# **The Water Room Procedure Manual**

# **Micro/Nano Fabrication Center**

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### IV MFC Deionized Water System Cleaning Manual

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# I Overview of the Water Room

The water room has 4 major areas. They are:

- 1 Treatment of incoming city water
- 2 Production of RO
- 3 Storage and distribution of RO
- 4 Waste treatment (neutralization)

There is some area overlap between the sections; much of the waste treatment area is in the pit below the treatment of the incoming city water. The map below shows the areas of each section:



# **II Key areas** (for more details see Ultra pure Water System Manual by Dr. J. F. O'Hanlon)

#### 1. Incoming water filters (Pretreatment)

In this area city water, about half from the city of Tucson and half for UA water wells comes into the water room. The water then goes through a pre-treatment system. Then the water passes into the RO units. There are 4 different types of filters in the pretreatment area, first the water passes into the multimedia filters, and here the large particles are removed. Next is the carbon bed whose job it is to remove chlorine and reduce total oxidizable carbon (TOC) levels. The removal of the chlorine is very import, if the chlorine is not removed, the RO membranes will be damaged. The next filter is a

water softener; this is used to reduce the hardness of the water. The final filter is 5 micron particle filter.

#### 2. **Reverse Osmosis pad**

In the RO pad, the water passes into a double pass RO system. In a RO system, each membrane has 3 water I/O's, input, good output and reject.



Figure 2.1.1 Basic Reverse Osmosis membrane in case

- 1. Input of water
- 2. Output of reject water (Concentrate)
- 3. Output of product water (Permeate)

The RO system we have is a double pass RO system. Here is a basic table that shows all of the inputs and outputs of the 5 tubes and the number of membranes in each tube.

Input	Outputs		
_		Permeate (prod-	Number of
From	Concentrate (reject)	uct)	Membranes
Pretreatment	into RO #3 input	input RO #4	4
Pretreatment	into RO #3 input	input RO #4	4
RO #1 & 2 Concen-			
trate	Drain	input RO #4	4
Permeate RO1,2 & 3	input to RO # 1 & 3	input RO # 5	3
		To Polish	
RO # 4	input to RO # 1 & 3	beds/storage tank	2
	Input From Pretreatment Pretreatment RO #1 & 2 Concen- trate Permeate RO1,2 & 3 RO # 4	InputOutputsFromConcentrate (reject)Pretreatmentinto RO #3 inputPretreatmentinto RO #3 inputRO #1 & 2 ConcentrateDrainPermeate RO1,2 & 3input to RO #1 & 3RO #4input to RO #1 & 3	InputOutputsFromConcentrate (reject)Permeate (prod-uct)Pretreatmentinto RO #3 inputinput RO #4Pretreatmentinto RO #3 inputinput RO #4PretreatmentDraininput RO #4Permeate RO1,2 & 3input to RO #1 & 3input RO #5RO #4input to RO #1 & 3beds/storage tank

After the water (permeate) complete the RO membranes, it will then pass thought a 254 nm Germicidal UV lamp. This lamp is just before the two polishing beds. The polishing beds take the resistively of the water from about 500 K-ohms-cm up to greater than 18.1 Meg ohm-cm. At this point the water is sent to the storage tank

#### 3. **DI Recirculation loop**

The DI recirculation loop has as it's key item a 2,000 gal HALAR storage tank. This tank is where all the DI water is stored, it is also the source for the water that is in the DI loop that runs to all the key tools in the fab, and where the DI loop completes it path, returning any unused water back to the same storage tank. There are 2 main pumps used one at a time to pump the water in the loop. After the pumps, the water goes though a set of 6 polish mix beds to keep the resistively over 18 Meg ohms-cm. Then a .1 micron filter and a 6,000 AMU filter. There is also a second UV lamp.

#### 4. Waste neutralization system

Any DI water used in the fab or chase area will be returned to the water room for waste neutralization. It is very important that this waste stream is treated by specification. Only some chemicals my be put into this waste stream, for details see waste stream on the MSDS section of the MFC web site (http://mfc.engr.arizona.edu/safety/MSDS.htm) In general, the chemicals that can NOT go down the drain (or into the waste neutralization system) are: solvents, metals, or large amounts of HF/BOE. Carrie over into the rinse from wafer processing is OK. The waste will automatically discharge into the county waste water system and is subject to the rules

of Puma County.

### 5. Cleaning skid

This unit is not in normal use. It is used only to clean the RO or the Ultrafilter and to wash the RO unit. For Details see the section marked "IV MFC Deionized Water System Cleaning Manual"

# TAKING A WATER SAMPLE

- 1. Water samples are taken twice a month around the 1<sup>st</sup> and the 15<sup>th</sup> of each month.
- 2. Get the ZIPLOCK BAG out of the refrigerator in the water room, which contains 2 SMALL PLASTIC BOTTLES and a chain of custody sheet. Place the bottles near the sink in the water room.
- 3. Place a clean 1 LITER PLASTIC BEAKER under the gray faucet in the sink.
- 4. Check the reading of the NT3 tank, pH meter should read and in SPEC (between 6 and 8 pH), then set the effluent ball value to AUTO (OPEN POSITION). Remember, if the pH is not in SPEC then wait until it is to take a sample
- 5. Turn the switch of the pump that is currently in use from AUTO to HAND.
- 6. CLOSE that valve on under the floor behind the RO System (do not over tighten or the valve will sheer off and break).
- 7. Water should now be flowing from the faucet in the sink, if not, open the value next to the sink.
- 8. Let the water overflow over the beaker for about 2 minutes, during this time dump the beaker 3 to 5 times.
- 9. Make sure that the beaker is full when taking water sample.
- 10. OPEN the value under the floor behind the RO System to stop the flow of water to the sink.
- 11. Reset the pump from HAND to AUTO.
- 12. Calibrate the HAND HELD pH METER (Procedures below, see STEP 2).
- 13. Use the hand held pH meter to take the pH water sample from the beaker.
- 14. Take the caps off both of the sample bottles.
- 15. Fill the bottles with the water sample from the beaker (a small amount of acid in the bottles is a preservative and will causes a smoky vapor as you fill it, this is a normal).
- 16. Put caps back on bottles and dry them off with a paper towel and return bottles back into the Ziploc bag.
- 17. Take out the chain of custody sheet and fill out completely, sign in the box at the top left, date and time in the appropriate boxes, then print your name then write "UA" where it says firm. In the box that reads, "Hand Held pH =" put the value you measured on the Hand Held pH Meter.
- 18. Place the chain of custody sheet in the Ziploc bag with both bottles and place back in the refrigerator.
- 19. Call Jeff Christiansen at Risk Management, 520-621-5861 and tell him that the Micro/Nano Fabrication Center has a water sample in the refrigerator ready for pickup. If he is not in, then leave a message that sample ready for pick with time and date you called.
- 20. These WATER SAMPLES are a MANDATORY REQUIREMENT of PIMA COUNTY WASTE WATER MANAGEMENT, if not completed, lab can be fined.

# 1. CALIBRATING HAND HELD PH METER

(Digi-sense Digital pH/mV/ORP meter 5938-00pH Cole-Parmer Instruments Corp., also called Barnnt 20 model # 559-3800)

- 1. Remove the **PROBE** from the probe storage bottle.
- 2. Empty the contents of the **PROBE STORAGE BOTTLE** out into the sink.
- 3. Refill the PROBE STORAGE BOTTLE with **pH 7 BUFFER SOLUTION**. (Solution left on the shelf next to the hand held pH meter.)
- 4. Put the probe back into the probe storage bottle.
- 5. Turn on the HAND HELD pH METER.
- 6. Use the screw on the right side of the meter that is labeled "STANDARDIZE" to adjust the readings until the meter reads 7.00 +- .02.
- 7. The Hand Held pH Meter is now calibrated and ready for use.

#### 2. CALIBRATING THE PH PROBES IN THE NEUTRALIZATION SYSTEM

(Located under grated flooring, mid way between sink and salt (brine) tank. The meter is a "pH/ORP transmitter +GF+SIGNET" part # 3-8710

- 1. Safety procedure requires two persons to complete this task.
- 2. Begin by shutting off all CHEMICAL DISCHARGE PUMPS.
- 3. Fill two (50ml) beakers one with **pH 7 BUFFER SOLUTION** the other with **pH 4 BUFFER SOLUTION**.
- 4. Remove **DOOR TO PIT**, use safety rope and cones around the opening leading to pit.
- 5. **ONE PERSON** will take the two beakers down into the acid pit.
- 6. Once in the pit, pull the **pH PROBE** out of **NT1** and place it in the **pH 7 BUFFER SOLUTION.**
- 7. The person above pit needs to hold the up and down buttons on the **PROBE CONTROLLER** until it prompts for a password.
- 8. Enter the **PASS WORD**: "up, up, up, down"
- 9. Go to the STANDARD OPTION by pressing the right arrow button.
- 10. Make sure the probe is in the **pH 7 BUFFER SOLUTION**.
- 11. Use the **UP**, **DOWN**, **AND RIGHT BUTTONS** to change the reading on the display until it reads 7.00.
- 12. To save the adjustment hold the **RIGHT ARROW BUTTON DOWN FOR 2 SECONDS** or until it flashes "SAVED" on the screen.
- 13. The person in the pit can now take the **PROBE** out of the pH 7 Buffer Solution and rinse the probe in the NT tank. Then insert the probe into the beaker with pH 4 Buffer Solution.

