

HIGH PURITY WATER

CLEARING IT UP

In 1987, the president of the United States, Ronald Reagan, hailed the success of the Montreal Protocol on substances that deplete the ozone layer. This protocol eventually led to limiting and, finally, an outright ban on chlorofluorocarbon containing chemicals (CFC's). Chief among them was Freon, which was widely used as cleaning solvent, especially in critical cleaning application.

By Bill Siegmund

This gave rise to the use of water as the primary cleaning agent and ultra pure water (UPW) for critical cleaning operations. To understand how water works, we will need to go back to the basics. We will discuss what water is, how we measure quality, treatment techniques and system design to purify water, as well as practical applications in critical cleaning optical lens coating operations.

WATER - A MOST UNIQUE SUBSTANCE ON EARTH

Barren and lifeless is the only way to describe earth without life-giving water. Water can not be created or destroyed. Water is continually recycled from sky to earth to sky in a natural circuit called the 'hydrologic cycle'. The hydrologic cycle has been called nature's purification treatment. The earth is more than 2/3 water, but less than 3% is fresh and most of that is tied up (until recently) in polar ice caps or underground. We use water in so many ways that it is perhaps unrealistic to expect it to meet all the demands we make upon it. Still, with the right treatment, water can and does meet all of its obligations. There is no new water on earth, so it is easy to see how the demand of an ever increasing population is contaminating this finite resource.

Water is unique: it is the only substance on earth that

at naturally occurring temperatures can exist in all states of matter: as ice it is a solid, we drink it, as a liquid, and as water vapor it is a gas. Water has a slightly blue color and does not conduct electricity (electrolyte), but instead it resists the flow of electrical current (resistor).

"Pure water exists so rarely that practically speaking it is non-existent."¹⁰

Water is the 'universal solvent' and dissolves a little of everything it comes into contact with. Contaminants can include:

- | dissolved minerals
- | dissolved gases
- | turbidity and sediment
- | color and organic matter
- | taste and odor
- | micro-organisms.

Dissolved minerals are primarily salts (metals) that are dissolved in water. The water can be perfectly clear, but all natural occurring water contains some degree of dissolved material measured as total dissolved solids (tds). There are different measures for each one of the impurities. You will see under 'treatment techniques and system design' that we will show them all. I would like to limit our discussion to the most commonly referred to quality indicators used in optical laboratories.

HOW WE MEASURE QUALITY

The most commonly used measurement for water quality relevant to critical cleaning are:

| parts per million (ppm)

is a measure of concentration, how many parts out of a million and can be expressed as milligram per liter mg / l

| micro Siemens per centimeter ($\mu\text{S} / \text{cm}$)

is a measure of specific conductance or a material's ability to conduct an electric current over a given distance (centimeter)

| mega ohms per centimeter ($\text{m}\Omega / \text{cm}$)

are a measure of specific resistance or a material's ability to impede the flow of electrical current over a given distance. 1 meg ohm is equal to 1 million ohms of resistance

Remember that water does not conduct electricity - it is a resistor. Also keep in mind that water is the universal solvent. What we are measuring is ionically charged particles dissolved in the water. As an example, if you were taking a bath in ultra pure water 10 $\text{m}\Omega / \text{cm}$ or better, and an electric fan fell in, you would not get a shock. If you add a teaspoon of salt, you're dead (please do not try this at home!).

Ionically charged particles are minerals (primarily salts) dissolved in water. They can be either (+) positively charged cation or (-) negatively charged

anion. We are measuring their presence (specific conductance) or absence (specific resistance) as a measure of water quality. The higher the specific conductivity, the lower the water quality. The higher the specific resistance, the higher the water quality.

High specific conductance = low water quality
High specific resistance = higher water quality
 (see figure 1)

TREATMENT TECHNOLOGIES

Deionization, reverse osmosis, and distillation are all demineralizing technologies. When we distill water, we use the fact that water can move from a liquid to a gas. By adding heat, we create a vapor and by cooling, we turn it back into a liquid. Only the dissolved minerals are left behind in the boiling chamber. Since few labs use distillation, we will leave that lay.

Reverse osmosis (ro) is also a demineralizing technology. To understand ro, it is helpful to understand osmosis. Osmosis is the movement of a solvent through a semi-permeable membrane from a lower to a higher concentration. The cells of our body will move needed nutrients through the cell wall (semi-permeable membrane) to supple nutrients. Goretex is an example of a semi-permeable membrane. It contains a plastic layer with pores large enough to let vapor out, but too small to not let liquid in.

With reverse osmosis, we use pressure to force a solvent-water with dissolved impurities - through a semi-permeable membrane from a higher to a lower concentration. Under pressure, the water flows down into the pores of the membrane and works its way to the permeate or product water side. Charged ions (mineral salts) are repelled from the membrane surface and are transported away (rejected) with main flow of water (reject water - see figure 2).

Reverse osmosis uses water to make water. The amount of water used versus the amount of product water produced is measured as a percentage called 'percent recovery'. Newer designed systems can run from 50% to 75% recovery and do not 'waste' water. In fact, some water is often recycled to the ro feed side or even used in other plant operations with little or no 'waste'.

