are uniform and shiny. Foulant can often be detected through visual examination; however, membrane appearance can be misleading as some foulants are not visible. The presence of foulant on the membrane surface can cause elevated delta pressure, loss in flow and damage if the foulant is abrasive. Additionally, the membrane surface is inspected for damage such as delamination. Delamination is the lifting of the thin-film membrane from the support layer and often occurs due to a positive pressure on the permeate side of the element.

The membrane surface contained a small amount of brown colored foulant which was found primarily in the feed spacer contact points. Random spots were observed across the membrane on all membrane leaves.

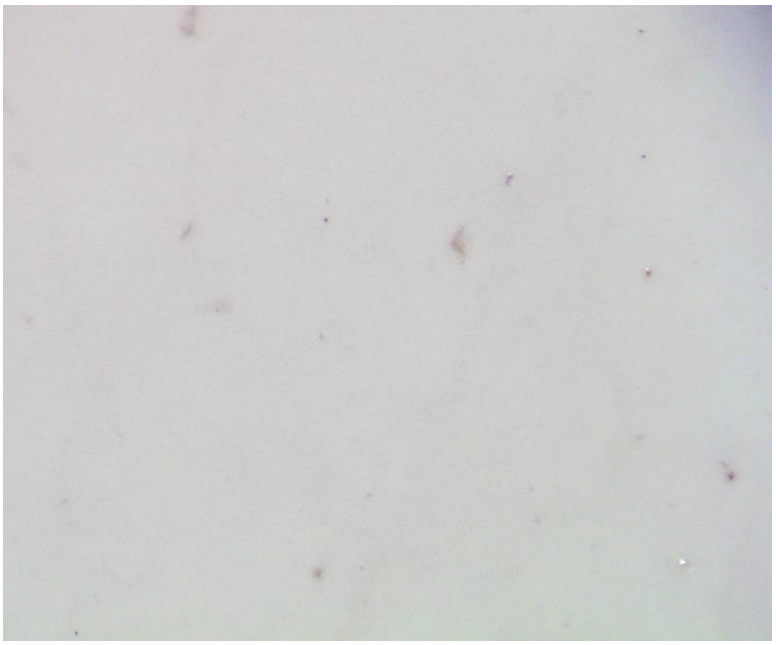
Exposed membrane surface of SN T4048772



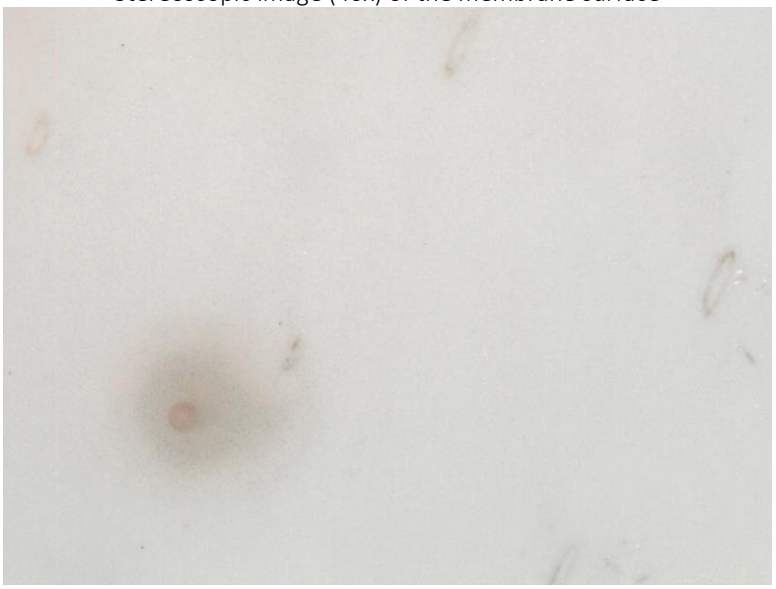
Exposed membrane surface from feed end of SN T4048772

Image of foulant scraped on the membrane surface

Image of spots observed on the membrane surface



Stereoscopic image (40x) of the membrane surface

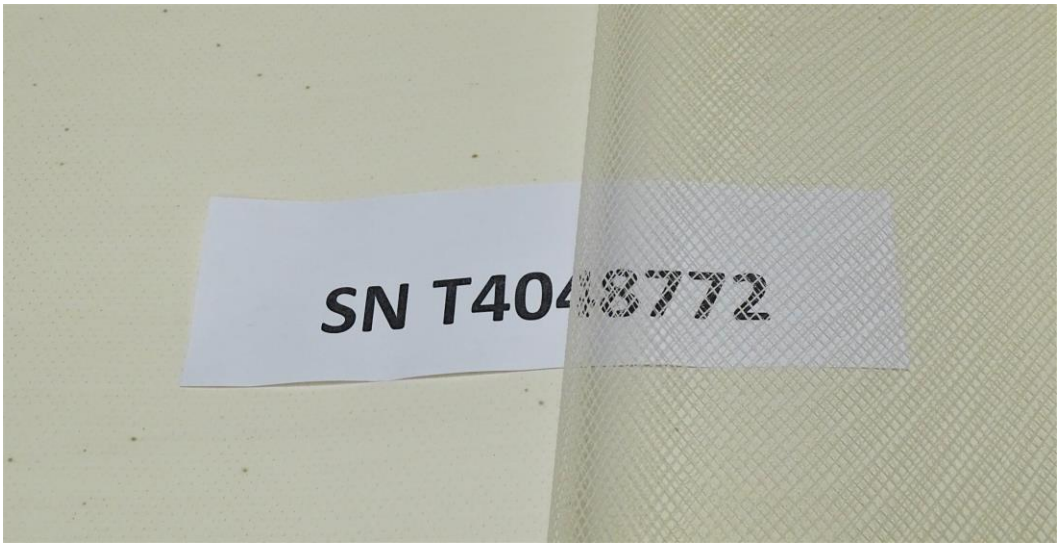


Stereoscopic image (40x) of a spot on the membrane surface

Feed Spacers

The feed spacer is a plastic net material designed to separate the membrane leaves, forming a flow path, and to promote turbulence within the feed water channels. Foulant blocking the feed channels causes more resistance for the feed water flowing through the element and results in higher than normal delta pressures.

Although some of the foulant was observed on the feed spacer they were in good mechanical condition and free of blockages.



Feed spacer of SN T4048772

Glue Lines

Membrane leaves are glued on three sides to separate the feed and permeate streams. The glue lines are inspected for specific damage, including glue flaps and pouching. Glue flaps refer to excess inactive membrane material located closest to the ends of the element. Flaps found on the feed end of the element can flare during operation, blocking the feed channels on the scroll end, potentially causing increased differential pressure. Pouching of the glue line, which is often a result of delamination, allows feed water to pass through the inactive membrane at the glue line, contaminating the permeate stream.

The glue lines were in good mechanical condition.

Permeate Carriers and Membrane Backing

The permeate carriers provide a path for permeate water to flow towards the permeate tube, which minimizes permeate-side pressure losses. New permeate carriers and membrane backing are uniform in color. Foulant found on the permeate side of the membrane leaves indicates contamination of the permeate stream.

Tan-colored spots were observed on the membrane backing and permeate carriers.

Membrane backing of SN T4048772

Foulant Analysis

Acid Testing

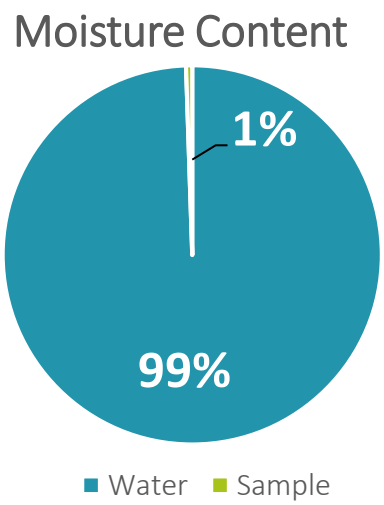
Acid testing is used to determine the presence of carbonates and metals on the membrane surface. In this test, several drops of dilute hydrochloric acid (HCl) were placed on the foulant surfaces. Effervescing indicates the presence of carbonates while a color change is associated with the presence of metals.

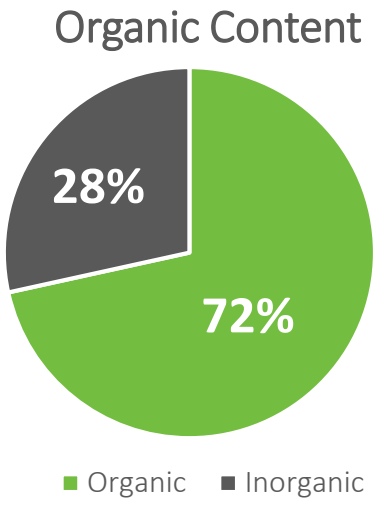
Acid testing was negative for the presence of carbonates and metals.

Foulant Density Measurement and Composition Testing

A sample collected from a known area of the membrane surface is weighed before and after drying to determine the foulant density (reported as dry foulant density – mg/cm^2) and moisture content of the sample. Different types of foulant materials exhibit higher moisture contents. Relative water concentrations greater than 95% indicate an extremely hydrated, biological material. Alternatively, scales (crystalline material) typically contain very little moisture. The organic content of the dehydrated material is then measured through loss on ignition (LOI) testing. If the organic content of the total solids is greater than 65%, it is considered primarily organic.

