

ICR Treatment Study Summary Report

Evaluation of Membrane Technology Using the Single Element Bench-Scale Test for Compliance with the Information Collection Rule

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Attachments: One zip disk containing the Data Collection and Treatment Study
Summary Report Spreadsheets, and Treatment Study Summary Report



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SECTION 1

CONCLUSIONS AND RECOMMENDATIONS

Recent developments have lead to the development of new, low-pressure nanofiltration (NF) membranes that are capable of removing significant amounts of total organic carbon (TOC) and low molecular weight synthetic organic compounds (SOCs). Such membranes show promise for making NF technologies more affordable. However, in many instances these membranes have shown a high fouling potential and require extensive particle control. To implement NF as a polishing treatment for lime softening to remove disinfection by-product (DBP) precursors and SOCs, greater pretreatment should be investigated prior to NF to control particle fouling. The Interim Enhanced Surface Water Treatment Rule would allow lime softening plants to acidify turbidity samples to account for the turbidity resulting from lime carryover though filtration (*Federal Register*). While such a measure is replicated in pretreating NF feed water (acidifying to control inorganic salt precipitation), the issue of lime carryover may be indicative of a larger problem associated with particle transport through filtration media and should be looked at again, especially with respect to particle fouling and NF.

NF has demonstrated superior water quality with respect to DBP and SOC removal. While blending of NF permeate water with conventionally treated plant water may produce a blended water quality that meets regulatory standards, any disinfection gained through NF will be negated. Blending should be conducted in light of all water quality objectives to produce a finished water that best meets each utilities individual needs.

To implement NF as a polishing treatment in the existing lime softening process at the Kansas City Missouri Water Services Department (KCMWSD), with a NF permeate capacity of 216 mgd, the cost of water would likely increase by approximately 78% over the current cost of water. This would raise the price of water from \$1.58 to \$2.81 per 1,000 gallons. Such an increase in price would have to be weighed with the benefits received from implementing an advanced treatment process like NF.

Should a utility look at implementing a new facility, an integrated membrane system may achieve multiple treatment objectives including softening, disinfection of chlorine resistant pathogens, and DBP control. Pilot testing from this study revealed that the cost of NF may be reduced by approximately \$0.05 to \$0.10 per 1,000 gallons. Implementation of an IMS system may also be comparable in price to conventional processes. The Zenon MF technology that was pilot tested required a high operating cost for electrical power consumed through the use of an air scour to control particle accumulation on the membrane surface. It would be recommended, for a utility considering a new facility, to pilot test a variety of MF and ultrafiltration (UF) technologies that do not require the use of an air blower. Such research may reduce the cost of implementing an integrated membrane system. Concentrate bleed must also be disposed of and evaluation of concentrate disposal may impact the cost of implementing a membrane process such as NF. Further work should also be conducted to evaluate re-use or disposal options for concentrate waste.

SECTION 2

BACKGROUND INFORMATION

As a Public Water System (PWS) serving more than 500,000 people with an average annual TOC exceeding 4 mg/L, the Kansas City Missouri Water Services Department (KCMWSD) elected to conduct membrane pilot tests to meet their Information Collection Rule (ICR) treatment study requirement. KCMWSD utilizes a softening treatment typical of most PWSs along the Missouri River. Water District No. 1 of Johnson County (WDNo1JC), Kansas is a neighboring PWS (within 20 river miles) that employs a similar treatment to that of KCMWSD. To prescreen candidate membranes for pilot testing, the KCMWSD agreed to host Johnson County's ICR treatment study as part of a collaborative research effort funded by the Missouri River Public Water Supplies Association (MRPWSA), the Electrical Power Research Institute (EPRI), and the American Water Works Association Research Foundation (AWWARF).

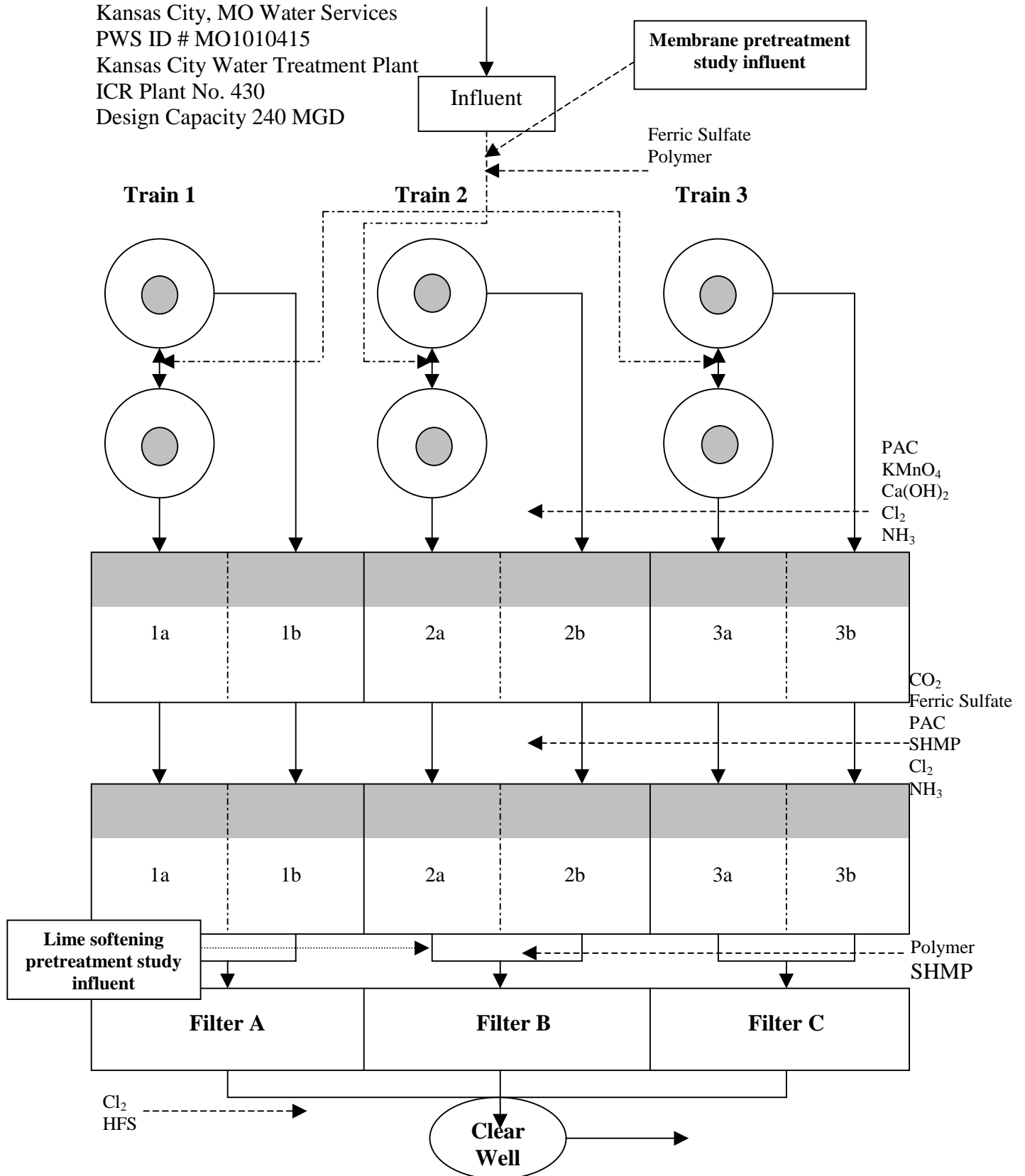
Due to the collaborative nature of this project, there were many research goals that required this treatment study to deviate from the standard ICR protocol. For pilot scale testing, the ICR requires that a single membrane be evaluated over four quarters to account for the effects of seasonal variation. The broad scope of this project satisfies the intent of this requirement while deviating from these constraints only slightly. To satisfy the intent of evaluating the effects of seasonal variation, one quarter of single element testing (from WDNo1JC's ICR treatment study) was grandfathered into the KCMWSD study so an alternative pretreatment, microfiltration (MF), could be tested ahead of NF. Only one quarter of ICR pilot scale membrane testing was performed using MF as a pretreatment. This testing scheme was to the liking of project participants and was approved by the EPA. Testing in this manner provides more design criteria critical for implementing NF in mid-western water plants treating high turbidity, high hardness surface water supplies. Only the fourth quarter of pilot testing was performed with new NF membranes using a MF pretreatment to provide design criteria for water plants considering new facilities.

BACKGROUND INFORMATION PERTAINING TO LIME SOFTENING PRETREATMENT

For the first three quarters of this study, lime softening followed by granular media filtration served as pretreatment for NF. A schematic diagram representing the lime softening treatment processes employed at the KCMWSD is presented in Figure 1. There are six separate treatment trains utilized during the treatment of Missouri River water at the KCMWSD. Water utilized during the ICR treatment study came from Train #2b. No chloramines were added to this train prior to granular filtration. The point at which treatment study influent water was drawn is indicated in Figure 1. Design Plant Parameters and Design Plant Chemical Parameters as reported in the *ICR Water Utility Database System* for each unit process in Figure 1 are listed in Appendix A.

Figure 1. Kansas City, Missouri Water Services Dept. plant schematic

Kansas City, MO Water Services
PWS ID # MO1010415
Kansas City Water Treatment Plant
ICR Plant No. 430
Design Capacity 240 MGD



As a source water, the Missouri River may be characterized as a high hardness source with periods of high turbidity and high total organic carbon (TOC) concentrations. As a result of the seasonal spikes in turbidity and TOC, the KCMWSD has corresponding difficulties in meeting proposed delivered water turbidity and total trihalomethane (TTHM) concentrations (0.3 for turbidity and 80/40 µg/L for Stage I/II TTHMs respectively). A summary of source and delivered water quality for the KCMWSD is presented in Table 1. Averages and standard deviations are calculated from daily values over 15 months of daily monitoring data (1998 average values monitored under the ICR are reported in the *Treatment Study Summary Report Spreadsheet*).

Table 1a. Source water quality

Water Quality Parameter	Average Yearly Concentration	Standard Deviation	Maximum Yearly Value	Minimum Yearly Value
Temperature (°C)	13	9.0	30	-2.1
pH	7.9	0.4	8.8	6.5
Turbidity (ntu)	170	370	3950	7
Alkalinity (mg/L as CaCO ₃)	172	22	436	96
Calcium Hardness (mg/L as CaCO ₃)	154	25	255	98
Total Hardness (mg/L as CaCO ₃)	247	39	382	165
TOC (mg/L)	4.67	1.2	11.73	2.06
UV ₂₅₄ (cm ⁻¹)	0.117	0.026	0.200	0.019
Bromide (µg/L)	< 0.1	0.0	0.2	< 0.01

Values taken from 15 months of daily monitoring data (January 1997 – June 1998)

Table 1b. Finished water quality

Water Quality Parameter	Average Yearly Concentration	Standard Deviation	Maximum Yearly Value	Minimum Yearly Value
Temperature (°C)	13.8	8.4	30	0.6
pH	9.7	0.3	10.8	8.7
Turbidity (ntu)	0.17	0.15	1.23	0.01
Alkalinity (mg/L as CaCO ₃)	50	9	100	20
Calcium Hardness (mg/L as CaCO ₃)	119	22	193	73
Total Hardness (mg/L as CaCO ₃)	148	18	223	103
TOC (mg/L)	2.34	0.37	4.60	1.19
UV ₂₅₄ (cm ⁻¹)	0.060	0.009	0.106	0.006
Bromide (µg/L)	< 0.1	0.0	0.1	< 0.1
Distribution System THM4 (µg/L)	13.8	15.8	97.1	2.5

Values taken from 15 months of daily monitoring data (January 1997 – June 1998)

BACKGROUND INFORMATION PERTAINING TO MEMBRANE PRETREATMENT

Integrated membrane treatment of surface water is a relatively new concept that has demonstrated superior particle removal, which is very important for successful NF operation. Information obtained during this portion of pilot testing is intended to develop design criteria critical for implementing new water treatment plants along the Missouri River. No full scale processes were used during this portion of the pilot study. Influent water was obtained prior to primary clarification as noted in Figure 1. Primary clarification was not employed due to the use of organic polymers, if carried over may irreversibly foul a polymeric membrane.

Integrated membrane applications show great promise for treating high turbidity, high hardness surface waters. The current regulatory trend is driving utilities to look at alternative processes that meet multiple treatment objectives. For mid-western water utilities, these objectives include softening, SOC removal, and reducing the risk of chlorine resistant pathogens while addressing DBP formation. In some cases, integrated membrane systems as a whole have demonstrated greater than a 10 log disinfection while providing softening and SOC removal though NF.

SECTION 3

MATERIALS AND METHODS

The objective of this study was to evaluate one nanofiltration (NF) membrane on the pilot scale using a softening or membrane pretreatment for three and one quarter(s) respectively. The membrane used for this pilot study was screening through bench and single element tests to determine if it would meet project goals. Goals for NF pilot evaluation included removing disinfection by-product (DBP) precursors (as required by the ICR), developing hydraulic criteria critical to design, and atrazine removal.