Ro as a demineralization technology is always a part of a system that combines treatment technologies, including micro filtration and carbon pre-treatment. Deionized water has the ionically charged minerals removed by ion exchanged. Usually two ion exchange resins are used. One resin (cation) removes positively

SPECIFIC CONDUCTANCE MICRO SIEMENS MU / CM*	* SPECIFIC RESISTANCE MEGA-OHMS P/CM (MΩ / CM)	PARTS PER MILLION AS CaCO ₃ PPM OR MG / L
0.056	18000,000	0.028
0.071	14000,000	0.030
0.100	10000,000	0.050
0.167	6000,000	0.083
0.500	2000,000	0.250
1.000	1000,000	0.500
1.667	600,000	0.833
2.500	400,000	1.250
10.000	100,000	5.000
20.000	50,000	10.000
40.000	25,000	20.000
80.000	12,500	40.000
312.500	3,200	156.250
625.000	1,600	312.500
2500.000	400,000	1250.000

Fig.1: Water quality equivalents

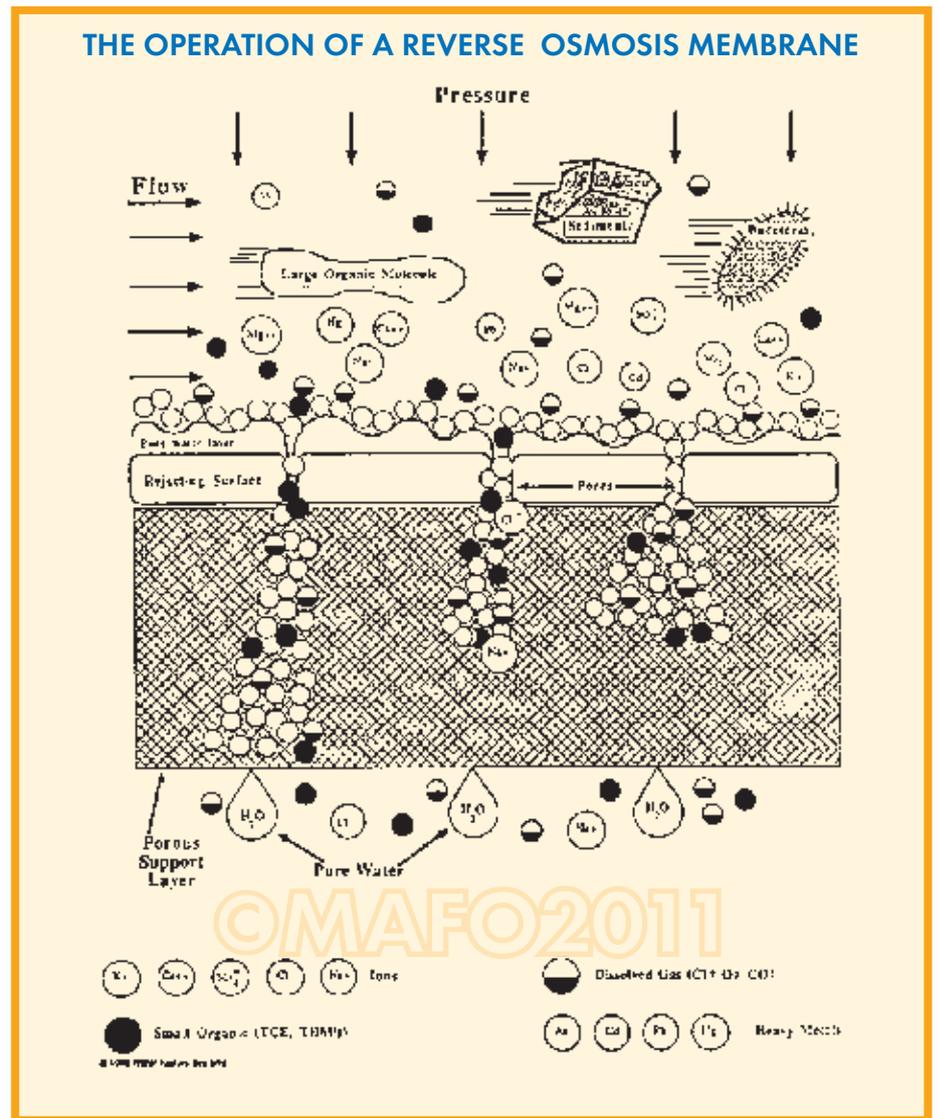


Fig. 2: Osmosis membrane

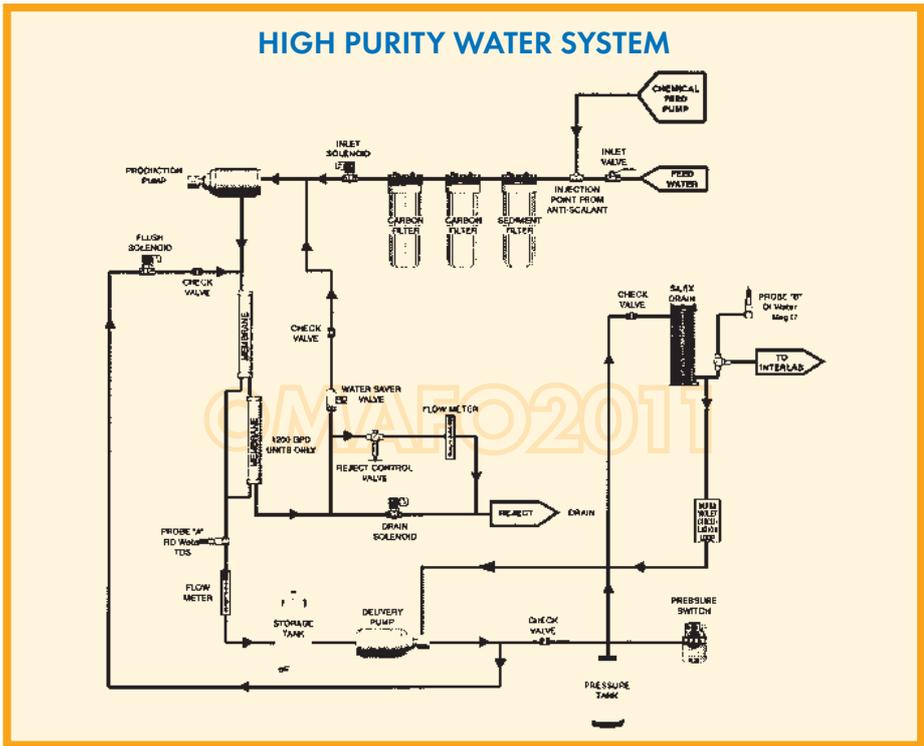


Fig. 3: Flow diagram
The feed water is first treated with an anti-scalant chemical. The purpose of this anti-scalant is to prevent the 'scaling' or 'fouling' of the membranes by the impurities in the water. Next the water is passed through a 5 micron sediment prefilter. This filter removes any large particulate material from the feed water. Finally the water is passed through two carbon cartridge filters. These carbon filters remove the chlorine from the water. Chlorine will severely damage the r.o. membrane. This concludes the pretreatment of the feed water.

Using a high pressure pump, the water is forced through the r.o. membrane. The impurities (TDS) are separated from the water by the r.o. membrane, which sends the impurities to the drain and the r.o. water to the storage tank.

When the interlab lens cleaner calls for water, the pressure switch activates the delivery pump. Water is pumped from the storage tank to the silex d.i. column where it is polished to d.i. water and then goes to the lens cleaner.

charged ions (calcium +, magnesium ++, sodium + etc.) and releases an equivalent amount of hydrogen ions (H+). The second resin (anion) removes the negatively charged ions (chloride-, sulfide - etc.) and releases an equivalent of hydroxide ions (OH-). The hydrogen and hydroxide ions combine to form water:



The dissolved minerals are attached to the resin beads and must be removed (regenerated) using hydrochloric acid and sodium hydroxide. Since this is a highly toxic process, it is rarely or never done in the lab. Regenerated tanks are 'exchanged', new for used.

