Technical Manual

MICRODYN-NADIR

MICRODYN® MF Modules with PP-Membranes

Technical Manual

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1 Crossflow-Microfiltration - Technical Information

Instructions for the Use of MICRODYN® MF Modules

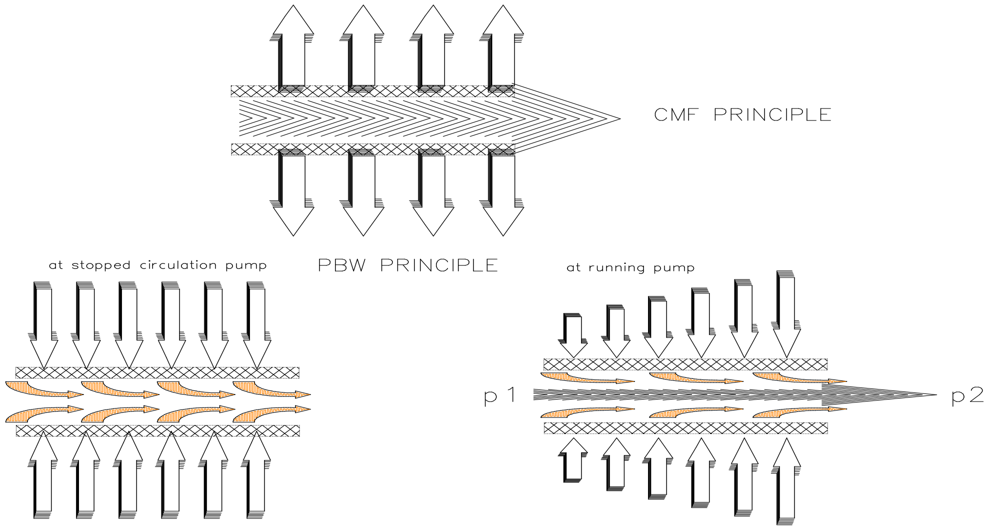

Figure 1. Principle of CMF and Periodical Back Pulse (PBP)

MICRODYN® MF modules have been developed for crossflow microfiltration (CMF), a modern filtration procedure for separating suspended solids and for low concentrated emulsions.

The separated particles form a deposit layer which is reduced to a very low thickness by means of:

a) shearing forces at the membrane surface due to tangential flow
b) periodic back Pulse (PBP) of the membrane.

Both procedures contribute to high and stable flux efficiency.

The microporous membranes are self-supporting and have a very narrow pore distribution. All module types can be cleaned with the periodical back pulse (PBP) technique.

2 Used Materials and Basic Module Design

MICRODYN modules contain a bundle of capillary or tubular membranes made of polypropylene (PP) that is tightly potted at both ends to a tubular casing and a cap. Tubular membranes are potted with polypropylene and capillary membranes are potted with polyurethane. (Figure 2) This way a space is formed for the clear permeate and the retentate.

The tubular membranes made of PP are welded in a housing of PP so this type of module consists completely of polypropylene.

The capillary membranes made of PP are cast into the housing with a confirmed polyurethane (FDA). The most important geometric dimensions can be seen from the data sheets. Additional materials, e.g. O-rings, are also listed in these sheets. O-rings with a certificate have to be ordered separately.