LIME SOFTENING PRETREATMENT OF INFLUENT WATER

The purpose of this portion of KCMWSD's ICR treatment study was to evaluate the effectiveness of NF as a polishing treatment in the existing water treatment facility for removing DBP precursors and atrazine. To determine the effectiveness of NF as a polishing treatment for an existing water treatment plant (WTP) it is important that water treated by NF membranes be subject to the same or a similar pretreatment as that seen in the water utility required to conduct the test.

The KCMWSD is a PWS who treats Missouri River water using a lime softening process. A schematic diagram of all full scale and bench scale processes, including chemical addition points prior to NF is presented in Figure 2. It is important to note that ICR requires no disinfection occur prior to NF to evaluate the ability of NF to remove DBPs. As a result, no chloramines were added to water from treatment train #2b (see Figure 1). Water from treatment trains #2a and #2b is mixed during final (granular) filtration. Since water from treatment train #2a has been exposed to chloramines, ICR treatment study influent had to be drawn prior to granular filtration. Filtration was replicated on the pilot scale.

Pretreatment design data for all processes prior to NF are listed in Table 2. Cartridge filtration after granular filtration was deemed necessary due to the fact that spiral wound NF membranes are sensitive to particulate fouling. Acid and scale inhibitors were also injected prior to NF to prevent the precipitation of inorganic salts on a membrane's surface.

Figure 2. Lime Softening Pretreatment to Nanofiltration

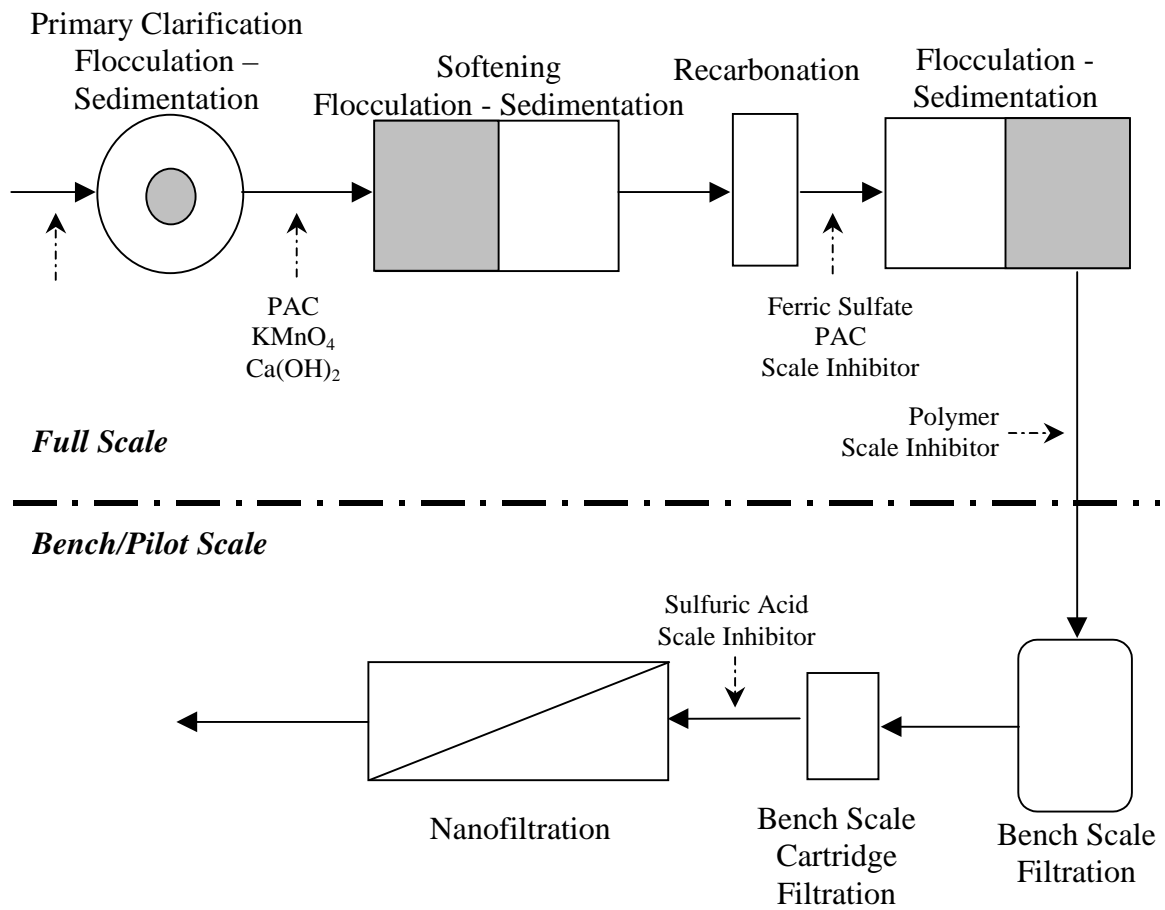


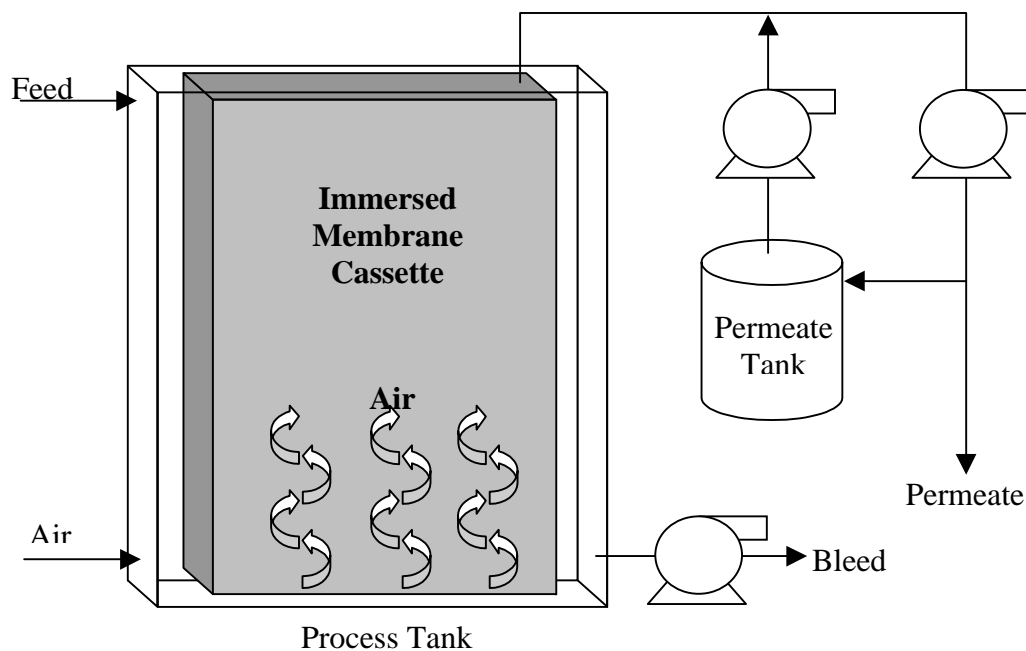
Table 2. Lime softening pretreatment design data (Train #2)

Unit Process	Process Description
Flocculation Basin (Full Scale)	Type of Mixer: Mechanical Liquid Volume (gal): 752,000 Short Circuiting Factor: 0.3 Baffling Type: Average Stage Sequence Number: 1 Stage Mean Velocity Gradient (sec^{-1}): 28 Stage Liquid Volume (gal): 752,000
Sedimentation (Full Scale)	Surface Area (ft^2): 52,780 Liquid Volume (gal): 9,882,000 Baffling Type: UN Short Circuiting Factor: 0.3
Flocculation Basin (Full Scale)	Type of Mixer: Mechanical Liquid Volume (gal): 1,944,000 Short Circuiting Factor: 0.4 Baffling Type: Average Stage Sequence Number: 1 Stage Mean Velocity Gradient (sec^{-1}): 71 Stage Liquid Volume (gal): 1,944,000
Sedimentation (Full Scale)	Surface Area (ft^2): 80,600 Liquid Volume (gal): 9,672,000 Baffling Type: UN Short Circuiting Factor: 0.4
Flocculation Basin (Full Scale)	Type of Mixer: Mechanical Liquid Volume (gal): 3,152,000 Short Circuiting Factor: 0.4 Baffling Type: Average Stage Sequence Number: 1 Stage Mean Velocity Gradient (sec^{-1}): 105 Stage Liquid Volume (gal): 3,152,000
Sedimentation (Full Scale)	Surface Area (ft^2): 30,600 Liquid Volume (gal): 3,777,400 Baffling Type: UN Short Circuiting Factor: 0.4
Filtration (Bench Scale)	Surface Area (ft^2): 1.4 Liquid Volume (gal): 4.6 Total Media Depth (in): 18 Media Type: Sand
Cartridge Filtration (Bench Scale)	Surface Area (ft^2): 0.1 Nominal Pore Size (μm): 5.0 Filter Material: Polypropylene Filter Life (gallons of water processed): 20,000
Nanofiltration (Pilot Scale)	

MEMBRANE PRETREATMENT OF INFLUENT WATER

Due to the time, location, and convenience of testing, the Zenon ZeeWeed[®] membranes were selected as the MF pretreatment process for this portion of KCMWSD's ICR treatment study. The ZeeWeed[®] membrane system uses a MF hollow fiber membrane. The membrane has a nominal pore size of 0.01 μm and is credited with a 2.5 log removal of viruses in the state of California. The membrane's polymeric composition is proprietary and not released by the manufacturer. The manufacturer does, however, indicate that the membrane material is resistant to oxidants such as chlorine and can operate at pHs ranging from 5 to 9. The system involves outside-in filtration through fibers that are immersed in a basin filled with feed water. The system operates under a vacuum induced by a centrifugal permeate pump. Airflow is introduced in the concentrate compartment at the bottom of the membrane cassette to scrub the outside of the fibers, reducing solids accumulation on the membrane skin. A flow schematic is shown in Figure 3.

Figure 3. Zenon process flow diagram



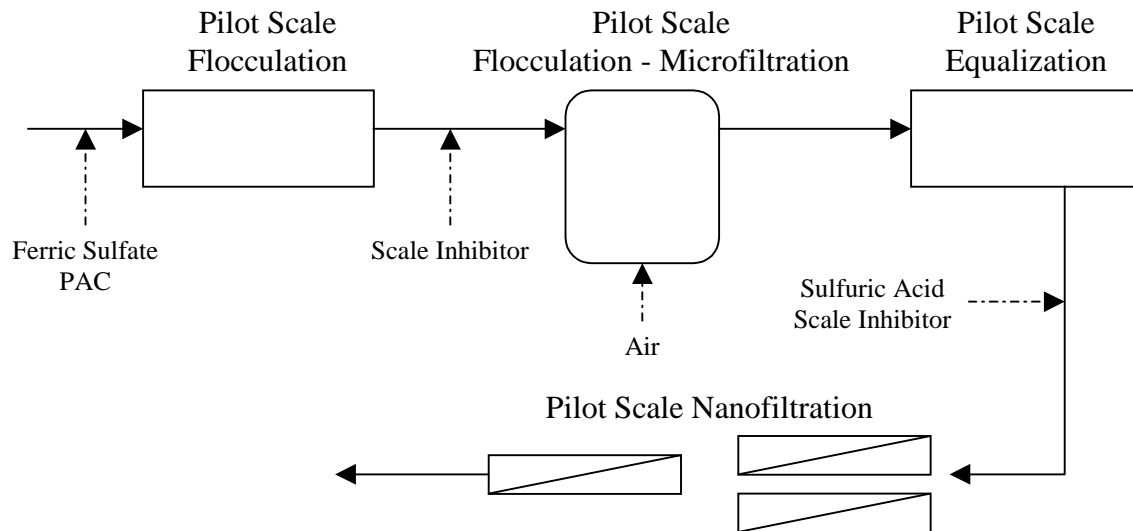
Regarding the raw water data presented in Table 1a, direct MF of Missouri River water would likely prove challenging. To manage the high turbidities and TOC concentrations typical of the Missouri River supply an inorganic coagulant, ferric sulfate, was added in-line with the MF membrane process. Jar tests were performed to select an optimum dose (18 mg/L as $\text{Fe}_2(\text{SO}_4)_3$) for colloidal destabilization and TOC removal. Pretreatment design data are presented in Table 3 for each of the unit processes preceding NF. A schematic of the pilot scale processes and points of chemical addition is presented in Figure 4. Flocculation basins for the in-line coagulation pretreatment are indicated in Figure 4. An additional flocculation basin, indicated in Table 3 represents the basin in

which the Zenon membranes are submerged. While air is primarily intended to scour solids from the membrane surface, it also provides mixing. The MF process, used to pretreat NF feed water, requires backwashing while the NF membrane system uses a constant feed of MF treated water. To supply the NF system with a constant supply of MF treated water, an equalization basin was provided between the MF and NF processes which is also presented in Figure 4.