TREATMENT TECHNIQUES, SYSTEM DESIGN

The treatment technique most commonly used in optical coating laboratories is mixed bed deionization (di). This system is usually applied using a carbon tank followed by two mixed bed (cation and anion) tanks in series because these tanks are only capable of producing water at 1 to 6 mΩ / cm. By using them in series, we can achieve the 10 mΩ / cm (+) required for most coating applications. However, this is by far the highest cost per gallon.

There is a technology worth mentioning, electric-deionization (edi). Edi uses electric current and resin to remove dissolved mineral salts. It is becoming cost

viable for small systems, but requires careful pre-treatment controls that are more cost effective on larger systems.

The most cost effective way to produce high purity water (hpw) is a combination of a reverse osmosis system and mixed bed di. The ro has the advantage of removing both dissolved and un-dissolved (non-ionically charged) contaminants. By removing both, you improve the overall quality of the product water and increase the 'life' of the mixed bed di by as much as 70% (see figure 3).

PRACTICAL APPLICATIONS

A properly designed high purity water system can reduce the cost per gallon while improving water quality and, therefore, yield. It must be integrated to the operation and contain adequate monitoring, as well as alarms to 'baseline' the quality to prevent fluctuation. Lab operators can count on the quality water being delivered to the washer / coater with little programmed maintenance. This can also extend the life of any recirculating 'on board' resins as well.

In a recent industry article 'anti-reflection coating quality' the author lists "control of all process variables that can be automatically controlled and alarmed." As one of several factors in 'quality creation' (2). The importance of proper cleaning and rinsing of a lens prior to coating can not be over stated, and therefore the importance of proper high purity water quality, as a controlled, alarmed variable, must not be overlooked.



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Bibliography
1 - Water treatment fundamentals; Seventh edition; Joseph P. Harrison, P.E., CWS-VI; Technical Editor; Copyright 2004; Water Quality Association; Chapter 1, pages 1-6
2 - 'Anti-reflective coating quality'; Arturo Colautti; MAFO ISSN 1614-1598 66527; 3/2010; page 28