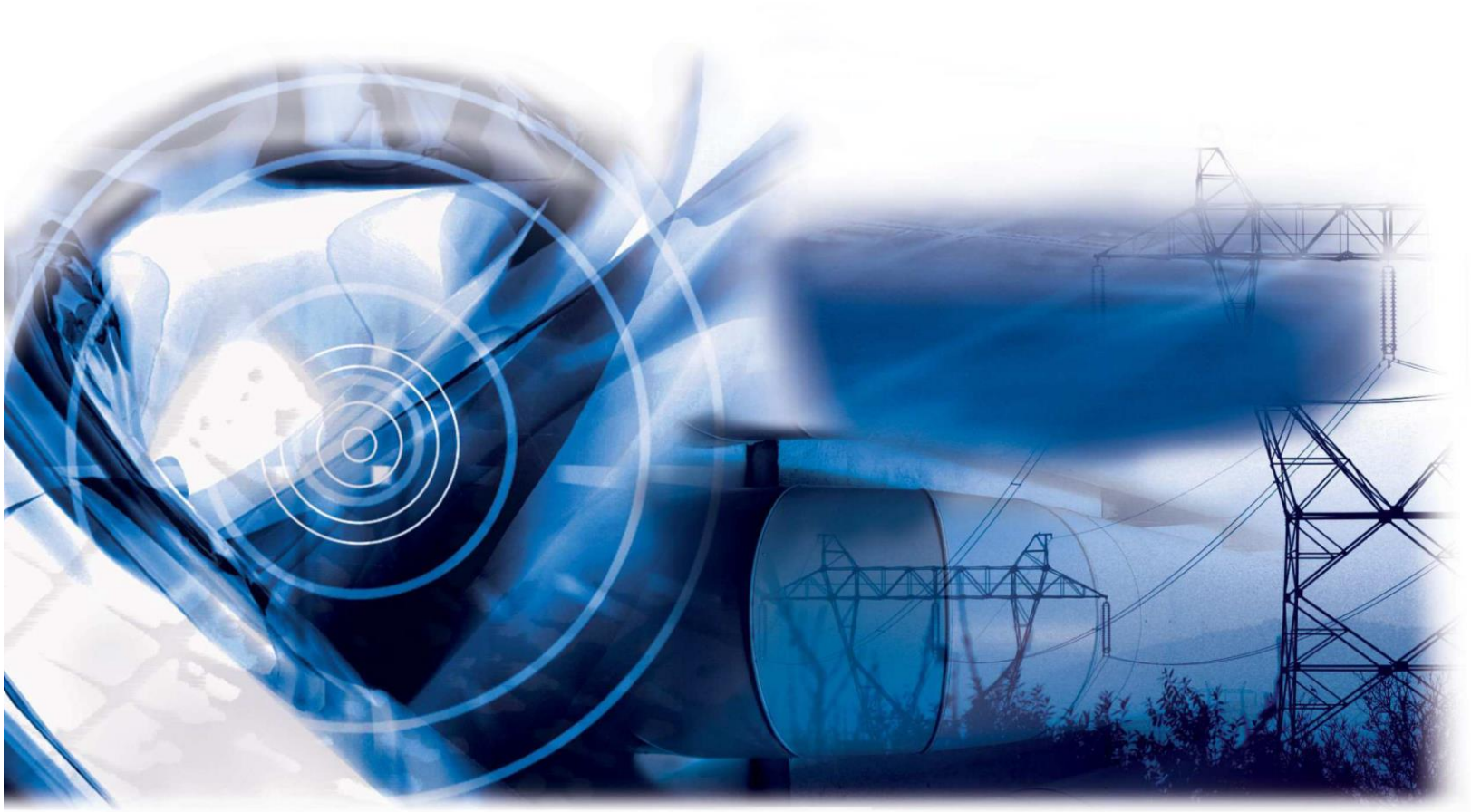
Liqui-Cel®
MEMBRANE CONTACTORS

Cleaning Guidelines



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SAFETY INFORMATION

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

Intended Use:
This Liqui-Cel Membrane Contactor is intended to add to 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.

