

DuPont Water Solutions

DuPont™ Ligasep™ Degasification Modules Technical Manual

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1 Introduction

1.1 Introduction to Degasification

1.1.1 How gases get into water

Nitrogen (N_2) and Oxygen (O_2) naturally dissolve into water that is in contact with air. Air is made up of mostly N_2 and O_2 and whenever a gas is in contact with water it will dissolve into water. A glass of water will contain around 8.5 ppm of oxygen and 14.1 ppm of nitrogen because it is in contact with the atmospheric air.

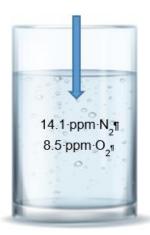


Figure 1. Ambient air composition at atmospheric pressure 79% N_2 and 21% O_2

Carbon dioxide (CO_2) gas present in water will originate from ambient CO_2 in air that will dissolve into water as well as from common minerals such as calcium carbonate ($CaCO_3$) and magnesium carbonate ($MgCO_3$) that can dissolve in water. These minerals will dissolve into carbon dioxide, calcium and magnesium ions. The amount of carbon dioxide dissolved into the water will depend on how much calcium carbonate and magnesium carbonate the water source has come in contact with as well as the pH and temperature of the water.

1.1.2 Oxygen and Water

Oxygen (O_2) is a strong oxidant that can oxidize certain metals that it comes in contact with within a water system. For this reason, there is a need to remove dissolved O_2 from water to protect any equipment that may become oxidized and damaged. Two major applications are impacted by the presence of oxygen reacting with metals, which affect various industries:

- 1. Any process that includes the use of steam boilers, which are found in the Power Generation industry but as well in the chemical, petrochemical and heavy industries in general,
- 2. Production of microelectronic components such as in the Semiconductor Manufacturing Industry.

1.1.3 Carbon Dioxide and Water

Water purity is often measured by its ability to conduct electricity. Ions in the water will allow the water to conduct electricity. Ultrapure water will have a very low conductivity indicating it has very few or virtually no ions in the water. Carbon dioxide will ionize and increase the conductivity of water.

The CO₂ reacts and dissolves in water as follow:

$$CO_{2(q)} + H_2O_{(aq)} \leftrightarrow H_2CO_{3(aq)} \leftrightarrow H^+ + HCO_{3(aq)}$$

Ion exchange will remove ions and can be used to remove carbon dioxide. As the level of carbon dioxide increases it becomes more economical to remove the carbon dioxide using a mechanical method rather than ion exchange. It is common to install a decarbonator (also known as a degasser) to remove dissolved carbon dioxide from water and improve the efficiency of the anionic resin.

Table 1. Typical dissolved CO₂ targets per application

Application	Typical Target Dissolved CO ₂ *
Anionic Resin	< 5 ppm
Ion Exchange Mixed Bed	< 5 ppm
Ultrapure Water	<1 ppm

^{*} Values may vary depending on the requirements of the project.

1.1.4 Hydrogen Sulfide and Water

Some natural water sources, such as brackish waters, are in a reduced state typically due to lack of oxygen (therefore referred to as anoxic or anaerobic) and the presence of iron, manganese, ammonium and/or hydrogen sulfide (H_2S).

 H_2S in ground water aquifers usually occurs at concentrations of 0.5 to 5 mg/L as the result of the dissolution of minerals in geologic deposits or as anaerobic bacterial activity on organic sulfur, elemental sulfur, sulfates and sulfites. The amount of sulfide dissolved in water is pH dependent as shown in the following equations:

$$H_2S + H_2O \Leftrightarrow H_3O^+ + HS^- pK_1 = 7.0$$

 $HS^- + H_2O \Leftrightarrow H_3O^+ + S_2^- pK_2 = 14.0$

 H_2S levels as low as 0.1 mg/L can adversely affect the performance of reverse osmosis or nanofiltration systems as H_2S can be oxidized and precipitate as elemental sulfur or metallic sulfides. The precipitates can foul the membranes as a black sooty or gray pasty substance. It can also corrode equipment such as pumps and valves.

 H_2S gas can be easily detected as it usually creates an odor described as the smell of rotten eggs and can represent a health risk to the population depending on the concentration and exposure time.

DuPont[™] Ligasep[™] Degasification Modules can be an excellent treatment option for H_2S removal. In order to have an efficient removal, the sulfide must be present as H_2S gas, being pH dependent, it means that the pH needs to be ≤ 6.0 before feeding into the Ligasep[™] module.

1.1.5 Ammonia and Water

Ammonia is a colorless inorganic compound of nitrogen and hydrogen with the formula NH_3 , usually in gaseous form with a characteristic pungent odor. Ammonia is irritating to the skin, eyes, nose, throat, and lungs.

 NH_3 reacts and dissolves in water forming the corresponding ionic species named ammonium (NH_4^+), in a pH dependent equilibrium, as follow:

$$NH_{3(q)} + H_2O \leftrightarrow NH_{4(qq)}^+ + OH^-$$

To remove ammonium from water through degasification, the process would include first the increase of the pH through the addition of NaOH (or another base) to ensure the equilibrium is fully displaced to the gas species. The gaseous NH_3 would transfer through the membrane. An option to safely manage NH_3 , which is a harmful gas, is made it react directly in the lumen side of the fiber with mineral acid to form a diluted ammonium salt solution. This ammonium salt could be the base of a fertilizer.

Applications:

- Removal of NH₃ in flue gas in Biomass Power Stations
- Industrial applications where process water waste containing NH₃ is discharged to sewer system.
- Pig, dairy, chicken farms
- Semiconductor plants

1.2 Degasification Methods

1.2.1 Thermal Method

Generally, the solubility of a gas in a liquid is lower at higher temperatures, therefore, heating an aqueous solution can expel dissolved gas. Ultrasonication and stirring can be used to improve the efficiency of the process. Although the process is simple, the gas removal process can be highly variable.

It is also important to consider that heating a solution can change the composition of the liquid and even decompose the solute. It can also catalyze a reaction between dissolved compounds and evaporate a portion of the liquid.

1.2.2 Chemical Method

This process consists in removing dissolved gases via chemical reaction.

1.2.2.1 CO₂ Removal

In the case where a project requires decarbonation, one method would be to inject caustic soda (NaOH) into the water in order to increase the pH. The increase in the pH will convert any dissolved carbon dioxide gas into bicarbonate and carbonate anions. These ions can then be removed by an additional process.

1.2.2.2 O₂ Removal

If oxygen should be removed, it is common to use reducing agents or scavengers to control dissolved oxygen in water. For example, ammonium sulfite is frequently used as a reductant because it reacts with oxygen to form sulfate ions. Although this method can be applied only to oxygen, the dissolved oxygen is virtually completely eliminated.

1.2.3 Physical Methods

1.2.3.1 Mechanical Deaeration

Mechanical deaeration can be accomplished in a packed tower. In a packed tower water is sprayed into the top of the tower and flows over trays or packing material. The water flows through the tower in thin films that increase the gas liquid contact area and improves the efficiency. The tower is designed to operate under a vacuum (to lower the total pressure of the gas mixture) or with a sweep gas (replace the gas in contact with water with another gas). When removing carbon dioxide air is often used to maintain a carbon dioxide free gas in contact with the water. In the case of carbon dioxide, the gas solubility is very high, so the air must be continuously replaced to assure that no carbon dioxide is in the gas in contact with the water.

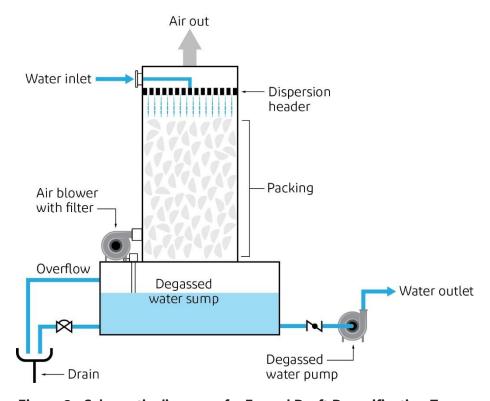


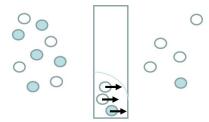
Figure 2. Schematic diagram of a Forced Draft Degasification Tower

Some examples of conventional deaerators are:

- Decarbonators
- Air or gas strippers
- Thermal deaerators
- Packed column deaerator
- Vacuum deaerator
- Air scrubbers

1.2.3.2 Membrane Degasification

A membrane is considered a selective barrier or interface between two phases, which has the ability to transport one component more readily than other because of differences in physical or chemical properties between the membrane and the permeating components. The transport of solutes or solvents through the membrane is a consequence of the driving force acting on them and the characteristics of the membrane to allow or resist their transport.



High Potential Membrane Low Potential

Figure 3. Schematic diagram of a two-phase system separated by a membrane

The performance of a membrane process will depend on:

- Driving force
- Properties of the membrane
- · Fluid properties
- Hydraulics

Table 2. Membrane processes comparison

Membrane Process	Phase 1	Phase 2	Driving Force
Microfiltration	Liquid	Liquid	Pressure
Ultrafiltration	Liquid	Liquid	Pressure
Nanofiltration	Liquid	Liquid	Pressure, Concentration
Reverse Osmosis	Liquid	Liquid	Pressure, Concentration
Membrane Degasification	Liquid	Gas	Partial pressure
	Liquid	Liquid	Partial pressure

There are different driving forces that are used in membrane processes. The most common in water treatment is the use of pressure in reverse osmosis systems for desalination. In the case of membrane degasification, the driving force is the partial pressure difference of the gases across the membrane, as explained by Henry's Law and Dalton's Law (refer to Section 1.3 for more details).

The membrane used in $DuPont^{TM}$ Ligasep TM is a hydrophobic hollow fiber that shows different permeability coefficients to gas species and prevents liquid from flowing across to the gas phase. The decrease of the partial pressure of a gas in the gas side will increase the driving force that favors the transport of that gas from the liquid side to the gas side.

The large area per volume, as can be found in hollow fiber modules, make this process more attractive than conventional deaerators. For example, packed degasification columns have surface areas ranging from 30 to 300 m^2/m^3 whereas membranes degasification modules have a surface area of 1600 to 6600 m^2/m^3 .1

In Ligasep $^{\text{m}}$ membrane degasification modules, liquid flows on the outside of the fiber while the sweep gas flows inside the fiber.

