3M™ Liqui-Cel™ EXF Series Membrane Contactors

Cleaning and Storage Guidelines

3M.com/Liqui-Cel

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To insure you are evaluating the most current information on our products, please review the English versions of our literature on our website. The English documents are the correct documents to be referenced.

Our 3M™ Liqui-Cel™ EXF-Series 2.5×8, 4×13, 4×28, 6×28, 8×20, 8×40, 8×80 and 10×28 Industrial Series Membrane Contactors are manufactured with Sound Engineering Practice where no CE mark is required due to their small size and low pressures and volumes. Our 10×28 high purity, 14×28 and 14×40 products have a product classification – Category 1 per PED 2014/68/EU and do carry a CE mark.

TABLES

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I. Safety Information

⚠ Please read and follow all safety information, warnings, and instructions in this manual. Failure to follow all product warnings and instructions could cause serious injury and property damage. Retain these instructions for future reference.

II. Intended and Restricted Use(s):

3M™ Liqui-Cel™ Membrane Contactors are intended to remove dissolved gases and bubbles from compatible liquids or to add gases to a liquid stream. Liqui-Cel products are for use in industrial separation applications of industrial fluids only, in accordance with the applicable product instructions and specifications. Certain limited Liqui-Cel products are also intended for use in Food and Beverage (F&B) applications. Refer to the specific Liqui-Cel product's data sheet to determine whether it includes a F&B designation and can be used for such applications.

3M Liqui-Cel Membrane Contactors may further be used to create the purified water that is processed by either distillation or some other equivalent process to create Water For Injection (WFI). Liqui-Cel products should not be used during the production of the WFI after the distillation or other equivalent process.

Since there are many factors that can affect a product's use, the customer and user remains responsible for determining whether the 3M product is suitable and appropriate for the user's specific application, including user conducting an appropriate risk assessment and evaluating the 3M product in user's application.

Restrictions on Use:

3M advises against the use of these 3M products in any application other than the stated intended use(s), since other applications have not been evaluated by 3M and may result in an unsafe or unintended condition. Do not use in any manner whereby the 3M product, or any extractable or leachable from the 3M product, may become part of or remains in a medical device, drug, cosmetic, or food or drink additive or supplement; or in applications involving life-sustaining medical applications or prolonged contact with internal bodily fluids or tissues. If you are considering using this 3M product for a restricted use, you must first contact 3M with information about your proposed application to request prior written authorization for supply for such use.

EXPLANATION OF SIGNAL WORD CONSEQUENCES	
⚠ **WARNING**	Indicates a hazardous situation which, if not avoided, could result in serious injury or death.
⚠ **CAUTION**	Indicates a hazardous situation which, if not avoided, could result in minor or moderate injury.
NOTICE	Indicates a situation which, if not avoided, could result in product or system damage.

⚠ WARNING

To reduce the risks associated with bursting and/or exposure to chemicals and contactor damage:

- To prevent buildup of pressure inside contactor, do not block or valve off all gas/vacuum ports during operation. If contactor is not in use but still full of process fluids, ensure the gas/vacuum side is open to atmospheric pressure.
- Do not exceed maximum operating pressure or temperature limits. Cleaning should be conducted at the minimal temperature and pressure required to clean the contactor, never exceeding the maximum operating pressure and temperature limits of the contactor.
- Implement workplace safety risk controls according to local applicable laws and regulations.
- All plumbing should be done in accordance with local regulations and code.
- Always make sure to verify proper connections within the contactor system.
- Never modify or alter the membrane contactor. Only 3M or parties authorized in writing may make changes/repairs to the equipment.
- Inspect contactor prior to installation or cleaning. Only use replacement parts supplied by 3M for this product.
- Inspect contactor to ensure no leaking, cracking, or other signs of damage on membrane contactor or gaskets.
- Ensure chemically compatible materials of construction are used within system.

To reduce the risks associated with asphyxiation related to discharge of sweep gas or vacuum pump exhaust into an enclosed space:

- Ensure system is properly installed, including adequate ventilation per local building codes and regulations.

To reduce the risks associated with explosion:

- Do not introduce dangerous (explosive, flammable, toxic or oxidizing) liquids or gases to the contactor or the system.
- Over-pressurization of liquid and gas should be prevented by the installation of proper pressure relief valves/safety systems.

To reduce the risks associated with impact, lifting or moving:

- Do not attempt to move the system while it contains liquid , or while in operation.
- Use appropriately rated lifting equipment for lifting or moving. Review the product datasheet or operating guide for weights.
- Always ensure the system is stable, level, and properly secured. Be sure the system cannot tip, roll, fall, slide or make any movement that may cause injury, damage to the unit, or damage to other system components. If needed, use shims to level the system.

⚠ CAUTION

To reduce the risks associated with hot surfaces:

- Do not touch the membrane contactor or liquid lines during operation. Surfaces may be hot.

To reduce the risks associated with environmental contamination:

- Exhaust gas should be vented in a safe manner and according to local regulations.

To reduce the risks associated with damaging the membrane contactor:

- Ensure contactor is properly aligned with piping, and flanges are adequately tightened after cleaning. Always conduct system checks in accordance with installation instructions and facility policies prior to operation.
- Ensure proper draining and flushing of contactor before maintenance, service, or shipping of contactors.

NOTICE

To reduce the risk of membrane contactor or system damage:

- Care must be taken not to drop, hit or impact the contactor.
- If the membrane contactor is used with air sweep, then the temperature should not exceed 35°C (95°F). For membrane contactors used with vacuum only this statement does not apply.
- For all membrane constactors, lumen side pressure should never exceed shell side pressure during cleaning and operation.
- 3M Liqui-Cel Membrane Contactors should be stored dry and in a sealed plastic bag or shrink wrap material to help prevent the introduction of contaminants into the contactor.
- Store 3M Liqui-Cel Membrane Contactors dry at temperatures < 49°C (120°F), but preferably at lower temperature such as <35°C (95°F) to not risk reduced lifetime. Membrane contactors should always be stored above freezing temperatures, and if stored at low temperature, they should be allowed to equilibrate to room temperature before use.
- 3M Liqui-Cel Membrane Contactors should be stored in their original box, or other opaque box, and should not be installed where they are exposed to direct sunlight.
- All plastic port extensions should be supported to prevent bending of extensions under excessive piping loads.