The wet foulant density was measured at 0.01 mg/cm^2. The moisture and organic contents are shown in the graphs below.

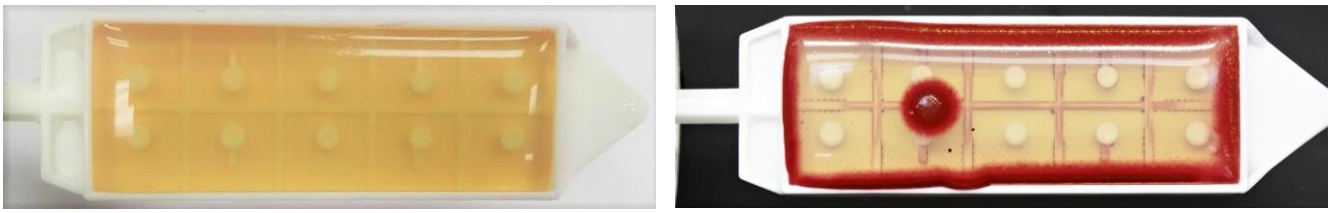




Biological Activity Testing

Dip slides for aerobic bacteria are exposed to the foulant material on the membrane surface. The slides are incubated for 72 hours and inspected for biological growth. Greater colony density, measured in colony-forming units (CFU)/cm^2, indicates a more biologically active sample.

The aerobic slide showed approximately 40 CFU/cm^2 after the 72 hour incubation period.

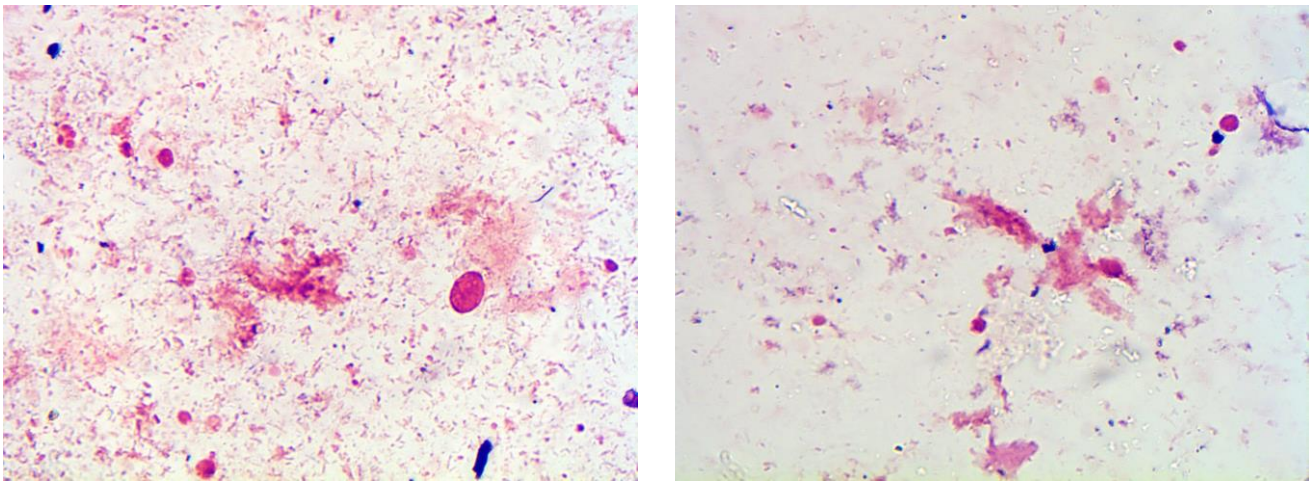


Aerobic bacteria slide before incubation (left) and after incubation (right)

Microbiological Analysis

This analysis is performed to identify microbiological components of the foulant removed from the membrane surface. Foulant samples are stained and examined with a light microscope at 1000x using an oil immersion lens. Gram positive bacteria are stained purple while Gram negative bacteria are stained pink.

Microbiological analysis performed on foulant scraped from the membrane surface identified bioslime, algae, yeast and bacteria.

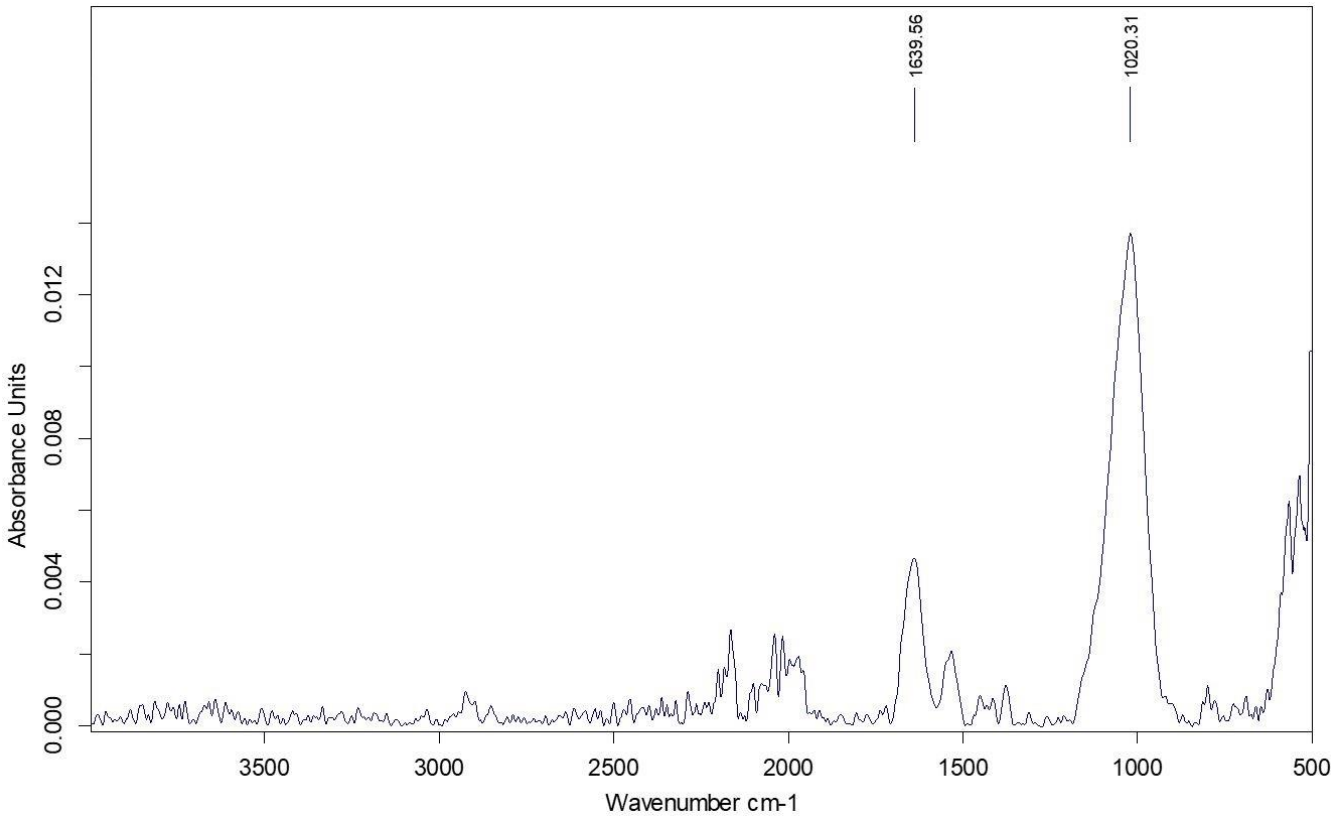


Light microscope images (1000x) of foulant scraped from SN T4048772

Fourier Transform Infrared Spectroscopy Analysis

Fourier Transform Infrared Spectroscopy (FT-IR) is an analytical technique used to identify functional groups (specific groups of atoms or bonds within molecules). Infrared radiation passes through a sample, with some of the radiation absorbed and some transmitted. A measurement and interpretation of this data produces a spectrum which can then be compared and matched to the known spectra for functional groups based on the wavenumber at which bands appear and their respective shapes (e.g. sharp, broad, strong, weak).

FT-IR spectroscopy performed on foulant scraped from the membrane surface displayed a strong, sharp peak at approximately 1000 cm^{-1} which is associated with carbohydrates and Si-O bond stretching. The double peak between 1650 and 1500 cm^{-1} is associated with amino acids (i.e. proteins) from microorganisms. The weaker bands between 2200 cm^{-1} and 2000 cm^{-1} are contributed by lipids (hydrophobic fatty acids).



FT-IR spectral image of foulant removed from the membrane surface of SN T4048772

Energy Dispersive Spectroscopy (EDS) Analysis

Energy Dispersive Spectroscopy analysis is used to determine the relative concentration of elements present in a sample. EDS analysis is performed on a dry membrane sample. The element sulfur is at least in part associated with the membrane support material (polysulfone) rather than a foulant layer. Avista's analysis of new membranes typically detects between 5.00 and 7.00 weight percentage. Relative concentrations below 5.00 percent indicate the presence of a foulant layer masking the membrane surface.