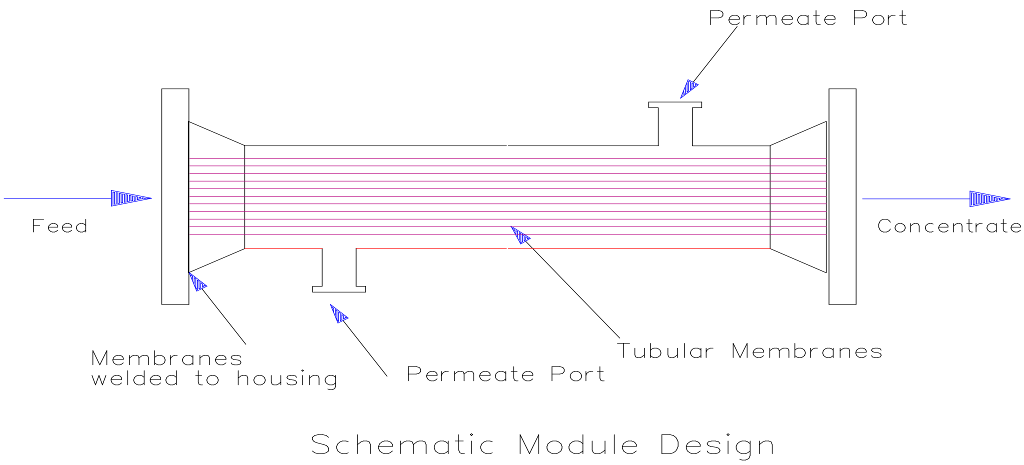

Figure 2. Schematic Module Design

3 Pore Size

MICRODYN® MF modules contain a membrane with a nominal pore size of 0.2 µm. These membranes are made symmetrically. The separation of substances happens at the membrane surface.

The pore size of 0.2 µm has been proved ideal for most separation requirements in solid-liquid and liquid-liquid filtration.

For special applications we provide pore sizes of 0.1 µm.

4 Module Position and Activation

MICRODYN® MF modules can be installed into a system vertically as well as horizontally. Each module has two permeate outlets, normally only one is used during filtration. The position of the outlet should be at the end of the module (low pressure side) if it is in a horizontal position.

A horizontal position is recommended for filtration of suspensions with a large amount of sedimenting solids. In the horizontal position the port for permeate must be the top outlet, to force the complete module to fill with liquid.

In vertical position the permeate outlet always has to be upwards so that all air bubbles can be removed from the module.

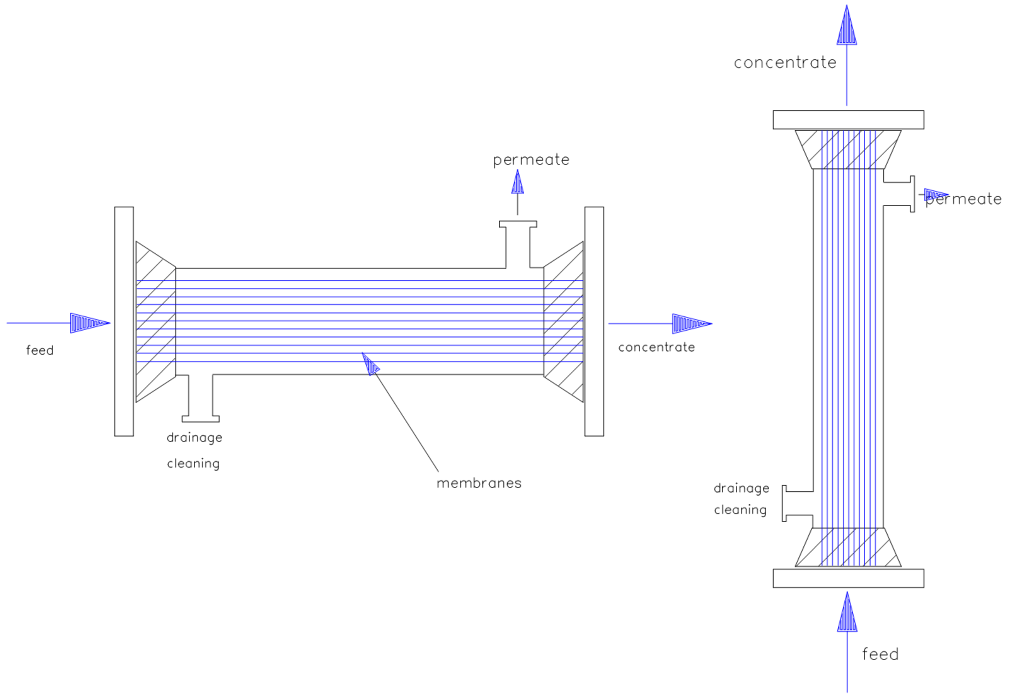

Figure 3. Module position and permeate outlet installed in a vertical and horizontal position

4.1 Activation of MICRODYN-Modules with Water Pressure

The PP membrane is hydrophobic in nature. Liquids like water with high surface tension of 0.072 N/m do not wet the membrane.