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

⚠ 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 www.3m.com/liqui-cel.
- If cleaning the shellside of the membrane only, the lumenside should be open at 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 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:

- Use face protection, protective gloves, and appropriate eye protection when handling and using all cleaning chemicals.
- Ensure any tubing or piping used to circulate the cleaning solution is not blocked, kinked, frayed, cracked or obstructing flow in any way. Ensure the all tubing and piping connections are leak-free.
- Avoid splashing of chemicals while preparing cleaning solutions or during pumping of solutions.
- Cleaning procedures should be conducted using the minimal temperatures and pressures required to clean 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 2.5x8-inch, 4-inch and 10-inch membrane contactors, lumen side pressure should never exceed shell side pressure during cleaning.
- Do not expose the membrane to air when liquid or gas temperatures exceed 30° C.

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.
- Filtered, de-chlorinated, and deionized water is recommended for mixing cleaning solutions. Water containing sparingly soluble compounds of Ca, Mg, Fe, etc. and silica (SiO2) 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.

IMPORTANT INFORMATION - PLEASE READ CAREFULLY

I. Intent of Document

There are many different types of contaminants that may adhere to the membrane. The cleaning protocol, which covers chemical cleaning agents, concentrations, time and flow rates, will be specific to each system. In all cleaning procedure discussed below it is assumed that cleaning is intended for the water-contact side of the membrane.

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 and chemical strength (caustic, acid, alcohol, etc.)

Changing any one of these parameters can affect the others. Therefore, it is important to develop a specific cleaning protocol 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 the best protocol for your applications are:

- Experimentation with *time* (frequency and duration), *temperature*, *chemical concentration* and cleaning liquid *flow rate* will determine the best method for cleaning the contactor.
- Refer to the Liqui-Cel® 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 protocol 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 a 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 Liqui-Cel Membrane Contactor ***Chemical Resistance Guide*** available at www.liqui-cel.com or contact a 3M representative.

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 exposure times for several chemicals, which can be used to clean or sanitize a 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 number of hours the contactor can be exposed to a specific chemical concentration.

Table 1: Sanitizing Guidelines

	Column 2	Column 3
Chemical	**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

* 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 (Note that these are very rough calculations and should at best be considered as estimates.)

Case 1: 2% hydrogen peroxide sanitation 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 cleaning chemical protocol be used?

Solution

a) Divide 4800 % - hours by 2%. Total maximum allowed cumulative exposure time is about 2400 hours.
b) Divide 2400 hours by 0.5 hours (30 minutes). Total number of cycles is about 4800.
*c) Using 365 cycles per year and an expected lifetime of 3 years, the total number of exposure cycles is 1095 (365 * 3 years). 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 the 3-year lifetime of the membrane.

Case 2 illustrates a cleaning protocol that we **DO NOT RECOMMEND**.

Case 2: 200ppm peracetic acid sanitization every day for 30 minutes.

a) What is the total exposure time for a solution of peracetic acid at 200ppm 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.
Should this cleaning chemical protocol be used?

Solution

a) Divide 4800ppm - hours by 200ppm. Total maximum allowable cumulative exposure time is about 24 hours.
b) Divide 24 hours by 0.5 hours (30 minutes). Total number of cycles is about 48.
*c) Using 365 cycles per year and an expected lifetime of 3 years, the total number of exposure cycles is 1095 (365 * 3 years).*
It would NOT be safe to use this chemical for daily cleaning for 30 minutes per day at 200ppm concentration at room temperature since the required number of cycles (1095) is much greater than the maximum number of 48 cycles.

However, the protocol 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 48 cycles.

Alternately, if concentration of peracetic acid were reduced to 50 ppm, total maxcimum allowable exposure time is about 96 hours, and it can be estimated that it is possible to sanitize the contactor 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 www.3m.com/liqui-cel.
- If cleaning the shellside of the membrane only, the lumenside should be open to atmosphere.

To reduce the risks associated with chemical exposure:

- Use face protection, protective gloves, and appropriate eye protection when handling and using all cleaning chemicals.
- Ensure any tubing or piping used to circulate the cleaning solution is not blocked, kinked, frayed, cracked or obstructing flow in any way. Ensure the all tubing and piping connections are leak-free.
- Avoid splashing of chemicals while preparing cleaning solutions or during pumping of solutions.
- Cleaning procedures should be conducted using the minimal temperatures and pressures required to clean the contactor.

⚠ CAUTION

To reduce the risks associated with damaging the membrane contactor:

- For all 2.5x8-inch, 4-inch and 10-inch membrane contactors, gas side pressure should never exceed liquid side pressure during cleaning.