- 14. The person above the pit then goes to the slope option and press the right arrow button
- 15. Use the up, down, and right buttons to change the reading on the display until it reads 4.00
- 16. To save the adjustment hold the **RIGHT ARROW BUTTON DOWN FOR 2 SECONDS** or until it flashes "SAVED" on the screen.
- 17. Hold down the up and down arrows until **PROGRAM MODE EXITS**.
- 18. Repeat the steps above for calibrating the probes in NT2 and NT3.

#### 3. BRINE TANK, ADDING SALT

- 1. **BRINE TANK** should be checked daily, salt should be added to the brine tank if below black line. (Tank can be filled with salt daily or every other day, but be sure to check a minimum of twice a week and the end of the week or beginning of Holiday).
- 2. Salt bags can be located on pallets by doorway to alley.
- 3. Remove **BRINE TANK LID**, open bag of salt, by pulling string, then empty bag in brine tank.
- 4. Fill the brine tank with salt to the **BLACK LINE** on the white tube in the tank.
- When the number of salt bags go below 25, it is time to order salt, let Gregg, Mike, Omid or Pat know so we can order more salt. We generally order 98 BAGS (2 PALLETS) from Home Depot, El Con Mall.

#### 4. MIX BED CYLINDER, CHANGING

- 1. If the small **LED light** on (water resistively is below 2 meg-ohm-cm) the mix bed cylinder is **RED**, mix bed needs to be changed.
- 2. There are **2 MIX BED CYLINDERS**. The first cylinder is labeled **CYLINDER 1** is and has the LED light on top of it. The second, cylinder is labeled **CYLINDER 2** is to the left of cylinder 1.
- 3. To begin changing mix beds, go to the main panel and turn the switch on the RO system to **OFF**.
- 4. Disconnect the hoses that go into CYLINDER 1 and CYLINDER 2.
- 5. Remove CYLINDER 1 and set it aside (be careful cylinders are very heavy).
- 6. Move CYLINDER 2 to the right where CYLINDER 1 was located.
- 7. Move a **NEW MIX BED CYLINDER** into the location where **CYLINDER 2** was.
- 8. Remove the white caps from the NEW MIX BED CYLINDER and place them

on the exhausted cylinder to be returned.

- 9. Reconnect the hoses to there proper places with respect to current cylinder position.
- 10. Make sure that the connections are as tight as possible, hand tightening is sufficient.
- 11. On the tag for the **NEW CYLINDER**, **WRITE THE DATE** and the **HOURS** from the **SMALL UV LIGHT** that is between the mix beds and the RO system.
- 12. Go to the main panel for the RO system and turn switch to HAND.
- 13. Watch the GAUGE next to the mix beds until it reads over 18.1 Mohms (this can take a while for the new cylinder to fill up). If the cylinder does not fill up to 18.0 Mohms, then the cylinder is defective. Call the vendor to bring out a new cylinder to replace defective one or USE EXTRA MIX BED CYLINDER, IF AVAILABLE.
- 14. Once the GAUGE reads above 18.1 Mohms switch the RO system to AUTO.
- 15. When out of **MIX BED CYLINDERS**, call the vendor and order 2 cylinders. We normally keep 2 cylinders on hand for changing cylinders as needed.
- 16. Cylinder changes should be done **TWICE A MONTH**, this depends on water usage in the lab.

#### 5. PRIMARY PUMP FOR NEUTRALIZATION SYSTEM

- This procedure is when you are switching from pump 1 to pump 2 to change from pump 2 to pump 1, change the numbers in the listings.
- 2. Turn **PUMP 1** to **OFF**.
- 3. Prepare pit with yellow chain and safety cone. Enter acid pit and close the VALUE on PUMP 1
- 4. Open the VALUE on PUMP 2.
- 5. Go to the **CONTROLLER PANEL** switch the **PUMP SELECTOR SWITCH** from **PUMP 1** to **PUMP 2**.
- 6. When finished, replace pit cover and remove the yellow chain and safety cone.
- 7. Turn PUMP 2 switch from to AUTO.
- 8. Switching NEUTRALIZATION PUMP once a month. Run on PUMP 2 (backup pump) for one week per month and run on PUMP 1 for the rest of the month.

#### 6. PRIMARY PUMP FOR RO SYSTEM

1. This procedure is when you are switching from PUMP 1 to PUMP 2 or

**PUMP 2** to **PUMP**. You can use these instructions for replacing pumps 1 to 2 or 2 to1.

- 2. Turn the **RO SYSTEM** switch to **OFF.**
- 3. Turn the RO PUMP 1 to OFF.
- 4. Turn the RO PUMP 2 to ON.
- 5. Turn the RO system to AUTO.
- 6. Switching **RO PUMP** once a month. Run on **PUMP 1** for one week per month (this is the backup pump) and run on **PUMP 2** for the rest of the month.

# 7. CHANGING PRIMARY OUTPUT PUMP FOR THE DI RECIRCULATION SYSTEM

- 1. This procedure is when you are switching from pump 1 to pump 2 or pump 2 to pump 1. You can use these instructions for replacing pumps 1 to 2 or 2 to 1.
- 2. Open valve **P2V** in
- 3. Slightly open valve **P2V** out(about 1 and a half turns)
- 4. Turn the pump 2 to **AUTO** and pump 1 switch **OFF** (both must be at the same time or it will cause a surge or disruption of water flow).
- 5. Close valve **P1V** in
- 6. Close valve P1Vout
- 7. Turn P2V out until the PRESSURE GAUGE reads 98 psi
- 8. Switch output pump once a month.
- 9. Run system on **PUMP 2** for one week per month (this is the backup pump) and run **PUMP 1** for the rest of the month.

#### 9. CHANGING POLISH BEDS, THE SIX TANKS ON THE DI LOOP

(This will be a walk through for polish bed 1 for all other polish beds just replace the follow the instructions below for changing polish beds.)

- 1. Open **PUMP VALVE**.
- 2. Close valves on PV1a and PV1b.
- 3. Disconnect the hoses going down POLISH BED 1.
- 4. Move, **POLISH BED 1** out (use caution, polish bed are very heavy).
- 5. Unscrew the **PVDF FITTINGS** attached to the top of polish bed 1.

- 6. Remove all the **TEFLON TAPE** from the fittings.
- 7. Replace with new Teflon tape on the fitting.
- 8. Place fittings back on the new polish bed.
- 9. Move the new polish bed 1 into the same location as the old polish bed 1.
- 10. Make sure to line up the right input and output hoses on the polish bed.
- 11. Reconnect the collars with the right hoses on the polish bed.
- 12. Open valve **PV1c**, then open valve **PV1a** slowly.
- 13. Let water flow to drain for about 1 minute.
- 14. Open valve **PV1b**, then close valve **PV1c**.
- 15. Repeat the above steps for replacing other polish beds. **PLEASE NOTE:** NO polish beds for **PV4** and **PV8**.
- 16. Once all beds have been changed, close PUMP VALVE.

# **10. CLEANING THE RO MEMBRANES**

- 1. Fill **RO/DI TANK** to at least 95% (250 gal tank on the skid).
- 2. TURN OFF the RO Water System using the main power breaker box. Disconnect FEED CONCENTRATE AND PERMEATE. Connect the H-TUBES to the feed and concentrate on each R/O mix bed. Connect the skid output to the feed side of the membrane. Leave the permeate open.
- 3. There are **5 TUBES** in the RO Water System. The **BOTTOM TUBE IS NUMBER 1** and the **TOP TUBE IS NUMBER 5**. The other tubes will be numbered in sequence 2-4.

# CLEANING PROCEDURES FOR EACH TUBE...

Start with RO TUBES 1 AND 2 (both tubes can be cleaned at the same time).

- 1. Fill water tank with 200 gallons of D.I. water and adjust temperature to 50 degrees Celsius. The tank temperature control system will over shoot on the temperature, it can go up to 80 C. Do not set the temperature set point above 50 degrees Celsius.
- 2. Flush 100 gallons of D.I. water through the system to drain. NOTE: Low pressure (no higher than 60psi) and no more than 24gpm
- Mix chemicals: RO Clean L403 (liquid); mix 1 gallon with the remaining 100 gallons of D.I. Water. (RO Clean L403 is from: Avista Technologies, Inc. 133 North Pacific Street San Marcos, CA 92069. Telephone 1.760.744.0536 MSDS is on line) use safety glasses to pour and mix the chemicals.
- 4. Run the first 20 gallons of this solution to NT-1 (drain), and then place the drain line back into the 250 gal cleaning tank to re-circulate.

