Liqui-Cel™ Membrane Contactors

3M™ Liqui-Cel™ EXF Series Membrane Contactors

Cleaning Guidelines

3M.com/Liqui-Cel

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NOTE: User safety and safe handling of the cleaning chemicals being used are not addressed in this document. Such chemicals should only be used by persons familiar with their use (for example, personnel who have received hazardous materials and communications training, are aware of the hazards of the chemicals, and have the appropriate protective equipment as specified in their organization's safety program and the chemical's material safety datasheet (MSDS). 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. Users of any substance should satisfy themselves by independent investigation that the chemical or material can be used safely. We may have described certain hazards, but we cannot guarantee that these are the only hazards that exist. Nothing herein shall be construed as a recommendation, certification or instruction to use any hazardous chemical, substance or material in an unsafe manner. THE INFORMATION CONTAINED HEREIN AND SELLER'S PRODUCTS ARE PROVIDED "AS IS" WITHOUT WARRANTY OF ANY KIND, EITHER EXPRESSED OR IMPLIED, INCLUDING, BUT NOT LIMITED TO, THE IMPLIED WARRANTIES OF MERCHANTABILITY, FITNESS FOR ANY PARTICULAR PURPOSE OR USE, OR NON-INFRINGEMENT OF INTELLECTUAL PROPERTY. IN NO EVENT SHALL SELLER BE LIABLE FOR ANY SPECIAL, INCIDENTAL, INDIRECT, OR CONSEQUENTIAL DAMAGES OF ANY KIND, OR ANY DAMAGES WHATSOEVER RESULTING FROM THE USE OF INFORMATION CONTAINED HEREIN AND/OR SELLER'S PRODUCTS.

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

ILLUSTRATIONS

SAFETY INFORMATION

Read, understand, and follow all safety information contained in these instructions prior to the use of this 3M™ Liqui-Cel™ EXF Series Membrane Contactor. Retain these instructions for future reference.

Intended Use:

This Liqui-Cel membrane contactor is intended to add or remove dissolved gases from non-dangerous liquid streams. It is expected that all users be fully trained in the safe operation of membrane contactors. Membrane contactors are intended for installation and operation by qualified installers and operators in accordance with all operating guidelines, installation instructions, and any other industry requirements. Use in any other application may not have been evaluated by 3M and may lead to an unsafe condition.

⚠ WARNING

To reduce the risks associated with explosion:

- Never exceed the maximum temperature and pressure ratings. Detailed temperature and pressure ratings can be found in the Operating Guide at 3M.com/Liqui-Cel.
- If cleaning the shellside of the membrane only, the lumenside should be open to atmospheric pressure.
- Only use replacement parts supplied by 3M for this product.

To reduce the risks associated with crush or impact related injuries:

- Always ensure that the membrane contactor is properly secured. Be sure the membrane contactor cannot tip, roll, fall, slide or make any movement that may cause injury or damage to other system components.
- Care must be taken not to hit or jar (shock) the membrane contactor.

To reduce the risks associated with lifting or moving:

- Always consult the product datasheet or operating guide for membrane contactor weights. Use appropriately rated lifting equipment for lifting or moving heavy membrane contactors.
- Drain liquid from the contactor before moving. **Do not** move a membrane contactor while it contains liquid.

To reduce the risks associated with chemical exposure:

- When handling, preparing, and using cleaning chemicals, appropriate Personal Protective Equipment (PPE) must be used wherever needed for the protection of eye, face, hand and skin of operator.
- Ensure any tubing or piping used to circulate the cleaning solution is not blocked, kinked, frayed, cracked or obstructing flow in any way. Ensure that all tubing and piping connections are leak-free.
- Avoid splashing of chemicals while preparing cleaning solutions or during pumping of solutions.
- 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.

⚠ CAUTION

To reduce the risks associated with hot surfaces:

- Do not touch the membrane contactor during a hot sanitization cycle and allow enough time for the surface of the membrane contactor to cool.

To reduce the risks associated with environmental contamination:

- At the end of useable life, dispose of the membrane contactor or cartridges in accordance with local regulations and laws.

To reduce the risks associated with damaging the membrane contactor:

- For all 2.5 × 8-inch, 4-inch and 10-inch membrane contactors, lumenside pressure should never exceed shellside pressure during cleaning.
- Do not expose the membrane to air when liquid or gas temperatures exceed 35°C.

NOTICE

- Avoid contacting membrane to surfactants or solvents to prevent membrane wet-out.
- To prevent oxidation, do not expose membrane to oxidizing species such as ozone, chlorine, hydrogen peroxide, peracetic acid, etc.
- Filtered, de-chlorinated, and deionized water is recommended for mixing cleaning solutions. Water containing sparingly soluble compounds of Ca, Mg, Fe, Al, and silica (SiO_2), etc. may precipitate on to the membrane surface when there is a pH shift in the water.
- Failure to follow any instructions in this guide will void any warranty, if any exists.
- Store dry membrane contactor(s) at temperatures < 49°C (120°F) with low to moderate humidity levels (<60% relative humidity).
- The membrane contactor(s) should not be stored where they are exposed to direct sunlight. Membrane contactors should always be stored in sealed bags or shrink wrap material and in the original box or other opaque box.

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 and/or property damage.
NOTICE	Indicates a situation which, if not avoided, could result in property damage.

EXPLANATION OF SAFETY AND RELATED SYMBOLS	
	Warning: Explosion
	Warning: Crush or Impact
	Caution: Lifting or Moving Hazard
	Caution: Possible Environmental Impact

I. Intent of Document

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. These procedure guidelines are intended for cleaning of the water-contact side of the membrane unless otherwise noted. For specific system and application cleaning procedures, contact your 3M representative.