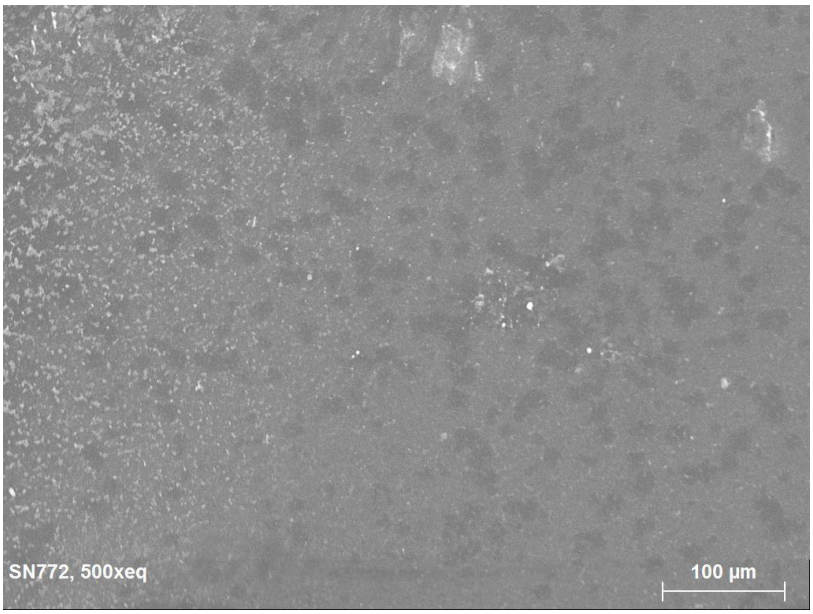
EDS analysis detected trace amounts (<0.50 wt%) of silicon and aluminum as the only inorganic elements present in the foulant. The sulfur weight percent (6.71 wt%) was above normal suggesting the presence of foreign sulfur.

Elements	SN T4048772 Weight Percent
Carbon	77.96
Oxygen	15.08
Sulfur	6.71
Silicon	0.13
Aluminum	0.12

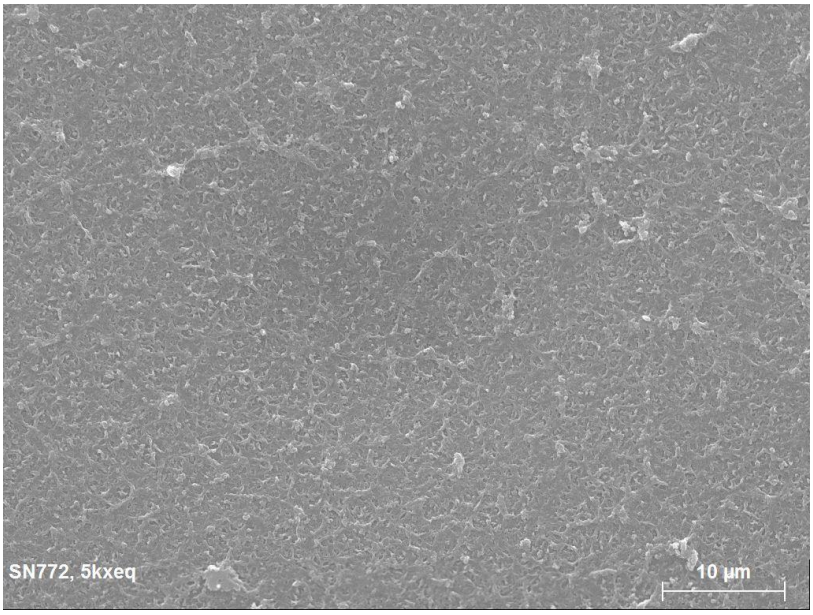
Scanning Electron Microscope (SEM) Imaging

SEM imaging is performed on the membrane surface to observe the topography of the foulant material. Foulant morphology can be an indicator of the type of foulant.

SEM images displayed patches of smooth foulant across the membrane. Random particles were also observed across the membrane.



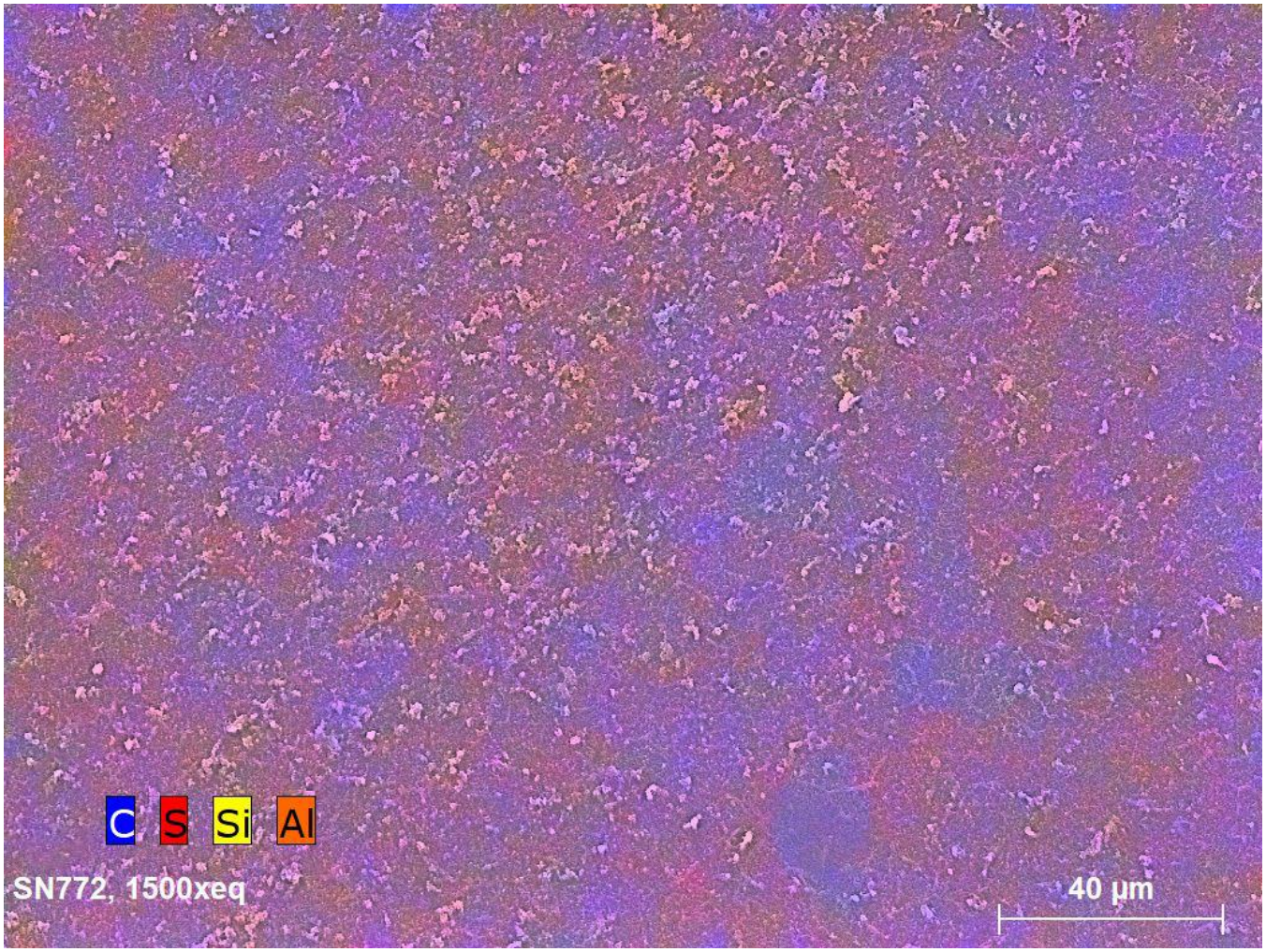
SEM image (150x) of the membrane surface of SN T4048772