- 5. Re-circulate the remaining 80 gallons through the system for 1 hour
- 6. Dump cleaning chemicals into NT-1
- 7. Rinse empty tank thoroughly, dumping waste into NT-1
- 8. Rinse membranes with regular city water for 15 minutes.

#### **Rinse RO3 Mix Bed**

- 1. Plug one side of the y-tube.
- 2. Fill water tank with 200 gallons of D.I. water and maintain temperature at 50 degrees Celsius. See above for details.
- 3. Flush 130 gallons of D.I. water through the system to drain. NOTE: Low pressure (no higher than 60psi) and no more than 12gpm.
- 4. Mix chemicals: RO Clean L403 (liquid); mix 1 gallon with the remaining 70 gallons of D.I. Water
- 5. Run 20 gallons of this solution to drain.
- 6. Re-circulate the remaining 50 gallons through the system for 1 hour.
- 7. Dump cleaning chemicals into NT-1.
- 8. Rinse empty tank thoroughly.
- 9. Rinse membranes with regular city water for 15 minutes.

#### **Rinse RO4 and RO5 Mix Beds**

- 1. Allow the plugged side of the y-tube to remain plugged.
- Fill water tank with 200 gallons of D.I. water and maintain temperature at 50 degrees Celsius.
- 3. Flush 100 gallons of D.I. water through the system to drain. NOTE: Low pressure (no higher than 60psi) and no more than 12gpm.
- Mix chemicals: RO Clean L403 (liquid); mix 1 gallon with 100 gallons of D.I. Water
- 5. Run 20 gallons of this solution to drain.
- 6. Re-circulate the remaining 80 gallons through the system for 1 hour.
- 7. Dump cleaning chemicals into NT-1.
- 8. Rinse empty tank thoroughly.
- 9. Rinse membranes with regular city water for 15 minutes.

#### **Repeat Steps**

Repeat all of the previous steps for the new chemical, RO Clean P111, with a ratio

of 15lbs/tube set. (RO Clean P111 is available for purchase from Avista

Technologies, Inc.)

Reconnect all **feed**, **concentrate**, and **permeate hoses** to there original configuration.

# **11. RO MEMBRANES CHANGING**

- 1. Fill the Storage tank to 100%, 2,000 gals.
- 2. Make sure that you have 17 new Membranes
- 3. Close the valve between the RO and the RO pre filters.
- 4. Turn the RO system to off
- 5. Disconnect and remove all piping that is going into the 5 filter tubes, check that the end caps have a line on them that needs to be lined up with a line on the tube, this is so that the manifolds can be reconnected properly.
- 6. Remove the snap ring from the left end of the tube and use a slide hammer to remove the end cap.
- 7. Have someone use a long skinny rod to push the membranes out from the right side of the system.
- 8. There are 4 membranes in each of the 3 bottom tubes, 3 membranes in the tube that is 4<sup>th</sup> from the bottom, and 2 membranes in the top tube. Note the order of all spacers and membranes in each tube.
- 9. Each membrane should come with an end o-ring a connector collar and 2 o-rings for the collar
- 10. Insert the collar o-rings in the collar, one on each end and rube some Glycerin on them.
- 11. The membrane should have an arrow printed on it indicating which direction water should flow through it. Put the membrane o-ring on the back end of membrane (the end opposite the direction the arrow is pointing) and rube Glycerin on it.
- 12. Rube glycerin on the ends of the membrane where the collars attach.
- 13. Each tube should have an arrow on it indicating which direction the water flows through the tube. Make sure that the arrow on the tube and the arrow on the membrane a pointing the same direction.
- 14. Insert the first membrane with a collar on the back but don't push it all the way in. Ready the next membrane/spacer and attach the front to the collar then use the second membrane to push in the first and so on until the tube is full.

- 15. Repeat this process for each tube.
- 16. Tubes 4 and 5 will also use spacers in them since they don't have 4 membranes, make sure to put the spacers in the same place they were when they were removed.
- 17. Once all membranes have been changed put the end caps back on the tubes and re insert the snap rings. The end caps should have a line on them that needs to be lined up with a line on the tube, this is so that the manifolds can be reconnected properly.
- 18. Reconnect the manifolds
- 19. Open the valves between the RO and RO pre filters
- 20. Turn the RO system back to auto.

#### **12. CLEANING THE NEUTRALIZATION SYSTEM**

- 1. Neutralization system needs to be cleaned about once a month
- 2. Preferred cleaning time is in the morning
- 3. Use a beaker to measure out 1 liter of liquid chlorine (xxx) (stored on top of the cabinet next to the pre-filtration system).
- 4. Pour the contents of the beaker into NT1
- 5. Rinse the beaker with water from the sink
- 6. Turn the out of spec ball valve switch to hand
- 7. Turn the effluent ball valve switch to off
- 8. Turn the currently selected pump switch to hand
- 9. Allow the system to circulate as long as possible without overflowing NT3
- 10. When finished set all switches back to there normal positions and let the system pump to drain.

# 14. PROGRAMMING THE AUTO-DIALER FOR THE NEUTRALIZATION SYSTEM

- 1. Press the button that says "Program"
- 2. To input the primary number press 7 01 then key in the entire phone number with a leading 1 and the area code then press "Enter"
- 3. The dialer will read back to you the number you entered and change the primary call number.
- 4. To input secondary numbers do the above steps replacing 01 with any number 02 through 16 the number will be put into the list at the select number position.

5. For further instructions see the **VERBATIM AUTO-DIALER** manual page 5.

### 15. CHARGING THE ACID AND CAUSTIC DRUMS

- 1. This should be done whenever one of the drums below the floor is at 50% or below
- 2. Use proper PPE (Apron, gloves, and face shield) Insure that there is a 55 gal. water drum next to the chemical drums that is full of water and is ready for use.
- 3. Insure that the drum is in the proper place for the transfer then open the drum of chemical to be pumped down to the holding tanks the acid is normally in the Black drum and the caustic is normally in the Blue drum, BUT YOU MUST ALWAYS CHECK!
- 4. Insert the SipHon pump into the drum and connect the hose to the proper connection on the floor. If you are facing the east wall then the connection on the right will be acid and the one on the left will be caustic.
- 5. Begin pumping until the chemical will flow on its own.
- 6. Watch the level of the chemical in the lower drum and stop the flow of chemical when it is about 4 inches from the top.
- 7. Pull the sipHon pump out of the top drum until it is not in the chemical so that it can drain, drain all the chemicals you can into one of the two drums.
- 8. Disconnect the hose from the floor connector.
- 9. Put the sipHon pump into the drum of water.
- 10. Insert the hose into the opposite side of the same drum
- 11. Pump water through the sipHon pump and then drain the water out of the sipHon pump. Return the sipHon pump to it's storage location.
- 12. Put the cap back on the chemical drum/s
- 13. About once a month the water drum should be pumped out into NT1 and refilled to about 80% with city water.
- 14. When a drum is completely drained, raise it out with water and dump the water content into NT1, repeat this process 3 or more times, when you are done, use pH paper to insure that the pH of the contents of the drum are 7 +\_\_\_\_\_1.5.

#### 16. FILLING OUT THE DAILY LOG SHEETS

1. There are 4 log sheets in the water room that need to be filled out daily one on the clipboard by the refrigerator, one on the RO system, one across from

the mix beds, and one by the loop resistively meters.

- 2. The log by the refrigerator should be filled out with the pH of NT3 and the current number of gallons pumped to drain.(make sure that you place the NT 3 pump auto/hand switch to hand and run the pump on NT3 for 60 seconds before you record the pH. If the pH of NT3 is not in spec contact lab manager or engineer. Reset the pump switch back to auto.
- 3. The log on the RO system should be filled out with the conductivity of the water going into the system, the conductivity of the water between 1<sup>st</sup> and 2<sup>nd</sup> pass and the conductivity of the water that comes out of second pass.
- 4. The log across from the mix beds should be filled out with whether the RO system is on or off and the level of water in the tank.
- 5. The log by the loop resistively meters should be filled out with the tank resistively and the loop supply resistively.

#### 17. FILLING OUT THE WEEKLY LOG SHEETS

Once a week the weekly log which is on a clipboard near the water logger computer needs to be filled out.

It should be pretty self explanatory.

Once the paper log is filled out it needs to be input into the spreadsheet file on the water logger computer.

#### **18. FILLING OUT THE MONTHLY WATER LOG**

At the beginning of every month a digital copy of the waste treatment log needs to be made from the last months data and emailed to Risk Management (**Environmental Programs** Health Safety Officer, Lloyd M. Wundrock ... 621-1590)

Get the last months waste treatment log from the water room

- Open the fill for the most resent months log in the <u>C:\MEL</u> file on the good computer in the library.
- Change the date at the top to be the month that you are filling out the log for
- Change the dates in the date column to be the dates for the month that you are filling out the log for.

Input the times that the meter was read for the corresponding day. If you don't have a value for the time on a particular day just leave it blank.

Input the values of the pH corresponding to the date they were recorded. If you don't have data for the pH on a particular day just leave it blank.

Record the dates and hand held pH values of the water samples in the area

where it says water samples.

