

Operation Procedure for CPD1

Bal-Tec CPD 030 Critical Point Dryer

Start Up

1. Sign-in to the logbook
2. Put on gloves to keep the system clean
3. Load samples into the sample holder, typically with methanol. If using the solid top on the sample holder do not tighten the top completely down so that any trapped gas can escape
4. Open the CO₂ gas cylinder. There should be ~800psi of pressure in the cylinder
5. Power on the CPD via the green switch
6. Power on the heat tape controller variac
7. Press the **Cooling** button and allow the system to cool to 10°C (this value can be checked by pressing the **Set Temp** button).

Running the system

8. Once cool, open the chamber and load the sample holder into the system making sure the notch is aligned over the side inlet hole on the left side of the chamber
9. Close and tighten the chamber lid
10. Press the **Medium In** button and allow the CO₂ liquid to fill the chamber to approximately 85% of the window height. This should give a pressure reading of 50-80bar on the pressure gauge.
11. Press **Medium In** again to stop the CO₂ fill
12. Let the chamber sit idle for 5 minutes
13. Press the **Medium Out** button to drain the CO₂ from the chamber. The needle valve on the back of the system may need to be adjusted. Allow the chamber to drain until the CO₂ is just ABOVE the sample surface
14. Press **Medium Out** to stop the drain
15. Press the **Medium In** button again and allow the CO₂ liquid to fill the chamber to approximately 85% of the window height. This should give a pressure reading of 50-80bar on the pressure gauge.
16. Press **Medium In** again to stop the CO₂ fill
17. Let the chamber sit idle for another 5 minutes
18. Press the **Medium Out** button to drain the CO₂ from the chamber. Allow the chamber to drain until the CO₂ is just ABOVE the sample surface
19. Press **Medium Out** to stop the drain
20. Press the **Medium In** button again and allow the CO₂ liquid to fill the chamber to approximately 85% of the window height for a 3rd time. This should give a pressure reading of 50-80bar on the pressure gauge
21. Press **Medium In** to stop the CO₂ fill
22. Press the **Cooling** button to stop the cooling
23. Press the **Heating** button to allow the chamber to start heating to 40°C (this value can be checked by pressing the **Set Temp** button). This will initiate the critical point heating of the CO₂. Reference the graph on page 8 of the system manual.
24. Close the CO₂ gas bottle
25. The chamber pressure should not exceed 100 bar

26. Watch for the critical change of the CO₂ in the chamber
27. Once the system reaches 40°C press the **Gas Out** button
28. Slightly open the **Metering Valve** until the gas flow on the flow meter reaches 160
29. As the flow in the flow meter drops continue to slightly open the **Metering Valve** more to keep the flow at around 160, while also keeping the temperature as close to 40°C as possible
30. Continue the adjustments of the **Metering Valve** until all the gas is out of the chamber (flow meter reads 0 and the valve is completely open)

Ending a Process and Shutting Down the System

31. Open the chamber and remove the sample holder
32. Remove samples from the holder as needed
33. Press **Gas Out** to close the gas out valve
34. Close the **Metering Valve** until it is just slightly hand tight (DO NOT OVER TIGHTEN)
35. Press the **Heating** button to shut off the heating
36. Power off the system via the green switch
37. **Change the main chamber o-ring**
38. Close the chamber
39. Power off the heat tape controller variac
40. Log out of the logbook