II. 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 guide you through the cleaning process. We recommend starting with cleaning chemicals that are generally used within your industry.

The initial performance of the contactor 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 cleaning frequency can be determined by monitoring decrease in system gas transfer efficiency or increase in pressure drop in liquid or gas phases.

For general questions about chemical resistance, refer to the 3M™ Liqui-Cel™ EXF Series Membrane Contactor Chemical Resistance Guide available at 3M.com/Liqui-Cel or contact a 3M representative.

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.

III. Chemical Compatibility/Sanitization/Detergents

NOTICE

- Avoid contacting membrane to surfactants or solvents to prevent membrane wet-out.
- Do not expose membrane to oxidizing species such as ozone, chlorine, hydrogen peroxide, peracetic acid, etc. to prevent membrane oxidation.

Table 1 shows the maximum recommended cumulative exposure times for several chemicals, which can be used to clean or sanitize a 3M™ Liqui-Cel™ Membrane Contactor. To determine the total exposure time as a function of concentration, divide the value shown in column 2 of Table 1 by the actual chemical concentration. The resulting value is the total maximum number of hours the membrane contactor can be exposed to a specific chemical concentration.

Table 1: Sanitization Guidelines

Chemical	Column 2	Column 3
	Concentration-hours at Room Temperature	Maximum Recommended Chemical Concentration*
Chlorine pH > 7	24000 ppm-hours	100 ppm
Hydrogen Peroxide	4800 %-hours	10% wt.
Peracetic acid	4800 ppm-hours	100 ppm

* Maximum allowable exposure times were determined when the fiber tensile strength and elongation values just began to decrease. The test conditions did not exceed these maximum concentrations and testing was completed at 23°C. Using higher concentrations is not recommended and at elevated temperatures the expected life is much shorter.

Exposure Time Calculations*

* The before mentioned exposure time calculations should be considered estimates only.

Case 1: Sanitization with 2% hydrogen peroxide every day for 30 minutes.

A. What is the total exposure time for a solution of hydrogen peroxide at 2% concentration at room temperature?
B. What is the maximum number of 30 minute cycles that the contactor can be subjected to using this solution at room temperature?
C. Assume the desired number of cycles will be 365 times per year and the contactor will have a lifetime of 3 years.
D. Should this sanitization procedure be used?

Solution

A. Divide 4800 % - hours by 2%. Total maximum allowed cumulative exposure time 2400 hours.
B. Divide 2400 hours by 0.5 hours (30 minutes). Total number of cycles ≈ 4800.
C. Using 365 cycles per year and a time frame of 3 years, the total number of exposure cycles is 1095 (365 * 3 years).
D. It would be safe to use this chemical for daily cleaning for 30 minutes per day at 2% concentration at room temperature since 1095 cycles < 4800 cycles.

The total life expectancy of a Liqui-Cel Membrane Contactor is affected by many factors, one of which is the chemical cleaning cycle. Do not assume the total number of exposure cycles can be used to predict the estimated lifetime of a contactor. Use this total number of cycles to judge whether the contactor lifetime will be affected by the cleaning cycle.

In the case above, compare the number of theoretical cleaning cycles (4800 cycles) to the desired number of cleaning cycles over the expected lifetime of the contactor (1095 cycles). The conclusion in this example is that cleaning cycles will probably not reduce membrane contactor life over the 3 year period.

Case 2 illustrates a sanitization procedure that we **DO NOT RECOMMEND**.

Case 2: Sanitization with 100 ppm peracetic acid every day for 30 minutes.

A. What is the total exposure time for a solution of peracetic acid at 100 ppm concentration at room temperature?

B. What is the maximum number of 30 minute cycles that the contactor can be subjected to using this solution at room temperature?

C. Assume the desired number of cycles will be 365 times per year and a time frame of 3 years. Should this sanitization procedure be used?

Solution

A. Divide 4800 ppm - hours by 100 ppm. Total maximum allowable cumulative exposure time is about 48 hours.

B. Divide 48 hours by 0.5 hours (30 minutes). Total number of cycles is about 96.

C. Using 365 cycles per year and a time frame of 3 years, the total number of exposure cycles is 1095 (365 * 3 years).

It would NOT be advisable to use this chemical for daily sanitization for 30 minutes per day at 100 ppm concentration at room temperature since the required number of cycles (1095) is much greater than the maximum number of 96 cycles.

However, 100 ppm paracetic acid could be used if the cleaning frequency was changed to 4 times per year for 3 years = 12 cycles, which is less than the maximum number of 96 cycles. Alternatively, if the concentration of peracetic acid were reduced to 50 ppm, total maximum allowable exposure time would be about 96 hours, leading to an estimate that the contactor could be sanitized for 30 minutes once a week for 3 years.

IV. Cleaning Solution Flow Rate and Backpressure Guidelines

⚠ WARNING

To reduce the risks associated with explosion:

- Never exceed maximum temperature and pressure ratings. Detailed temperature and pressure ratings can be found in the Operating Guide at 3M.com/Liqui-Cel.
- If cleaning the shellside of the membrane only, the lumenside should be open to atmospheric pressure.
- Only use replacement parts supplied by 3M for this product.