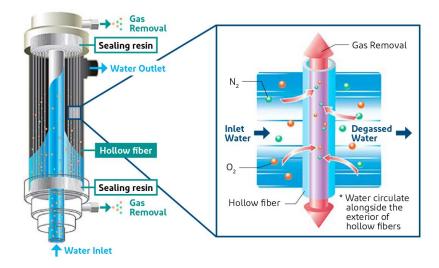


Figure 4. Operation of membrane degasification modules

1.3 Degasification Principles

In order to understand the mechanics behind gas removal, it is important to review three chemical engineering principles:

- Henry's Law
- Dalton Law of Partial Pressure

These principles are simplified below:

1.3.1 Henry's Law

When a gas is in contact with a liquid, it dissolves until an equilibrium is established. The concentration of a gas into a liquid, under equilibrium conditions, is related to its partial pressure. This is governed by a chemical engineering principal called Henry's Law:

Membrane contactors: fundamentals, applications and potentialities page 130

$$P_i = H_i ullet C_i$$
 Eq. 1

Where,

- P_i gas partial pressure
- H_i Henry's constant
- C_i concentration of component gas (i) dissolved into the water

Figure 5. Gas-liquid equilibrium

Henry's constant (H_i) can be calculated using the following equation:

$$H\left(T
ight)=H^{o}$$
 $ullet$ $\exp\left[rac{-\ sol}{R}\left(rac{1}{T}-rac{1}{T^{o}}
ight)
ight]$

Where,

H(T) Henry's constant at temperature T

H° Henry's constant at reference temperature (25 °C)

 $-\Delta_{sol}H$ Enthalpy of solution

T Temperature

T° Reference temperature (25 °C)

Note: Henry constant is specific for each gas component.

1.3.2 Dalton Law of Partial Pressure

In a gas mixture each gas exerts its own pressure or partial pressure. The total gas pressure is the sum of all the partial pressures in the gas mixture. The partial pressure of a gas is the total pressure times the % fraction of the gas in the gas mixture.

$$P_T = P_1 + P_2 + P_3 + \ldots = \frac{P_1}{Y_2}$$
 Eq. 3

Where,

 P_T total pressure of the gas mixture

P_i partial pressure of gas component (i)

Yi % fraction of the gas component (i) in the gas mixture

Example air is made up of 21% oxygen and 79% nitrogen. At one atmosphere oxygen exerts a partial pressure of 0.21 atmospheres and nitrogen 0.79 atmospheres. Oxygen and nitrogen will dissolve in water proportional to these partial pressures.

If we replace the air in contact with the water with nitrogen, the partial pressure of the nitrogen will increase to 1.0 atmospheres and the oxygen partial pressure will decrease to 0 atmospheres. Additional nitrogen will dissolve into the water and oxygen will subsequently move out of the water.

1.3.3 Degasification Methods

Combining equations 1 and 3, the concentration of a gas in water can therefore be calculated using the following equation:

$$C_i = rac{P_i}{H_i} = rac{(P_T ullet Y_i)}{H_i}$$
 Eq. 4

Where,

C_i concentration of component gas (i) dissolved into the water

H_i Henry's constant for gas component (i)

P_T total pressure of the gas mixture

 $Y_i\,$ % fraction of the gas component (i) in the gas mixture

Equation 5 can therefore be used to illustrate how degasification works. To reduce the concentration of the gas component in water (x_i) , it is important to reduce the partial pressure of the same gas component in the gas phase (p_i) .

The first method to do so is to reduce the overall gas pressure (P_T) . This can be done by applying a vacuum on the gas phase.

$$\downarrow C_i = \frac{(\downarrow P_T \cdot Y_i)}{H_i}$$

The second method is to reduce or eliminate the fraction of the gas component (y_i) in the gas phase. This is done by replacing the sweep gas used to degasification to a gas that does not have the component. For example, air is made up of 21% oxygen and 79% nitrogen. At one atmosphere of pressure, oxygen exerts a partial pressure of 0.21 atmospheres and nitrogen 0.79 atmospheres. Oxygen and nitrogen will dissolve in water proportional to these partial pressures.

If we replace the air in contact with the water with nitrogen gas, the partial pressure of the nitrogen will increase to 1.0 atmospheres and the oxygen partial pressure will decrease to 0 atmospheres. Additional nitrogen will dissolve into the water and oxygen will subsequently move out of the water.

$$\downarrow C_i = \frac{(P_T \cdot \downarrow Y_i)}{H_i}$$

The third method is to combinate the two method above. For example, to use nitrogen gas as a sweep gas under vacuum.

$$\downarrow C_i = \frac{(\downarrow P_T \cdot \downarrow Y_i)}{H_i}$$

These gas removal principles will be used to lower the partial pressure of the gas in contact with the water to remove the gas from the water.

The above formulas were used to illustrate the different method of degasification and are not meant to be used for system sizing. Please contact your DuPont Water Solutions representative for a basic process design of a degasification system using $DuPont^{TM}$ Ligasep TM .

1.3.4 Factors Affecting Membrane Degasification Performance

Gas removal percentage is the key performance parameter of a membrane degasification system. The gas removal percentage of a membrane degasification system are mainly influenced by variable parameters including:

- Water side
 - Water flowrate
 - Water temperature
- Gas side
 - Vacuum level
 - Composition of the sweep gas
- Number of modules in series
- Maintenance and operation

The following graphs show the impact of each of those parameters when the other three parameters are kept constant. In practice, there is normally an overlap of two or more effects. Figure 6, Figure 7, Figure 8 and Figure 9 are qualitative examples of membrane degasification performance.

1.3.4.1 Water flowrate

The percentage gas removal will vary depending on the contact time or retention time the liquid has inside the module. A longer contact time or retention time will allow for more time for the gas to permeate through the membrane resulting in a higher gas removal.

The contact time is inversely proportional to the flowrate. As the volume is set inside a degasification module, the higher the flowrate, the lower the contact time.

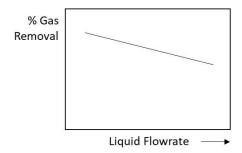


Figure 6. Percentage (%) gas removal behavior with liquid flowrate

1.3.4.2 Water Temperature

Liquid temperature impacts the performance of the DuPont™ Ligasep™ module in different ways. The most important being:

- Water density and viscosity
- Solubility of the gas in water
- Gas permeability through the membrane.

As a combination of these properties, the following behavior will be observed:

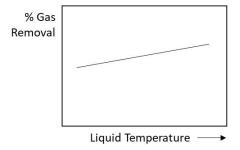


Figure 7. Percentage (%) gas removal behavior with temperature

It is recommended to consider the minimum water temperature to design a degasification system that meets your required performance.

1.3.4.3 Vacuum Level

As explained in Section 1.2.1, in order to reduce the concentration of the undesired dissolved gas in the liquid outlet, it is important to reduce the partial pressure of the sweep gas as much as possible. When operating at a deeper vacuum, closer to absolute vacuum, low levels of dissolved gasses can be reached.

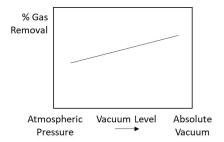


Figure 8. Percentage (%) gas removal behavior with vacuum level

1.3.4.4 Number of modules in series

As a two-pass reverse osmosis system is used to produce a very low conductivity product, the percentage gas removal can also be improved by having more than one module in series. In most applications, it is common practice to have two (2) modules in series to have a gas removal above 99%.

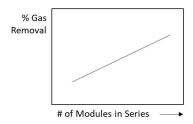


Figure 9. Percentage (%) gas removal behavior with number of modules in series

1.3.4.5 Maintenance and operation of the system

Outside DuPont Water Solutions scope of supply, some recommendations to ensure proper maintenance and operation of the DuPont™ Ligasep™ membrane degasification system are:

- Maintenance of auxiliary equipment. A properly maintained vacuum pump or blower will ensure that we achieve the adequate vacuum level and sweep gas flowrate. Please consult with your vacuum pump or blower supplier for more details on how to maintain your equipment.
- Maintain gas filter is clean to ensure proper sweep gas cleanliness, flowrate and pressure through the gas line.
- Monitor the pressure differential across the Ligasep™ module to ensure the fibers are not fouled. Fouling can
 damage and reduce performance of the system.
- Ensure the feed water is not in an oxidative state. Oxidants will damage the fibers of the Ligasep™ module.
- In the case your system has a water trap, ensure its emptied from water on a regular basis. The frequency will depend on the operating conditions of the system.

Please contact your equipment supplier for a detailed maintenance plan of the degasification system. Also review the rest of this manual for additional recommendations.

2 Product Information

2.1 Ligasep™ Degasification Modules Description

DuPont™ Ligasep™ Degasification Modules use a proprietary Polymethylpentene (PMP) hollow fiber membrane that provides an efficient transfer of gases between a liquid and a gas. These modules are ideal for deoxygenation, decarbonation, and gas control of liquids.

Ligasep™ Degasification Modules utilizes a hollow fiber membrane with a skin layer that reduces the passage of water vapor through the membrane. Low water vapor passage across the membrane reduces vapor condensate build up on the gas side and allows blowers and other vacuum pump technologies to be used to create a vacuum on the gas side of the membrane.

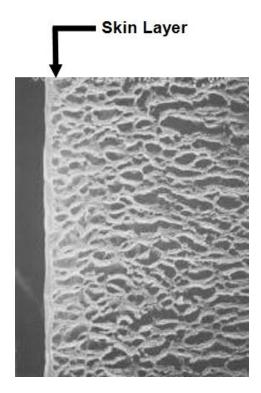


Figure 10. Fiber cross section

2.2 Fiber Types

There are two fiber types available:

- The "LS" fiber is typically used in applications with gases that have a low solubility in water, such as O_2 , and where high levels of removal are required.
- The "HS" fiber is designed for more efficient contact between the sweep gas and the liquid, which is ideal for gases that have a high solubility in water, such as CO₂, H₂S, and NH₃.

