

# Instruction for the Denton DCP-1 critical point dryer using CO<sub>2</sub>

Total time for this procedure is about 30–45 min.

## PPE and SOP

- PPE: safety glasses and nitrile gloves
- SOP: flammables

## load the prep

1. begin heating 3 L of water on a hot plate to use in the later step: **vaporize the CO<sub>2</sub>**
2. close both Inlet Valve and Exhaust Valve (clockwise)
3. load the chamber (make sure the specimens are completely submerged in ethanol all the time)
  - load specimens into specimen baskets (with cover)
  - pour enough ethanol in the chamber to cover the specimens
  - lower the basket into the chamber; use the lifter, if necessary
4. close the chamber



o-ring properly seated



yoke turned counterclockwise and properly engaged



support the chamber to avoid pushing it down while turning the Hand Knob

- lower the Top Closure Yoke below the chamber flange, make sure the o-ring sits flush on the flange, and turn the yoke **counterclockwise** to engage the flange
  - Warning:* If the yoke is turned **clockwise**, it may appear to hold but will be blown off at high pressure!
- turn the hand knob clockwise to seal the chamber

## CO<sub>2</sub>-ethanol exchange

1. fill the stainless steel beaker to ~1" from the top with ~2.5 L of tap water to maintain the chamber at 15–25 °C
2. carefully bring the beaker up under the chamber until you can swing the shelf into position to support the beaker
3. add more water to completely submerge the fittings of the inlet and outlet of the Exhaust Valve
4. open the CO<sub>2</sub> tank valve
5. open the Inlet Valve to introduce liquid CO<sub>2</sub> into the chamber and pressure gauge should register 800 to 900 psi

*Caution:* If the pressure is below 800 psi, you could run out of CO<sub>2</sub> during this session. Contact Facility staff to replace the tank.

6. put the waste ethanol container under the Exhaust tube
7. slightly open the Exhaust Valve to allow a small but even flow of CO<sub>2</sub> to flush the ethanol out of the chamber but keep the pressure at 800–900 psi in the chamber
8. continue to bleed off CO<sub>2</sub> for 5 to 10 min. to completely replace the ethanol, collect the exhaust periodically on brown paper towel until the CO<sub>2</sub> can sublime away completely leaving no traces of ethanol

## vaporize the CO<sub>2</sub>

1. close Exhaust Valve
2. close Inlet Valve
3. close CO<sub>2</sub> tank valve
4. remove the beaker and dump the water
5. fill the beaker with the water you have been heating and adjust it to 55 °C

*Note:* It is a good idea to keep some water heated to 60 °C on hand in case you need to further adjust the water temperature. You are going to lower the chamber pressure at elevated temperature (>45 °C) to "go around" the critical point of CO<sub>2</sub> at 1072 psi and 31 °C in the next few steps!

6. install the beaker to immerse the fittings of the Exhaust Valve; when the chamber warms up to ~45 °C the pressure will rise >1650 psi, passing over the critical point of CO<sub>2</sub>
7. open the Exhaust Valve slowly to vent the chamber at about 100 psi/min; make sure the water temperature do not drop below 45 °C

*Note:* It should take ~15 min. to completely vent the chamber and you need to adjust the valve continuously to maintain the flow.

8. open the Exhaust Valve completely
9. support the beaker, swing out the shelf, lower the beaker, empty and leave it over the sink to dry
10. dry the chamber and Top Closure Assembly carefully to avoid having water dripping back into the chamber as you open it, short bursts of air directed at the junctions will help
11. unscrew the Hand Knob on the Top Closure completely while supporting the chamber from below

12. rotate the yoke clockwise and lift it up to remove it from the chamber

*Note:* If it is difficult to get them apart, hold the chamber and use your thumb to push the top off.

13. check the top flange and inside wall of the chamber for water, wipe dry if necessary

*Caution:* Water on the top flange and tapered surface may be ok, otherwise it might have gotten into your sample!

14. remove the dried specimens and replace the Top Closure assembly on the chamber
15. clean up the work area and submit your usage using the google form