To reduce the risks associated with chemical exposure:

- When handling, preparing, and using cleaning chemicals, appropriate Personal Protective Equipment (PPE) must be used wherever needed for the protection of eye, face, hand and skin of operator.
- Ensure any tubing or piping used to circulate the cleaning solution is not blocked, kinked, frayed, cracked or obstructing flow in any way. Ensure that all tubing and piping connections are leak-free.
- Avoid splashing of chemicals while preparing cleaning solutions or during pumping of solutions.
- 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.

⚠ CAUTION

To reduce the risks associated with chemical exposure:

- For all 2.5 × 8-inch, 4-inch and 10-inch membrane contactors, lumenside pressure should never exceed shellside pressure during cleaning.

NOTICE

- Avoid contacting membrane to surfactants or solvents to prevent membrane wet-out.
- Filtered, de-chlorinated, and deionized water is recommended for mixing cleaning solutions. Water containing sparingly soluble compounds of Ca, Mg, Fe, Al, and silica (SiO2), etc. may precipitate on to the membrane surface when there is a pH shift in the water.
- To prevent oxidation, do not expose membrane to oxidizing species such as ozone, chlorine, hydrogen peroxide, peracetic acid, etc.

It is important 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. The flow rates listed in Table 2 are for single units and should be used only as a guideline. Depending on the nature of the fouling, the flow rate should be adjusted accordingly.

We recommend using de-ionized water when preparing cleaning solutions for rising after cleaning. If deionized water is not available, filtered and de-chlorinated water that does not contain sparingly soluble compounds of Mg, Fe, Al, silica (SiO2), etc. 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.

Warning: Pressure on the liquid side should always exceed the pressure on the gas side. Applying a higher flow/pressure on the gas side may cause damage to the cartridge.

NOTE: Shellside = Outside of fiber. Liquid flows on the shellside in typical operation during typical cleaning.
Lumenside = Inside of fiber. Gas flows on the lumenside in typical operation. Lumenside cleaning is less frequent but may be required under some circumstances.

To prepare a cleaning solution when using untreated raw water, it is important to know the water chemistry. We recommend using de-ionized water when preparing cleaning solutions for rising after cleaning. If deionized water is not available, filtered and de-chlorinated water that does not contain sparingly soluble compounds of Mg, Fe, Al, silica (SiO2), etc. 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.

Table 2: Cleaning Solution Flow Rate Guidelines

Contactor Size	Liquid Flow Rate	Gas Side Flow Rate
2.5 × 8	1 – 2 gpm (0.23 – 0.45 m³/h)	≤0.5 gpm (≤ 0 .11 m³/h)
4 × 13	5 – 15 gpm (1.1 – 3.4 m³/h)	4 - 6 gpm (0.9 – 1.4 m³/h)
4 × 28, 6 × 28, 8 × 20	10 – 30 gpm (2.3 – 6.8 m³/h)	3 - 7 gpm (0.68 – 1.60 m³/h)
8 × 40	40 - 60 gpm (9.0 – 13.6 m³/h)	5 - 10 gpm (1.1 – 2.3 m³/h)
8 × 80	60 - 80 gpm (13.6 – 18.2 m³/h)	5 – 10 gpm (1.1 – 2.3 m³/h)
10 × 28	30 – 40 gpm (4.5 – 9.0 m³/h)	10 - 20 gpm (2.3 – 4.5 m³/h)
14 × 28	50 – 60gpm (11.4 – 13.6 m³/h)	10 - 20 gpm (2.3 – 4.5 m³/h)
14 × 40	100 – 150 gpm (22.7 – 34.1 m³/h)	10 – 20 gpm (2.3 – 4.5 m³/h)

Note: The liquid flow rates shown in Table 2 are guidelines only and may need to be adjusted depending on the condition of the membrane contactor. Always use the minimum solution pressure required to generate the desired cleaning solution flow rate. Under no circumstances should the maximum flow and maximum pressure ratings of the membrane contactors be exceeded. Maximum pressure ratings can be located on product datasheets or in the Operating Guide. If the cleaning solution pressure needed to generate the desirable flow rate exceeds the maximum pressure rating of the membrane contactor then clean using a lower flow rate.

V. Cleaning in Place (CIP) and Hot Water Sanitization

⚠ WARNING

To reduce the risks associated with explosion:

- Never exceed maximum temperature and pressure ratings. Detailed temperature and pressure ratings can be found in the Operating Guide at 3M.com/Liqui-Cel.
- If cleaning the shellside of the membrane only, the lumenside should be open to atmospheric pressure.
- Only use replacement parts supplied by 3M for this product.

To reduce the risks associated with chemical exposure:

- When handling, preparing, and using cleaning chemicals, appropriate Personal Protective Equipment (PPE) must be used wherever needed for the protection of eye, face, hand and skin of operator.
- Ensure any tubing or piping used to circulate the cleaning solution is not blocked, kinked, frayed, cracked or obstructing flow in any way. Ensure that all tubing and piping connections are leak-free.
- Avoid splashing of chemicals while preparing cleaning solutions or during pumping of solutions.
- Cleaning should be conducted at the minimal temperature and pressure required to clean contactors, never exceeding the maximum operating pressure and temperature limits of the contactor.

⚠ CAUTION

To reduce the risks associated with hot surfaces:

- Do not touch the membrane contactor during a hot sanitation cycle, and allow enough time for the surface of the membrane contactor to cool before touching.

To reduce the risks associated with damaging the membrane contactor:

- For all 2.5 × 8-inch, 4-inch and 10-inch membrane contactors, lumenside pressure should never exceed shellside pressure during cleaning.