Table 3. Membrane pretreatment design data

Unit Process	Process Description
Flocculation Basin (Pilot Scale)	Type of Mixer: Mechanical Liquid Volume (gal): 250 Type of Mixer: Pneumatic Liquid Volume (gal): 185
Microfiltration (Pilot Scale)	Surface Area (ft ²): 500 Filter Media: Proprietary Nominal Pore Size (μm): 0.01 Filtration Mode: Outside-in
Equalization Basin (Pilot Scale)	Liquid Volume (gal): 55
Nanofiltration (Pilot Scale)	

Figure 4. Membrane pretreatment to nanofiltration



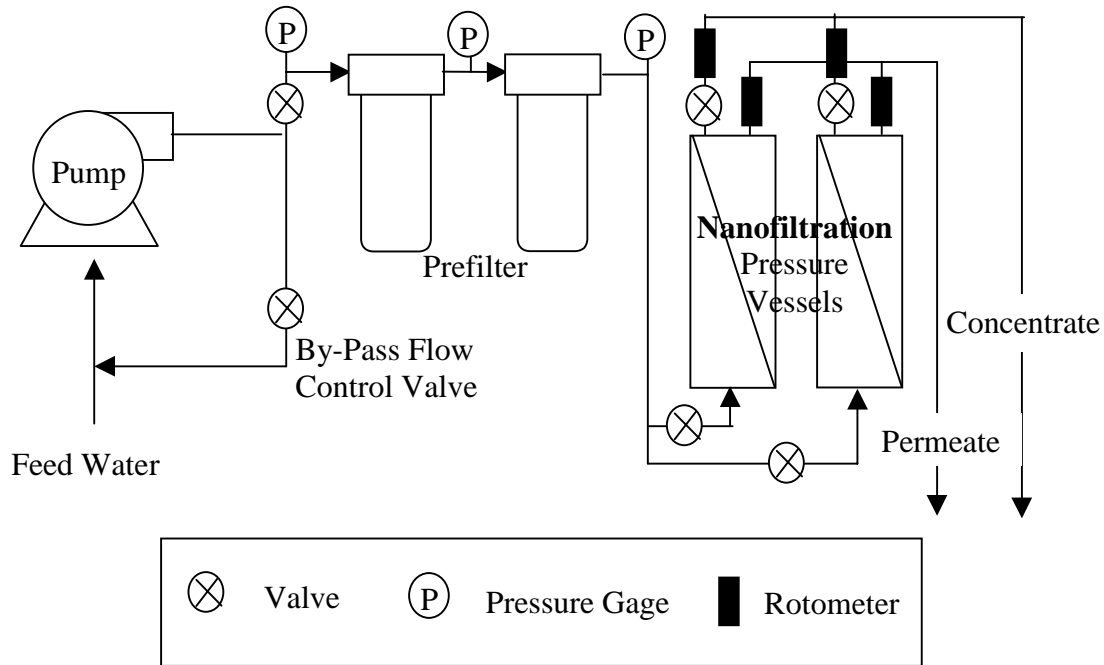
PRELIMINARY SELECTION OF CANDIDATE MEMBRANES

Preliminary selection of NF membranes consisted of a literature review followed by rapid bench scale membrane screening tests (RBSMTs) and then further screening with the Single Element Bench Scale Membrane Test (SEBST). Upon completion of the literature review, nine candidate membranes were identified from four different manufacturers. Table 4 presents a summary of these membranes along with their physical and chemical properties. Membrane manufacturers were then contacted and asked to provide a sample membrane, suitable for RBSMT screening or data that would prove candidacy based on study objectives (i.e. atrazine removal). Eight membrane samples were obtained for RBSMT evaluation, which consisted of monitoring TOC, UV₂₅₄, atrazine, and total dissolved solids (TDS) removal at a normalized flux. Membrane samples used for RBSMT evaluations were 2" x 12" spiral wound modules that fit into pressure vessels rated for 125-psi maximum pressure. Figure 5 presents a schematic of the bench scale apparatus used for RBSMT evaluations.

Table 4. Physical and chemical properties of commercially available NF membranes

Membrane	Average Operating Pressure (psi)	MWCO (Daltons)	Active Layer Composition	Application
<i>Sepa-Osmonics</i>				
ST	400	160-210	Cellulose acetate	Softening
MS	200	100-150	Polyamide	Softening
<i>Purification Products</i>				
B-22-1	100	~ 200	Polyamide	Softening
B-22-9	100	~ 200	Polyamide	Softening
<i>Hydranautics</i>				
NTR-7450	143	10000	Sulfonated polyether sulfone	Organics
NTR-7410	143	6000	Sulfonated polyether sulfone	Organics
PVD1	143	100-300	Polyvinyl alcohol derivative	Softening
ESNA	75	~ 200	Polyamide	Organics-Softening
<i>Dow FilmTec</i>				
NF200B	60	~ 300	Polypiperazine	Micro-pollutants

Figure 5. Rapid bench-scale membrane screening test apparatus



For ICR purposes, the Hydranautics NTR-7450 and NTR-7410 membranes were eliminated from further consideration due to the fact that their molecular weight cut-offs (MWCOs) were greater than 1000 Daltons. However, these membranes were included in the preliminary screening to determine the fractionation of source water NOM and to determine if the membranes' negative surface charge alone was capable of repelling 215.68 Dalton atrazine molecules. In lieu of providing membrane samples, Dow FilmTec provided data which proved candidacy for their membrane. Remaining candidate membranes were either selected or eliminated based on their rejection of TOC and atrazine. To be considered for single element ICR testing, membranes were required to have a minimum of 80% and 60% atrazine and TOC rejection respectively with mass balance errors (Error_{MB}) falling within a range explainable by experimental error. Mass balance error was calculated as follows:

$$C_{\text{C(calc)}} = (C_{\text{F}} - R \times C_{\text{P}}) / (1 - R) \quad \text{Error}_{\text{MB}} = ((C_{\text{C}} - C_{\text{C(calc)}}) / C_{\text{C}}) \times 100\%$$

Where: $C_{\text{C(calc)}}$ is a calculated concentration of a solute based on feed, C_{F} , and permeate, C_{P} , concentrations at the operating recovery, R .

SINGLE ELEMENT BENCH SCALE SCREENING

To meet the ICR treatment study requirements for WDNo1JC and to prescreen candidate membranes for KCMWSD's pilot-scale ICR study, two single element membrane pilot plants were installed on the premises of the KCMWSD's water treatment facility. Each

pilot plant, as illustrated in Figure 6, was capable of housing one 4" x 40" spiral wound membrane element.

With the consent of the EPA, testing four membranes over two quarters was accepted as an alternative to testing 2 membranes over four quarters as required by the ICR due to the collaboration between WDNo1JC and KCMWSD. The best performing membrane, in terms of water quality and hydraulic performance, from two quarters of SEBST evaluation was chosen for a year long pilot study in fulfillment of KCMWSD's ICR treatment study requirements.

In accordance with the intent of the ICR, each of the four selected membranes were operated for two, thirty-day quarters (60 days per membrane, 240 days total). Daily monitoring of hydraulic parameters, pH and TDS were preformed as specified in the *ICR Manual for Bench and Pilot-Scale Treatment Studies*.¹ An overall system recovery of $75\% \pm 5\%$ was also employed. Fluxes were normalized daily to an average temperature representative of the historical average temperature for the testing period. Normalization was achieve by varying the permeate flow based on the following equations:

$$Q_T = Q_{Tave} \times 1.03^{(T-Tave)}$$
$$F_{Tave} = Q_{Tave} / A$$

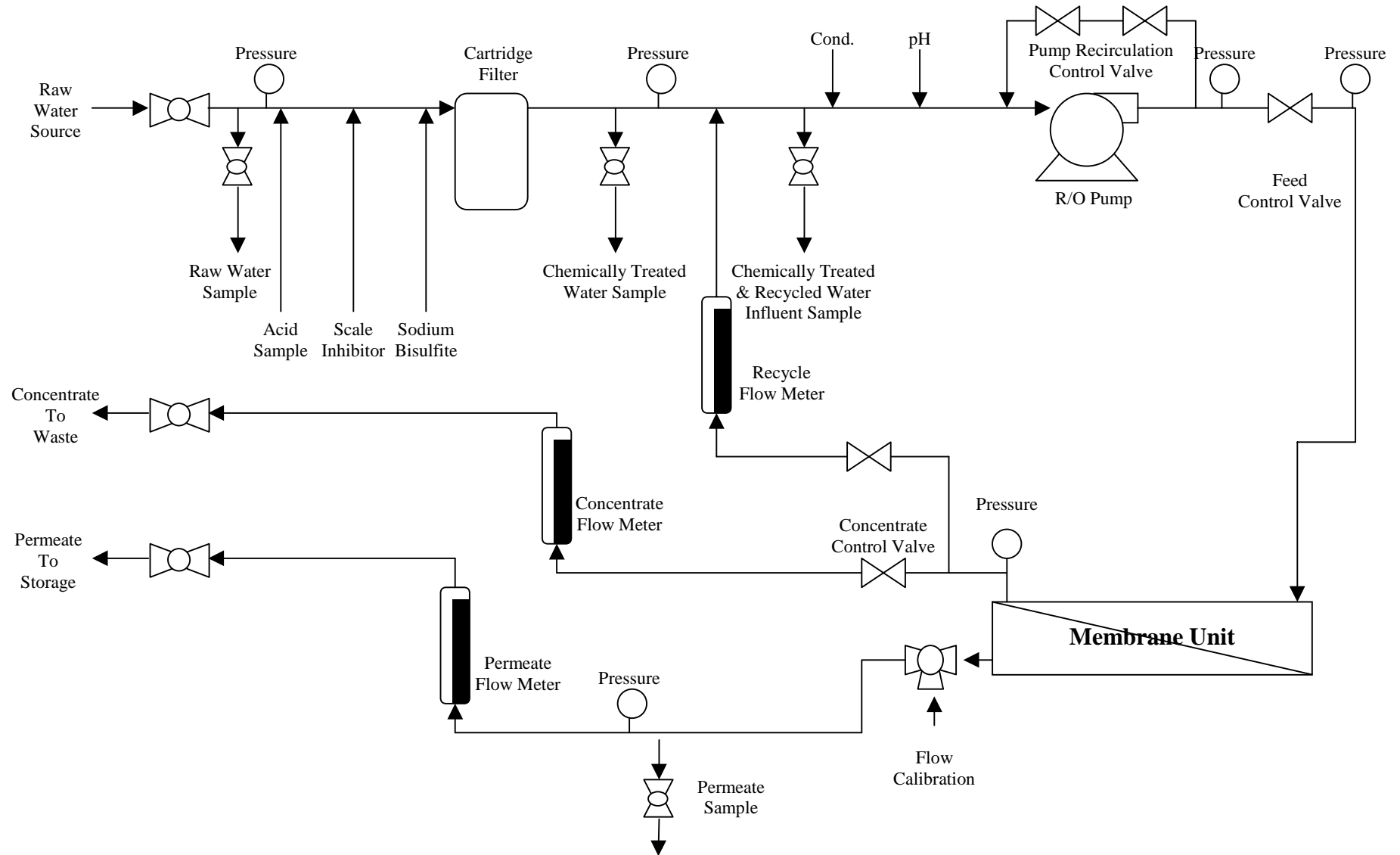
Where: Q_T = permeate flow at ambient temperature T = ambient temperature
 Q_{Tave} = permeate flow at average temperature A = membrane surface area
 T_{ave} = average quarterly temperature F_{Tave} = Flux at average temperature

To design system operating conditions, each selected manufacturer was contacted and asked to provide basic information about their membrane elements. Appendix B lists the physical and chemical properties as reported by the manufacturer for their corresponding membrane elements.

Typically, operating conditions such as flux, recovery, cross flow velocity and pH limit the overall system performance. Membrane manufacturers generally provide a recommendation for standard operating conditions and some computer models, such as *RO DESIGN* by Hydranautics, have been developed to aide in system design. Due to the brevity of this study, most system variables where held constant during both quarters. Fluxes were normalized to 12 gfd at 7°C and 13°C for single element screening quarters one and two respectively.

As indicated in Figure 2, NF feed water, pretreated by lime softening was subject to bench scale sand and cartridge filtration. In order to control mineral scaling, pH was adjusted to a target value of 6.0 with sulfuric acid. In addition, 4 mg/L of sodium hexametaphosphate (SHMP) or Hypersperse SI 300 (Argo Scientific; San Marcos, California) was added as a scale inhibitor.

Figure 6. Single element bench scale membrane test schematic



Samples were drawn from the SEBST system as prescribed by the ICR. Table 5 provides the sampling matrix for ICR SEBST evaluations. It should be noted that permeate and feed water TDS concentrations were measured online. The TDS feed probe was located improperly by the pilot plant manufacturer for ICR evaluations. As noted in Figure 6, the sample probe has been located within the recirculation loop instead of in the feed stream prior to acid and scale inhibitor addition. Therefore, the TDS concentrations measured were bulk concentrations and not feed concentrations.

