Preparation of Reagent Water

1 INTRODUCTION

Preparation of reagent water is one of the most important processes in the laboratory. Purified water is used daily for the preparation of reagent solutions and standards, as well as for critical cleaning of glassware and other apparatus. The operation and maintenance of the equipment used to produce reagent water is discussed in this SOP.

2 SCOPE AND APPLICATION

2.1 OVERVIEW

Reagent water is prepared by first distilling softened tap water using an automated electric still. Tap water flows through a water softener, through a condenser which also acts as a pre-heater, and into a stainless steel electric boiler. Water flows continuously into and out of the boiler to reduce the buildup of scale. Steam generated in the boiler moves upward and through a tin-lined stainless steel condenser. Condensed steam drains out the end of the condenser as distilled water. Distilled water is collected in an internal storage tank. A sensor turns the still on automatically when the water level in the storage tank falls below a certain level. Distilled water is pumped from the storage tank throughout the laboratory building to four distilled water facets marked with a white “DW” button on the handle.

Distillation effectively removes all ionized solids, high boiling point organics, and bacteria from the softened tap water. Thus, distilled water may be used for some less critical applications such as pre-rinsing. However, it does not remove ionized gases and low boiling point organics. In addition, some contaminants are re-introduced into the distilled water from the supply and circulating pipes, pumps, and valves that make up the distribution system. Thus, for most critical applications, the distilled water must be further purified before it can be used for most applications.

A Barnstead pressure cartridge system (PCS) is used to “polish”, or further purify distilled water using a pack of four purification cartridges and a filter. The first pre-treatment cartridge removes colloids, some organics, and bacteria. Next, water flows through two ultra-high purity deionization cartridges, and a trace-level organics adsorption cartridge. The distilled, deionized, and organic-free water (DDO) is finally passed through a 0.2 µm pore size hollow fiber filter to complete treatment. Reagent water produced from this combination of distillation and polishing is suitable for most

critical applications. Continuous monitoring of polished water resistivity indicates the level of ionic impurities. All cartridges are changed every four months, or more often if water resistivity falls below 17.0 MΩ-cm, or if other measurements indicate a need for new cartridges.

3 REQUIRED TRAINING

4 EQUIPMENT AND MATERIALS

4.1 DISTILLATION

4.1.1 Water Softener and salt for feed water pretreatment.
4.1.2 Electric Distiller, 240 V, 3 gpm (Pure Water, C-50)
4.1.3 Distribution pipes (1/2" PVC), valves, and faucets.

4.2 POLISHING SYSTEM

4.2.1 Barnstead Pressure Cartridge System (PCS) Nanopure (Model #D11901), digital purity meter, inlet and outlet valves (outlet valve constructed of Teflon), and delivery tubing (Teflon-lined Tygon).

4.3 CONSUMABLES (LISTED AS INSTALLED FROM LEFT TO RIGHT)

4.3.1 1 ea. Nanopure Diamond Pack, Barnstead (Thermo Fisher #D50280).
4.3.2 2 ea. 10" BB DI Water Filter Cartridge (Pure & Secure LLC, #25501).

4.4 REAGENT WATER STORAGE

4.4.1 20-liter polyethylene carboys with and without spigots for DDW and DW storage and transport.
4.4.2 1 to 4-liter combusted glass bottles for DDO storage.

5 SAFETY PRECAUTIONS

5.1 SAFETY PRECAUTIONS

5.1.1 Always depressurize system before changing cartridges. Routine maintenance of the polishing system results in some spillage of water, and may present a shock hazard. Do not allow water to contact electrical outlets or equipment. Sponge up excess water after changing cartridges.

6 SOLUTIONS AND REAGENTS

7 STANDARD SOLUTIONS

8 PROTOCOL

8.1 SERVICE INTERVAL

8.1.1 Cartridges must be periodically replaced, and the service interval will depend on the amount of water used, as well as the quality of the feed water. The digital purity meter monitors the resistivity of the purified water, and thus the amount of ionized dissolved solids present. Theoretically, pure water at 25° C with no dissolved solids will have a resistivity of 18.2 MΩ-cm (specific conductance = 0.055 µmho-cm). ASTM Type I reagent water must have a resistivity of >16.67 MΩ-cm (specific conductance = 0.060 µmho-cm) which allows for a range of total dissolved solids in reagent water up to ~10 ppb. Most of this conductance in deionized water initially comes from carbon dioxide dissolved from the surrounding air.

8.1.2 When the ion exchange cartridges on the PCS system become saturated, they will begin to bleed off dissolved ions into the reagent water stream. Bleed off from the cartridges will be indicated when the resistance of the reagent water stays below 17 MΩ-cm while the water is flowing. The conductance measured while water is not flowing cannot be used to indicate water purity. If the resistance indicated by the digital purity meter consistently reads below 17 MΩ-cm with the outlet valve open and the water running, it is time to change the cartridges.

8.1.3 Resistivity only indicates the concentration of ionic impurities present and is not sensitive to organics, particulates, or other neutral molecules. Since the amount of dissolved organics is not continuously monitored, it is important to change cartridges more frequently than indicated by a drop in resistivity. Thus, if TOC reagent blanks indicate any change in dissolved organics, or if any other sensitive measurements indicate a possible contamination of the reagent water, it is likely that it is time to change the cartridges as well. As a general rule, cartridges should be changed every 3-4 months, or more frequently if otherwise indicated.

8.1.4 Pre-treatment cartridges must be periodically replaced as well. The service interval will depend on the amount of water used, as well as the quality of the feed water. Replace cartridges when the light on the system head of both cartridges turns, and remains, red.