Close-up SEM image (5000x) of the membrane surface of SN T4048772

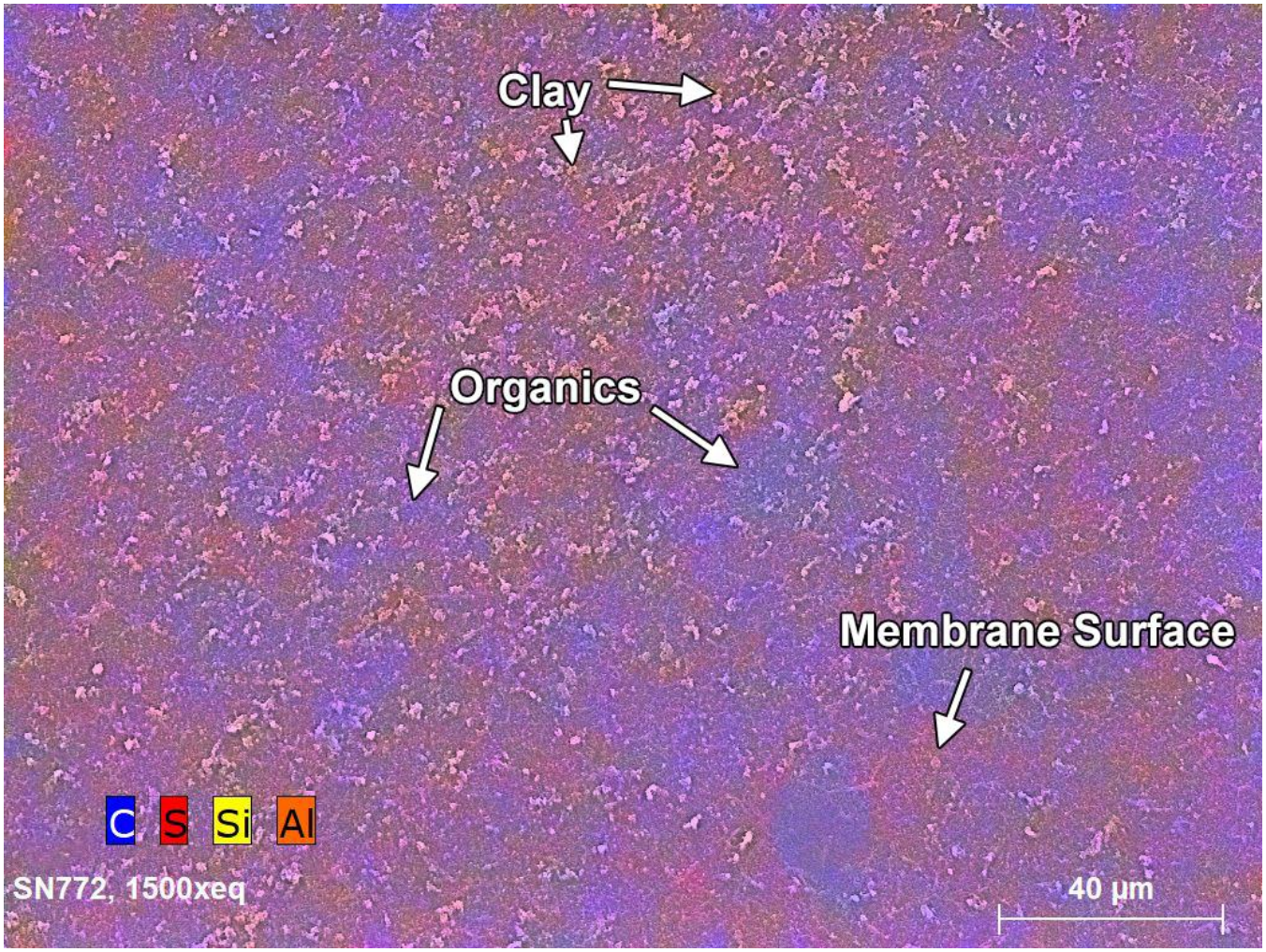
Chromatic Elemental Imaging^{SM} (CEI^{SM})

CEI is a high-resolution imaging technique used to determine the spatial distribution of elements in a foulant sample. Each element is assigned a color (shown in a legend on the bottom left corner of the CEI image) and the colors correspond to the location of the elements in the sample. An element's color intensity is associated with its concentration in the sample (i.e. elements present with higher relative concentrations are displayed with greater color intensity in the image). Additionally, a blending of colors signifies a compound (material composed of more than one element such as calcium carbonate).



CEI image (1500x) of the membrane surface

CEI[SM] identified patches of organics (high carbon content-dark blue) across the membrane. The patches were relatively thin allowing the signal from the membrane (sulfur-red) to be visible through some of the patches. Clay (yellow-orange) particles were observed embedded within and above the organic material.



CEI image (1500x) of the membrane surface with labels

Flat Sheet Performance and Cleaning Study

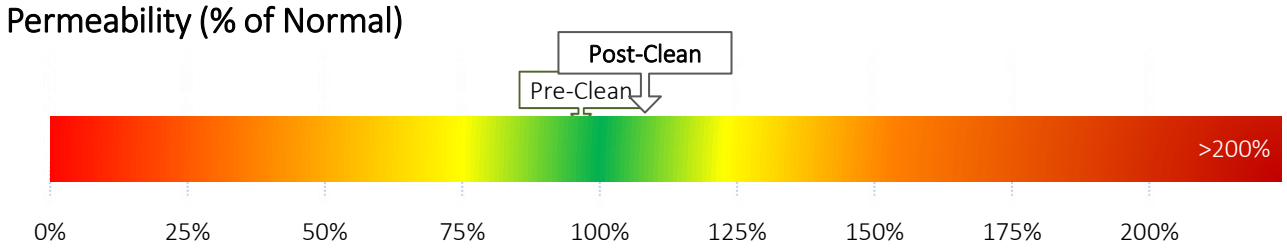
To evaluate flat sheet performance, membrane samples harvested from the full element are tested for permeability and salt passage. The raw flow and conductivity measurements from the test are used to calculate the permeability and salt passage constants, which are independent of pressure, temperature and salt content of the feed stream. The permeability constant is measured in cm/s/atm and the salt passage constant in cm/s. Discrepancies between the flat sheet and full element performance can indicate the presence of mechanical damage.

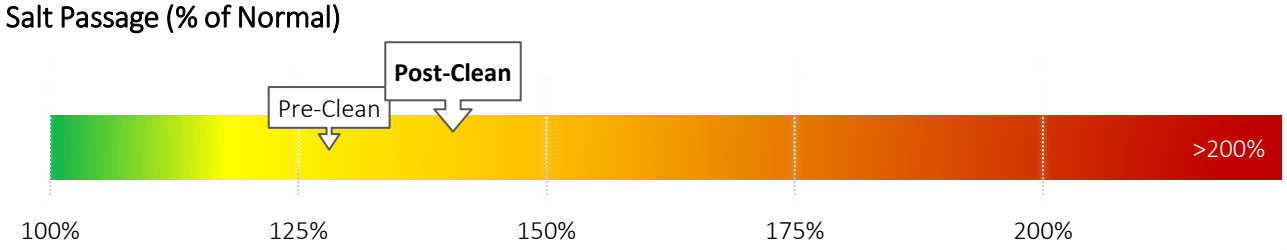
The flat sheet samples are then cleaned with various Avista chemicals to determine the most effective cleaner combinations and contact times. Cleaner efficacy is based on overall improvement in permeability and salt passage constant as well as visual foulant removal.

Flat sheet samples harvested from the full element produced normal permeability and 116% of normal salt passage during baseline cell testing. Flat sheet samples were cleaned with RoClean P111 (2% by weight in RO/DI water and heated to approximately 35 degrees Celsius and circulated) for 2 hours which removed the visual foulant and increased flow by approximately 11%; however, salt passage remained high before and after cleaning.

SN T4048772	Permeability Constant	Salt Passage Constant
Pre-Clean	1.33E-04 Normal	12.48E-06 128% of Normal
Post-Clean	1.47E-04 Normal	13.26E-06 136% of Normal
Manufacturer's Specifications	1.10 to 1.37E-04 Normal Range	5.67 to 9.75E-06 Normal Range

Note: testing conducted with dechlorinated city water from San Marcos, CA





Testing for Flat Sheet Damage

Fujiwara Testing

Fujiwara testing is a qualitative analysis which determines if a polyamide (PA) thin-film membrane has been exposed to an oxidizing halogen, such as chlorine, bromine, or iodine. A color change does not occur if the membranes has not been exposed to halogens. Common symptoms of halogen oxidation include increased flow and loss in permeate quality.

Fujiwara testing was negative for the presence of halogens (e.g. chlorine) in the membrane structure.

Example of a negative Fujiwara color change

Dye Test

Cleaned flat sheet samples were exposed to dye in a cell test apparatus at 100 psi for 15 minutes. Physically and/or chemically damaged membranes will absorb the dye on the membrane surface. Dye penetration through the membrane backing indicates severe physical and/or chemical damage.

Dye passed through the membrane in the areas where the spots were identified indicating severe damage in these areas.

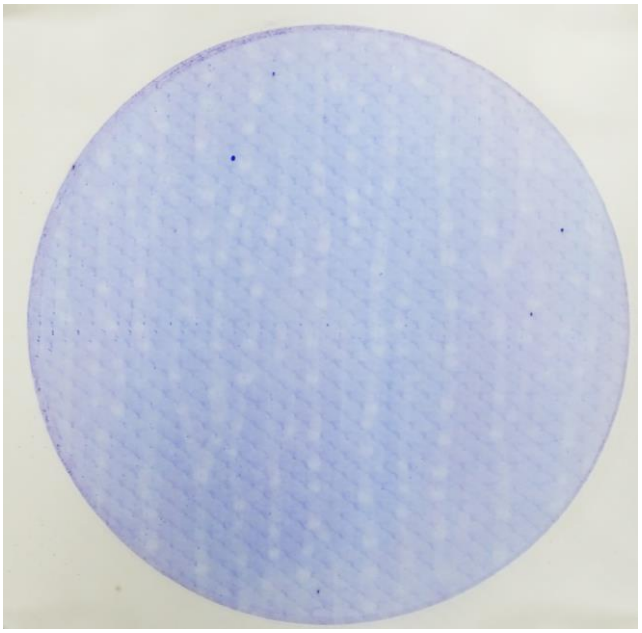
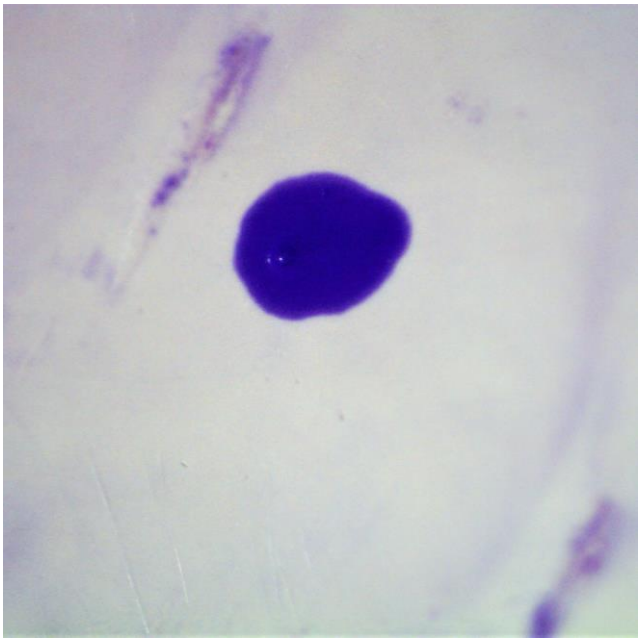