Table 5. SEBST sampling matrix

Parameter	Frequency	Location	# of Samples
<i>Hydraulic</i>			
Flow	Daily	P, C, R	124
Pressure	Daily	P, C, I	124
Temperature	Daily	I	124
TDS	Daily	F, P, C	124
pH	Daily	F, P, C	124
<i>Water Quality</i>			
pH	Weekly	F, P, C	20
Total Hardness	Weekly	F, P, C	20
Calcium Hardness	Weekly	F, P, C	20
Alkalinity	Weekly	F, P, C	20
TDS	Weekly	F, P, C	20
Turbidity	Weekly	F, P, C	20
TOC	Weekly	F, P, C	25
UV254	Weekly	F, P, C	25
Bromide	Weekly	F, P	25
SDS-THM4	Weekly	F, P	25
SDS-HAA9	Weekly	F, P	25
SDS-TOX	Weekly	F, P	25
SDS-Chlorine Demand	Weekly	F, P	25
Atrazine	Weekly		

F: Feed
P: Permeate
C: Concentrate
I: Inlet
R: Recycle

PILOT TESTING PROTOCOL

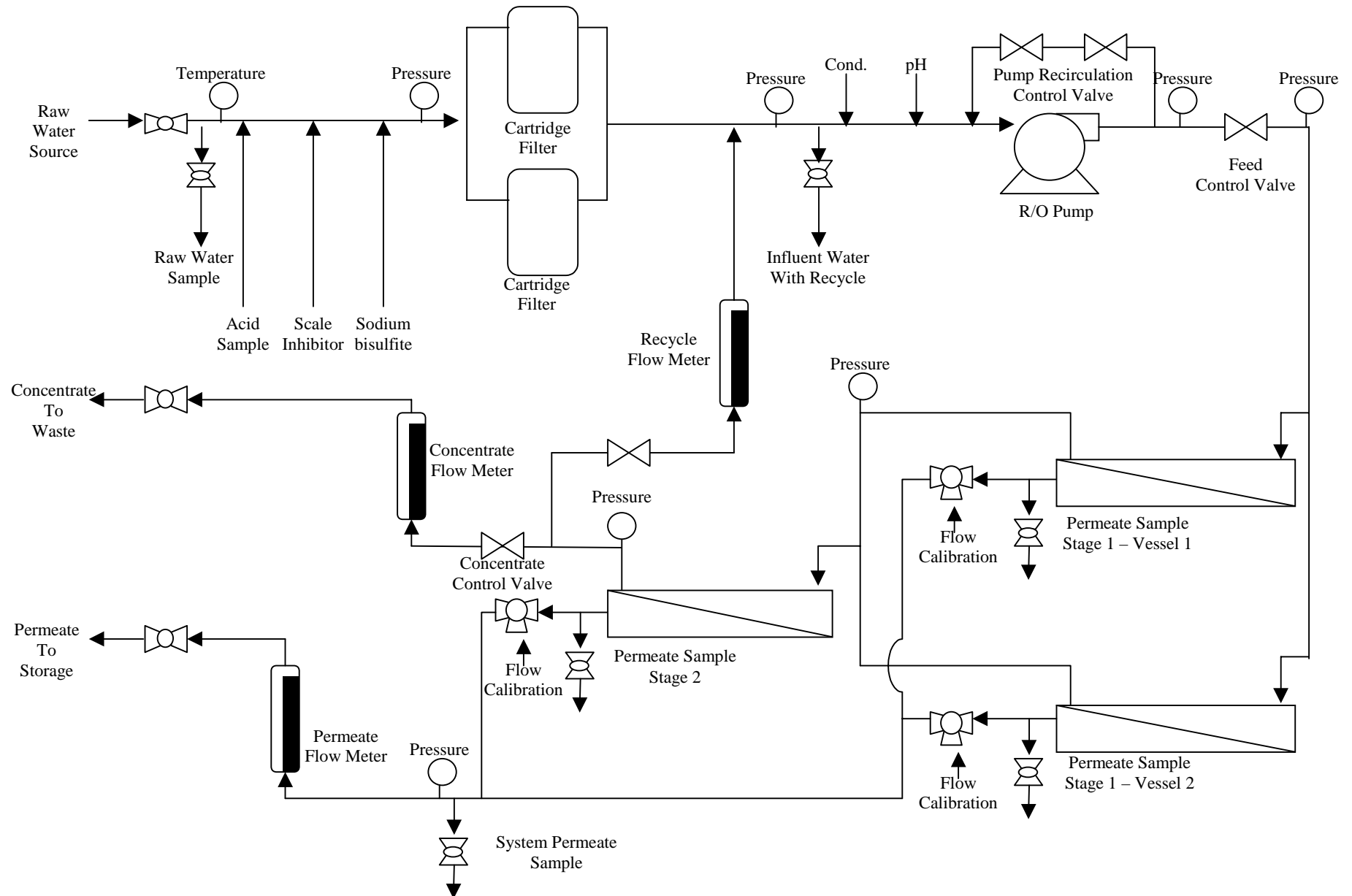
Upon completion of SEBST evaluations, membranes were evaluated based on water quality and hydraulic performance. One membrane, meeting all water quality and

hydraulic goals, was then retained for pilot testing in fulfillment of KCMWSD's ICR treatment study.

The ICR requires that NF be evaluated at the pilot scale for a total of 6,600 hours over a period of four, 70-day quarters. Pilot testing was performed at a recovery of 93% and the flux for each quarter of evaluation was set to 15 gfd, normalized to a temperature indicative of the average quarterly temperature. Flux normalization was achieved in a similar manner to that described previously in the SEBST procedure. To maintain hydrodynamic conditions specified by the manufacturer, it was necessary to implement a recirculation flow of stage two concentrate water back to the head of stage one. This practice will likely compromise staged water quality measurements, but was necessary to prevent fouling of stage 2 membrane elements.

Figure 7 presents a flow diagram of the two stage, twelve element pilot plant used for this ICR treatment study. As indicated in Figure 7, in order to control mineral scaling, pH was adjusted to a target value of 6.0 with sulfuric acid. In addition, 4 mg/L of sodium hexametaphosphate (SHMP) or Hypersperse SI 300 (Argo Scientific; San Marcos, California) was added as a scale inhibitor.

Figure 7. Two stage, twelve element nanofiltration pilot plan



Samples were drawn from the pilot system as prescribed by the ICR. Table 6 provides the sampling matrix for ICR pilot scale membrane evaluations. All water quality parameters were measured as grab samples. As indicated in Figure 7, there was no feed water sample port after acid addition and prior to the addition of recirculated concentrate water prior to stage one. Daily feed water (after acid addition) grab samples were acquired by turning off the recirculation loop, after all other water quality samples had been monitored, and drawing water from the sample port labeled “Influent Water With Recycle”.

MEMBRANE CLEANING

Membranes were considered to require a chemical cleaning when a 15% increase in pressure or productivity (MTC_w) decline was observed or when permeate water quality (TDS rejection) decayed by more than 15%. Typical foulants that hinder membrane production, making cleaning necessary include sparingly soluble materials such as: calcium carbonate scale, calcium sulfate scale, metal oxides, silica and organic or biological deposits.

Cleaning procedures recommended by most manufacturers require either an acid or alkaline cleaning solution based on the nature of fouling and the observed symptoms of said fouling. Appendix C lists a recommended cleaning procedure as provided by Hydranautics for various types of fouling conditions categorized by symptoms and cleaning action(s) required. It is important to note that all the membranes used during this study were considered sensitive to chlorine. Under no circumstances were any of the membranes exposed to chlorine during cleaning conducted as part of this treatment study.

ATRAZINE CHALLENGES

Technical grade atrazine was acquired from Novartis Crop Protection (Greensboro, North Carolina). NF membranes were challenged with atrazine on a quarterly basis. A stock solution of atrazine was prepared and injected into the feed water via a metering pump to produce a feed water concentration of approximately 6-10 $\mu\text{g/L}$.

ANALYTICAL METHODS

Analytical methods employed during this ICR study, listed in Table 7, were performed by the Kansas City Missouri Water Services Laboratory and Carollo Engineers. Analytical protocols required by the *DBP/ICR Analytical Methods Manual* were followed. The Kansas City Missouri Water Services Laboratory is a certified laboratory for ICR analysis.

Minimum reporting levels (MRLs) are also reported in Table 7. The MRL for total organic carbon (TOC) was determined on a weekly basis by the Kansas City Missouri Water Services Laboratory. In the case of TOC, the MRL was equal to twice the

Table 6. Two stage NF pilot sampling and monitoring requirements

Parameter	Raw	Acidified feed	Chemically treated & filtered	Influent w/ recycle*	Stage I & II permeate	Stage II influent	System permeate	System concentrate	# of samples	Std. Meth
Hydraulic										
Flow					D		D	D	672	
Pressure		D	D	D	D	D	D	D	1560	
Temperature				D					224	
Direct Flow meas.							D	D	224	
TDS	D		D	D	D	D	D	D	1560	2510
pH	D		D	D	B	B	B	D	1344	4500-H ⁺
Water Quality										
Total Hardness			B	B	B	B	B	B	784	2340
Calcium Hardness			B	B	B	B	B	B	784	3111, 3120
Alkalinity			B	B	B	B	B	B	784	2320
Turbidity			B	B	B	B	B	B	784	2130
TOC			B	B	B	B	B	B	800	5310
UV ₂₅₄			B	B	B	B	B	B	800	5910
Bromide			B				B		800	4110, 4500-Br ⁻
SDSTHM4			B				B		250	5710 & 6232
SDSHAA6			B				B		250	5710 & 6251
SDSTOX			B				B		250	5710 & 5320
SDS Cl ₂ Demand			B				B		250	2350 B.
Atrazine										EPA Method 505

B: Bi-weekly

D: Daily

* The water quality parameters need only be monitored if concentrate recycle is used.

minimum detection level. A low level calibration check standard equal to the determined MRL was prepared and analyzed immediately following to instrument calibration. A relative percent difference (RPD) was calculated after two injections of the low-level calibration check standard. TOC analysis would only proceed once an $RPD \leq 10\%$ was achieved.

Table 7. Analytical methods and minimum reporting levels

Analyte	Method	Minimum Reporting Level
Alkalinity	2320 B	5 mg/L as CaCO_3
Ammonia	EPA 350.1	0.03 mg/L
Bromide	EPA 300.0	10 $\mu\text{g/L}$
Calcium Hardness	3120 B	0.16 mg/L as CaCO_3
Chlorine Residual	4500-Cl G	0.5 mg Cl as Cl_2/L
DCAA, TCAA, MBAA, DBAA, BCAA, BDCAA*, CDBAA*, TBAA*	6251 B	1 $\mu\text{g/L}$
MCAA	6251 B	2 $\mu\text{g/L}$
pH^{JCE}	4500- H^+ B	Not Applicable
TDS^{JCE}	2510 B	1 mg/L
Temperature ^{JCE}	2550 B	Not Applicable
THM4	EPA 502.2	1 $\mu\text{g/L}$
Total Hardness	5710 C	0.65 mg/L as CaCO_3
TOC	5310 B	0.2 mg/L
TOX	5320 B	25 $\mu\text{g Cl}^-/\text{L}$
Turbidity ^{JCE}	2130 B	0.05 ntu
UV_{254}	5910	0.009 cm^{-1}
Atrazine*	EPA 505	0.05 $\mu\text{g/L}$

^{JCE} Analyses performed by John Carollo Engineers staff

* Not required by the ICR

SDS testing, DBP and water quality analyses was performed in accordance with ICR procedures and only reagent grade chemicals were used. To ensure that residual concentrations of free chlorine were met after 48 hours of incubation, multiple samples were chlorinated at varying TOC: Cl_2 ratios. A summary of SDS conditions employed is listed in Table 8.

Table 8. SDS incubation conditions

Incubation Conditions	SEBST Quarter #1	SEBST Quarter #2
Time (hours)	48	48
pH	7.0	7.0
Temperature (°C)	7	13
Free Cl ₂ Residual (mg/L)	0.5 – 1.0	0.5 – 1.0

Optional haloacetic acids (HAAs), bromodichloroacetic acid (BDCAA), chlorodibromoacetic acid (CDBAA) and tribromoacetic acid (TBAA) were analyzed in addition to the six required haloacetic acids. The DBP/ICR analytical methods require that the optional HAAs be analyzed using EPA method 552.2 and MRLs be set at 1, 2 and 4 µg/L for DBCAA, CDBAA and TBAA respectively. However, *Standard Method 6251B* was used to analyze these compounds and a single standard of 10 µg/L was used for calibration. Regardless, these compounds have been included in the Treatment Studies Spreadsheets and MRLs have been set at 1 µg/L.

SECTION 4

RESULTS AND DISCUSSION

FEED WATER QUALITY

Table 9 summarizes the feed water used for bench, single element, and pilot scale evaluations. Average values and standard deviations of water quality constituents monitored during SEBSTs and pilot tests are listed for each quarter of testing. Alkalinity and pH values represent an aggressive water, which is necessary to prevent fouling of inorganic salts on NF membranes.

Although PAC was added on the full scale to the lime softening plant at the KCMWSD, primarily as a measure for removing SOCs and taste and odor causing compounds, proposed stage II MCLs for THM4 and HAA5 were exceeded on the average during most quarters of testing. This would indicate the need for an advanced treatment process such as NF when using free chlorine as a residual disinfectant.

Lime softened feed water used for bench, single element, and pilot scale NF tests was subject to pilot scale sand and cartridge filtration. In this case, filtered feed water may be characterized as having a turbidity less than 1 ntu and a Silt Density Index (SDI) ranging from 3 to 5. Due to the relatively high SDI, particle fouling was expected to be an issue.

MF pretreatment proved to be much more effective at removing particles. As indicated in Table 9, MF consistently reduced raw water turbidity to less than 0.2 ntu. Silt density index values never exceed 1, which would indicate that particle fouling would not be an issue for NF. As expected, the raw river water than has only been subject to limited in-line coagulation and MF produced more DBPs than the lime softened water, which had been subject to permanganate oxidation in addition to coagulation.