8.2 INSTALLATION OF PRE-TREATMENT CARTRIDGES

8.2.1 Close blue shutoff valve on both the left and right side of the system.

8.2.2 Place a tub to collect spillage under the pre-treatment cartridges (#25501). Purge air from the system by pressing on each red pressure release valve several times.

8.2.3 Unscrew the pre-treatment cartridge canister from the head, and drain contents into sink. Remove old cartridge and place in sink to drain. Remove new cartridge

from shipping bag and place in canister with rubber gasket, and arrows on the side of the canisters, facing upward.

8.2.4 Align the top ring of the cartridge with the boss on the bottom of the system head. Using one hand to support the bottom of the canister, and hold it in position, rotate the canister counterclockwise to screw it onto the head. Make sure that the grooved O-ring on the head stays in place while screwing the canister on. Continue rotating until tight. Repeat procedure for second cartridge.

8.2.5 Open blue shutoff valve on the right side of the system to allow water to fill the cartridges. Purge air from the system using the red pressure release valves one last time before opening the outlet valve on the right side of the system.

8.2.6 Record the date, the conductivity of the storage tank in the distiller, and the working hours of the distiller in the excel file, and the cartridge re-order date in the excel file (Attachment 1).

8.3 INSTALLATION OF BARNSTEAD DIAMOND PACK CARTRIDGE

8.3.1 Close the blue inlet valve connected to the tubing on the left side of the unit.

8.3.2 De-pressurize the unit. First, place in “Idle” Mode (leave the power on). From “Idle,” press the enter button. From “Air Purge?” press the up arrow. From “Depressurize” press the up arrow, then press enter.

8.3.3 Open the door to the unit. Unscrew the wingnuts positioned on top of the white connector box above the pack itself.

8.3.4 Pull out the used cartridge. Once no more water leaks out, it can be discarded.

8.3.5 Remove new cartridge from shipping bag and box. Remove the four red caps.

8.3.6 Slide new cartridge into place and replace the wingnuts attached to the connector box.

8.3.7 Open the blue inlet valve connected to the tubing on the left side of the unit.

8.3.8 Air purge the system. First, place in “Idle” Mode (leave the power on). From “Idle,” press the enter button. From “Air Purge?” press enter. Water will begin to move through each of the four packs in order, purging air. Watch to ensure that water fills all four packs. Do not stop the air purge until no more air is heard inside the system.

8.3.9 While the system is purging itself of air, unscrew the hollow fiber filter bulb from the top right hand side of the unit. Allow the water to drain out of the bulb before detaching the tubing. Re-attach tubing to new bulb before screwing it into place.

8.3.10 Once air is purged from the system, dispense water as normal out of the line. Some air will remain stuck in the filter bulb, but it will flush out over time. If air is heard inside the system, repeat the air purge. Once you are sure that water is moving through the system normally, the door can be closed.

8.3.11 Write the date and your initials on a piece of tape and attach to the diamond filter pack. Record the date in the excel file (Attachment 1), and re-order another diamond pack.

8.4 USING PCS REAGENT WATER

8.4.1 Always monitor the filling of carboys and other containers to prevent spillage and

overflow of reagent water over the counters and floors. Reagent water is expensive to produce and should not be wasted. Frequently check the digital reagent water purity meter to make sure that the resistance does not fall below 17 MΩ-cm while water is flowing.

8.4.2 Distilled deionized water (DDW) can be drawn off and stored in polyethylene carboys for use anywhere throughout the lab, as well as for field use. Keep carboys at least half full of reagent water at all times so that a sufficient supply is always available. Organic-free water (DDO) should be used directly from the polishing line, or stored in combusted glass containers (See comments section).

9 DATA REDUCTION AND STATISTICS

10 QUALITY ASSURANCE

Always run reagent blanks to check for contamination of reagent water. Monitor resistivity continuously, and change cartridges at the first sign of cartridge bleed.

11 ADDITIONAL INFORMATION

11.1 REFERENCES

11.1.1 APHA (1992) Method 1080 Reagent-Grade Water. In: Standard Methods for the Examination of Water and Wastewater. 18th Edition. American Public Health Association, American Water Works Association, Water Environment Federation. Washington, D.C.

11.1.2 Barnstead (1989) Barnstead PCS Owner's Manual. Barnstead Thermolyne Corporation. Dubuque, IA.

11.1.3 USEPA (1979) Laboratory Services (Ch. 2). In: Handbook for Analytical Quality Control in Water and Wastewater Laboratories. US Environmental Protection Agency. Environmental Monitoring and Support Laboratory, Cincinnati, OH.

11.2 COMMENTS

11.2.1 Distilled deionized water (DDW) is drawn off from the polishing system and stored in 19-liter polyethylene carboys at various locations throughout the lab. DDW is suitable for most analytical applications at the Laboratory except for trace-level organic analysis. Reagent water stored in polyethylene carboys cannot be considered to be organic-free since plasticizers can be leached from the container. If the reagent water is to be stored and used as organic-free (DDO), then it should be drawn off in pre-combusted glass containers.

11.3 ATTACHMENTS

11.3.1 EXCEL file: Water Usage.

12 PREVIOUS ISSUES AND CHANGES

Document File Name	Issue	Issue Effective Dates	Author
Water-001	001	June 9th, 1995 – June 11th, 2015	Unknown
Water-002	002	June 11th, 2015 – April 12th, 2018	Monica Hollrah
07_02_10.003 Water	003	April 12th, 2018 - Present	Victoria Wickham

12.1 ISSUE CHANGES

12.1.1 Issue 001:

- Unknown

12.1.2 Issue 002:

- Unknown

12.1.3 Issue 003:

- Moved SOP to new format
- Re-wrote SOP to accommodate new practices/water system setup
- Added/Expanded on instructions on how to change the filters to reflect current systems

13 DIAGRAMS, FIGURES, AND PHOTOGRAPHS