NOTICE

- Avoid contact with surfactants/solvents or oxidants (e.g. ozone, chlorine) to the hydrophobic membrane to prevent wet-out or oxidation.
- Filtered, de-chlorinated, and deionized water is recommended for mixing cleaning solutions. Water containing metals such as Mg, iron, Al, and SiO_2 may precipitate on to the membrane surface when there is a pH shift in the water.

It is important to apply a backpressure to the system to ensure that the system is completely full of cleaning solution before cleaning. During actual cleaning maintaining of backpressure may not be needed. To increase the cleaning solution backpressure, slowly and partially close the outlet flow valve. Refer to Table 2 for general guidelines. 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.

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

Table 2: Cleaning Solution Flow Rate and Backpressure Guidelines

Contactor Size	Cleaning Solution Flow Rate on Shell Side	Cleaning Solution Flow Rate on Lumen Side
MiniModule® *	≤ 0.08 gpm (≤ 300 ml/min)	≤ 0.13 gpm (≤ 500 ml/min)
2 x 6	≤ 0.26 (≤ 1 lit/min)	Not Applicable
2.5 x 8	1 – 2 gpm (0.23 – 0.45 m^3/h)	≤0.5 gpm (≤ 0 .11 m^3/h)
4 x 13	5 – 15 gpm (1.1 – 3.4 m^3/h)	4 - 6 gpm (0.9 – 1.4 m^3/h)
4 x 28, 6 x 28, **8 x 20**	10 – 30 gpm (2.3 – 6.8 m^3/h)	3 - 7 gpm (0.68 – 1.60 m^3/h)
8 x 40	40 - 60 gpm (9.0 – 13.6 m^3/h)	5 - 10 gpm (1.1 – 2.3 m^3/h)
8 x 80	60 - 80 gpm (13.6 – 18.2 m^3/h)	5 – 10 gpm (1.1 – 2.3 m^3/h)
10 x 28	30 – 40 gpm (4.5 – 9.0 m^3/h)	10 - 20 gpm (2.3 – 4.5 m^3/h)
14 x 28	50 – 60gpm (11.4 – 13.6 m^3/h)	10 - 20 gpm (2.3 – 4.5 m^3/h)
14 x 40	100 – 150 gpm (22.7 – 34.1 m^3/h)	10 – 20 gpm (2.3 – 4.5 m^3/h)

* MiniModules® operate with liquid on the lumenside.

To prepare a cleaning solution when using untreated raw water, it is important to know the water chemistry. We recommend using water that has been filtered and de-chlorinated. We recommend using de-ionized water for cleaning if possible. We also recommend paying attention to sparingly soluble compounds of Ca, Mg, Fe, Al, etc. and insoluble silica, SiO_2.These elements can precipitate onto the membrane when there is a pH shift in the water.

V. Cleaning In Place (CIP) and Hot Water Sanitization (HWS)

⚠ 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 www.3m.com/liqui-cel.
- If cleaning the shellside of the membrane only, the lumenside should be open to atmosphere.

To reduce the risks associated with chemical exposure:

- Use face protection, protective gloves, and appropriate eye protection when handling and using all cleaning chemicals.
- Ensure any tubing or piping used to circulate the cleaning solution is not blocked, kinked, frayed, cracked or obstructing flow in any way. Ensure the all tubing and piping connections are leak-free.
- Avoid splashing of chemicals while preparing cleaning solutions or during pumping of solutions.
- Cleaning procedures should be conducted using the minimal temperatures and pressures required to clean 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 damaging the membrane contactor:

- For all 2.5x8-inch, 4-inch and 10-inch membrane contactors, gas side pressure should never exceed liquid side pressure during cleaning.
- Do not expose the membrane to air when liquid or gas temperatures exceed 30° C.

NOTICE

- Avoid contact with surfactants/solvents or oxidants (e.g. ozone, chlorine) to the hydrophobic membrane to prevent wet-out or oxidation.
- Filtered, de-chlorinated, and deionized water is recommended for mixing cleaning solutions. Water containing metals such as Mg, Fe, Al, and SiO_2 may precipitate on to the membrane surface when there is a pH shift in the water.