The most simple, cleanest and safest way to activate the membrane in the module(s) is the use of water at higher pressure. First step is to fill the module(s) bubble free with water (25 °C) at both sides (feed and permeate side). A pressure of 6 bar has to be applied to the module(s) for 1 minute. Normally this is the tap line pressure. (Please notice the difference to the max. operating pressure given in the data sheet).

After this procedure the pressure has to be released to zero. The water must be exchanged and the procedure must be repeated 4 to 6 times. Now the module is activated and can be used for filtration.

4.2 Activation with Wetting Agents

For all filtration purposes where the module has to be tested by a bubble point test for process safety reasons, the module has to be activated by means of water-soluble liquids with a surface tension of less than 0.035 N/m.

Appropriate liquids to be used are:

- a mixture of water and 50 vol. % of isopropanol or
- a mixture of water and 25 vol. % tert. butanol (not inflammable)
- surfactants

With this method it is necessary to run the module in a system for about 30 min in crossflow mode until a sufficiently high flux is reached.

After wetting, the module is flushed either with water or with the solution to be filtered to rinse any remaining agents.

As long as it stays in fluid, the membrane is activated. If the membrane dries up it must be activated again as per description. The membrane's separation properties will not change by drying.

Heavily gasifying liquids (carbonated beverages or beer) require a counter-pressure on the permeate side (e.g. 1 bar) in order to prevent the release of gases. Gases will deactivate the membranes.

In the PBP process a part of the permeate is pushed back by compressed gas (e.g. air). Care must be taken, that the gas is not pressed into the membrane. Otherwise the membranes will be made a partially hydrophobic with reduced flux. A reactivation has to be performed.

5 Shutdown of a System

During a long shutdown, the plant must be cleaned completely (washing with water) and filled with e.g. a 1% NaOH solution to avoid bacterial contamination.

Used in filtration of liquid food e.g. fruit juice or wine, the system has to be cleaned very well, to leave a minimum of organic material in the system. A 2% NaOH solution with 1% ethyl alcohol usually prevents a cultivation of mildew.

The membranes will not dry in a closed system. If the membrane dries up it must be activated again. The membrane's separation properties will not change by drying.

6 Operating and Back Pulse Pressures

In CMF, a liquid is pumped through the MICRODYN modules. The hydraulic pressure drops from the inlet value p1 to the outlet value p2 due to the liquid flow. It is necessary that the filtrate pressure pF is lower than p2 for filtration to occur.

