



User Guide

3M™ Liqui-Cel™ Membrane Contactors for TransMembrane ChemiSorption Operation

For the following products:
MM-0.5×1 Series for TMCS
EXF-2.5×8 Series for TMCS
EXF-8×20 Series for TMCS
EXF-14×28 Series for TMCSz

[3M.com/Liqui-Cel](https://www.3m.com/Liqui-Cel)

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Note: While membrane contactors can be utilized in many applications with a variety of compatible fluids, this User Guide is written for ammonia gas removal (abatement) from aqueous solutions using an acid to absorb and chemically convert ammonia. This process is referred to as **Transmembrane Chemisorption (TMCS)**. Contact your 3M representative if you have questions that are not answered in this guide.

I. Safety Information

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

Intended Use and Product Selection:

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

3M Liqui-Cel Membrane Contactors may further be used in the production of water for various pharmaceutical products upstream of the final water sterilization step

Certain limited Liqui-Cel Membrane Contactors may further be used in Transmembrane Chemisorption (TMCS) processes where the removed gasses are absorbed into a liquid phase and converted by chemical reaction.

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

Restrictions on Use:

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

If your process may result in dangerous concentrations of explosive, flammable, toxic or oxidizing liquids or gases in the membrane contactor or the system, contact 3M prior to use.

Explanation of Signal Word Consequences	
⚠ WARNING	Indicates a hazardous situation which, if not avoided, could result in serious injury or death.
⚠ CAUTION	Indicates a hazardous situation which, if not avoided, could result in minor or moderate injury.
NOTICE	Indicates a situation which, if not avoided, could result in product or system damage.

Read entire product manual. Failure to follow all product instructions and warnings could cause personal injury and/or property damage.

⚠ WARNING

- **To reduce the risks associated with liquid bursting or gas explosion and/or exposure to chemicals and membrane contactor damage:**
 - Do not introduce gas alone into the membrane contactor without liquid in the shell side except when following 3M storage guidelines.
 - Do not exceed maximum operating pressure or temperature limits.
 - Implement workplace safety risk controls according to local applicable laws and regulations.
 - Always use appropriate personal protective equipment (PPE) when installing, servicing, operating, cleaning or disposing of the membrane contactor.
 - All plumbing should be done in accordance with local regulations and code.
 - To prevent buildup of pressure inside the membrane contactor, do not block or valve off all gas/vacuum ports during operation or downtime.
 - Ensure chemically compatible materials of construction are used within system.
 - Always make sure to verify proper connections within the membrane contactor system.
 - Never modify or alter the membrane contactor. Only 3M or parties authorized in writing may make changes/repairs to the equipment.
 - Inspect membrane contactor prior to cleaning or installation. Only use replacement parts supplied by 3M for this product.
 - Inspect the membrane contactor to ensure no leaking, cracking, or other signs of damage on membrane contactor, gaskets and tubing or piping
- **To reduce the risks of asphyxiation [or other health hazards], accidental gas explosion, or environmental contamination:**
 - Ensure proper system ventilation and discharge of any gases being used in or generated during membrane contactor operation, cleaning and drying, (including sweep gas, vacuum pump, or blower discharge,) according to all applicable building codes and regulations.
- **To reduce the risks associated with fire and explosion:**
 - Do not introduce explosive, flammable, toxic or oxidizing liquids or gases in dangerous concentrations into the membrane contactor or the system.
 - Over-pressurization of liquid and gas should be prevented by the installation of proper pressure relief valves/safety systems.
- **To reduce the risks associated with acid spraying or leakage and property damage:**
 - Proper safety equipment, such as flange covers and/or skid enclosures or shielding, must be installed in the facility to protect against possible spraying from system components, bursting or release of acid under pressure.

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

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

CAUTION

- **To reduce the risks associated with hot surfaces and hot exhaust gases:**

- Do not touch the membrane contactor or liquid lines during operation or cleaning and drying. Surfaces may be hot.
- Avoid close proximity to blower exhaust

- **To reduce the risks associated with environmental contamination:**

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

- **To reduce the risks associated with damaging the membrane contactor:**

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

NOTICE - To reduce membrane contactor or system damage:

- Care must be taken not to drop, hit or impact the membrane contactor.
- If the membrane contactor is used with air sweep, then the temperature should not exceed 35°C (95°F). For membrane contactors used with vacuum only this statement does not apply.
- For all membrane contactors, lumen side pressure should never exceed shell side pressure during cleaning and operation. Always refer to operating and cleaning guidelines for the use application.
- To avoid contamination of the process fluid, gloves are recommended when handling the membrane contactors.
- All plastic port extensions should be supported to prevent bending of extensions under excessive piping loads.
- 3M™ Liqui-Cel™ Membrane Contactors should be stored dry and in a sealed plastic bag or shrink wrap material to help prevent the introduction of contaminants into the membrane contactor.
- 3M Liqui-Cel Membrane Contactors should be stored in their original box, or other opaque box, and should not be installed where they are exposed to direct sunlight.
- Store 3M Liqui-Cel Membrane Contactors dry at temperatures < 50 °C (122 °F), but preferably at lower temperature such as <35 °C (95 °F), to not risk reduced lifetime. Membrane contactors should always be stored above freezing temperatures, and if stored at low temperature, they should be allowed to equilibrate to room temperature before use.
- Do not use thread sealant to connect fittings to membrane contactor.
- Use care if using a metal fitting to connect to a plastic connector on the membrane contactor.
- Do not allow membrane contactors containing microporous hollow fiber membranes to come into contact with surfactants, oil, or organic solvents, such as pure alcohols, glycol, acetone, etc., to reduce the risk of membrane wet out. SP-series membrane contactors containing polyolefin membrane are not subject to this restriction.
- To protect the membrane contactors, prefiltration equipment should be inspected and maintained in accordance to 3M™ Liqui-Cel™ Membrane Contactors Inlet Water & Sweep Gas Operating Guidelines, in the Technical Resources section at 3M.com/Liqui-Cel.
- Suspended solids, biological contaminants, or the precipitation of soluble or insoluble salts on the membrane surface may lead to membrane plugging.
- Filtered, de-chlorinated, and deionized water is recommended for mixing cleaning solutions. If a pH shift occurs water containing sparingly soluble compounds of Ca, Mg, Fe, Al, and silica (SiO₂) etc. could precipitate from the solution and block or damage the membrane. Ensure that your water is free of these compounds.
- Cumulative exposure of the membrane to oxidants, such as ozone, chlorine, hydrogen peroxide, peracetic acid, etc., should be restricted to reduce the risk of membrane oxidation.
- Avoid water hammer (sudden pressure spikes) in system.