- In cell E6 input the value that is in cell F36. If there is no value in cell F36 the use the value in cell F35.
- In the column titled current gallons of water input the values of total gallons from the log sheet. In the areas where there is no recorded value the what needs to be done is take the value before the break in data and the value after and find the difference. Then divide by the number of days that the value wasn't recorded plus 1. This will give you the average pumped over the break per day. Then take the value before the break in data and add this value to it. And input that for the first blank value and then take this new value and add the average per day to it again and input that in the next blank. Repeat this until you have data values again.
- When the log rolls over from 100000 back to 0 then for the log sheet in column F input the value with all 6 digits but where column E is supposed to equal that cell input the value as it is not as a 100000s number.
- If one of the values in column E doesn't change then it needs to be changed to equal the cell in column F that is above the cell.
- If the month doesn't have 31 days in it then delete the formatting of the cells that don't have a date.

#### 19. CALIBRATING THE NI MULTIPLIERS FOR THE PH PROBES

- These multipliers need to be calibrated at least once a month and whenever the NI water logger gets restarted.
- Look at the pH meter reading under the floor in the acid pit for NT1 and remember its value
- Adjust the multiplier for NT1 pH until the displayed value is within about +-.1 of the value displayed on the meter under the floor
- Repeat this process for the meters for NT2 and NT3
- Record the new multipliers on the text file on the computer. The file is under C:/data xxx

#### **20. REPLACING THE PH PROBES**

Remove the probe that needs to be change from the tank.

Push in the probe tip and turn it until it stops then pull it out

insert the new tip and turn it until it locks into place

Put the probe back into its tank.

Run a probe calibration on the new probe as per this document.

#### **21. BACKING UP THE NI WATER LOGS**

- Use a USB flash drive and save all the files located in the C:\data on the water logger computer.
- Move the files from the flash drive to where ever you want to store the backups, also make a CD copy of all data, mark the CD with the date and subject and give it to the lab manager.

#### 22. CHANGING THE BATTERY FOR THE PLC LOGIC CONTROLLER

This battery has been changed once in the history of the system., it should be done every 2 to 3 years

Find and record the type of battery being used. Order a new batter and replace the old one. This will keep the program in the PLC even if the power to the PLC is lost.

Record with a label the date the new battery was installed.

#### 23. CHANGING THE UV LAMP AFTER THE RO SYSTEM

Turn the RO system to off

Remove the cover of the UV lamp case

Disconnect the cap from the bottom of the bulb.

Pull the bulb up a little so that you can grab it

Remove the cap from the top of the bulb and pull it out of the case from the top.

Insert a new bulb and attach the cap to the top.

Let the bulb go all the way in to the case slowly, and then connect the cap to the bottom

Put the cover back on the case

Turn the RO system back to auto.

#### 24. CHANGING THE RO PRE-FILTERS

Turn the RO system to off

Close the valves before and after the filter container

Open the valve at the bottom on the back of the filter container to drain the water from the container. Once all the water is drained close this valve

Open the top of the container.

Remove the old filters and replace them with new, also inspect the O-ring, if bad replace.

Close the top of the container and make sure not to crimp the O-ring

Open the valve on top of the container

Open the input valve to the container

When water starts spraying from the valve on top of the container, close the top valve and open the output valve from the container.

Switch the RO system back to auto.

#### **25. CHANGING THE FF FILTERS**

Bypass the FF filter housing.

Open the top of the housing

Make sure to wear rubber gloves when changing these filters.

Unscrew the nut on that holds the filters in place

Remove the plate from the top of the filters

Remove all 6 filters

- Insert new filters with the O-rings toward the bottom of the housing making sure to push the filter into place in the bottom plate, inspect the O-ring on the top cover, if damaged, replace.
- Once all 6 filters are in put the plate back on the top of the new filters and screw it down with the nut.

Close the top and make sure not to crimp the O-ring

Open the blow off valve on top of the housing

Put the system back in line.

When water starts to spray from the blow off valve close it.

# IV MFC Deionized Water System Cleaning Manual

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# 1. Introduction

The purpose of this manual is to provide procedures and information on cleaning the MFC Deionized Water System. The MFC Deionized Water System consists of the laboratory loop, the production loop and the storage tank. The manual also describes procedures for cleaning the actual components of the system that filter and deionize the water. This manual is a work in progress, and as such, should be revised and updated as new information is obtained.

The DI water system, as any key item in the lab, needs to have as much prior notification of shutdown given as time permits. Try to give a pre DI shutdown notification notices at least 2 weeks prior. This notice should be on all lab doors, the website and e-mailed to all users (students, professors and outside users) a second notice of 2 days should also go out by the same posting.

This notice should state the length of time planed for the action (duration of flush 24 hours), but also give a down side time period up to 48 hours. "All clear" notification will be posted on doors, web site and by e-mail when complete.

Request notification from faculty/students for any tools NOT compatible or sensitivity to the H2O2 (3%) clean processes being used.

# 2. Materials

1. Two 55 gallon drums of 30% un-stabilized technical grade hydrogen peroxide.

Vendor: VWR International 1 800 932 5000 www.vwrsp.com

Hydrogen Peroxide Solution, 30%, GR ACS Product Number: HX0635-31 Unit Price: \$1,210.57

2. Five new 0.1u final filters.

Vendor: Fluid Conditioning Co. LLC 480 496 4131

CUNO Electropor ER Nylon Filter Cartridges, 0.1 Micron Rated Product Number: 70003-03H-101ER Unit Price: \$258.85

3. Six new deionizing polishing beds.

Vendor: Ionics 602 437 1355 www.ionics.com

4. One hydrogen peroxide titration test kit.

Vendor: Hach Company 1 800 227 4224 www.hach.com

Hydrogen Peroxide Test Kit, Model HYP-1 Product Number: 2291700 Unit Price: \$49.10

5. One hydrogen peroxide strip test kit.

Vendor: OzoneLab 250 265 4461 www.ozoneservices.com

QUANTOFIX Peroxide 100 No Product Number Unit Price: \$33.50

- One 55 gallon drum of reverse osmosis water.
   One pneumatic transfer pump

# 3. Cleaning Procedures

# 1. Deionized Water System Preparation

The purpose of this section is to prepare the MFC Deionized Water System for the transfer of hydrogen peroxide into the system. Before beginning this section, be sure to the UF CIP Skid tank and a 55 gallon drum with deionized water. This water will be needed later in the cleaning procedure when the system will be unable to provide deionized water. The procedures for filling the tank of the skid can be found in section 6. These procedures can also be used to fill the 55 gallon drum.

A basic floor plan of the water room can be seen in Figure 1. The floor plan should aide in determining the locations of system equipment described in the sections below.



FIGURE 1. Basic floor plan of the water room.

#### 1.1. Turn off all UV lamps in the system.

This will prevent the UV lamps from decomposing the hydrogen peroxide as it circulates through the deionized water system.

The two UV lamps are located immediately west of the HALAR tank. Turn off the UV lamps by pressing the power button on each UV lamp control panel. The first control panel is located north of the deionized water pumps. The second control panel is located west of the deionized water pumps. The locations of each panel can be seen in Figure 1. A picture of the UV control panel is shown below.



FIGURE 2. UV control panel.

1.2. Turn off the reverse osmosis (RO) water skid.

This will allow the input of the HALAR tank, through the mixed polishing bed lines, to be used for transferring the hydrogen peroxide into the deionized water system. This will also prevent the RO system from pumping water into the 2000-gallon HALAR tank during the hydrogen peroxide circulation, allowing the correct solution of hydrogen peroxide to be maintained throughout the cleaning process.

Both RO water pumps are located near the RO control panel. The first RO pump is north of the RO control panel. The second RO pump is below the RO control panel. The control panel can be found along the north wall of the water room above the neutralization pit. The locations of the RO water skid and the RO control panel can be seen in Figure 1.

Note: The system has two RO pumps, one of which is on at one time. Thus, by turning off the RO pump that is on, both pumps will be off.

The RO pump switch, which is labeled "RO System," is located in the center of the east most RO control panel. Turn off the RO pump by turning the pump's switch from the auto position to the off position. The RO control panel is shown below.



FIGURE 3. RO control panel.

1.3. Turn off the deionized water pumps.

This will prevent disrupting the transfer of hydrogen peroxide.

The two deionized water pumps are located on the ground beneath the system's UV lamps. Turn off the deionized water pumps by turning the each pump's switch from the auto position to the off position. The deionized water pump switches are located on the control panel south of the deionized water pumps as seen in Figure 1. A picture of this control panel can be seen in Figure 4.

Note: The system has two deionized water pumps, one of which is on at one time. Thus, by turning off the deionized water pump that is on, both pumps will be off.



FIGURE 4. Deionized water pump control panel.

1.4. Close all normal drains in the facility.

This will prevent the hydrogen peroxide solution from being consumed during the hydrogen peroxide circulation.

Be sure to open the bypass lines of each normal drain in the facility. This will eliminate dead legs and allow the hydrogen peroxide to constantly flow through out the system.

The following list indicates the normal drains in the facility.