Image of dye uptake on the membrane surface

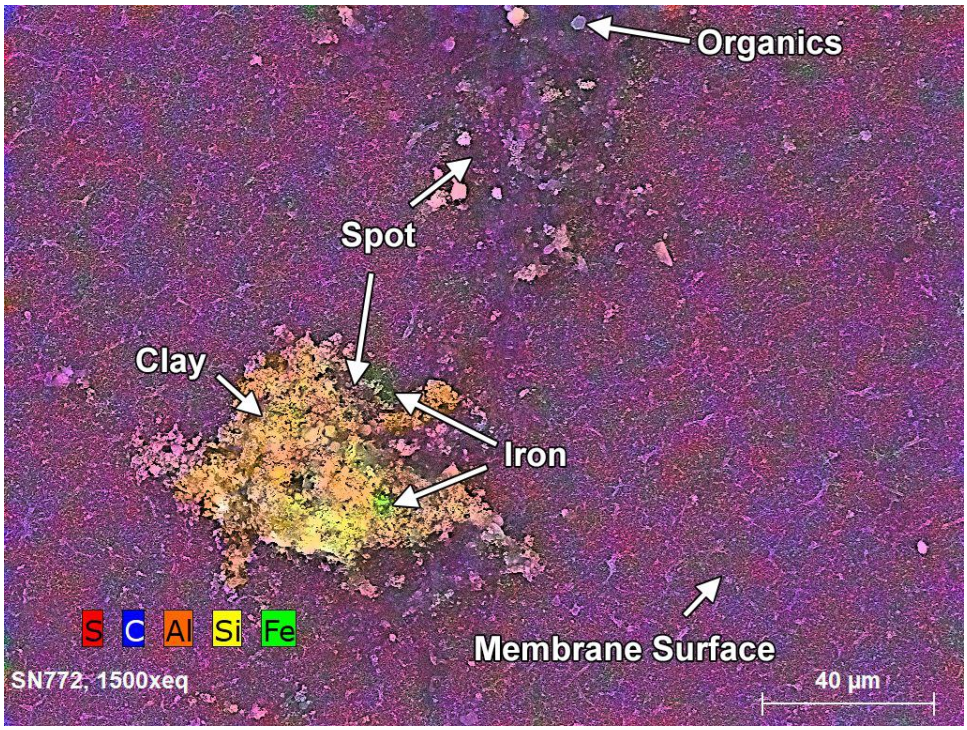


Stereoscopic image (20x) of dye uptake on the tan-colored spots of the membrane surface

Spot Analysis

EDS analysis and CEI detected iron in the areas where the spots were observed which was not in present in areas away from the spots.

Elements	SN T4048772 Away from Spot Weight Percent	SN T4048772 At Spot Weight Percent
Carbon	77.96	72.68
Oxygen	15.08	20.08
Sulfur	6.71	6.56
Silicon	0.13	0.33
Aluminum	0.12	0.26
Iron	ND	0.09



CEI image (1500x) of a spot observed on the membrane surface

Certification by Laboratory

Report Number	Report Content	Element Serial Number	Report Date
WO#120219-3	Standard Spiral Autopsy	T4048676 T4048771 T4048772	February 20th, 2020

We the undersigned being the technical specialists in membrane autopsy and related testing procedures and protocol for Avista Technologies certify to the best of our knowledge and belief that the tests listed in this report have been conducted following Avista's standard testing practices and that the results are accurate and complete.

By signing this certificate neither the laboratory employees nor their employer makes any warranty, expressed or implied, concerning the cleaning study results.

Date: 02/20/2020

Signed:

Megan Lee
Laboratory Services Manager

Jaime La Cuesta
Laboratory Services Chemist

Appendix B

Data Record for CCRO Testing at Padre Dam

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Routine Monitoring Surrogate Data

					Eurofins Report	816785	818050	819161	820491	821847	823300	824554	825619
					Eurofins locator	201907180462	201907250335	201908010338	201908090225	201908150946	201908220632	201908290704	201909050548
PRO FEED	Parameter	Units	Method	MRL	MDL	7/18/19	7/25/19	8/1/19	8/8/19	8/15/19	8/22/19	8/29/19	9/5/19
	Strontium	mg/L	EPA 200.7	0.1	0.002	0.34	0.38	0.38	0.32	0.29	0.31	0.3	0.34
	Magnesium	mg/L	EPA 200.7	0.1	0.003	18	18	18	15	15	16	15	16
	Total Organic Carbon	mg/L	SM5310C/E415.3	0.3	0.042	7.2	6.7	6.6	7.0	6.5	6.9	7.2[A]	6.9
	Sulfate	mg/L	EPA 300.0	0.5	0.06	180	190	180	170	180	180	180	180
	Total Dissolved Solids (TDS)	mg/L	E160.1/SM2540C	10	4.2	620	660	660	610[A]	620	600	600	620
	Orthophosphate	mg-P/L	EPA 365.1	0.01	0.007	0.19	0.28	0.31	0.19	0.26	0.21	0.11	0.071
	Conductivity (Field Grab)	µS/cm					1123	1123	1048		1015	1035	

					Eurofins Report	816785	818050	819161	820491	821847	823300	824554	825619
					Eurofins locator	201907180461	201907250336	201908010337	201908090224	201908150945	201908220631	201908290705	201909050549
PRO PERMEATE	Parameter	Units	Method	MRL	MDL	7/18/19	7/25/19	8/1/19	8/8/19	8/15/19	8/22/19	8/29/19	9/5/19
	Strontium	µg/L	EPA 200.8	0.3	0.016	0.13[B]	0.17[B]	0.12[B]	0.092[B]	0.082[B]	0.087[B]	0.0889[B]	0.1[B]
	Magnesium	mg/L	EPA 200.7	0.1	0.003	ND	0.0062[B]	ND	0.006[B]	ND	0.0034[B]	ND	ND
	Total Organic Carbon	mg/L	SM5310C/E415.3	0.3	0.042	0.15[B]	0.086[B]	0.057[B]	0.12[B]	0.054[B]	0.066[B]	0.21[A,B]	0.085[B]
	Sulfate	mg/L	EPA 300.0	0.5	0.06	0.11[B]	0.061[B]	0.074[B]	ND	ND	ND	ND	ND
	Total Dissolved Solids (TDS)	mg/L	E160.1/SM2540C	10	4.2	12	15	12	11	16	7[B,D]	9[B,D]	9[B]
	Orthophosphate	mg-P/L	EPA 365.1	0.01	0.007	ND	ND	ND	ND	ND	ND	ND	ND
	Conductivity (Field Grab)	µS/cm					29.45	25.68	23.8		23.3	22.44	

					Eurofins Report	816785	818050	819161	820491	821847	823300	824554	825619
					Eurofins locator	201907180464	201907250337	201908010340	201908090227	201908150948	201908220634	201908290706	201909050550
CCRO FEED	Parameter	Units	Method	MRL	MDL	7/18/19	7/25/19	8/1/19	8/8/19	8/15/19	8/22/19	8/29/19	9/5/19
	Strontium	mg/L	EPA 200.7	0.1	0.002	1.3	1.5	1.5	1.2	1.2	1.3	1.2	1.3
	Magnesium	mg/L	EPA 200.7	0.1	0.003	69	74	69	57	62	63	61	65
	Total Organic Carbon	mg/L	SM5310C/E415.3	0.3	0.042	27	26	27	26	29	28	29[A]	28
	Sulfate	mg/L	EPA 300.0	0.5	0.06	710	750	740	660	700	720	740	750
	Total Dissolved Solids (TDS)	mg/L	E160.1/SM2540C	10	4.2	2400	2600	2600	2400[A]	2400	2300	2400	2400
	Orthophosphate	mg-P/L	EPA 365.1	0.01	0.007	0.69	0.65	0.98	0.8	1.0	0.85	0.47	0.26
	Conductivity (Field Grab)	µS/cm					3912	4073	3685	3657	3603	3702	

					Eurofins Report	816785	818050	819161	820491	821847	823300	824554	825619
					Eurofins locator	201907180463	201907250338	201908010339	201908090226	201908150947	201908220633	201908290707	201909050551
CCRO PERMEATE (Tank)	Parameter	Units	Method	MRL	MDL	7/18/19	7/25/19	8/1/19	8/8/19	8/15/19	8/22/19	8/29/19	9/5/19
	Strontium	µg/L	EPA 200.8	0.3	0.016	0.69	3.3	11	29	110	2	0.38	0.34
	Magnesium	mg/L	EPA 200.7	0.1	0.003	0.022[B]	0.16	0.54	1.5	5.1	0.11	0.015[B]	0.013[B]
	Total Organic Carbon	mg/L	SM5310C/E415.3	0.3	0.042	0.21[B]	0.19[B]	0.38	0.68	2.3	0.2[B]	0.27[A,B]	0.13[B]
	Sulfate	mg/L	EPA 300.0	0.5	0.06	0.41	1.6	5.3	16	59	0.61	0.38	0.31
	Total Dissolved Solids (TDS)	mg/L	E160.1/SM2540C	10	4.2	36	49	56	90	230	66	54	50
	Orthophosphate	mg-P/L	EPA 365.1	0.01	0.007	ND	ND	0.009	0.015	0.0089	ND	ND	ND
	Conductivity (Field Grab)	µS/cm					90.73	120.8	182.9	418.5	123.7	107.8	