NOTICE

- Avoid contacting membrane to surfactants or solvents to prevent membrane wet-out.
- Filtered, de-chlorinated, and deionized water is recommended for mixing cleaning solutions. Water containing sparingly soluble compounds of Ca, Mg, Fe, Al, and silica (SiO_2), etc. may precipitate on to the membrane surface when there is a pH shift in the water.
- To prevent oxidation, do not expose membrane to oxidizing species such as ozone, chlorine, hydrogen peroxide, peracetic acid, etc.

A. Cleaning the Membrane

A membrane contactor may lose its performance due to chemical or biological fouling or due to membrane mineral scaling. 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. Contactors and membranes can be chemically cleaned, at warm temperature if needed. The procedures described below includes guidelines only. Actual procedures used by a customer may vary depending on the condition of membranes and should be developed in consultation with a technical representative of the contactor manufacturer. In all procedures described below, it is assumed that cleaning is done only on the water-contact side, i.e. shellside, of the membrane. In some cases, the gas side, i.e. lumenside, of the membrane may also have to be cleaned. All solutions should circulate on the shellside unless there is a special need to clean the lumenside.

B. Sanitization of the Cleaned Membrane

Once the membrane has been cleaned, hot water sanitization can begin using the following guidelines. Do not exceed 85°C during HWS. Only SS vessels are recommended for HWS.

Table 3: HWS Guidelines for Stainless Steel Housings with X40 Fiber in 4 and 10-inch sizes and for X-50 in 4-inch only.

Maximum Temperature	Maximum Operating Pressure	Maximum number of sanitizations
85°C (185°F)	30 psig (2.11 kg/cm²)	1000

NOTE: During HWS, the lumenside should have a small, continuous purge flow of N_2 or another inert gas to protect the membrane from oxidation. In addition, always purge the lumenside with gas after the HWS process is complete. We recommend using an inert gas for purging. If an inert gas is not available, air can be used only after the contactor has cooled down to room temperature. (Air sweep can be used for a final lumen purge only once the contactor has cooled down to room temperature.)

The water temperature during sanitization should be accurately controlled to ensure the temperature never exceeds 85°C (185°F).

VI. CLEANING PROCEDURE FOR BIOLOGICAL FOULING REMOVAL

⚠ WARNING

To reduce the risks associated with explosion:

- Never exceed maximum temperature and pressure ratings. Detailed temperature and pressure ratings can be found in the Operating Guide at 3M.com/Liqui-Cel.
- If cleaning the shellside of the membrane only, the lumenside should be open to atmospheric pressure.
- Only use replacement parts supplied by 3M for this product.

To reduce the risks associated with chemical exposure:

- When handling, preparing, and using cleaning chemicals, appropriate Personal Protective Equipment (PPE) must be used wherever needed for the protection of eye, face, hand and skin of operator.
- Ensure any tubing or piping used to circulate the cleaning solution is not blocked, kinked, frayed, cracked or obstructing flow in any way. Ensure that all tubing and piping connections are leak-free.
- Avoid splashing of chemicals while preparing cleaning solutions or during pumping of solutions.
- Cleaning should be conducted at the minimal temperature and pressure required to clean contactors, never exceeding the maximum operating pressure and temperature limits of the contactor.

⚠ CAUTION

To reduce the risks associated with hot surfaces:

- Do not touch the membrane contactor during a hot sanitation cycle, and allow enough time for the surface of the membrane contactor to cool before touching.

To reduce the risks associated with damaging the membrane contactor:

- For all 2.5 × 8-inch, 4-inch and 10-inch membrane contactors, lumenside pressure should never exceed shellside pressure during cleaning.

NOTICE

- Avoid contacting membrane to surfactants or solvents to prevent membrane wet-out.
- Filtered, de-chlorinated, and deionized water is recommended for mixing cleaning solutions. Water containing sparingly soluble compounds of Ca, Mg, Fe, Al, and silica (SiO_2), etc. may precipitate on to the membrane surface when there is a pH shift in the water.
- To prevent oxidation, do not expose membrane to oxidizing species such as ozone, chlorine, hydrogen peroxide, peracetic acid, etc.

A. Biological Fouling Removal

Typically, a decrease in contactor gas transfer efficiency or an increase in liquid or gas pressure drop through contactor indicates that the contactor requires cleaning. If fouling has not penetrated the membrane pore structure, a surface cleaning of the wetted side of the membrane (normally the shellside) is usually sufficient. If the performance is not restored after two cleaning cycles, then use the procedures described below in Section B.

Table 4: Recommended Cleaning Procedure for Normal Biological Fouling

Step	Process	Description	Time (min.)
1	Water flush / once through	5 micron filtered, ambient to cold water	5
2	Alkaline wash / recirculated	2% to 6% w/w caustic (NaOH or KOH) solution, using 5 micron filtered water. Suggested temperature ambient to 122 °F (ambient to 50 °C)	30 min to 4 hrs.
3	Water flush / once through	5 micron filtered water	5 to 15 minutes.
4	Drain contactor		
5	Acid rinse / recirculated	10% w/w citric, or 1 to 6% nitric, phosphoric, or hydrochloric acids or a combination, using filtered (5 micron) water at ambient temp.	30 min to 2 hrs.
6	Water flush / once through	5 micron filtered, ambient to cold, water	Until acceptable cleaning solution pH is achieved
7	Purge gas side	CO_2, N_2, or air at maximum available flow rate without exceeding flow or pressure rating. If operating in combo mode, use maximum sweep gas in combination with the vacuum pump.	Minimum of 15 minutes and until no water droplets appear from exit gas port

NOTE: Do not use any commercial cleaner that contains surfactants.