- Photolithography Sink (Two Drains)
- Photolithography SRDs (Two Drains)
- LPCVD Sink (Two Drains)
- Raised Class 10 Sink (Several Drains)
- Raised Class 10 SRD (One Drain)
- Climax Bay CMP (Several Drains)
- Climax Bay SRDs (Three Drains)
- Climax Bay Chase Sink (One Drains)
- Chase Sink (Two Drains)
- Class 10 CMP (Several Drains)
- Class 10 Sink (Several Drains)
- Dicing Saw (One Drain)
- Graduate Research Sink (Jun) (One Drain)

1.5. Bypass all deionizing polishing beds.

This will prevent the hydrogen peroxide solution from entering the deionizing polishing beds, which can damage the filter media in the beds.

The deionizing polishing beds are located near the west wall of water room as seen in Figure 1. Open the large bypass valve located at the west end of the deionizing polishing bed loop. This valve, labeled PVmb, is shown below.



FIGURE 5. Deionizing polishing bed bypass valve, PVmb.

Close all input, output and drain valves of the deionizing polishing beds. This includes all PV1, PV2, PV3, PV4, PV5, PV6, PV7 and PV8 valves. Disconnect and remove all of the deionizing polishing beds. Have these beds picked up by the vendor after the deionizing water system clean has been completed.

1.6. Remove all 0.1u final filters from the system.

This will prevent exposing the 0.1u final filters to the hydrogen peroxide, as hydrogen peroxide will damage the filters. The final filters can tolerate a hydrogen peroxide concentration between 3% and 5% for one hour.

The 0.1u final filters reside in a filter housing located in the northwest corner of the water room. The location of the 0.1u final filters and the filter housing can be seen in Figure 1. To remove the filters from the filter housing, open the filter housing by loosening, but not completely removing, the nuts of the bolts that fasten the top of the filter housing to the

body of the filter housing. Open the top of the filter housing.

Note: The top of the filter housing may need to be pried off the filter housing body using a crow bar. Take care when prying the top of the filter housing to avoid damaging the unit.

Remove the gasket and o-ring from top lip of the filter housing body. Loosen and remove the Teflon nut that secures the top Teflon filter support. Remove the top Teflon filter support. Remove all five 0.1u final filters simply by pulling them up and out of the slots in the bottom Teflon filter support.

Note: Only an o-ring secures the 0.1u final filters to the bottom Teflon filter support. The pressure exerted by the o-ring on the walls of the slots in the bottom Teflon filter support secures the filters to the housing. This is depicted in the schematic in Figure 6.



FIGURE 6. Schematic of the connection of the 0.1u final filters and the bottom Teflon filter support.

Place the Teflon filter support back into the filter housing and secure it with the Teflon nut. Place the gasket and o-ring back onto the top lip of the filter housing body. Close the top of the filter housing. Fasten the top of the filter housing to the filter housing body by tightening the nuts on the bolts.

Note: Although the 0.1u final filters are removed from their housing, the housing itself should not be bypassed during the hydrogen peroxide circulation. This will allow the hydrogen peroxide to clean the filter housing.

1.7. Bypass both Ultrafiltration Modules in the system.

This will ensure that the hydrogen peroxide will not damage the Ultrafiltration Modules during the beginning of the hydrogen peroxide circulation, when the hydrogen peroxide concentration of hydrogen peroxide is close to 3%. A 3% concentration of hydrogen

peroxide is the maximum concentration the modules can withstand.

The Ultrafiltration Modules are located along the north wall in the northwest corner of the water room. See Figure 1. Insert the bypass piece between valves V(UF-XX) and V(UF-YY). Open these valves. Close valves V(UF-11), V(UF-12), V(UF-21) and V(UF-22) to isolate the modules from the rest of the deionized water system. See Figure 7.



FIGURE 7. UF Modules schematic.

1.8. Clean the neutralization pit.

This will prevent the sump pump in the neutralization pit from clogging. This becomes extremely important when flushing the hydrogen peroxide from the system, which entails draining the HALAR tank into the neutralization pit.

Using city water from a standard garden hose, flush all debris in the neutralization pit into the sump area. Remove as much debris as possible from the sump area.

1.9. Drain Neutralization Tank (NT) 3.

This will ensure enough volume in NT3 to neutralize all of the water from the hydrogen peroxide flush.

Both of the neutralization discharge pumps are located on top of NT3. Each pump's switch can be found on the neutralization control panel on the east wall of the water room. See Figure 1 for the location of the neutralization control panel. Place the neutralization discharge pump into manual mode by moving the pump's switch from the auto position to the manual position. The neutralization control panel can be seen below.



FIGURE 8. Neutralization control panel.

Note: The system has two neutralization discharge pumps, one of which is on at one time. Thus, by turning off the neutralization discharge pump that is on, both pumps will be off. 1.10.Adjust the water level in the HALAR tank.

This will allow the correct concentration of the hydrogen peroxide to be obtained in the deionized water system.

Adjust the water level in the HALAR tank to 50% of the tank's capacity, or 1000 gallons. 110 gallons of 30% hydrogen peroxide, will be transferred into the HALAR tank. The total solution will be in the amount of 1110 gallons, 110 gallons of which will be hydrogen peroxide. This results in a solution of approximately 3% hydrogen peroxide. You will also need one drum(55 gals) of deionized water to purge the lines.

If the HALAR tank has less than 50 % of its capacity in use, add water to the tank using the RO system. Turn on the RO pump by turning the pump's switch from the off position to the hand, or manual position. Once the HALAR tank has 50% of its capacity in use, turn off the RO pump by turning the pump's switch from the hand position to the off position.

If the HALAR tank has more the 50% of its capacity in use, open the valve labeled TVdr, located at the bottom of the northwest arch of the HALAR tank. This will drain the water from the tank into the pit of neutralization system. Once the HALAR tank has 50% of its capacity in use, close the valve at the bottom of the tank. The location of this valve can be found in Figure 1. The valve itself can be seen below.



FIGURE 9. The HALAR tank drain valve, TVdr.

# 2. Hydrogen Peroxide Transfer

This section details the procedures used to transfer the two 55 gallon drums of hydrogen peroxide into the deionized water system.

2.1. Don personal protective equipment.

This will protect the person monitoring the hydrogen peroxide pneumatic pump from hydrogen peroxide splashes or leaks.

The transfer of hydrogen peroxide into the HALAR tank requires at least two people. One person must monitor the pneumatic pump at all times checking for leaks, stabilizing the pump and coordinating with the second person to power the pump. The second person will control the feed line into the pump.

2.2. Hydrogen peroxide pneumatic pump set up.

The hydrogen peroxide will be transferred using a pneumatic pump. The pneumatic pump can be seen in Figure 10.



FIGURE 10. Pneumatic pump.

Place the pneumatic pump in a large plastic bin to isolate the pump and protect other equipment from hydrogen peroxide leaks and/or splashes. Position a stand or cart near the mixed polishing beds located along the north wall of the water room, west of the RO system. The location of the mixed polishing beds can be seen in Figure 1. Place the bin with the pneumatic pump on the cart or stand so that the top of the bin is chest high. The height of the bin will aid in connecting the pneumatic pump to the input of the HALAR tank, which is through the mixed polishing bed lines.

Note: The cart or stand must be sturdy, as the pneumatic pump will vibrate.

2.3. Connect the pneumatic pump to the deionized water system.

Connect the input of the pump to a clean piece of Teflon tubing five feet in length. The other end of this tubing will be put into the drums of hydrogen peroxide. Connect the output of the pump to the output line of the second mixed polishing bed. Figure 11 shows the output line of the second mixed polishing bed. This line leads directly into the HALAR tank.



FIGURE 11. Mixed Polishing Beds.

Power the pump by connecting the pump's feed line to a compressed air or nitrogen gas line. Several gas lines can be found in between the HALAR tank and the UV lamps, as shown in Figure 1. A picture of these gas lines can be seen below.



FIGURE 12. Pneumatic pump gas lines.

2.4. Transfer the hydrogen peroxide into the HALAR tank.

Place the free end of the Teflon tubing connected to the pump's input into one of the drums of hydrogen peroxide. Begin pumping the hydrogen peroxide into the HALAR tank by opening the gas valve allowing gas into the pneumatic pump's feed line. When drum of hydrogen peroxide is empty, indicated by large quantities of air in the pump's input line, turn off the pump. Place the free end of the pump's input Teflon tubing in the second drum of hydrogen peroxide and transfer it into the HALAR tank. When the drum of hydrogen peroxide is empty, turn off the pump. Pour any hydrogen peroxide left in the drums into a plastic bin. Transfer the remaining hydrogen peroxide into the HALAR tank.

Note: The person monitoring the pneumatic pump must wear PPE.

2.5. Transfer deionized water into the HALAR tank.

This will flush the input and output lines of the pneumatic pump and the output line of the mixed polishing beds. It is important that this step be completed, as any hydrogen

peroxide left in the output line of the mixed polishing beds can damage the filter media in the beds.