ATTENTION:

Disposal

At end of life, dispose of the membrane contactor or cartridges in accordance with all applicable local and government regulations.

Hazards from Chemicals

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

II. Technology Overview

3M™ Liqui-Cel™ Membrane Contactors add gases to or remove dissolved gases from compatible liquid streams without dispersion. The dissolved gas can be transferred from liquid water and desorbed into a gas stream or absorbed into another liquid stream.

A membrane contactor can contain thousands of either microporous or dense asymmetric hollow fibers that are placed inside a contactor housing and are arranged with uniform spacing to allow for high flow capacity and utilization of the total membrane surface area. Because the hollow fiber membrane is hydrophobic, aqueous liquids will not penetrate the membrane pores. Unlike dispersed-phase contactors, such as packed columns, membrane contactors provide a constant interfacial area for transfer over the entire range of flow rates.

Table 1: 3M Liqui-Cel Membrane Contactor Characteristics for TMCS

TMCS Module Summary
<ul style="list-style-type: none"> • Four-port module design configuration for two liquid stream operation • Chemically resistant components suitable for wide range of pH-values • Array of product sizes available to meet different flow rates, from feasibility testing to full-scale production

Visit our web site at 3M.com/Liqui-Cel or contact your 3M sales representant for more information on applications and available product options.

A. 3M Liqui-Cel Membrane Contactor Description and Design

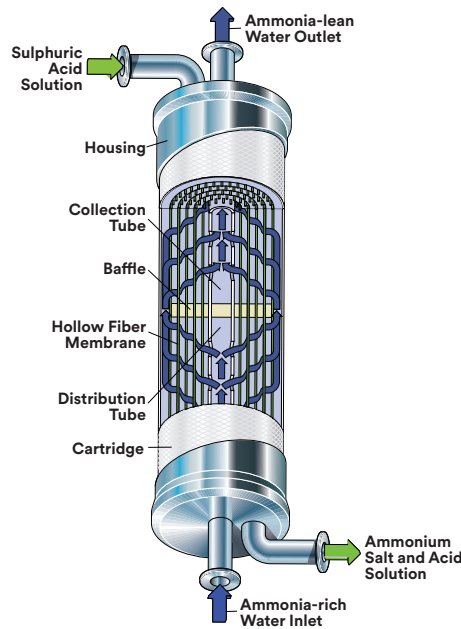
Regardless of the membrane type and the membrane contactor variant used, the separation principle in 3M Liqui-Cel Membrane Contactors differs substantially from other membrane separations such as filtration and gas-gas separation. With 3M Liqui-Cel Membrane Contactors there is no convective flow through the hollow fiber membrane pores. Instead, the membrane acts as an inert support that brings the liquid and gas phases into direct contact without dispersion. The transfer between the liquid and gas phases is primarily governed by the partial pressure difference of each gas species between the two phases.

The degassing surface area per unit volume in a membrane contactor is an order of magnitude higher than traditional technologies such as packed columns, forced draft deaerators and vacuum towers. This greater surface area-to-volume ratio leads to a reduction in system size at a comparable level of performance. there is no need to disperse one fluid phase into another, and there is no risk of flooding or non-uniform flow distribution. In addition, the in-line operation under pressure eliminates or reduces the need for transfer pumps or booster pumps after the membrane contactor.

B. Hollow Fiber Membrane Type

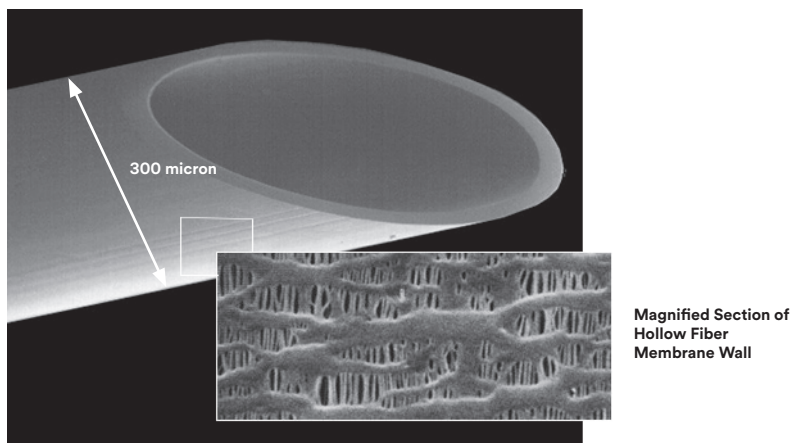
Liqui-Cel Membrane Contactors can be offered with different types of membranes. The EXF-series membrane contactors for water applications typically use symmetrical microporous polypropylene hollow fiber membrane. See Figure 2. These membranes are hydrophobic and non-selective.

Figure 1: 3M™ Liqui-Cel™ Membrane Contactor EXF Series for TMCS Operation



Our 3M Liqui-Cel Membrane Contactors offer a center baffle design for optimized flow distribution, also called (EXF) design. See Figure 1. For TMCS operation the releasing phase enters the inlet of the contactor into a distribution tube and flows over the outside of the hollow fiber membrane (shell side) around the central baffle. The releasing phase flows further into a collection tube and exits outlet of the contactor in a degassed state. The hollow fibers are open from one end to the other and allow the receiving phase to flow in countercurrent direction at the inside of the hollow fiber membrane (lumen side).

Figure 2: Scanning Electron Microscope (SEM) Images of Hollow Fiber Membrane



Among the fiber options available in 3M Liqui-Cel Membrane Contactors, the X50 fiber is recommended for TMCS because of its high porosity, reduced wall thickness and large inner diameter. These characteristics contribute to increasing the mass transfer, lowering the mass transfer resistance and lowering the pressure drop for lumen liquid.

III. Important Information on Product Protection

A. Chemical Concentration Guidelines

Unless pH of the feed water is already high, bases are typically added (e.g. 50% NaOH) to raise pH of the feed water for enhanced release of ammonia gas to be transferred across the microporous hydrophobic membrane. Concentrated acids are typically used (i.e. 96% H₂SO₄) to make-up the acid recirculation tank solution, which is pumped through the contactor. But there are limits to the recommended maximum acid concentration, as shown in Table 2.

Table 2: Chemical Selection and Maximum Concentrations (at 50°C) to Contactor Inlet

Chemical	Molar Concentration	Percent % (wt.)
Sulfuric Acid	≤ 5M	≤ 50
Phosphoric Acid	≤6M	≤ 60
Hydrochloric Acid	< 10 M	≤ 37
Citric Acid	<2.5 M	< 40
Nitric Acid	DO NOT USE	
Caustic Soda	<10 M	<32

B. Membrane Oxidation

Exposing the hollow fiber membrane inside a 3M™ Liqui-Cel™ Membrane Contactor to oxidizing species may reduce the life of the contactor. Prior to operation, a risk assessment to identify any potential risk of membrane exposure to oxidizing agents is strongly recommended. It is the sole responsibility of the purchaser and operator to review all potential risks.

