Chapter 2 Reagent Water

Pure water- no such thing. Pure water is H_2O. Water is the universal solvent and often called aggressive because it dissolves everything. Oils, gases, metals, salts, organics, inorganics all dissolve in water, some more than others. This dissolution of materials of course adds contaminants to the water. Municipal water supplies contain various amounts of these contaminants depending upon the source of the water, surface water or groundwater. Since analysis is the measurement of contaminants in water, the water used to make chemical reagents, dilutions, calibration blanks must be as free of the contaminants as possible. To prepare reagent grade water the contaminant must be removed through the use of various water treatment processes. There is no one treatment process used to remove all contaminants. The most common commercial systems use activated carbon, microfiltration, ultrafiltration and reverse osmosis, followed by deionization or distillation.

Figure: An example of a laboratory reagent grade water production system.

Granular Activated Carbon

Granular activated carbon is primarily used to remove chlorine residual and organics. Activated carbon has lots of surface area and removes organics primarily by absorption. The efficiency of the absorption is tied to the type of carbon material, the flow rate, and the nature of the organic materials. Not all organic materials are effectively removed. Chlorine residual is effectively removed by activated carbon. Because chlorine is so efficiently removed, living bacteria that escape chlorine become embedded in the downstream carbon and can begin to reproduce. This may become a major source of biofouling in other downstream treatment units.

Microfiltration

Microfiltration also commonly called a sediment filter removes TSS particles. Most of the cartridge filters are 1-5 micron nominal filters which are used to protect the downstream reverse osmosis unit from TSS fouling. The size of a nominal filter designates the average size of the particle removed. For instance, a 1 micron nominal filter will catch most 1 micron particles but some larger particles will manage to get through and likewise some smaller particles will not. An absolute 1 micron filter will remove all 1 micron particles but is much more expensive. The feedwater to the microfilter passes through the filter, catching particles in the filter matrix.

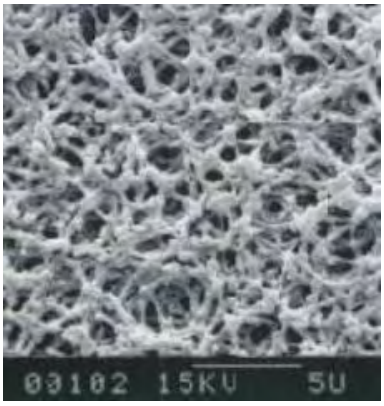
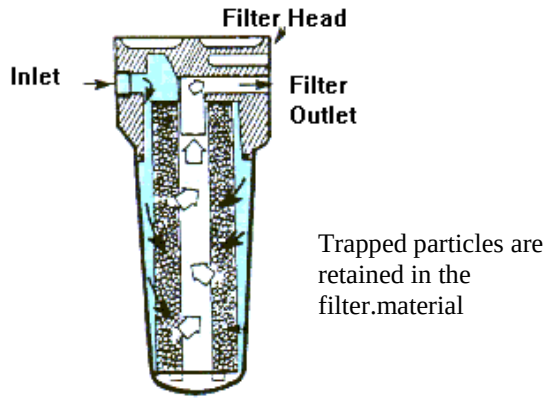


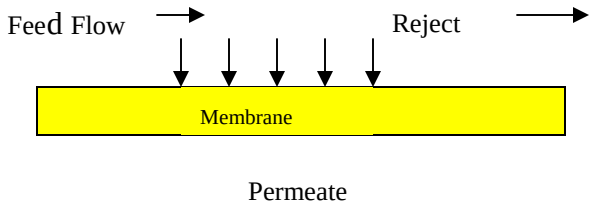
Figure: Notice the different size pores in this nominal filter. The tortuous path through the filter will catch most but some particles will manage to get through.



Microfilter plugging can be monitored by determining the differential pressure across the filter. Some commercial units will have a pressure gauge before and after the filter.
As the filter plugs, the difference in pressure will increase indicating the filter is plugging. Most cartridge filters are removed and replaced with a new filter.

Ultrafiltration

Ultrafiltration is a cross flow membrane separation process. In a cross flow system, a feed stream is introduced into the membrane element under pressure and passed over the membrane surface. A portion of the feed water passes through the membrane at 90° and is called permeate.



The rejected materials are flushed away in a stream called the reject or retentate. The cross-flow direction scouring the membrane surface and reduce membrane fouling. 90-95% of the feedwater is often recovered as permeate. Ultrafiltration is not as widely used in reagent grade water production as microfiltration but is valuable when organic contaminates are a concern. Ultrafiltration removes particles in the 0.01- 0.1 micron range. Ultrafilters are also classified by the molecular weight of the particle they reject. This molecular weight cut off (MWCO) is helpful to laboratories analyzing for low level organics. Large molecular weight organic contaminants such as those from surface water can be effectively removed by ultrafiltration. Small molecular weight organics are likely to pass through. Microorganisms, including viruses are theoretically all removed by ultrafiltration.