A. Cleaning the Membrane

Contactor may lose its performance due to chemical or biological fouling or due to membrane 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. Contactor and membrane can be chemically cleaned, at warm temperature if needed. The procedure described below includes guidelines only. Actual procedure used by customer may vary depending on condition of membrane and should be developed in consultation with technical representative of contactor manufacturer. In all procedure

described below it is assumed that cleaning is done only on the water-contact side of membrane. In some cases the gas side 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, sanitization can begin using the following guidelines. Do **not** exceed 85 ºC during HWS. Only SS vessels are recommended for HWS.

Table 3: Hot Water Sanitization (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 sanitations
85 ºC (185 ºF)	30 psig (2.11 kg/cm^2)	1000

NOTE: During HWS the lumenside should have a small, continuous flow of N2 or other inert gas to protect the membrane from oxidation. In addition, always purge the lumenside with gas after the Hot CIP 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.

The water temperature during the sanitization should be accurately controlled to ensure the temperature never exceeds 85 ºC (185 ºF).

VI. Cleaning Protocol 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 www.3m.com/liqui-cel.
- If cleaning the shellside of the membrane only, the lumenside should be open to atmosphere.

To reduce the risks associated with chemical exposure:

- Use face protection, protective gloves, and appropriate eye protection when handling and using all cleaning chemicals.
- Ensure any tubing or piping used to circulate the cleaning solution is not blocked, kinked, frayed, cracked or obstructing flow in any way. Ensure the all tubing and piping connections are leak-free.
- Avoid splashing of chemicals while preparing cleaning solutions or during pumping of solutions.
- Cleaning procedures should be conducted using the minimal temperatures and pressures required to clean 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 2.5x8-inch, 4-inch and 10-inch membrane contactors, gas side pressure should never exceed liquid side pressure during cleaning.
- Do not expose the membrane to air when liquid or gas temperatures exceed 30° C.

NOTICE

- Avoid contact with surfactants/solvents or oxidants (e.g. ozone, chlorine) to the hydrophobic membrane to prevent wet-out or oxidation.
- Filtered, de-chlorinated, and deionized water is recommended for mixing cleaning solutions. Water containing metals such as Mg, iron, Al, and SiO2 may precipitate on to the membrane surface when there is a pH shift in the water.

A. Biological Soil 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 soil 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 ***Severe Biological Soil Cleaning Protocol, section VI-B***.

Table 4: Recommended Cleaning Procedure For Normal Biological Fouling

Step	Description	Chemical Solution	Time
1	Water flush / once through	10 micron filtered, ambient to cold water	5 min
2	Alkaline wash / recirculated	2% to 6% w/w caustic (NaOH or KOH) solution, using 10 micron filtered water. Suggested temperature ambient to 122 ºF (ambient to 50 ºC)	30 min to 4 hrs.
3	Water flush / once through	10 micron filtered, ambient to cold, water	5 to 15 minutes.
4	Acid rinse / recirculated	10% w/w citric, or 3% Nitric or phosphoric, or 3% hydrochloric or a combination of 3% Nitric and 3% phosphoric acid solution or 3% Nitric and 3% HCL using filtered (10 micron) water at ambient temp.	30 min to 2 hrs.
5	Water flush / once through	10 micron filtered, ambient to cold, water	Until acceptable cleaning solution pH is achieved
6	Purge lumens	CO_2, N_2, air at maximum flow rate. If operating in combo mode, use maximum sweep gas in combination with the vacuum pump.	Until no water droplets appear from exit sweep port

Do not use any commercial cleaner that contains surfactants.

B. Recommended cleaning procedure for severe 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 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, a ***Severe Biological Soil Cleaning Protocol*** is recommended.

This uses an alcohol-water solution and then a final drying step. The cleaning frequency depends on the types and concentrations of proteins. In order to prevent wet-out, a daily cleaning protocol should be used until an appropriate cleaning frequency is determined.

