University of Minnesota -- MN Nano Center
Standard Operating Procedure

Equipment Name: Critical Point Dryer

Badger Name:	P2 Etcher CP Dryer Tousimis	**Revision Number:**	6
Model:	915 B	**Revisionist**:	Lage von Dissen
Location:	PAN Bay 2	**Date:**	3/23/2020

1. Description

The Critical Point Dryer is capable of drying various size wafers (maximum of 6 inch wafer) down to 1 square cm pieces. Liquid carbon dioxide replaces the solvent used in the last rinse process. The pressure and heat are then increased to change the liquid carbon dioxide to a supercritical fluid, allowing it to behave like a liquid and a gas. The pressure is slowly decreased to atmosphere thereby not allowing carbon dioxide to condense. Circumventing the typical liquid-to-vapor phase transition allows for the minimizing of stiction (capillary adhesion) in MEMS/NEMS devices and micro-nano porous structures that have been submerged in an alcohol.

2. Safety

- **a.** Safety glasses are mandatory when using this system.

3. Restrictions/Requirements

- **a.** Must be a qualified user on CPD.
- **b.** **No acid, acetone, or water can go into the CPD**. Wafers need to be rinsed 3-4 times in water (if possible) then 3-4 times in Ethanol, Methanol, or IPA before loading into the CPD. **Acid, water, and acetone will DAMAGE the chamber**.
- **c.** DO NOT use vacuum grease in the CPD.
- **d.** DO NOT adjust the metering valves unless you've received special instructions.
- **e.** Complete log book and bar code in and out with each use.
- **f.** Clean up all rinsing dishes when finished.

4. Required Facilities

- **a.** 120 Volt Power
- **b.** LCO_2: 2 tanks

5. Definitions

- **a.** CPD: Critical Point Dryer
- **b.** LCO_2: Liquid Carbon Dioxide

6. Setup

a. Enable the "P2 Etcher CP Dryer Tousimis" system in Badger.

b. Go into the chase, behind the CPD system and check the weight of the CO_2 cylinders. The scales located above the gas cylinders will read "0 lbs." with a brand new cylinder, otherwise they will display a negative number which continues to decrease as more and more runs have been completed. If either of the scales read "- 40 lbs." or less (e.g. "- 45", etc.), do not use the system. Report a problem in Badger stating the weight displayed and that a cylinder replacement is needed.

c. **In the chase, verify that both CO_2 gas cylinder bottle valves are open.** The bottle valves are located at the top of each gas cylinder. If the valve is open, it should turn fairly easily. Toggle the valve a half-turn in either direction and then return it to its original position to verify that the valve turns easily. If it doesn't, then it is likely closed, in which case you should turn the valve counter-clockwise at least two full turns to open it. Go back into bay 2 when finished with this step, and then you can proceed to the next step.

d. Verify metering valve settings are set for the default values given below:

Cool: 0.55
Fill: 0.35
Vent: 0.26
Bleed: 0.15

Other values can be set if approved by an NFC staff member. Note: The metering valves need to be set back to the default values once any non-standard run has been completed.

e. Wafers need to be rinsed 3-4 times in water (if possible) then 3-4 times in Ethanol, Methanol, or IPA before loading into the CPD. Acid, water, and acetone will DAMAGE the chamber. The rinses will minimize the chance of any of the above getting into the CPD.

f. Turn power switch to "**ON**". The "**ON/OFF**" power switch is located on the lower right side of the system. The green LED on the **VENT** button will illuminate. This indicates that the power is **ON** and the machine is in the standby **VENT** mode. Let the machine stand for 3-5 minutes. This will allow all internally heated components to "warm-up".

7. Operating Instructions

a. Press the **VENT** button once. The **VENT** **LED** will begin to blink. This indicates that the **VENT** solenoid is closed.