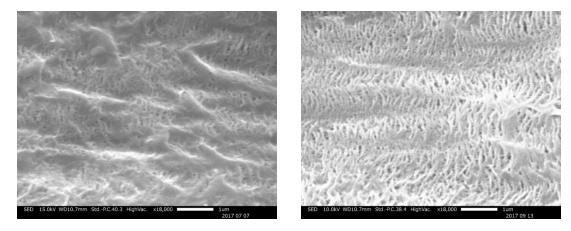


Figure 11. Surface of the LS fiber (left) and HS fiber (right)

2.3 Fiber Characteristics

Table 3. Fiber characteristics

Parameter	Value	
Fiber material	PMP	
Fiber outer diameter	180-240 µm	
Wall thickness	30-40 µm	
Gas permeability	High	
Water vapor transfer	Low	

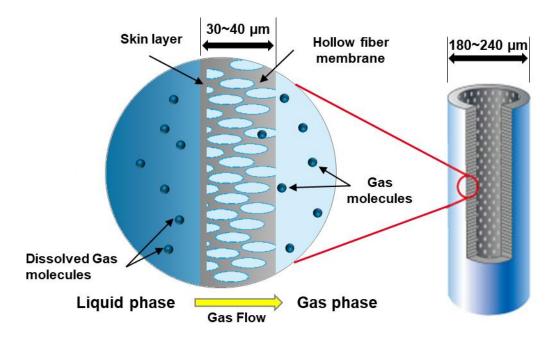


Figure 12. Characteristics of PMP fiber

2.3.1 Advantages to low water vapor transfer

Advantages for O2 removal

Low water vapor transfer across the skinned membrane allows the module to remove dissolved oxygen to very low levels under vacuum only. Operation under vacuum only eliminates the need to use a sweep gas to remove dissolved oxygen to very low levels. This allows the technology to be used in applications where nitrogen is not available.

Microporous (non-skinned) membranes allow water vapor to transfer through the pores adding a large load to a vacuum system. Large vapor loads prevent a deep vacuum from being applied to the gas side of the membrane.

Advantages for CO₂ removal

Low water vapor transfer across the skinned membrane prevents liquid water from accumulating on the gas side of the membrane. Elimination of water on the gas side of the membrane allows the vacuum to be drawn on the gas side using a regenerative blower instead of a liquid ring vacuum pump.

2.4 Membrane Degasification Advantages

There are several reasons to consider DuPont™ Ligasep™ Degasification Modules as an excellent choice for degasification as opposed to conventional technologies:

- Improved and more consistent product quality: the hollow fiber provides a barrier between the liquid and the
 gas, which will reduce the risk of cross contamination between them and offer consistent final product quality,
 hence improving process reliability.
- Smaller footprint and light weight: Ligasep™ modules have 10x the contact area compared to a conventional degasification tower. This can lead to a reduced cost of land acquisition, building design, and transport.
- Ease of design and operation: Ligasep™ modules offer much more stable water quality and production capacity than a conventional degasification tower.
- Modular design: Ligasep[™] degasification systems can be skid mounted and integrated as part of an existing or new system. Process design is less complicated, and control is more automated than with conventional pretreatment. Additionally, Ligasep[™] modules can be individually isolated for repair, maintenance or replacement without compromising the plant output.
- Lower environmental impact: membrane degasification does not require any chemicals for degasification. This can lead to lower chemical consumption and less environmental concerns for wastewater disposal.

2.5 Chemical Tolerance

Chemical agents are required in systems using $DuPont^{TM}$ Ligasep TM Degasification Modules for removal or control of inorganic, organic, and biological fouling in the membrane. However, in order to maximize the life of the modules and membranes, it is advisable to reduce as much as possible their exposure to those chemicals, and never exceed the maximum stated tolerance.

Table 4 provides information on the chemical tolerance of the Ligasep™ Degasification Modules for generic chemicals.

Table 4. Chemical tolerance

Chemical	Concentration	Endurance Time @ 25°C
Chlorine	10 ppm	1,000 h
	100 ppm	500 h
	300 ppm	60 h
	500 ppm	50 h
Hydrogen Peroxide	3.5 wt%	200 h
Hydrochloric Acid	3.5 wt%	200 h
Nitric Acid	3 wt%	900 h
Sodium Hydroxide	3.5 wt%	10,000 h
Citric Acid	3 wt%	10,000 h

Notes:

- Please do not use higher concentrations than specified in the data above.
- Tolerance time decreases under high temperature conditions.

3 Applications

3.1 Deionized water production

Water purity is often a measure of its ability to conduct electricity. Ions in the water will allow the water to conduct electricity. Deionized or ultrapure water will have a very low conductivity indicating it has very few or virtually no ions in the water. Any carbon dioxide present in the water will ionize and increase the conductivity of water.

Carbon dioxide in water is present as an equilibrium mixture of the dissolved gas CO_2 , the weak acid H_2CO_3 , and the bicarbonate (HCO_3^{-1}) and carbonate (CO_3^{-2}) anions associated with bicarbonate and carbonate alkalinity. The exact equilibrium ratio of these four species depends on the pH and temperature of the water as shown in Figure 13 below.

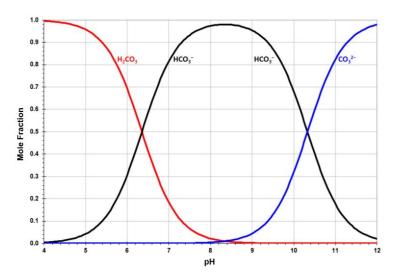


Figure 13. Ionization of carbon dioxide solutions as functions of the pH at 77°F (25°C)

There are several different processes to produce deionized water and CO₂ is a concern in all three:

- Ion exchange demineralization
- Double-pass reverse osmosis with an ion exchange mixed bed polisher
- Double-pass reverse osmosis with an electrodeionization system

3.1.1 Ion exchange demineralization

In an ion exchange demineralization system, HCO_3^{-2} and CO_3^{-2} alkalinity are converted to dissolved CO_2 and H_2CO_3 as they pass through the cation exchange resin bed due to a reduction in pH. As the water then enters the anion exchange resin bed, CO_2 is converted back to bicarbonate or carbonate anions, as the pH increases, which exchange onto the anion resin.

3.1.2 Double-pass reverse osmosis with an ion exchange mixed bed polisher

Typically, the pH of RO permeate is acidic with a pH range between 4-5. The low pH will convert any bicarbonate and carbonate alkalinity into dissolved CO_2 gas. If the CO_2 is not removed prior to entering the mixed bed polisher, it will be converted by the ion exchange system to bicarbonate and carbonate anions, which will exchange onto the anion resin in the mixed bed polisher.

In the cases above, depending on the CO₂ levels;

- Accounting for this load in new designs can result in large mixed bed polisher vessels which increase capital
 expenses.
- For existing systems that see an increase in CO₂, reduction in run length and increased chemical consumption can be observed, resulting in increased operating costs and water usage.

3.1.3 Double-pass reverse osmosis with an electrodeionization system

In an electrodeionization (EDI) system, CO₂ in the feed water will combine with hydroxide ions (OH⁻) (created by electrolysis) when it enters the dilute chamber.

 CO_2 is typically present in significant concentrations in RO permeate water and is removed in the EDI module through a combination of bicarbonate and carbonate anions.

Since CO_2 is nonionic it can diffuse back through the cation membrane into the diluting chamber and as such may need to be removed from the dilute chamber more than once.

Consequently, occurrence of CO_2 and alkalinity at more than the recommended values can greatly degrade EDI product water quality.

Removing CO₂ before the EDI system will allow for:

- Improve process reliability
- Increased lifespan of EDI modules
- Improved removal of weakly ionized species like Boron and Silica
- Improved EDI product quality
- · Improve system recovery
- Eliminate cross contamination from sweep gas compared to decarbonators.
- Eliminates the use of chemicals to remove dissolved CO₂ improving the permeate quality of the second pass Reverse Osmosis system.
- Cost effective operation

3.2 Ultrapure Water (UPW)

Ultrapure water (UPW) is essential to properly fabricate today's integrated circuits. It is the primary cleaning solvent used to rinse all contaminants and remnants of silicon etched away during the production process. The geometry of today's integrated circuits is so minute and complex that even the smallest contaminant can prevent a circuit from functioning properly. These contaminants decrease the production yield of usable circuits.

As the degree of integration becomes increasingly more complex, the semiconductor industry requires higher levels of water purity. In fact, the ultrapure water now required by the semiconductor industry typically must have a resistivity of $18.2 \text{ M}\Omega\text{.cm}$, while ionic and non-ionic contaminants must approach non-detectable limits.

A single water treatment process cannot efficiently deliver water of such a high purity level. Experience shows that optimum water treatment solutions take advantage of the specific capabilities of reverse osmosis (RO), ion exchange (IX), EDI and now, DuPont™ Ligasep™ membrane degasification.

Ultrapure water treatment systems vary depending on the source of the water to be processed and the ultimate purity required by the complexity of the semiconductor device. In general terms, the ultrapure water systems has the following steps:

- 1. The first step is the pre-treatment, to remove any suspended solids through ultrafiltration or multimedia filtration.
- 2. The second step is the primary loop where the goal is to demineralize water. A typical train consists of a double pass RO followed by a polishing unit such as a mixed bed or EDI unit. In this step, the removal of dissolved CO₂ is necessary to optimize the operation of the polishing unit. We've discussed the advantages of Ligasep™ in the previous slides.
- 3. The third step is the polishing loop where the demineralized water is further treated by a polishing unit to produce ultrapure water. In the polishing loop, achieving a very low concentration of dissolved O₂ is critical to ensure the highest water quality in the production of microelectronics. The desired dissolved oxygen concentration is usually less than 1 ppb.
- 4. The last application is in the removal of NH₃ in the wastewater generated in the manufacturing process. The treated wastewater can therefore be reused or discharged.