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 Fouling Procedure

Step	Description / Flow Schematic	Chemical Solution	Time
1	Water flush/Once-through	Filtered (10 micron) water	5 min
2	Wet-Out Membrane Pressurize shell side up to the max allowable operating pressure for the contactor and let liquid come out of lumen ports	Isopropanol (IPA) or 2-Propanol	15 min – 2 hrs.
4	Alkaline wash / recirculate	2-5% w/w. caustic (NaOH or KOH) solution using filtered (10 micron) water. Suggested temperature 86 ºF to 122 ºF (30 ºC to 50 ºC)	1 to 4 hrs.
5	Drain contactor		
6	Acid rinse / recirculated	5% w/w citric, or 3% Nitric or phosphoric, or 3% hydrochloric or a combination of 3% Nitric and 3% phosphoric acid solution or 3% Nitric and 3% HCL using filtered (10 micron) water at ambient temp.	1 to 2 hrs.
7	Drain contactor		
8	Water flush/Once-through	Filtered (10 micron) water – ambient temperature. Flush until pH in = pH out.	20 to 30 min
9	Drying	Inert gas is preferred. Clean, dry, oil free air can also be used. Do not exceed 122 ºF (50 ºC) gas temperature when using air to dry the contactors	See section VIII
10	Membrane Integrity Test		See section IX

* Note that the air temperature should not exceed 30 ºC (86 ºF) in normal operations. Higher temperatures are only recommended for short cleaning/drying cycles.

Illustration 1: Flow Schematic for Normal Biological Soil Removal

F = Flow Indicator

P = Pressure Gauge

⋈ = Valve

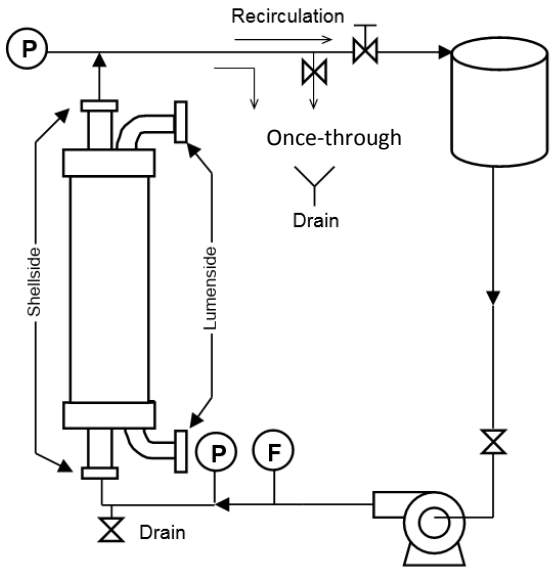
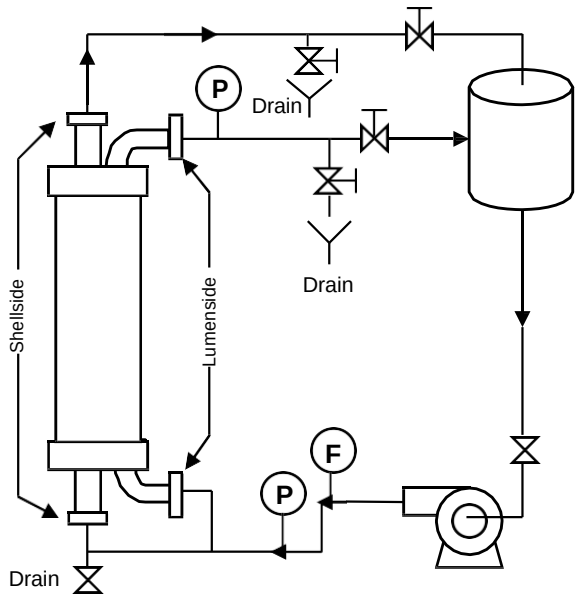


Illustration 2: Flow Schematic for Severe Biological Soil Removal

F = Flow Indicator **P = Pressure Gauge** **⋈ = Valve**	

VII. Cleaning Protocol for Mineral Deposits 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 www.3m.com/liqui-cel.
- If cleaning the shellside of the membrane only, the lumenside should be open to atmosphere.

To reduce the risks associated with chemical exposure:

- Use face protection, protective gloves, and appropriate eye protection when handling and using all cleaning chemicals.
- Ensure any tubing or piping used to circulate the cleaning solution is not blocked, kinked, frayed, cracked or obstructing flow in any way. Ensure the all tubing and piping connections are leak-free.
- Avoid splashing of chemicals while preparing cleaning solutions or during pumping of solutions.
- Cleaning procedures should be conducted using the minimal temperatures and pressures required to clean the contactor.

⚠ CAUTION

To reduce the risks associated with damaging the membrane contactor:

- For all 2.5x8-inch, 4-inch and 10-inch membrane contactors, gas side pressure should never exceed liquid side pressure during cleaning.
- Do not expose the membrane to air when liquid or gas temperatures exceed 30° C.