crossflow microfiltration periodic back pulse

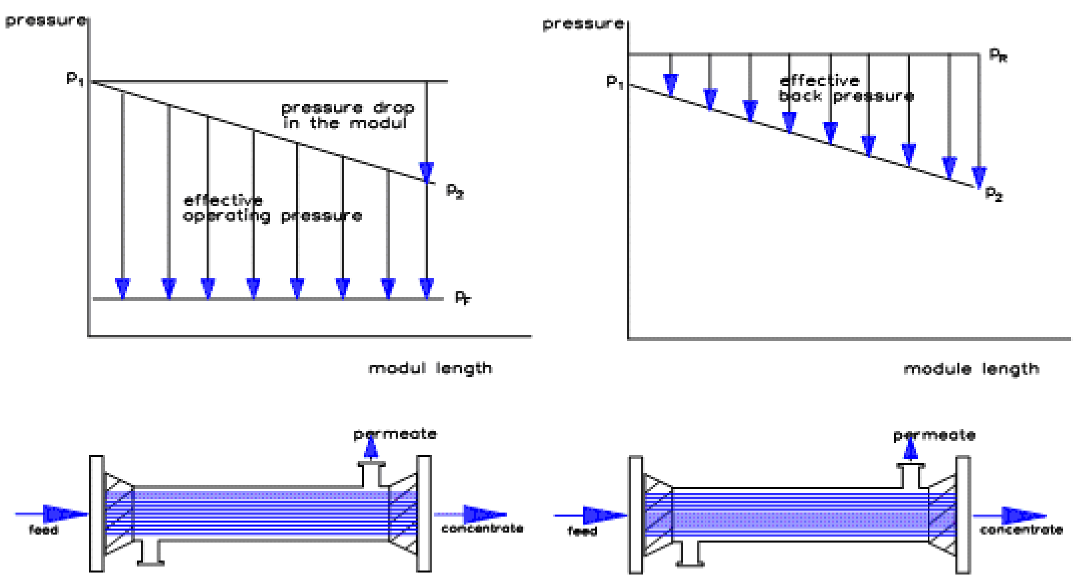

Figure 4. Pressure situation during filtration and PBP

The allowable operating pressure (p1 - pF) depends on the membrane and the temperature. These values are listed in the data sheets of the modules.

In PBP mode, PF must increase to a value higher than P1. This procedure is an implosion stress for the membrane. The PBP-pressure (pressure above outlet pressure P2) indicated in the data sheets should not be exceeded to prevent the membrane tubes from imploding.

Air bubbles in the module always affect the efficiency of the Back Pulse. Generally the permeate outlet should be placed at the drain end of the module. In vertical position the permeate outlet always has to be on the upper side.

7 Speed Recommendations

The favourable flow velocity for membrane tubes ranges from 1 to 3 m/s, for capillary membranes from 1 to 2 m/s.

The PBP intervals of 1 to 30 min are recommended, depending on the product. Generally the PBP filtrate volume is less than 5 % of the total filtrate quantity. The PBP run 2 to 5 s.

8 Pressure Loss of MICRODYN-Modules

The energy consumption of a crossflow microfiltration system is determined mainly by the circulating volume and the pressure losses of the module. The pressure loss is affected by:

- the properties of the circulating fluid (e.g. rheologic behavior, viscosity and the concentration of suspended particles),
- the velocity resistance in the circulation (modules, fittings, piping system),
- the conditions of operation (flow velocity).

Usually the greatest velocity resistance in the circulation occurs in the parallel or serially operated modules. Here only the relationship between the pressure drop and the mean linear velocity of a liquid in the MICRODYN modules is considered. The pressure drop Δp of MICRODYN modules using water at 20 °C is shown in the following figure.

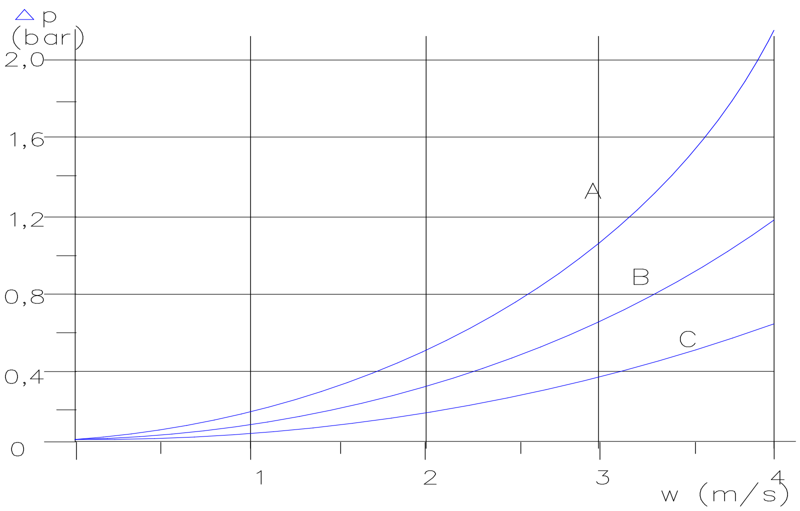

Figure 5. Pressure drop vs. speed of feed

Curve A: For modules with capillary membranes; d_i = 1.8 mm and 1 m in length

MD 150 CP 2N

Curve B: For modules with tubular membranes; d_i = 5.5 mm and 3 m in length

MD 150 TP 2L

Curve C: For modules with tubular membranes; d_i = 5.5 mm and 1.5 m in length

MD 090 TP 2N

MD 150 TP 2N

For liquids other than pure water the following equation can be used to calculate the pressure drop of an MICRODYN module.