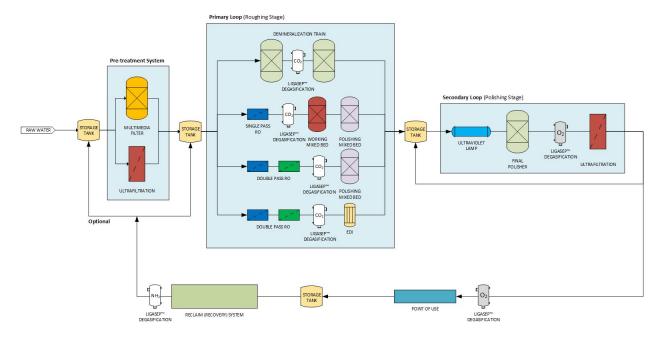


Figure 14. General Water Chain for Microelectronics

3.3 De-alkalinization system

In a de-alkalization system using a weak acid cation (WAC) resin in the H form, the system removes alkalinity and partially softens the water. The WAC resin exchanges hydrogen ions for hardness ions associated with alkalinity. The resulting low pH converts bicarbonate and carbonate alkalinity to dissolved ${\rm CO_2}$ gas which is then removed by degasification.

Reliably and effectively removing as much CO_2 as possible by degasification reduces the amount of alkalinity that could form post treatment due to equilibrium conversion of CO_2 and protects sensitive downstream processes and equipment from potential CO_2 related corrosion.

3.4 Boiler feedwater or make-up water

Boiler feedwater or make-up water are generally demineralized water that require very low conductivity. The feedwater quality required will vary depending on the operating pressure of the boilers. The following tables shows examples of different boilers with conductivity and dissolved oxygen concentration.

Table 5. Examples of boiler feedwater requirements

Boiler Operating Pressure	Maximum Conductivity	Dissolved Oxygen Level
Low Pressure (< 300 psig)	30 μS/cm	< 7 ppb
Medium Pressure (< 900 psig)	10 μS/cm	< 7 ppb
High Pressure (< 2000 psig)	10 μS/cm	< 7 ppb

Note: these are general requirements, please consult with your boiler supplier for specific requirements.

Demineralized water generally has a high level of dissolved gasses. These gasses can cause many corrosion problems inside the boilers.

- Oxygen causes pitting that is particularly severe because of its localized nature.
- Carbon dioxide corrosion is frequently encountered in condensate systems and less commonly in water distribution systems.
- · Ammonia, particularly in the presence of oxygen, readily attacks copper and copper-bearing alloys.

The resulting corrosion leads to damage on boiler heat transfer surfaces and reduces efficiency and reliability.

In order to meet industrial standards for both oxygen content and the allowable metal oxide levels in feedwater, nearly complete oxygen removal is required. Membrane degasification can offer a reliable and efficient process to reduce O_2 to 5 ppb or less. Other benefits are:

- Reduce or eliminate the use of oxygen scavengers.
- · Reduction in chemical consumption and operating cost.
- Improve process reliability
- Protect downstream equipment from corrosion
- Eliminate cross contamination from strip gas compared to deaerators.

3.5 Steam Power Generation

Steam Power plants generate steam to create a force to push a series of blades mounted on a shaft (similar to a propeller). As the shaft rotates it converts the mechanical energy to electrical energy. These blades are made of metal and are prone to oxidation. If the metals in the turbine blades start oxidizing, they will become damaged and impact the performance of the turbine.

3.6 Semiconductor Manufacturing Industry

Semiconductor manufacturing plants use a large volume of water to rinse the silicon wafer as they go through different processing steps. The wafer may go through 10-20 individual processing steps and each step will be followed by a rinse to remove chemicals used in the process. Oxygen in the water will react and oxidize metals used in the integrated circuit. The oxides will impact the circuits and create defects.

Table 6. Typical dissolved oxygen targets per application

Application	Typical Target D.O. *
Integrated circuits	<1 ppb
TFT Displays	< 50 ppb
Power plants	< 7 ppb
Boiler Feedwater	< 7 ppb

^{*} Values may vary depending on the requirements of the project.

4 System Design

4.1 Introduction

An entire membrane degasification system consists of the pre-treatment, the membrane degasification section and the post-treatment section. Since a degasification system is complementary to a large water treatment system, the options for pre-treatment and post-treatment units can vary considerably.

The pre-treatment step should be designed to ensure that the liquid fed to the membrane degasification modules comply with the feedwater quality guidelines shown in Section 4.6 of this manual. Post-treatment is employed to achieve the required product quality.

In this section, the design of a membrane degasification system is addressed.

4.2 Design Parameters

In order to design a DuPont™ Ligasep™ Degasification system, the following information is required:

Table 7. Ligasep™ design requirements

Gas to be Removed	Value	Units
Liquid Flowrate		m³/hr (gpm)
Liquid Temperature		
Minimum		°C (°F)
Maximum		°C (°F)
Dissolved gas inlet concentration		mg/L (ppm)
Desired dissolved gas outlet concentration		μg/L (ppb)
Pressure requirements (if any)		bar (psig)
pH value (for CO ₂ applications)		pH Units

4.3 Understanding operating modes

4.3.1 Vacuum only mode

Vacuum only mode is an effective method to remove gases from liquids using the two vacuum ports.

This process is when a vacuum is applied to inside the hollow fiber using a vacuum pump. The applied vacuum lowers the partial pressure of the gasses inside of the hollow fiber. This creates a driving force to move from the dissolved gasses in the water phase into the gas phase. The gas will then be emitted through the vacuum pump. Removal efficiency is controlled by the vacuum pressure because, the lower the gas pressure in contact with the water, the lower the amount of gas that will be dissolved into the water.

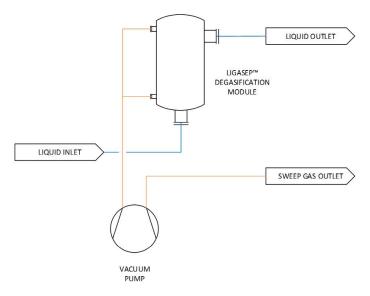


Figure 15. Vacuum only mode with vacuum pump

Note: For a more detailed process and instrumentation diagram (P&ID), please contact your DuPont Water Solutions representative.

4.3.2 Sweep mode

Sweep mode is a process in which gas flows through the inside of the hollow fiber countercurrent to the flow of water on the outside of the fiber. When a gas that is different from the gas to be removed water flows on the inside of the fiber the concentration and corresponding partial pressure of the gases in contact with the water is changed. This change in partial pressure creates a driving force to move the dissolved gasses in the water phase into the gas phase.

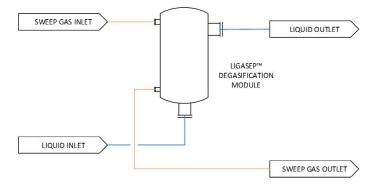


Figure 16. Sweep mode

Note: For a more detailed process and instrumentation diagram (P&ID), please contact your DuPont Water Solutions representative.

4.3.3 Combo mode

This is a mode in which vacuum mode is combined with the sweep mode. Combo mode is a process in which gas flows through the inside of the hollow fiber countercurrent to the flow of water on the outside of the fiber while a vacuum is simultaneously applied. When a gas that is different from the gas to be removed water flows on the inside of the fiber and a vacuum is also applied the concentration total gas pressure is changed. This will correspondingly lower the partial pressure of the gases in contact with the and create a driving force to move the dissolved gasses in the water phase into the gas phase. This is the most effective method in removing dissolved oxygen or carbon dioxide from water.

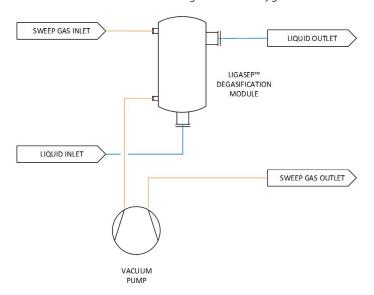


Figure 17. Combo mode with vacuum pump

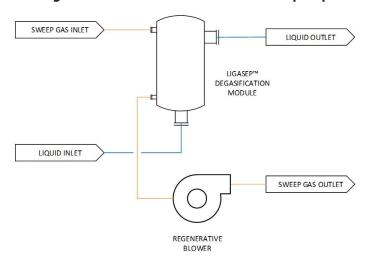


Figure 18. Combo mode with regenerative blower

Note: for a more detailed process and instrumentation diagram (P&ID), please contact your DuPont Water Solutions representative.

Table 8. Operating modes summary table

Operating Mode	Sweep Gas	Applied Vacuum
Vacuum Only		•
Sweep	•	
Combo	•	•

4.4 Applying Vacuum

When applying a vacuum on the gas side of the $DuPont^{TM}$ LigasepTM Degasification Module, the gas pressure inside the fibers is lowered. This lowers the pressure of the gas in contact with the liquid and in effect, the partial pressure of the gasses in contact with the water. This creates a driving force to remove dissolved gasses from the water.

The Ligasep™ Degasification Module has a unique skinned membrane that reduces the water vapor transport across the membrane. This allows the Ligasep™ Degasification Module to be operated at a vacuum below the vapor pressure of water with little water vapor passing through the membrane. When operating at a deeper vacuum, low levels of dissolved gasses can be reached.

The Ligasep™ Degasification Modules can be operated two ways when using vacuum:

- Vacuum only mode
- Combo mode

In both cases, vacuum can be applied using:

- Dry vacuum pump
- Liquid ring vacuum pump
- Regenerative blower in suction mode
- Diaphragm vacuum pump

The selection of the adequate vacuum pump or blower will depend on:

- Target gas (application)
- Desired vacuum level
- Sweep gas flowrate
- Cost

Table 9. Comparative table of different types of vacuum pumps

	Vacuum Level	Target Gas			
Vacuum Pump Type	Torr	CO ₂	02	Tolerance to Vapor	Liquid Trap
Liquid Ring	> 50	•	•	Yes	No
Rotary Vane	>1		•	Depends on make and models	Yes
Dry	>1		•	Yes	Yes
Diaphragm	>1		•	Yes	Yes

Table 10. Comparative table of different types of blowers

Target Gas					
Blower Type	CO ₂	02	Generate Pressure	Liquid Trap	
Positive Displacement	•		High	Yes	
Centrifugal	•		Low	Yes	
Regenerative	•		High	Recommended	

4.4.1 Liquid Ring Vacuum Pump

There are several types of vacuum pumps available in the market that could potentially be used in membrane degasification systems. Liquid-ring vacuum pumps are the most common types of vacuum pumps used in membrane degasification systems because of their availability, simple operation and it can handle any potential vapor condensate present in the vacuum line.

If there is any condensed water vapor present in the gas outlet line, it will be mixed with the water seal of the liquid ring vacuum pump, which will then be expelled out of the system through its outlet.