There are many possible causes of membrane oxidation induced by various species in the liquid streams. For reference, some common oxidizing species are listed in Table 3 below along with their relative oxidative strengths. Increased temperature will accelerate oxidation by these species.

Table 3: Oxidative Potential of Different Species in Water

Oxidizing Species	Relative Oxidative Strength (eV)
F ₂	3.10 eV
OH·	2.80 eV
O·	2.42 eV
O ₃	2.07 eV
H ₂ O ₂	1.70 eV
Cl ₂	1.36 eV

Note: The above list is not all-inclusive

Our recommendation is to remove free chlorine, peroxides, ozone, and any other oxidizing species from water prior to the 3M Liqui-Cel Membrane Contactor.

Common examples of oxidation risk

Peroxides and ozone in water - the presence of peroxides and ozone are also important considerations. They are used extensively as disinfectants, germicides and to destroy dissolved oxidizable carbon in many water purifications processes. If these species are present in the liquid flowing through the membrane contactor, they will oxidize the membrane over time.

Chlorine - water and or liquids flowing through the contactor may contain high levels of chlorine. Chlorine may also cause the membrane hollow fibers to oxidize. It is recommended to remove all chlorine from liquids flowing through the contactor.

Trace dissolved metals, such as iron and nickel, will act as a catalyst in the presence of chlorine or other oxidizing agents. The result is rapid oxidation of the membrane, especially at warm water temperatures.

C. Surfactants and Organic Compounds

Avoid contact with surfactants and organic solvents with low surface tension (e.g. alcohols, grease, oils) when using microporous polypropylene membrane. These will render the membrane hydrophilic. The membrane will need to be cleaned and dried before the contactor can be put back into service.

Because the membrane is hydrophobic it generally acts as barrier to passage of dissolved non-volatile substances in water. But any volatile component will be able to pass across the gas-filled pores into the acid stream.

IV. TMCS Mode for Ammonia Abatement (Removal)

The **Transmembrane Chemisorption (TMCS)** is a specific operating mode for membrane contactors and can be used for various gas transfer applications. In this mode, using ammonia as an example, ammonia is physically removed (desorbed or stripped) from a feed water, diffuses across the membrane wall (trans-membrane), and is then absorbed into and chemically reacts with (chemical-sorption) a liquid acid stream. The process is also known as ammonia abatement. The process is shown as a single hollow fiber illustration in Figure 3.

The two aqueous liquid phases are separated by the wall of the hydrophobic microporous membrane. the feed water containing ammonia flows outside (shell side) of hollow fibers. An acid solution (pH<1-2) flows in counter-current direction inside (lumen side) the hollow fibers. Ammonia gas passes through the air-filled pores and chemically reacts with an acid (e. g. sulfuric acid) to form ammonium salt. The driving force for ammonia transfer is the ammonia concentration differential between feed water phase and acid phase.

Figure 3: Cutaway of a Single Microporous Hollow Fiber Showing TMCS of Ammonia

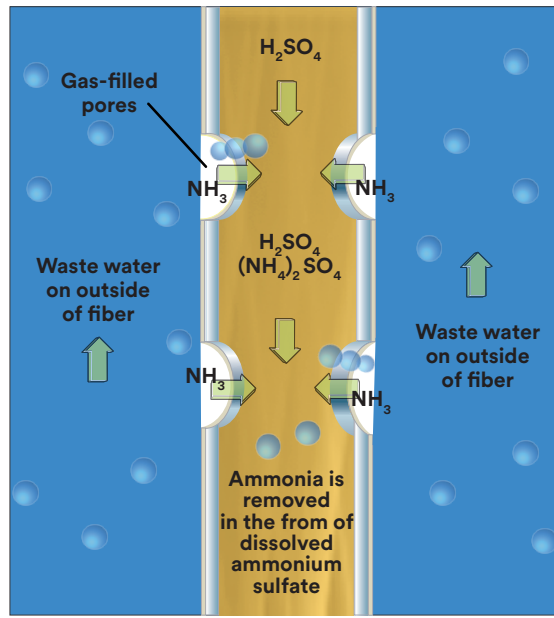
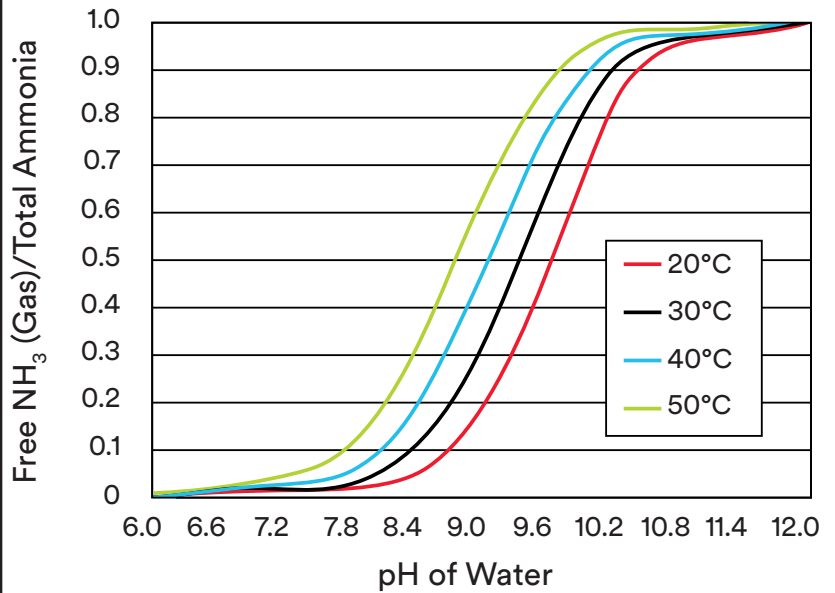


Figure 4: Ionization Equilibrium of Free NH₃(g) in Water as Function of pH and Temperature



Reaction of ammonia with sulfuric acid to form ammonium sulfate is depicted in Equation 1, Table 4.