$$P_M = \left(k + \lambda \frac{l}{d_i}\right)\frac{\rho}{2}w^2 \qquad \text{(equation 1)}$$

P_M = pressure loss of one module (N/m ²)

k = module specific constant (specific pressure drop at inlet and outlet of a module)

k = 2 for tubular membrane d_i = 5.5 x 10^{-3} m

k = 4 für Kapillarmembranen mit d_i = 1.8 x 10^{-3} m

λ = friction factor

l = length of the membrane tubes or capillaries (m)

d_i = inner diameter of the membrane tubes resp. -capillaries (m)

ρ = density of liquid in (kg/m³)

w = mean linear velocity within the membrane tubes resp. -capillaries (m/s)

The friction factor λ depends on the type of flow within the membrane tube (laminar or turbulent), and also on wall roughness if turbulent flow occurs. The Reynolds number can be calculated from the following equation:

$$\mathrm{Re} = \frac{w d_i \rho}{\eta} \qquad \text{(equation 2)}$$

whereas η is the dynamic viscosity of the liquid in Pa·s. For water the value of η is 10^{-3} Pa·s.

For Reynolds numbers below 2300 (laminar flow condition) the following relation for the friction coefficient λ is valid:

$$\lambda = \frac{64}{\mathrm{Re}} \qquad \text{(equation 3)}$$

For Reynolds numbers higher than 2300 please take the value from the following figure 6:

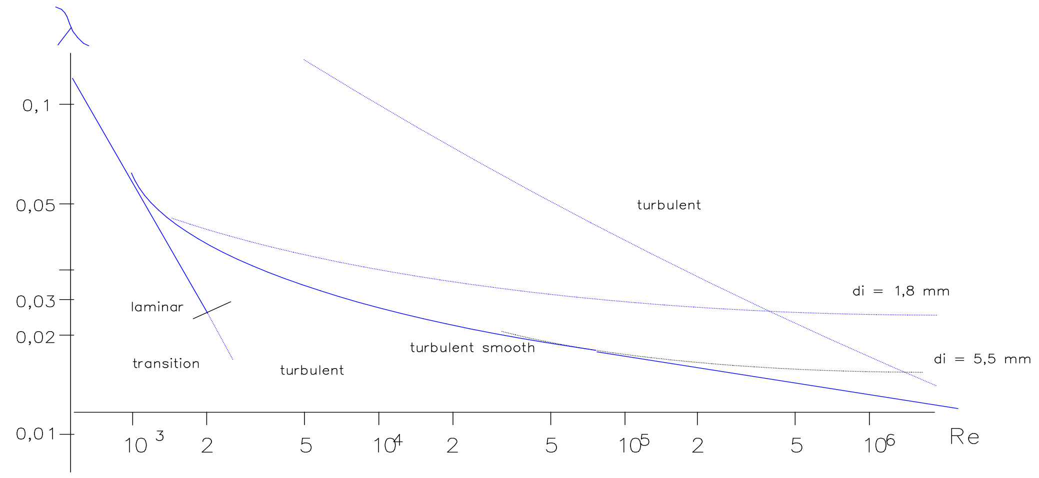

Figure 6. Friction factor as a function of Reynolds number

9 Resistance to Chemicals

Modules with tubular membranes and plastic casing are made entirely of polypropylene.

Capillary membranes are potted with polyurethane. Both polymers are resistant to aqueous salt solution, diluted inorganic acids as well as concentrated caustic solutions. Both polymers are not resistant to hypochlorite solutions and oxidants.