The main disadvantage of a liquid-ring vacuum pump is the need to have a clean water supply for the pump seal. The temperature of the water can impact the performance of the vacuum pump, please consult your pump supplier for more details. This liquid seal water supply can be considered as a waste of water which needs to be properly handled and treated.

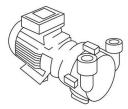


Figure 19. Schematic diagram for a liquid-ring vacuum pump

4.4.2 Options of Vacuum Pumps for Oxygen Removal

For vacuum only mode, it is recommended to use a dry vacuum pump in order achieve the required vacuum level for O_2 removal.

A rotary vane vacuum pump can also be used. Care must be taken when designing the system to avoid any water to enter the vacuum as it can emulsify with the oil seal and damage the vacuum pump.

Diaphragm pumps can also be an alternative to be used for smaller systems.

In all three cases, it is recommended to use a trap that ensures retention of any vapor condensate or liquids originating from the $DuPont^{TM}$ LigasepTM modules.

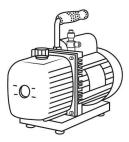


Figure 20. Schematic diagram for a vacuum pump

4.4.3 Regenerative Blower

An alternative would be to use a regenerative blower in suction mode for CO_2 removal. The blower's objective is to move air from the inlet to the outlet of the $DuPont^{TM}$ LigasepTM modules. However, not all blowers are suitable for LigasepTM operation.

The blower has to be sized and able to overcome the pressure drop across the Ligasep $^{\text{m}}$ module on the gas side of the membrane at the specified sweep gas flowrate.

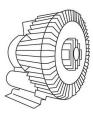


Figure 21. Schematic diagram of a regenerative blower

4.5 Steps to Design a Ligasep™ Degasification System

The main criteria for the design of the degasification system are:

- Desired gas removal percentage
- Vacuum level required
- Motor size
- Cost

The following steps are taken to design a membrane degasification system:

Step 1: Consider feed source, feed quality, feed/product flow, and required product quality

The membrane degasification system design depends on the available feed water and the application. Therefore, the system design information according to Table 7 should be collected first.

Step 2: Required pre-treatment

It is critical to evaluate the inlet liquid quality to ensure that it meets the feedwater guidelines shown in Section 4.6 of this manual. If there are one or many parameters outside the maximum limits indicated, it important to design a pretreatment adequate to condition the liquid to meet these guidelines.

For pre-treatment recommendations, please contact your DuPont Water Solutions representative.

Step 3: Select the operating mode and sweep gas

Select the operating mode and required sweep mode based on the gas to be removed from the liquid, as shown in Table 11.

Note: please refer to Section 4.3 for more information on the different operating modes.

Step 4: Select fiber type

The selection of the fiber type will depend on the gas to be removed from the liquid. Table 11 indicates the different types of fibers to be used according to the gas being removed.

Note: please refer to Section 2.2 for detailed information on the different fiber types available.

Step 5: Select module size and number of trains

The module size selection depends on the liquid flowrate to be treated. To ensure that the configuration is correct, it is recommended to ensure that the flowrate per train does not exceed the maximum flowrate per module as indicated on the product datasheets.

To calculate the flowrate per train Q_{Train} , divide the total liquid flowrate Q_{Total} by the total number of trains desired N_{Trains} . If the flowrate per train is higher than the maximum flowrate of the selected module, a large module can be selected or add a train to reduce to the flowrate per train.

$$Q_{Trains} = rac{Q_{Total}}{N_{Trains}}$$

Step 6: Select between a vacuum pump or regenerative blower

It is important to select whether the system will use a vacuum pump or regenerative blower. The type of vacuum unit to be used will depend on the gas to be removed, operating mode, sweep gas and final concentration of dissolved gas required. Table 9 shows the type of vacuum unit recommended per application.

Step 7: Analyze and optimize the membrane degasification system

The chosen system should then be analyzed and refined to meet the requirements of the project using the $DuPont^{\mathbb{M}}$ Ligasep $^{\mathbb{M}}$ sizing tool.

Table 11. Design Selection Criteria

Gas to be Removed	Target Concentration*	LS Fiber	HS Fiber	Operating Mode	Recommended Sweep Gas	Recommended Vacuum Source
	< 5 ppb	•		Vacuum Only		Dry Vacuum Pump
02	< 1 ppb	•		Combo	N_2	Liquid Ring Vacuum Pump
	< 10 ppb	•		Sweep	N_2	
<u></u>	< 5 ppm		•	Combo	Atmospheric Air	Liquid Ring Vacuum Pump
CO ₂	< 10 ppm		•	Sweep	Atmospheric Air	Regenerative Blower
H ₂ S **			•	Combo	Atmospheric Air	Liquid Ring Vacuum Pump
NH ₃ **			•	Liquid-Liquid Extraction	Acidic Solution	Chemical Pump

^{*} Target concentrations can vary depending on operating conditions (inlet concentrations, flow, pressure, number of modules in series), please contact your DuPont Water Solutions representative for detailed technical proposal.

^{**} For NH₃ or H₂S removal applications, please contact your DuPont Water Solutions representative.

4.6 Feedwater Quality Guidelines

Table 12 summarizes the limits of quality parameters of the feedwater. It is recommended to respect these limits to ensure successful operation of the membrane degasification system. Otherwise, more frequent cleaning and/or sanitization may become necessary. The concentrations correspond to the entry to the membrane for a continuous feed stream, including any influences to the feedwater from dosing chemicals or piping materials in the pretreatment line.

Table 12. Guidelines for feedwater quality

Component	Maximum Level
Total Suspended Solids	<1 ppm
Total Dissolved Solids	Under saturation limits
Total Organic Carbon	<1 ppm
Oil & Grease	< 0.1 ppm
Free Chlorine	< 0.1 ppm
Oxidizer	Not detectable
pH Range	1 – 13
Turbidity	< 0.5 NTU
SDI ₁₅	<3

In the event the water is of lower quality, please contact your DuPont Water Solutions representative.

4.7 Gas Supply Guidelines

Table 13. Guidelines for gas supply quality

Component	Maximum Level
Sweep Gas Particle Filtration	5 μm nominal
Sweep Gas Temperature (ambient air)	40 °C (104 °F)
Oil & Grease	None

These requirements must be met for sweep and combo mode.

The sweep gas temperature is limited if the sweep gas has oxygen present, such as in using ambient air for CO_2 removal. A high ambient air temperature can cause the oxygen to oxidize the fiber and damage it.

A high operating temperature can cause fiber breakage through shrinking. Please refer to the product datasheet for the maximum allowable operating temperature.

4.8 Typical system configurations

4.8.1 Modules in Series

As a two-pass reverse osmosis system is used to produce a very low conductivity product, the percentage gas removal will increase by having more than one module in series in one train.

This type of configuration has the following characteristics:

- The liquid outlet of the first module will connect to the liquid inlet of the second module in the series.
- Gas inlet are connected in parallel
- Gas outlet are connected in parallel
- Gas connections should always be connected in parallel otherwise, the second and subsequent modules will be
 fed with a mixture of the sweep gas and gas removed from the water in the previous module, affecting the gas
 removal objectives.

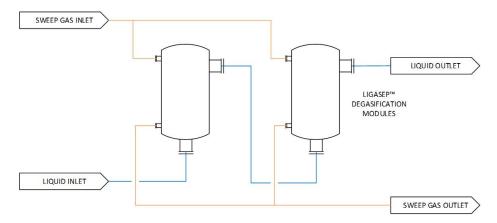


Figure 22. Two (2) modules in series

Note: For a more detailed process and instrumentation diagram (P&ID), please contact your DuPont Water Solutions representative.

4.8.2 Modules in Parallel

Since the modules has a maximum liquid capacity, in order to increase the overall system flowrate, it is important to have more than one module in parallel. Each module in parallel can be installed with its own auxiliary equipment or can share it with the other trains in the system. Each train can have one module or several in series, depending on the requirements of the project.

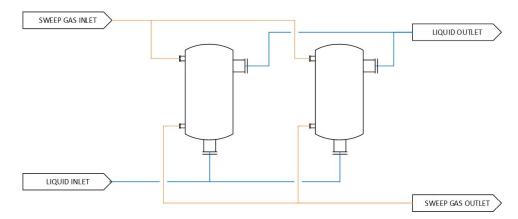


Figure 23. Two (2) trains in parallel

Note: For a more detailed process and instrumentation diagram (P&ID), please contact your DuPont Water Solutions representative.

4.9 Minimum instruments required

Table 14. Minimum instruments required

Parameter	Liquid Inlet	Liquid Intermediate	Liquid Outlet	Gas Inlet	Gas Outlet
Flowrate	•••		•	• ⁽²⁾	
Temperature	•••			• • • ⁽²⁾	
Pressure	•••	••	••	•••	
Vacuum Level				•	•••
pH (CO ₂) ⁽¹⁾	•••		•	•	•
ORP	•••				
Dissolved Oxygen (3)	•		•		

- ••• Mandatory
- Recommended
- Optional
- 1. pH measurement could be a method to monitor the performance of a membrane degasification system. However, the applicability of this parameter will depend on the application and the liquid inlet quality.
- 2. applicable in sweep or combo mode.
- 3. for oxygen removal application.

5 Installation

5.1 General installation recommendations

5.1.1 Handling product packages and unpacking

DuPont™ Ligasep™ Degasification Modules can be damaged due to improper handling and storage. The modules are shipped inside a cardboard box.

The following guidelines are intended to provide a framework for successful unpacking and installing Ligasep™ modules:

- Care must be taken not to hit or jar (shock) the modules to minimize the possibility of internal damage.
- Open the cardboard box with care not to damage the Ligasep™ module inside the box.
- Remove the documentation and any lose accessories delivered with the module.
- Remove the packaging on top of the module.
- Remove the module from the box and install it on the frame of the system. Do not carry or manipulate the modules by the flanges or gas port connections.

If you have any questions, please contact your DuPont Water Solutions representative.

5.1.2 Place of installation

Install Ligasep™ Degasification Modules in accordance with conditions specified in the Product Data Sheet. Some additional aspects to consider are:

- Select a place with no exposure to direct sunlight. Additionally, consider ambient temperature ranges over the year (minimum and maximum) to avoid freezing conditions or excessive warm temperatures.
- No contact with dust, moisture, corrosive gas or liquid.
- Enough space should be considered as part of the design for easy access to the modules for maintenance, inspection, repair and replacement.

