

# 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

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## 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. check to make sure both o-rings for the Top Closure Yoke are in good condition, clean with ethanol as needed
3. check to make sure the chamber is clean, blow off any dust with compressed air
4. close both Inlet Valve and Exhaust Valve (clockwise)
5. load the chamber (make sure the specimens are completely submerged in ethanol all the time)
  - i. load specimens into specimen baskets (with cover)
  - ii. pour enough ethanol in the chamber to cover the specimens
  - iii. lower the basket into the chamber; the lifter can make loading and subsequent unloading easier
6. 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

- i. 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!

- ii. 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> valve on the tank with the tag "USE THIS TANK"
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, the tank is probably exhausted. Follow the **tank switch** procedure below.
6. put the waste ethanol container under the Exhaust tube
7. slightly open the Exhaust Valve to allow a gentle but even flow of CO<sub>2</sub> to flush the ethanol out of the chamber, keep the pressure at 800–900 psi
  - Caution:* You will see dry ice coming out of the exhaust, first as chunks and then in a fine stream when most of the ethanol is being replaced. If you do not see any dry ice, the tank is exhausted. Follow the **tank switch** procedure below.
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

### tank switch

- i. close Exhaust Valve
- ii. close Inlet Valve
- iii. close the CO<sub>2</sub> valve on the tank with the tag "USE THIS TANK FIRST"
- iv. open the CO<sub>2</sub> valve on the tank with the tag "USE THE OTHER TANK"
- v. open the Inlet Valve and continue with CO<sub>2</sub>-ethanol exchange
- vi. notify facility staff after the session

## 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 compressed 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
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



# Appendix

## CPD baskets

description	quantity	diameter (mm)	height (mm)	remarks
large metal mesh basket, no lid	1	24	59	chamber can hold 1
large metal mesh basket, no lid	2	23	17	
large metal screen basket with lid	3	23	17	2 removable 4-compartment partitions, chamber can hold 2
small metal screen basket with lid	2	14	9	2 removable 4-compartment partitions, chamber can hold 3
small plastic mesh basket with cap	>10	13	9	total height is 25 mm with cap, chamber can hold 2
small metal mesh basket with lid	1	12	21	
small metal mesh basket with screw cap	2	10	10	chamber can hold 3
modified BEEM capsule, cap with nylon screen	>5	7	8–14	DIY, size 00, chamber can hold 9
glass tube, cap with copper mesh grid	12	4	18	2 mm internal diameter