Table 4: Ammonia Reaction with Sulfuric Acid and Ammonia Dissociation in Water

$2\text{NH}_3 + \text{H}_2\text{SO}_4 \rightarrow (\text{NH}_4)_2\text{SO}_4$	Equation 1: Full reaction of ammonia with sulfuric acid
$\text{NH}_3(\text{g}) + \text{H}_2\text{O} \leftrightarrow \text{NH}_4^+(\text{aq.}) + \text{OH}^-$ High pH Products Low pH Products	Equation 2: Ammonia dissociation in water; depends on pH value

The amount of free ammonia gas in water is a function of pH and temperature, as shown in Figure 4. Ammonia solutions at pH >10.5 and >35°C contain nearly 98% free ammonia NH₃(g), which can diffuse through the pores of each hollow fiber. Free ammonia NH₃(g) is a gas and when present in water, it dissociates into its ionic species, called ammonium NH₄⁺ ion, per Equation 2. Only NH₃(g), but not NH₄⁺ ion, can diffuse across the microporous membrane. To drive the formation of free NH₃(g) it may be necessary to increase the pH of feed water by adding a base such as sodium hydroxide (NaOH). The amount of base needed will depend upon the initial pH level and the temperature of the feed water.

As in other gas transfer applications, ammonia removal is influenced by the feedwater temperature. The gas solubility decreases, and the gas diffusivity increases with higher temperature. Higher the temperature of the feed water, higher the transfer rate of ammonia. But at the same time, at higher temperature water vapor transfer rate will also increase, causing a dilution effect in the acid phase.

In TMCS process, the ammonia transfer driving force across membrane increases with increasing free ammonia concentration in feed water, all other parameters remaining the same. As a result, ammonia transfer rate is more favorable at higher inlet ammonia concentration and less favorable at very low inlet ammonia concentration.

The acid solution helps maximize the ammonia driving force. For example, a sulfuric acid solution almost instantaneously reacts with ammonia gas to form ammonium sulfate, making ammonia concentration effectively zero in acid phase. The maximum theoretical ammonium sulfate concentration for TMCS is the saturation solubility of the salt, which is 43.5g/100g water at 20°C. However, the highest achievable ammonium sulfate concentration in TMCS process will also depend upon the inlet ammonia level in wastewater, inlet acid concentration, and amount of transferred ammonia and water vapor. Oversaturation or salt precipitation at the acid-membrane interface, due to local concentration gradients, should be avoided since it will form scales on membrane surface and can even block the flow in the hollow fiber lumen. The choice of acid will determine the reaction product (salt) as shown in Table 5.

Table 5: Examples of Acid Types and Reaction Products with Ammonia

Acid type	Formula	Products	Formula
Sulfuric	H ₂ SO ₄	Ammonium sulfate, ammonium bisulfate	(NH ₄) ₂ SO ₄ , NH ₄ HSO ₄
Phosphoric	H ₃ PO ₄	Ammonium phosphate and ammonium hydrogen phosphates	(NH ₄) ₃ PO ₄ , (NH ₄) ₂ HPO ₄ , NH ₄ H ₂ PO ₄
Hydrochloric	HCl	Ammonium chloride	NH ₄ Cl

For practical reasons there are some general considerations regarding ammonia removal using TMCS process. Table 6 below lists of the optimum ranges for the most important operating parameters. The actual parameters may be different due to local requirements and situations.

Table 6: Ideal Process Parameter for TMCS of Ammonia

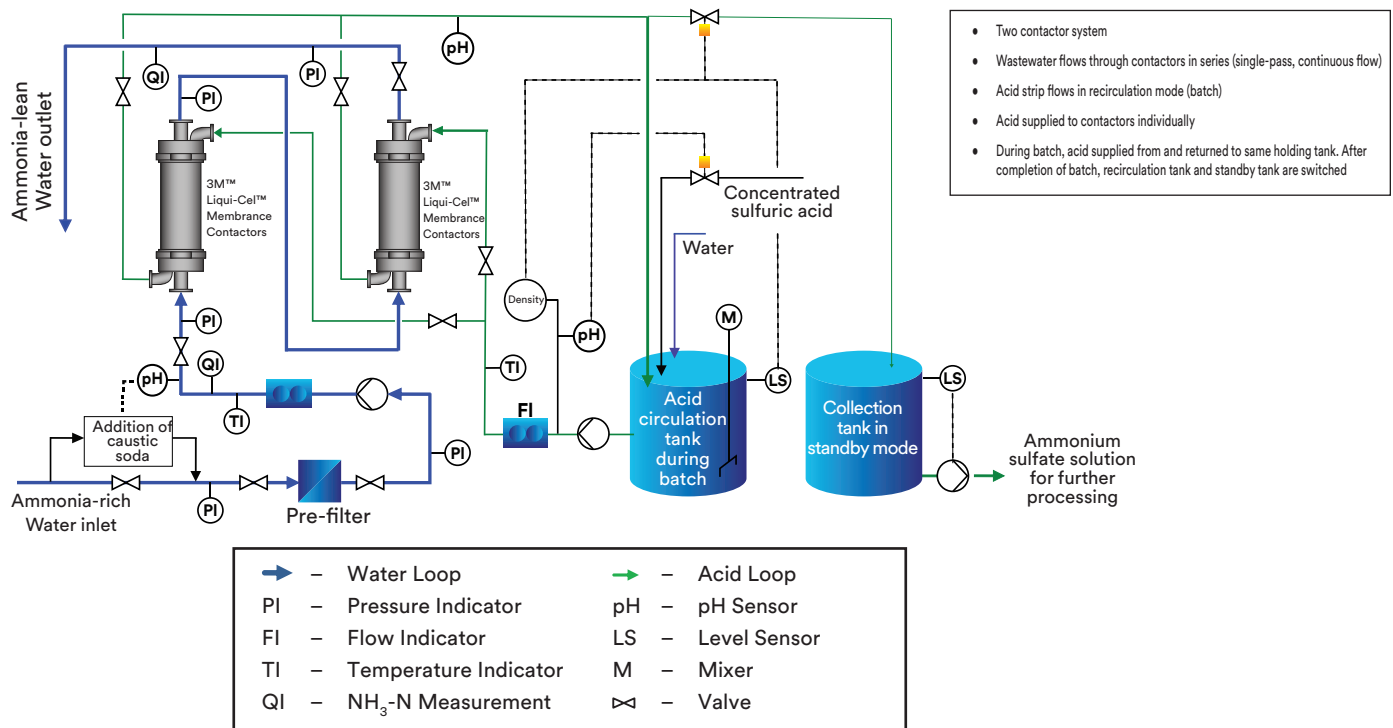
Operating parameter	Recommended Level	Unit
NH ₃ (g) inlet concentration	> 200	ppm (mg/L)
Wastewater pre-filtration (absolute)	<5 absolute	µm
Temperature range	>35-50 (95-122)	°C (F)
Wastewater stream pH	≥ 11	-
Acid stream pH	≤1-2	-

V. General System Design Guidelines

A. Flow Pattern Configurations

A schematic process diagram of a typical plant for TMCS of ammonia is shown in Figure 5. For each contactor, the wastewater flows from bottom to top through the shell side of vertically installed membrane contactor. The acid solution flows through the lumen side of contactor from top to bottom. Feed water flows through the contactors in series, while acid should always be fed to contactors in parallel. The ammonium salt solution is pumped from circulation tank in batch mode. If the maximum level of the circulation tank is reached, the solution is redirected to the collection tank.