Place the free end of the Teflon tubing connected to the pump's input into the drums deionized water. Begin pumping the water into the HALAR tank by opening the gas valve allowing gas into the pneumatic pump's feed line. Pump approximately 15 gallons of water into the HALAR tank. Turn off the pneumatic pump.

2.6. Disconnect the pneumatic pump to the deionized water system.

After both drums of hydrogen peroxide have been emptied into the HALAR tank, disconnect the pneumatic pump's input and output lines and gas feed line. Reconnect the output line of the mixed polishing beds to the beds.

Note: The mixed polishing beds will be needed to fill the HALAR tank during the hydrogen peroxide flush.

# 3. Hydrogen Peroxide Circulation

This section describes the process of circulating the 3% hydrogen peroxide through the facility.

3.1. Turn on the deionized water pumps.

This will allow the 3% hydrogen peroxide solution to circulate through the deionized water system.

Turn on the deionized water pump by moving the pump's switch from the off position to the manual position.

Note: Placing the pump in the manual mode will allow the pump to operate when the HALAR tank capacity is below 30%. The deionized water pumps must not operate when the level of the HALAR tank is below 10%.

Also, open the valve of the deionized water pump not in use to allow hydrogen peroxide to clean the pump's input and output lines, but do not turn the pump on. Both deionized water pumps should not be on simultaneously.

3.2. Allow the hydrogen peroxide solution to circulate.

Allow the 3% hydrogen peroxide solution to continuously circulate through the system for three hours.

Note: During this time, the percentage of the HALAR tank in use should remain fairly constant, fluctuating by 3%. If the percentage of the HALAR tank drops more than 5%, a normal drain in the facility is open or a leak exists. Either of these problems should be tended to immediately.

3.3. Open all normal drains in the facility.

This will allow the hydrogen peroxide solution to flow through each entire line of the system, as well as clean the normal drains themselves.

Open one normal drain in the facility at a time. Allow each drain to consume approximately 1% of the HALAR tank's capacity, or 20 gallons of the hydrogen peroxide solution. Close the drain after it has been cleaned.

This process requires at least two people. One person must open the normal drains in the facility. The second person must monitor the HALAR tank percentage. Both people

need to be in contact with each other in order to open and close the normal drains at the proper time.

Note: During this process, do not allow the HALAR tank level to drop below 10%. If this occurs, turn off the deionized water pump and add water to the tank by moving the RO pump switch from the off position to the manual position. Stop adding water to the tank when the amount of water in the tank is large enough to complete the cleaning of the normal drains without the level of the tank dropping below 10%.

3.4. Turn off the deionized water pumps.

This will prevent breaking any lines or valves when introducing the Ultrafiltration Modules into the system.

Turn off the deionized water pump by moving the pump's switch from the manual position to the off position.

3.5. Introduce the Ultrafiltration Modules into the system.

This will allow the 3% hydrogen peroxide solution to clean the modules. As stated above, a concentration of 3% hydrogen peroxide is the maximum concentration the modules can withstand, by this time in the process, the hydrogen peroxide concentration will be at or below 2%.

To introduce the Ultrafiltration Modules into the system, open valves V(UF-11), V(UF-12), V(UF-12) and V(UF-22). Close valves V(UF-XX) and V(UF-YY). See Figure 1.

3.6. Turn on the deionized water pumps.

This will allow the 3% hydrogen peroxide solution to circulate through the system, which now includes the Ultrafiltration Modules.

Turn on the deionized water pump by moving the pump's switch from the off position to the manual position.

Note: Placing the pump in the manual mode will allow the pump to operate when the HALAR tank capacity is below 30%. The deionized water pumps must not operate when the level of the HALAR tank is below 10%.

3.7. Allow the hydrogen peroxide solution to circulate.

Allow the 3% hydrogen peroxide solution to continuously circulate through the system and the Ultrafiltration Modules for one hour.

# 4. Hydrogen Peroxide Flush

This portion of the manual specifies the procedures used to flush the hydrogen peroxide out of the deionized water system. It is extremely important that the system be flushed thoroughly to remove all, below 0.2ppm, hydrogen peroxide. Any hydrogen peroxide left in the system could damage system components.

4.1. Turn off the deionized water pumps.

This will prevent damaging the pumps after the deionized system has been completely drained.

The deionized water pump can be turned off by moving the pump switch from the manual position to the off position.

#### 4.2. Bypass both Ultrafiltration Modules in the system.

To bypass the Ultrafiltration Modules, open valves V(UF-XX) and V(UF-YY). Close valves V(UF-11), V(UF-12), V(UF-21) and V(UF-22). See Figure 7. This will isolate the modules from the rest of the deionized water system.

Note: The Ultrafiltration Modules will be cleaned and flushed using the UF CIP Skid. This process will occur during the hydrogen peroxide flush. The procedures for using the UF CIP Skid can be found in Section 6.

4.3. Empty the HALAR tank.

This will drain the majority of the hydrogen peroxide solution still in the deionized water system.

Open valve TVdr, which is located at the bottom of the northwest arch of the HALAR tank to drain the water from the tank into the pit of neutralization system. See Figure 1 for the location of the valve and Figure 9 for a picture of the valve itself. Once the HALAR tank is empty, close the valve.

Note: The seven-segment display that shows the percentage of the tank in use will never read zero. Thus, to determine if the tank has been completely emptied, check the HALAR tank's drain line in the pit of the neutralization system. If the hydrogen peroxide solution no longer exits the line to, the tank is empty.

4.4. Open all normal drains in the facility.

This will purge the hydrogen peroxide solution remaining in the deionized water system loop.

Open all normal drains in the facility simultaneously. Once all of the normal drains have run dry, close them. This includes the lines of the deionizing polishing bed lines.

4.5. Turn on the reverse osmosis water pump.

This will add clean water to the HALAR tank and further dilute the hydrogen peroxide solution.

Turn on the RO system by moving the RO pump's switch from the off position to the auto position. Once the HALAR tank more than 25% of its capacity in use, turn off the RO pump by moving the RO pump's switch from the hand position to the off position.

4.6. Turn on the deionized water pump.

This will allow the diluted hydrogen peroxide solution to circulate through the deionized water system.

The pump can be turned on by moving the pump switch from the off position to the manual position.

Note: Placing the pump in the manual mode will allow the pump to operate when the HALAR tank capacity is below 30%.

Also, open the valve of the deionized water pump not in use to allow hydrogen peroxide to clean the pump's input and output lines, but do not turn the pump on. Both deionized water pumps should not be on simultaneously.

4.7. Allow the diluted hydrogen peroxide solution to circulate.

Allow the diluted hydrogen peroxide solution to continuously circulate throughout the system for 10 to 20 minutes.

4.8. Open all normal drains in the facility.

This will allow the diluted hydrogen peroxide solution to flush each line of the system.

Open all normal drains in the facility simultaneously. This includes the deionizing

polishing bed lines. Keep these drains open until the HALAR tank has 15% of its capacity in use. Once the HALAR tank is using only 15% of its capacity, close all of the normal drains.

4.9. Turn off the deionized water pumps.

This will prevent damaging the pumps after the deionized system has been completely drained.

Turn off the deionized water pump by moving the pump's switch from the manual position to the off position.

4.10.Empty the HALAR tank.

This will drain the remainder of the diluted hydrogen peroxide solution still in the HALAR tank.

Open the valve located at the bottom of the northwest arch of the HALAR tank to drain the water from the tank into the pit of neutralization system. Once the HALAR tank is empty, close the valve used to drain the tank.

4.11.Repeat the hydrogen peroxide flush process.

Repeat sections 4.4 through 4.9 two more times. Afterwards, testing for hydrogen peroxide can begin.

# 5. Hydrogen Peroxide Testing

(Note: The Hach Hydrogen Peroxide Test Kit procedures can be found in the Appendix section 2.4).

This section describes testing the deionized water system for hydrogen peroxide. The hydrogen peroxide testing requires that the HALAR tank be filled to at least 25% of its capacity and that one of the deionized pumps be turned on to circulate the water through the system.

#### 5.1. 1pp to 100ppm hydrogen peroxide testing.

This will allow the MFC to test for higher concentrations of hydrogen peroxide in the deionized water system.

- 1. Turn on one of the sinks in the chase and let it run for 5 minutes.
- 2. Rinse a clean beaker in the water from the sink.
- 3. Fill half of the beaker with water from this sink.
- 4. Dip a QUANTOFIX Peroxide 100 test strip into the water sample and measure the amount of hydrogen peroxide in the water.
- 5. If more than 1ppm of hydrogen peroxide exists in the deionized water system, repeat sections 4.4 through 4.9 again.
- 6. If less than 1ppm of hydrogen peroxide exists in the deionized water system, proceed to section 5.2.

# 5.2. 0.2ppm to 10ppm hydrogen peroxide testing.

This will also allow the MFC to test for lower concentrations of hydrogen peroxide in the deionized water system.

- 1. Turn on one of the sinks in the chase and let it run for 5 minutes.
- 2. Rinse all glassware from the Hach Hydrogen Peroxide Test Kit with water from the sink.
- 3. Fill the glass sample cell to the bottom of the line marked at the top of the sample cell.
- 4. Add 1mL of Ammonium Molybdate solution to the sample cell.