- Do not allow membrane contactors containing microporous polypropylene membranes to come into contact with surfactants, oils or organic solvents, such as detergent solutions, enzymatic cleaners, pure alcohols, glycol, acetone, etc., to reduce the risk of membrane wet out.
- To protect the membrane contactors, prefiltration equipment should be inspected and maintained in accordance to the 3M™ Liqui-Cel™ Membrane Contactors Inlet Water & Sweep Gas Operating Guidelines, in the Technical Resources section at 3m.com/Liqui-Cel.
- Suspended solids, biological contaminants, or the precipitation of soluble salts on the membrane surface may lead to membrane plugging.
- Liquid pressure should always exceed gas pressure inside the membrane contactor.
- Filtered, de-chlorinated, and deionized water is recommended for mixing cleaning solutions. If a pH shift occurs water containing sparingly soluble compounds of Ca, Mg, Fe, Al, etc. and silica (SiO2) etc. could precipitate from the solution and block or damage the membrane. Ensure that your water is clean of these compounds.
- At end of life, dispose of the membrane contactor or cartridges in accordance with all applicable local and government regulations.
- Cumulative exposure of the membrane to oxidants, such as ozone, chlorine, hydrogen peroxide, peracetic acid, etc., should be restricted to reduce the risk of membrane oxidation.
- Avoid water hammer (sudden pressure spikes).

Note regarding Hazards from Chemicals

The chemicals that User selects to use in connection with the membrane can present their own hazards. User should follow all safety information and related requirements provided by the chemical supplier and applicable regulations, as well conduct User's own workplace safety, hazard and application assessment. This document cannot and does not address all safety and/or safe handling requirements that different chemicals could present. User is responsible for ensuring that chemicals are only used by persons familiar with their use and hazards (for example, personnel who have received hazardous material training), and who have the appropriate protective equipment as specified in their organization's safety program and the chemical's material safety datasheet (SDS). User assumes all responsibility for the suitability and fitness for use as well as for the protection of the environment and for health and safety involving such chemicals.

III. Intent of Document

A membrane contactor may lose its performance due to chemical or biological fouling or due to mineral scaling. There are many different types of contaminants that may adhere to the membrane. Membrane performance can also decrease or be completely lost because of loss of its hydrophobic functionality which can happen when membrane "wets out" or gets severely oxidized.

The procedures described in the following sections are guidelines only. Actual procedures used by customer may vary depending on the condition of membranes and should be developed in consultation with a technical representative of the contactor manufacturer. Note, however, that cleaning may not always restore system performance completely, or contactor may have exceeded their lifetime and contactor replacement will be required instead of cleaning.

There are several levels and types of cleaning: routine/preventative, moderate fouling treatment, severe fouling treatment, mineral scale removal, and particle blockage. In all procedures described below, it is assumed that cleaning is done only on the water-contact side of the membrane. In some cases, the gas-contact side of the membrane may also have to be cleaned. Consult with a technical representative of the contactor manufacturer for gas-contact side cleaning procedure.

IMPORTANT NOTES FOR CLEANING SOLUTION SELECTION:

Do NOT use strong oxidizing agents.

Do NOT use any chemicals that contain detergents or surfactants.

Surfactants may allow liquids to pass through the membrane. This phenomenon is called break-through or wet-out. The membrane can usually be restored to a hydrophobic state by rinsing the detergent from the contactor and then drying the contactor, but this is a time-consuming process.

There are many different types of contaminants that may adhere to the membrane. The cleaning procedure, which covers chemical cleaning agents, concentrations, time and flow rates, will be specific to each system. Cleaning procedures contained in this document should be treated as guidelines only and may require modification to suit individual systems and applications. For specific system and application cleaning procedures, contact your 3M representative. Note: These cleaning instructions are for EXF series products but can be partially used for SP or MM series products.

IV. Cleaning Parameters

There are four parameters that affect the cleaning process:

- Time (duration and frequency)
- Temperature
- Mechanical shearing on membrane surface by flowing liquids
- Chemical type (caustic, acid, alcohol, etc.) and chemical strength

Changing any one of these parameters can affect the others. Therefore, it is important to develop a specific cleaning procedure for your application. These guidelines will help you develop your specific cleaning procedure. We recommend starting with cleaning chemicals that are generally used within your industry but use oxidizer and surfactant free chemicals.

The performance of the contactor before cleaning should be monitored to establish its baseline performance. This baseline performance can be compared to the performance of the contactor after cleaning. Other considerations for establishing a cleaning procedure for your system and application are:

- Experimentation with time (frequency and duration), temperature, chemical concentration and cleaning liquid flow rate will help you determine the optimal procedure for cleaning the membrane contactor.
- Refer to the 3M Liqui-Cel EXF Series Membrane Contactor product data sheet for maximum temperature and pressure ratings. Take into account the rise in temperature that occurs during a physical or chemical reaction such as mixing of water with caustic soda or sulfuric acid, or mixing of acids and bases or from pumping.
- An aggressive cleaning procedure may clean the contactor in a shorter time period, but can also reduce the contactor service life.

Generally, the required cleaning frequency can be determined by monitoring decrease in system gas transfer efficiency or increase in pressure drop in liquid or gas phases. Contactors are normally cleaned when one of the following occurs: (1) effluent dissolved oxygen concentration from system approaches an upper control limit set by customer, and/or (2) The water pressure drop (DP) in the system exceeds an upper control limit set by customer.