B. Recommended cleaning procedure for severe biological membrane fouling

Wet-out refers to a condition of the membrane when it has 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 biological deposits that have penetrated the membrane pore structure will restore the membrane to a hydrophobic state. To remove the proteins adhering to the polymer surface, the following procedure is recommended.

This procedure uses an alcohol-water solution and then 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 absolutely necessary.

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.

Table 5: Severe Biological Fouling Cleaning Procedure

Step	Process	Description	Time (min.)
1	Water flush/Once-through	Filtered (5 micron) water	5 min
2	Wet-out the membrane by pressurizing shellside with IPA or 50/50 IPA-water mixture. Do not exceed the maximum allowable operating pressure for the contactor. Let liquid come out of both lumen (gas) ports. Then, cap off lumen (gas) ports and let the contactor soak.	Isopropanol (IPA) or 2-Propanol	Recommended soak time: at least 1 hr. Depending on severity of fouling, an extended soak time may be needed.
3	Alkaline wash / recirculate	2-5% w/w. caustic (NaOH or KOH) solution using filtered (5 micron) water. Suggested temperature 86 °F – 122°F (30 °C – 50°C).	1 to 4 hrs.
4	Drain contactor		
5	Acid rinse / recirculated	10% w/w citric, or 1 to 6% nitric, phosphoric, or hydrochloric acids or a combination, using filtered (5 micron) water at ambient temp.	1 to 2 hrs.
6	Drain contactor		
7	Water flush/Once-through	Filtered (5 micron) water – ambient temperature.	20 to 30 min
8	Drying	Inert gas is preferred. Clean, dry, oil free air can also be used. Do not exceed 122°F (50°C) gas temperature when using air to dry the contactors.	See section IX
9	Membrane Integrity Test		See section X

* Note that the air temperature should not exceed 35°C (95°F) in normal operations. Higher temperatures are only recommended for short cleaning/drying cycles.

Illustration 1: Flow Schematic for Normal Biological Fouling Removal

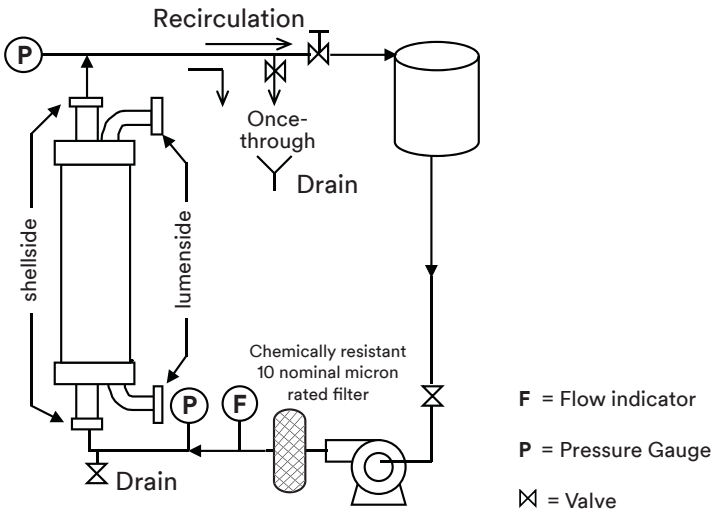
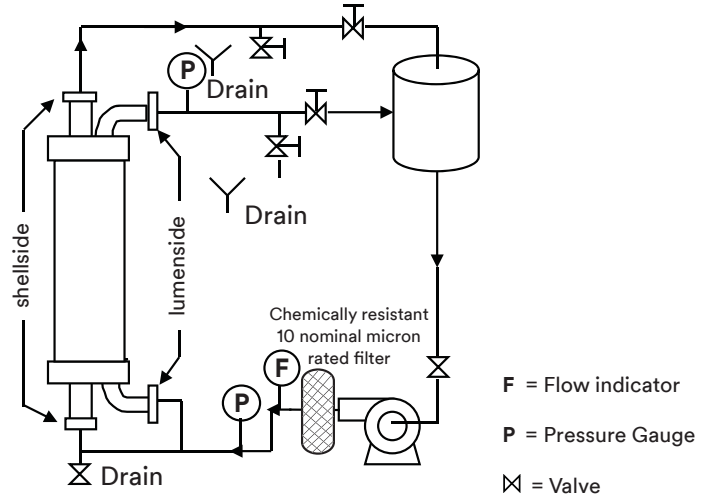


Illustration 2: Flow Schematic for Severe Biological Fouling Removal



VII. CLEANING PROCEDURE FOR MINERAL SCALING REMOVAL

WARNING

To reduce the risks associated with explosion:

- Never exceed maximum temperature and pressure ratings. Detailed temperature and pressure ratings can be found in the Operating Guide at 3M.com/Liqui-Cel.
- If cleaning the shellside of the membrane only, the lumenside should be open to atmospheric pressure.
- Only use replacement parts supplied by 3M for this product.

To reduce the risks associated with chemical exposure:

- When handling, preparing, and using cleaning chemicals, appropriate Personal Protective Equipment (PPE) must be used wherever needed for the protection of eye, face, hand and skin of operator.
- Ensure any tubing or piping used to circulate the cleaning solution is not blocked, kinked, frayed, cracked or obstructing flow in any way. Ensure that all tubing and piping connections are leak-free.
- Avoid splashing of chemicals while preparing cleaning solutions or during pumping of solutions.
- Cleaning should be conducted at the minimal temperature and pressure required to clean contactors, never exceeding the maximum operating pressure and temperature limits of the contactor.