MRL = method reporting limit (Eurofins Eaton Analytical) MDL = method detection limit ND = non-detect (i.e., < MDL)

[A] *Target analyte detected in blank at or above method acceptance criteria.*

[B] *The analyte was either detected at or greater than the MDL and less than the MRL, or did not meet any one of the required QC criteria.*

[C] *Sample received with inadequate chemical preservation, but preserved by the laboratory.*

[D] *Result estimated. Minimum 2.5 mg dried residue for SM2540 was not met.*

Routine Monitoring Surrogate Data

			827001	828424	829762	830780	832036	833490	834822	836230	837574	838840
			201909120497	201909190340	201909230382	201910020595	201910090666	201910160406	201910230621	201910310675	201911070575	201911140599
PRO FEED	**Parameter**	**Units**	**9/12/19**	**9/19/19**	**9/25/19**	**10/2/19**	**10/9/19**	**10/16/19**	**10/23/19**	**10/30/19**	**11/7/19**	**11/14/19**
	Strontium	mg/L	0.31	0.32	0.29	0.28	0.29	0.28	0.28	0.3	0.27	0.29
	Magnesium	mg/L	15	15	14	13	14	14	14	15	14	14
	Total Organic Carbon	mg/L	7.0	7.2	6.5	6.5	6.5	6.5	7.1	6.8	6.7	7.1
	Sulfate	mg/L	160	180	160	170	170	170	170	170	170	180
	Total Dissolved Solids (TDS)	mg/L	620	620	600	590	600	580	560	590	620	620
	Orthophosphate	mg-P/L	0.088	0.084	0.14	0.11	0.084	0.086	0.096	0.089	0.077	0.097
	Conductivity (Field Grab)	µS/cm	1057	1053	1041	1025	1023	986.5	978.7	995.7	1020	1008

			827001	828424	829762	830780	832036	833490	834822	836230	837574	838840
			201909120496	201909190339	201909230383	201910020596	201910090667	201910160407	201910230622	201910310676	201911070576	201911140600
PRO PERMEATE	**Parameter**	**Units**	**9/12/19**	**9/19/19**	**9/25/19**	**10/2/19**	**10/9/19**	**10/16/19**	**10/23/19**	**10/30/19**	**11/7/19**	**11/14/19**
	Strontium	µg/L	0.12[B]	0.09[B]	0.1[B]	0.14[B]	0.067[B]	0.071[B]	0.064[B]	0.062[B]	0.053[B]	0.053[B]
	Magnesium	mg/L	ND	0.0053[B]	ND	ND	ND	0.0031[B]	ND	ND	ND	ND
	Total Organic Carbon	mg/L	0.11[B]	0.15[B]	0.055[B]	0.11[B]	0.1[B]	0.074[B]	0.053[B]	0.1[B]	ND[C]	0.064[B,C]
	Sulfate	mg/L	ND	ND	ND	ND	ND	0.16[B]	ND	ND	0.23[B]	ND
	Total Dissolved Solids (TDS)	mg/L	13	15	12	14	13	12	9[B]	11	17	18
	Orthophosphate	mg-P/L	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
	Conductivity (Field Grab)	µS/cm	22.37	23.4	22.43	20.33	20.56	24.56	20.07	20.96	19.9	19.33

			827001	828424	829762	830780	832036	833490	834822	836230	837574	838840
			201909120499	201909190342	201909230384	201910020597	201910090668	201910160408	201910230623	201910310677	201911070577	201911140601
CCRO FEED	**Parameter**	**Units**	**9/12/19**	**9/19/19**	**9/25/19**	**10/2/19**	**10/9/19**	**10/16/19**	**10/23/19**	**10/30/19**	**11/7/19**	**11/14/19**
	Strontium	mg/L	1.2	1.3	1.1	1.1	1.1	1.1	1	1.2	1.1	1
	Magnesium	mg/L	56	62	54	51	55	53	53	57	57	53
	Total Organic Carbon	mg/L	29	24	28	25	26	26	33	26	27	24
	Sulfate	mg/L	610	680	670	670	680	670	670	700	680	710
	Total Dissolved Solids (TDS)	mg/L	2400	2400	2300	2300	2400	2200	2200	2300	2400	2300
	Orthophosphate	mg-P/L	0.34	0.33	0.59	0.5	0.34	0.34	0.39	0.39	0.3	ND
	Conductivity (Field Grab)	µS/cm	3682	3705	3709	3566	3617	3368	3334	3555	3692	3585

			827001	828424	829762	830780	832036	833490	834822	836230	837574	838840
			201909120498	201909190341	201909230385	201910020598	201910090669	201910160409	201910230624	201910310678	201911070578	201911140602
CCRO PERMEATE (Tank)	**Parameter**	**Units**	**9/12/19**	**9/19/19**	**9/25/19**	**10/2/19**	**10/9/19**	**10/16/19**	**10/23/19**	**10/30/19**	**11/7/19**	**11/14/19**
	Strontium	µg/L	0.33	0.93	0.35	0.31	0.41	0.69	2.6	12	33	15
	Magnesium	mg/L	ND	0.016[B]	0.0059[B]	0.014[B]	0.016[B]	0.036[B]	0.14	0.63	1.7	0.83
	Total Organic Carbon	mg/L	0.2[B]	0.24[B]	0.18[B]	0.15[B]	0.15[B]	0.12[B]	0.53	0.36	0.85	0.51[C]
	Sulfate	mg/L	0.25	0.28	0.3	0.24	0.28	0.51	1.9	7.2	22	10
	Total Dissolved Solids (TDS)	mg/L	74	70	60	50	49	46	44	62	120	92
	Orthophosphate	mg-P/L	ND	ND	ND	ND	ND	ND	ND	ND	0.01	ND
	Conductivity (Field Grab)	µS/cm	106.7	117.7	111.1	89.07	87.71	79.21	82.12	114.1	215	135.9

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[C] *Sample received with inadequate chemical preservation, but preserved by the laboratory.*

[D] *Result estimated. Minimum 2.5 mg dried residue for SM2540 was not met.*

Appendix B - Data Record for CCRO Testing at Padre Dam

Routine Monitoring Surrogate Data

			842607	842606	844071	845417	845431
				201912050382	201912120631	201912190402	201912190441
PRO FEED	**Parameter**	**Units**	**12/4/19**	**12/5/19**	**12/11/19**	**12/18/19**	**12/19/19**
	Strontium	mg/L		0.33	0.32	0.31	0.31
	Magnesium	mg/L		17	18	18	18
	Total Organic Carbon	mg/L		8.8	6.3	6.9	7.1
	Sulfate	mg/L		170	170	140	150
	Total Dissolved Solids (TDS)	mg/L		580	630	600	590
	Orthophosphate	mg-P/L		0.032	0.045	0.036	0.034
	Conductivity (Field Grab)	µS/cm	1033	994.1	1051	1045	1021

			842607	842606	844071	845417	845431
				201912050383	201912120632	2019121990403	201912190442
PRO PERMEATE	**Parameter**	**Units**	**12/4/19**	**12/5/19**	**12/11/19**	**12/18/19**	**12/19/19**
	Strontium	µg/L		0.042[B]	0.063[B]	0.03[B]	0.031[B]
	Magnesium	mg/L		ND	0.005[B]	ND	0.004[B]
	Total Organic Carbon	mg/L		0.1[B,C]	0.079[B,C]	0.1[B,C]	0.043[B,C]
	Sulfate	mg/L		ND	ND	ND	ND
	Total Dissolved Solids (TDS)	mg/L		ND	12	ND	ND
	Orthophosphate	mg-P/L		ND	0.011	ND	ND
	Conductivity (Field Grab)	µS/cm	18.66	22.76	15.82	15.13	15.49

			842607	842606	844071	845417	845431
			201912050388	201912050384	201912120633	201912190404	201912190444
CCRO FEED	**Parameter**	**Units**	**12/4/19**	**12/5/19**	**12/11/19**	**12/18/19**	**12/19/19**
	Strontium	mg/L	1.4	1.4	1.2	1.1	1.2
	Magnesium	mg/L	71	69	69	64	66
	Total Organic Carbon	mg/L	28	37	33	28	28
	Sulfate	mg/L	710	700	680	590	610
	Total Dissolved Solids (TDS)	mg/L	2500	2400	2500	2400	2300
	Orthophosphate	mg-P/L	0.1	0.1	0.16	0.14	0.12
	Conductivity (Field Grab)	µS/cm	3659	3638		3785	3755