5.1.3 Mechanical installation

- It is recommended to install the Ligasep™ modules vertically, the liquid inlet port should be facing down as shown in Figure 24. This configuration will allow for adequate drainage of the modules.
- The modules should be installed firmly onto the skid. It should not move, vibrate or suffer any damage during operation.
- All plastic port extensions should be supported to prevent bending of extensions under excessive piping loads.
- If you support Ligasep[™] modules with a clamp, make sure not to loosen the fitting. Do not apply too much pressure when fixing Ligasep[™] modules with a U-band as too much pressure may damage the module. Putting a saddle between Ligasep[™] modules and the clamp may prevent damage to the module from the support frame.
- All pipes should be firmly supported and fixed, make sure these pipes will not influence the stability of the system when operating.



Figure 24. Vertical installation of a DuPont™ Ligasep™ module

5.1.4 Hydraulic installation

- · Upstream process pipework should be thoroughly flushed prior to installation and commissioning.
- Make sure that connections do not suffer from excessive pressure or forces due to the module installation, hydraulics and piping weight. Piping and supports design should follow sound engineering practices.
- Do not shake or shock the Ligasep™ Degasification Module. Shaking or shocking may damage the module even if it is a light impact.
- Because the housing of the Ligasep™ module is composed of polypropylene, excess pressure to the connector may cause damage to the connecting port. Excess pressure to connecting port may shave thread and cause particles to appear inside the module.
- Do not touch hollow fibers.

5.1.4.1 Water inlet

The water inlet consists of the pipeline connected from the water source to the module inlet flange.

- Install security devices, such as a pressure reducing valve, safety valve or pressure switches, to make sure that pressure will not exceed the maximum allowable operating pressure as indicated in the product datasheets.
- Water hammer, a hydraulic shock to the membrane element, can also happen when the system is started up
 before all air has been flushed out. This could be the case at initial start-up or at operational start-ups, when the
 system has been allowed to drain. In starting up a partially drained degasification system, the pump may behave
 as if it had little or no backpressure. It will suck water at great velocities, thus hammering the elements. Water
 hammers could also with automated valves with quick actuators causing sudden opening and shut down of the
 valves.
- Size and design piping system in order to allow equivalent hydraulics at the inlet of modules installed in parallel.

5.1.4.2 Water outlet

The liquid outlet consists of the pipeline connected from the module outlet flange to the next module or exiting the degasification system.

• It is recommended to install a pressure gauge and sample valve between modules installed in series for troubleshooting purposes.

5.1.4.3 Gas inlet

The gas inlet consists in the gas line from the sweep gas source to the $DuPont^{m}$ Ligasep degasification modules. These recommendations are applicable for sweep and combo mode where the sweep gas is ambient air. There isn't an inlet gas line in vacuum only mode.

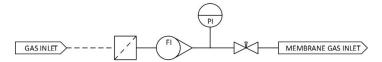


Figure 25. Installation of ambient air inlet line

- An air filter should be installed on the inlet side of the Ligasep™ moduler to prevent dust and other debris that may be in the air near the membrane degasification system. A 5.0 µm filter is recommended.
- No excess torque must be given to a joint, the maximum torque will be dependent of the materials used for the gas line.
- Materials used for piping and connections should be adequate for vacuum operation.
- Prevent gas leaks in the gas lines to avoid any reduction in removal efficiency.
- Avoid long pipes or loops and minimize objects that may cause pressure loss in the gas line.

5.1.4.4 Pressurized Gas installation recommendations

The gas inlet consists in the gas line from the sweep gas source to the Ligasep[™] degasification modules. These recommendations are applicable for sweep and combo mode where the sweep gas is pressurized, such as compressed air or compress nitrogen.

In the case of sweep mode, it is critical that the pressure of the sweep gas is lower than the liquid pressure to prevent any damage to the fiber.

In the case that compressed air is used, make sure it is oil-free and dry.

- 1. Use a pressure regulating valve to control the feed gas pressure ensuring that the sweep gas pressure is lower that the water pressure. The required gas pressure will be dependent on the system design and configuration.
- 2. The maximum allowed gas pressure is 0.5 barg (7.2 psig).
- 3. After the pressure control valve, it is recommended to install a gas flowmeter, pressure gauge and needle valve. This is important to ensure the flowmeter operates close to atmospheric pressure in order to read the correct flowrate without the need of any pressure calibration.
- 4. The pressure gauge should be a compound pressure gauge (vacuum/pressure) in order to ensure the gauge doesn't get damaged during operation. The range of measurement of a compound gauge is typically from -30 in Hg vac to 15 psi. This is applicable for combo mode for a O_2 removal application.

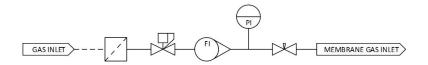


Figure 26. Installation of nitrogen inlet line

5.1.4.5 Gas outlet

The gas outlet consists in the gas line from gas outlet ports of the $DuPont^{TM}$ LigasepTM modules to the vacuum pump or blower. These recommendations are applicable for vacuum mode and combo mode.

- Installed vacuum lines must always be facing downwards to smoothly purge liquid derived from vapor.
- Install a vacuum gauge or transmitter near the module.
- · Correct interconnecting piping.
- A leak valve may be required to avoid the cavitation of a liquid-ring pump operating in vacuum mode. Please
 refer to Section 4.3 for more details on the different operating modes of a system with Ligasep™ modules.
- Install a drain valve at the lowest point of the gas line.

5.1.4.6 Liquid Trap

Although the vapor transfer through the membrane is low, the sweep gas exiting the module may be saturated with vapor which can condensate in the piping and auxiliary equipment.

In the case sensitive equipment to vapor is used, it is recommended to install a liquid trap to capture and separate the condensate from the gas stream and protect the equipment. The trap can also capture any liquid leaked due to damaged fibers or damages inside the module.



Figure 27. Schematic diagram of a liquid trap

5.2 Liquid Ring Vacuum Pump installation recommendations

- 1. Liquid ring vacuum pumps require a liquid seal supply to ensure the vacuum seal is operating adequately.
- 2. The liquid seal should comply with the recommended flowrate and pressure by the pump manufacturer.
- 3. To control the liquid seal supply flow and pressure, it is recommended to install a control valve and pressure compound gauge. The control valve will help regulate the pressure to ensure a slightly positive pressure is fed to the liquid ring vacuum pump. This will ensure that the vacuum pump will not cavitate and adequate vacuum is achieved by the vacuum pump.
- 4. It is also recommended to install a solenoid valve on the liquid seal supply line to stop the supply of water when the vacuum pump is not operating.
- 5. Check valve should be rated to operate under vacuum. A regular check valve will not operate adequately and will cause water from the vacuum pump to flow back into the inside of the fiber. If this occurs, the modules need to be put out of services and dried as per our drying procedure (Section 6.3) before being put back in service.
- 6. The liquid seal, along with any condensed water coming from the membrane degasification modules, will exit the vacuum pump through its outlet port. This stream can be sent back to the feed of the process for further treatment or discharged.
- 7. To separate the gas mixed in with the liquid seal, it is recommended to install an air-water separator. The gas will exhaust from the top of the separator and the liquid will drain from the bottom.

8. It is not recommended to use a variable frequency drive to modulate the speed of the motor as it can cause excessive heating of the motor and damage it. A variable frequency drive or soft started can be used to ramp up the startup of the blower.

It is highly recommended to consult the installation recommendations from the pump supplier.

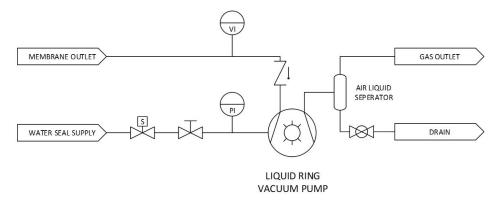


Figure 28. Installation of a liquid ring vacuum pump

5.3 Dry or Oil-Free Vacuum Pump installation recommendations

- It is important to ensure the vacuum line is designed and sized according to the operating conditions of the system.
- Installed vacuum lines must always be facing downwards to smoothly purge liquid derived from vapor.
- In projects where there is a high potential for vapor condensate transfer, it is recommended to use a liquid trap to protect the vacuum pump.
- It is not recommended to use a variable frequency drive to modulate the speed of the motor as it can cause excessive heating of the motor and damage it. A variable frequency drive or soft started can be used to ramp up the startup of the blower.

It is highly recommended to consult the installation recommendations from the pump supplier.

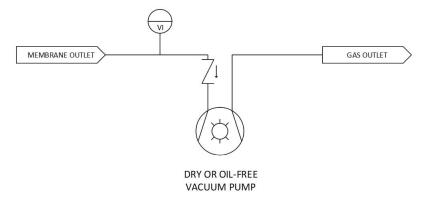


Figure 29. Typical installation of a dry or oil-free vacuum pump

5.4 Blower installation recommendations

- Vacuum line should be connected from the outlet gas ports of the DuPont™ Ligasep™ modules to the suction port of the blower.
- It is important to ensure the vacuum line is designed and sized according to the operating conditions of the system.
- Installed vacuum lines must always be facing downwards to smoothly purge liquid derived from vapor.
- In projects where there is a high potential for vapor condensate transfer, it is recommended to use a liquid trap to protect the blower.
- The exhaust line of the blower should be designed according to project specifications and requirements of the end-user.
- It is not recommended to use a variable frequency drive to modulate the speed of the motor as it can cause excessive heating of the motor and damage it. A variable frequency drive or soft started can be used to ramp up the startup of the blower.

It is highly recommended to consult the installation recommendations from the blower supplier.

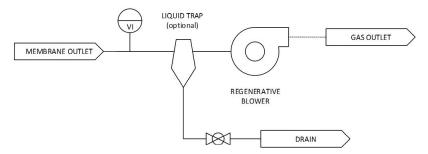


Figure 30. Typical installation of a regenerative blower

6 Operation

6.1 Start-up Procedure

Proper start-up of DuPont™ Ligasep™ Degasification Modules is essential to prepare the membranes for operating service and to prevent membrane damage due to excessive pressure, flow, or hydraulic shock. Following the proper start-up sequence also helps ensure that system operating parameters conform to design conditions so that water quality and productivity goals can be achieved. Measurement of initial system performance is an important part of the start-up process. Documented results of this evaluation serve as benchmarks against which ongoing system operating performance can be measured.