Historical Missouri River atrazine concentrations are presented as an exceedance diagram, Figure 8. As indicated, the maximum contaminant level (MCL) for atrazine is exceeded 10% of the time. Typically atrazine concentrations are highest during the spring due to run-off from agricultural lands which surround the Missouri River basin. Atrazine challenges to the SEBST and pilot systems were performed to mimic the concentrations seen in the Missouri River with feed water concentrations ranging from 6 – 10 µg/L.

Table 9. ICR Treatment Study Feed Water Quality

Water Quality Parameter	LS-SEBST January – April Average (SD)	LS-SEBST April – July Average (SD)	LS-Pilot NF April – July Average (SD)	LS-Pilot NF July – September Average (SD)	LS-Pilot NF October – December Average (SD)	MF-Pilot NF January - April Average (SD)
Temperature (°C)	10.1 (3.5)	20.5 (4.6)	22.5 (5.3)	25.4 (2.2)	11.5 (7.1)	8.6 (4.2)
pH	6.9 (1.0)	6.7 (0.9)	6.3 (21.1)	7.7 (1.0)	7.1 (1.0)	6.7 (0.3)
Turbidity (ntu)	1.5 (0.8)	0.80 (0.7)	0.41 (0.2)	0.48 (0.1)	0.39 (0.2)	0.13 (0.05)
Alkalinity (mg/L as CaCO ₃)	23.5 (8.4)	28.0 (15.9)	31.8 (10.5)	40.6 (7.1)	31.5 (18.7)	89.1 (17.1)
Calcium Hardness (mg/L as CaCO ₃)	149.5 (40.9)	157.3 (27.0)	146.2 (37.1)	138.7 (20.0)	118.0 (42.3)	151.6 (41.2)
Total Hardness (mg/L as CaCO ₃)	195.1 (43.7)	172.5 (26.5)	300.9 (86.0)	294.5 (23.2)	291.3 (48.8)	405.5 (40.5)
Bromide (µg/L)	74.3 (30.1)	77.8 (14.4)	BMRL	BMRL	BMRL	64.5 (40.5)
TOC (mg/L)	2.3 (0.4)	2.6 (0.9)	2.2 (0.3)	4.6 (2.93)	2.7 (0.8)	3.2 (0.5)
UV ₂₅₄ (cm ⁻¹)	0.07 (0.1)	0.06 (0.0)	0.053 (0.0)	0.057 (0.0)	0.062 (0.0)	0.067 (0.0)
SDS-THM4 (µg/L)	14.8 (11.0)	43.1 (3.9)	47.7 (10.3)	45.9 (18.3)	42.3 (10.3)	80.3 (29.1)
SDS-HAA5 (µg/L)	9.6 (6.0)	27.5 (10.8)	24.7 (6.4)	36.7 (17.4)	25.4 (4.8)	55.1 (21.9)
SDS-HAA6 (µg/L)	12.0 (7.8)	33.3 (11.9)	30.1 (6.7)	41.9 (18.0)	29.4 (4.9)	63.1 (24.4)
SDS-TOX (µg Cl ⁻ /L)	78.1 (29.8)	123 (14.0)	133 (21.0)	122 (33.1)	100 (45.5)	147 (36.1)
SDS-Chlorine Demand (mg/L)	2.0 (0.4)	2.2 (0.2)	2.3 (0.1)	2.1 (0.5)	2.7 (0.3)	2.9 (0.3)

LS = Lime Softening Pretreatment

MF = Microfiltration Pretreatment

NF = Nanofiltration

BMRL = Below Minimum Reporting Level

SDS = Simulated Distribution System

THM4 = Trihalomethanes (chloroform, bromodichloromethane, chlorodibromomethane, bromoform)

HAA5 = Haloacetic acids (chloroacetic acid, dichloroacetic acid, trichloroacetic acid, bromoacetic acid, and dibromoacetic acid)

HAA6 = Haloacetic acids (chloroacetic acid, dichloroacetic acid, trichloroacetic acid, bromoacetic acid, and dibromoacetic acid, bromochloroacetic acid)

PRELIMINARY MEMBRANE SCREENING

Bench Scale Screening

Candidate membranes were screened on the bench-scale to determine which membranes would best meet study objectives. Pre-screening consisted of RBSMT evaluations of eight membranes for TOC, UV₂₅₄, atrazine, and TDS removal. The results of these evaluations and the recoveries employed during prescreening are presented in Table 10. Due to bench-scale system limitations, recoveries were limited to those achievable through single pass operation.

Table10. Results of RBSMT evaluation on candidate membranes

Membrane	Recovery	TOC Rejection	UV ₂₅₄ Rejection	Atrazine Rejection	TDS Rejection
<i>Sepa</i>					
ST	13%	64%	69%	35%	93%
MS	30%	81%	73%	98%	90%
<i>Purification Products</i>					
B-22-9	80%	45%	14%	47%	4%
B-22-1	70%	77%	70%	81%	81%
<i>Hydranautics</i>					
NTR-7450	33%	17%	67%	4%	32%
NTR-7410	77%	7%	4%	9%	0%
PVD1	32%	57%	96%	92%	82%
ESNA	26%	63%	92%	96%	90%

During prescreening of candidate membranes, mass balance calculations for water quality constituents were conducted to ensure the soundness of experimental data and also to ensure that atrazine removal was the product of rejection and not adsorption to the membrane. Table 11 lists the rejection of atrazine with corresponding errors in mass balance for membranes screened using the RBSMT.

Table 11. Atrazine rejection

Membrane	Scale of Test	Atrazine Observed Rejection	Error _{MB}
Sepa ST	RBSMT	35%	-17 %
Sepa MS	RBSMT	98%	-2 %
B-22-1	RBSMT	81%	8%
B-22-9	RBSMT	47%	- 410%
NTR-7450	RBSMT	4%	- 41%
NTR-7410	RBSMT	9%	-1 %
PVD1	RBSMT	92%	- 43%
ESNA	RBSMT	96%	- 2%

Feed Concentrations 4-6 µg/L

Note: A negative Error_{MB} may indicate adsorption to the membrane

Candidate membranes screened using the RBSMT may be separated into three categories based on the Error_{MB} associated with their respective atrazine rejections. The first category may be described as those membranes having an $\text{Error}_{\text{MB}} \pm 10\%$, which may be attributed to experimental error. A second category, those membranes with an Error_{MB} between -10% and -40% may be characterized as those membranes that may exhibit atrazine removal through adsorption of atrazine on the membrane surface. The third category of membrane, those membranes with an Error_{MB} lower than -40% , are membranes who adsorb atrazine. Extended operation of category two and three membranes would likely show an increase in atrazine concentrations in the permeate stream over time.

Based on study objectives (80% atrazine and 60% TOC removal respectively), taking the pre-selection Error_{MB} of atrazine for each membrane into account, the Sepa MS, Purification Products B-22-1, and the Hydranautics ESNA membranes were selected for SEBST evaluation. The Dow FilmTec NF200B was also selected based on data that the manufacturer provided which indicated that this membrane would also meet study objectives.

SINGLE ELEMENT PILOT TEST MEMBRANE SCREENING

Permeate Water Quality

Permeate water from tested NF membranes on the single element bench scale may be characterized as high purity water, low in organic carbon, alkalinity and mineral content. A summary of NF permeate water collected as part of SEBST evaluations is presented in Table 12. Values of water quality parameters listed in Table 12 are the average weekly values from 4 weeks of testing. Of the four membranes tested, average permeate TDS concentrations ranged from 4.1 mg/L for the Sepa MS to 195.9 mg/L from the NF200B. Having the highest quarterly average permeate hardness concentrations, the NF200B membrane appears to have the lowest rejection of divalent cations such as calcium and magnesium. The B-22-1 and ESNA had similar concentrations of alkalinity and TDS in their permeate waters. Trends in average permeate alkalinity concentrations mirror TDS concentrations while bromide concentrations were frequently reduced to below detection levels for all membranes tested.

The application of traditional NF and RO membranes has been limited in the past due to high operating pressures and the need for post treatment of permeate water for remineralization and LSI adjustment. Low alkaline, low pH waters have been characteristic of NF permeate waters. The future of NF lies in the development of membranes that may operate at low pressures while rejecting fewer inorganic constituents. This puts membranes like the ESNA and the NF200B are on the cutting edge of NF due to the reduced rejection of inorganics while rejecting significant amounts of organics (i.e. TOC).

Observed rejections of atrazine for SEBST evaluations of the B-22-1 and ESNA membranes were relatively lower than the rejections observed during the bench scale tests, 64% and 84% respectively. It should be noted that recoveries employed during the

bench scale tests were lower than the 75% recovery employed during the SEBST due to system limitations. As recovery increases, at a constant feed water concentration of atrazine, the bulk concentration of atrazine would increase likewise. With a greater concentration of atrazine in the bulk stream, the driving force for atrazine mass transfer would be greater, thereby forcing more atrazine to the permeate stream and ultimately producing a lower observed rejection. Rejection of atrazine for the Sepa MS membrane remained superior at close to 100% rejection (permeate concentrations of atrazine were below detection). The NF200B membrane rejected only 50% of feed water atrazine, which is lower than rejections observed by other researchers (Ventresque, et. al. 1997). Source water chemistry as well as membrane chemistry has been noted to effect the rejection of atrazine from NF membranes and it is likely that the solution chemistry softened Missouri River water played a role in this discrepancy (Devitt and Wiesner, 1998).

The need for remineralization and treatability of concentrate are limitations to the implementation of NF on the full scale. A membrane that may pass sufficient amount of inorganics while rejecting a maximum amount of organics represents the future of nanofiltration. However, inorganic compounds that may pass such a membrane will likely include bromide. Ultimately, any organics passing which are precursors to DBP formation would subject to form a greater percentage of brominated DBPs since the bromide to TOC ratio would increase and brominated DBP formation is favored kinetically to chlorinated DBP formation. Figures 9-12 present the percent DBP yield from available TOC in feed and permeate waters during this study. There is a marked increase in brominated DBP yield in the permeate water from the NF200B (Figure 12) which, as discussed passed the most significant concentration of TDS and hardness during both quarters of SEBST evaluation. Examination of permeate THM yield from other membranes does not indicate this shift toward brominated DBP formation.

Table 12. Single Element NF Permeate Water Quality

Water Quality Parameter	Sepa MS Quarter 1 Ave (SD)	Sepa MS Quarter 2 Ave (SD)	B-22-1 Quarter 1 Ave (SD)	B-22-1 Quarter 2 Ave (SD)	ESNA Quarter 1 Ave (SD)	ESNA Quarter 2 Ave (SD)	NF200B Quarter 1 Ave (SD)	NF200B Quarter 2 Ave (SD)
TDS (mg/L)	4.1 (1.7)	11.9 (1.4)	95.8 (31.4)	52.1 (15.6)	34.8 (17.3)	70.3 (7.8)	75.0 (53.4)	195.9 (30)
pH	7.3 (1.1)	6.3 (0.4)	6.4 (0.4)	6.6 (0.7)	6.4 (0.2)	7.0 (0.6)	7.7 (1.2)	5.9 (0.7)
Alkalinity (mg/L as CaCO ₃)	5.0*	5.0*	10.0 (1.0)	8.8 (3.0)	8.9 (1.5)	9.0 (1.6)	11.8 (5.9)	34.0 (15.8)
Calcium Hardness (mg/L as CaCO ₃)	4.0*	1.9 (2.1)	12.2 (11.5)	8.0 (4.6)	2.8 (1.1)	3.0 (0.9)	19.8 (12.3)	53.9 (24.9)
Total Hardness (mg/L as CaCO ₃)	6.9*	1.9 (2.1)	15.2 (13.8)	8.6 (5.0)	3.3 (1.7)	3.2 (1.2)	28.5 (17.8)	58.6 (23.5)
Bromide (µg/L)	BMRL	BMRL	85.0 (21.1)	50*	60*	BMRL	78.8 (16.5)	BMRL
TOC (mg/L)	0.4 (0.1)	0.3 (0.1)	0.4 (0.1)	0.4 (0.2)	0.3 (0.2)	0.4 (0.1)	0.5 (0.1)	0.8 (0.2)
UV ₂₅₄ (cm ⁻¹)	0.02 (0)	0.2 (0)	0.02 (0)	0.02 (0)	0.02 (0)	0.02 (0)	0.2 (0)	0.03 (0)
SDS-THM4 (µg/L)	3.4 (6.4)	2.7 (0.5)	18.1 (14.3)	4.8 (1.9)	2.6 (0)	3.2 (0.6)	1.4 (1.6)	15.7 (7.9)
SDS-HAA5 (µg/L)	4.9 (6.3)	5.5 (1.8)	15.3 (8.0)	3.9 (1.1)	2.1 (0.1)	1.7 (0.5)	1.6 (0.2)	4.8 (1.9)
SDS-TOX (µg Cl ⁻ /L)	BMRL	BMRL	32.0*	25.0* (5.9)	BMRL	BMRL	BMRL	25*
SDS-Chlorine Demand (mg/L)	0.3 (0.1)	0.4 (0.1)	0.7 (0.2)	0.6 (0.1)	0.5 (0.3)	0.6 (0.1)	0.5 (0.3)	0.8 (0.1)
Atrazine (µg/L) [†]	0.05	NA	2.15	NA	0.63	NA	2.79	NA

* All but one week (which is reported) was below detection

[†] Not required by the ICR

NA = Not available

Membrane Productivity on the Single Element Bench Scale

Membrane productivity is referred to as the mass transfer coefficient of water (MTC_w) by the ICR, but is more commonly called specific flux. A membrane's MTC_w is expressed as volume of water produced per unit area of membrane surface per unit time per unit of applied pressure (gfd/psi or L/hr.m².bar). MTC_w is a measure of how productive a membrane may be through the application of pressure. To achieve an equal production, a membrane with a low MTC_w would require either more pressure or more membrane surface area. Either may result in a greater capital or operating cost. Thus, the examination of MTC_w decline (gfd/psi/day) during a pilot study is an important element in cost analysis.