V. Cleaning Solution Flow Rate Guidelines

Table 1 lists recommended cleaning solution flow rates for different contactors. It has two columns for flow rate: one for normal shell side cleaning and the other for cleaning lumen side of contactor, if necessary.

Table 1: Cleaning Solution Flow Rate Guidelines

Contactor Size	Shell Side Flow	Lumen Side Flow
2.5×8	1 - 2 gpm (0.23 - 0.45 m3/h)	< 0.5 gpm (<0.11 m3/h)
4×13	4 - 10 gpm (0.91 - 2.27 m3/h)	4 - 6 gpm (0.91 - 1.36 m3/h)
4×28, 6×28, 8×20	4 - 10 gpm (0.91 - 2.27 m3/h)	3 - 7 gpm (0.68 - 1.59 m3/h)
8×40, 8×80	20 - 30 gpm (4.54 - 6.81 m3/h)	5 - 10 gpm (1.14 - 2.27 m3/h)
10×28	20 - 40 gpm (4.54 - 9.08 m3/h)	10 - 20 gpm (2.27 - 4.54 m3/h)
14×28, 14×40	50 - 60 gpm (11.36 - 13.63 m3/h)	10 - 20 gpm (2.27 - 4.54 m3/h)

We recommend using de-ionized or softened water when preparing cleaning solutions for rinsing after cleaning. If deionized water is not available, filtered and de-chlorinated water, that does not contain sparingly soluble compounds of Ca, Mg, Fe, Al, etc. and silica (SiO2), can be used. It is important to know the water chemistry because these compounds can precipitate onto the membrane when there is a pH shift in the water.

It is necessary to apply a backpressure to ensure that the system is completely full of cleaning solution before cleaning. To increase the cleaning solution backpressure, partially close the outlet flow valve. During cleaning maintaining backpressure is not required. Flow rates listed in Table 1 are for single units and guidelines only. Depending on nature of the fouling, flow rates should be adjusted accordingly.

VI. Recommended Cleaning Procedure for Routine/ Preventative Maintenance for Fouling

Table 2 below offers general recommendations of routine cleaning as preventative maintenance of 3M™ Liqui-Cel™ Membrane Contactors exposed to normal city water or other waters, such as ground water, surface water, or seawater. Suggested cleaning is weekly or monthly, depending on water quality. Figure 1 shows typical flow diagram for cleaning. If possible, reverse the direction of the water flow (back-flow) during cleaning.

Before beginning cleaning, verify the following parameters are under control and therefore are NOT the reasons for performance excursion: (a) water temperature is no more than 2°C lower than design value; (b) vacuum level in contactor is no more than 20% above system design value (such as obtained from GasCAD); (c) Nitrogen flow rate and purity level are at design value, and (d) Feed water flow rate is no more than 10% above design value.

Table 2: Recommended Cleaning Procedure for Routine and Preventative Maintenance

Use RO Permeate or Softened Water to prepare Cleaning Solutions or for flushing. Do not use commercial cleaners with surfactants.

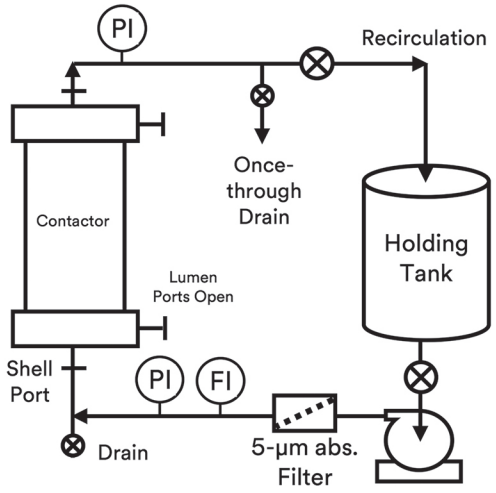
Step	Process	Description	Suggested Duration
1	Water flush	Flush contactor once-through with 5-micron (abs.) filtered water, at ambient temperature	5-15 min.
2	Alkaline cleaning	Recirculate solution of NaOH or KOH at pH 12, prepared with 5-micron (abs.) filtered water, on shell side of contactor, at ambient to 40°C temperature; faster and more effective cleaning at warmer temperature	30 min. to 1 hr.; soak time optional
3	Drain Contactor and flush with water (optional) for 5 min.		
4	Acid cleaning	Recirculate solution of citric, nitric, hydrochloric, or phosphoric acid at pH 1, prepared with 5-micron (abs.) filtered water, on shell side of contactor, at ambient temperature	30 min. to 1 hr.; soak time optional
5	Drain Contactor		
6	Water flush	Flush contactor once-through with 5-micron (abs.) filtered water, at ambient temperature	Till effluent pH is acceptable
7	Drain Contactor		

Cleaning Tips:

1. It is recommended to begin the cleaning cycle using caustic to remove biofouling. Monitor pH and add more caustic if pH decreases >0.5 pH units during cleaning. Replace caustic solution with fresh solution if it is strongly colored or turbid.

2. Acid cleaning will remove mineral scaling and will also help flush out and neutralize the caustic. Monitor pH and add more acid if pH increases >0.5 pH units during cleaning. Replace acid solution with fresh solution if it is strongly colored or turbid.

Figure 1: Flow Schematic for Cleaning for Routine Maintenance or for Moderate Fouling



VII. Recommended Cleaning Procedure for Moderate Fouling

If the above procedure is not effective in maintaining system performance, then increase the chemical concentrations or clean for longer time, as suggested in Table 3 below for moderate fouling.