- 1. Before start-up, make sure water feed inlet and outlet are closed.
- 2. Flush all piping prior to membrane inlet to drain.
- 3. Gradually open inlet valve to ensure feed pressure is under the design condition until the modules are full and all air has been purged from the module.
- 4. Start up the vacuum pump (or blower) in accordance with the specifications prepared by the manufacturer. Ensure vacuum is showing on the vacuum gauge; if not, check the rotation of the vacuum pump or blower.
- 5. In cases where a sweep gas is used, make sure sweep gas inlet pressure is always lower than the pressure of the liquid outlet.
- 6. Gradually raise the gas flowrate to the specified value.
- 7. Gradually open outlet valve and ensure flowrate and water feed pressure are under design conditions.
- 8. If designed vacuum level is not reached, check for leaks in the vacuum line.
- 9. Observe product quality, temperature, and pressure specified in the specification sheet.
- 10. <u>Do not</u> use any oxidizing agents, such as NaOCl, O₃, strong acids, strong bases, organic solvents, oil, or any other liquid which has not been confirmed as compatible by DuPont Water Solutions.

6.1.1 Flow Diagram

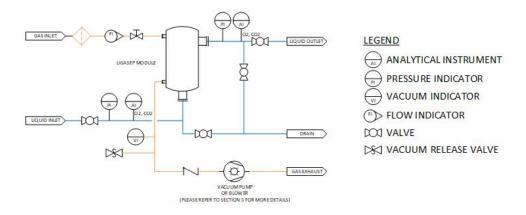


Figure 31. Process flow diagram for start-up procedure

6.2 Shut-down Procedure

Proper shut-down of DuPont™ Ligasep™ Degasification Modules is essential to prevent bacterial growth inside the module and to maintain performance once the modules are put back into service.

6.2.1 Short-term Shut-down Procedure

Short-term shut-down is defined as a shut-down period less than 3 or 4 days.

- 1. Open the vacuum relief valve of the vacuum exhaust pipe. Upon confirming that the vacuum has been released, shut down the vacuum pump or blower.
- 2. Close the valve on the liquid inlet side, then close the valve on the liquid outlet side.
- 3. Drain water from membrane degasification module opening the drain valve at the bottom of the degasification module.

Combo or Sweep Mode

- 1. Purge the whole circuit for longer than one hour with nitrogen or inert gas.
- 2. Close the exit valve of the gas line.
- 3. Water will then be saturated with nitrogen and this prevents bacterial growth.
- 4. Maintain the nitrogen pressure at a low pressure of 1 2 psi during shut-down.

Vacuum Mode

1. Shut down vacuum pump or blower and close all system and drain valves.

6.2.2 Long-term Shut-down Procedure

Long-term shut-down is defined as a shut-down period longer than 3 or 4 days.

- 1. Follow each recommended steps on short term shut down up to draining system
- 2. Connect compressed air to vacuum line with vacuum isolation valve closed and air line closed
- 3. Dry the module as per the Drying Procedure.
- 4. Allow over night air flow
- 5. Remove air line and connect N₂ line
- 6. Purge system with N_2 for about 5 to 10 minutes, close drain valve and pressurize system with N_2 at about 10 psi, close all valves and leave system pressurized with N_2

The module may also be disconnected and removed from the system and stored as indicated in the storage procedure.

6.2.3 Restart after Shut-down

- 1. Let the water start to flow after flushing the inside of the module.
- 2. Discharge the drain of the vacuum exhaust pipe before starting the vacuum pump.

6.2.4 Flow Diagrams

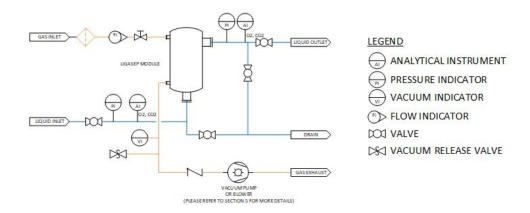


Figure 32. Process flow diagram for shut down procedure

6.3 Drying Procedure

Drying DuPont™ Ligasep™ Degasification Modules for long-term shut-down or when the fibers are wetted out:

- 1. Close the isolation valve of the degasification system.
- 2. Drain the degasification module by opening drain valves located at the bottom of the modules.
- 3. In order to completely dry the membrane, any water inside the pore of the membrane must be evaporated. Due to the large surface area, this process can take as long as 24 hours.
- 4. Introduce air into the top gas inlet port and liquid outlet port, exhausting the air from the bottom gas and liquid ports. This will allow air to flow on both sides of the membrane. The air used in this step needs to be clean, filtered, dry, and oil-free.
- 5. Follow air flow and time recommendation from Table 15.

Table 15. Recommended conditions to dry modules

Ligasep™ Model	Flowrate (N L/min)	Temperature Range	Maximum Pressure	Time
LDM-040	80	15 - 40°C (59 - 104°F)	≤7 bar (101.5 psig)	≤ 24 h
LDM-120	80	15 - 40 C (59 - 104 F)	s / bar (101.5 psig)	≤ 24 h

6.3.1 Flow Diagram

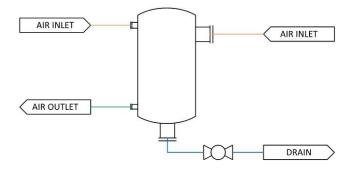


Figure 33. Process flow diagram for drying procedure

6.4 Handling, Preservation, and Storage

DuPont™ Ligasep™ Degasification Modules can be damaged due to improper handling and storage. The following guidelines are intended to provide a framework for successful handling and storage of these modules.

If you have any questions, please contact your DuPont Water Solutions representative.

6.4.1 Handling

Proper handling of modules is critical. Care must be taken not to hit or jar (shock) the modules to minimize the possibility of internal damage. It is also important to not carry or manipulate the modules by the flanges or gas port connections.

6.4.2 Storage

It is recommended that the modules be stored in a dry, heat-sealed plastic bag or shrink-wrap material in their original box to prevent the introduction of contaminants into the modules.

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

Temperature: Store the modules dry in their original boxes at temperatures not to exceed 40°C (104°F). Modules 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 modules be stored at low to moderate humidity levels (< 60% relative humidity). Humidity will not affect the components of the module but exposure at high humidity levels may affect the integrity of the cardboard boxes.

Store the membrane modules in the horizontal position, in cardboard boxes with the appropriate supports and filling materials. They may also be packaged in individual bags that need to be adequately sealed and stored on pallets or inside wooden crates. It is important not to stack the modules on top of each other.

Modules should be stored in a safe location where they are not at risk of falling, being crushed, or being impacted. Care should be taken to secure the membrane modules and containers to ensure stability and to avoid any possible injury resulting from falling, leaning, or any other accident.

6.4.3 Exposure to Sunlight

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

6.4.4 Freezing Conditions

In cold climates, it is critical to not allow the modules and the remaining water inside them to freeze.

6.5 Record Keeping

The performance of a DuPont™ Ligasep™ Degasification Module is influenced by feedwater composition, water and air flow, temperature, and vacuum level of the gas line.

Plant data recording is strongly recommended because it allows an early identification of potential problems (e.g., fouling). Corrective measures are much more effective when taken early.

A computer program called **Ligasep Log Sheet** is available to record operating data and graphing pressure drop on the liquid and air side. This program is available at www.dupontwatersolutions.com and requires Excel software.

When applicable, the program can also be used to record operating data for ion exchange system, whether it be for an anionic resin or mixed bed.

6.5.1 Daily Record

It is highly recommended to record and monitor the operation on a daily basis to ensure proper operation and optimal performance:

- Liquid flowrate
- Liquid inlet pressure
- Liquid outlet pressure
- Liquid temperature
- Sweep gas flowrate
- Inlet vacuum level (vacuum and combo mode only)
- Outlet vacuum level (vacuum and combo mode only)
- Liquid level in the vacuum trap (visual inspection)

In oxygen removal applications, it is recommended to also monitor:

- Inlet oxygen concentration
- · Outlet oxygen concentration

In CO_2 removal for ion exchange applications, it is recommended to monitor the throughput of the anionic resin column to confirm that it is similar to the design calculations.

7 Troubleshooting

7.1 Wet out conditions

In order for a membrane degasification system to operate efficiently, the interface between phases must be carefully controlled. The hydrophobic property of the PMP hollow fibers membranes, prevents water from passing through the pores as long as the pressure is kept below a critical threshold known as breakthrough pressure.

The breakthrough pressure is drastically reduced in presence of detergents and surfactants because they reduce the surface tension of the liquid allowing it to permeate through the membrane. Once the liquid has entered the fiber's pore, the fiber is known to be "wet out".

Once the fiber is wet out, the membrane will need to be cleaned and dried as explained in Sections 7.3 and 6.3 of this manual, respectively.

7.2 Symptoms

Trouble with the performance of a DuPont™ Ligasep™ Degasification Module normally means at least one of the following:

- Dissolved gas removal is lower than projected.
- Liquid flowrate is lower than the designed flowrate.
- Increase in pressure drop in the liquid or gas side of the modules.
- Presence of liquid inside the gas piping.

7.2.1 Dissolved gas removal is lower than projected

If the low performance occurs during the start-up of the Ligasep™ Degasification Module, it is important to review and correct the following:

- Dust plugs used during transportation should be removed.
- Sweep gas is contaminated and needs to be changed or cleaned before use; this may also require replacing or cleaning the gas filtration system.
- The sweep gas flow is low or not uniform through the modules.
- Vacuum level is less than required which may be due to a leak in the gas line, incorrect installation of the check valve, or damage to the vacuum pump or blower.
- Condensation inside the degasification module or vacuum line.
- Liquid temperature is lower than the design temperature.
- · Liquid flowrate is higher than the design flow or not balanced among the different modules.

A performance decline over time is generally due to gradual fouling of the membrane fibers which will necessitate cleaning to restore performance. Please refer to the Ligasep $^{\text{TM}}$ Degasification Modules Cleaning Procedure for specific instructions to clean the modules.