CAUTION

To reduce the risks associated with hot surfaces:

- Do not touch the membrane contactor during a hot sanitation cycle, and allow enough time for the surface of the membrane contactor to cool before touching.

To reduce the risks associated with damaging the membrane contactor:

- For all 2.5 × 8-inch, 4-inch and 10-inch membrane contactors, lumenside pressure should never exceed shellside pressure during cleaning.

NOTICE

- Avoid contacting membrane to surfactants or solvents to prevent membrane wet-out.
- Filtered, de-chlorinated, and deionized water is recommended for mixing cleaning solutions. Water containing sparingly soluble compounds of Ca, Mg, Fe, Al, and silica (SiO_2), etc. may precipitate on to the membrane surface when there is a pH shift in the water.
- To prevent oxidation, do not expose membrane to oxidizing species such as ozone, chlorine, hydrogen peroxide, peracetic acid, etc.

The inlet water should be treated to prevent mineral precipitation. For example, changes in pH due to carbon dioxide removal may initiate a precipitation reaction.

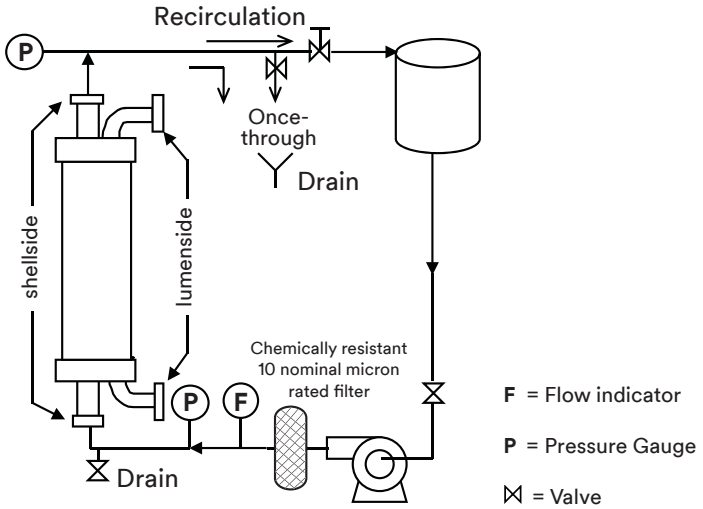
If the performance of the contactor decreases and the inlet water source is not treated to remove minerals such as calcium carbonate, it is likely that a layer of mineral scale has formed on the wetted side (normally the shellside) of the contactor. A simple acid cleaning followed by a water flush should restore the performance. The contactor does not need to be dried after this procedure.

Table 6: Cleaning Procedure for Scaling Deposit Removal

Step	Process	Description	Time (min.)
1	Water flush/ Once-through	Filtered (5 micron) water	5 min
2	Acid wash / recirculate (repeat if necessary)	10% w/w citric, or 1% to 6% Nitric, phosphoric, or hydrochloric acids or a combination, using filtered (5 micron) water ambient temperature	30 min to 2 hours
3	Drain contactor		
4	Water flush / Once through	Filtered (5 micron) water Flush until pH in = pH out	Until acceptable cleaning solution pH is achieved

If silica, aluminum or a combination of these is found in the inlet water source, it is likely that they will precipitate on the membrane surface. If CO_2 is used as a sweep gas, precipitation can occur depending on the concentration and the water pH shift. For aluminum precipitation, follow the mineral deposit removal procedure. For silica precipitation refer to the Severe Biological Fouling Removal Procedure, section VI, but use higher caustic concentration (up to 6% by weight) and increase the temperature to maximum operating limit of contactor. If possible, the flow rate of the cleaning solution should be similar to the process water flow rate used during normal operation. In addition, it is advisable to keep the direction of the water flow consistent during cleaning.

Illustration 3: Flow Schematic for Acid Cleaning to Remove Scaling Deposits



VIII. CLEANING PROCEDURE WHEN PARTICLE FOULING IS SUSPECTED

⚠ WARNING

- **NOTE:** Never use this procedure for 3M™ Liqui-Cel™ SP Series Membrane Contactors.

To reduce the risks associated with explosion:

- Never exceed maximum temperature and pressure ratings. Detailed temperature and pressure ratings can be found in the Operating Guide at 3M.com/Liqui-Cel.
- If cleaning the shellside of the membrane only, the lumenside should be open to atmospheric pressure.
- Only use replacement parts supplied by 3M for this product.

To reduce the risks associated with chemical exposure:

- When handling, preparing, and using cleaning chemicals, appropriate Personal Protective Equipment (PPE) must be used wherever needed for the protection of eye, face, hand and skin of operator.
- Ensure any tubing or piping used to circulate the cleaning solution is not blocked, kinked, frayed, cracked or obstructing flow in any way. Ensure that all tubing and piping connections are leak-free.
- Avoid splashing of chemicals while preparing cleaning solutions or during pumping of solutions.
- Cleaning should be conducted at the minimal temperature and pressure required to clean contactors, never exceeding the maximum operating pressure and temperature limits of the contactor.

⚠ CAUTION

To reduce the risks associated with hot surfaces:

- Do not touch the membrane contactor during a hot sanitation cycle, and allow enough time for the surface of the membrane contactor to cool before touching.

To reduce the risks associated with damaging the membrane contactor:

- For all 2.5 × 8-inch, 4-inch and 10-inch membrane contactors, lumenside pressure should never exceed shellside pressure during cleaning.