Figure 5: Generic P&ID with Two Membrane Contactors in Series for TMCS of Ammonia



Successful operation of a 3M™ Liqui-Cel™ Membrane Contactor system in the TMCS mode depends on a well-designed fluid path. It is important to follow these recommendations when designing a system:

Piping

- Do not overtighten threaded or flanged connections.
- Select appropriate materials for aggressive media. Avoid piping materials that may not be compatible with corrosive acids and bases.
- Threads, pipe sealant, and pipe tape should be used with extreme caution to prevent leaking. Any leak will cause risk to operational safety.
- Avoid long runs of piping and loops. Minimize the use of elbows and other sources of pressure loss.
- Design flow manifolds to handle the load of the entire flow.
- Ensure ability to drain at lowest point in piping. We recommend vertical mounting the membrane contactors. Only vertically mounted membrane contactors facilitates drainage of shell and lumen side.

Pump Type and Sizing

- Contact a 3M representative to estimate the related pressure drops in feed water and acid phases based on liquid volume flow rate and membrane contactor size. This will determine the size of the required pumps.
- A pump with corrosion resistant materials is recommended. There are many types and brands of pumps; choose the one that satisfies your needs.
- The chemical dosing pumps should be designed to handle the aggressive media in required concentration and volume. Filling, operation, and maintenance require special attention from authorized and trained personal.

B. Recommended System Instrumentation

Pressure gauges (isolation valves are optional)

- Near contactor wastewater inlet
- Near contactor wastewater outlet
- Acid inlet
- Acid outlet

Flowmeters

- Acid inlet
- Feed water inlet

Pressure Relief Valve and Alarm

- Acid inlet (discharged to safe location w/alarm)
- Feed water inlet

Valves

- Inlet and Outlet Isolation valves for feed water
- Inlet and Outlet Isolation valves for acid
- Manual flow control valves, optional
- Low point drain valve on feed water line
- Low point drain valve on acid line
- Drain valves between contactors (optional)

Pressure switches

- Feed water inlet high pressure switch to shut down or warn operator
- Acid inlet high pressure switch to shut down or warn operator

Temperature measurement

- Feed water inlet
- Acid inlet

Determination of Series and Parallel Configuration

Each type of 3M™ Liqui-Cel™ Membrane Contactor has recommended minimum / maximum feed water and acid flow rates, determined by the hydraulic limit of each contactor size. For system flow rates that exceed the individual membrane contactor's maximum flow rate, it is advisable to split the flow into parallel trains. To determine a first indication of minimum number of parallel trains, divide the total system feed water flow rate by the maximum flow rate provided in Table 7. The actual number of parallel trains and membrane contactors in series is determined by the required gas removal performance and liquid pressure drop.

After calculating the minimum number of parallel trains, additional membrane contactors can be added in series to obtain the desired dissolved gas concentration at system outlet. The number of membrane contactors in series will be a function of the required ammonia outlet concentration and the maximum allowable system pressure drop, specified by customer. If the maximum allowed system water pressure drop is reached, one can add additional parallel trains of membrane contactors to achieve lower ammonia outlet and lower system pressure drop.

The typical recommended liquid flow rates for TMCS of ammonia listed in Table 7 are guidelines. The ratio of acid to feed water flow rates is generally in the range of 0.5 to 1.5 depending upon the acid concentration and pH value. The acid concentration/pH should be monitored since water vapor transfer into the acid side dilutes the acid stream.

Table 7: Typical Recommended Liquid Flow Rates for TMCS

3M Liqui-Cel Membrane Contactor	Feed water stream (shell side)	Acid stream (lumen side)
0.5×1	5-30 mL/min	5-30 mL/min
2.5×8	20-100 L/hr	20-100 L/hr
8×20	0.25-2.0 m ³ /hr (1-9 gpm)	0.25-2.0 m ³ /hr (1-9 gpm)
14×28	5-20.4 m ³ /hr (22-90 gpm)	2-9.1 m ³ /hr (9-40 gpm)

Liquid-Stream Configuration

The following are guidelines for designing a TMCS process with 3M Liqui-Cel Membrane Contactors.

- Vertical mounting of contactor is recommended since only vertical orientation allows for self-draining of each stream.
- It is recommended that feed water flows in upward direction from bottom of contactor to top of contactor.
- Flow directions of feed water and acid must be countercurrent to each other.
- If the feed water pressure coming into the Membrane Contactor system is greater than the maximum operating pressure, a pressure regulator is strongly recommended. For maximum pressure ratings and maximum operating temperature refer to the table 8.

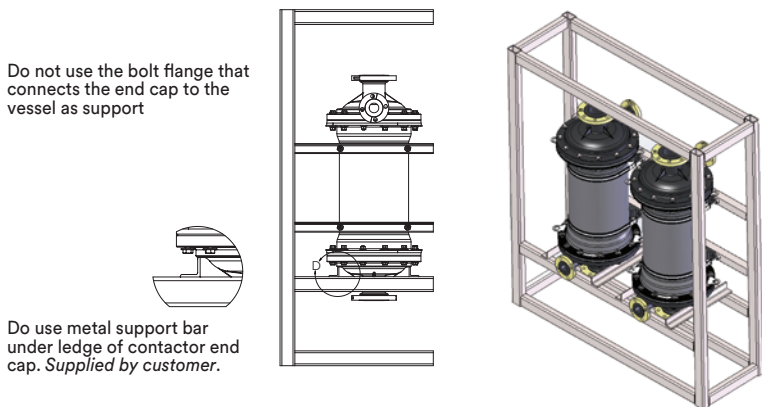
- To avoid damage from water hammer, always use slow closing valves on the downstream side of the membrane contactor system.
- Feed water side low-point drains, pressure and temperature indicators should be included in the design.

It is generally recommended that the membrane contactors not be operated when the ambient air temperature is below freezing. During shutdowns, the membrane contactors must be protected from freezing temperatures. Protection options include placing system in a heated environment or electric heat tracing. If a system (after draining) is transported by airfreight or by truck during freezing temperatures, it is necessary to dry the membrane contactors prior to shipment. Refer to the *Liqui-Cel Cleaning and Storage Guidelines*.