Note: The dropper used for the Ammonium Molybdate bottle cap is marked at 0.5mL and 1.0mL.

- 5. Open one Sulfite 1 Reagent powder pillow and add the contents of the pillow to the sample cell.
- 6. Cap the sample cell with the white plastic cap and invert repeatedly to mix.

7. Wait five minutes before proceeding.

Note: This is the prepared sample.

Note: Not all of the powder must dissolve. If hydrogen peroxide is present a transparent blue color will develop. The color can be very dark, but it will be transparent. Any opaque or cloudy mixtures signify sample contamination, such as chlorine, or more than 10 mg/L of hydrogen peroxide.

# High Range Test Instructions (1 drop = 1 mg/L hydrogen peroxide):

- 1. Fill the plastic measuring tube full with the prepared sample.
- 2. Pour the sample from the plastic tube into the flask.
- 3. Add Sodium Thiosulfate Titrant drop by drop to the flask, while constantly swirling the solution.

Note: Do not pause for more than 15 seconds while adding the drops, as this will cause low results.

- 4. Count each drop as it is added.
- 5. Continue to add Sodium Thiosulfate Titrant until the sample loses all blue color.

Note: Each drop used to bring about the color change is equal to 1mg/L, 1ppm, of hydrogen peroxide. As soon as possible, rinse the glass sample cell and cap, the plastic measuring tube and the flask with clean water.

If more than 1ppm of hydrogen peroxide exists in the deionized water system, repeat sections 4.4 through 4.9 again.

# Low Range Test Instructions (1 drop = 0.2mg/L hydrogen peroxide):

- 1. Pour the entire prepared sample from the glass sample cell into the flask.
- 2. Add Sodium Thiosulfate Titrant drop by drop to the flask, while constantly swirling the solution.

Note: Do not pause for more than 15 seconds while adding the drops, as this will cause low results.

- 3. Count each drop as it is added.
- 4. Continue to add Sodium Thiosulfate Titrant until the sample loses all blue color.

Note: Each drop used to bring about the color change is equal to 0.2mg/L, 0.2ppm, of hydrogen peroxide. As soon as possible, rinse the glass sample cell and cap, the plastic measuring tube and the flask with clean water.

If more than 0.2ppm of hydrogen peroxide exists in the deionized water system, repeat

sections 4.4 through 4.9 again.

When the results of the test indicate there is less than 0.2ppm of hydrogen peroxide in the deionized water system, the hydrogen peroxide flush is complete.

It may take 8 or my DI water cycles to drop the hydrogen peroxide levels below 0.2 ppm.

# 6. UF CIP Skid Cleaning Procedures

This section details the cleaning procedures of the Ultrafiltration Module using the UF CIP Skid. The UF CIP Skid is a hot water sanitizer, thus no chemistry is needed. The UF CIP Skid cleaning procedures requires that the Ultrafiltration Modules already be bypassed. Before using the skid, rinse and clean it two times using DI water.

6.1. Fill the skid tank.

This water will be used to clean the Ultrafiltration Modules.

The sections of hose used to connect the Ultrafiltration Modules to the UF CIP Skid can be found in a white cardboard box located on the top of the tank of the skid.

Connect the small skid hose from the deionized water output valve. This valve is located on the north wall of the water room, east of the Ultrafiltration Modules. Its location can be seen in Figure 1. Below is a picture of the valve.



FIGURE 13. Deionized water output valve.

Connect the other end of this hose to any of the inputs of the UF CIP Skid tank, or place it in the tank itself. Open the valve and fill 95% of the tank with water.

Note: The skid must be filled with deionized water to prevent the filters from becoming



more polluted. Figures 14 and 15 show schematics of the UF CIP Skid.

FIGURE 14. General UF CIP Skid schematic.



FIGURE 15. UF CIP Skid schematic.

6.2. Connect the skid to the Ultrafiltration Modules.

Connect one end of one of the large skid hoses to the input of the Ultrafiltration Modules. The input is shown in Figure 16. Open valves V(UF-14) and V(UF-24).



FIGURE 16. Bottom portion of the UF Module schematic with the input indicated.

Connect the other end of this hose to the output of the UF CIP Skid. The output of the skid is indicated in Figure 17.



FIGURE 17. Top view of the UF CIP Skid with the output indicated.

Connect the second large skid hose to the output of the Ultrafiltration Modules. The output is shown in Figure 18. Open valves V(UF-13) and V(UF-23). Also, open the UF Modules output valve, UFVclrt.



FIGURE 18. Top portion of the UF Modules schematic with the output indicated.

Connect the other end of this skid hose to the input of the UF CIP Skid. The input of the skid is indicated in Figure 19.



FIGURE 19. Top view of the UF CIP Skid with the input and secondary input indicated.

6.3. Open the Ultrafiltration Modules drain valve.

This will ensure that the contaminated water from the reject line of the Ultrafiltration Modules flows to drain and not back into the HALAR tank. A schematic of the Ultrafiltration Modules can be seen in Figure 20.



FIGURE 20. Schematic of the Ultrafiltration Module.

Open the Ultrafiltration Modules drain valve located on the north wall of the water room, west of the Ultrafiltration Modules. The drain valve, UFVcldr, can be found in Figure 1.

Close the HALAR tank reclaim valve. This valve is located next to the Ultrafiltration Modules drain valve on the north wall of the water room, west of the modules.

6.4. Turn on the UF CIP Skid.

Turn the UF CIP Skid on by moving the skid's breaker switch from the off position to the on position. The UF CIP Skid breaker switch, shown in Figure 21, includes breakers 38, 40 and 42.



FIGURE 21. UF CIP Skid breaker switch.

The skid's breaker switch can be found inside the breaker panel along the west wall of the water room. The breaker panel can be found in Figure 1.

Note: The breaker switch is in the off position if the switch is on the right side. The breaker switch is in the on position if the switch is on the left side.

6.5. Turn on the sanitization pump.

This will allow the water in the tank of the UF CIP Skid to circulate through the Ultrafiltration Modules.

Turn on the sanitization pump by pressing the green start button on the control panel of the skid.



FIGURE 22. UF CIP Skid control panel.

6.6. Adjust the temperature set point.

The temperature set point is the temperature at which the heating unit of the UF CIP Skid will heat the water in the skid's tank to.

To adjust the temperature set point, press the button labeled "p" on the TI-703 PID controller located on the control panel of the skid. This will allow the user to view the temperature set point stored in the controller. We want the system to run at  $80 + 5^{\circ}$  C.

The controller does not control the over shoot of the temperature very well, set the control to a temperature set point of  $60^{\circ}$  C,

Note: The maximum temperature of water that can be used without damaging the modules is 90° C. Thus, do not adjust the temperature set point above this point.

6.7. Turn on the heating unit.

Turn on the heating unit of the UF CIP Skid by turning the heating unit's switch from the off position to the on position.

6.8. Allow the water temperature to ramp up.

Allow the water temperature to ramp up as it circulates through the Ultrafiltration Modules. The temperature ramp should take at least 15 minutes.

The temperature of the water is ramped up while the water circulates through the Ultrafiltration Modules. This avoids thermally shocking the modules, which will shorten the life of the modules.

The controller does not control the over shoot of the temperature very well, set the control to a temperature set point of  $60^{\circ}$  C, Let the system heat up, the temperature should not go over 85.

If the temperature stabilizes below 75° C, slowly increase the set point by pressing the button with an arrow pointing upwards; if the temperature stabilizes above 85° C slowly decrease the set point by pressing the button with an arrow pointing downwards.

Note: The maximum temperature of water that can be used without damaging the modules is 90° C. Thus, do not adjust the temperature set point above this point.

6.9. Allow the hot water to circulate.

Once the water temperature has reached  $80 + 5^{\circ}$  C, allow the water to circulate through the Ultrafiltration Modules for one hour, keep an eye on the temperature, if the temp goes over  $82^{\circ}$  C, shut down the heater, till the temp drops below  $73^{\circ}$  C.

6.10.Allow the water temperature to ramp down.

Allow the water temperature to ramp down to the ambient temperature of the water room as the water circulates through the Ultrafiltration Modules. The temperature ramp should take at least 15 minutes.

Allowing the water temperature to ramp down is just as important as allowing the water temperature to ramp up. This avoids thermally shocking the modules, which will shorten the life of the modules.

Simply turning off the heating unit should suffice. Turn off the heating unit by turning the heating unit's switch from the on position to the off position.

6.11.Turn off the sanitization pump.(after the water temperature is within 5° C of room temperature.

This will stop the circulation of water through the Ultrafiltration Modules.

Turn off the sanitization pump by pressing the red stop button on the control panel of the skid.

6.12.Disconnect the skid from the Ultrafiltration Modules.

Close valves V(UF-13), V(UF-23), V(UF-14), V(UF-24) and UFVclrt. These can be seen in Figures 16 and 18.

Disconnect all skid hoses from the skid and the Ultrafiltration Modules and place them back into the white cardboard box. This includes two large skid hoses and one small skid hose. Place the box on top of the tank of the skid.