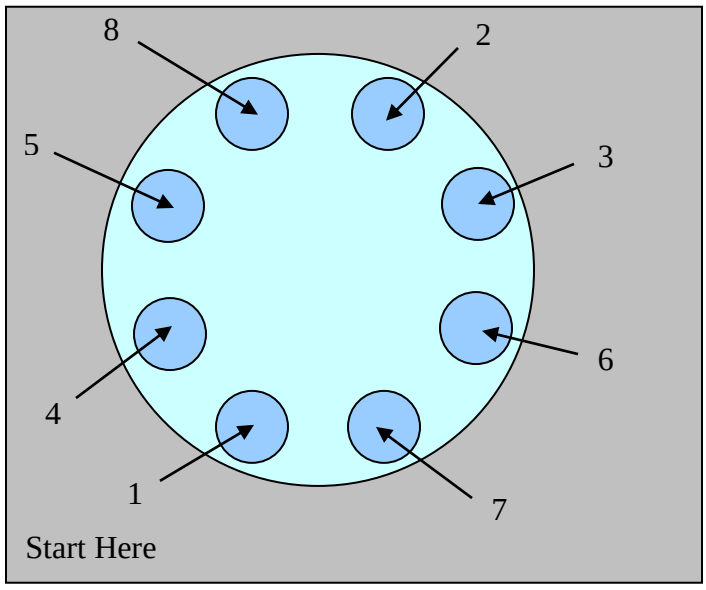
b. Loosen the knurl nuts on the top of the machine and remove the lid. Place the lid and knurl nuts on top of clean wipes next to the machine.

c. Put the correct number of space-filling inserts into the chamber that will minimize the movement of any wafers or pieces. Load sample into the correct size holder.

d. At this point, you can fill the chamber with enough alcohol (IPA, methanol, or ethanol) to cover your wafer(s) or pieces. **NEVER EXPOSE CHAMBER TO ANY ACIDS, ACETONE, or WATER!**

e. Carefully and quickly transfer your wafer(s) or pieces from your own container into the main chamber of the machine. For best results, minimize any exposure time to air.

f. Carefully place the chamber lid on top of the chamber. Use your hand to evenly tighten the 8-knurled nuts around the circumference of the chamber lid. Then, use the wrench and uniformly tighten each knurl nut in a "Star Pattern". Tighten no more than 15˚ at a time. Do this at least twice.



g. Once the chamber lid is secured, it is time to set the **PURGE** timer located to the right of the push button switches. Positions on the purge timer are calibrated at 5-minute intervals. Setting the "purge timer" indicator arrow to the #1 position will give you a 5-minute purge time, setting it to the #2 position will give you a 10-minute purge time, etc. The general rule of thumb for purging:

¼ chamber full of alcohol = 15 minute purge time
½ chamber = 20 minute purge time
¾ chamber = 25 minute purge time

h. After the initial warm up, press the **COOL** button. The **COOL LED** light will go on, and the **VENT** light will turn off. As the temperature slowly begins to drop, you will hear the LCO_2 circulating through the machine. The machine will continue to cool itself until the chamber temperature reaches 0-10^oC. Once this temperature is reached, proceed to the next step.

i. Press the **FILL** button and the machine will begin to fill the chamber with LCO_2. **From this point forward, the machine will automatically cycle through all the drying sequence steps until the process terminates.** During the **FILL** mode, the LCO_2 will enter the chamber for 8 minutes. You may hear the **COOL** cycle on / off during the **FILL** mode as the chamber temperature is automatically maintained between 0-10^oC.

j. Once all of the above mentioned cycles have been completed, the chamber will then vent itself. Once the **VENT** LED is illuminated, wait 15 minutes for the chamber to come to atmospheric pressure.

k. Loosen the knurl nuts in the reverse order from the way that they were tightened.

l. Remove the samples from the chamber.

m. Close the chamber lid and lightly tighten the knurl nuts with your hand.

n. Turn the power off by using the **ON/OFF** switch on the lower right side of the machine.

o. Complete the log book (if available). Empty out the condenser located to the right of the system. To do this, open the black-handled quarter-turn ball valve so that it is parallel with the outlet. Once all liquid has drained out from the condenser, close the black handled quarter-turn ball valve. Put all beakers used during the rinse process away. Leave the area clean and tidy.

p. Disable the "P2 Etcher CP Dryer Tousimis" from Badger.

8. Problems/Troubleshooting

a. If there is a leak during any time during the run, please contact a staff member. If it is coming out of the chamber lid seal, you may have not tightened the knurled nuts sufficiently prior to pressing the **FILL** button.

b. If there is liquid left in the chamber after your run, then the purge time likely needs to be increased, and you'll have to repeat your run.