To satisfy the requirements of the ICR for WDN01JC, each of the four membranes selected for SEBST evaluation were operated for two, 30-day quarters. Figures 13–20 present the results of daily hydraulic monitoring for each quarter of SEBST evaluation. As required by the ICR, each SEBST was operated at a recovery of 75%. Cleaning events are indicated on each graph.

Figures 13 and 14 present the results of operating the Sepa MS membrane. This membrane is characterized by a high operating pressure, as noted in Table 4, operating at approximately 150 psi. During the first quarter of evaluation, the transmembrane pressure (TMP) increased steadily over the first ten days of operation to reach 200 psi after which it stabilized for the following ten days. A chemical clean was performed at day 24 due to a break through of PAC from the pilot scale sand and cartridge filters. PAC breakthrough continued plagued operation during the last 7 days of operation as noted with a sharp increase in TMP to 225 psi. During the second quarter of Sepa MS evaluation, the first 5 days of operation saw a steady increase in TMP. This increase was attributed to inorganic deposits on the membrane brought on by the rapid hydrolysis of SHMP to ortho-phosphate. SHMP hydrolysis was enhanced by an increase in seasonal temperatures. As a result, scale inhibitor was switched from SHMP to Hypersperse SI 300 as noted in Figure 14. TMP increased steadily during the second quarter of Sepa MS operation and the test was terminated with a TMP of 190 psi.

Two issues arose during the operation of the B-22-1 membrane that are noteworthy. During the first quarter of testing (Figure 15) the MTC_w rapidly decreased and operation stabilized at an unexpectedly high transmembrane pressure (TMP) upon initial operation. This TMP was much higher than that reported by the manufacturer (Table 4) and that seen during bench-scale pre-screening. This rapid increase in TMP was mistaken for compaction of the membrane's porous matrix and the B-22-1 was operated at this high TMP throughout the first quarter. Due to this high operating pressure, a recovery of 75% could not be maintained and a lower flux (12 L/hr.m²) was required for operation. Prior to storage, this membrane was cleaned (caustic followed by acid wash) and the second quarter of B-22-1 operation (Figure 16) yielded a TMP of 45 psi. Due to recovery in TMP and MTC_w , it is assumed that this membrane experienced some form of rapid fouling which was either biological or inorganic in nature during the first day of operation during quarter number one.

Testing with the ESNA membrane was performed at a flux of 20 L/hr.m^2 at T_{ave} and a 75% recovery. The ESNA membrane operated at a TMP of approximately 60-psi as seen in Figures 17 and 18. Particle fouling triggered the first chemical cleaning as indicated on day 26 in Figure 17. Again, PAC broke through pilot scale filters prior to NF and rapidly fouled the membrane. However, chemical cleaning during the second quarter of ESNA evaluation was attributed to inorganic fouling and ineffective anti-scalant addition due to the hydrolysis of SHMP.

The NF200B membrane operated in a manner similar to that of the ESNA. During the first quarter of SEBST evaluation, the TMP ranged from 50 psi to 90 psi from beginning to end as shown in Figure 19. Particle fouling was successfully controlled during the first quarter by scouring the membrane using high recirculation velocities for a short period of time. The second quarter of testing lead to results that are reported in Figure 20. Chemical cleaning was required after 20 days of operation. Subsequently, two additional cleanings were required during the last 15 days of testing. As with the second quarter of ESNA evaluation, fouling was attributed to inadequate scale inhibition by SHMP at elevated temperatures.

Rates of MTC_w decline were calculated for each membranes based on the negative slope of daily operating values between chemical cleaning intervals as seen in Figures 10–17. Based on cleaning criterion (15% MTC_w decline), an average cleaning interval was calculated using these MTC_w decline rates. Rates of MTC_w decline and calculated chemical cleaning intervals are reported in Table 13.

Table 13. Membrane productivity and cleaning

Membrane Quarter	Average Rate of MTC _w Decline L/hr.m ² .bar.day (gfd/psi.day)	Average Cleaning Interval days	Initial MTC _w L/hr.m ² .bar (gfd/psi)	Final MTC _w L/hr.m ² .bar (gfd/psi)	MTC _w After Cleaning L/hr.m ² .bar (gfd/psi)
Sepa MS Quarter 1	-7.1 x 10 ⁻³ (-2.9 x 10 ⁻⁴)	41	1.97 (0.08)	1.47 (0.06)	1.72 (0.07)
Sepa MS Quarter 2	-1.5 x 10 ⁻² (-6.3 x 10 ⁻⁴)	24	2.46 (0.1)	1.72 (0.07)	NC
B-22-1 Quarter 1	-1.6 x 10 ⁻² (-6.8 x 10 ⁻⁴)	20	2.22 (0.09)	0.49 (0.02)	1.47 (0.06)
B-22-1 Quarter 2	-4.2 x 10 ⁻² (-1.7 x 10 ⁻³)	16	5.91 (0.24)	5.66 (0.23)	NC
ESNA Quarter 1	-5.4 x 10 ⁻² (-2.2 x 10 ⁻³)	20	4.68 (0.19)	3.20 (0.13)	5.17 (0.21)
Membrane Quarter	Average Rate of MTC _w Decline L/hr.m ² .bar.day (gfd/psi.day)	Average Cleaning Interval days	Initial MTC _w L/hr.m ² .bar (gfd/psi)	Final MTC _w L/hr.m ² .bar (gfd/psi)	MTC _w After Cleaning L/hr.m ² .bar (gfd/psi)
ESNA Quarter 2	-5.1 x 10 ⁻² (-2.1 x 10 ⁻³)	15	5.17 (0.21)	3.20 (0.13)	5.17 (0.21)
NF200B Quarter 1	-9.8 x 10 ⁻² (-4.0 x 10 ⁻³)	10	6.65 (0.27)	3.44 (0.14)	NC
NF200B Quarter 2	-6.6 x 10 ⁻² (-2.7 x 10 ⁻³)	13	5.91 (0.24)	2.71 (0.11)	4.43 (0.18)

MTC_w = Mass transfer coefficient of water (or specific flux)

NC = No cleaning performed during quarter

These data summarize the hydraulic operation of each membrane in a manner that is suitable for determining design criteria. Also, calculated chemical cleaning intervals would likely vary according to pretreatment practices. Comparison of lime softening followed granular filtration to alternative pretreatments (i.e. UF or MF) for NF would also provide data critical to design and are evaluated as part of the pilot scale testing presented later in this report.

Summary Of Single Element Testing

Based on the hydraulic and water quality monitoring performed on using the SEBST, the Hydranautics ESNA membrane best meets project goals and was selected for pilot scale testing.

PILOT SCALE ICR TESTING

Permeate Water Quality on the Pilot Scale

As required by the ICR, pilot scale water quality was monitored biweekly across four quarters. Table 14 presents the pilot scale permeate water using the Hydranautics ESNA membrane. The first three quarters present only the data collected while lime softening was used as a pretreatment (LS-Pilot NF1 through LS-Pilot NF3). The fourth quarter of testing presents results from new ESNA membranes where MF served as a pretreatment. Reported values are the average of six bi-weekly samples taken across ten weeks of monitoring.

NF permeate water from the ESNA membrane operated on the pilot scale, as with single element results, indicate a highly purified water, low in TDS, hardness and TOC which is slightly aggressive in nature. Total hardness, TDS and alkalinity values for NF permeate using MF as a pretreatment are quite higher than those concentrations seen using lime softening as a pretreatment. In fact, finished water hardness from the ESNA membrane using MF as a pretreatment is high enough for distribution without remineralization. While the alkalinity is sufficiently high, it may be advisable to adjust the pH above a value of 7.0 to protect the distribution system from corrosion.

Average SDS-DBP values are all within the range acceptable for Stage I and Stage II D/DBP concentrations. TOC concentrations during the second quarter (July – September) of pilot tests are higher than those seen during quarters one and three (in all cases lime softening serves as pretreatment). This may be explained by observing the feed water quality data presented in Table 9 which indicates that feed water TOC for the NF pilot system is, on the average, much higher than those seen during quarters one and three. SDS-DBP yield from the MF treated water is also high and the elevated NF permeate concentrations seen during quarter number four may be explained by a combination of high feed water TOCs and a difference in pretreatment. Consequently, NF permeate from the MF treated water exhibits higher SDS-DBP concentrations while still achieving regulatory standards on the average.

Table 14. Pilot scale permeate water quality

Water Quality Parameter	LS-Pilot NF1 April – July Average (SD)	LS-Pilot NF2 July – September Average (SD)	LS-Pilot NF3 October – December Average (SD)	MF-Pilot NF4 January – April Average (SD)
TDS (mg/L)	22.9 (8.0)	22.0 (3.0)	20.7 (3.2)	174.0 (27.5)
pH	5.0 (2.5)	7.4 (1.6)	5.9 (0.4)	6.3 (0.3)
Alkalinity (mg/L as CaCO ₃)	5*	7.2 (2.4)	BMRL	33.5 (8.1)
Calcium Hardness (mg/L as CaCO ₃)	1.7 (1.3)	0.3 (0.1)	0.4*	40.6 (11.1)
Total Hardness (mg/L as CaCO ₃)	2.3 (1.2)	BMRL	BMRL	64.7 (16.7)
Bromide (µg/L)	BMRL	BMRL	BMRL	40 (0.0)
TOC (mg/L)	0.4 (0.1)	0.8 (0.5)	0.4 (0.1)	0.9 (0.3)
UV ₂₅₄ (cm ⁻¹)	0.019 (0.002)	0.021 (0.003)	0.029 (0.006)	0.012 (0.002)
SDS-THM4 (µg/L)	2.3 (1.4)	3.5 (1.7)	0.9 (1.0)	19.0 (10.8)
SDS-HAA5 (µg/L)	2.9 (1.4)	11.9 (9.1)	4.4 (8.5)	9.8 (6.0)
SDS-HAA6 (µg/L)	3.3 (1.7)	11.9 (9.1)	4.4 (8.5)	15.6 (10.0)
SDS-TOX (µg Cl/L)	BMRL	BMRL	BMRL	36 (9.2)
SDS-Chlorine Demand (mg/L)	0.5 (0.1)	0.5 (0.2)	0.5 (0.1)	0.8 (0.2)

LS = Lime softening pretreatment

MF = Microfiltration pretreatment

NF# = Quarter of nanofiltration pilot testing

* Only one sample reported (others below detection level)

BMRL = Below minimum reporting level

Membrane Productivity On The Pilot Scale

The ICR requires that NF be evaluated at the pilot scale for a total of 6,600 hours over a period of four, 70-day quarters. Pilot testing of the ENNA membrane was performed at a recovery of 93% and the flux for each quarter of evaluation was set to 15 gfd (25 L/hr.m²) normalized to a temperature indicative of the average quarterly temperature. Three quarters of pilot testing where lime softening serves as a pretreatment for NF are presented in Figures 21-23. In each case, scale inhibitor and acid addition was standard. A detailed summary of pilot testing interruptions and condition changes throughout the course of pilot testing are presented in tabular form in Appendix D.

During the first quarter of pilot scale testing (Figure 21), chemical cleans were required on days 20 and 27 respectively. These cleanings were required due to the hydrolysis of SHMP to ortho-phosphate. Hydrolysis of SHMP occurs under most conditions, but is enhanced by exposure of the stock solution to the open atmosphere and elevated temperatures. Due to the increase in seasonal temperature, use of SHMP was discontinued and an alternative scale inhibitor, Hypersperse SI 300 was used. In addition

to sequestering calcium carbonate and calcium sulfate scale, Hypersperse SI 300 is also capable of sequestering silica scale, which may be an issue at recoveries greater than 90%. An additional fouling event was observed on day 50, which appeared to remedy itself over the course of 5 days. No attempt was made to clean the membrane since the primary operator was not available.

The first 30 days of the second quarter of the pilot-scale evaluation, Figure 22, were characterized by pH control difficulties. Fouling events on days 10 and 15 were the result of pH control failure that, in part, was due to an elevated source water alkalinity. Chemical cleaning was not performed immediately upon occurrence of the second fouling event that began on day 15. Again this was due to the fact that the primary operator was not available to perform the cleaning. These two fouling events are a testimony to the need for pretreatment with acid to a pH of 6. In addition, recycle of stage 2 concentrate to the head of stage 1 was implemented on day 35. Recycle was implemented to normalize the stage 1 and stage 2 cross flow velocity to 0.6 ft/s and reduce fouling observed in stage 2.