			842607	842606	844071	845417	845431
			201912050389	201912050385	201912120633	201912190405	201912190444
CCRO PERMEATE (Tank)	**Parameter**	**Units**	**12/4/19**	**12/5/19**	**12/11/19**	**12/18/19**	**12/19/19**
	Strontium	µg/L	80	1.1	0.48	0.34	0.46
	Magnesium	mg/L	4.4	0.059[B]	0.023[B]	0.017[B]	0.031[B]
	Total Organic Carbon	mg/L	1.7[C]	0.13[B,C]	0.13[B,C]	0.12[B,C]	0.083[B,C]
	Sulfate	mg/L	43	1.3	0.84	0.54	0.63
	Total Dissolved Solids (TDS)	mg/L	190	36	60	56	72
	Orthophosphate	mg-P/L	0.007	ND	ND	ND	ND
	Conductivity (Field Grab)	µS/cm	371	90.64		113.61	108.8

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[C] *Sample received with inadequate chemical preservation, but preserved by the laboratory.*

[D] *Result estimated. Minimum 2.5 mg dried residue for SM2540 was not met.*

Appendix B - Data Record for CCRO Testing at Padre Dam

MS2 Challenge Test Data

Report Number BVL-734
Date of Test: 9/25/19

Sample ID#	Sample Location	MS2 (pfu/mL)
19BVL-225-001	PRO Feed (1 of 3)	2.30E+06
19BVL-225-002	PRO Feed (2 of 3)	2.00E+06
19BVL-225-003	PRO Feed (3 of 3)	2.40E+06
19BVL-225-004	CCRO Feed (1 of 3)	1.10E+07
19BVL-225-005	CCRO Feed (2 of 3)	9.60E+06
19BVL-225-006	CCRO Feed (3 of 3)	1.00E+07
19BVL-225-007	PRO Permeate (1 of 3)	3.00E+00
19BVL-225-008	PRO Permeate (2 of 3)	6.00E+00
19BVL-225-009	PRO Permeate (3 of 3)	3.00E+00
19BVL-225-010	CCRO Permeate (1 of 3)	2.90E+02
19BVL-225-011	CCRO Permeate (2 of 3)	3.20E+02
19BVL-225-012	CCRO Permeate (3 of 3)	4.30E+02

Report Number: BVL-2625
Date of Test: 10/30/19

Sample ID#	Sample Location	MS2 (pfu/mL)
19BVL-378-001	PRO Feed (1 of 3)	3.60E+06
19BVL-378-002	PRO Feed (2 of 3)	4.60E+06
19BVL-378-003	PRO Feed (3 of 3)	2.50E+06
19BVL-378-004	CCRO Feed (Tank) (1 of 3)	1.90E+07
19BVL-378-005	CCRO Feed (Tank) (2 of 3)	1.60E+07
19BVL-378-006	CCRO Feed (Tank) (3 of 3)	3.70E+07
19BVL-378-007	PRO Permeate (1 of 3)	4.00E+00
19BVL-378-008	PRO Permeate (2 of 3)	6.00E+00
19BVL-378-009	PRO Permeate (3 of 3)	2.00E+00
19BVL-378-010	CCRO Permeate (Tank) (1 of 3)	5.00E+05
19BVL-378-011	CCRO Permeate (Tank) (2 of 3)	3.70E+06
19BVL-378-012	CCRO Permeate (Tank) (3 of 3)	1.00E+05
19BVL-378-013	CCRO Permeate (Early) (1 of 3)	1.00E+05
19BVL-378-014	CCRO Permeate (Early) (2 of 3)	5.50E+06
19BVL-378-015	CCRO Permeate (Early) (3 of 3)	1.00E+05
19BVL-378-016	CCRO Permeate (Middle) (1 of 3)	1.00E+05
19BVL-378-017	CCRO Permeate (Middle) (2 of 3)	1.00E+05
19BVL-378-018	CCRO Permeate (Middle) (3 of 3)	2.00E+05
19BVL-378-019	CCRO Permeate (Late) (1 of 3)	2.00E+05
19BVL-378-020	CCRO Permeate (Late) (2 of 3)	1.00E+05
19BVL-378-021	CCRO Permeate (Late) (3 of 3)	1.00E+05
19BVL-378-022	CCRO Feed-Brine (Early) (1 of 3)	2.10E+07
19BVL-378-023	CCRO Feed-Brine (Early) (2 of 3)	4.60E+07
19BVL-378-024	CCRO Feed-Brine (Early) (3 of 3)	2.70E+07
19BVL-378-025	CCRO Feed-Brine (Middle) (1 of 3)	4.10E+07
19BVL-378-026	CCRO Feed-Brine (Middle) (2 of 3)	6.20E+07
19BVL-378-027	CCRO Feed-Brine (Middle) (3 of 3)	4.10E+07
19BVL-378-028	CCRO Feed-Brine (Late) (1 of 3)	4.60E+07
19BVL-378-029	CCRO Feed-Brine (Late) (2 of 3)	6.60E+07
19BVL-378-030	CCRO Feed-Brine (Late) (3 of 3)	6.20E+07
19BVL-378-031	Field Blank PRO Permeate	1.00E+00
19BVL-378-032	Field Blank CCRO Permeate (Tank)	<1

Report Number: BVL-3902
Date of Test: 12/11/19

Sample ID#	Sample Location	MS2 (pfu/mL)
19BVL-535-001	PRO Feed (1 of 3)	3.70E+06
19BVL-535-002	PRO Feed (2 of 3)	3.60E+06
19BVL-535-003	PRO Feed (3 of 3)	4.50E+06
19BVL-535-004	CCRO Feed (Tank) (1 of 3)	2.30E+07
19BVL-535-005	CCRO Feed (Tank) (2 of 3)	1.60E+07
19BVL-535-006	CCRO Feed (Tank) (3 of 3)	1.00E+04
19BVL-535-007	PRO Permeate (1 of 3)	2.00E+00
19BVL-535-008	PRO Permeate (2 of 3)	2.00E+00
19BVL-535-009	PRO Permeate (3 of 3)	1.00E+00
19BVL-535-010	CCRO Permeate (Tank) (1 of 3)	5.00E+00
19BVL-535-011	CCRO Permeate (Tank) (2 of 3)	1.00E+01
19BVL-535-012	CCRO Permeate (Tank) (3 of 3)	4.00E+00
19BVL-535-013	CCRO Permeate (Early) (1 of 3)	3.00E+00
19BVL-535-014	CCRO Permeate (Early) (2 of 3)	4.00E+00
19BVL-535-015	CCRO Permeate (Early) (3 of 3)	5.00E+00
19BVL-535-016	CCRO Permeate (Middle) (1 of 3)	9.00E+00
19BVL-535-017	CCRO Permeate (Middle) (2 of 3)	5.00E+00
19BVL-535-018	CCRO Permeate (Middle) (3 of 3)	5.00E+00
19BVL-535-019	CCRO Permeate (Late) (1 of 3)	2.00E+01
19BVL-535-020	CCRO Permeate (Late) (2 of 3)	2.00E+01
19BVL-535-021	CCRO Permeate (Late) (3 of 3)	1.00E+01
19BVL-535-022	CCRO Feed-Brine (Early) (1 of 3)	5.60E+07
19BVL-535-023	CCRO Feed-Brine (Early) (2 of 3)	3.60E+07

Report Number: BVL-3902 (continued)
Date of Test: 12/11/19

19BVL-535-024	CCRO Feed-Brine (Early) (3 of 3)	6.80E+07
19BVL-535-025	CCRO Feed-Brine (Middle) (1 of 3)	6.70E+07
19BVL-535-026	CCRO Feed-Brine (Middle) (2 of 3)	4.30E+07
19BVL-535-027	CCRO Feed-Brine (Middle) (3 of 3)	6.40E+07
19BVL-535-028	CCRO Feed-Brine (Late) (1 of 3)	6.80E+07
19BVL-535-029	CCRO Feed-Brine (Late) (2 of 3)	7.40E+07
19BVL-535-030	CCRO Feed-Brine (Late) (3 of 3)	6.50E+07
19BVL-535-031	Field Blank PRO Permeate	<1
19BVL-535-032	Field Blank CCRO Permeate (Tank)	<1

Report Number BVL-3875
Date of Test: 12/17/19

Sample ID#	Sample Location	MS2 (pfu/mL)
19BVL-573-001	PRO Feed (1 of 3)	4.00E+06
19BVL-573-002	PRO Feed (2 of 3)	6.90E+06
19BVL-573-003	PRO Feed (3 of 3)	5.90E+06
19BVL-573-004	CCRO Feed (Tank) (1 of 3)	1.90E+07
19BVL-573-005	CCRO Feed (Tank) (2 of 3)	2.10E+07
19BVL-573-006	CCRO Feed (Tank) (3 of 3)	2.10E+07
19BVL-573-007	PRO Permeate (1 of 3)	2.00E+00
19BVL-573-008	PRO Permeate (2 of 3)	5.00E+00
19BVL-573-009	PRO Permeate (3 of 3)	4.00E+00
19BVL-573-010	CCRO Permeate (Tank) (1 of 3)	1.00E+00
19BVL-573-011	CCRO Permeate (Tank) (2 of 3)	1.00E+00
19BVL-573-012	CCRO Permeate (Tank) (3 of 3)	1.00E+00
19BVL-573-013	CCRO Permeate (Early) (1 of 3)	4.00E+00
19BVL-573-014	CCRO Permeate (Early) (2 of 3)	1.00E+00
19BVL-573-015	CCRO Permeate (Early) (3 of 3)	1.00E+00
19BVL-573-016	CCRO Permeate (Middle) (1 of 3)	5.00E+00
19BVL-573-017	CCRO Permeate (Middle) (2 of 3)	1.00E+00
19BVL-573-018	CCRO Permeate (Middle) (3 of 3)	5.00E+00
19BVL-573-019	CCRO Permeate (Late) (1 of 3)	1.00E+00
19BVL-573-020	CCRO Permeate (Late) (2 of 3)	1.00E+00
19BVL-573-021	CCRO Permeate (Late) (3 of 3)	2.00E+00
19BVL-573-022	CCRO Feed-Brine (Early) (1 of 3)	4.40E+07
19BVL-573-023	CCRO Feed-Brine (Early) (2 of 3)	6.30E+07
19BVL-573-024	CCRO Feed-Brine (Early) (3 of 3)	4.00E+07
19BVL-573-025	CCRO Feed-Brine (Middle) (1 of 3)	7.50E+07
19BVL-573-026	CCRO Feed-Brine (Middle) (2 of 3)	6.90E+07
19BVL-573-027	CCRO Feed-Brine (Middle) (3 of 3)	7.80E+07
19BVL-573-028	CCRO Feed-Brine (Late) (1 of 3)	7.90E+07
19BVL-573-029	CCRO Feed-Brine (Late) (2 of 3)	6.50E+07
19BVL-573-030	CCRO Feed-Brine (Late) (3 of 3)	1.00E+06
19BVL-573-031	Field Blank PRO Permeate	<1
19BVL-573-032	Field Blank CCRO Permeate (Tank)	<1