Table 3: Recommended Cleaning Procedure for Moderate Fouling

Step	Process	Description	Suggested Duration
1	Water flush	Flush contactor once-through with 5-micron (abs.) filtered water, at ambient temperature	5-15 min.
2	Alkaline cleaning	Recirculate 2-6 wt.% caustic (NaOH or KOH) solution prepared with 5-micron (abs.) filtered water; suggested cleaning solution temperature ambient to 122°F (50°C)	30 min. to 4 hrs.
3	Drain Contactor		
4	Acid cleaning	Recirculate 10 wt.% citric acid, or 1-6 wt.% nitric, phosphoric, hydrochloric, or mixed acid solution prepared with 5-micron (abs.) filtered water; suggested cleaning solution temperature ambient	30 min. to 2 hrs.
5	Drain Contactor		
6	Water flush	Flush contactor once-through with clean water, filtered to 5-micron absolute, at ambient temperature	Till effluent pH is acceptable
7	Drain Contactor		
8	Gas purge (optional) to flush out lumen side	Blow air or an inert gas through lumen side of contactor at maximum available flow rate without exceeding flow or pressure rating	Minimum 15 min. or till no water drips out of gas exit port

VIII. Recommended Cleaning Procedure for Severe fouling

A surface cleaning of the wetted side of the membrane is usually enough, per the cleaning procedures described above. If performance is not restored after attempting routine and moderate cleaning procedures, fouling may have penetrated the membrane pore structure. At this point the procedure for severe fouling described in Table 4 and Figure 2 might be needed. This procedure requires that the membrane be 'wetted out' before any cleaning is done. Wet-out refers to a condition of the membrane when it lost its hydrophobic property, thus allowing liquids to pass through the pore structure. Wet-out can also occur when the membrane is exposed to surfactants/detergents, to organic solvents, or to protein-containing liquids such as beer, wine or fruit juice. Removing deposits that have penetrated the membrane pore structure will restore the membrane to a hydrophobic state. This procedure uses an alcohol-water solution and requires a final extensive drying step. Since this procedure requires resources that may not always be available in the field, it should be used only when it absolutely cannot be avoided. The drying step is critical for removing any remaining liquid from the pore structure. If liquid remains in the pore structure, any liquids introduced into the contactor during operation will pass through the membrane. Therefore, the contactor must be dried before it is put back into service. Contact a 3M representative to learn more about contract cleaning services available in our facility for your convenience.

Figure 2: Recommended Flow Schematic - Cleaning for Severe Fouling

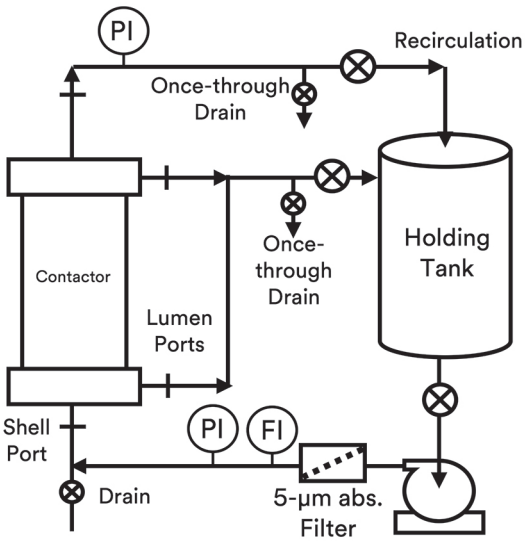


Table 4: Recommended Cleaning Procedure for Severe Fouling

Step	Process	Description	Suggested Duration
1	Water flush	Flush contactor once-through with clean water, filtered to 5-micron absolute, at ambient temperature	5-15 min.
2	Drain Contactor		
3	Membrane wet-out	Pressurize shell side of contactor with 50 vol% Isopropanol-water or ethanol-water solution, not exceeding max pressure rating. Allow alcohol solution to come out of both lumen ports, then cap off lumen ports and let contactor soak	Min. 1 hr.
4	Alkaline cleaning	Recirculate 2-6 wt.% caustic (NaOH or KOH) solution prepared with 5-micron (abs.) filtered water; suggested cleaning solution temperature 86-122°F (30-50°C)	1 to 4 hrs.
5	Drain Contactor		
6	Acid cleaning	Recirculate 10 wt.% citric acid, or 1-6 wt.% nitric, phosphoric, hydrochloric, or mixed acid solution prepared with 5-micron (abs.) filtered water; suggested cleaning solution temperature ambient	1 to 2 hrs.
7	Drain Contactor		
8	Water flush	Flush contactor once-through with clean water, filtered to 5-micron absolute, at ambient temperature	Until flush water effluent pH is acceptable
9	Drain Contactor		
10	Gas purge (optional) to flush out lumen side	Blow air or an inert gas through lumen side of contactor at maximum available flow rate without exceeding flow or pressure rating	Min. 15 min. or till no water drip out of gas exit port
11	Drying	Dry contactor thoroughly using procedure described separately later in this guide. Warm gas would dry contactor much faster. Inert gas is preferred for drying. Clean and dry air could be used but air temperature should not exceed 122°F (50°C)	Refer to section on drying procedure later
12	Integrity test membrane and contactor (described in later section)		

IX. Cleaning Procedure for Mineral Scaling Removal

If the performance of the contactor decreases and the inlet water source is not treated to remove minerals, such as silica or calcium carbonate, it is likely that a layer of mineral scale has formed on the water-contact side of the membrane. An acid cleaning followed by a water flush should restore the performance in case of carbonate scaling. Acid cleaning procedure is a part of the moderate cleaning procedure. Follow procedure listed in Table 2 and Figure 1 but skip steps 2 and 3 (caustic cleaning and flush). The contactor does not need to be dried after this procedure.

If the scaling is due to silica precipitation also, use the recommended cleaning procedure for moderate biological fouling (Table 3) but use higher caustic concentration (up to 6 wt.%) and increase the temperature to maximum operating limit of contactor. If possible, the cleaning solution flow rate should be like the process water flow rate used during normal operation. The contactor does not need to be dried after these procedures.