Additional Information for Vertically Mounting of 14x28 Membrane Contactors

It is recommend using 3M mounting kits and an additional steel bar support under the membrane contactors to adequately support the full weight of the 14x28 membrane contactors. Do not place the membrane contactor load on the bolt flange that is used to secure the end caps onto the housing. Also, do not place the membrane contactor load on the piping materials. Please refer to the 14x28 vertical skid mounting drawings in Figure 6. These will provide you will more detailed information. A basic view of the metal support that is located under the bottom end caps is depicted below.

Figure 6 Supporting 14x28 Membrane Contactors



C. Maximum Operating Temperature and Pressure Guideline

There are two pressure ratings that operators and system builders should pay close attention to when operating and installing membrane contactors: a shell side (liquid) housing (or vessel) pressure rating and a lumen side (acid) pressure rating.

Note: The shell side **feed water outlet** pressure can be higher or lower than **acid inlet** pressure if the pressure differential is low (<1 bar). However, if acid pressure is higher, and there is a membrane leak, then acid could leak into the feed water phase. Never exceed pressure/temperature limits provided in this guide and on contactor data sheets.

The maximum recommended operating pressures and temperatures for different contactors are listed in Table 8 for 3M™ Liqui-Cel™ membrane contactors for ammonia TMCS application. **Note that for all membrane contactor sizes, higher operating temperatures may reduce membrane contactor service life.**

IMPORTANT NOTE: In most cases, liquid flows on the shell side (outside the fibers) of the membrane contactor and the acid stream flows on the lumen side (inside the fibers) of the membrane contactor. If conducting a pressurized gas test to check for system and seal integrity on a dry membrane contactor during installation and first start-up, **DO NOT** exceed the pressure limits given for the lumen side as gases will pass through the membrane during the test when liquid is not present. It is important not to over pressurize the gas side as the end caps have a lower pressure limit than the vessel in most cases.

Table 8: Membrane Contactors Pressure/Temperature Limits while Operating in TMCS Mode

Contactor size:	0.5x1		
Feed Water (shell side) Maximum Pressure Limits			
5-40 °C (41-104 °F)	Less than 1 bar gauge		
Acid side (lumen side) Pressure Limits			
5-40 °C (41-104 °F)	Less than 1 bar gauge		
Contactor size:	2.5x8	8x20*	14x28*
Feed Water (shell side) Maximum Pressure Limits			
5-50 °C (41-122 °F)	3.1 barg (45 psig)	3.1 barg (45 psig)	3.1 barg (45 psig)
Acid side (lumen side) Pressure Limits			
5-50 °C (41-122 °F)	3.1 barg (45 psig)	3.1 barg (45 psig)	3.1 barg (45 psig)
*European Union Pressure Equipment Directive (2014 / 68 / EU) Group 1 liquid			

The acid side pressure limits may need to be derated depending upon the acid selection. To obtain the maximum acid side operating pressure, divide the pressure from Table 8 by the derating factor in Table 9.

Example: using H₂SO₄ with 14×28 contactor

Acid side pressure rating from Table 8 = 3.1 bar

Derating factor = 1.1

New Operating Pressure = 3.1 bar / 1.1 = 2.8 bar at 50C

Table 9: Membrane Contactor Pressure Derating Factors

Contactor	Derating Factors, 50C		
	H ₂ SO ₄ (<50%)	H ₂ PO ₄ (<60%)	HCl (25%-30%)
2.5×8	1.1	1.4	1.75
8×20 IND	1	1	1
14×28	1.1	1	1

D. Membrane Contactor Weights for System Fabrication

The system framework should be designed to support the maximum liquid-full weights of the membrane contactors. The weights listed in Table 10 are for one membrane contactor so the total number of membrane contactors will need to be taken into consideration when designing the skid support structure.

Table 10: Membrane Contactor Weight

3M™ Liqui-Cel™ Membrane Contactor	Housing / Endcap Material	Dry weight	Weights with both shell and lumen sides water-filled
		Kg (lbs.)	Kg (lbs.)
0.5×1	HD Polyethylene	7.9 grams	18.6 grams
2.5×8	Polypropylene / Polypropylene	0.5 (1.1)	0.9 (1.9)
8×20	PVC / Engineered Thermoplastic	12 (27)	25 (54)
14×28	PVC / Engineered Thermoplastic	54 (118)	113 (249)

E. Filtration Requirements

The inlet shell and lumen streams need to be pre-filtered. The proper filtration arrangement depends on the water source so a comprehensive water quality analysis should be completed. The optimal filtration and pre-treatment arrangement will depend on several variables, including the water source, operating conditions, biological matters, dissolved organics, Turbidity, Total Suspended Solids (TSS), Total Hardness, Silica, Fat, Oil and Grease (FOG), Total Dissolved Solids (TDS) and other factors. A small filtration pilot system can reveal valuable information to be used to design the final pre-filtration system. 3M produces a complete line of filtration products, contact your local 3M sales representative for further details.

Tables 11 and 12 provide the minimum recommended filtration guidelines to prevent potential membrane fouling and blockage due to particles which will create a high pressure drop across the membrane contactors and restrict the flow through the membrane contactor system.

The potential for a pH shift caused by the removal or addition of certain chemicals, may help prevent fouling of the membrane surface or may cause scaling. Consult your water treatment specialist to answer these questions.

Feed water should be free of surfactants/solvents or oxidants (e.g. hydrogen peroxide, ozone, chlorine) to prevent wet out or oxidation of the hydrophobic membrane. Small amounts of chlorine and oil can be removed by activated carbon. Biological fouling can be reduced with regular, frequent chemical cleaning or sanitization procedure. See *Liqui-Cel Cleaning and Storage Guidelines* for additional information).

Table 11: Inlet Water Quality Guidelines

Quality Indicator	Units	Recommended Level	Prevention / Control	Shell side Cleaning
Turbidity	NTU	< 0.5	flocculation/UF/NF/RO	no treatment
Colloids	Silt Density Index (SDI)	< 3	flocculation/UF/NF/RO	no treatment
Total Suspended Solids	ppm (mg/L)	<5	flocculation/UF/NF/RO	no treatment
Particle Size	Micron, absolute rating [1]	≤5 for typical particles [3]	Pre-filtration	Back washing (not for carbon black fines)
Suspended Oil	ppm	≤ 10	filtration	(hot) caustic
pH	Units	0.5 – 14		-
Silica – Colloidal	ppm	< 10	antiscalants	hot caustic
Surfactants	ppm	0		water/alcohol (>25%) cleaning & drying
Total Hardness [2]/ Langelier Saturation Index (LSI)	ppm/Langelier Saturation Index	10 ppm ≤ 0 LSI	softening/antiscalants	Acid cleaning

[1] Do not use Nominal rated filters, only Absolute rated filtered, recommended 99.9% particle removal efficiency (Beta 1000).