6.13.Drain the skid tank.

Connect one end of a standard garden hose to the drain of the skid. Place the other end of the hose in the neutralization pit. Open the drain valve. Once all of the water had drained from the skid, close the drain valve and disconnect the garden hose. The drain valve can be seen in Figure 9.



FIGURE 23. Top view of the UF CIP Skid with the drain indicated.

6.14. Turn off the UF CIP Skid.

Turn off the UF CIP Skid by moving the skid's breaker switch from the on position to the off position. The UF CIP Skid breaker switch includes breakers 38, 39 and 40.

# 7. Final Deionized Water System Preparation

This section details the final preparation of the deionized water system. It describes bringing the system back into normal operating mode. Thus, it is the last section of the cleaning process. At this point the HALAR tank should be at least 30% full, and one of the deionized water pumps should be on.

7.1. Turn on the reverse osmosis water pump.

This will fill the HALAR tank to the appropriate percentage.

Turn on the RO system by moving the RO pump's switch from the off position to the auto position. Leave the RO pump in this state.

7.2. Turn off the deionizing water pump.

This will prevent water from entering the 0.1u final filter housing, allowing the new 0.1u final filters to be installed.

Turn off the deionized water pump by turning the pump's switch from the manual position to the off position.

7.3. Install the new 0.1u final filters.

Note: Wear latex gloves to avoid contaminating the 0.1u filters with oil from skin.

Open the 0.1u final filter housing by loosening, but not completely removing, the nuts of the bolts that fasten the top of the filter housing to the body of the filter housing. Open the top of the filter housing.

Note: The top of the filter housing may need to be pried off the filter housing body using a crow bar. Take care when prying the top of the filter housing to avoid damaging the unit.

Remove the gasket and o-ring from top lip of the filter housing body. Loosen and remove the Teflon nut that secures the Teflon filter support. Remove the Teflon filter support. Install the 5 new 0.1u final filters into the filter housing by simply inserting a filter into each of the 5 slots. Apply pressure to fit the filters in the slots snuggly.

Note: Only an o-ring secures the 0.1u final filters to the bottom Teflon filter support. The pressure exerted by the o-ring on the walls of the slots in the bottom Teflon filter support secures the filters to the housing. This is depicted in the schematic in Figure 6.

Place the Teflon filter support back into the filter housing. Verify that the tops of the five 0.1u final filters are inserted into the five cutouts in the Teflon support. Secure the Teflon support with the Teflon nut. Place the gasket and o-ring back onto the top lip of the filter housing body. Close the top of the filter housing. Fasten the filter housing top to the filter housing body by tightening the nuts on the bolts.

Note: When the deionized water pump is turned on the new 0.1u filters will cause a drop in the resistivity of the water. This is due to a large amount of air that is in each filter. The resistivity of the water should return to normal or 18.2 M $\Omega$ , in two to three hours.

7.4. Introduce the Ultrafiltration Modules into the system.

Close valves V(UF-14), V(UF-15), V(UF-24) and V(UF-25). Open valves V(UF-11), V(UF-12), V(UF-21) and V(UF-22). Finally, close valves V(UF-XX) and V(UF-YY). Use Figure 7 to aid in closing and opening the valves.

7.5. Turn on the deionized water pump.

This will allow deionized water to circulate through the deionized water system.

Turn on the deionized water pump by moving the pump's switch from the off position to the auto position.

7.6. Introduce the new deionizing polishing beds into the system.

This is the final step of the deionized water system clean.

Before connecting the new deionizing polishing beds, drain two gallons of water from each of the input and output valves of the polishing bed lines. Drain each valve individually.

Connect the new deionizing polishing beds to the polishing bed lines. There should only be six deionizing polishing beds.

Introduce each bed individually. Open the drain valve of the first deionizing polishing bed. Open the input valve of the bed.

Note: It is normal for the valve and bed line to vibrate and produce a hissing noise when opening the valve.

Wait 30 to 60 seconds for the water to fill the bed.

Note: Since the drain valve is open, the water exiting the bed will flow to drain, ensuring

that any particulates loose in the bed are removed and do not proceed to the next stage of the system.

Open the output valve of the bed. Close the drain valve of the bed. Follow these procedures for each bed.

After all deionizing polishing beds have been introduced, close the large bypass valve, PVMB, located at the west end of the deionizing polishing bed loop.

# 8.0 CLEANING THE RO MEMBRANES

- 1. Fill **RO/DI TANK** to at least 95% (> 1900 gals in the tank on the skid).
- 2. **TURN OFF** the RO Water System using the main power breaker box. Disconnect **FEED CONCENTRATE AND PERMEATE**. Connect the **H**-**TUBES** to the feed and concentrate on each R/O mix bed. Connect the skid output to the feed side of the membrane. Leave the permeate open.
  - 3. There are **5 TUBES** in the RO Water System. The **BOTTOM TUBE IS NUMBER 1** and the **TOP TUBE IS NUMBER 5**. The other tubes will be numbered in sequence 2-4.

### **CLEANING PROCEDURES FOR EACH TUBE...**

Start with **RO TUBES 1 AND 2** (both tubes can be cleaned at the same time).

- 9. Fill water tank with 200 gallons of D.I. water and adjust temperature to 50 degrees Celsius. The tank temperature control system will over shoot on the temperature, it can go up to 80 C. Do not set the temperature set point above 50 degrees Celsius.
- 10. Flush 100 gallons of D.I. water through the system to drain. NOTE: Low pressure (no higher than 60psi) and no more than 24gpm
- Mix chemicals: RO Clean L403 (liquid); mix 1 gallon with the remaining 100 gallons of D.I. Water. (RO Clean L403 is from: Avista Technologies, Inc. 133 North Pacific Street San Marcos, CA 92069. Telephone 1.760.744.0536 MSDS is on line) use safety glasses to pour and mix the chemicals.
- 12. Run the first 20 gallons of this solution to NT-1 (drain), and then place the drain line back into the 250 gal cleaning tank to re-circulate.
- 13. Re-circulate the remaining 80 gallons through the system for 1 hour
- 14. Dump cleaning chemicals into NT-1
- 15. Rinse empty tank thoroughly, dumping waste into NT-1
- 16. Rinse membranes with regular city water for 15 minutes.

#### **Rinse RO3 Mix Bed**

10. Plug one side of the y-tube.

- 11. Fill water tank with 200 gallons of D.I. water and maintain temperature at 50 degrees Celsius. See above for details.
- 12. Flush 130 gallons of D.I. water through the system to drain. NOTE: Low pressure (no higher than 60psi) and no more than 12gpm.
- 13. Mix chemicals: RO Clean L403 (liquid); mix 1 gallon with the remaining 70 gallons of D.I. Water
- 14. Run 20 gallons of this solution to drain.
- 15. Re-circulate the remaining 50 gallons through the system for 1 hour.
- 16. Dump cleaning chemicals into NT-1.
- 17. Rinse empty tank thoroughly.
- 18. Rinse membranes with regular city water for 15 minutes.

#### **Rinse RO4 and RO5 Mix Beds**

- 10. Allow the plugged side of the y-tube to remain plugged.
- Fill water tank with 200 gallons of D.I. water and maintain temperature at 50 degrees Celsius.
- 12. Flush 100 gallons of D.I. water through the system to drain. NOTE: Low pressure (no higher than 60psi) and no more than 12gpm.
- Mix chemicals: RO Clean L403 (liquid); mix 1 gallon with 100 gallons of D.I. Water
- 14. Run 20 gallons of this solution to drain.
- 15. Re-circulate the remaining 80 gallons through the system for 1 hour.
- 16. Dump cleaning chemicals into NT-1.
- 17. Rinse empty tank thoroughly.
- 18. Rinse membranes with regular city water for 15 minutes.

#### **Repeat Steps**

Repeat all of the previous steps for the **new chemical**, **RO Clean P111**, with a ratio of 15lbs/tube set. (RO Clean P111 is available for purchase from Avista Technologies, Inc.)

Reconnect all **feed**, **concentrate**, and **permeate hoses** to there original configuration.

# 9. Appendix

1. Contact Information

For information regarding the cleaning the deionized water system contact:

Contact the current company supporting the water room

For information regarding the using the UF Skid contact:

Dave Wisnut: Ionics Engineer Cell Phone – 602 689 4295 Office Number – 602 437 1355

For information regarding the Ultrafiltration Modules contact:

Thomas Redding: Pall Corporation Engineer Office Number – 516 801 9124

Christian Besendorfer: Flow Solutions Sales Engineer Office Number – 602 68610

#### General information

#### http://www.hach.com

Free & Total Chlorine Test Strips, 0-10 mg/L, 50 tests **Product #:** 2745050 **US Price:** \$12.00 (used for general testing of chlorine levels before water goes into the RO unit)

Hydrogen Peroxide Test Kit, Model HYP-1, Drop Count Titration, 100 tests **Product #:** 2291700 **US Price:** \$52.10 (used to test the level of H2O2 after a major H2O2 flush)