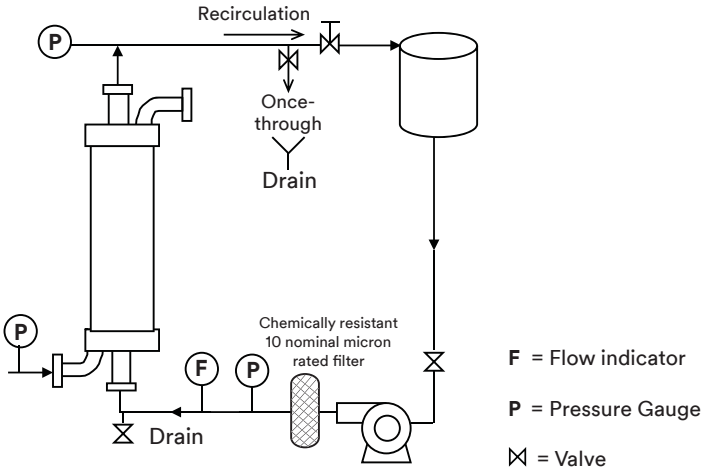
NOTICE

- Avoid contacting membrane to surfactants or solvents to prevent membrane wet-out.
- Filtered, de-chlorinated, and deionized water is recommended for mixing cleaning solutions. Water containing sparingly soluble compounds of Ca, Mg, Fe, Al, and silica (SiO_2), etc. may precipitate on to the membrane surface when there is a pH shift in the water.
- To prevent oxidation, do not expose membrane to oxidizing species such as ozone, chlorine, hydrogen peroxide, peracetic acid, etc.

Follow the steps described in sections VI-A and VII with the following exceptions:

- Backflush the cleaning solutions (i.e. introduce the cleaning solutions in the direction opposite of the normal operating flow direction).
- Once the cleaning solution is flowing into the contactor, introduce clean, dry, and oil-free compressed air into one gas port and in the same direction as the liquid flow. Valve off or cap the other gas port.
- Regulate the air pressure 5-10 psig 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.
- At the end of the cleaning procedure, shut off the air supply first, then the liquid.

Illustration 4: Flow Schematic for Particle Fouling



IX. MEMBRANE DRYING

⚠ WARNING

To reduce the risks associated with explosion:

- Never exceed maximum temperature and pressure ratings. Detailed temperature and pressure ratings can be found in the Operating Guide at 3M.com/Liqui-Cel.
- Only use replacement parts supplied by 3M for this product.

⚠ CAUTION

To reduce the risks associated with damaging the membrane contactor:

- For all 2.5 × 8-inch, 4-inch and 10-inch membrane contactors, lumenside pressure should never exceed shellside pressure during cleaning.
- Do not expose the membrane to air when liquid or gas temperatures exceed 35°C.

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 8 and 9 provide reference points for flow rates and drying times.

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 the Bulk Water Removal schematic on page 12. Use clean, dry and filtered (0.2 micron) gas at the flow rates shown in Table 8. Keep the lower lumen and shellside ports open.

Discontinue the gas flow when the water discharge rate decreases to a few drips. Close the bottom shellside port when finished.

Table 7: Bulk Water Removal Conditions

3M™ Liqui-Cel™ EXF Series Membrane Contactor Size	Gas Flow Rate scfm*
2.5 × 8	3 scfm (1.7 m³/hr)
4 × 13	10 scfm
4 × 28, 6 × 28, 8 × 20	20 scfm (17 m³/hr)
8 × 40	40 scfm
8 × 80	80 scfm
10 × 28 and 14 × 28	50 scfm (120 m³/hr)
14 × 40	100 scfm

* Maximum gas pressure = 10 psig (0.7 kg/cm²)

B. Final Drying

The final drying step involves flowing a clean, dry, filtered (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 Table 9 for estimated flow rate and drying time guidelines. Keep the lower shellside port closed.

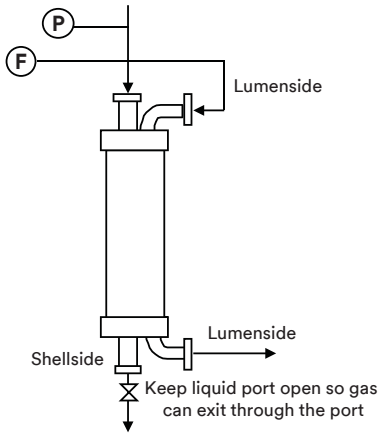
Table 8: Final Drying Conditions

3M™ Liqui-Cel™ EXF Series Membrane Contactor Size	Gas Flow Rate*	Estimated Drying Time
2.5 × 8	1 scfm (1.7 m³/hr)	1 hr @ 60°C (140°F)
4 × 13	5 scfm	4 hr @ 60°C (140°F)
4 × 28	10 scfm (17 m³/hr)	4 hr @ 60°C (140°F)
6 × 28	25 scfm (40 m³/hr)	8 hr @ 60°C (140°F)
8 × 20	20 scfm	8 hr @ 60°C (140°F)
8 × 40	30 scfm	16 hr @ 60°C (140°F)
8 × 80	60 scfm	24 hr @ 60°C (140°F)
10 × 28	40 scfm (120 m³/hr)	16 hr @ 60°C (140°F)
14 × 28	50 scfm (130 m³/hr)	24 hr @ 60°C (140°F)
14 × 40	100 scfm	24 hr @ 60°C (140°F)

* Maximum gas pressure = 10 psig (0.7 kg/cm²)