[2] Total Hardness along with LSI is needed to predict if the water will scale. Total Hardness alone is not enough.

[3] Special care should be taken to flush to drain the carbon bed thoroughly after each filling of the carbon bed before TMCS start-up

Table 12: Acid Inlet Quality Guidelines

Quality Indicator	Units	Recommended Level	Prevention / Control	Lumen side Cleaning
Total Suspended Solids	ppm (mg/L)	< 5	Pre-filtration	Flushing
Particle Size	Micron, absolute rating [1]	≤ 5 for typical particles [2]	Pre-filtration	Flushing
Surfactants	ppm (mg/L)	0		water/alcohol (>25%) cleaning & drying

[1] Do not use Nominal rated filters, only Absolute rated filtered, recommended 99.9% particle removal efficiency (Beta 1000).

[2] Special care should be taken to flush to drain the carbon bed thoroughly after each filling of the carbon bed before TMCS start-up

F. Membrane Fouling and Scaling

Feed water may contain species that are soluble in water in acidic pH ranges but insoluble in alkaline pH ranges. If water pH increases as the water passes through the membrane the insoluble species might precipitate on the membrane surface. Precipitation of solids can also occur when water is treated with flocculation chemicals followed by a change in pH. The thin coating or deposits will prevent normal gas transfer through the membrane and the membrane contactor's removal efficiency will drop. This process is reversible by cleaning the membrane contactor with an acid solution (refer to the *Liqui-Cel Cleaning and Storage Guidelines*). The acid solution may dissolve surface precipitates on membrane and will restore the contactor degassing performance to original manufacturer's specifications.

When the contactors are installed downstream from carbon beds, the beds should be flushed to drain after the carbon change-out until all carbon fines are flushed out.

A chemical CIP (Clean-In-Place) system is recommended to clean and restore the performance of the Liqui-Cel Membrane Contactors. Please refer to the *Liqui-Cel Membrane Contactor Cleaning and Storage Guidelines* available on-line at 3M.com/Liqui-Cel or from your 3M representative.

VI. Startup, Shutdown and Downtime Procedures

A. General instructions

The TMCS system has four components:

- Acid circulation
- Feed water stream
- Acid addition
- Caustic addition

Note: Mixing in the acid circulating tank is suggested to avoid precipitation or sedimentation of ammonium sulfate and ensure a homogenous solution.

Refer to Figure 5 for equipment layout.

B. Initial Start-up

- Make sure that all connections are tight
- Open all water valves except the inlet valve to contactors
- Fill acid circulating tank with sufficient volume of water for filling the piping and membrane contactors and open all valves to the acid circulation pump and each module.
- Turn on circulation pump for complete filling and venting of the acid circulation system
- Slowly open water inlet valve to the system to introduce feed water to the system. Making sure that water inlet pressure and water flow rate through the membrane contactor never exceed the maximum operating limits.
- Adjust feed water flow rate to the desired levels by adjusting the appropriate valves on the system.
- Make sure there are no leaks.

C. System Start-up

- Ensure acid circulation and wastewater flow pumps are operating properly. Then start acid dosing pump.
- Wait for pH to reach 1-2 before turning on the caustic dosing pump
- Monitor process conditions (flow, pressure, pH, temperature)

D. Shutdown Procedure

- Stop caustic dosage and wastewater flow
- Stop acid circulation and acid addition
- Leave the lumen side (acid) valves open. Do not close these valves during shutdown. This avoids pressure build-up in the lumen side of the membrane contactor.

E. Downtime Guidelines

Composition of the feedwater will determine how one should handle the membrane contactors for downtimes longer than 3 days. During downtime the wastewater side should be flushed with demineralized water. We recommend wastewater side to remain filled with clean high-pH water (pH > 11). The acid pumps should be stopped and the acid outlet valve should be open to the tank to prevent pressure buildup on acid side because of osmosis (water from feed water side drawn into the acid side).

VII. Maintenance

Maintaining a system performance Log is recommended for 3M™ Liqui-Cel™ Membrane Contactors. Recording data is required to trouble shoot the system so a weekly or bi-weekly data log is required. The performance log should record critical information as shown in the example below.

Table 13: Typical Maintenance Log

Date	Operator	Feed water parameters				Acid parameters				System Performance		
		Feed water flow rate	Feed water temperature, Inlet / Outlet	Feed water pressures, inlet / outlet	Feed water pH, inlet / outlet	Acid flow rate	Acid temperature, inlet / outlet	Acid pressures, inlet / outlet	Acid pH, inlet / outlet	NH ₃ or NH ₄ + concentration at system water inlet	NH ₃ or NH ₄ + concentration at system water outlet	

This log will show the user a pattern of consistent performance or a decline in performance over time. If the contactors are still performing to the stated performance, no maintenance is required. If a decline in performance is noted, it may be time to clean the membrane or replace. Refer to the Troubleshooting section below for further details.

VIII. 3M™ Liqui-Cel™ Membrane Contactor FAQ

What factors affect service life?

The expected service life of 3M Liqui-Cel Membrane Contactors varies depending on many factors. It is important to note that membrane system designs can vary considerably. Membrane contactor lifetime can be affected by water quality, system performance specifications and operating conditions.

One must separate membrane failure and system performance issues before deciding when to replace membrane contactors. System design (i.e.- number of membrane contactors) is determined by the design engineer and depends upon the safety factor used. So, the number of contactors needed to meet system performance can vary. Therefore, the replacement time for each system design will vary since the system performance begins at different points. For example, a system designed with excess capacity may not be as sensitive to system performance degradation compared to systems designed without extra capacity.

Membrane contactor life is affected by water temperature, oxidation, particle, and organic fouling (water quality) and frequency of cleaning.

What is the allowable operating temperature range for TMCS process?

Recommended continuous operating temperatures for 3M™ Liqui-Cel™ Membrane Contactors in TMCS applications typically range from 5-50 °C (41 – 122 °F).

TMCS uses acid streams which undergo a temperature increase because of heat of reaction. Take the heat of reaction rise into consideration for setting inlet feed water temperature. Operating at higher temperatures may reduce membrane contactor service life: a membrane contactor in a system operating at 25 °C (77 °F) may last longer than in a system operating at 50 °C (122 °F). Exceeding the membrane contactor’s temperature operating limit is likely to permanently damage the membrane contactor.