Reverse Osmosis

Reverse Osmosis is another cross flow membrane separation process. Following the removal of particles by microfiltration or ultrafiltration, the water is forced under pressure through a semi permeable thin film composite (TFC) membrane. RO membranes remove approximately 99% of dissolved ionic materials (TDS) including both multivalent and monovalent salts. Essentially all dissolved and suspended materials rejected by the membrane go to drain. The RO permeate usually flows to a storage tank then shuts off when the tank is full. One of the drawbacks of reverse osmosis is that at least half of the feed flow is rejected with each pass through the RO membrane. This reject flow is required to minimize scaling and fouling on the membrane surface. The RO permeate is considered low grade reagent water (reagent grade Type III). This quality water has very little use in analysis but makes a great economical source of water for deionization and distillation units. The major advantage of using RO is as a pretreatment for deionization and distillation. Because RO removes 99% of the TDS, it extends the service cycle of deionization resin and reduces scaling and carryover problems with distillation. The RO membrane is subject to fouling from TSS, microorganisms, colloidal particles, and scaling. Pretreatment with activated carbon, microfilters, and ultrafilters, extends the life of the RO membrane. Monitoring the percent salt

rejection and conductivity will help identify problems with the RO.

Deionization

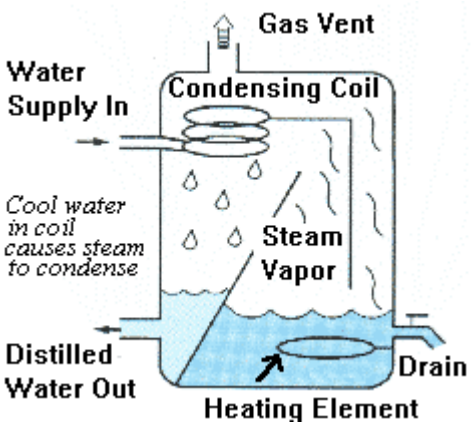
Deionization refers to the removal of ions using exchange resin. There are two major types of resin, strong acid cation resin and strong base anion resin. Strong acid cation resin exchanges all positive ions (Na^{+}, Ca^{+2}, Mg^{+2}) for hydrogen ions (H+). Strong base anion resin exchanges all negative ions (Cl^{-} , HCO_3^{-} , SO_4^{-2}) for hydroxide ions (OH-). The hydrogen ions (H+) and hydroxide ions (OH-) react to form "pure" water (H_2O). This water has now been deionized. The cation resin and anion resin can be purchased either as separate beds or together as a mixed bed. In either case, the resin has a limited capacity. The more ions in the feedwater to the resin, the faster the resin becomes used or exhausted. Once the resin is exhausted it must be regenerated or replaced. The quality of the deionized water (DI water) is ideally monitored continuously using inline meters or at least monitored monthly by testing for conductivity or resistivity, TOC, chlorine residual, and heterotrophic plate count.

A high quality DI water (Type I) will have a resistivity of 18.3 megohms. To reach this level the water must be flowing. Laboratories requiring ultrapure water usually have a continuously flowing/recirculating system with taps at the point of use. Remember "pure" water is very aggressive. As soon as the water leaves the recirculating system, contaminants are immediately introduced. The first contaminant is usually carbon dioxide from the air. Carbon dioxide reacts with the water to form carbonic acid which drops the pH. A pH around 5 should not be alarming. In addition, pH is very difficult to measure in ultrapure water and is seldom accurate. As the water quality deteriorates the resistivity will drop. A resisitivity below 10 megohm indicates the resin is nearing exhaustion and a replacement cartridge should be available or the resin bed should be regenerated. Ion exchange resin is not 100% efficient at removing ions and often a second mixed bed ion exchange cartridge is used as a polishing resin. For trace analysis, the contaminant level in the DI product water should be measured for each specific test. .

Distillation

Like deionization, the feedwater to a still is usually product water from the RO storage tank. RO product water will drastically drop the maintainance required and reduce the carryover of solids into the distillate. Distillation works by heating the water in a boiler. The steam produced goes through a series of glass tubes and eventually passes a condenser coil containing cooling water. The steam condenses, is collected and taken to the storage vessel. The distillation unit is usually all borosilicate glass to minimize metal corrosion and carryover to the storage tank. Like deionized water, the water quality should be tested monthly for resistivity, chlorine residual, HPC, and TOC and any other constituents for individual tests.

Deionized or distilled water used for microbiological analysis must also be checked annually for cadmium, chromium, copper, nickel, lead, and zinc. These trace metals may be found in plumbing and can be harmful when running microbiological tests.



There are instances where double or triple distilled water is needed to produce water without trace contaminants.

UV Sterilization

Ultraviolet sterilization often follows deionization or distillation. A lamp composed of mercury is used to produce light with a wavelength of 254 nm. The 254 nm wavelength is considered a bactericidal wavelength which is capable of sterilizing microorganisms. The UV light destroys the microorganisms ability to reproduce but does not cause lysis. For this reason, another 0.2 micron absolute microfilter is often found after the UV to "collect" the sterilized microorganisms.

Reagent Grade Water

Reagent grade water is classified primarily on its resistivity. For most wastewater laboratory applications, medium-quality reagent water with a resistivity of >1.0 megohm (conductivity <1 uS) is acceptable. Medium grade water is produced by distillation and deionization.

High quality reagent water has a minimum resisitivity of >10 megohms (conductivity <0.1 uS). This quality of water is very unstable and must be produced continuously. This quality reagent water is often in a closed loop system where the water is constantly returned to a polishing mixed bed demineralizer to maintain its high resistivity. Water line drops to the sink from high purity closed loop lines should be very short to reduce chances for biofilm formation.

High-quality water is used for all trace metal and trace organic analysis. In addition to resistivity, this quality water is often tested for specific contaminants to document that there is no background contamination that will affect trace analysis.