Aromatic and aliphatic hydrocarbons with low boiling points such as petrol ether, benzene, and carbon tetrachloride diffuse into the polymers and will cause swelling and decreasing mechanical strength. Before filtration of these substances, consultation with MICRODYN-NADIR is necessary.

Exceptions are low volatile substances such as greases, oil and waxes. They cause negligible swelling and low mechanical strength reduction only.

Peroxides may be used in small amounts for cleaning. The concentration must be discussed for the individual case.

The chemical resistance of MICRODYN modules is listed in the following table. Appendix 1 gives the chemical resistance for MICRODYN modules with tube membranes (TP-Modules) of the and with capillary membranes (CP-Modules). The resistance depends on many factors such as temperature, concentration and intensity of the mechanical stress. The data in the table are for reference only.

10 Chemical Membrane Cleaning

In order to obtain a high filtration capacity on a long-term basis, a chemical cleaning of the membrane is required at times. Non-oxidizing acids or caustic solutions within the pH-range of 1—14 are appropriate chemicals.

Media which were developed for membrane cleaning in particular generally contain additional purifying agents like sequestering agents, surfactants or active enzymes.

⚠ **WARNING**

Hypochlorite solutions in any concentration are not permitted

In many cases a very good result can be reached by flushing the cleaning solution from the permeate side through the membrane.

Employing crossflow to the membrane at low pressure level has a favourable effect on the cleaning process, as it is very effective and saves cleaning agents. Unspent chemicals may be pumped down for further use.

Starting this recommended way of cleaning, the module has to be cleared through the lower outlet. The cleaning agent is pumped in and pressed through the membranes. Then it must act in the module for a period of about 30 minutes to 1 hour. Afterwards a new cleaning agent is pumped through the module to replace the contaminated solution. The next step is a thorough flushing with water. When employing a combined cleaning e.g. alkaline – acidic this step is repeated with the new cleaning agent. The flushing with water removes all residues of the cleaning agent.

Although a cleaning with chemicals in flow direction is possible, dissolved substances may penetrate the membrane structure. This can produce a contrary effect if the substances are not dissolved completely. Furthermore the amount of chemicals needed is higher. A low pressure overflow of the membrane has a favourable effect on the cleaning process.

The temperatures indicated in the data sheet must not be exceeded.

Detailed cleaning instructions must be worked out on-site and depend on the product.

Generally the following rules apply:

Organic fouling of the membrane are predominantly handled with alkaline cleaning agents, esp. NaOH with a concentration level of about 1 – 5% at a temperature of 20 – 60°C.

Cleaning aids (tensides) speed the dissolution of fouling and of oily and greasy substances.

For cleaning of inorganic fouling e.g. Ca/Mg carbonates (“water hardeners”), ferrous compounds, mostly acidic agents are employed. Organic acids e.g. citric acid, oxalic acid, mixtures of hydrochloric acid and oxalic acid have proved favourable due to their complex-building properties.

In case of serious problems with module cleaning we advise consultation of MICRODYN-NADIR.

The latest published information is available on MICRODYN-NADIR’ s homepage. MICRODYN-NADIR assumes no liability for the completeness of the same. Liability of any kind which is not covered by the warranty as specified in MICRODYN-NADIR 's Conditions of Sale cannot be accepted.