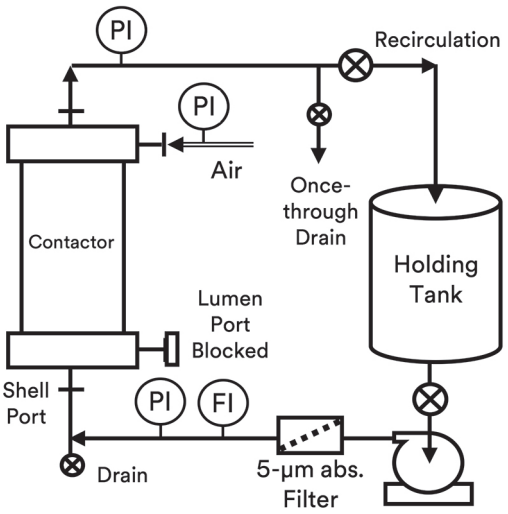
X. Cleaning Procedure for Particle Blockage in Contactor

NOTES: This procedure is intended for 3M™ Liqui-Cel™ EXF Series Membrane Contactors. For SP series, only follow steps 1 & 2.

Refer to Figure 3 below.

1. Follow the steps described earlier in sections V and VI with the following exceptions:
2. Backflush the cleaning solutions (i.e. introduce the cleaning solutions on the shell side in the direction opposite of the normal operating flow direction). Use maximum liquid inlet pressure of 15 to 30 psig (1 to 2 bar).
3. Once the cleaning solution is flowing into the contactor, valve off or cap one gas port. Then slowly introduce clean, dry, and oil-free compressed air into the other gas port. Avoid sudden spike in gas pressure. Air inlet pressure should not exceed the max lumen pressure.
4. Regulate the air pressure 5-10 psig (0.4 to 0.8 bar) GREATER than the liquid pressure, such that the air will bubble vigorously into the cleaning solution. This statement is only valid for air bubbling when attempting to remove particle contamination. During normal operation the liquid pressure should always exceed the gas pressure.
5. At the end of the cleaning procedure, shut off the air supply first, then slowly open the gas outlet port, then stop liquid flow.

Figure 3: Recommended Flow Schematic - Cleaning for Shell Side Particle Blockage



XI. Contactor Sanitization

Sanitization is intended to reduce the incidence of microbiological, chemical and physical hazards in plants where contactors are used by reducing pathogens, yeast and other biological matters. After the contactor system is cleaned using any of the appropriate cleaning procedures outlined in sections V, VI, and VII, the last operation prior to re-start the system is to sanitize the equipment. In some operations, sanitization procedure is practiced on a regular basis even if no chemical cleaning is done.

3M Liqui-Cel Membrane Contactors can be sanitized by two methods

- Chemical sanitization
- Hot Water (85°C, 185°F) sanitization

DO NOT USE steam

DO NOT USE beta or gamma radiation sterilization methods

Chemical Sanitization

Table 5 shows the normally recommended concentration and exposure times for several sanitizing chemicals. Since these chemicals slowly oxidize the membrane during every use, their use should be limited.

If a daily sanitization routine is needed, our only recommendation is caustic or hot water. Caustic concentration and condition should be as described before for routine/preventative procedure.

Table 5: Recommended Chemical Sanitization Guidelines (room temperature)

Chemical	Maximum Concentration	Time Duration	Frequency
Chlorine*	100 ppm	15 min.	Once/month
Hydrogen Peroxide	3%	15 min.	Once/month
Peracetic Acid	50 ppm	15 min.	Once/month

* Use Hypochlorite at high pH only; Avoid Chlorine Dioxide

Hot Water Sanitization (HWS)

Hot water sanitization of contactors, with or without chemical cleaning, should be done following the guidelines below. Refer to Figure 4 for a schematic flow diagram.

Hot Water Sanitization is commonly used to combat microbial growth in the pharmaceutical and food and beverage industries. Performed frequently as a prevention strategy, hot water sanitization is a requirement for high purity water (HPW) for United States Pharmacopeia (USP) and European Pharmacopoeia (Ph. Eur.).

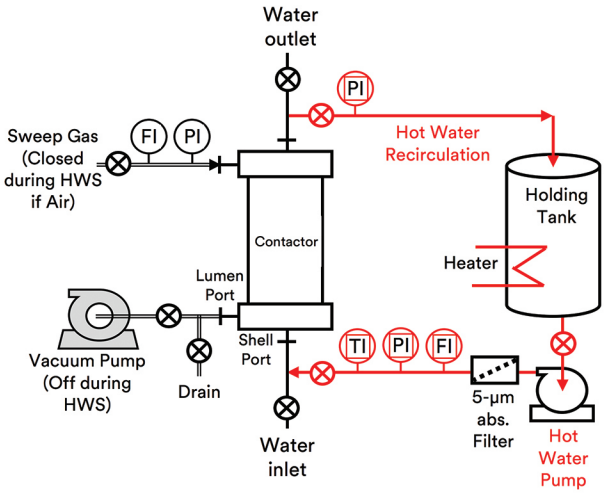
Only 3M Liqui-Cel Membrane Contactors available in SS housing should be used for HWS. They are EXF-4×13 Series, EXF-4×28 Series, EXF-8×20 Series, and EXF-10×28 Series. Table 6 below describes the recommended process condition for HWS.

Table 6: Recommended Process Requirements During Hot Water Sanitization

Water temperature	Max 85°C (185°F)	
Water pressure	Max 2 bar (29 psi)	
Heating/cooling rate	2-3°C per min. (3-5°F per min.)	
Sanitization time	30-60 min after reaching sanitization temperature	
Min. recommended inert gas flow rate (optional) during HWS	4×13	1 lpm
	4×28	1 lpm
	8×20	2 lpm
	10×28	2 lpm
Vacuum/Air sweep	Must be switched off during HWS	

Before starting the HWS process, note the operating mode: vacuum, sweep mode or combo mode (sweep gas with vacuum). If the membrane contactors are being operated in vacuum or combo mode, the vacuum must be turned off before starting the HWS process. If contactors are operating in combo or sweep mode using air, then the sweep gas supply must also be turned off to prevent oxygen in the air oxidizing the membrane at high temperatures. If inert sweep gases such as nitrogen or carbon dioxide are used, sweep gas flow should be maintained at least at a minimum flow rate. For example, a minimum gas flow of 2 L/min for each EXF-10×28 contactor is recommended during HWS process. During hot HWS sanitation, isolate the contactor(s) from process lines and connect the contactor to the hot water loop (see Figure 5). Check that any other sweep gas being maintained is flowing at minimum recommended flow rate for your product(s). Leave the gas outlet port open during HWS.