Report Number BVL-3904
Date of Test: 12/18/19

Sample ID#	Sample Location	MS2 (pfu/mL)
19BVL-573-001	CCRO Feed (Tank) (1 of 4)	1.40E+07
19BVL-573-002	CCRO Feed (Tank) (2 of 4)	1.50E+07
19BVL-573-003	CCRO Feed (Tank) (3 of 4)	1.40E+07
19BVL-573-004	CCRO Feed (Tank) (4 of 4)	1.70E+07
19BVL-573-005	CCRO Permeate (Test 1) (1 of 3)	1.30E+07
19BVL-573-006	CCRO Permeate (Test 1) (2 of 3)	1.40E+07
19BVL-573-007	CCRO Permeate (Test 1) (3 of 3)	1.10E+07
19BVL-573-008	CCRO Permeate (Test 2) (1 of 3)	8.10E+06
19BVL-573-009	CCRO Permeate (Test 2) (2 of 3)	8.60E+06
19BVL-573-010	CCRO Permeate (Test 2) (3 of 3)	7.10E+06
19BVL-573-011	CCRO Permeate (Test 3) (1 of 3)	1.00E+07
19BVL-573-012	CCRO Permeate (Test 3) (2 of 3)	9.30E+06
19BVL-573-013	CCRO Permeate (Test 3) (3 of 3)	9.10E+06
19BVL-573-014	CCRO Permeate (Control) (1 of 3)	1.00E+00
19BVL-573-015	CCRO Permeate (Control) (2 of 3)	1.00E+00
19BVL-573-016	CCRO Permeate (Control) (3 of 3)	1.00E+00

Test 1: Interconnector O-ring compromise
Test 2: Brine endcap o-ring compromise
Test 3: Feed endcap o-ring compromise

Appendix B - Data Record for CCRO Testing at Padre Dam

RO O-ring Compromise Surrogate Data

EUROFINS FOLDER **845426**
Event Date: **12/18/19**

Parameter	Unit	CCRO Permeate (Tank)				CCRO Feed (Tank)			
		Control	Test 1	Test 2	Test 3	Control	Test 1	Test 2	Test 3
Eurofins ID	--	201912190405	201912190428	201912190429	201912190430	201912190404	201912190425	201912190426	201912190427
Grab Conductivity	µS/cm	113.6	2232	1395	2318	3785	3754	3779	3799
Grab pH	units	5.39	6.67	6.39	6.63	6.76	6.85	6.75	6.77
Total Chlorine	mg/L	0	0	0	0	0	0	0.2	0
Magnesium	mg/L	0.017 [A]	41	28	39	64	65	66	66
Sulfate	mg/L	0.54	350	250	360	590	590	580	620
Orthophosphate	mg/L as P	ND	0.088	0.063	0.089	0.14	0.15	0.15	0.16
Strontium	µg/L	0.34	0.71	0.5	0.62	1.1	1.1	1.2	1.2
TDS	mg/L	54	1400	1000	1400	2400	2400	2400	2400
TOC	mg/L	0.12	9.9	7.4	9.8	28	26	26	25

[A] *The analyte was either detected at or greater than the MDL and less than the MRL, or did not meet any one of the required QC criteria.*

Test 1: Interconnector O-ring compromise *Test 2: Brine endcap o-ring compromise* *Test 3: Feed endcap o-ring compromise*

Refer to Routine Surrogate Data Record for MRL (method reporting limit) of analytes

Cycle Assessment Surrogate Data

EUROFINS FOLDER **836217**
Event Date: **10/30/19**

Parameter	Unit	CCRO Vessel Permeate			CCRO Feed-Concentrate Recirculation		
		Early	Middle	Late	Early	Middle	Late
Eurofins ID	--	201910310596	201910310597	201910310598	201910310592	201910310594	201910310595
Time	--	11:39	12:07	12:35	11:39	12:07	12:35
Time in CCD	--	1:16	5:01	8:30	1:16	5:01	8:30
Vol-R	%	46	67.3	76.4	46	67.3	76.4
Grab Conductivity	µS/cm	63.03	125.8	177.3	6360	10610	14090
Grab pH	units	5.95	5.76	6.07	6.68	6.82	6.91
Total Chlorine	mg/L	0.1	0	0.1	0.2	0.2	0.2
Magnesium	mg/L	0.23	0.6	1	110	200	270
Sulfate	mg/L	2.6	6.9	12	1200	2200	3100
Orthophosphate	mg/L as P	ND	ND	ND	0.65	1.2	1.8
Strontium	µg/L	4.6	12	19	2300	4000	5500
TDS	mg/L	35	68	98	4400	7900	11000
TOC	mg/L	0.19 [A]	0.34	0.63	50	88	140

ND = non-detect (i.e., < MDL)

[A] *The analyte was either detected at or greater than the MDL and less than the MRL, or did not meet any one of the required QC criteria. Refer to Routine Surrogate Data Record for MRL (method reporting limit) of analytes*

Cycle Assessment Surrogate Data

EUROFINS FOLDER **844081**
Event Date: **12/11/19**

Parameter	Unit	CCRO Vessel Permeate			CCRO Feed-Concentrate Recirculation		
		Early	Middle	Late	Early	Middle	Late
Eurofins ID	--	201912120649	201912120647	201912120648	201912120646	201912120647	201912120648
Time	--	13:32	13:54	14:24	13:32	13:54	14:24
Time in CCD	--	3:00	7:00	9:30	3:00	7:00	9:30
Vol-R	%	53	70	77	53	70	77
Grab Conductivity	µS/cm	67.32	108	137.2	8849	13110	15650
Grab pH	units	5.67	5.64	5.78	7.13	7.25	7.31
Total Chlorine	mg/L	0	0	0	0.2	0.2	0
Magnesium	mg/L	0.012 [A]	0.027 [A]	0.036 [A]	190	300	360
Sulfate	mg/L	0.51	0.92	1.2	1600	2500	3200
Orthophosphate	mg/L as P	ND	ND	ND	0.43	0.64	0.78
Strontium	µg/L	0.24 [A]	0.48	0.64	3600	5400	6800
TDS	mg/L	39	64	80	6200	9700	12000
TOC	mg/L	0.092 [A]	0.12 [A]	0.13 [A]	86	100	120

ND = non-detect (i.e., < MDL)

[A] The analyte was either detected at or greater than the MDL and less than the MRL, or did not meet any one of the required QC criteria. Refer to Routine Surrogate Data Record for MRL (method reporting limit) of analytes

Cycle Assessment Surrogate Data

EUROFINS FOLDER **845427**
Event Date: **12/17/19**

Parameter	Unit	CCRO Vessel Permeate			CCRO Feed-Concentrate Recirculation		
		Early	Middle	Late	Early	Middle	Late
Eurofins ID	--	201910310596	201910310597	201910310598	201910310592	201910310594	201910310595
Time	--	12:28	13:00	13:16	12:28	13:00	13:16
Time in CCD	--	3:30	6:00	10:15	3:30	6:00	10:15
Vol-R	%	55	70	78	55	70	78
Grab Conductivity	µS/cm	68.58	108.3	128.4	8961	13290	15870
Grab pH	units	6.25	5.69	5.94	7.18	7.29	7.36
Total Chlorine	mg/L	0	0.1	0.1	0.1	0.2	0.4
Magnesium	mg/L	0.0073 [A]	0.015 [A]	0.025 [A]	170	270	330
Sulfate	mg/L	0.38	0.68	0.82	1500	2400	2900
Orthophosphate	mg/L as P	ND	0.013	ND	0.42	0.67	0.79
Strontium	µg/L	0.18 [A]	0.39	0.46	4000	4700	5800
TDS	mg/L	39	66	82	6500	10000	12000
TOC	mg/L	0.27	0.12	0.11	68	110	140

ND = non-detect (i.e., < MDL)

[A] The analyte was either detected at or greater than the MDL and less than the MRL, or did not meet any one of the required QC criteria. Refer to Routine Surrogate Data Record for MRL (method reporting limit) of analytes