NOTICE

- Avoid contact with surfactants/solvents or oxidants (e.g. ozone, chlorine) to the hydrophobic membrane to prevent wet-out or oxidation.
- Filtered, de-chlorinated, and deionized water is recommended for mixing cleaning solutions. Water containing metals such as Mg, iron, Al, and SiO_2 may precipitate on to the membrane surface when there is a pH shift in the water.

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 protocol. Also, note that phosphoric acid is more efficient for removing hard mineral deposits or other precipitated deposits.

Table 6: Cleaning Protocol for Mineral Deposit Removal

Step	Description / Flow Schematic	Chemical Solution	Time (min.)
1	Water flush/Once-through	Filtered (10 micron) water	5 min
2	Acid wash / recirculate (repeat if necessary)	5% w/w citric, or 3% Nitric or phosphoric, or a combination of 3% Nitric and 3% phosphoric acid solution using filtered (10 micron) water – ambient temperature	30 min to 2 hours
3	Drain contactor		
4	Water flush / Once through	Filtered (10 micron) water Flush until pH in = pH out	5 to 10 min

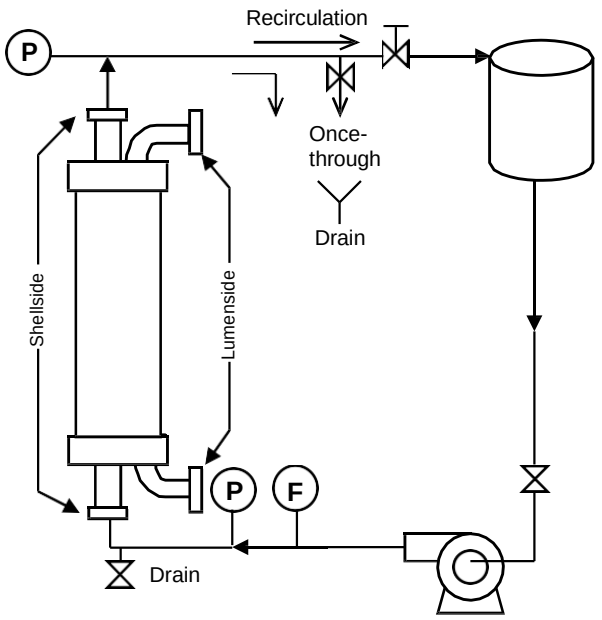
If silica, aluminum or a combination of these is found in the inlet water source, it is likely that they will precipitate on the membrane surface. If CO_2 is used as a sweep gas, precipitation can occur depending on the concentration and the water pH shift. For aluminum precipitation follow the mineral deposit removal procedure. For silica precipitation refer to the **Biological Soil Removal Procedure, section VI**, but increase the caustic concentration to 5.5% by weight and increase the temperature to 50 ºC. 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 Mineral Deposits

F = Flow Indicator

P = Pressure Gauge

⋈ = Valve



VIII. Cleaning Protocol When Particle Fouling Is Suspected

⚠ 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 www.3m.com/liqui-cel.
- If cleaning the shellside of the membrane only, the lumenside should be open to atmosphere.

To reduce the risks associated with chemical exposure:

- Use face protection, protective gloves, and appropriate eye protection when handling and using all cleaning chemicals.
- Ensure any tubing or piping used to circulate the cleaning solution is not blocked, kinked, frayed, cracked or obstructing flow in any way. Ensure the all tubing and piping connections are leak-free.
- Avoid splashing of chemicals while preparing cleaning solutions or during pumping of solutions.
- Cleaning procedures should be conducted using the minimal temperatures and pressures required to clean the contactor.

⚠ CAUTION

To reduce the risks associated with damaging the membrane contactor:

- For all 2.5x8-inch, 4-inch and 10-inch membrane contactors, gas side pressure should never exceed liquid side pressure during cleaning.
- Do not expose the membrane to air when liquid or gas temperatures exceed 30° C.

NOTICE

- Avoid contact with surfactants/solvents or oxidants (e.g. ozone, chlorine) to the hydrophobic membrane to prevent wet-out or oxidation.
- Filtered, de-chlorinated, and deionized water is recommended for mixing cleaning solutions. Water containing metals such as Mg, iron, Al, and SiO_2 may precipitate on to the membrane surface when there is a pH shift in the water.