Figure 4: Recommended Flow Schematic for Hot Water Sanitization



Follow the next steps:

1. Start circulating water through the contactor. As the water circulates, heat the water slowly by increasing the temperature at the rate of 2-3°C (3.6-5.4°F) per minute until 80-85°C (176-185°F) is reached. This maximum temperature should be maintained for 30 – 60 minutes. The hot water temperature should not exceed 85°C during this process.

2. During hot water recirculation, it is normal to see a slow drip of condensed water at the lumen outlet port. This is from condensation of hot water vapor that passes through membrane pores during HWS onto the lumenside. The condensation rate depends on the liquid temperature.

3. The flow rate of hot water through the contactor is not critical, but a minimum flow rate is recommended. For example, 50 gpm for each 10×28 contactor. Do not exceed the contactor's maximum allowable water pressure of 2 bar (30 psig).

4. The final step of the hot HWS process is to slowly cool the water at the rate of 2-3°C (3.6-5.4°F) per minute. After cool-down, restart or re-adjust the sweep gas flow rate, and then re- apply the vacuum. See Figure 4.

5. When sweep gas and/or a vacuum are applied after a hot HWS procedure, a large amount of liquid condensate water may come out of the vacuum port. This normal occurrence is due to the lumen side filling with water vapor condensate. Unless you are using a liquid-ring vacuum pump a liquid water trap should be installed in the vacuum line to collect this water and protect the vacuum pump.

To clear the lumenside of any water vapor after cooling down, it is advisable to purge with a sweep gas for 5-10 minutes at <0.5 bar (<7 psi) inlet gas pressure before re-applying the vacuum to the contactor. However, if an inert gas is not available, the vacuum should only be reapplied after the cartridge has cooled down. Additionally, we recommend mounting the contactors vertically. If a vacuum is used on the system, the vacuum should be pulled from the gas port on the bottom of the contactor. This and the downward sweep gas flow facilitate the draining of condensed water from the lumenside of vertically mounted contactors.

Lumenside (gas-side) Cleaning

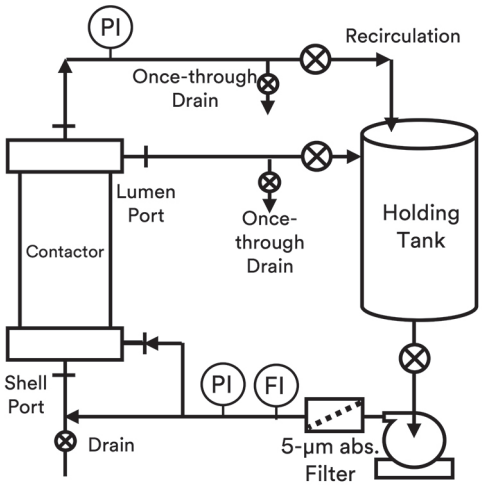
Cleaning guidelines addressed so far in this document assume shellside flow since that is the liquid-contact side of the membrane that is most prone to fouling. Most system designs where the operations run 24/7, the gas side of the membrane is under inert gas conditions and biological growth is usually not present. However, whenever the operation is not continuous, and the contactor is full of static water for long time, special precautions should be taken to avoid or remove biological growth.

Biological growth may also occur due to liquid leaking through broken fibers, which may occur over time. Process in the beverage market, like soft drink, beer, wine or juice processing may schedule lumenside cleaning more frequently.

If lumenside cleaning is necessary, follow the recommended flow guidelines in the rightmost column of Table 1. Lower liquid flowrates are needed on lumen side relative to shell side due to the pressure drop through the fiber lumens. See Figure 5. for a process schematic diagram for simultaneous shell side and lumen side cleaning.

Note that during lumenside cleaning the liquid solution should be introduced slowly to avoid sudden over-pressurization and inlet liquid pressure should not exceed 30-45 psig (2-3 bar).

Figure 5: Process Flow Schematic for combined shell and lumen side cleaning



Biocide Guidelines

The addition of biocides to the feed water is intended to control the incidence of microbiological growth on the membrane surface. For 3M™ Liqui-Cel™ Membrane Contactors non-oxidizing biocides are recommended to minimize membrane oxidation and membrane lifetime reduction. Some biocides that have been tested on Liqui-Cel contactors over limited time and did not show detrimental effect on membrane are Quaternary Ammonium, DBNPA, Glutaraldehyde, and Hydroxymethyl phosphonium sulfate.

XII. Membrane Drying

The drying process involves two steps:

A. Bulk Water Removal

B. Final Drying

Bulk Water Removal quickly removes water from the contactor prior to passing the drying gas through. Removing excess liquid water beforehand makes the final drying much faster. The purpose of Final Drying is to evaporate any remaining water from the contactor. Dry air, nitrogen, and carbon dioxide gas can be used to facilitate drying. Tables 7 and 8 provide reference points for flow rates and drying times.

⚠ WARNING

To reduce the risks associated with asphyxiation related to discharge of sweep gas or vacuum pump exhaust into an enclosed space:

- Ensure system is properly installed, including adequate ventilation per local building codes and regulations.

⚠ CAUTION

To reduce the risks associated with hot surfaces:

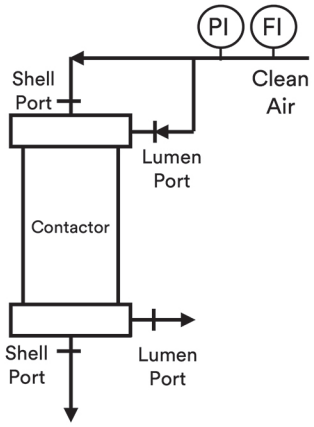
- Do not touch the membrane contactor or liquid lines during operation. Surfaces may be hot.