11 Appendices

Resistance of MICRODYN-Modules

<u>SYMBOLS</u>

+ resistant at operating conditions as per data sheet

/ limited resistance, swelling (only limited operating conditions)

- not resistant

MEDIUM	TP-Modules		CP-Modules	O-Ring resist.	
	20°C	60°C	40°C	EPDM	NBR
Acetone	+	/	/	+	-
ethanol, 99 %	+	+	+	+	+
Ethylacetate	/	-	-	-	-
Ethylether	+	+	-	/	-
ethylene glycol	+	+	+	+	+
formic acid (dil.)	+	+	+	/	-
amylic alcohol	+	+	-	+	/
aniline dye	+	+	-	-	-
petrol ether, Kp 100-140 C	/	-	/	-	-
Benzene	/	-	-	-	-
succinic acid (dil.)	+	+	+	-	+
Beer	+	+	+	+	+
bromic water, cold saturated	-	-	-	-	-
butanol	+	/	/	+	+
butyl	/	-	/	/	-
butyl glycol	+	+	+	+	+
calcium chlorid sol.aqueous	+	+	+	+	+
Chlorobenzene	+		+	-	-
Chloroform	+	-	-	-	-
chlorosulfonic acid	-	-	-	-	-
chloric water	+	/	-	+	-
Cyclohexane	+	+	/	-	+
Cyclohexanol	+	/	/	-	+
Cyclohexanon	/	+	/	-	-
diethanol amine	+	+	+	-	/
Dichlorethylene	/	+	/	-	-
dichlorobenzene, cold sat.	/		/	-	/
dimethyl amine	/	-	/	+	/
ferric chloride (III), sat.	+	+	+	+	+
Vinegar	+	+	+	+	+
acetic acid, 10%	+	/	+	+	-

MEDIUM	TP-Modules		CP-Modules	O-Ring resist.	
	20°C	60°C	40°C	EPDM	NBR
hydrofluoric acid, 10%	+	+	/	+	+
formaldehyde, 30%, aqueous	+	+	+	+	+
juices, aqueous	+	+	+	+	+
fructose, aqueous, cold sat.	+	+	+	+	+
galvanic baths	+	/	+	+	+
Gelatine	+	+	+	+	+
glucose, aqueous	+	+	+	+	+
Glycerol	+	+	+	+	+
Glycol	+	+	+	+	+
Hexane	+	/	/	-	+
Isopropanol	+	+	+	+	+
potassium hydroxide, aqueous	+	+	+	+	-
potassium permanganate (2n)	-	-	-	+	-
saline, saturated	+	+	+	+	+
linseed oil	+	+	+	-	+
machine oil	+	/	/	-	+
sea water	+	+	+	+	+
Methanol	+	+	+	+	/
methylenechloride	+	-	-	-	-
Molasses	+	+	+	+	+
Milk	+	+	+	+	+
lactic acid, aqueous, 10%	+	+	+	+	+
mineral oils	+	/	+	-	+
mineral water, commercial qual.	+	+	+	+	+
natrium sulfite, 40%	+	+	+	+	+
sodium hydroxide solution(2n)	+	+	+	+	+
sodium hydroxide solution,52%	+	+	+	/	+
Nitrobenzene	/	/	-	-	-
fruit juices	+	+	+	+	+
oleum, 100%	-	-	-	-	-
olive oil	+	+	+	/	+
petrol ether	+	+	+	/	+
peracetic acid, 0.2%(see note)	-	-	-	+	-
plant oils	+	/	+	-	/
Pyridine	/	-	-	/	-
nitric acid (2n), aqueous	/	-	/	+	-
chloric acid, 30%ig, aqueous	+	/	+	+	+
sulfuric acid (2n), aqueous	+	/	+	+	+
soap solution	+	+	+	+	+

MEDIUM	TP-Modules		CP-Modules	O-Ring resist.	
	20°C	60°C	40°C	EPDM	NBR
silicon oil	+	+	+	/	+
sodium carbonate solution	+	+	+	+	+
cold saturated, aqueous	+	+	+	/	+
soy bean oil	+	+	+	+	+
starch solution	-	-	-	-	/
Terpentine	-	-	-	-	-
carbon tetrachloride	/	-	-	-	-
Toluene	+	+	-	-	-
trichloro acetic acid	+	/	/	/	/
Water	+	+	+	+	+
tartaric acid, aqueous, 10%	+	+	+	+	+
Whisky	+	+	+	+	+
Xylol	-	-	-	-	-
Citric acid, aqueous, 10%	+	+	+	+	+