The performance decline can also be due to damage to the vacuum pump or blower that may require maintenance, repair, or replacement. Please consult with your pump or blower manufacturer for more details.

7.2.2 Low flow

Low flow may be due to a flow restriction upstream of the degasification system. In such case, it is important to review the process train to ensure that there are no other underlying issues that will reduce the flowrate of the system.

Another cause for low flow is fouling, which can be caused by:

- Inorganic compounds or scaling
- Biological and organic compounds
- Particulates and colloidal matter

Please refer to the DuPont™ Ligasep™ Degasification Modules Cleaning Procedure for specific instructions to clean the modules.

7.2.3 Increase in pressure drop on the liquid side

An increase in pressure drop is generally due to fouling of the membrane inside the Ligasep™ Degasification Modules. Fouling can be caused by:

- Inorganic compounds or scaling
- Biological and organic compounds
- · Particulates and colloidal matter

Please refer to the Ligasep™ Degasification Modules Cleaning Procedure for specific instructions to clean the modules.

7.2.4 Increase in pressure drop on the gas side

The sweep gas needs to be clean, particulate and oil-free, before it can be used inside the Ligasep™ Degasification Modules. An increase in pressure drop on the gas side is an indication that the lumen side of the fiber is fouled, typically by particulates.

To solve this issue, it is important to ensure that the gas filters are cleaned or replaced. If the pressure drop remains high, the degasification modules will require cleaning. Please refer to the Ligasep™ Degasification Modules Cleaning Procedure for specific instructions to clean the modules.

Another cause can be that the gas sweep flowrate exceeds the maximum flowrate per module and needs to be corrected accordingly.

7.2.5 Presence of liquid inside of the fiber

In DuPont™ Ligasep™ Degasification Modules, the inside of the fiber needs to be dry and free of liquid to ensure proper performance of the module. However, there are conditions that might introduce liquid inside the fiber.

First, it is important to ensure that the mechanical installation is correct:

- Ensure that the liquid supply is not connected to the gas port of the Ligasep™ Degasification Modules.
- When using a liquid ring type of vacuum pump, ensure that the check valve of the vacuum pump is operating correctly and is rated to operate under vacuum. If the check valve and the solenoid valve of the liquid seal supply are not operating correctly, liquid can flow back into the modules.

If the mechanical installation is correct, the membranes inside the degasification modules may have "wet out", allowing liquid to pass through the membrane. This condition can be due to:

- Contamination by surfactants, oil, or alcohols
- Biofouling of the fibers
- Cleaning the modules with chemicals that contain surfactants

To remedy this situation, it is important to first clean the module according to the DuPont™ Ligasep™ Degasification Module Cleaning Procedure. The module will then need to be dried according to the Ligasep™ Degasification Module Drying Procedure before being placed back into service.

7.3 Cleaning Procedure

The following are general recommendations for cleaning Ligasep $^{\text{TM}}$ Degasification Modules. More detailed procedures for cleaning a system containing Ligasep $^{\text{TM}}$ Degasification Modules are typically included in the operating manual provided by the system supplier.

7.3.1 Introduction

The surface of the hollow-fiber membranes in a Ligasep[™] Degasification Module is subject to fouling by foreign materials that may be present in the feedwater, such as suspended solids, colloids, organics, and biological matter. The term "fouling" includes the build-up of various kinds of layers on the membrane surface, including scaling.

Pretreatment of the feedwater prior to the Ligasep™ Degasification Modules is required to reduce these contaminants.

Membrane fouling is generally characterized as one, or a combination of:

- Inorganic fouling and scaling
- Particulate and colloidal fouling
- Microbial and biological fouling
- · Organic fouling

The fouling of membrane surfaces results in the gradual performance decline of the Ligasep $^{\text{m}}$ Degasification Modules in terms of lower sustainable gas transfer and high axial differential pressure.

The initial performance of the Ligasep™ Degasification Modules should be monitored and registered in order to establish a baseline for comparing the performance of the Ligasep™ Degasification Modules when they are clean and new against their current performance. For this purpose, DuPont can supply a data log sheet with the most important parameters to monitor.

The cleaning frequency will depend on the feedwater quality, performance, and differential pressure across the Ligasep $^{\text{M}}$ Degasification Modules.

7.3.2 Cleaning Requirements

Ligasep™ Degasification Modules should be cleaned to restore membrane performance when one or more of the below parameters are encountered during regular operation:

- Axial pressure differential increase by more than 3 psid.
- Outlet gas level exceeds design specification.

7.3.3 Safety Precautions

- 1. When using any chemical indicated here in subsequent sections, follow accepted safety practices. Consult the chemical manufacturer for detailed information about safety, handling, and disposal.
- 2. When preparing cleaning solutions, ensure that all chemicals are dissolved and well-mixed before circulating the solutions through the modules.
- 3. It is recommended that the elements be flushed with good-quality chlorine-free water (20°C minimum temperature) after cleaning. RO permeate or DI water is preferred for the cleaning solution; in cases when RO permeate is not available, please discuss project-specific conditions of water quality with DuPont Water Solutions Technical Service to evaluate if chlorine-free potable water or another water source may be used.
- 4. Care should be taken to operate initially at reduced flow and pressure to flush the bulk of the cleaning solution from the elements before resuming normal operating pressures and flows. Despite this precaution, cleaning chemicals will be present following cleaning. Therefore, the permeate must be diverted to drain for at least 30 minutes or until the water is clear when starting up after cleaning.
- 5. During recirculation of cleaning solutions, the maximum temperature must not be exceeded (50°C). It is important to 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.
- 6. Consult the chemical tolerance chart to ensure the cleaning solution does not exceed chemical concentration limits
- 7. Do not use surfactants or formulated cleaning solutions without consulting a Dupont Water Solutions representative.

7.3.4 Cleaning Conditions

- The cleaning solution flowrate and pressure should be equal to the service flowrate of the system.
- Refer to DuPont™ Ligasep™ Degasification Modules product data sheets to confirm the maximum allowable flow, temperature, and pressure ratings.
- The cleaning solution should be fed into the Ligasep™ Degasification Modules from the inlet port and circulated in the normal service flow direction.

7.3.5 Recommended Equipment

If a manual cleaning is not enough to restore performance, a Clean-in-Place system can be used to perform the cleaning of the DuPont™ Ligasep™ Degasification Modules.

Such a system will require the following equipment:

- Clean-in-Place tank
- Clean-in-Place pump
- 5-µm cartridge or bag filter
- Pressure gauges
- Flowmeter
- Necessary valves and piping

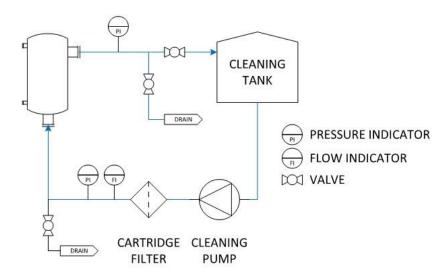


Figure 34. Process flow diagram for cleaning

7.3.6 Wetted-out Conditions

When the fibers of a DuPont™ Ligasep™ Degasification Module have been wetted out, the fibers have lost their hydrophobic properties and the contaminants, as well as water, have been allowed to permeate through from the liquid side of the fiber into the lumen side.

In this case, it is necessary to clean the lumen side of the fiber as well in order to restore performance. This can be done by using the following configuration:

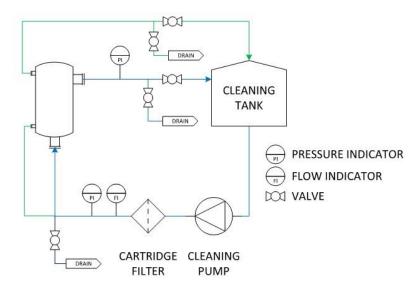


Figure 35. Process flow diagram for cleaning when wet out

Procedure:

- Prepare the cleaning solution based on the type of fouling experienced by the Ligasep™ Degasification Module.
 - Ensure no air bubbles are present in the cleaning solution.
- 2. Isolate the train that requires cleaning using the appropriate isolation valves.
- 3. Open cleaning valves of the train to be cleaned.
- 4. Recirculate the cleaning solution using the cleaning pump and set cleaning conditions as indicated above.
- 5. Continue recirculating for a period of 2 hours.
- 6. Allow a soaking for 1 hour.
- 7. Rinse the cleaning solution out of the modules until the solution is clear or when the pH has neutralized.
- 8. If the lumen has also been cleaned, it is important to employ proper drying procedures before putting the modules back in service (refer to Section 6.3 for further details on the drying procedure).

7.3.7 Extreme Fouling

In cases of extreme fouling, an air scour can be introduced into the cleaning process.

To do so, introduce clean, dry, oil-free air into the gas port of the Ligasep™ Degasification Module at a pressure 5 – 10 psig higher than the pressure on the liquid. This will create an air scouring effect allowing to dislodge the foulant from the membrane surface.

Note: During wet-out or complete pour plugging, no air will cross the membrane.

7.3.8 Cleaning Chemicals

Table 16 lists general cleaning chemicals that are effective to recover membrane performance. Generic acid cleaners and alkaline cleaners are standard, widely available cleaning chemicals. Acid cleaners are used to remove inorganic precipitates (including iron), while alkaline cleaners are used to remove organic fouling (e.g., biological matter).

Table 16. General cleaning solutions guidelines

			Cleaning Solution		
Targeted	HCl @ pH range of	H_2SO_4 @ pH range of	Citric Acid ¹ @ pH	NaOH at a pH range of	Hydrogen Peroxide @
Foulant	2 – 3 @ ambient	2 – 3 @ ambient	range of 2 – 3 @	10 - 12 @ ambient	1 – 3.5 wt% @ ambient
	temperature ²	temperature ²	ambient temperature 2	temperature ²	temperature ²
Inorganic	Preferred	Alternative	Alternative		
Particulate	Preferred	Alternative	Alternative		
Or					
Colloidal					
Microbial				Preferred	Alternative, up to 200 h
or					of cumulative exposure
Biological					
Organic				Preferred	

^{1.} When using Citric Acid, the solution should be thoroughly rinsed out due to organic loading onto the membrane.

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^{2.} Please consult a DuPont Water Solutions representative for chemical limits at different temperatures.