What are the maximum allowable chemical concentrations?

It is required that concentrations be kept within operating limits. See Table 2 earlier in the document.

What factors influence membrane oxidation

Oxidation is a complex process that is a function of the oxidizer chemistry and concentration, temperature, and concentration of dissolved metals. The hollow fiber membranes have various tolerances to oxidizing species, but the presence of oxidizers may reduce service life. Exposing the hollow fiber inside the membrane contactor to oxidizing species could cause hollow fiber degradation and/or modify properties of the hollow fiber surface. These changes may allow water to pass through the membrane, and in extreme cases, may make the hollow fiber brittle resulting in reduced system performance or even membrane contactor failure. The presence of a dissolved material, such as soluble iron, could accelerate the oxidation reaction due to catalytic action.

Exposure to oxidizing species, such as free chlorine, UV radiation, chlorine dioxide, oxygen at high temperature in the feed water will reduce service life. Refer to Tables 10 and 11: Water and Acid Inlet Quality Guidelines.

Cleaning chemicals may also reduce service life. See the *Liqui-Cel Cleaning and Storage Guidelines* at 3M.com/Liqui-Cel.

To help reduce risk of oxidation, it is recommended to minimize exposure to high liquid temperatures. Chemicals used for Clean-In-Place (CIP) processes should also be thoroughly reviewed to ensure the chemicals do not contain oxidizing species.

See section III-B.

How do you control particulate blockage of the membrane contactor?

Influent water to membrane contactors should be pre-filtered to remove insoluble particulates. The minimum pre-filtration requirement is 5-micron absolute at 99.9% particle removal efficiency. Insoluble particles can build-up over time and clog up the fluid flow channels inside the membrane contactors, increasing pressure drop. Insoluble or colloidal silica could cause increased pressure-drop. Refer to Tables 11 and 12.

What could cause biofouling or organic fouling?

Biofouling inside the membrane contactor can occur if the influent water has high levels of Biological Oxygen Demand (BOD) and Chemical Oxygen Demand (COD). Microbiological fouling can also occur if the system has long shutdown periods.

Organic compounds can also create biofouling. For example, oils, or other natural organic matter (NOM).

Please refer to the *Liqui-Cel Membrane Contactor Cleaning and Storage Guidelines* available on-line at 3M.com/Liqui-Cel or from your 3M representative.

What could cause membrane scaling?

Scale formation on the membrane surface is possible if the feed water contains high hardness and silica levels.

Please refer to the *Liqui-Cel Membrane Contactor Cleaning and Storage Guidelines* available on-line at 3M.com/Liqui-Cel or from your 3M representative.

What is the procedure to follow if I see system performance decline?

Compare all the following parameters versus the original system design.

- A significant change in water temperature
- A significant change in acid concentration
- Feed water flow rate

What do I monitor to determine if cleaning or a replacement is necessary?

Performance should be based on monitoring of outlet dissolved ammonia concentration.

Is liquid pressure-drop a good indicator for cleaning or replacement?

Pressure drops in the liquid phases may be suitable parameters to determine if cleaning is required. Refer to the Liqui-Cel Cleaning and Storage Guidelines at 3M.com/Liqui-Cel for more details. However, cleaning or replacement decisions are best determined from both (1) system-outlet ammonia levels, and (2) pressure drop in the first contactor in the feed water line and pressure drop in acid lines.

How do I check for chemical compatibility?

For general guidelines, contact your 3M representative.

What precautions should I take when using contactors in plastic vessels?

Plastic vessels should not be subjected to sudden temperature and pressure changes. To prevent premature failure of the thermoplastic fittings, accurate alignment and proper support of those connections is critical. Pipe that is not properly supported will put additional stress on the plastic fittings, which can lead to a fitting failure. Use proper plastic pipe support guidelines when installing plastic vessels.

IX. 3M™ Liqui-Cel™ Membrane Contactor Storage, Handling and Operating Environment

3M Liqui-Cel Membrane Contactors can be damaged through improper handling and storage. If you have any questions refer to *Liqui-Cel Cleaning and Storage Guidelines* at 3m.com/Liqui-Cel or contact your 3M representative.

Container/packaging: 3M Liqui-Cel Membrane Contactors should be stored dry and in a sealed plastic bag or shrink wrap material to help prevent the introduction of contaminants into the contactor.

Temperature: Store 3M Liqui-Cel Membrane Contactors dry at temperatures < 50°C (122°F) but preferably at lower temperature such as <35°C (95°F) to not risk reduced lifetime. Membrane contactors should always be stored above freezing temperatures, and if stored at low temperature, they should be allowed to equilibrate to room temperature before use.

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

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

Store the membrane contactors in the horizontal position. 10×28-inch membrane contactors with stainless steel housings may be packaged in cardboard boxes or wooden crates. 14×28-inch and 8×20-inch membrane contactors are packaged in cardboard boxes. Membrane contactors should be stored in a safe location where they are not at risk of falling, being crushed or impacted. Always ensure the membrane contactor, and any systems using membrane contactors, are stable, level, and properly secured. Be sure the membrane contactors/system cannot tip, roll, fall, slide or make any movement that may cause injury, damage to the unit, or damage to other system components.

X. Troubleshooting

Various contactor performance problem descriptions, probable causes and suggested corrective actions are listed in Table 14.

Table 14: Troubleshooting Guidelines

Problem Description	Probable Cause	Corrective Action
Outlet ammonia concentration above specification	Membrane contamination/fouling	Clean contactor. Refer to <i>Liqui-Cel Cleaning and Storage Guidelines</i> .
	Liquid temperature below design specification	Raise temperature or add more contactors.
	Liquid flow rate higher than design specification	Reduce flow rate or add more contactors.
	Low acid flow rate or concentration	Increase acid flow rate. Use higher acid concentration
	Unequal liquid flow through contactor trains	Verify train flow rate. Adjust valves accordingly to equalize flow rates.
Increase in feed water and/or acid pressure drops	Particulate accumulation on shell and/or lumen side	Check filters and replace if needed.
		Flush contactors. Refer to <i>Liqui-Cel Cleaning and Storage Guidelines</i> at 3m.com/Liqui-Cel .
		Replace contactors.
Sudden increase or decrease of pH in feed water or acid outlets	Seal O-rings may not be sealing	Contact a 3M representative.
	Membrane leakage	Perform a contactor integrity test. Refer to <i>Cleaning and Storage Guidelines</i>
	Surfactants or oils/fats may have been introduced into contactors, leading to membrane wet out	Drain and remove all liquids. Rinse, clean and dry the contactors. Refer to <i>Cleaning and Storage Guidelines</i> .

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