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

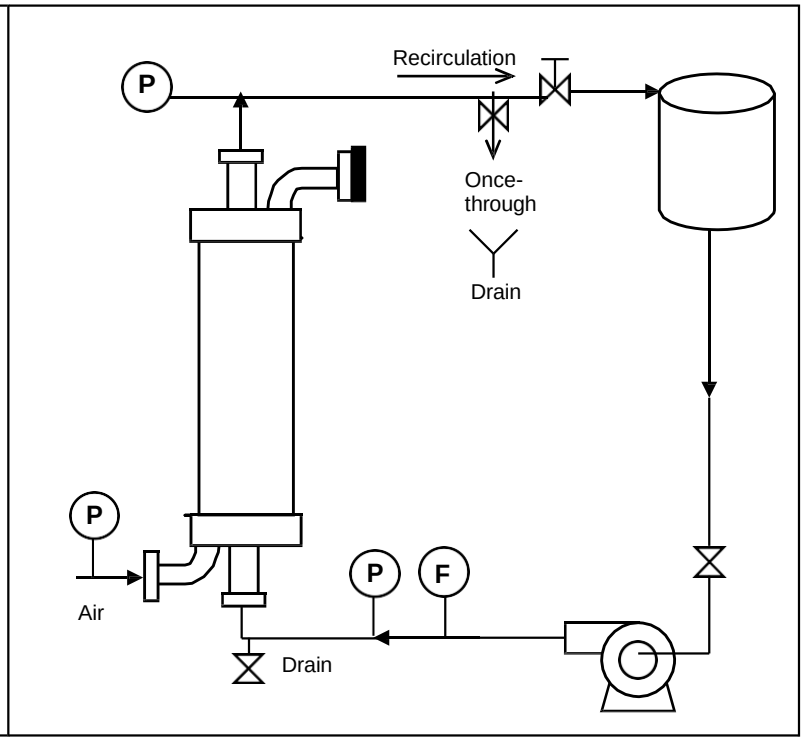
- Backflush the cleaning solutions (i.e. introduce the cleaning solutions in the direction opposite of the normal operating flow direction).
- Once the cleaning solution is flowing into the contactor, introduce clean, dry, and oil free compressed air into one gas port and in the same direction as the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

Illustration 4

F = Flow Indicator

P = Pressure Gauge

⋈ = Valve



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 www.3m.com/liqui-cel.

⚠ CAUTION

To reduce the risks associated with damaging the membrane contactor:

- For all 2.5x8-inch, 4-inch and 10-inch membrane contactors, gas side pressure should never exceed liquid side pressure during cleaning.
- Do not expose the membrane to air when liquid or gas temperatures exceed 30° C.

The drying process involves two steps:

A. Bulk Water Removal
B. Final Drying

Bulk Water Removal quickly removes liquid from the contactor prior to passing the drying gas through. 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 is not suggested for 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 20. 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

Liqui-Cel® Membrane Contactor Size	Gas Flow Rate scfm*
MiniModules and 2 x 6	0.5 scfm (0.84 m^3/hr)
2.5 x 8	3 scfm (1.7 m^3/hr)
4 x 13	10 scfm
4 x 28, 6 x 28, 8 x 20	20 scfm (17 m^3/hr)
8 x 40	40 scfm
8 x 80	80 scfm
10 x 28 and 14 x 28	50 scfm (120 m^3/hr)
14 x 40	100 scfm

*Maximum gas pressure = 10 psig (0.7 kg/cm^2)

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

Liqui-Cel® Membrane Contactor Size	Gas Flow Rate*	Estimated Drying Time
2 x 6	0.5 scfm (0.84 m^3/hr)	1 hr @ 60 ºC (140 ºF)
2.5 x 8	1 scfm (1.7 m^3/hr)	1 hr @ 60 ºC (140 ºF)
4 x 13	5 scfm	4 hr @ 60 ºC (140 ºF)
4 x 28	10 scfm (17 m^3/hr)	4 hr @ 60 ºC (140 ºF)
6 x 28	25 scfm (40 m^3/hr)	8 hr @ 60 ºC (140 ºF)
8 x 20	20 scfm	8 hr @ 60 ºC (140 ºF)
8 x 40	30 scfm	16 hr @ 60 ºC (140 ºF)
8 x 80	60 scfm	24 hr @ 60 ºC (140 ºF)
10 x 28	40 scfm (120 m^3/hr)	16 hr @ 60 ºC (140 ºF)
14 x 28	50 scfm (130 m^3/hr)	24 hr @ 60 ºC (140 ºF)
14 x 40	100 scfm	24 hr @ 60 ºC (140 ºF)