The third quarter of pilot-scale evaluation (Figure 23) was performed at a normalized flux and recovery comparable to that of quarter number one. An increase in TMP from approximately 60 to 115 psi over the entire course of testing is coupled with nearly a 35% loss in MTC_w (specific flux). Other researchers have also noted that the ESNA membrane is significantly susceptible to fouling. Scanning electron microscope pictures of this membrane have revealed an exceptionally rough surface that would be prone to accumulation of particulates or biological growth (Riess et al., 1999). As a result of these findings, Hydranautics is working to develop a new smoother version of the ESNA membrane. Suspicious fouling events observed in previous quarters lead to the daily addition of sodium bisulfite as a preventative measure for bio-fouling control. Sodium bisulfite was added to the pilot feed water once a day for 30 minutes at a concentration of 500 mg/L to quench dissolved oxygen and in effect suffocate any aerobic organisms that could potentially foul the membrane. No significant increase in permeate water TDS was observed during bisulfite addition.

The fourth quarter of pilot scale ICR testing was performed from January to April of 1999 using MF as a pretreatment. Figure 24 presents the results of the NF testing during this period. Membrane surface area in the NF pilot was decreased on day 12. This was done so that the flux could be increased while operating in a hydrodynamic range acceptable to the manufacturer's specifications. In addition, challenging source water limited the amount of permeate available from the MF pretreatment pilot plant also requiring the decrease in NF surface area. As with the lime softening pretreatment NF results, these data indicate that the ESNA is susceptible to a great deal of irreversible fouling. From start to end of this testing period, 35% of the initial MTC_w (specific flux) is lost. A chemical cleaning was initiated on day 42 and no productivity was recovered.

One may expect that the MF pretreatment would produce the best productivity data since particle fouling is limited and most, if not all, biological organisms are removed. However, when comparing productivity data presented in Table 15, the lime softening

pretreatment seemed to operate competitively. To allow comparison of the effects associated with seasonal variation with a single pretreatment (lime softening), the single element data collected during the months January and March of 1998 for the ESNA membrane have been included in this table. It can be noted that the chemical cleaning frequency for this quarter of testing is quite high and exceeds the frequencies required during other quarters of testing. This may only be a factor of the scale of testing, or it may indicate that this quarter is the most challenging in terms of treatment. If so, perhaps further testing of the ESNA membrane with MF pretreatment would produce cleaning frequencies that out perform the lime softening pretreatment NF productivity data.

Table 15. ESNA membrane productivity and cleaning

Quarter	Average Rate of MTC _w Decline L/hr.m ² .bar.day (gfd/psi.day)	Average Cleaning Interval days	Initial MTC _w L/hr.m ² .bar (gfd/psi)	Final MTC _w L/hr.m ² .bar (gfd/psi)	MTC _w After Cleaning L/hr.m ² .bar (gfd/psi)
SEBST*	- 5.3 x 10 ⁻² (- 2.2 x 10 ⁻³)	18	4.9 (0.20)	3.2 (0.13)	5.17 (0.21)
LS-NF1	- 2.0 x 10 ⁻² (- 8.4 x 10 ⁻⁴)	53	5.3 (0.22)	4.9 (0.20)	5.3 (0.22)
LS-NF2	- 2.0 x 10 ⁻² (- 8.4 x 10 ⁻⁴)	42	4.2 (0.17)	3.5 (0.14)	3.9 (0.16)
LS-NF3	- 1.5 x 10 ⁻² (- 6.3 x 10 ⁻⁴)	55	3.3 (0.13)	2.9 (0.11)	3.3 (0.13)
MF-NF4	- 2.0 x 10 ⁻² (- 8.4 x 10 ⁻⁴)	46	4.6 (0.19)	3.7 (0.15)	3.7 (0.15)

MTC_w = Mass transfer coefficient of water (or specific flux)

* Single element ESNA results (average of January & March 1998 data)

LS = Lime softening pretreatment

MF = Microfiltration pretreatment

NF# = Quarter of nanofiltration pilot testing

Permeate And Feed Water Blending

There are three water treatment goals that must be weighed when evaluating NF as a polishing treatment for softened Missouri River water. Reduction of DBP precursors such as TOC, removal of atrazine and disinfection are all accomplished through nanofiltration. If the goal of NF in water treatment was solely reduction of DBPs and/or atrazine, then feed water and permeate water may be blended to achieve a blended water quality that meets regulatory guidelines. However, should water be blended then any disinfection achieved through NF would be negated.

During all quarters of testing Stage II concentrations for both trihalomethanes and haloacetic acids were exceeded within a 10% factor of safety. Table 16 lists the maximum permeate to total water blend ratios and calculated blended water quality for the ESNA membrane. In each case the controlling treatment objective was a Stage II

D/DBP treatment standard. Reported conditions represent the worst occurrence of DBP yield during a testing quarter. In most testing quarters, Stage I treatment standards were exceeded and would indicate the need for an advanced treatment technology such as NF when using free chlorine as a residual disinfectant. Stage II treatment standards however, produced the highest blend ratios and were therefore reported in lieu of any Stage I blending. Of interest in Table 12 data are the blend ratios for the single element NF tests and the MF-NF tests. The SEBST produced the lowest blend ratio when compared to the pilot scale data for quarters NF1 through NF3. This may be indicative of the seasonal variation, or may again be a factor of the scale of testing. The MF pretreated NF permeate failed to meet the Stage II regulations within a 10% factor of safety. While the actual average concentration for Stage II THM4 is within regulatory guidelines, the individual occurrence data, which was used to calculate the blend ratio, was slightly greater than the regulatory limit with a 10% factor of safety applied. These data would indicate that direct NF would be required at all times using MF as a pretreatment during this quarter of testing.

Table 16. Blending ratios and blended water quality for controlling DBPs

Membrane	Controlling Treatment Objective	Value of Listed Parameter at Blend Ratio to Achieve Controlling Treatment Objective				
		Permeate: Total Flow Blend Ratio	TOC (mg/L)	SDS-THM4 (µg/L)	SDS-HAA5 (µg/L)	SDS-TOX (µg Cl ⁻ /L)
SEBST*	SDS-THM4 36 µg/L	0.23	2.1	36.0	23.0	NA
LS-NF1	SDS-THM4 36 µg/L	0.47	1.4	36.0	17.5	NA
LS-NF2	SDS-HAA5 27 µg/L	0.70	0.9	25.3	27.0	NA
LS-NF3	SDS-HAA5 27 µg/L	0.83	0.7	7.8	27.0	NA
MF-NF4	SDS-THM4 36 µg/L	> 1.0	1.0 [‡]	37.1 [‡]	20.0 [‡]	51.0 [‡]

* Single Element ESNA blending calculations

[‡] Value presents direct NF, concentration of control DBP greater than MCL with 10% factor of safety

NA = Not available, permeate concentrations below minimum detection level

LS = Lime softening pretreatment

MF = Microfiltration pretreatment

NF# = Quarter of nanofiltration pilot testing

Due to the nature of this study, blend ratios should also be calculated with atrazine as a treatment objective. With a 10% factor of safety applied to the maximum contaminant level (MCL) for atrazine ($MCL_{\text{atrazine}} = 3.0 \mu\text{g/L}$), blended water quality atrazine concentrations would be targeted for a concentration of $2.7 \mu\text{g/L}$. Table 17 lists the concentrations of atrazine in NF feed and permeate waters from each membrane along with the average flux employed.

Table 17
Atrazine blend ratios and hydraulic operating conditions

Pretreatment	Feed Concentration µg/L	Permeate Concentration µg/L	Permeate: Total Flow Blend Ratio	Flux gfd
Lime softening (LS – NF1)	9.0	0.3	0.72	15
Microfiltration (MF-NF4)	14.1	0.6	0.84	15

The lime softening – NF data presented in this table represents the first quarter of pilot testing (LS – NF1). From these data it is evident that atrazine would control the amount of NF required since the blend ratio of 0.72 exceeds the blend ratio of 0.47 required to treat the same water to comply with DBP regulations. The NF permeate where MF serves as pretreatment had a lower blend ratio than the corresponding DBP treatment data. From these data, it is evident that if NF is used to achieve multiple treatment objectives, such as DBP and SOC removal, care must be taken to ensure that blending achieves all treatment goals.

COST ANALYSIS

Lime Softening Pretreatment

This ICR study served as compliance for the Kansas City Missouri Water Services Department (ICR Plant No. 430) who has facility rated for a 240-MGD capacity. To implement NF as a polishing treatment for existing lime softening processes, the plant capacity would be reduced to 216 MGD assuming all water would be treated by NF operating at a 90% recovery. The investment required to implement NF as a polishing treatment for lime softening is 146.2 million dollars. This value includes capital costs, building costs and pre- and post-treatment implementation costs.

A summary of all factors included in pricing a NF system for implementation are presented in Table 18. The plant factor is a value representing the utilization of plant capacity over the course of one year and was calculated based on the total water delivered in 1998.

Table 18. Calculation of Investment and Water Cost for Integrating NF in an Existing Lime Softening Process

DESIGN PARAMETERS			
Plant capacity, MGD	216	Labor rate, \$/hr	30
Design flux, gfd	15	Staff, # people	15
Investment, \$	146.2 mil	Plant life, years	30
Interest rate, %	10	Membrane life, years	5.0
Plant factor, %	40	Membrane cost, \$/element	600
Engineering & Administration (as % of investment)	20.0	Number of elements	35340
Contingency (as % of investment)	10.0	Inhibitor cost, \$/lb	3.71
Power Cost, \$/kWhr	0.05	Inhibitor dosing, mg/L	4.0
Power consumption, kWhr/kgal	3.06	Acid cost, \$/lb	0.07
Maintenance (as % of investment)	3.0	Acid dosing, mg/L	104.7
NF WATER COSTS			
Capital cost, \$/kgal	0.48		
Engineering & administration fees, \$/kgal	0.10		
Contingency, \$/kgal	0.05		
Power cost, \$/kgal	0.13		
Labor cost, \$/kgal	0.02		
Chemical cost, \$/kgal	0.18		
Membrane replacement cost, \$/kgal	0.13		
Maintenance, \$/kgal	0.14		
TOTAL WATER COST, \$/kgal	1.23		

KCMWSD currently charges their customers \$1.58 per 1000 gallons of water produced. Implementation of NF as a polishing treatment to existing unit processes would likely increase the total cost of water by at least 78% over the current value. Therefore, the estimated cost of water treated by an integrated NF – lime softening system would be \$2.81 per 1000 gallons of finished water.

Membrane Pretreatment

For this cost analysis it is assumed that all water is to be treated by NF and no MF – NF permeate water blending occur. This treatment scenario would require 240 mgd of Zenon permeate water capacity while operating at 90% recovery of feed water. With this capacity, a NF system operating at 90% recovery would produce 216 mgd of finished water. Again, the plant factor (at a value of 40) is a value that represents the use of plant capacity over the course of an entire year and was based on historical demand values for the year 1998. Since the IMS facility scenario presented would be a new facility, a distribution system pumping station would also need to be included in the costs and have been included as part of the Zenon MF system.

Table 19 presents the costs associated with the Zenon MF treatment process as well as pumping for distribution. Of interest, the cost of the air scour is approximately 90% of the Zenon power cost. Air blowers have been noted to be extremely inefficient in terms of power usage. To operate a cross flow membrane system, were no air is used may

reduce the power cost, however the capital costs could be impacted. Treatment with a cross flow membrane system should also be investigated to develop more design criteria.

Table 19. Calculation of Investment and Water Cost for Zenon MF Treating the Missouri River Supply

DESIGN PARAMETERS			
Plant capacity, MGD	240	Labor rate, \$/hr	30
Design flux, gfd	30	Staff, # people	15
Investment, \$	150.6 mil	Plant life, years	30
Interest rate, %	10	Membrane life, years	5
Plant factor, %	40	Membrane cost, \$/cassette	
Engineering & Administration (as % of investment)	20.0	Number of cassettes	16,000
Contingency (as % of investment)	10.0	Fe ₂ (SO ₄) ₃ cost, \$/lb (as Fe)	0.048
Power Cost, \$/kWhr	0.05	Fe ₂ (SO ₄) ₃ dosing, mg/L (as Fe)	5.0
Power consumption (MF system), kWhr/kgal	0.76	Inhibitor cost, \$/lb	3.71
Power consumption (pump station), kWhr/kgal	0.65	Inhibitor dosing, mg/L	1.0
Maintenance (as % of investment)	5.0		
WATER COSTS			
Capital cost, \$/kgal		0.50	
Engineering & administration fees, \$/kgal		0.10	
Contingency, \$/kgal		0.05	
Power cost, \$/kgal		0.06	
Labor cost, \$/kgal		0.01	
Chemical cost, \$/kgal		0.05	
Membrane replacement cost, \$/kgal		0.16	
Maintenance, \$/kgal		0.18	
TOTAL WATER COST, \$/kgal		1.11	

The cost of NF after MF pretreatment, presented in Table 20, are lower than those seen from polishing lime softened water. This is expected since fouling by particles would be reduced and the overall extension of life for the NF membranes is anticipated. Chemical costs were slightly reduced due to a decrease in source water pH and acid consumption. In the end, one may anticipate a reduction of approximately \$0.05 to \$0.10 per 1,000 gallons of NF water produced by having a membrane pretreatment.