Vacuum at ambient temperature is not suggested as a substitute for warm gas drying as residual water may remain even after several hours.

A. Bulk Water Removal

To reduce the drying time after cleaning, it is recommended that bulk water be removed by purging the contactor with gas (normally air) at room temperature for a short period of time (typically, less than one hour). Introduce gas through the top shellside and lumenside ports. See Figure 6 for schematic. Use clean, dry and filtered (preferably 0.2 micron) gas at the flow rates shown in Table 7. Keep the lower lumen and shellside ports open.

Figure 6: Recommended Flow Schematic for Bulk Water Removal



Stop the gas flow when the water discharge rate decreases to a few drips.

Table 7: Recommended Air Flow Rate Range for Bulk Water Removal

3M™ Liqui-Cel™ EXF Series Membrane Contactor Size	Recommended Air Flow Rate for Bulk Water Removal*
2.5×8	1-3 SCFM (1.7-5.1 m3/hr)
4×13	5-10 SCFM (8.5-17 m3/hr)
4×28, 6×28, 8×20	10-20 SCFM (17-34 m3/hr)
8×40	20-40 SCFM (34-68 m3/hr)
8×80	40-80 SCFM (68-136 m3/hr)
10×28	20-50 SCFM (34-85 m3/hr)
14×28	30-60 SCFM (51-102 m3/hr)
14×40	50-100 SCFM (85-170 m3/hr)

* Recommended max air pressure 10 psig (0.7 bar)

Final Drying

The final drying step involves flowing a clean, dry, oil-free filtered (preferably 0.2 micron) gas into the top shellside port. Using a warm gas will reduce drying time. Nitrogen is preferred in the final drying step as hot air can shorten the membrane life. See Figure 7 for schematic and Table 8 for estimated flow rate and drying time guidelines. After a few minutes, close lower shell side port and keep it closed during this procedure.

Figure 7: Recommended Flow Schematic for Final Drying Step

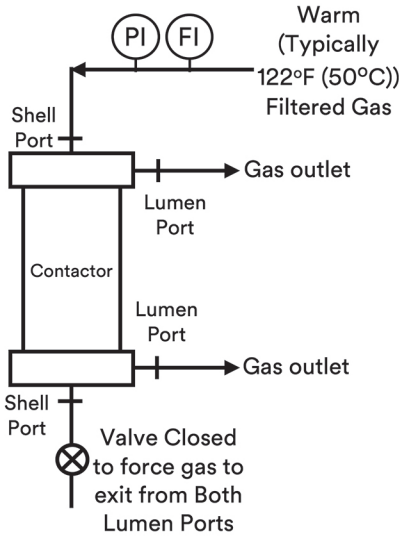


Table 8: Recommended Air Flow Rate and Duration for Final Drying

3M™ Liqui-Cel™ EXF Series Membrane Contactor Size	Recommended Air Flow Rate for Final Drying*	Recommended Duration for Drying
2.5×8	1-3 SCFM (1.7-5.1 Nm3/hr.)	1 hr. @ 50°C
4×13	3-5 SCFM (5.1-10.2 Nm3/hr.)	4 hrs. @ 50°C
4×28, 6×28, 8×20	5-20 SCFM (8.5-34 Nm3/hr.)	8 hrs. @ 50°C
8×40	20-40 SCFM (34-68 Nm3/hr.)	16 hrs. @ 50°C
8×80	20-40 SCFM (34-68 Nm3/hr.)	24 hrs. @ 50°C
10×28	20-50 SCFM (34-85 Nm3/hr.)	16 hrs. @ 50°C
14×28	30-60 SCFM (51-102 Nm3/hr.)	24 hrs. @ 50°C
14×40	50-100 SCFM (85-170 Nm3/hr.)	24 hrs. @ 50°C

* Recommended max air pressure 30 psig (2 bar)

XIII. Membrane Contactor Integrity Test

After cleaning and complete drying, it is recommended that an integrity test be done to validate that the contactor hydrophobicity has been restored. The integrity test can also be performed on contactors any time to verify integrity. This test involves mounting the contactor vertically, pressurizing the shell side with water, and measuring the drip rate from the lower lumen side port. Table 9 lists the integrity test procedure. The test starts by pressurizing contactor shell side with water. Do not apply any strip medium to the gas side. During this procedure, it is normal for a small amount of liquid water to transmit from the shell side to the lumen side and drip out of bottom lumen side. The expected drip rates for different contactor sizes have been measured at ambient temperature and are reported in Table 10. The drip rates will depend on the fiber type.

Table 9: Membrane Contactor Integrity Test Procedure; Contactor to be Mounted Vertically

Step	Description
1	Relieve lumenside pressure. Blow-out lumen side with nitrogen or oil-free air. Open lumen ports to atmosphere.
2	Close the shell side outlet valve.
3	Fill shell side with filtered (5-micron abs.) water. Slowly apply 60 psig (4 bar) pressure to the shell side.
4	Measure drip rate from the bottom lumen port for 1 hour. Compare against Table 10 values.
5	Release the shell side pressure by slowly opening the outlet valve. Drain the contactor.

Table 10: Expected Range of Drip Rates Out of Lumen Port During Contactor Integrity Test

3M™ Liqui-Cel™ EXF Series Membrane Contactor Size	Expected Drip Rate for Various Contactors, mL/hr		
	X50 membrane	X40 membrane	XIND membrane
2.5×8	< 3	< 1	Not Applicable
4X13	6 to 13	2 to 4	Not Applicable
4X28	17 to 33	4 to 8	Not Applicable
6×28	34 to 69	9 to 17	Not Applicable
8×20	43 to 86	11 to 21	11 to 21
8×40	Not Applicable	26 to 52	Not Applicable
8×80	Not Applicable	52 to 104	Not Applicable
10×28	106 to 212	26 to 53	106 to 212
14×28	186 to 373	47 to 93	Not Applicable
14×40	283 to 565	71 to 141	Not Applicable

Note that if the water temperature during integrity test is higher than ambient temperature the drip rates are likely to be higher than what are listed in Table 10.