* Maximum gas pressure = 10 psig (0.7 kg/cm^2)

C. Drying Schematics

Illustration 5: Bulk Water Removal/Initial Drying Step

F = Flow Indicator
P = Pressure Gauge
⋈ = Valve

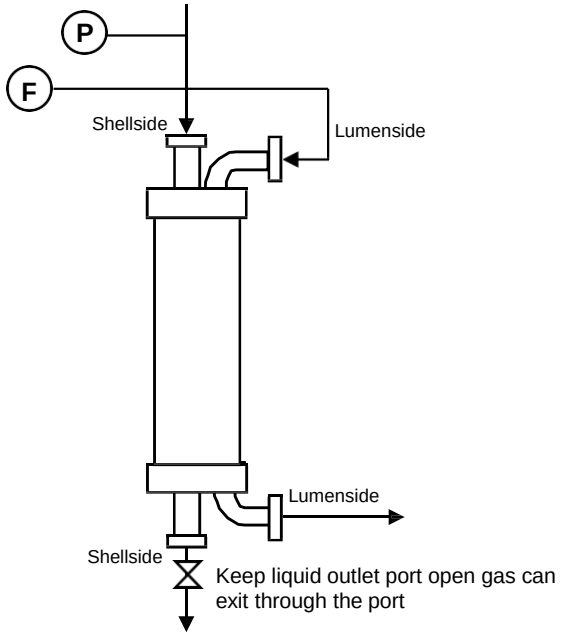
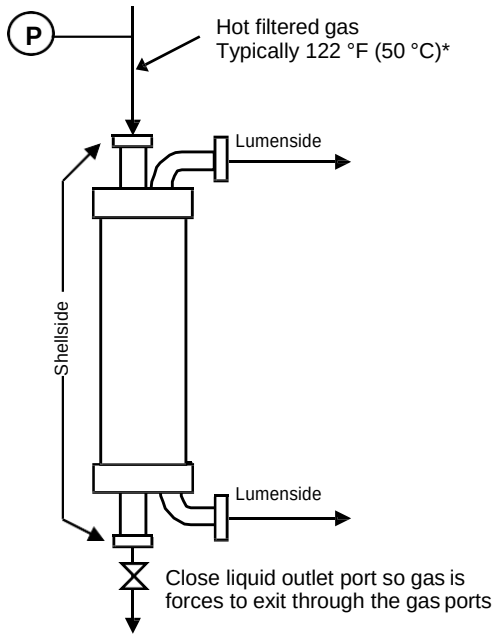


Illustration 6: Final Drying

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/cm^2) 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 protocol 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 Lumen side Port

Fiber Type	**X50 Fiber**	**X40 Fiber**	**XIND**
Condensation Rate Per m^2 of active membrane area)	0.8 to 1.6 ml/hr	0.2 - .4 ml/hr	0.8 to 1.6 ml/hr

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 Position: Store the contactors in the horizontal position. Ten inch contactors with SS housings are packaged in wooden crates. Fourteen inch, 10 inch FRP, 8 x 20 inch and 6 inch contactors are packaged in cardboard boxes. 8 x 40 inch and 8 x 80 inch contactors are individually bagged, then cradled on pallets. When storing or stacking contactors care should be taken to secure the contactors and ensure stability 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.

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 (704) 587-8888 to obtain an RMA form. Complete the form and return it by email to your representative or fax to (704) 587-8585, Attn: 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 6.0. 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 protocol. 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 protocol is recommended: (5.25% available chlorine) diluted 1:500 with filtered water (final concentration = ~100ppm 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 100ppm 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 shell and tube side 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 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.
Fax to: (704) 587-8585, Attn: Liqui-Cel Membrane Contactor Technical Service or email the form to your 3M representative.

Product Use: Many factors beyond 3M's control and uniquely within user's knowledge and control can affect the use and performance of a 3M product in a particular application. Given the variety of factors that can affect the use and performance of a 3M product, user is solely responsible for evaluating the 3M product and determining whether it is fit for a particular purpose and suitable for user's method of application.

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