Table 20. Calculation of Investment and Water Cost for NF in an Integrated Membrane System

DESIGN PARAMETERS			
Plant capacity, MGD	216	Labor rate, \$/hr	30
Design flux, gfd	15	Staff, # people	10
Investment, \$	146.2 mil	Plant life, years	30
Interest rate, %	10	Membrane life, years	10.0
Plant factor, %	40	Membrane cost, \$/element	600
Engineering & Administration (as % of investment)	20.0	Number of elements	35340
Contingency (as % of investment)	10.0	Inhibitor cost, \$/lb	3.71
Power Cost, \$/kWhr	0.05	Inhibitor dosing, mg/L	4.0
Power consumption, kWhr/kgal	3.06	Acid cost, \$/lb	0.07
Maintenance (as % of investment)	3.0	Acid dosing, mg/L	55.0
NF WATER COSTS			
Capital cost, \$/kgal	0.48		
Engineering & administration fees, \$/kgal	0.10		
Contingency, \$/kgal	0.05		
Power cost, \$/kgal	0.13		
Labor cost, \$/kgal	0.01		
Chemical cost, \$/kgal	0.16		
Membrane replacement cost, \$/kgal	0.07		
Maintenance, \$/kgal	0.14		
TOTAL WATER COST, \$/kgal	1.14		

COST SUMMARY

Total water cost for an integrated MF – NF system, using the Zenon technology as the preliminary membrane treatment has been estimated to cost \$2.25 per 1,000 gallons of finished water. *This cost does not include administrative overhead associated with plant operation, distribution system maintenance and other fees associated with the cost that the KCMWSD provided as the cost of water to their customers.* However, if we were to compare this to a lime softening pretreatment, where the combined finished water cost is only \$2.81 per 1,000 gallons, the implementation of NF as a polishing treatment to a conventional process, such as lime softening, may seem comparable in cost. The added benefits associated with reducing risk of microbial pathogens and carcinogenic by-products may provide an impetus for water utilities considering future facilities to look at an IMS. Pilot testing a cross flow (or a gravity membrane system that is new to the drinking water market) may reduce costs associated with an integrated membrane system and should therefore be investigated.

SECTION 5

QA/QC SUMMARY

Quality Assurance (QA) and Quality Control (QC) measures conducted during this study were performed in accordance to the procedures outline in the *DBP/ICR Analytical Methods Manual*. Of particular interest to the ICR treatment studies were the methods associated with measuring and reporting DBPs and DBP precursors including TOC, Bromide, TOX, THMs, and HAAs. Calibration procedures that were employed by the Kansas City Missouri Water Services Laboratory (KCMWSL) (ICR Lab # ICRMO004) for each of these analytes are summarized in the following:

CALIBRATION SUMMARY

Total Organic Carbon (TOC)

Calibration for TOC samples, measured using Standard Method 5310B, was performed on a daily basis. Calibration consisted of two blank (distilled deionized water) injections followed by injection of a low, medium and high level calibration standard. Preparation of low level calibration standards was performed after blank samples had been analyzed. The low level calibration standard was prepared at a concentration equal to twice the detected concentration obtained from the blank analysis, which was never less than 0.2 mg/L. Prepared separately from the calibration standards, low, medium and high level check standards were analyzed to verify calibration. Medium and high level (calibration and check) standard concentrations were 4 and 10 mg/L respectively.

After all calibration standards had been analyzed, a low-level calibration check standard was analyzed. Two injections of this standard were analyzed and used to compute a relative percent difference (RPD) which was required to be $\leq 20\%$. After the low level check standard had successfully verified the instrument calibration, 10 ICR treatment study samples were analyzed in duplicate (as laboratory duplicates). RPDs for samples whose measured concentration were ≤ 2.0 mg/L were required to be $\leq 20\%$ while samples whose concentrations exceeded 2 mg/L only required an RPD $\leq 10\%$.

Once 10 ICR treatment study samples had been analyzed, a medium or high level check standard was then analyzed. The RPD of the medium and high level check standard was required to be $\leq 10\%$. If the RPD of the medium or high-level check standard did not meet this criteria, the previous 10 samples would be reanalyzed. If any of the pervious 10 ICR treatment study samples failed to meet the acceptance criteria with respect to sample RPD, the sample was then included in the next 10 ICR treatment study samples to be analyzed in duplicate. This procedure of analyzing 10 ICR treatment study samples followed by a check standard was repeated until 30 treatment study samples had been analyzed. After analyzing 30 treatment study samples, a medium or high level check standard was analyzed, which marked the end of analyses under the current calibration.

Bromide

Calibration for Bromide samples, measured using EPA Method 300.0, was performed on a daily basis. Calibration consisted of two blank (distilled deionized water) injections followed by injection of a low, medium and high level calibration standard. Prepared separately from the calibration standards, low, medium and high level check standards were analyzed to verify calibration. Low, medium and high level (calibration and check) standard concentrations were 0.02, 0.10 and 0.30 mg/L respectively.

After all calibration standards had been analyzed, a low-level calibration check standard was analyzed. One injection of this standard was analyzed and used to compute a surrogate recovery (detector response/average calibration curve detector response). For the low-level calibration check standard, the surrogate recovery was required to be between 50 and 150%. After the low level check standard had successfully verified the instrument calibration, 10 ICR treatment study samples were then analyzed as laboratory duplicates.

Following the analysis of 10 ICR treatment study samples, a medium or a high-level calibration check standard was analyzed. If the surrogate recovery of the check standards ranged between 90 and 110%, analysis of the next 10 ICR treatment study samples could then proceed. If the check standard surrogate recovery exceeded the specified range, all 10 ICR treatment study samples would be reanalyzed. The process of analyzing 10 ICR treatment study samples, followed by a medium or high level check standard was continued until all the samples had been analyzed. The very last sample analyzed was always a check standard. No more than 30 ICR treatment study samples would be analyzed in a single day. This number was never reached in practice.

Total Organic Halogen (TOX)

Calibration for TOX samples, measured using Standard Method 5320 B, was performed multiple times randomly throughout each day samples were analyzed. Calibration consisted of three blank injections (two laboratory and one system blank) followed by injections of low, medium and high NaCl standards whose concentrations were 25, 200 and 500 respectively. Prior to injection of standardized NaCl solutions, background concentrations of blanks were checked and made sure not to exceed 0.8 µg Cl⁻/L and 12.5 µg Cl⁻/L for laboratory and system blanks respectively.

After all calibration standards had been analyzed, a low-level calibration check standard was analyzed. One injection of this standard was analyzed and used to compute a surrogate recovery (detector response/average calibration curve detector response). For the low-level calibration check standard, the surrogate recovery was required to be between 75 and 125%. After the low level check standard had successfully verified the instrument calibration, 7 ICR treatment study samples were then analyzed as laboratory duplicates.

Following the analysis of 7 ICR treatment study samples, another laboratory blank was analyzed. If the interference concentration did not exceed 0.8 µg Cl⁻/L, a medium or a high-level calibration check standard was analyzed. If the surrogate recovery of the check standards ranged between 85 and 115%, TOX analyses for the day were complete. If the laboratory blank exceeded its maximum allowable interference concentration, or if the check standard surrogate recovery exceeded the specified range, all 7 ICR treatment study samples would be reanalyzed (if possible) the next day. The very last sample analyzed each day was always a check standard.

Trihalomethanes (THMs)

Calibration for THM samples, measured using EPA Method 502.2, was performed on a daily basis. Calibration consisted of one laboratory blank injection followed by injection of a low, medium and high level calibration standard. Prepared separately from the calibration standards, low, medium and high level check standards were analyzed to verify calibration. Low, medium and high level (calibration and check) standard concentrations were 1, 20 and 40 µg/L respectively.

After all calibration standards had been analyzed, a low-level calibration check standard was analyzed. One injection of this standard was analyzed and used to compute a surrogate recovery (detector response/average calibration curve detector response). For the low-level calibration check standard, the surrogate recovery was required to be between 50 and 150%. After the low level check standard had successfully verified the instrument calibration, 10 ICR treatment study samples were then analyzed as laboratory duplicates.

Once 10 ICR treatment study samples had been analyzed, a medium or high level check standard was then analyzed. The surrogate recovery of the medium or high level check standard was required to be 80 to 120%. If the surrogate recovery of the medium or high-level check standard did not meet this criteria, the previous 10 samples would be reanalyzed. This procedure of analyzing 10 ICR treatment study samples followed by a check standard was repeated until 30 treatment study samples had been analyzed. After analyzing 30 treatment study samples, a medium or high level check standard was analyzed, which marked the end of analyses under the current calibration. While this procedure was understood as practice, on no occasion did the laboratory ever analyze more than 30 ICR treatment study samples.

Haloacetic Acids (HAAs)

Calibration for HAA samples, measured using Standard Method 6251 B, was performed on a daily basis. Calibration consisted of one laboratory blank injection followed by injection of a low, medium and high level calibration standard. Prepared separately from the calibration standards, low, medium and high level check standards were analyzed to verify calibration. Low, medium and high level (calibration and check) standard concentrations were 1 (2 for MCAA), 20 and 40 µg/L respectively. Although BDCAA, CDBAA and TBAA are not recognized as accepted analytes by the ICR using Standard

Method 6251 B, they were reported by the KCMWSL with an MDL equal to 1 µg/L. These HAAs were included due to the fact that the chemists at the KCMWSL had great success in achieving superior surrogate recoveries for these compounds over a wide range of concentrations including very low concentrations (lower than the MCL).

After all calibration standards had been analyzed, a low-level calibration check standard was analyzed. One injection of this standard was analyzed and used to compute a surrogate recovery (detector response/average calibration curve detector response). For the low-level calibration check standard, the surrogate recovery was required to be between 50 and 150%. After the low level check standard had successfully verified the instrument calibration, 10 ICR treatment study samples were then analyzed as laboratory duplicates.

Once 10 ICR treatment study samples had been analyzed, a medium or high level check standard was then analyzed. The surrogate recovery of the medium or high level check standard was required to be 80 to 120%. If the surrogate recovery of the medium or high-level check standard did not meet these criteria, the previous 10 samples would be reanalyzed. This procedure of analyzing 10 ICR treatment study samples followed by a check standard was repeated until 30 treatment study samples had been analyzed. After analyzing 30 treatment study samples, a medium or high level check standard was analyzed, which marked the end of analyses under the current calibration. While this procedure was understood as practice, on no occasion did the laboratory ever analyze more than 30 ICR treatment study samples.

Performance Evaluation (PE) Study Results

In accordance with the ICR, the KCMWSL conducted PE studies to verify that the Laboratory was performing in a manner that was acceptable to the EPA for the purpose of reporting ICR monitoring results. Table 21 summarizes the results of the PE study conducted by the KCMWSL as the analyses pertain to the ICR treatment study. While the results in these PE studies indicate that the laboratory was performing in a satisfactory manner, the PE studies were conducted as an internal challenge only. All ICR monitoring samples (treatment study samples excluded) were shipped out and analyzed by Montgomery Watson Laboratories.

Table 21. US EPA ICR Performance Evaluation (PE) Study Summary

		Method Number	True Value	Measured Value	% Recovery
PE Study #6 2/20/98	UV ₂₅₄ (cm ⁻¹)	5910	0.435	0.415	95%
	TOC (mg/L)	5310C	4.70	4.91	104%
	TOX (µg Cl ⁻ /L)	5320B	50.5	NR	
	Bromide (µg/L)	EPA 300.0	169	NR	
	Haloacetic Acids	6251B			
	MCAA (µg/L)		6.05	9.40	155%
	MBAA (µg/L)		11.9	15.40	129%
	DCAA (µg/L)		12.0	9.68	81%
	TCAA (µg/L)		9.03	6.86	76%
	BCAA (µg/L)		5.96	5.57	93%
	DBAA (µg/L)		15.1	12.70	84%
	Trihalomethanes	EPA 502.2			
	Chloroform (µg/L)		36.5	34.70	95%
	BDCM (µg/L)		32.2	34.50	107%
	CDBM (µg/L)		14.7	14.60	99%
	Bromoform(µg/L)		26.5	23.80	90%
PE Study #7 4/29/98	UV ₂₅₄	5910	0.361	0.347	96%
	TOC	5310C	1.22	1.38	113%
	TOX	5320B	188	173	92%
	Bromide	EPA 300.0	448	NR	
	Haloacetic Acids	6251B			
	MCAA		5.94	5.32	90%
	MBAA		11.1	9.41	85%
	DCAA		24.0	17.40	73%
	TCAA		15.0	12.70	85%
	BCAA		12.1	9.77	81%
	DBAA		14.0	12.30	88%
	Trihalomethanes	EPA 502.2			
	Chloroform		17.0	16.70	98%
	BDCM		11.0	9.90	90%
	CDBM		28.1	27.90	99%
	Bromoform		18.2	17.00	93%
PE Study #8 8/22/98	UV ₂₅₄	5910	0.072	0.070	97%
	TOC	5310C	2.62	3.10	118%
	TOX	5320B	80.3	73.0	91%
	Bromide	EPA 300.0	325	NR	
	Haloacetic Acids	6251B			
	MCAA		13.0	9.99	77%
	MBAA		16.0	12.50	78%
	DCAA		14.2	11.10	78%

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