If the measured drip rates significantly exceed the range stated in Table 10, there is a possibility that membrane contactor integrity has been compromised. There are three conditions which will compromise contactor integrity and cause the contactor to leak more than the normally expected drip rate:

- Membrane wet-out
- A fiber break/cartridge damage
- Seal failure

Membrane wet-out can occur when solutions containing surfactants or proteins, such as beer, juice, wine, fermentation broth or other organic solutions pass through the contactor. This is typically a reversible condition once the contactor is cleaned. An integrity test can be performed to verify the restoration of the membrane's hydrophobic property.

A fiber break/cartridge damage can occur due to membrane oxidation and/or over-pressurization of the contactor. A fiber break/cartridge damage cannot be reversed and typically the contactor or cartridge may have to be replaced. Seal failure can be caused due to displacement of the seals or damaged seals. This may be solved by replacing or re-seating the seals.

Contact a 3M representative for additional help.

XIV. Contactor Storage and Handling Guidelines

After cleaning and complete drying of contactors, it is possible to store membrane contactors before using again. If drying is not completed, then contactors must be stored with preservatives, such as Sodium Metabisulfite or dilute caustic solution, on both shell and lumen sides. Storage under an inert gas such as nitrogen or CO_2 is an option. Drain contactor and fill with 0.5 bar (7 psig) inert gas before storage.

Container/packaging: If cleaning and drying are completed, 3M™ Liqui-Cel™ Membrane Contactors should be stored dry and in a sealed plastic bag or shrink wrap material, to keep all ports closed, to help prevent the introduction of contaminants into the contactor.

Temperature: 3M Liqui-Cel Membrane Contactors can be stored dry at temperatures < 49°C (120°F), but preferably at lower temperature such as <35°C (95°F) to not risk reduced lifetime. Membrane contactors should always be stored above freezing temperatures, and if stored at low temperature, they should be allowed to equilibrate to room temperature before use.

Exposure to Sunlight: 3M Liqui-Cel Membrane Contactors should be stored in their original box, or other opaque box and should not be installed where they are exposed to direct sunlight.

⚠ **Storage and Handling:** Care must be taken not to drop, hit or impact the membrane contactor. Use appropriately rated lifting equipment for lifting or moving. Review the product datasheet or operating guide for weights at 3M.com/Liqui-Cel.

⚠ Store the membrane contactors in the horizontal position. 10×28-inch membrane contactors with stainless steel housings may be packaged in cardboard boxes or wooden crates. 14×28-inch, 10×28-inch with FRP housings, 8×20-inch and 6×28-inch membrane contactors are packaged in cardboard boxes. 8×40 inch and 8×80-inch membrane contactors are individually bagged, then cradled on pallets. Membrane contactors should be stored in a safe location where they are not at risk of falling, being crushed or impacted. Always ensure the membrane contactor, and any systems using membrane contactors, are stable, level, and properly secured. Be sure the membrane contactors/system cannot tip, roll, fall,

slide or make any movement that may cause injury, damage to the unit, or damage to other system components.

For more detailed storage information, refer to the Start-up Guide and the Design & Operating Guide of the 3M™ Liqui-Cel™ Membrane Contactors.

XV. Contactor Decontamination for Return to 3M

In the event that a contactor needs to be returned to 3M for analysis, it must be cleaned and dried. A Returned Material Authorization (RMA) form must be obtained from 3M before a contactor is returned. Please follow the instructions below when returning a contactor.

In the USA call 3M at (980) 859-5400 to obtain an RMA form. Outside the USA, contact your 3M representative. Complete the RMA form and return it by email to your representative, Attn: 3M™ Liqui-Cel™ Membrane Contactor Technical Service.

- If Non-Hazardous materials (water, air, nitrogen, oxygen, and carbon dioxide) were used, clean and dry the contactor, then place it in a clean, leak-proof plastic bag.
- Write the RMA number on the outside of the shipping box.
- **If Hazardous Materials** were used in the contactor, follow the cleaning procedure in section VI above. Provide a **Safety Data Sheet (SDS)** for any chemical(s) introduced into the contactor to your product representative. Even though these chemicals need to be flushed from the contactor prior to shipment, the SDS is required information to safe-guard our personnel when handling the returned contactor. Place the contactor in a clean, leak-proof plastic bag. Write the RMA number on the outside of the shipping box.

3M Liqui-Cel Membrane Contactors should never be used with human blood or blood products. If non-human (or other non-primate) blood or blood products were used in the contactor, follow your established moderate cleaning procedure. In addition, flush the contactor with water until the rinsed water is completely clear. Continue rinsing for 30 more minutes to ensure the complete removal of any blood product.

Prior to returning the contactor to 3M, it must be sanitized. The following sanitizing procedure is recommended: Flush the contactor with filtered water with 100 ppm available free chlorine. Adjust the pH >10 with caustic prior to using the hypochlorite solution.

Recommended contact time and temperature with the contactor is 30 minutes at 70°F – 100°F (21°C – 38°C). The active chlorine level should be maintained at 100 ppm during sanitization. The entire cartridge needs to be contacted with this solution to kill bacteria or viruses. Therefore, both the shellside and lumenside flow paths need to be decontaminated.

Flush the contactor with water to remove residual chlorine. Dry the contactor as per procedure described earlier and place the contactor in a leak-proof plastic bag. Write the RMA number on the outside of the shipping box.

It is important to send a copy of the RMA form to 3M prior to shipping. Email the RMA form to your representative, Attn: 3M Liqui-Cel Membrane Contactor Technical Service. Ensure authorization has been received from 3M prior to returning products.

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