C. Drying Schematics

Illustration 5: Bulk Water Removal/Initial Drying Step

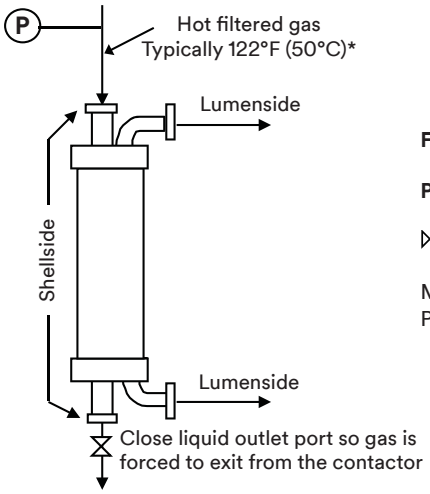


F = Flow indicator

P = Pressure Gauge

⋈ = Valve

Illustration 6: Final Drying



F = Flow indicator

P = Pressure Gauge

⋈ = Valve

Maximum Gas Pressure = 10 psig (0.70 kg/cm²)

X. MEMBRANE CONTACTOR INTEGRITY TEST

There are three conditions which will cause the contactor to leak:

- Membrane wet-out
- A fiber break
- O-ring/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. This test involves pressurizing the shellside with water and measuring the drip rate leaving the lower lumenside port. The integrity test should be completed after cleaning.

Table 9: Membrane Contactor Integrity Test

Steps	
1.	Relieve lumenside pressure. Blow-out lumenside stream with nitrogen or oil-free air. Open the lower lumenside port connection so an observation can be made.
2.	Close the shellside outlet valve.
3.	Fill the shellside with filtered (10-micron) water. Slowly apply 60 psig (4.2 kg/cm²) pressure to the shellside.
4.	Measure the drip rate from the lumenside port for 1 hour.
5.	Release the shellside pressure by slowly opening the outlet valve. Drain the contactor.

When the liquid side of contactor is pressurized with water but no strip medium is applied to the gas side, it is normal for a small amount of liquid water to transmit from the shellside to the lumenside and exit through the lumenside port. To establish a baseline, this drip rate can be compared to a new contactor. The drip rates will depend on the fiber type. Table 11 provides a guideline for typical drip rates during normal operating conditions at ambient temperatures.

If the contactor drips at a higher rate than the value listed in Table 11, either the cleaning procedure needs to be repeated, a fiber is broken, or a seal is damaged. Contact a 3M representative for additional help.

Table 10: Typical Drip Rates out of Lumenside Port

Contactor size	Normal drip rate (approx.), ml/hr for fiber type		
	X50 Fiber	X40 Fiber	XIND
2.5×8	< 3	< 1	N/A
4×13	6 to 13	2 to 4	N/A
4×28	16 to 32	4 to 8	N/A
6×28	34 to 67	8 to 17	N/A
8×20	42 to 85	11 to 21	42 to 85
10×28	105 to 210	26 to 52	105 to 210
14×28	176 to 352	44 to 88	N/A

XI. STORAGE AND HANDLING GUIDELINES

The Membrane Contactor that you have purchased can be damaged through improper handling and storage. The following guidelines are intended to provide a framework for successful storage of these contactors. If you have any questions, please contact your 3M representative.

Handling: Proper handling of contactors is critical. Care must be taken not to hit or jar (shock) the contactor to minimize the possibility of internal damage. It is recommended that the contactors be stored in a dry, heat-sealed plastic bag or shrink wrap material [0.08 mm (0.003 in.) wall thickness] in their original box to prevent the introduction of contaminants into the contactor.

Important Note: All plastic port extensions should be supported to prevent bending of extensions under excessive piping loads.

Temperature: Store the contactor dry in their original boxes at temperatures not to exceed 49°C (120°F). Contactors stored at very low temperatures < 5°C (41°F) should be allowed to equilibrate to room temperature prior to introducing water.

Humidity: It is recommended that contactors be stored at low to moderate humidity levels (< 60% relative humidity). Humidity will not affect the components of the contactor but exposure at high humidity levels may affect the integrity of the cardboard boxes.

Storage: Store the membrane contactors in the horizontal position. 10×28-inch membrane contactors with stainless steel housings are packaged in 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. Care should be taken to secure the membrane contactors and containers to ensure stability and to avoid any possible injury resulting from falling, leaning or any other accident.

Shelf Life: Membrane samples from contactors stored for 4 years (room temperature, low to moderate humidity, heat-sealed bag but not stored in a box) have shown no changes in physical properties (hollow fiber tensile strength and elongation).

Exposure to Sunlight: Contactors should not be stored where they are exposed to direct sunlight. Contactors should always be stored in sealed bags or shrink wrap material and in the original box or other opaque box.

Product Use: Many

XII. 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.

Call 3M at (980) 859-5400 to obtain an RMA form. Complete the 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 **Material Safety Data Sheet (MSDS)** 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 MSDS 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 RGA number on the outside of the shipping box.

  If non-human (or other non-primate) blood or blood products were used in the contactor, follow your established normal 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: (5.25% available chlorine) diluted 1:500 with filtered water (final concentration = ~100 ppm available chlorine). Adjust the pH >10 using caustic prior to adding 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 the duration of the cleaning cycle. 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.

  Dry the contactor as per section IX and place the contactor in a leak-proof plastic bag. Write the RGA 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.

Technical Information: The technical information, recommendations and other statements contained in this document are based upon tests or experience that 3M believes are reliable, but the accuracy or completeness of such information is not guaranteed.

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